Influences on crystal structure due to hydrogen bonding and other effects

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Declaration

The work described in this thesis was carried out in the Department of Chemistry at the University of Durham between October 1997 and September 1998. This work has not been submitted, either complete, or in part, for a degree in this or any other University. All the work is my own unless stated to the contrary.

Charlotte Brooks

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Influences on Crystal Structure Due to Hydrogen Bonding and Other Effects

Abstract

One of the most fundamental pieces of information available about a compound is the molecular structure, and one of the best methods for obtaining structural information for a crystalline compound is to study the diffraction pattern obtained when the crystal interacts with an X-ray beam. The diffraction pattern can be analysed to give not only the molecular structure, but also information on the packing arrangements in the crystal, the 3-Dimensional crystal structure.

This information on the molecular structure and packing within the crystal allow the study of weak intra and intermolecular interactions such as hydrogen bonds. In this project two different methods will be used to study hydrogen bonds. Firstly using a statistical approach, where one type of intramolecular hydrogen bond (the N-H...π bond between neighbouring residues on a peptide chain) is analysed using crystallographic data obtained from the Cambridge Structural Database. Secondly the diffraction data from a several very different compounds are solved and the resulting structures analysed in terms of molecular and crystal structure; how and why the molecules pack as they do in the crystal lattice, and influences of hydrogen bonds on this packing.

The effect of changes in the molecular structure on the molecular packing and crystal structure is also considered by the analysis of diffraction data from several groups of compounds where there are small changes in molecular structure in going across the group. The effects considered are; the effect of changing a methyl group for a butyl group in the 1,3-dimethyl-2-imidazolidinone co-crystallised with methane-diphenol-3,5,3',5'-tetra^{t}butyl, the effect of changing the methyl group for an amino or ethyl group in triphenylmethylphosphonium co-crystallised with *p-tert*-butylcalix[4]arene and finally the effect of changing the metal in the compounds M-bis(2,6-di^{t}butyl-4-methylphenoxy) bis(triphenylaminophosphonium) and M-bis(trimethylsilylamide) bis(triphenylmethylphosphonium), where M is an alkali earth metal.

Introduction

The molecular structure of a compound is one of the most fundamental pieces of information available on the compound. After all, how else can the chemist be sure of what they have made? But molecules are too small to be looked at in the way we would look at everyday macroscopic or even microscopic objects. Even given the most powerful microscope in the world, an individual molecule, let alone an individual atom, would indistinguishable. This is because each atom is far smaller than the wavelength of the light being used to look at it. There are many techniques of deducing the structure of a compound but diffraction is the closest we can come to actually looking at an individual molecule.

Of course, more information is available from a diffraction pattern than the molecular structure. The 3-Dimensional structure and information on weak interactions such as hydrogen bonds can be analysed. Intramolecular hydrogen bonds can influence the shape the molecule takes, while intermolecular hydrogen bonds can influence not only the shape, but also the way the molecules interact and therefore the packing within the crystal. The packing within the crystal can influence/explain many of the macromolecular properties of the crystal, from morphology to magnetic and conductive properties.

In this project hydrogen bonds are studied using two different methods. Especial consideration will be given to the effect of the hydrogen bonds on the molecular and crystal structure. The first method uses a statistical approach is used to study one type of intramolecular hydrogen bonds using crystallographic data obtained from the Cambridge Structural Database. The second method is the solving of diffraction data from a several very different compounds, the resulting structures are analysed in terms of molecular and crystal structure; how and why the molecules pack as they do in the crystal lattice and influences of hydrogen bonds on this packing.

The analysis of diffraction data is also used to study the effect of changes in the molecular structure on the molecular packing and crystal structure. Several groups of compounds are considered where there are small changes in molecular structure in going across the group. The groups are: 1,3-dimethyl-2-imidazolidinone or 1,3-dibutyl-2-imidazolidinone co-crystallised with methane-diphenol-3,5,3',5'-tetra^{t}butyl where the effect of changing a methyl group for a butyl group is considered. Triphenylmethylphosphonium or

triphenylaminophosphonium or triphenylethylphosphonium co-crystallised with *p-tert*-butylcalix[4]arene where the effect of changing the methyl group for an amino or ethyl group in a large molecular system is considered. Finally the effect of changing the metal in the compounds M-bis(2,6-di^{t}butyl-4-methylphenoxy) bis(triphenylaminophosphonium) and M-bis(trimethylsilylamide) bis(triphenylmethylphosphonium), where M is an alkali earth metal, is considered.

Chapter 1

Hydrogen Bonding

Hydrogen bonds are one of, if not the most important interactions for ordering molecules in a crystal[1,2]. Indeed many compounds that will not crystallise under normal circumstances, will crystallise in the presence of a solvent that can form hydrogen bonds, and will crystallise in such a way as to make the solvent an integral part of the crystal.

The molecules in a crystal pack in such a way as to optimise the hydrogen bonding, and preferably to form hydrogen bond networks which run through out the entire crystal. Given this, it is may be possible to deduce the molecular packing of a given molecule from an analysis of the hydrogen sites, though this usually proves easier said than done. Working in the opposite direction, 'crystal engineering[3]'; the building of a given crystal structure by choosing molecules such that they will pack in the desired manner, is heavily dependent on a correct prediction of the hydrogen bonding patterns.

Prediction of the hydrogen bond patterns is difficult because of the wide variety of types of hydrogen bond[2,4,5]. Not only do the classical hydrogen bonds, O..H..O, N..H..N and O..H..N, need to be considered, but also the weaker O..H-C and N..H-C and even O-H..π, N-H..π, and C-H..π bonds, where π is an area of electron density due to the presence of a π-bond or an aromatic ring. Often it is more favourable for the molecule to orientate such that several of these weaker hydrogen bonds form instead of a single classical hydrogen bond.

What is a Hydrogen Bond?

Classically hydrogen is considered to have a valence of one, i.e. it can only form one bond. This logical since a hydrogen atom only has a single electron with which it can form only one normal covalent bond. However, sometimes a hydrogen atom is obviously involved in two bonds, the weaker of which is referred to as a hydrogen bond.

A-H..B

A hydrogen bond is an interaction that occurs between a polarisable bond containing a hydrogen atom, e.g. O-H, N-H, and an electronegative atom such as oxygen, nitrogen or a halogen. What is happening is that a covalent bond forms between the atom A and the hydrogen atom, this bond is made from the single electron from the hydrogen, and an electron

from atom A, and this electron pair is located between the hydrogen and atom A. If atom A is fairly electronegative then the electron pair is pulled closer to the atom A leaving the hydrogen nucleus 'exposed'. Any area of electron density occurring nearby, e.g. another electronegative atom which can donate electron density from a lone pair of electrons or a π-bond, will interact with the with the electropositive hydrogen nucleus[1,6].

$$\overset{\delta-}{A}-\overset{\delta+}{H}..\overset{\delta-}{B}$$

In cases where A = B, e.g. both atoms are oxygen, the hydrogen may be equally shared between the two atoms, with both bonds being the same length. These symmetric hydrogen bonds usually only occur in strongly hydrogen bound systems[7].

Hydrogen bonds are usually identified by analysing the interatomic distances, obtained from diffraction data. The best identification is an H..B distance that is much less than the sum of the Van der Waals radii[7]. Unfortunately unless the X-ray diffraction data are very good, the positions of the hydrogen atoms are only poorly defined, or not found at all but generated in the calculated positions. This is because X-rays are diffracted by electrons, and hydrogen only has one electron. So when identifying hydrogen bonds from X-ray diffraction data it is sometimes better to consider the A...B distance instead of the H..B distance. A and B are usually well defined since they have more electrons than hydrogen. In a hydrogen bond the A...B distance is notably less than the sum of the Van der Waals radii of the two atoms. The amount that the bond is shorter than the sum of the Van der Waals radii gives a measure of the strength of the hydrogen bond. A strong hydrogen bond, as defined by Emsley[7], is one where the A...B distance is more than 0.3Å shorter than the Van der Waals radii. Hydrogen bonds tend to be linear, but this is not always the case and is in no way enough to define a hydrogen bond.

In this report I will be studying hydrogen bonds in two different ways. First by using a statistical / theoretical approach to analyse the occurrence of one specific hydrogen bond (N-H..π in peptide chains) in the Cambridge Crystallographic Database[8]. Secondly by a detailed analysis of the hydrogen bonding patterns in several different crystals, studied by X-ray diffraction (see chapters 7 and 8).

Hydrogen bonding between amino and phenyl groups

Hydrogen bonds are often described as weak interactions but the strength of these 'weak interactions' covers a wide range, depending on the strength of the proton donor and proton acceptor. The hydrogen bond studied in this chapter is one of the weaker of the acknowledged hydrogen bonds, the bond between the donor N-H, and the acceptor phenyl ring.

The existence of a hydrogen bond between an N-H donor and a benzene ring acceptor is fairly well recognised and defined[9,10]. There has been a lot of recent study of this type of bond[5,9,10]. Studies of the interaction between gas phase ammonia and benzene[10] show it to be a weak bond with a bond energy of approximately 2 to 4 Kcal mol^{-1} and an N...ring plane distance of 3.6Å. Though weak, a bond strength of 2 to 4 Kcal mol^{-1} is still strong enough to be biologically significant. Geometrically the bond is very soft, that is, the N-H bond can be at a wide number of angles to the ring plane not only the expected normal (90°) angle, with very little change in energy[11].

It has been suggested that hydrogen bonds of this type could help to form the secondary structure[12,13,14] (α helixes and β folds etc.) of proteins containing Phenylalanine, Tyrosine or Tryptophan (the amino acid groups that contain a phenyl or phenol ring). There have been various studies of the effect of this type of hydrogen bond between residues at different spacings along the peptide chain.

A study by Armstrong *et al*[12] showed that the interaction between an amino group on a histidine (not the chain backbone) and a phenyl ring four residues along (i.e. an interaction between residues i and i+4) helps stabilise an α helix.

A study by Worth and Wade[13] of the interaction between an amino group on the chain backbone and a ring on the i+2 residue shows it to be important not as a driving force for folding but as a correction. Thus allowing otherwise unsatisfactory hydrogen bond donor capacity to partially fulfilled, i.e. when the peptide is on the end of a β sheet.

A study by T. Steiner[14] suggests that the interaction between an amino group on the chain backbone and a ring on the i+1 residue may be important in stabilising the structure when there are no stronger interactions possible. The geometry of this system is such that it has to be a very weak distorted bond, infact it is so weak and distorted that it is hard to decide is it is a bond at all or if it is simply a non-bonded forced contact.

It is the last of these interactions that will be considered here. By looking at the distance between the amino group on the chain backbone and the phenyl ring, where the

relation between the ring and the N-H is i+1, for all cases where such a relationship occurs in the Cambridge Structural Database[8], I will attempt to find the frequency of occurrence of such an interaction.

Cambridge Structural Database[8] Search:

Method:

The search was carried out in the main structural database not the protein database. This was because although the protein database would be the more obvious starting point, the data held on it is of a lower resolution, and rarely, if ever, is the structure detailed enough to allow analysis of hydrogen bonds. The reason behind the low resolution is the large size of the protein molecules. The main database holds information on many smaller peptide chains and is designed to allow detailed searched for a specified geometry or interaction.

Since the interaction of interest is between the ith and i+1 peptide residues, the search fragment was two peptide residues where one of the residues containing a phenyl ring:

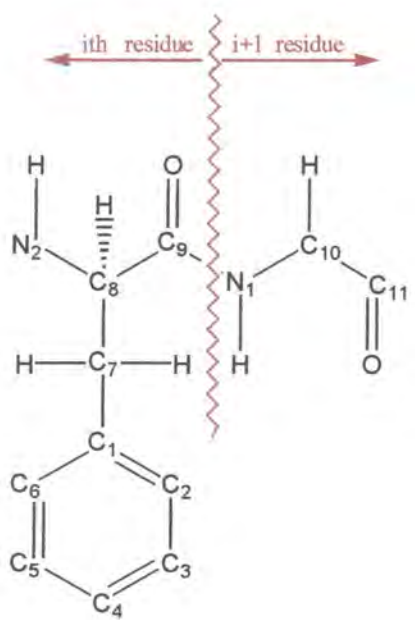


Diagram 1.1: the search fragment.

Using the '3-D constraints' menu various parameters (distances and angles) were defined and recorded for each result found. These parameters were:

NAME	DISCRIPTION
DISCEN	Distance between centre of ring on residue i and N of N-H on residue i+1.
DISCEH	Distance between centre of ring on residue i and H of N-H on residue i+1.
DISC1N	Distance between C_1 on ring on residue i and N of N-H on residue i+1.
DISC1H	Distance between C_1 on ring on residue i and H of N-H on residue i+1.
TORNCC	Torsion angle about the C_9 C_8 bond
TORCCC	Torsion angle about the C_8 C_7 bond

The two torsion angles can be said to define the N_1 - C_1 distance, since rotation about the C_9 - N_1 bond only influences the position of the hydrogen atom of the N-H group, while rotation about the C_7-C_1 bond only influences the ring orientation. Since the ring centre lies on a continuation of the C_7-C_1 bond vector the ring centre position is not influenced by the rotation about the C_7-C_1 bond, and therefore the two torsion angles can be said to define the N - ring centre distance as well.

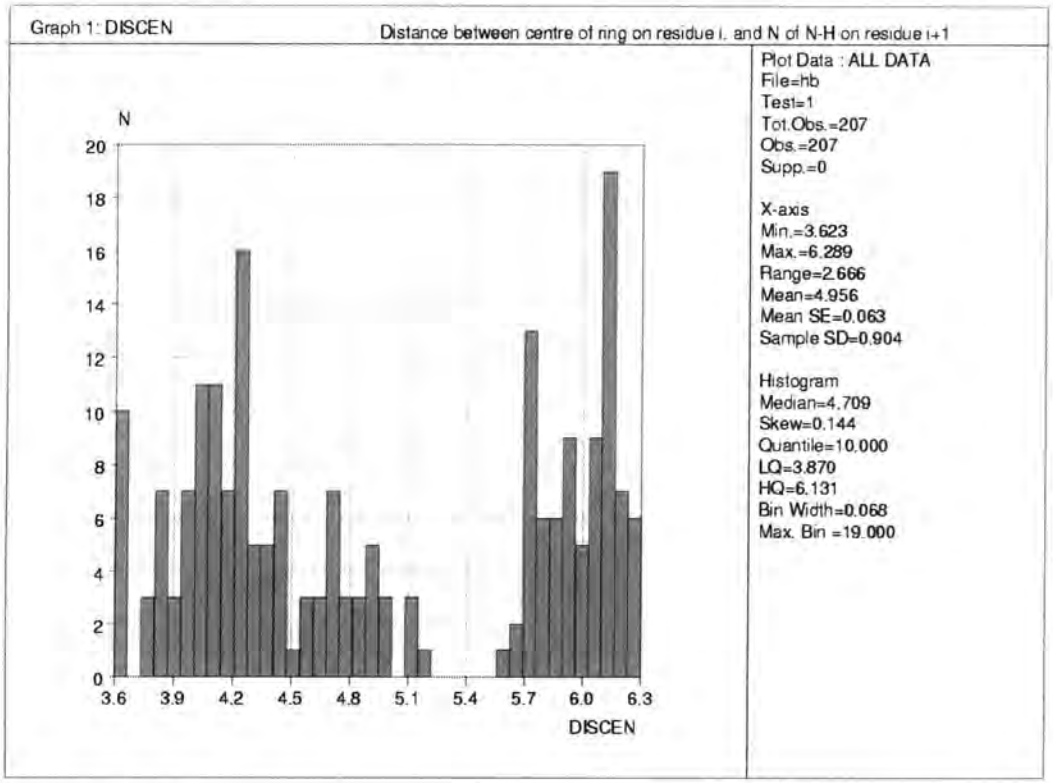
The search was carried out for organic molecules only.

The data was analysed using the VISTA program. Histogram plots or scattergrams were generated, as appropriate, for all the parameters measured.

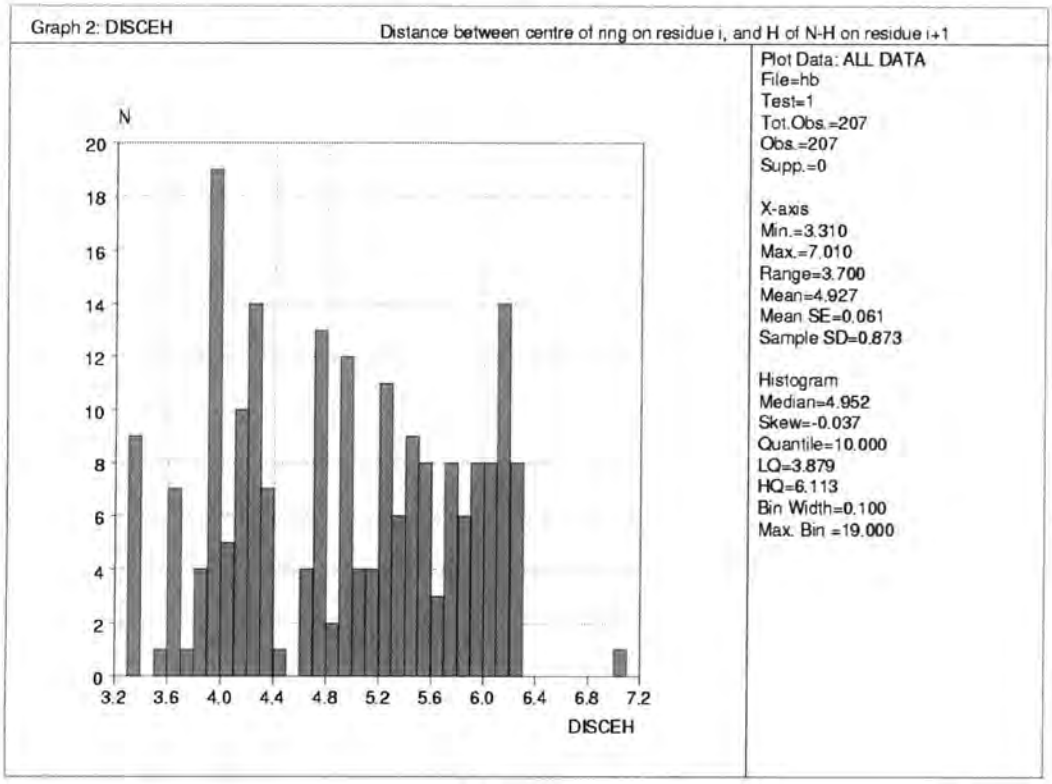
Results:

Number of occurrences of the search fragment: 207

Number of different molecules containing search fragment at least once: 82



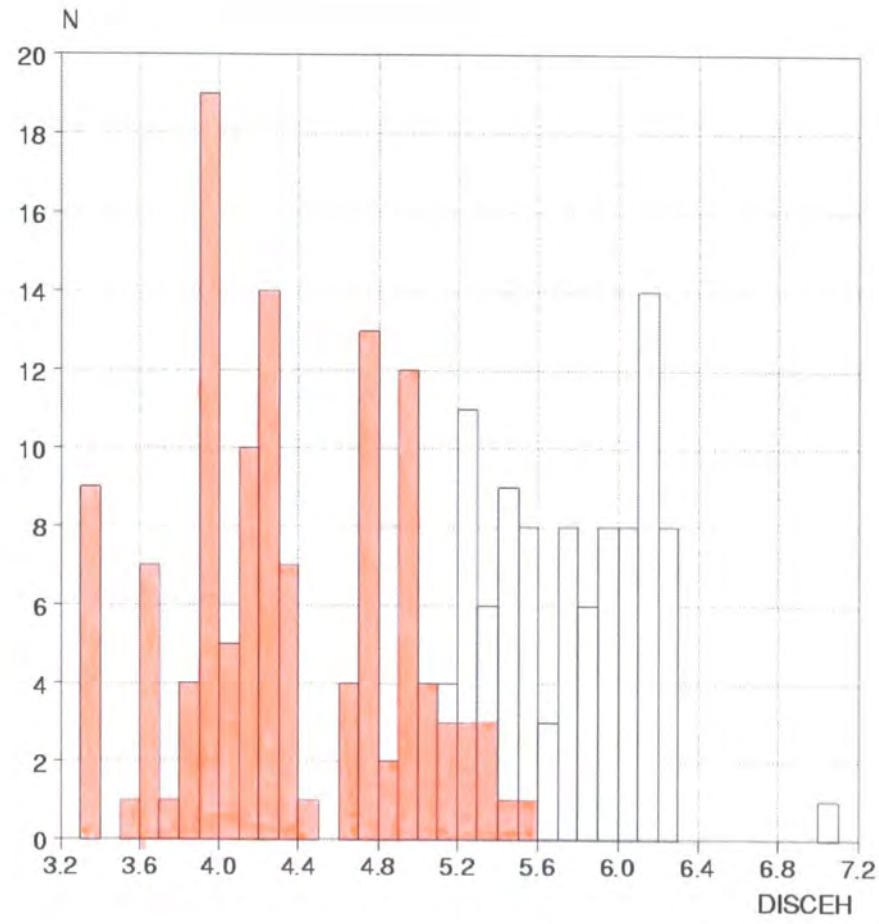
It can be seen that the bond lengths separate into two distinct groups or clusters, one cluster has bond lengths that range between 3.60 Å and 5.22Å the other cluster has longer bond lengths ranging between 5.62Å and 6.30Å. 3.6Å is the N_1 – ring plane distance for the ideal case of gas phase ammonia - benzene system[10], but 5.22Å is far longer then any acknowledged hydrogen bond.



At first sight this graph does not show the nice separation seen in graph 1, but if the part of the graph corresponding to the shorter bond length cluster only is highlighted then the graph clearly separates into two clusters again, only now the clusters overlap (see graph 3).

Graph 3: DISCEH

Distance between centre of ring on residue i, and H of N-H on residue i+1



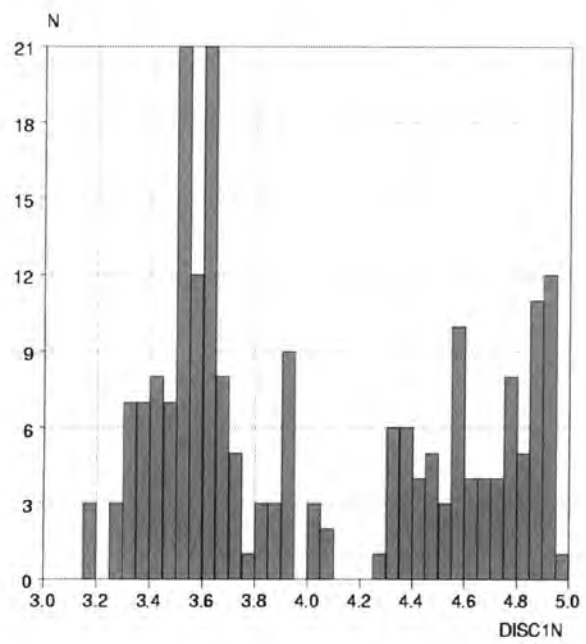
Plot Data: ALL DATA
File=hb
Test=1
Tot.Obs.=207
Obs.=207
Supp.=0

X-axis
Min.=3.310
Max.=7.010
Range=3.700
Mean=4.927
Mean SE=0.061
Sample SD=0.873

Histogram
Median=4.952
Skew=-0.037
Quantile=10.000
LQ=3.879
HQ=6.113
Bin Width=0.100
Max. Bin =19.000

data corrisponding to shorter bond length cluster only.

Graph 4: DISC1N — Distance between C1 of ring on residue i and N of N-H on residue i+1



Plot Data: ALL DATA
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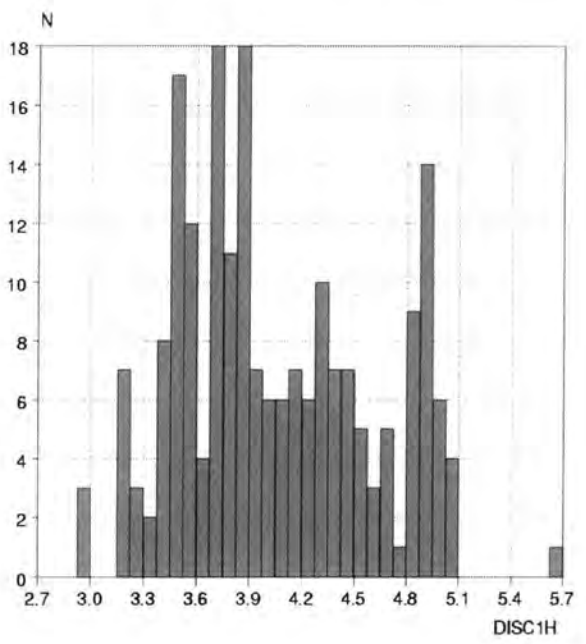
Test=1
Tot.Obs.=207
Obs.=207
Supp.=0

X-axis
Min.=3.176
Max.=4.978
Range=1.802
Mean=4.025
Mean SE=0.039
Sample SD=0.566

Histogram
Median=3.830
Skew=0.344
Quantile=10.000
LQ=3.385
HQ=4.863
Bin Width=0.050
Max. Bin =21.000

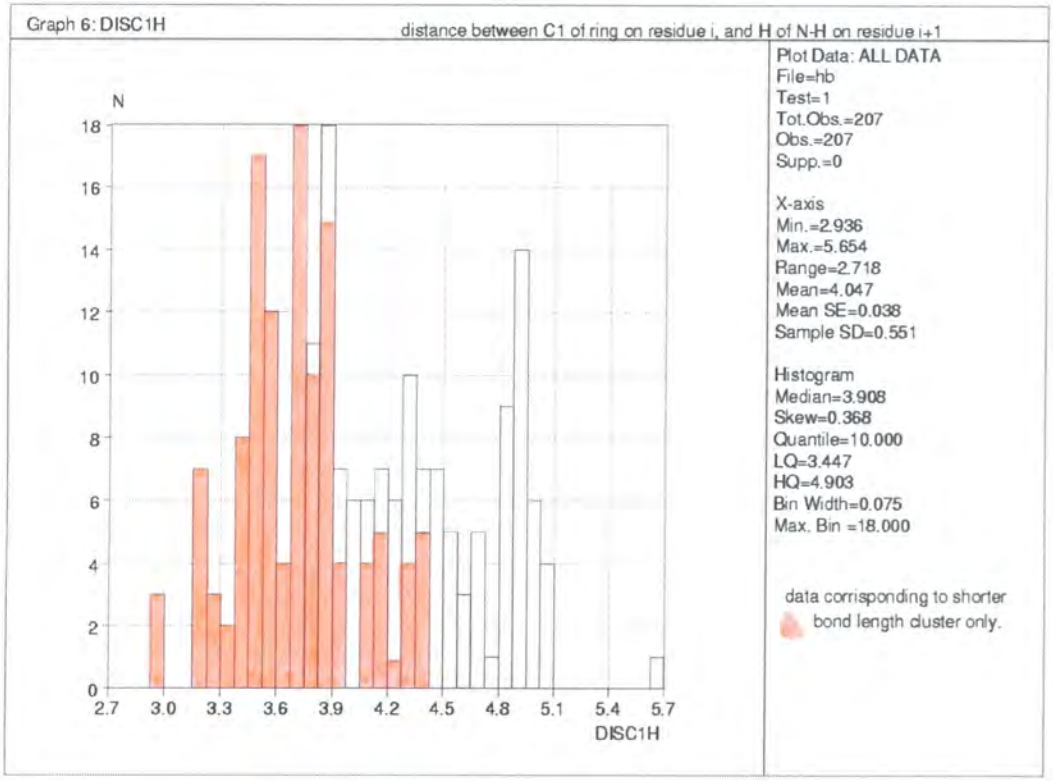
Graph 5: DISC1H — Distance between C1 of ring on residue i, and H of N-H of residue i+1



Plot Data: ALL DATA
File=hb
Test=1
Tot.Obs.=207
Obs.=207
Supp.=0

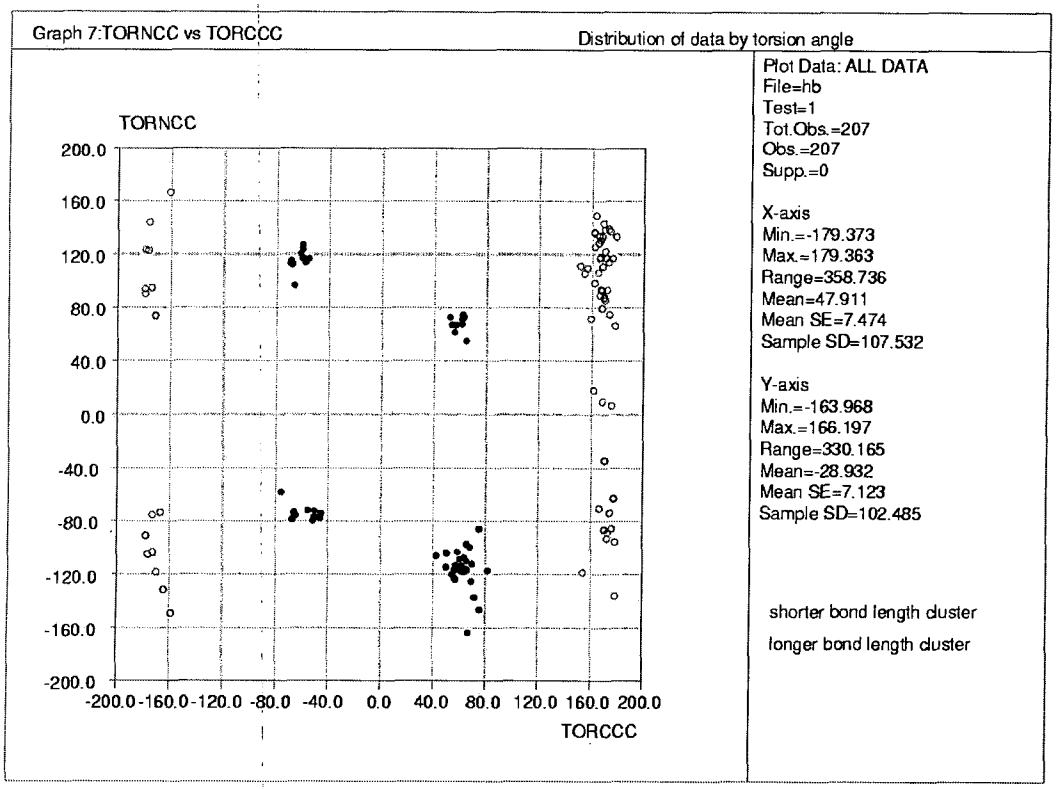
X-axis
Min.=2.936
Max.=5.654
Range=2.718
Mean=4.047
Mean SE=0.038
Sample SD=0.551

Histogram
Median=3.908
Skew=0.368
Quantile=10.000
LQ=3.447
HQ=4.903
Bin Width=0.075
Max. Bin =18.000



The data for the distance between C_1 of the ring on residue i and the amine group on residue i+1, shows similar distributions to that seen for the corresponding data for the ring centre. Once again, the data corresponding to the H of the amine group on residue i+1 shows, at first sight, no obvious separation into the two distinct clusters, as was seen for the data corresponding to the N_1 of the amine group on residue i+1. That is, no obvious separation is seen until the part of the graph corresponding to the shorter bond length cluster is highlighted, then the graph clearly separates into two overlapping clusters again.

The distances tend to be shorter in the C_1 - amine data, than in the ring centre – amine data, this is due to the steric restraints imposed by the short length of chain between the two groups.



The results are clearly grouped in terms of torsion angles, this is logical if it is accepted that these two torsion angles can be considered to define the $N_1 \ldots C_1$ distance (see method).

The data corresponding to the longer bond length cluster appears at first to be grouped in three distinct areas. However, when it is remembered that it is angles that are being analysed here, and therefore -180° ≡ +180°, it is seen that the data actually lies in one group centred around the +180°/-180° value for TORCCC, and with any value from –160° to +160° for TORNCC.

The data corresponding to the shorter bond length cluster is grouped in four distinct areas. One with both torsion angles at –50° to -60°, one with both torsion angles at +50° to +60°, one with TORCCC at +40° to +60° and TORNCC centred on -120°, and the final cluster with TORCCC centred on +120° and TORNCC at –40° to -60°

Conclusions:

The most interesting result from the search is the distinct clustering of the distances into two ranges. The cluster with the longer bond length is far too long to exhibit any intramolecular interaction. This cluster has fewer data items and a TORCCC torsion angle ≈180°. If TORCCC is at 180° then the N_1-C_1 (and the N_1-ring centre) distance lies in a narrow range of values dependent on rotation about TORNCC, but such that all the distances are at the long distance end of all possible N_1-C_1 (and the N_1-ring centre) distances for this fragment.

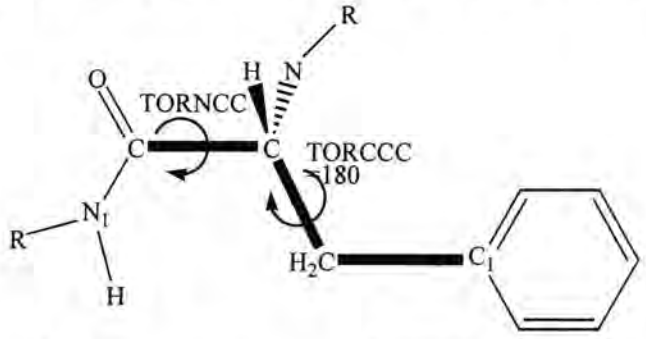


Diagram 1.2:peptide fragment with torsion angle TORCCC =180°

When TORCCC = 180° there does not seem to be a great distinction between TORNCC angle nearly the full range of angles from 180° to – 180° are seen though there is a slight preference for the 80° to 160° and –80° to –160° range.

The cluster with shorter bond length range exhibits bond lengths from those equal to that seen in the gas phase ammonia – benzene case, i.e. unmistakably a hydrogen bond, to longer than could ever be justifiably called a hydrogen bond. This is a problem that often occurs in the identification of hydrogen bonds: there is no definite cut of point above which the hydrogen bond is said not to exist and below which it is said to exist. The interaction just gets weaker and weaker until it is of negligible influence. It is interesting to note the skewed nature of this section of the graph, instead of a symmetric bell shaped graph that might have been expected the data are skewed towards the shorter bond lengths.

The data for the short bond length cluster are all grouped at torsion angle combinations of ±60° and ±120°, these are the staggered conformations, which, in general, will usually coincide with the local energy minima for any molecule. The combinations seen are:

TORCCC /°	TORNCCC /°
60	60
-60	-60
60	-120
-60	120

There are however twelve combinations of these staggered conformations that are not seen these are the four other sign combinations of the angular configurations that are found and the eight combinations with TORCCC = ±120°.

Using the CAche computer-modelling program, the $N_1 - C_1$ distances and energies were calculated for all these combinations, using the standard molecular mechanics MM2 procedure, and using a short peptide chain consisting of two peptide residues as a model:

Diagram 1.3: model for the CAche computer-modelling calculations.

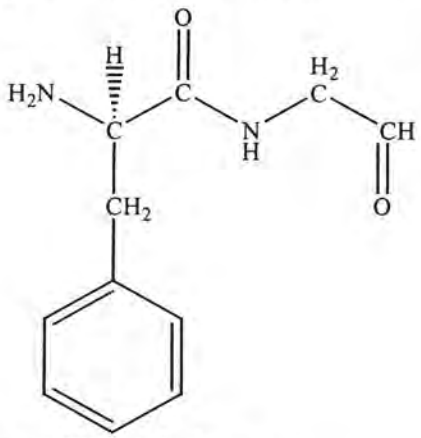
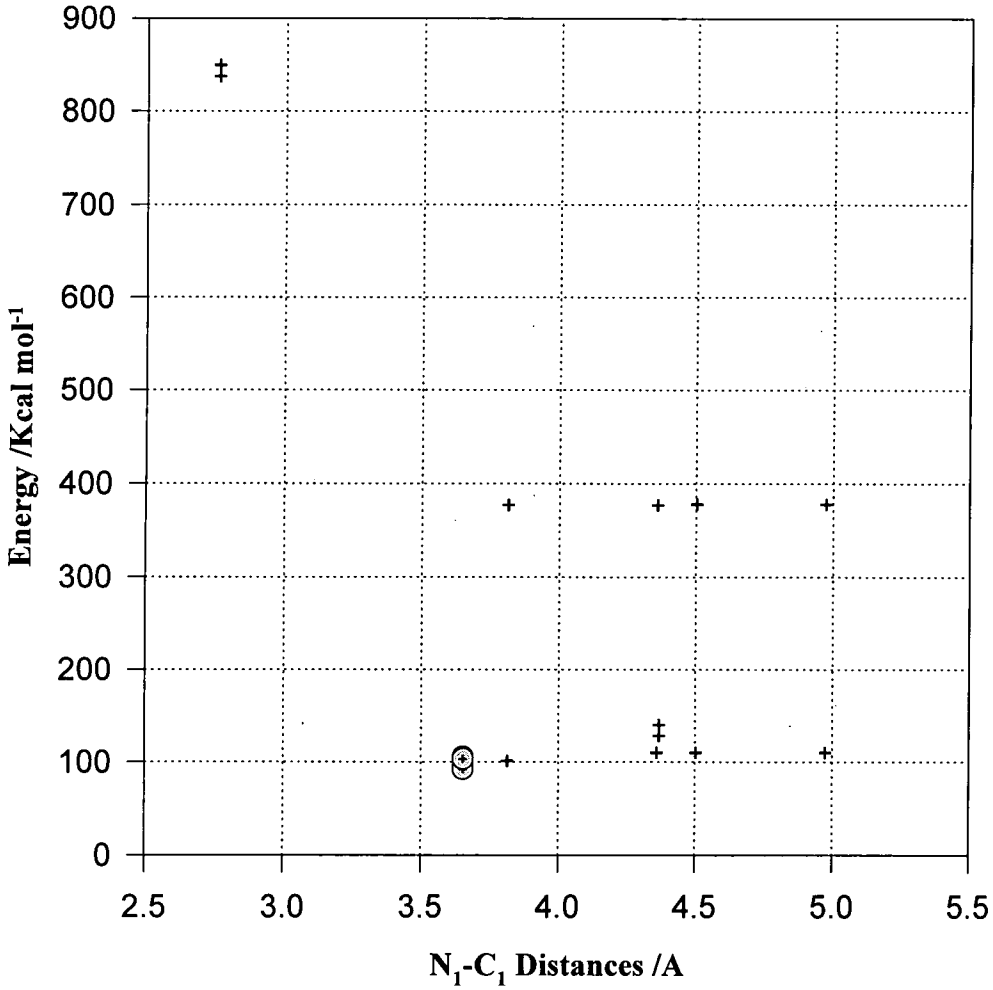


Table 1.1: results from CAche computer-modelling calculations.

TORCCC /°	TORNCC /°	DISTANCE/Å (CALCULATED)	ENERGY/Kcal mol^{-1} (CALCULATED)
60	60	3.653	94
-60	-60	3.653	105
60	-120	3.653	92
-60	120	3.653	103
60	-60	2.759	837
-60	60	2.759	849
60	120	4.368	128
-60	-120	4.368	140
120	120	4.974	110
-120	-120	4.974	378
120	120	4.360	110
-120	-120	4.360	377
120	60	4.503	110
-120	-60	4.503	378
120	-60	3.814	101
-120	60	3.814	377

Graph 8:

Results from CAche computer-modelling calculations



The combinations of angles that are seen in the data base search results are shown as shaded in grey.

It can be seem that the combinations of angle that are seen in the data base search results all correspond to the shorter distances, the only exception to this is for the 60, –60 and –60, 60 combinations, which although they have a shorter distance, have a much higher energy. There are combinations which seem to have a similar energy to the combinations that are seen and should be equally likely to be found, except that the $N_1 - C_1$ distance is far longer, so no hydrogen bond interaction could occur.

Of course this is not the only explanation of the torsional angular dependence, and further work is required in this area, especially with reference to the angle between the N-H bond and the phenol ring, and with comparison to more detailed chemical modelling studies. But I feel that these results suggest that there is, at least, some kind of interaction occurring.

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Chapter 2

X-ray crystallography

The technique

X-ray crystallography is a technique for the study of molecular structure, on an atomic scale, of phases of matter that exhibit long range order; i.e. crystalline solids. The X-ray beam is directed onto a crystal, and is diffracted by the atoms which comprise the regular arrangement of molecules in the lattice. Diffraction occurs by the X-rays being scattered by electrons, this often means that hydrogen atoms are not distinguishable since they have only one electrons to scatter the X-ray beam, and that electron is involved in bonding, not located at the hydrogen nucleus.

Diffraction patterns

The definition of diffraction is the interaction of electromagnetic radiation with an object in space. When a beam of radiation is passed through a grating where the size of the holes is of the same order of magnitude as the wavelength the beam is split. The difference in path length of the parts of the beam from the different holes in the grating to a point further on in space lead to constructive and destructive interference, which in turn leads to a pattern of peaks of intensity – the diffraction pattern.

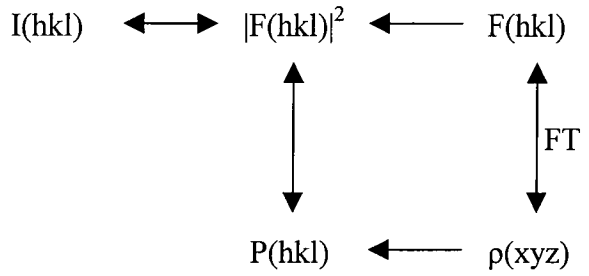
From an observed diffraction pattern it is possible to calculate information on the grating that caused it. The regular arrangement of atoms in a crystal is, in effect, a 3-Dimensional diffraction grating. The atomic spacing i.e. the size of the holes in the grating is of the same order of magnitude as the wavelength of X-rays, this is why X-ray radiation is used to study the diffraction patterns of crystals.

The position of the atoms cause a phase shift which effects the intensity of the diffraction pattern peaks and, therefore, the intensity of the peaks. This gives information on the positions of the atoms within the unit cell.

This all may sound fairly straightforward. However, while the pattern of the diffraction peaks is easily to obtained by recording the position of each peak and analysing the

data, which can be done, for example, on a P.C. attached to the diffractometer, the intensity data pose more of a problem.

The measured intensity I(hkl) can be converted to $|F(hkl)|^2$ (the relative structure factor). F(hkl) can be converted to the ρ(xyz) (electron density – real space), but of course although F(hkl) can be converted to $|F(hkl)|^2$, $|F(hkl)|^2$ cannot be converted to F(hkl) since $\sqrt{(F(hkl)^2)} = +F$ or $-F$. This is the phase problem.



This is why once the initial trial structure is arrived at, the structure is solved by iterative refinement. The procedure is to take an initial guess at the electron density, convert ρ(xyz) to F(hkl) by fourier transform procedures convert F(hkl) to $|F(hkl)|^2$ and compare to the experimentally derived $|F(hkl)|^2$'s, adapt the model and re-compare the data to see if the fit is improved. If model is improved, adapt the new model and re-compare. If the model is not improved then discard the new model and adapt the original model in some other way.

The initial trial structure is found either from the Patterson function which can be calculated direct from $|F(hkl)|^2$, or more often from direct methods. To describe either of these methods in any detail would take more space than is available here but put very very simply the Patterson function method involves the calculation of the vector relationship between atoms, since these interatomic vectors can be calculated from $|F(hkl)|^2$ the phase problem is not encountered, this method works very well for crystals containing a few heavy atoms. The positions of the heavy atoms are found using the Patterson function, then the lighter atoms identified by an iterative process. Direct methods is where possible phase combinations are tried, given certain assumptions (such as the peaks of electron density must be discrete and the electron density cannot be negative) most of the phase combinations can be discarded and the resulting solution can be used to start modelling the actual structure.

This is a very much simplified description of a couple of the principles behind X-ray crystallography. There is neither the time nor the space for a full explanation here. For a more complete description see ref. 1.

The initial trial structure was determined using the SHELX-86 (Sheldrick, 1990) program,[2] and the refinement carried out using the SHELX 93 (Sheldrick, 1993) program,[3] The molecular graphics were generated using SHELX-PLUS (Sheldrick, 1991).[4]

Through out this year I have been learning both the extents and the limitations of X-ray crystallography as an experimental technique. It is very important to remember that the final structural solution is still only a model. The accuracy of the model is given by the R1 and wR2 values, but it is always important to think about how these values are calculated. For a compound where most of the X-ray diffraction is arising from a heavy metal atom, which is well defined in the model, good R1 and wR2 values may be obtained despite the fact the lighter atoms are only poorly defined. The use of restraints and constraints can help to force the model to take a more chemically and physically logical form, usually with very little effect on the accuracy of fit to the experimental data. However care must always be taken not to force the model to take some preconceived form that is not accounted for in the data.

The equipment: The diffractometers

The diffractometer directs an X-ray beam at the crystal and records the position of the intensity peaks. The RIGAKU 4 circle diffractometer[5] measures each diffraction peak individually. The crystal can be rotated to almost* any given orientation by movement of three different angles;

ϕ - rotates the crystal on the goniometer head,

χ - rotates the goniometer head about a vertical circle,

ω - rotates the χ circle with relation to the X-ray beam,

which together with 2θ, the circle that carries the detector, give the four circles of the machine's name. This means that any diffraction peak can be brought to lie on the detector and recorded. At the start of the experiment the detector scans through a zig-zag pattern and records the first ten peaks found. The recorded angles of these peaks are used to determine the unit cell, this is then checked against the next ten peaks. This information is enough to allow the matrix and the positions of the rest of the peaks to be determined and the crystal and detector can be driven to the correct orientation to allow individual measurement of each peak in the main data collection.

The SMART-CCD (Siemens SMART Version 4.050 (Siemens Analytical X-ray Instruments, 1995))[6] works on the same principle but instead of measuring each peak separately the SMART has an 'area detector' which is effectively a sophisticated electronic substitute for the photographic paper used in the original diffraction experiments! The positions and intensities of a large number of peaks can be measured simultaneously, greatly reducing the data collection time.

Unless otherwise stated all the experiments in this report used a fairly standard set up. For the RIGAKU 4 circle diffractometer[5] the standard set up was using copper X-ray radiation (λ=1.54178Å), and scanning in 2θ-ω, with a scan rate on 8 or 16 °min^{-1} depending on the crystal diffraction quality. For the SMART-CCD[6] the standard set up was using molybdenum X-ray radiation (λ=0.71073Å), scanning in ω, with a scan width of 0.3°, and a scan rate of 10 or 20 sec/frame depending on the crystal diffraction quality.

The Cryostream unit:

The cryostream unit is used to cool the crystal. This often leads to improved results since when the atoms are vibrating less, the atomic positions can be better defined. The cooling system on both diffractometers was the Oxford Cryosystem Cold N_2 gas cooler.

Setting up

The crystals were either mounted on a glass fibre using 'Araldite' epoxy resin, or covered in oil (perfloropolyether M.W.2700) and picked up on the end of the glass fibre in a drop of oil which was then frozen to hold the crystal in place.

A suitable crystal was chosen; a suitable crystal is one that is a single crystal[†], not twinned. This can be checked by rotating the crystal while viewing, using a microscope, under two, crossed, polarising filters; the crystal should undergo sharp uniform extinction.

The X-ray beam used was approximately 0.8mm in diameter on both machines. The crystal had to be in the very centre of the beam where the X-ray intensity was approximately uniform, therefore a crystal of less than 0.5 mm in any dimension was used. If the crystal was too big, it was cut down to size using a very sharp razor blade. If possible the crystal should

---

[*] Some angles are physically unobtainable due to factors such as the path of the X-rays being blocked by the goniometer head or by the χ- circle etc.

[†] The refinement program used can model a twinned crystal, but it is preferable to use a single crystal in the first place.

be approximately equal size in all directions, since the X-ray beam undergoes absorption by the crystal, which depends on the path length in the crystal. This absorption reduces the intensities recorded such that:[1]

$$I = I_0 e^{-\mu t}$$

where: I = intensity of the X-rays
I_0 = intensity of incident beam
μ = linear absorption coefficient dependent on the crystal
t = path length of beam through crystal

This error can be modelled and the data adjusted to correct for the effects of absorption.

The crystal must stay stationary in the centre on the beam as it is rotated by the diffractometer. This was achieved by selecting a crystal of approximately equal size in all directions, and with a regular shape (this depends on the crystal morphology of the sample), and positioning the crystal carefully at the centre of all the circles, so that the crystal does not appear to move when any of the axes are rotated.

References:

1) a) Crystal Structure Analysis, A Primer. J. P. Glusker, K. N. Trueblood. New York, Oxford. Oxford University Press. 1985.

b) Crystal Structure Analysis for Chemists and Biologists. J. P. Glusker, M. Lewis, M. Rossi. VCH Publishers. New York, Wienheim, Cambridge. 1994.

2) G. M. Sheldrick, *Acta. Crystallogr.* 1990 **A46** 467

3) G.M. Sheldrick, (1993). SHELX 93. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.

4) G. M. Sheldrick, (1993). SHELXTL-Plus. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, U.S.A.

5) a) MSC/AFC. Molecular Structure Corporation (1991).

b) Molecular Structure Corporation (1991). MSC/AFC Diffractometer Control Software. (1991). MSC, 3200 Research Forest Drive, Woodlands, TX77381, U.S.A.

6) Smart Version 4.050. Seimens Analytical X-ray Instruments, Madison, U.S.A., 1995.

Chapter 3

Crystal Growth

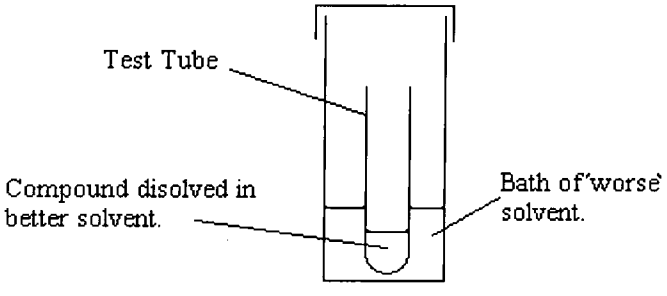
There are four main methods of crystal growth, slow evaporation of solvent, cooling of a supersaturated solution, growth from a fluid phase, and gel methods.

Slow Evaporation of Solvent[1,2]:

A concentrated solution of compound in the solvent of choice was made up using clean, dust free glassware. If necessary the solution is filtered to remove any undissolved residue and/or dust specs. The top of the test tube containing the solution was covered with a piece of parafilm with a few holes pricked in it, these holes allowed the slow evaporation of the solvent while reducing the introduction of dust.

The evaporation of the solvent increases the concentration of the solution to the point of nucleation and crystal growth. The removal of any dust and residue decreases the number of nucleation sites promoting the growth of a few large single crystals, instead of a lot of small intergrowing crystals. The choice of solvent is important[1,3], polar solvents promote hydrogen bonding linkages between the solvent and the compound while non-polar compounds promote hydrogen bonding linkages between and within molecules of the compound. The optimum solvent is usually found by a process of trial and error, but consideration of polarity and hydrogen bonding sites can reduce the range of solvents tried.

Other variations on this method are to use a mixture of solvents usually one in which the compound is soluble, and another in which it is not, so the solubility is decreased over time by the slow introduction of the solvent in which the compound is less soluble. There are various methods for the slow introduction of second solvent. One very effective way is to place a test tube containing the compound dissolved in the 'better' solvent (the one it does dissolve in) in a bath of the 'worse' solvent (the one it is not, or is only sparing soluble in); the solvents slowly mix by vapour diffusion. (see diagram below)

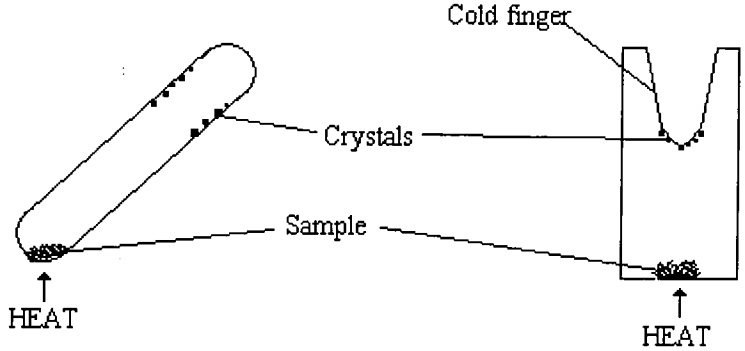


Cooling of a Supersaturated Solution[1,2]:

This method works on the principle that the solubility of almost all compounds decreases as the temperature drops. So starting from a saturated or preferably supersaturated solution, the temperature is slowly decreased. This has the effect of lowering the solubility to the point where crystal growth occurs. The decrease in temperature is achieved by cooling the sample to room temperature (if made as a hot solution), then moving sample to a cold room (16-14°C), then to that of a fridge (4°C), to that of a freezer (≈ -20°C). Again the use of clean, dust free glassware is important to reduce the number of nucleation sites, and the choice of solvent is important in controlling the hydrogen bonding.

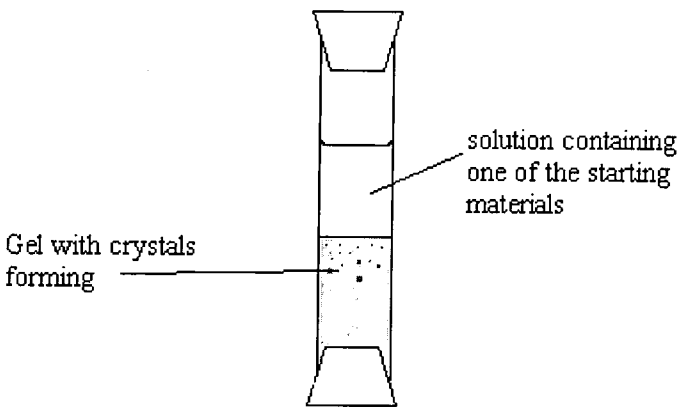
Growth from a Fluid Phase[1,2]:

Cooling of a vapour or liquid to below the melting point of the compound can be a very effective way of growing crystals, if the compound is thermally stable enough to be heated above its melting or boiling point. For compounds that sublime, growth from a vapour is often the best method of crystallisation. The compound is heated in one part of the apparatus (usually a sealed glass tube) the vapour diffuses to a cooler part of the apparatus where it crystallises out. Growth from a liquid is carried out simply by very slowly cooling the pure molten compound until crystals, or preferably a single crystal grows. A temperature gradient across the molten sample helps to promote growth. Obviously for growth from a vapour and, especially, from a liquid a large amount of sample is required.



Gel Methods[4]:

This can be a very good way of growing crystals, especially crystals of compounds that form very fragile crystals; the gel acts as a support for the growing crystal. However unlike the previous methods this technique is not a method of recrystallisation but depends on the reaction of two solutions to give a third insoluble, or at least, much less soluble compound. Both of the starting materials must be water soluble or at least soluble in ethanol. There are various methods of gel crystallisation, one of the simplest is the two component system: a gel is made (usually a silica gel e.g. tetramethyloxysilane gel) with one of the starting materials dissolved in it, a solution containing the second starting material is placed on top of the gel. The second starting material slowly diffuses into the gel and reacts with the first starting material forming the product, the slow rate of diffusion into the gel means that the product is formed slowly enough to form perfect crystals. The crystals grow within channels or fissures within the gel, so the gel supports them without actually interacting or reacting. This technique is mainly used for inorganic compounds and also for proteins.



The Use of Co-crystallising agents[2,5]:

More than one chemically independent molecule can be included in a crystal. Often one of these molecules is included solely to induce crystallisation of the other. For instance, the introduction of extra hydrogen bond acceptors to a compound with an excess of hydrogen donors, or reduction in the motion of a long chain by tying it down with hydrogen bonds, thus allowing crystals to grow from a compound that on its own would usually form an oil. One of the major disadvantages of using co-crystallising agents is that if crystallisation does not occur, the sample must either be discarded or be purified to remove the co-crystallising agent before further attempts can be made at crystallisation. Also care has to be taken to carefully

check any crystals grown since often they are of the co-crystallising agent only, not both compounds, this occurs since the co-crystallising agent is usually a compound that is very easy to recrystallise.

As can be seen from all these methods, the important factors when attempting any crystal growth experiment are:

- Use only very pure samples.
- Reduce the numbers of nucleation sites by using clean dust free equipment.
- Consider the effect of the solvent used.
- Achieve slow growth by very slowly adjusting the conditions to achieve nucleation and growth i.e. slow evaporation, slow cooling, slow diffusion, etc.
- Consider the likely position and effect of hydrogen bonds in the crystal, would the use of a co-crystallising agent help?

The Experiment:

The compounds, from which I attempted to grow crystals, were all medium sized organic species. Since, for all the compounds, there was only a small amount of each sample available and since there was very little information available on the melting and boiling points and thermal stability of the compounds, growth from a liquid or vapour was not attempted. Gel methods were also not used as the compounds were being recrystallised, not formed from starting materials. So the techniques used (with varying degrees of success) were slow evaporation of solvent and cooling of a supersaturated solution, with in some cases the use of co-crystallising agents.

Of the eight compounds which I attempted to grow crystal of, two gave diffraction quality crystal (details of which are given in chapter 4), two were abandoned as routes of interest by the chemists before I managed to grow crystals, and four are still being worked on. Unfortunately I cannot give full details of the recrystallisation attempts as all the compounds are the property of Xenova Discovery Ltd.

References:

1) a) M.C.Etter, D.A.Jahn, B.A.Donahue, R.B.Johnson, C.Ojala. *J. Cryst. Growth.* 1986, **76**, 645.

   b) Advances in Crystal Growth. P.M.Dryburgh, B.Cockayne, K.G. Barraclough. Prenice Hall. New York, London. 1987.

   c) Crystal Growth and Characterization. R.Ueda, J.B.Mullin. North-Holland Publishing Company. Amsterdam, Oxford. 1975.

   d) Crystal Growth. B.R.Pamplin. Pergamon Press. Oxford, New York. 1975.

2) http://crystal.chemie.unibas.ch/xtl/services/growing.html

3) Y.Tanaka, M.Matsuoka. *J. Cryst. Growth.* 1990, **99**, 1130.

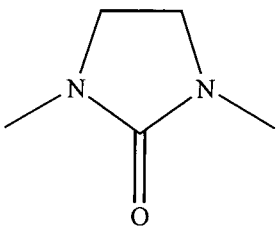
4) M.C.Robert, F.Lefaucheux, *J. Cryst. Growth.* 1988, **90**, 358.

5) V.R.Pedireddi, W.Jones, A.P.Chorlton, R.Docherty. *Chem. Commun.* 1996, 987.

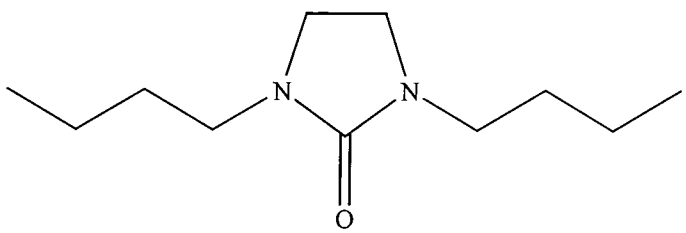
Chapter 4

Hydrogen bonding in methane-diphenol-3,5,3',5'-tetra^{t}butyl co-crystals

Co-crystals of 1,3-dimethyl-2-imidazolidinone (DMI) with methane-diphenol-3,5,3',5'-tetra^{t}butyl and 1,3-dibutyl-2-imidazolidinone (DBI) with methane-diphenol-3,5,3',5'-tetra^{t}butyl, were studied. The only difference between these two co-crystals is in the length of the 'arms' on the imidazolidinone molecule.

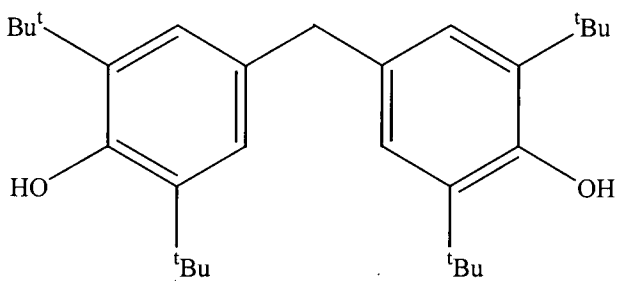


DMI



DBI

Co-crystallized with:



Methane-diphenol-3,5,3',5'-tetra^{t}butyl

Diagram 4.1: The molecular species in the co-crystals

The bonding patterns and crystal structure of these two co-crystals were studied and compared, with the aim of understanding the effect of extending the imidazolidinone molecule's 'arms' on the crystal structure.

Crystal Data:

Name:	1,3-dimethyl-2-imidazolidinone with methane-diphenol-3,5,3',5'-tetra^{t}butyl	1,3-dibutyl-2-imidazolidinone with methane-diphenol-3,5,3',5'-tetra^{t}butyl.
Formula	C5 H10 N2 O1 / C29 H44 O2	C11 H22 N2 O1 / C29 H44 O2
Colour	Yellow	Pale yellow
Morphology	Block	Needle
Size	0.40 × 0.36 × 0.24 mm	0.30 × 0.20 × 0.15 mm
Instrument	Smart-CCD[1]	Smart-CCD[1]
Temp/K	150(2)	150(2)
a	13.4162(2)Å	14.0667(3)Å
b	14.1096(10)Å	13.4124(3)Å
c	19.22689(3)Å	20.9124(5)Å
α	105.30(1)°	90°
β	102.31(1)°	94.71(1)°
γ	94.58(1)°	90°
Volume / z	3394.0(1)Å^3 / 4	3932.2(2) Å^3 / 4
Crystal system	Triclinic	Monoclinic
Space group	P-1	P2(1)/n
R(int)	0.0454	0.1523
Absorption correction	None	None
Mu mm^{-1}	0.066	0.065
Total data unique	15361	9002
Data I>2σ	9265	4385
R1 (all data)	0.1088	0.2219

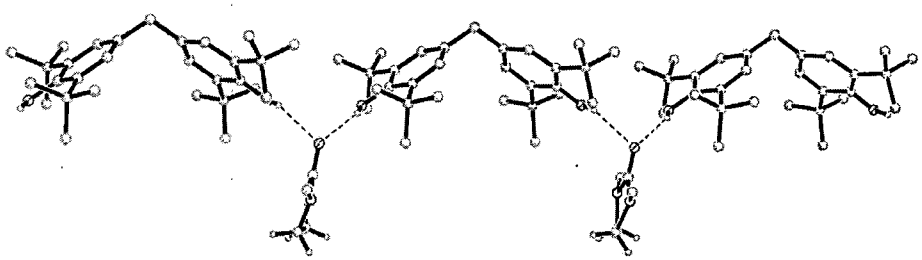
See also

Appendix B:	Compound 1, page 102	Compound 2, page 111

Crystal Structure and packing:

The structure of these co-crystals is very interesting. Looking only at the unit cells they appear to be completely different, in different space groups and different sized cells. The DMI co-crystal has four molecules (ie two DMI and two methane-diphenol molecules) in the independent part of the unit cell, while the DBI co-crystal has only two molecules. However the packing motif in 2-dimensions is identical, only when the 3rd dimension is considered does the packing show any differences that could lead to the different cells. The relative orientation of the molecules differs only slightly between the co-crystals.
These compounds show some very interesting hydrogen bonding interactions. First the two classical hydrogen bond donors join to the classical hydrogen bond acceptor to form zigzagging chains.

A:



B:

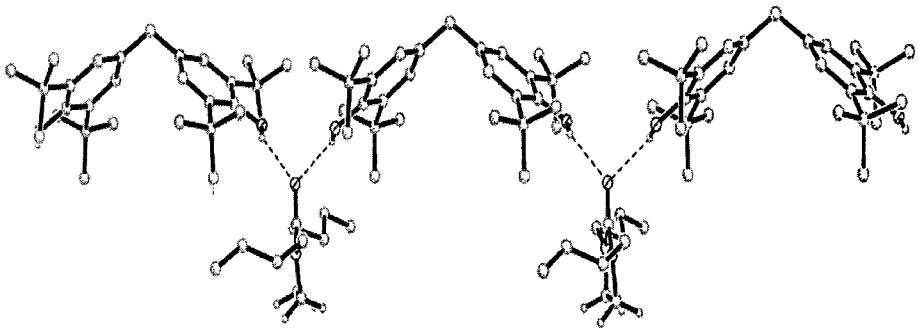
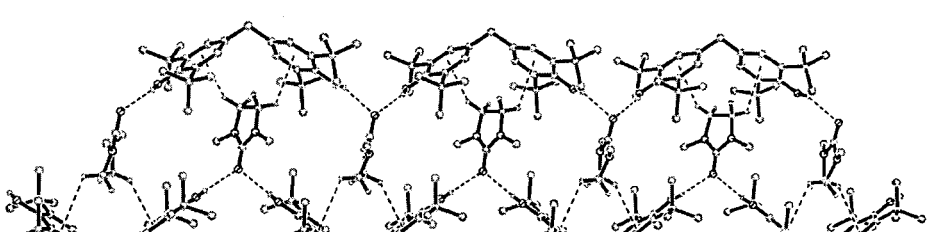


Diagram 4.2: Classical hydrogen bonded chain A) DMI co-crystal, B) DBI co-crystal.

The chains then form double chains, by a non-classical C-H…π hydrogen bond interaction (see also chapter 6) between the C-H of the imidazolidinone and the phenol rings of the methane-diphenol. Each of the imidazolidinone molecules fits into the cleft of the methane-diphenol of the neighbouring chain.

A:



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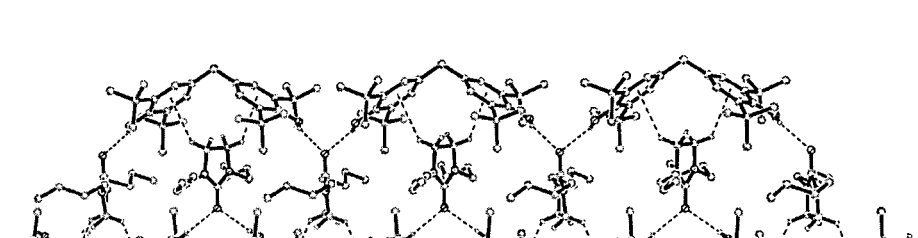
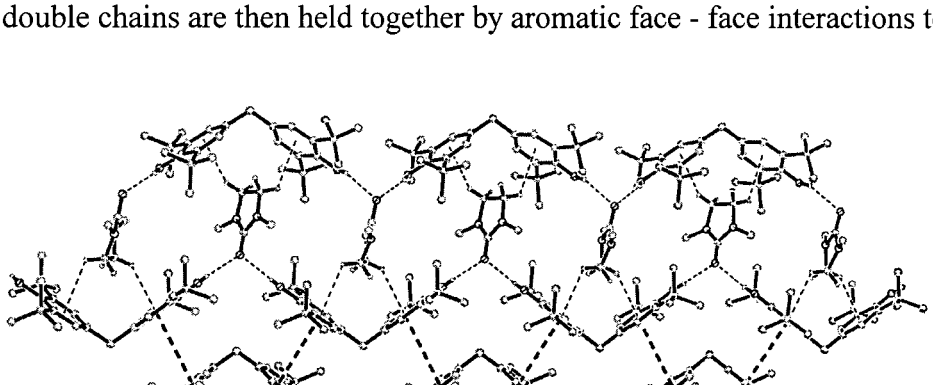


Diagram 4.3: Double chains A) DMI co-crystal, B) DBI co-crystal.

These double chains are then held together by aromatic face - face interactions to give sheets
A:



B:

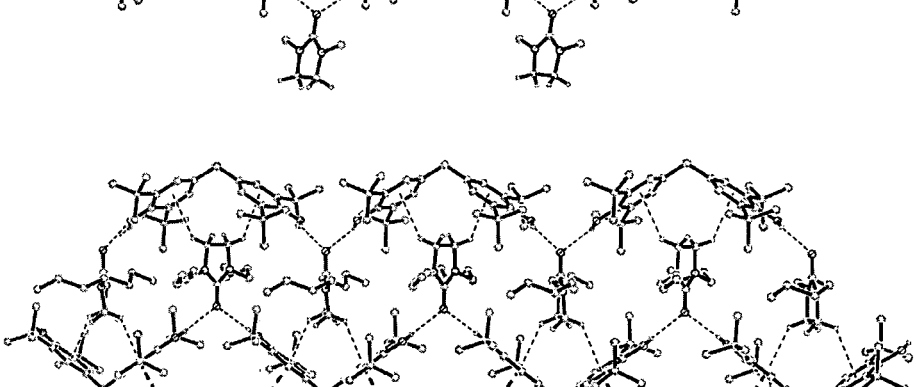
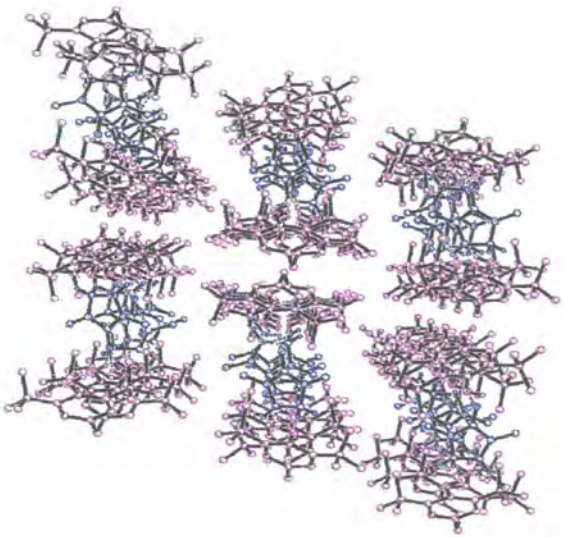


Diagram 4.4: Aromatic face-face interactions holding the double chains together, A) DMI co-crystal, B) DBI co-crystal.

These sheets then stack, as shown below. It is only here that the differences between the two co-crystals become apparent.

A:



B:

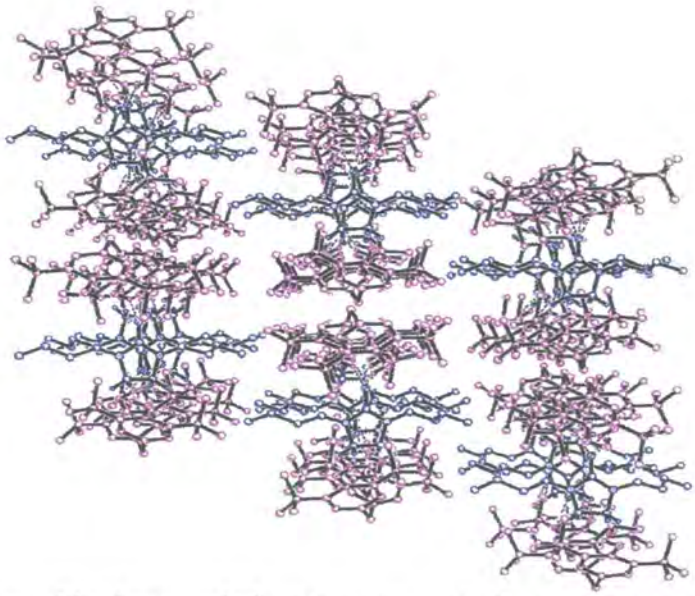


Diagram 4.5: Stacking of the sheets, each sheet is running vertically perpendicular to the plane of the page (aromatic face-face interactions are not shown, but link two vertically adjacent double chains). A) DMI co-crystal, B) DBI co-crystal.

The amount each sheet is displaced relative to the adjacent sheets is different in the different co-crystals. The separation between the sheets also changes, being greater in the DBI co-crystal for the obvious reason of the longer 'arms' on the imidazolidinone molecule, this agrees with the larger cell volume for this crystal.

The diagram below shows clearly why the packing within each sheet is constant despite the change in molecular structure, the methyl / butyl groups on the imidazolidinone molecule are

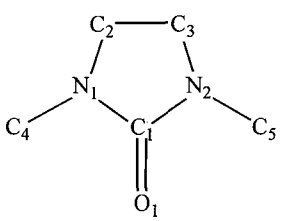
directed perpendicularly out from the sheet thus they do not influence the packing within the sheet itself.

It might seem strange that such a small change in the structure in only one dimension can have such a large impact on the space group and unit cell, but of course it takes only a small shift in relative position of two molecules to remove any symmetry between them. The space group and unit cell assignment for both co-crystals have been very carefully checked.

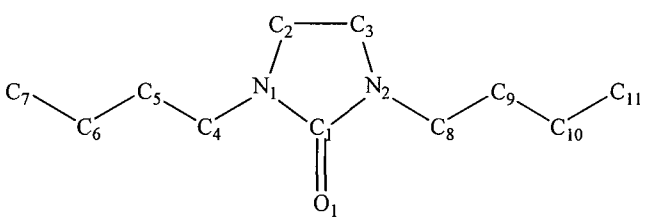
These crystals are a nice example of three different types of intermolecular interaction: classical hydrogen bonding, non-classical hydrogen bonding, and aromatic face-face interactions, all coming together in a single crystal to influence the structure.

The numbering system used is such that the corresponding atoms in each crystal are given the same numbers. But in the DMI co-crystal where there are two of each molecule type in the independent unit 100 or 200 is added to each number to distinguish between the molecules: ie carbon C20 in the DBI co-crystal corresponds to C120 and C220 in the DMI co-crystal. The occurrence of two molecules of each type (instead of just one) in the independent unit in the DMI co-crystal doubles the number of independent interactions seen.

Numbering scheme for the compounds:

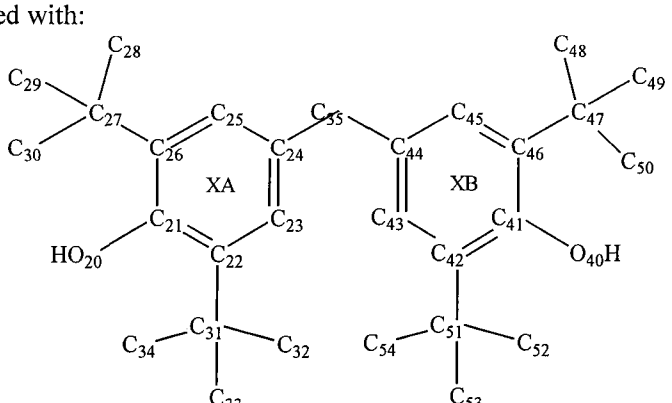


DMI



DBI

Co-crystallized with:



Methane-diphenol-3,5,3',5'-tetra^{t}butyl

Diagram 4.6: Numbering scheme used

Table 4.1: Hydrogen bond distances in the DMI co-crystal.

TYPE: A…H…B	ATOM (/RING) A	ATOM H	ATOM (/RING) B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...O	O(101)	H(120)	O(120)	2.963(2)	2.315(28)	136(2)
O...H...O	O(101)	H(140)	O(140)	2.872(2)	2.191(30)	139(3)
O...H...O	O(201)	H(220)	O(220)	2.837(2)	2.191(28)	133(2)
O...H...O	O(201)	H(240)	O(240)	2.826(2)	2.136(36)	137(3)
π...H...C	X(1A)	H(203A)	C(203)	3.485(24)	2.842(24)	123.2(5)
π...H...C	X(1B)	H(202A)	C(202)	3.466(23)	2.571(24)	150.3(5)
π...H...C	X(2A)	H(102A)	C(102)	3.538(24)	3.004(25)	115.0(4)
π...H...C	X(2B)	H(103A)	C(103)	3.602(23)	2.776(24)	141.2(5)
π... π	X(1A)		X(1A)	5.799(47)		
π... π	X(1B)		X(1B)	4.543(49)		
π... π	X(2A)		X(2A)	4.343(49)		
π... π	X(2B)		X(2B)	4.251(47)		

Table 4.2: Hydrogen bond distances in the DBI co-crystal.

TYPE: A...H...B	ATOM (/RING) A	ATOM H	ATOM (/RING) B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...O	O(1)	H(20)	O(20)	2.907(4)	2.239(4)	136.5(1)
O...H...O	O(1)	H(40)	O(40)	2.949(4)	2.287(4)	135.9(1)
π...H...C	X(1A)	H(2A)	C(2)	3.545(40)	2.749(40)	137.7(9)
π...H...C	X(1B)	H(3A)	C(3)	3.488(40)	2.746(40)	132.1(9)
π... π	X(1A)		X(1A)	4.438(80)		
π... π	X(1B)		X(1B)	4.063(79)		

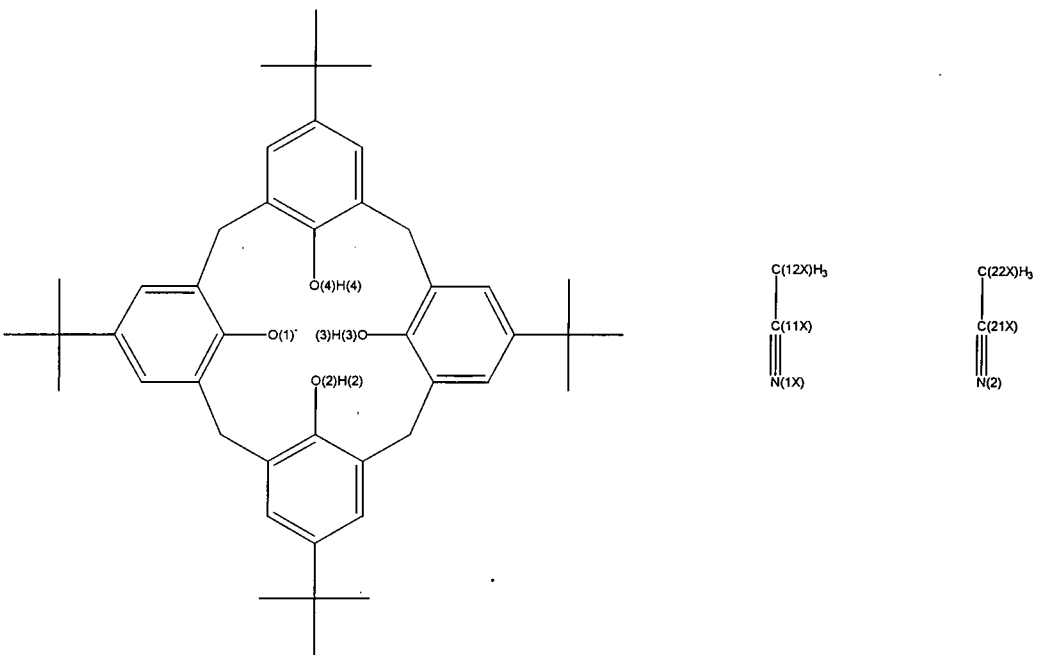
References:

1) SMART Version 4.050. Seines Analytical X-ray Instruments, Madison, U.S.A., 1995

Chapter 5

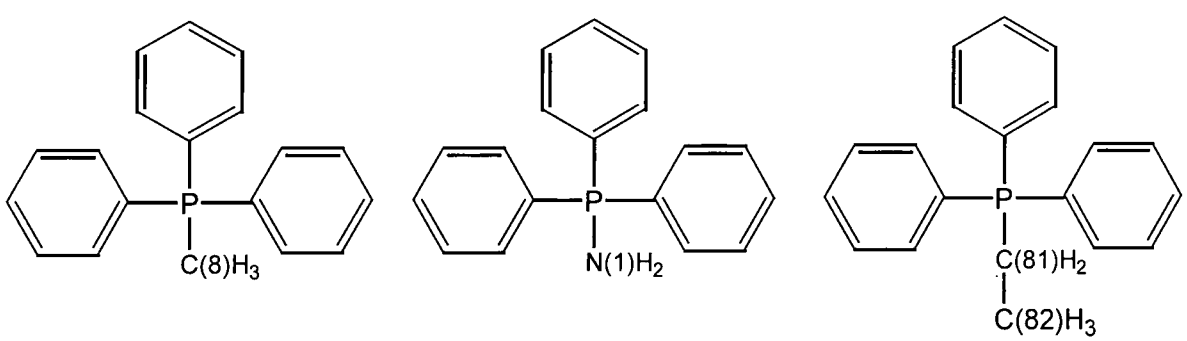
p-tert-butylcalix[4]arene with triphenyl phosphonium species.

p-tert-butylcalix[4]arene[1] was co-crystallised with three different triphenyl phosphonium species; triphenylmethylphosphonium Ph_3PCH_3, triphenylaminophosphonium Ph_3PNH_2 and triphenylethylphosphonium $Ph_3PCH_2CH_3$. The effect of this small change in structure on the unit cell and molecular packing was considered.



p-tert-butylcalix[4]arene + 2x solvent (acetonitrile)

with



Triphenylmethylphosphonium or triphenylaminophosphonium or triphenylethylphosphonium

Diagram 5.1: the molecular species in the co-crystals.

Crystal Data:

Name:	*p-tert*-butylcalix[4]arene and triphenylmethylphosphonium	*p-tert*-butylcalix[4]arene and triphenylaminophosphonium	*p-tert*-butylcalix[4]arene and triphenylethylphosphonium
Formula	C44 H52 O4 C19 H18 P1 (solvent 2×N1 C2 H3)	C44 H52 O4 C18 H17 N1 P1 (solvent 2×N1 C2 H3)	C44 H52 O4 C20 H20 P1 (solvent 2×N1 C2 H3)
Colour	Colourless	Colourless	Pale yellow
Morphology	Block	Block	Block
Size /mm	0.50×0.40×0.05	0.65×0.15×0.15	0.5×0.5×0.3
Instrument	Smart-CCD^2	Smart-CCD^2	Smart-CCD^2
Temp/K	150(2)	150(2)	150(2)
a	18.3934(3)Å	18.161(4)Å	12.8544(2) Å
b	12.6138(2)Å	12.592(3)Å	21.8410(1) Å
c	25.3686(4)Å	25.652(5)Å	21.0271(1) Å
α	90°	90°	90°
β	90.422(1)°	90.63(3)°	90.210(1)°
γ	90°	90°	90°
Volume / z	5885.6(2) Å^3 / 4	5866(2) Å^3 / 4	5903.4(1) Å^3 / 4
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	P2(1)/c	P2(1)/c	P2(1)/n
R(int)	0.1002	0.1332	0.0926
Absorption correction	None	None	Multiscan
Mu mm^{-1}	0.095	0.096	0.096
Total data unique	13478	13400	13525
Data I>2σ	8126	8803	8853
R1 (all data)	0.1476	0.1679	0.1397
Other	One of the tButyl groups on the calixarene exhibited Disorder	No disorder	The highest peak after refinement was 1.03eÅ^{-3} this peak was 1.54Å from a hydrogen on one of the ^{t}butyl groups on the calixarene. However I was unable to account for it by any simple disordering of the ^{t}butyl group.
See also Appendix B	Compound 3, page 118	Compound 4, page 127	Compound 5, page135

Crystal structure and Packing:

Calixarenes are bowl shaped molecules designed to trap or cage smaller molecules. Each of these three adducts show the same host – guest relationship between the calixarene and one of the solvent molecules (acetonitrile). This is a perfect example of a trapped molecule. A single molecule of the acetonitrile is trapped in the bowl of the calixarene. The acetonitrile is aligned so that the electropositive end is close to the electronegative base of the bowl and the electronegative end is pointing out of the bowl.

The trapping of a molecule in this way demonstrates very clearly an important factor in the way molecules pack in a crystal, that is that they will always pack so as closely as possible so as not to leave any empty voids in the crystal. The empty void that would exist inside the calixarene bowl is nicely filled by a molecule of the solvent; acetonitrile. This packing feature occurs in all the calixarene adducts considered here, regardless of whether the acetonitrile in question is involved in any other interactions.

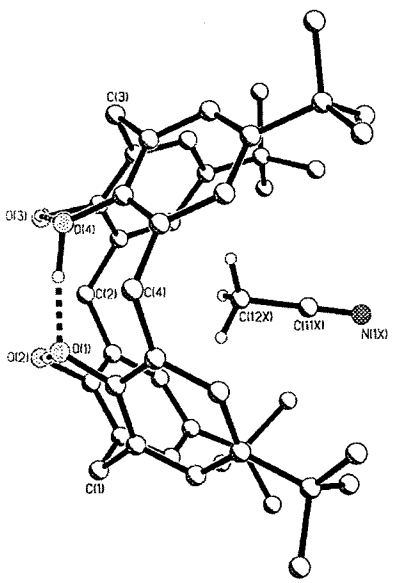


Diagram 5.2: Acetonitrile trapped in the calixarene cage.

This is where the similarity between the three adducts ends. While the effect of changing the -CH_3 for -NH_2 had a negligible effect on the packing of the molecules and, therefore, a negligible effect on the size and shape of the unit cell, the effect of changing either of these

groups for an ethyl -CH_2CH_3 has a very noticeable effect. Changing from a methyl or amino to an ethyl group may only give a small increase in size in relation to the whole compound, but it causes a great change the packing arrangement of the molecules within the unit cell. The unit cells of the three adducts are:

Calixarene with triphenylmethylphosphonium: P2(1)/c

Cell: a = 18.393(1)Å $\alpha = 90°$

b = 12.614(1)Å $\beta = 90.422(1)°$

c = 25.369(1)Å $\gamma = 90°$ Vol: 5885(1)$Å^3$

Calixarene with triphenylaminophosphonium: P2(1)/c

Cell: a = 18.161(4)Å $\alpha = 90°$

b = 12.592(3)Å $\beta = 90.63(3)°$

c = 25.652(5)Å $\gamma = 90°$ Vol: 5866(2)$Å^3$

Calixarene with triphenylethylphosphonium: P2(1)/n

Cell: a = 12.854(1)Å $\alpha = 90°$

b = 21.841(1)Å $\beta = 90.21(1)°$

c = 21.027(1)Å $\gamma = 90°$ Vol: 5903$Å^3$

The methyl and amino species adducts are in the same space group and have nearly identical unit cell dimensions. This similarity follows through to the packing where the only variation is a slight change in the solvent positions. The ethyl adduct is in a closely related space group to the methyl and amino adducts space group, but the actual cell and the packing of the molecules is very different. The two space groups both are of the P2(1)/n type, but the cells cannot be converted in to each other. The change in packing has not resulted in a loss or gain in the degree of symmetry in the crystal.

The Crystal structure and packing of *p-tert*-butylcalix[4]arene with triphenylaminophosphonium or triphenylmethylphosphonium.

Methyl and amino groups, though a little different in electronegativity and shape, are very similar in size. This may help to explain why there is no change in the packing arrangement of the molecules.

The structure is easiest to analyse by looking at the packing of the calixarenes and the tri-phenyl phosphonium species separately.

The calixarenes are packed in sheets with each bowl facing the same direction. The sheets stack alternately, i.e. with bowl base to bowl base and bowl top to bowl top, each sheet was displaced by half a molecule compared to the previous and next sheets so each bowl fits in the hollow between the four bowls beneath. The trapped acetonitrile points into the space between two sheets where the bowl tops meet. The second acetonitrile is hydrogen bonded to the base of the calixarene bowl so a row of acetonitrile separates two sheets of calixarene packed bowl base to bowl base.

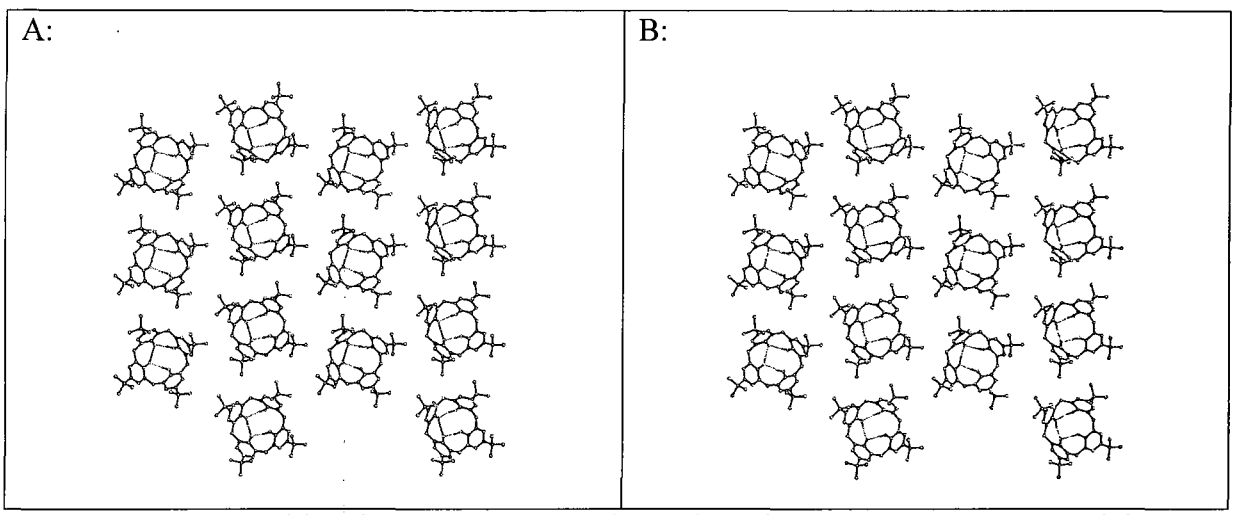


Diagram 5.3: Packing of the calixarene molecules, packing within a sheet viewed down a axis . A)methyl species, B)amino species.

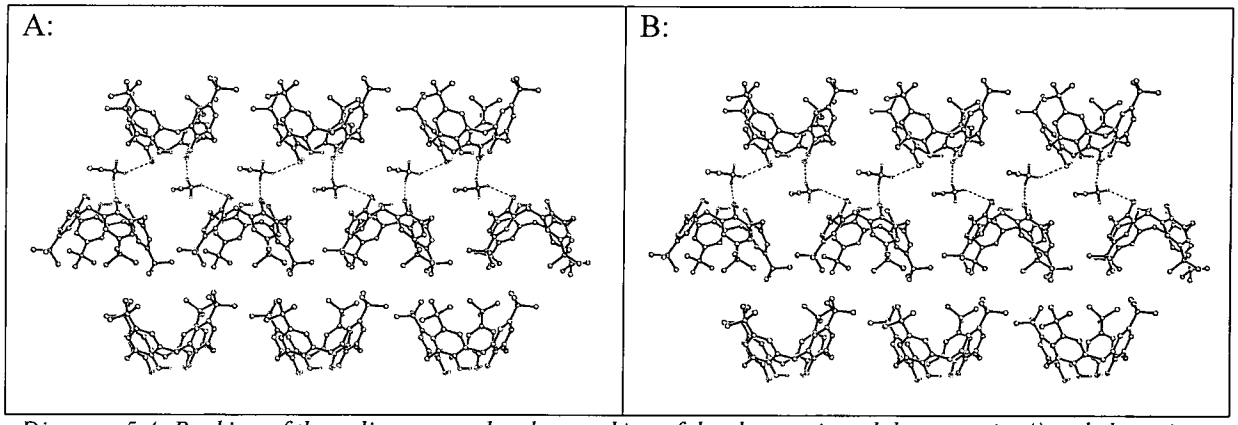


Diagram 5.4: Packing of the calixarene molecules, packing of the sheets, viewed down c axis. A)methyl species, B)amino species.

Although it is not very clear in the diagrams above, the acetonitrile position has very slightly shifted between compounds.

The triphenylphosphonium species also packs in sheets, each triphenylphosphonium is hydrogen bonded to the 'second' acetonitrile so the sheets of triphenylphosphonium species lie in between the two sheets of calixarene packed bowl base to bowl base.

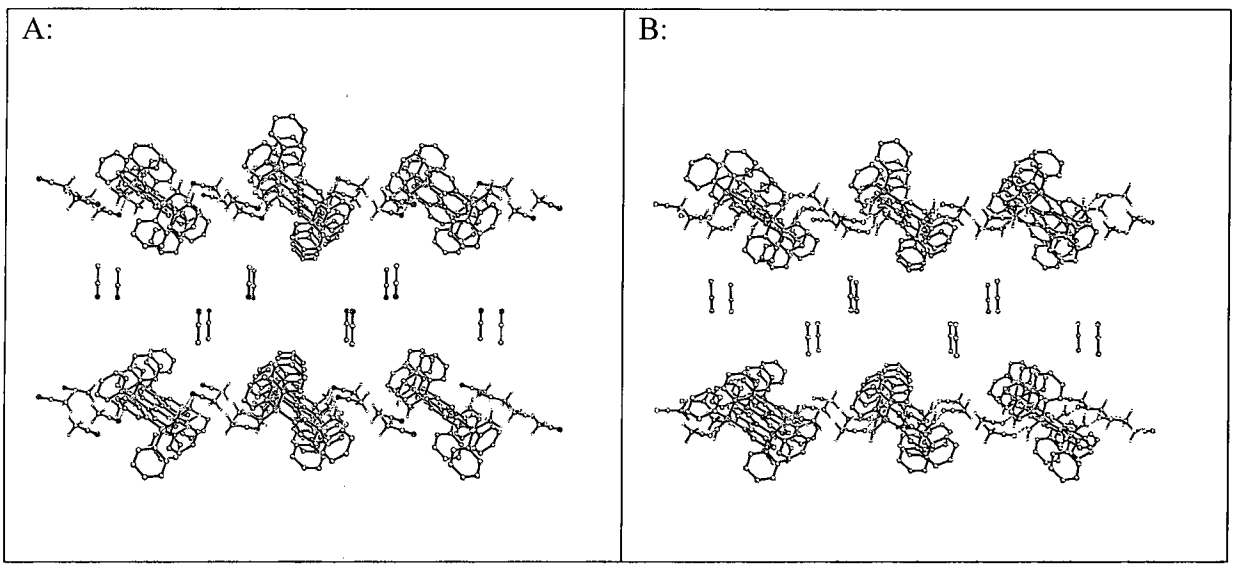


Diagram 5.5: two triphenyl phosphonium species sheets, viewed end on down b axis, acetonitriles give the position of the calixarene molecules. A)methyl species, B) amino species

Putting these two parts of the structure together gives the full crystal structure.

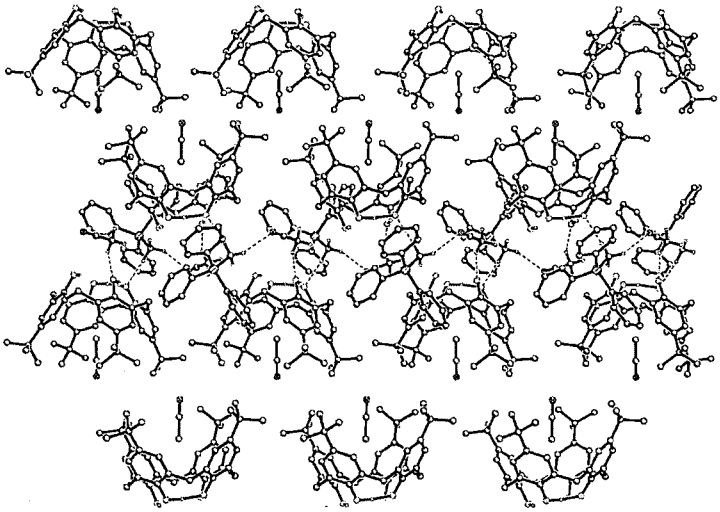


Diagram 5.6(a): The full crystal structure, methyl species.

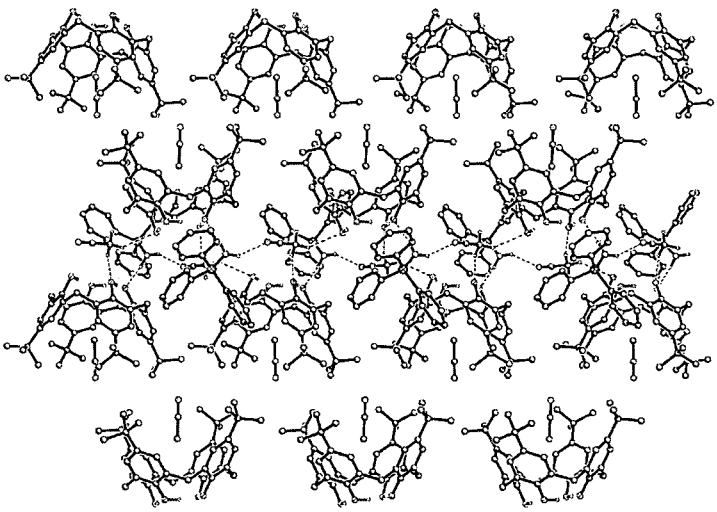


Diagram 5.6(b): The full crystal structure, amino species.

Table 5.1: Hydrogen bond distances.

For *p-tert*-calixarene and triphenylmethylphosphonium

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...C	O(2)	H(2XA)	C(22X)	3.211(4)	2.30(1)	154(1)
O...H...C	O(3)	H(2XB)	C(22X)	3.432(5)	2.63(2)	140(2)
N...H...C	N(2X)	H(8B)	C(8)	1.808(5)	1.61(4)	85(2)
O...H...C	O(1)	H(8C)	C(8)	3.153(4)	2.19(4)	55(2)
O..H..O	O(1)	H(2)	O(2)	2.551(3)	1.63(4)	176(4)
O..H..O	O(4)	H(3)	O(3)	2.721(3)	1.89(5)	163(5)
O..H..O	O(1)	H(4)	O(4)	2.521(3)	1.47(5)	173(5)
O..H..O	O(2)		O(3)	2.945(3)		

Table 5.2: Hydrogen bond distances.

For *p-tert*-calixarene and triphenylaminophosphonium

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...C	O(2)	H(2XA)	C(22X)	3.201(5)	2.25(1)	162.(2)
O...H...C	O(3)	H(2XB)	C(22X)	3.370(5)	2.57(2)	139(2)
N...H...N	N(2X)	H(1A)	N(1)	2.992(6)	2.27(5)	147(5)
O...H...N	O(1)	H(1B)	N(1)	2.745(5)	1.86(5)	173(4)
O..H..O	O(1)	H(2)	O(2)	2.572(4)	1.66(5)	178(5)
O..H..O	O(4)	H(3)	O(3)	2.728(4)	1.85(5)	171(5)
O..H..O	O(1)	H(4)	O(4)	2.556(4)	1.65(6)	169(6)
O..H..O	O(2)		O(3)	2.957(4)		

The Crystal structure and packing of *p-tert*-butylcalix[4]arene with triphenylethylphosphonium.

The ethyl group is much bigger than either the methyl or amino groups in the adducts considered above. This has a large effect on the way in which the molecules pack. Instead of the calixarene packing in sheets with all the molecules orientated in the same direction, the calixarenes are orientated in a more complexed pattern as seen below.

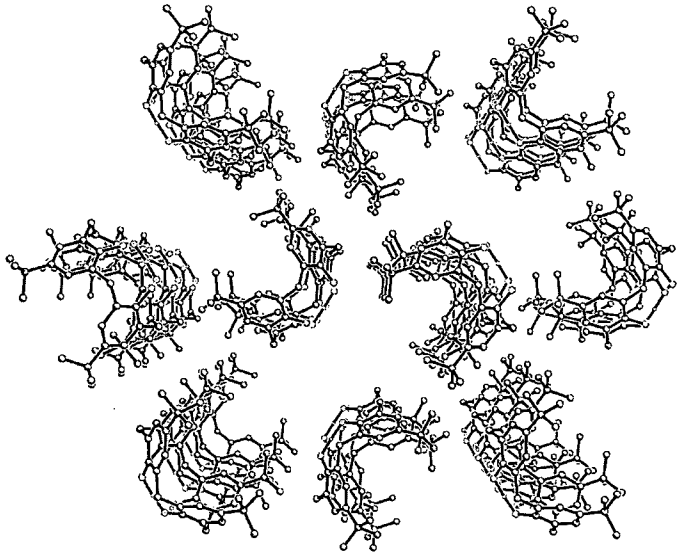


Diagram 5.7: Packing of the calixarene molecules.

The packing is best visualised as being built up from distinct columns or stacks, where each column consists of triphenylethylphosphonium stacked about a screw axis. Then there is a non-classical hydrogen bond interaction between the -CH_2- of the ethyl group and O(2) of the calixarene. There is also another non-classical hydrogen bond between the CH of one of the phenyl rings on the triphenylethylphosphonium molecule and first (trapped) acetonitrile molecule. These interactions define the orientation of the rest of the molecules about the triphenylethylphosphonium stack.

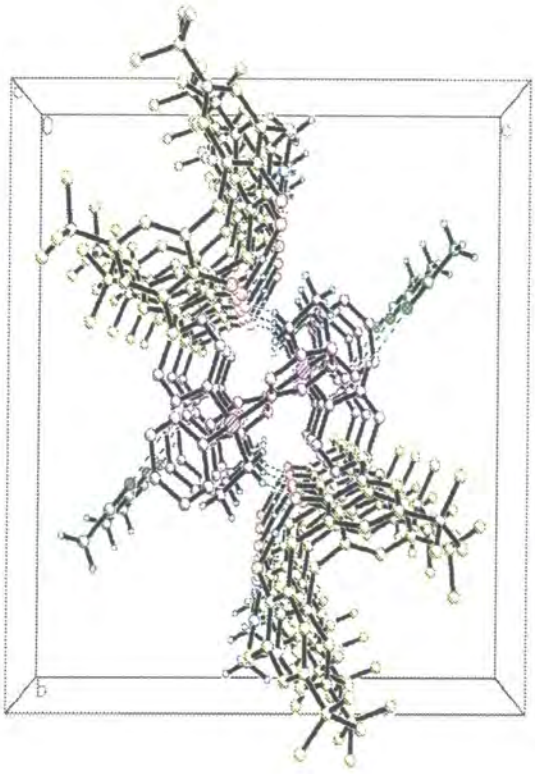


Diagram 5.8: Packing of the molecules about a triphenylethylphosphonium stack, viewed down stack.

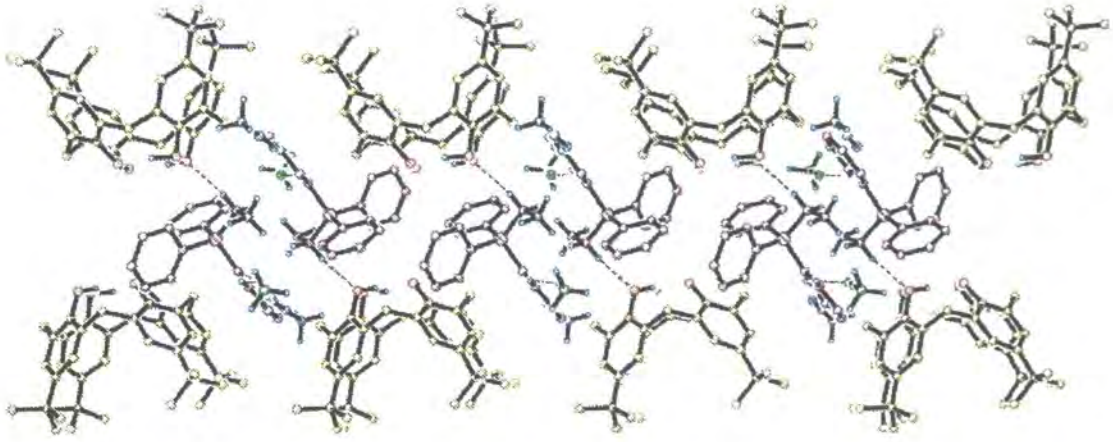
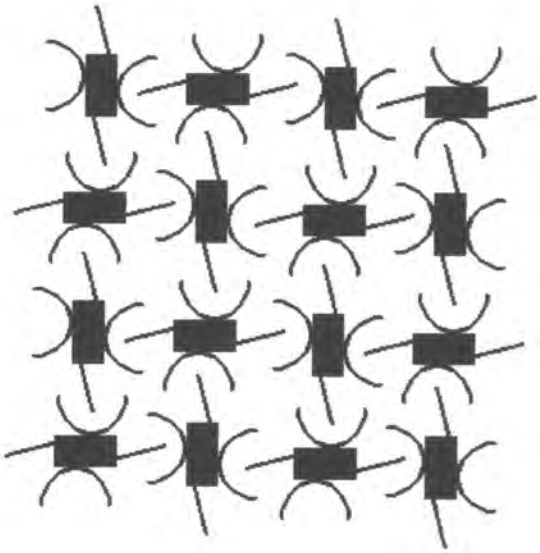


Diagram 5.9: Packing of the molecules about a triphenylethylphosphonium stack, viewed along stack

If one of these stacks (viewed down the stack) is considered as a simple pictogram:

Then the packing can be clearly demonstrated:



i.e: in the real crystal:

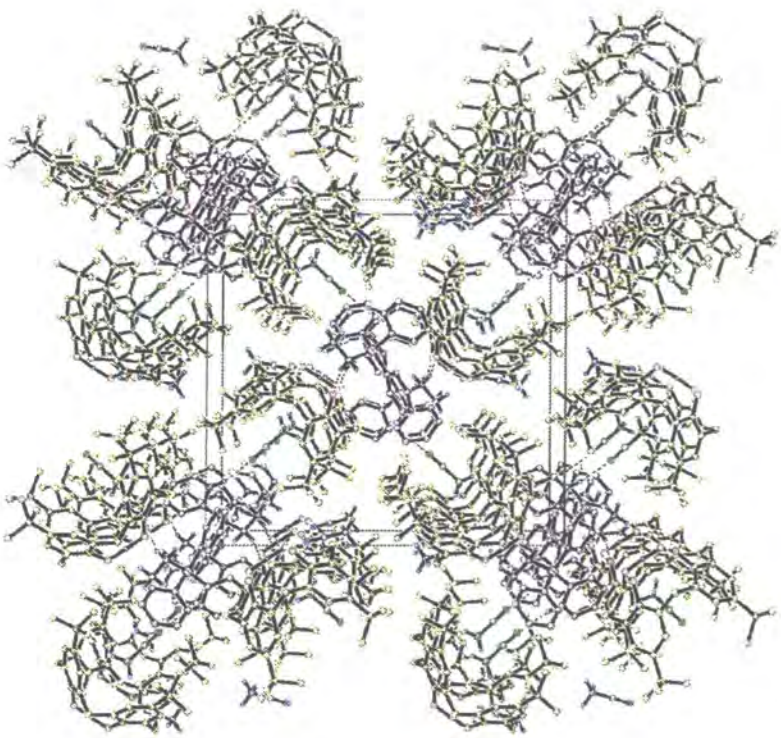


Diagram 5.10: Packing of the stacks (viewed down the stacks), in the actual crystal.

In the previous packing arrangement (seen for the methyl and amino species adducts) the trapped acetonitrile was a space filler only, with no interactions outside the calixarene bowl. Now this acetonitrile (green in the diagram above) is involved in a hydrogen bond to the ethyl group of the triphenylethylphosphonium molecule, however the second acetonitrile (blue in the diagram above) has no interaction or effect on the packing except as a space filler.

Table 5.3: Hydrogen bond distances.

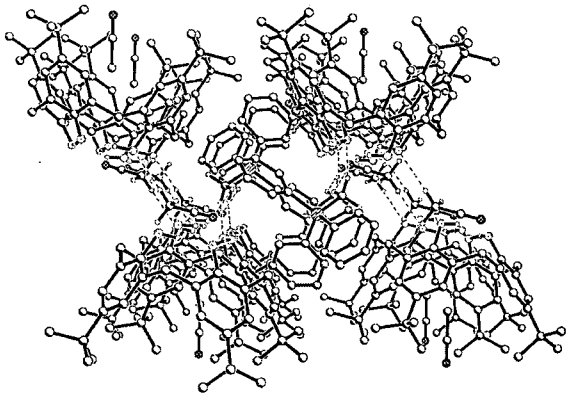
For p-tert-calixarene and triphenylethylphosphonium

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...C	O(2)	H(81B)	C(81)	3.225(4)	2.35(4)	164(3)
N...H...C	N(1X)	H(52)	C(52)	3.336(5)	2.58(1)	136(1)
O..H..O	O(1)	H(2)	O(2)	2.596(3)	1.691(6)	173(4)
O..H..O	O(4)	H(3)	O(3)	2.655(3)	1.629(5)	173(4)
O..H..O	O(1)	H(4)	O(4)	2.491(3)	1.329(6)	167(5)
O..H..O	O(2)		O(3)	2.964(3)		

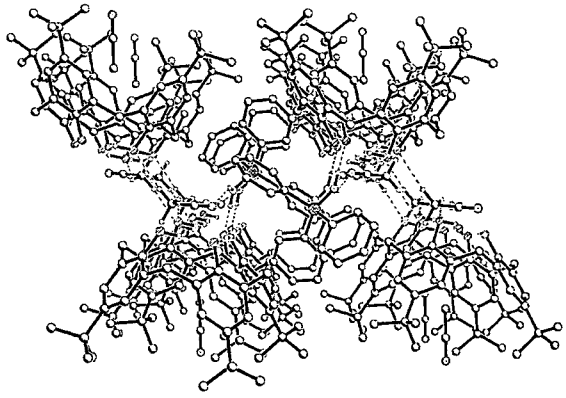
Conclusions:

The small variation of phosphonium molecule in changing from methyl/amino species to an ethyl species results in a big change in packing, but there are still strong similarities between the two structures. The calixarene and triphenylphosphonium part of the motif considered above (see below) is common to both structures, the differences arise in the solvent (acetonitrile) positions and the packing of this common motif.

A:



B:



C:

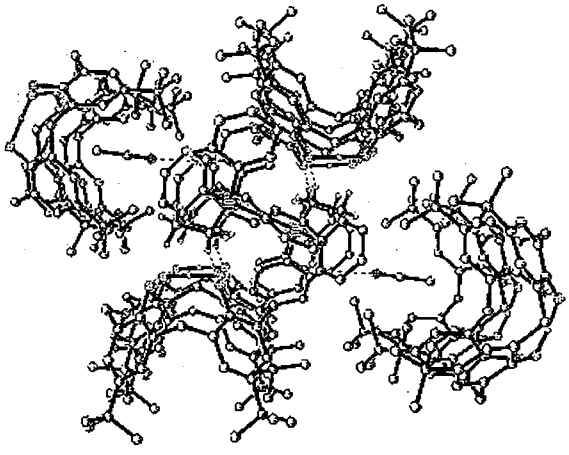


Diagram 5.11: Packing of the common motif, A)methyl species B)amino species C)ethyl species.

The changes in the space group and packing appear to arise from small changes in the size of the triphenyl phosphonium group and not from changes in electronic or other properties. This conclusion is drawn from the fact that the methyl and amino groups which are the same size but have different electronic properties exhibit the same space group, while methyl and ethyl groups that exhibit the same electronic properties but have different sizes show a change in space group and packing.

References:

1) Calixarenes. C.D.Gutsche. Royal Society of Chemistry, Cambridge. 1989.

2) SMART Version 4.050. Siemans Analytical X-ray instruments, Madison, U.S.A., 1995

3) G.M.Sheldrick. SHELXL 93. Program for Refinement of Crystal Structures. University of Göttingen, Germany.

Chapter 6

Effect of the metal on the structure of various metal phosphonium complexes.

A range of organometallic complexes were analysed and their structures compared. The range consisted of three different alkaline earth metals (calcium, strontium, and barium) and two different sets of ligands, giving six compounds in total. The effect of increasing the metal size with in the alkaline earth group, and the effect of changing the ligand type on the structure was considered.

The compounds considered are:

Series 1	Series 2
$(Me(C_6H_2)^tBu_2)_2$-Ca-$(N(H)PPh_3)_2$	$((Me_2Si)_2N)_2$-Ca-$(CH_2PPh_3)_2$
$(Me(C_6H_2)^tBu_2)_2$-Sr-$(N(H)PPh_3)_2$	$((Me_2Si)_2N)_2$-Sr-$(CH_2PPh_3)_2$
$(Me(C_6H_2)^tBu_2)_2$-Ba-$(N(H)PPh_3)_2$	$((Me_2Si)_2N)_2$-Ba-$(CH_2PPh_3)_2$

In each of these cases the metal is in the 2^+ state, the corresponding standard ionic radii[1] for the 2^+ state are; Ca^{2+} = 0.99Å, Sr^{2+} = 1.12Å, Ba^{2+} = 1.34Å. Only two of the four ligands in each set undergo a major change between the series, changing from 2,6-di^{t}butyl-4-methylphenoxy to hexamethyldisilylamide. The change in the phosphonium based ligand is only slight, see also chapter 5.

Experimental Details: Series 1

Crystal Data:

Name:	Calcium bis(2,6-di^{t}butyl-4-methylphenoxy) bis(triphenylaminophosphonium)	Strontium bis(2,6-di^{t}butyl-4-methylphenoxy) bis(triphenylaminophosphonium)	Barium bis(2,6-di^{t}butyl-4-methylphenoxy) bis(triphenylaminophosphonium)
Formula	C66 H78 N2 O2 P2 Ca	C66 H78 N2 O2 P2 Sr	C66 H78 N2 O2 P2 Ba
Colour	Colourless	Colourless	Colourless
Morphology	Block (elongated)	Block (Rhombic prism)	Block (plate like)
Size /mm	0.30×0.15×0.10	0.35×0.25×0.20	0.50×0.30×0.10
Instrument	Smart-CCD[2]	Smart-CCD[2]	Smart-CCD[2]
Temp/K	150(2)	150(2)	150(2)
a	25.6442(2) Å	25.6929(2) Å	12.5952(1) Å
b	11.5399(1) Å	11.6965(1) Å	14.8787(2) Å
c	19.8180(1) Å	19.9288(3) Å	18.6550(3) Å
α	90°	90°	80.227(1)°
β	90°	90°	71.041(1)°
γ	90°	90°	69.526(1)°
Volume / z	5864.86(7) Å^3 / 4	5988.94(11) Å^3 / 4	3091.27(7) Å^3 / 2
Crystal system	Orthorhombic	Orthorhombic	Triclinic
Space group	Pca2(1)	Pca2(1)	P-1
R(int)	0.1140	0.0955	0.0263
Absorption correction	Multiscan[3]	Multiscan[3]	Psi scan[4]
Mu mm^{-1}	0.206	0.997	0.736
Total data unique	13450	13720	14074
Data I>2σ	9289	9901	11873
R1 (all data)	0.1075	0.0864	0.0487
See also Appendix B:	Compound 6, page 144	Compound 7, page 160	Compound 8, page 168

Experimental Details: Series 2

Crystal Data:

Name:	Calcum bis(trimethylsilylamide) bis(triphenylmethylphosphonium)	Strontium bis(trimethylsilylamide) di(triphenylmethylphosphonium)	Barium bis(trimethylsilylamide) bis(triphenylmethylphosphonium)
Formula	C50 H70 N2 P2 Si4 Ca (solvent 2×C7 H8)	C50 H70 N2 P2 Si4 Sr (solvent 2×C7 H8)	C50 H70 N2 P2 Si4 Ba
Colour	Yellow	Yellow	Yellow
Morphology	Block	Sphere	Block
Size /mm	0.50×0.10×0.10	0.30×0.30×0.30	0.25×0.10×0.10
Instrument	Smart-CCD[2]	Smart-CCD[2]	Smart-CCD[2]
Temp/K	150(2)	150(2)	150(2)
a	30.598(6) Å	30.612(5) Å	9.882(2) Å
b	12.393(3) Å	12.435(2) Å	23.521(5) Å
c	22.023(4) Å	22.039(3) Å	24.193(5) Å
α	90 Å°	90°	90°
β	129.03(3)°	129.028(6)°	98.91(3)°
γ	90 Å°	90°	90°
Volume / z	6488(2) $Å^3$ / 4	6517(2) $Å^3$ / 4	5556(1) $Å^3$ / 4
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	C2/c	C2/c	P2(1)/n
R(int)	0.0627	0.0980	0.2066
Absorption correction	Multiscan[3]	Multiscan[3]	Multiscan[3]
Mu mm^{-1}	0.258	0.987	0.890
Total data unique	7208	7139	12724
Data I>2σ	4782	4230	6283
R1 (all data)	0.1154	0.1257	0.1824
See also Appendix B:	Compound 9, page 168	Compound 10, page 173	Compound 11, page 187

Crystal structure and packing

The first point of interest to note is that the effect of changing the set of ligands gives a completely different structure regardless of which metal is used. This suggests that the overall shape or properties of the molecule is more important in determining the crystal structure, not the identity of the metal itself. Even though there is only a very slight difference in the phosphonium ligands in the different ligand sets, the structures bear no relation to each other, and the interactions at the phosphonium ligands are completely different.

The next point is that in both series the effect of changing the calcium for a strontium ion has a negligible effect on the unit cell either in terms of space group, size, or packing. However the effect of changing either of these ions for a barium ion causes a complete change in all of the above three factors. It is not surprising that when the structure changes it is such a complete change, in fact it would be quite hard to conceive of a change in any one of these factors (with the possible exception of small changes in size), that could occur without changing all of them.

The effect of changing calcium for strontium in the first series (with 2,6-di^{t}butyl-4-methylphenoxy ligand) caused a slight increase in the unit cell size. This is logical given the fact that strontium is larger than calcium. However changing calcium for strontium in the second series (with hexamethyldisilylamide ligand) has no effect on the unit cell size (within the error limits).

Crystal structure and packing in Series 1

Taking a closer look at the packing in series 1 highlights a number of interesting points. While there are no classical hydrogen bonds in any of the compounds in this series (the only classical donor/acceptor group, two NH groups, are buried near the centre of the molecule), there are a number of non-classical C-H...π bonds[5,6] in each case. This type of bonds are among the weakest of the accepted hydrogen bonds, and it is unlikely that that they have a great influence on the packing of the crystal but nevertheless they are worth considering.

For the barium compound the hydrogen bonding pattern is very simple to visualise. The barium complex forms chains two molecules wide, related by an inversion centre. It is very interesting to note the 'double', non-symmetrical, C-H...π...H-C hydrogen bond leading from one of the 2,6-di^{t}butyl-4-methylphenyl groups, this is a fairly unusual hydrogen bonding system.

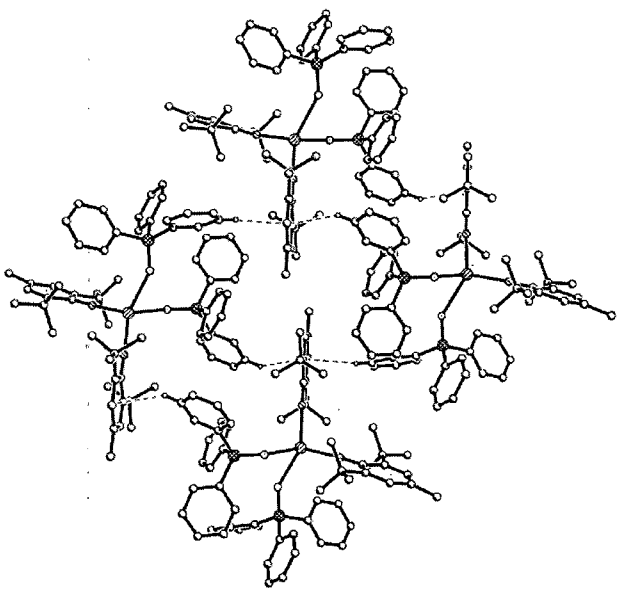


Diagram 6.1: segment of the hydrogen bonded chain.

Each of these chains is bound to the next and vica versa by another set of inversion related hydrogen bonds.

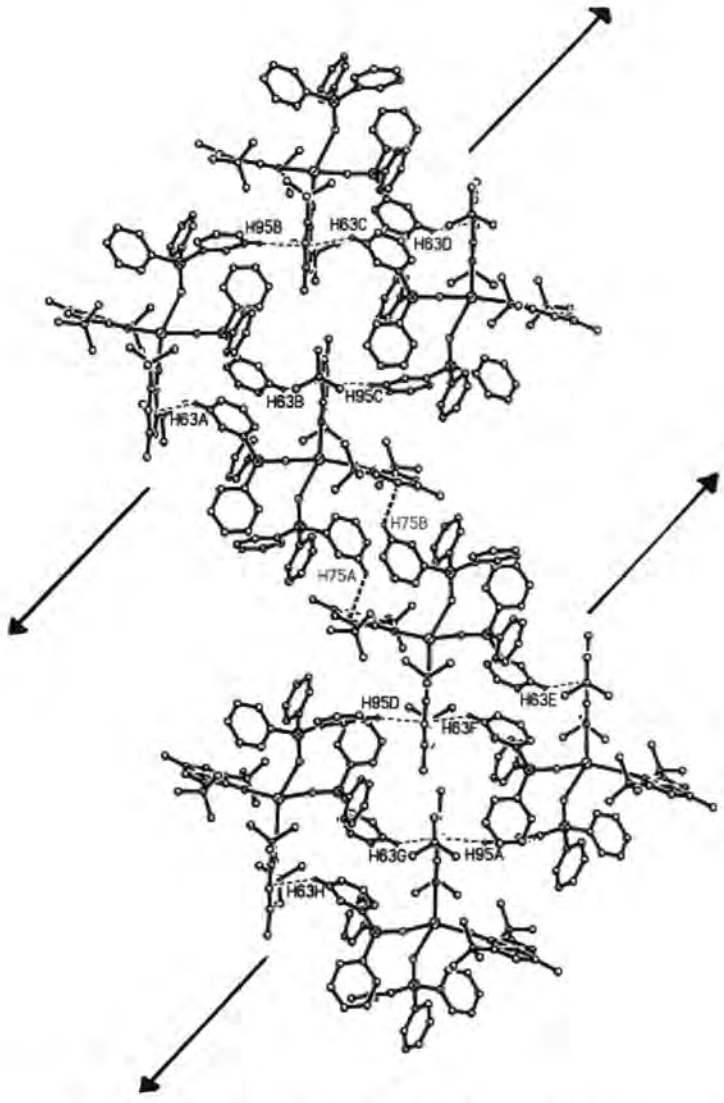


Diagram 6.2: Hydrogen bonds joining the chains (each chain runs from bottom left to top right in diagram).

Table 6.1 Hydrogen bonding distances in barium species

TYPE: A…H…B	RING CENTER A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
π...H...C	C(1)-C(6)	H(63)	C(63)	3.427(9)	2.576(9)	149.4(2)
π...H...C	C(1)-C(6)	H(95)	C(95)	3.658(9)	2.827(9)	146.8(2)
π...H...C	C(21)-C(26)	H(75)	C(75)	3.398(10)	2.753(9)	125.9(2)

The hydrogen bonding pattern in the calcium and strontium compounds is far more complicated; involving seven, non-classical C-H...π hydrogen bonds[5,6] with each molecule linked to five other symmetry generated molecules. The two compounds are in nearly identical configurations since when the two are compared, the weighted route mean squared difference between the two sets of co-ordinates is 0.6729 Å.

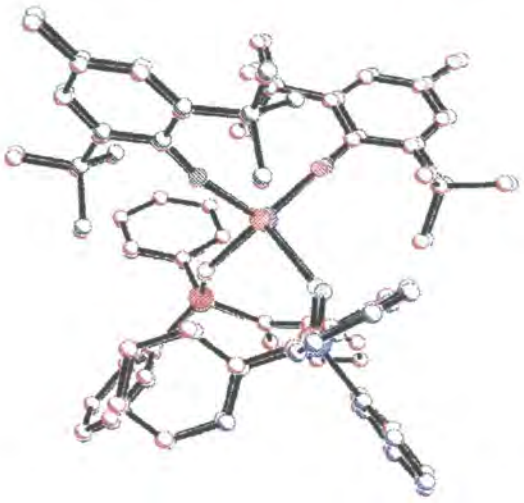


Diagram 6.3: comparison of the comparative structure of calcium and strontium compounds for series 1, calcium species = red, strontium species = blue.

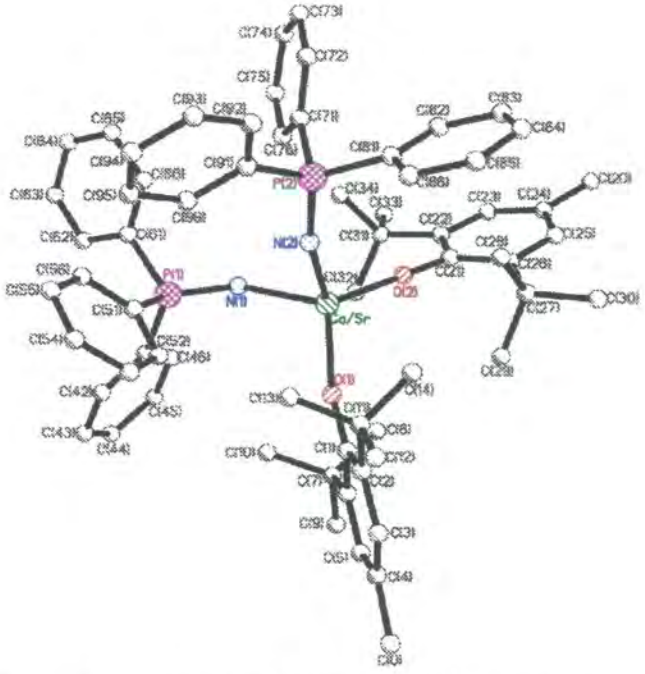


Diagram 6.4: numbering system used in bis(2,6-di^{t}butyl-4-methylphenoxy)bis(triphenylaminophosphonium)-calcium/strontium.

Given the identical packing it is a logical extension that the hydrogen bonding pattern will be identical, and this is what occurs, with only slight variations in bond lengths and angles.

Table 6.2: Hydrogen bonding distances for calcium and strontium species

TYPE: A...H...B	RING CENTER A	ATOM H	ATOM B	DISTANCE A...B /Å		DISTANCE A...H /Å		ANGLE A-H-B /°	
				Calcium	Strontium	Calcium	Strontium	Calcium	Strontium
π...H...C	C(1)-C(6)	H(55)	C(55)	3.721(16)	3.775(17)	2.861(16)	2.909(17)	151.1(3)	152.1(3)
π...H...C	C(1)-C(6)	H(85)	C(85)	3.501(17)	3.539(19)	2.816(18)	2.892(20)	129.8(4)	126.3(5)
π...H...C	C(21)-C(26)	H(65)	C(65)	3.700(18)	3.698(19)	2.951(18)	2.954(19)	136.6(4)	136.1(5)
π...H...C	C(71)-C(76)	H(30A)	C(30)	3.738(17)	3.781(17)	2.977(37)	3.010(40)	135.3(7)	136(1)
π...H...C	C(71)-C(76)	H(45)	C(45)	3.933(18)	4.078(21)	3.102(18)	3.263(22)	147.1(3)	145.1(4)
π...H...C	C(81)-C(86)	H(33B)	C(33)	3.795(17)	3.805(16)	2.816(64)	2.829(68)	174.9(5)	174(2)
π...H...C	C(91)-C(96)	H(43)	C(43)	3.862(18)	3.875(19)	3.079(18)	3.087(19)	140.8(3)	141.3(3)

It can be seen from the above table that in each case there are two 'double' C-H...π...H-C bonds of the type that were seen in the barium compound. These 'double' C-H...π...H-C bonds lead from ring C(1)-C(6) and C(71)-C(76). Again these bonds are non-symmetric about the ring. They are not noticeably any weaker (longer) that 'single' π...H-C bonds system despite the fact that two $H^{\delta+}$ are interacting with the same area of π density (though from opposite sides of the ring). It is worth noting that the H bonds for the barium compound are far fewer (if overall slightly shorter/stronger) than for the calcium and strontium compounds. This fact coupled with the fact that calcium and strontium complexes both take the same form, perhaps indicating that the Pca2(1) is the preferred form until the increase in metal size forces the compound take the P-1 form.

Crystal structure and packing in Series 2

In this series as in series 1, the calcium and strontium compounds take the same crystal structure, where as barium exhibits a completely different structure. The calcium and strontium compounds, despite the slight difference in metal size, have unit cells that are identical in size within the experimental errors. When the structures are studied more closely, it is seen that while there is a very good agreement between the molecular structure, the weighted route mean squared deviation between the metallic species co-ordinates is only 0.0752 Å. While the solvent appears to completely change position from one compound to the other, the two positions are in fact symmetry equivalents of each other, and therefore, effectively the same position.

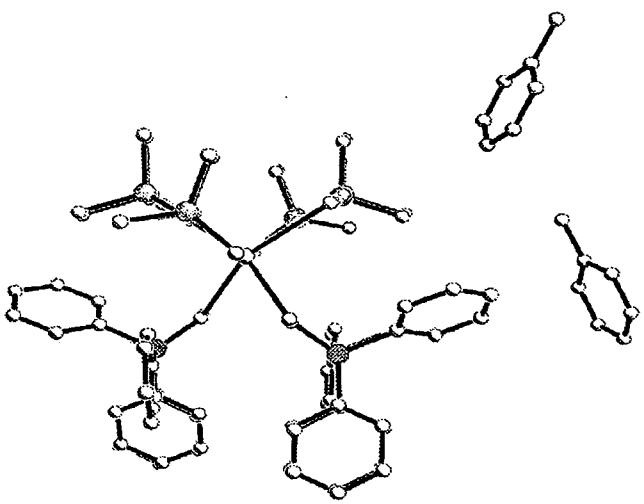
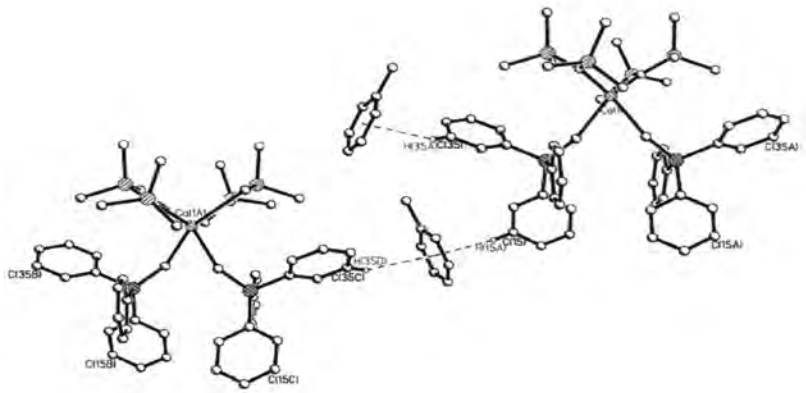


Diagram 6.5: comparison of the comparative structure of calcium and strontium compounds for series , calcium species = red, strontium species = blue. Solvent positions are symmetry equivalents.

The solvent must be filling an empty void in the structure. The occurrence of this partially filled void perhaps explains why there is no change in the unit cell dimensions on changing to a larger metal, the compound simply expands into the empty space.

The hydrogen bonding pattern for these compounds is very simple, as before there are no accessible classical hydrogen bond donors/acceptors. However in the previous complexes these compounds exhibit very few hydrogen bonds of any type, and the only bond seen is that to the π ring of the solvent. In both compounds this is a double set of 'double' C-H...π...H-C bonds.

A:



B:

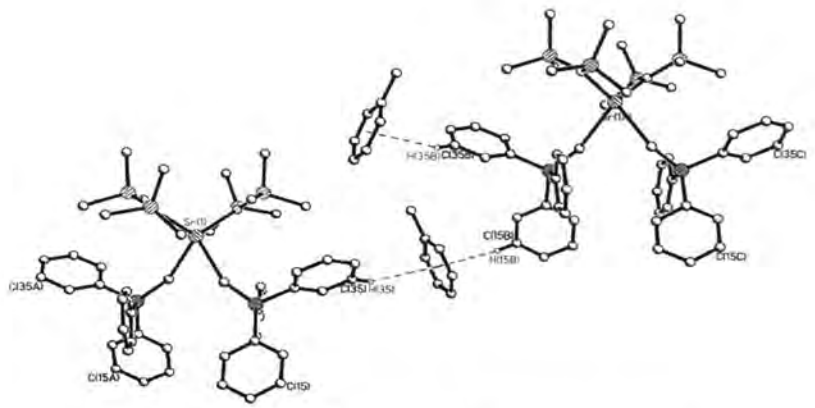


Diagram 6.6:Hydrogen bonding patterns, A) calcium species, B) strontium species.

Table 6.3: Hydrogen bonding distances for calcium and strontium species.

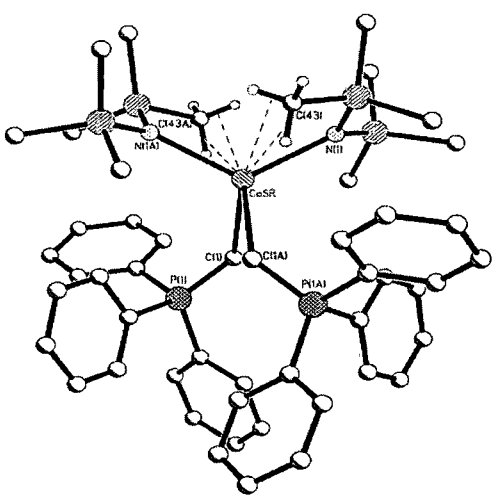
TYPE: A...H...B	RING CENTER A	ATOM H	ATOM B	DISTANCE A...B /Å		DISTANCE A...H /Å		ANGLE A-H-B /°	
				Calcium	Strontium	Calcium	Strontium	Calcium	Strontium
π...H...C	C(1X)-C(6X)	H(15)	C(15)	3.624(11)	3.596(12)	2.885(11)	2.875(12)	135.5(2)	135.3(3)
π...H...C	C(1X)-C(6X)	H(35)	C(35)	3.803(11)	3.777(11)	2.992(11)	2.973(11)	144.1(2)	145.6(3)

It can be seen that there is a good agreement between the hydrogen bond lengths/angles for the calcium and strontium species. It is also worth noting that the bond lengths in the strontium species are shorter than in the calcium species, this agrees with the theory that the close agreement in the cell size is due to the molecule expanding into the empty space that is partially filled by the solvent.

The barium species exhibits completely different space group (with lower symmetry), different packing and a different molecular structure. There is no longer any solvent included in the structure, which could of course be the reason for the difference in structure since different morphologies often can be obtained by varying the solvent (see chapter 3). However, since in this case the preparatory conditions for all the compounds are identical, the lack of solvent in the crystal must be a feature of the packing structure. There are no hydrogen bonds of any type in this compound.

A final point about the compounds in this series is the occurrence of agostic type bonds[7]. An agostic bond is where the C-H sigma bond interacts with an electron deficient metal centre in a three-centre two-electron interaction. Interactions of this type are very important in the formation of transition intermediates during metal catalysis reactions. In this case, the compounds exhibit an unusually short C-H...metal distance, and a distinct distortion in the metal centre geometry from the ideal tetrahedron to a distorted octahedron.

A:



B:

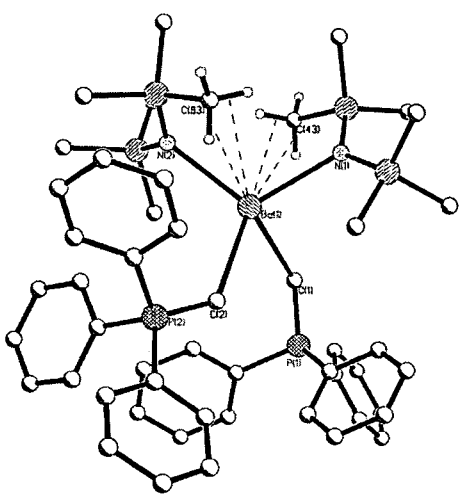


Diagram 6.7:Agostic bonds in bis(hexamethyldisilylamide) bis(triphenylmethylphosphonium)-metal compounds, A) calcium/strontium species, B) barium species

The compounds exhibit four of these 'unusually short' C-H...metal distances, from two methyl groups with two agostic bonds per methyl group, (in the calcium / strontium species the methyl groups are symmetrically related since the metal lies on a special position).

Table 6.4: Agostic bond distances.

	C...M	Distance /Å	H...M	Distance /Å	σ...M	Distance /Å
Ca	C(43)...Ca	3.15(3)	H(43A)...Ca	2.94(5)	C(43)-H(43A) | Ca	3.04(4)
			H(43C)...Ca	2.88(5)	C(43)-H(43C) | Ca	3.06(4)
Sr	C(43)...Sr	3.19(3)	H(43A)...Sr	2.95(5)	C(43)-H(43A) | Sr	3.01(4)
			H(43C)...Sr	2.90(5)	C(43)-H(43C) | Sr	3.06(4)
Ba	C(43)..Ba	3.42(3)	H(43A)...Ba	3.17(9)	C(43)-H(43A) | Ba	3.28(9)
			H(43C)...Ba	3.12(9)	C(43)-H(43C) | Ba	3.22(9)
Ba	C(83)..Ba	3.49(3)	H(83A)...Ba	3.30(9)	C(83)-H(83A) | Ba	3.32(9)
			H(83B)...Ba	3.13(9)	C(83)-H(83B) | Ba	3.23(9)

Angles about the metal for each species

Angles in Calcium bis(hexamethyldisilylamide) bis(triphenylmethylphosphonium)

N1_$1	121.06 (0.37)						
C1_$1	111.72 (0.28)	111.69 (0.25)					
C1	111.69 (0.25)	111.72 (0.28)	82.51 (0.46)				
X1B_$1	66.06 (0.37)	152.23 (0.42)	73.69 (0.37)				
X1B	66.06 (0.37)	90.61 (0.42)	73.69 (0.37)	152.23 (0.42)	132.63 (0.62)		
X1A_$1	77.37 (0.35)	65.15 (0.40)	169.66 (0.39)	89.57 (0.45)	17.64 (0.51)	115.55 (0.45)	
X1A	65.15 (0.40)	77.37 (0.34)	89.57 (0.45)	169.66 (0.39)	115.55 (0.45)	17.64 (0.51)	99.02 (0.76)
Ca1-	N1	N1_$1	C1_$1	C1	X1B_$1	X1B	X1A_$1

Angles in Strontium bis(hexamethyldisilylamide) bis(triphenylmethylphosphonium)

N1	120.01 (0.33)						
C1	113.56 (0.27)	111.10 (0.26)					
C1_$1	111.10 (0.26)	113.56 (0.27)	81.08 (0.40)				
X1B_$1	64.23 (0.50)	90.88 (0.54)	152.84 (0.44)	75.40 (0.38)			
X1B	90.88 (0.54)	64.23 (0.50)	75.40 (0.38)	152.84 (0.44)	130.39 (0.66)		
X1A_$1	63.89 (0.53)	79.35 (0.37)	167.50 (0.39)	88.49 (0.51)	14.71 (0.60)	116.28 (0.47)	
X1A	79.35 (0.37)	63.89 (0.53)	88.49 (0.51)	167.50 (0.39)	116.28 (0.47)	14.71 (0.60)	102.64 (0.89)
Sr1-	N1_$1	N1	C1	C1_$1	X1B_$1	X1B	X1A_$1

Angles in Barium bis(hexamethyldisilylamide) bis(triphenylmethylphosphonium)

N2	112.08 (0.22)						
C2	122.97 (0.24)	109.95 (0.24)					
C1	92.36 (0.24)	125.58 (0.25)	93.10 (0.25)				
X1B	57.02 (0.49)	68.85 (0.48)	178.25 (0.47)	88.64 (0.47)			
X1D	85.67 (0.51)	57.60 (0.54)	85.86 (0.44)	176.79 (0.56)	92.40 (0.59)		
X1A	56.01 (0.52)	84.17 (0.48)	162.78 (0.48)	70.21 (0.48)	18.94 (0.56)	110.57 (0.60)	
X1C	72.35 (0.52)	55.82 (0.54)	103.18 (0.45)	161.96 (0.46)	75.11 (0.59)	17.96 (0.56)	92.86 (0.61)
Ba1-	N1	N2	C2	C1	X1B	X1D	X1A

Conclusions:

The effect of changing the ligand set (i.e. changing between the two series) is to change completely the structure of the crystal. This indicates that the crystal structure is dependent on the overall molecular properties or shape, not the metal type.

In both series the same relationship between metal type and crystal structure in seen, with calcium and strontium compounds showing the same structure as each other, but these are both different from the barium structures. Given that the ionic radii[1] for each ion are 0.99Å for Ca^{2+}, 1.12Å for Sr^{2+}, and 1.34Å for Ba^{2+} it can be seen that the increase in size in going from Ca^{2+} to Sr^{2+} is less than the increase in size in going from Sr^{2+} to Ba^{2+}. This almost certainly explains many of the changes in crystal structure observed.

A very similar compound to those of series 2 is studied in chapter 8. This compound, [sodium-(hexamethyldisilylamide) (triphenylethylphosphonium)] exhibits a slight change in the phosphonium ligand and has the alkali metal sodium in place of the alkaline earth metal seen in series 2. The alkali metal has of course an oxidation state of +1 and so is only bound to two ligands not the four seen in series 1 and 2. It can be seen that the compound shows a completely different molecular structure forming a dimer, and of course the crystal has a completely different packing arrangement from those seen in this chapter.

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7) a)M.Brookhart, M.L.H.Green. *J. Organomet. Chem.* 1983, **250,** 395.

b)P.L.A.Popelier, G.Logothetis. *J. Organomet. Chem.* 1998, **555,** 101.

c)D.Braga, F.Grepioni, K,Biradha, G.R.Desiraju. *J. Chem. Soc., Dalton Trans.* 1996, **20,** 3925

Chapter 7

Crystals Studied: Miscellaneous Organics

[5,6'(5',8'-dihydroxy-1',4'-naphthoquinone)]-2-methylpent-2-ene phenyl acrylate

Crystal Data:

Name:	[5,6'(5',8'-dihydroxy-1',4'-naphthoquinone)]-2-methylpent-2-ene phenyl acrylate
Formula	C25 H22 O6
Colour	Red
Morphology	Block
Size	0.5 × 0.1 × 0.1 mm
Instrument	Smart CCD[1]
Temp/K	100 (2)
a	6.16770(10)Å
b	15.7416(3)Å
c	10.5141(2)Å
α	90°
β	90.7630(10)°
γ	90°
Volume / z	977.4(3) $Å^3$ / 2
Crystal system	Monoclinic
Space group	P2(1)
R(int)	0.0276
Absorption correction	Psi scan[2]
Mu mm^{-1}	0.097
Total data unique	4360
Data I>2σ	4011
R1 (all data)	0.0489
Other	The crystals were air/moisture sensitive and also sensitive to sunlight. The extent of the sensitivity was unknown, so the crystals were treated as very sensitive and mounted quickly under oil (perfloropolyether M.W. 2700). The data were collected on the Smart CCD area detector[1], since data collection was much faster with an area detector than on the Rigaku 4 circle diffractometer[3]. The setting up of the crystal and the data collection were carried out, as far as possible, in the dark, and at low temperature (100K). In fact the crystals were stable enough to last over two weeks under oil without any visible decay, but by the time this had been discovered the data collection had been long completed!
See also Appendix B:	Compound 12, page 187

Data Solution and refinement:

The structure of the molecule was unknown, only the molecular formula (C25 H22 O6) was given. The structure was therefore analysed by fitting the q peak patterns to known chemical groups. The benzene ring and the naphthoquinone rings were easy to identify by their shape. The alchohol and aldehyde groups, on the naphthoquinone or ester, were distinguishable by the bond lengths. The ether oxygen on the ester had originally been input as a carbon, but it became apparent both from the bond lengths, and the apperence of a q peak ≈ 0.33Å from the atom in the correct position for a lone pair of electrons on an oxygen atom, that the atom was actually an oxygen. The structure was checked by careful study of all bond lengths and angles and comparison to known bond lengths for identical chemical environments, taken from the Cambridge Structural Database[4], and also by careful checking of the position of the residual q peaks. It is worth carrying out similar checks even if the structure modelled is identical to the expected structure.

Crystal structure and packing:

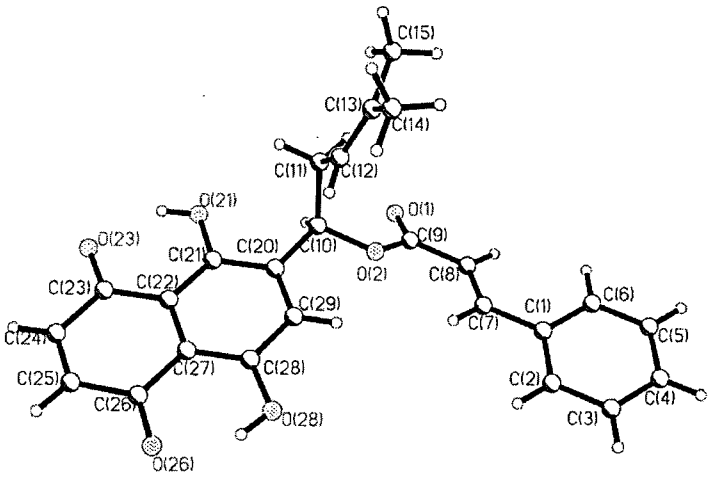


Diagram 7.1: [5,6'(5',8'-dihydroxy-1',4'-naphthoquinone)]-2-methylpent-2-ene phenyl acrylate.

The compound had two alcohol groups on the naphthoquinone as well as the two aldehyde groups, and the ester of course had another aldehyde group and the linking ether oxygen. It would seem reasonable to expect that these alcohol groups would be involved in intermolecular hydrogen bonding to build up a lattice framework. At least, this is a reasonable assumption until the position of the OH group is considered: The bulk of the molecule beyond the naphthoquinone reduces the ways a second molecule could position to form a hydrogen bond, and the two aldehyde groups on the 1 and 4 positions of the naphthoquinone group are in perfect position to form intramolecular hydrogen bonds to the alcohol groups.

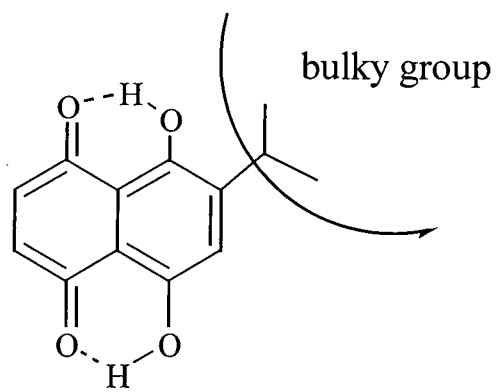


Diagram 7.2: why the alcohol groups on 5,8-dihydroxy-1,4-naphthoquinone forms intra not intermolecular bonds.

As there is a lack of O-H hydrogen bond donors, the crystal lattice is stabilised by weaker O..H..C hydrogen bonds. A bond between O(1), the C=O of the ester, and H(25), on the 2 position of the naphthoquinone group, forms chains of molecules with the naphthoquinone group fitting into the space between the ester and the $CH_2CH=C(Me)_2$ branches.

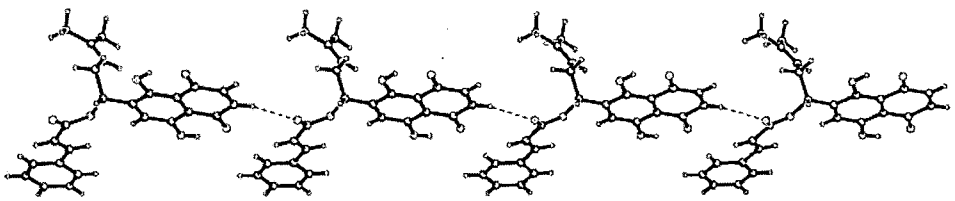


Diagram 7.3: the chain formed by a hydrogen bond between O(1) and H(25).

These chains are then linked together by a bond between O(28), one of the alcohol oxygens, and H(10), the hydrogen on the carbon C(10) the tertiary carbon in the middle of the molecule. So each molecule is stacked in columns with all the ester branches pointing in the same direction and nearly superimposed. This gives a sheet of molecules.

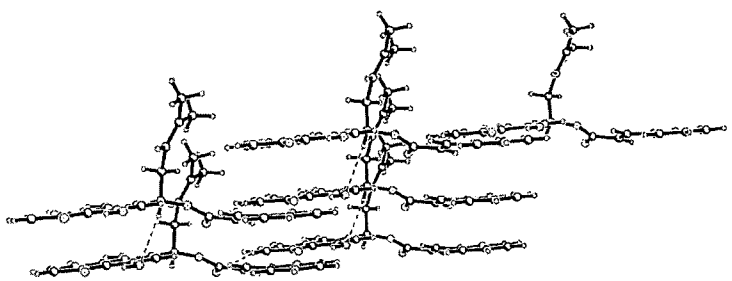


Diagram 7.4: the sheet formed by a hydrogen bond between O(28) and H(10) on separate chains.

The sheets are then packed on top of each other such that each sheet had been reflected horizontally before being stacked vertically.

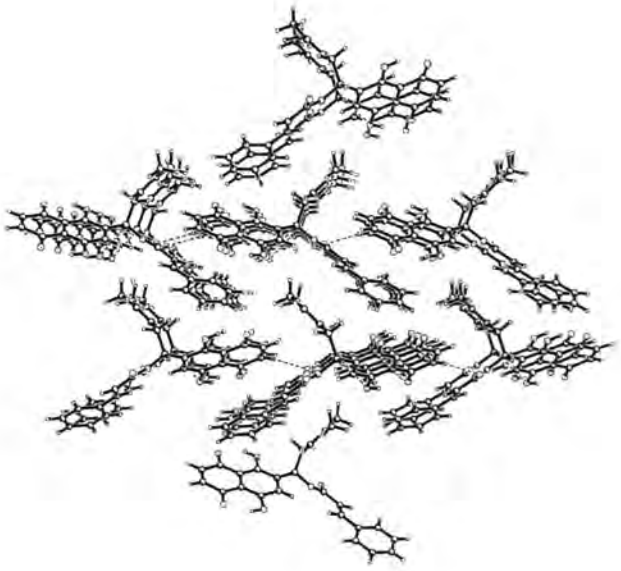


Diagram 7.5: the packing of the sheets.

Table 7.1: Hydrogen bond distances.

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...O	O(23)	H(21)	O(21)	2.589(2)	1.856(7)	144.90(1.02‡)
O...H...O	O(26)	H(28)	O(28)	2.587(2)	1.846(2)	146.23(8)
O...H...C	O(28)	H(10)	C(10)	3.357(3)	2.547(3)	137.91(6)
O...H...C	O(1)	H(25)	C(25)	3.356(3)	2.450(3)	159.37(7)

‡ The exceptionally large error in this angle is probably due to a wrong assumption in the model. The hydrogen was generated in an ideal position and the thermal motion, U, only allowed to refine. In-fact this hydrogen is probably not in this ideal position. But, given the way the model is fixed, the only way it can allow for this wrongly positioned hydrogen atom, is to increase the value of U for this hydrogen atom, and thus increase the error. A better model would be to allow this hydrogen atom to refine freely.

(2-methyl(diethanoic acid)amine)phenoxyethanoic acid

Crystal Data:

Name:	(2-methyl(diethanoic acid)amine)phenoxyethanoic acid
Formula	C13 H15 O7 N1
Colour	Brown
Morphology	Plate
Size	0.25 × 0.20 × 0.05 mm
Instrument	Smart CCD[1]
Temp/K	296
a	10.6417(3)Å
b	9.6898(3)Å
c	13.1969(4)Å
α	90°
β	94.031(2)°
γ	90°
Volume / z	1357.44(7) $Å^3$ / 4
Crystal system	Monoclinic
Space group	P2(1)/n
R(int)	0.2209
Absorption correction	Multiscan[5]
Mu mm^{-1}	0.120
Total data unique	3110
Data I>2σ	1036
R1 (all data)	0.2747
Other	The data were collected at room temperature because the cryostream was temporarily out of use and this was a structure that did not require low temperature analysis. The crystal was quite a poor diffractor at room temperature but enough data were collected to solve the structure.
See also Appendix B:	Compound 13, page 191

Crystal structure and packing:

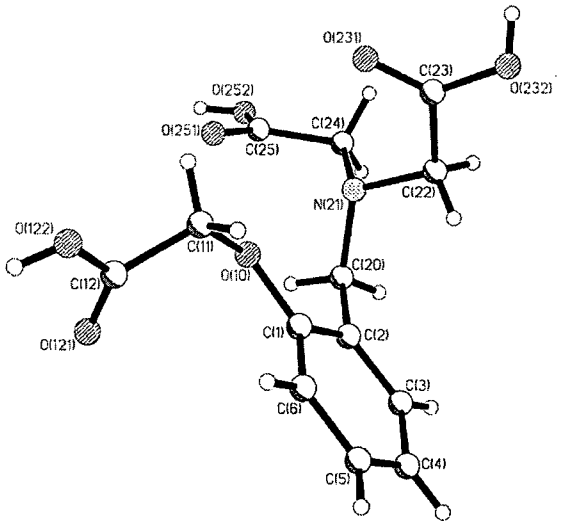


Diagram 7.6: (2-methyl(diethanoic acid)amine)phenoxyethanoic acid

The first thing to notice about the packing of the crystal is the way all the phenyl rings are aligned in the same orientation, which when viewed from the side appear to be in rising rows like steps, however these steps are in-fact not orientated directly over each other. This alignment of planes (though not necessarily this pattern) is a fairly common arrangement for benzene rings and other flat systems to take.

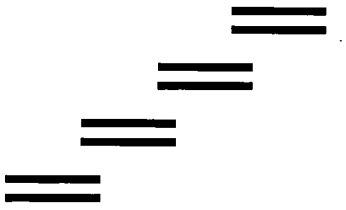


Diagram 7.7:schematic of the packing of the benzene rings in the crystal, viewed down the a axis (phenyl rings in cross-section).

The next point to note is that none of the three carboxylic acid groups have formed the dimer system that is so often seen with this type group, and which has been suggested as a crystal engineering 'synthon'[6].

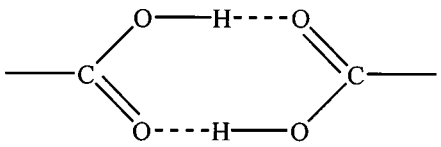


Diagram 7.8: carboxylic acid dimer

This dimer may be the commonly observed arrangement of hydrogen bonds for carboxylic acid systems, but this is not how the bonding occurs in this crystal. Instead two of the acid groups form a single hydrogen bond each, to the reciprocal groups another molecule related by a central inversion centre, i.e.:

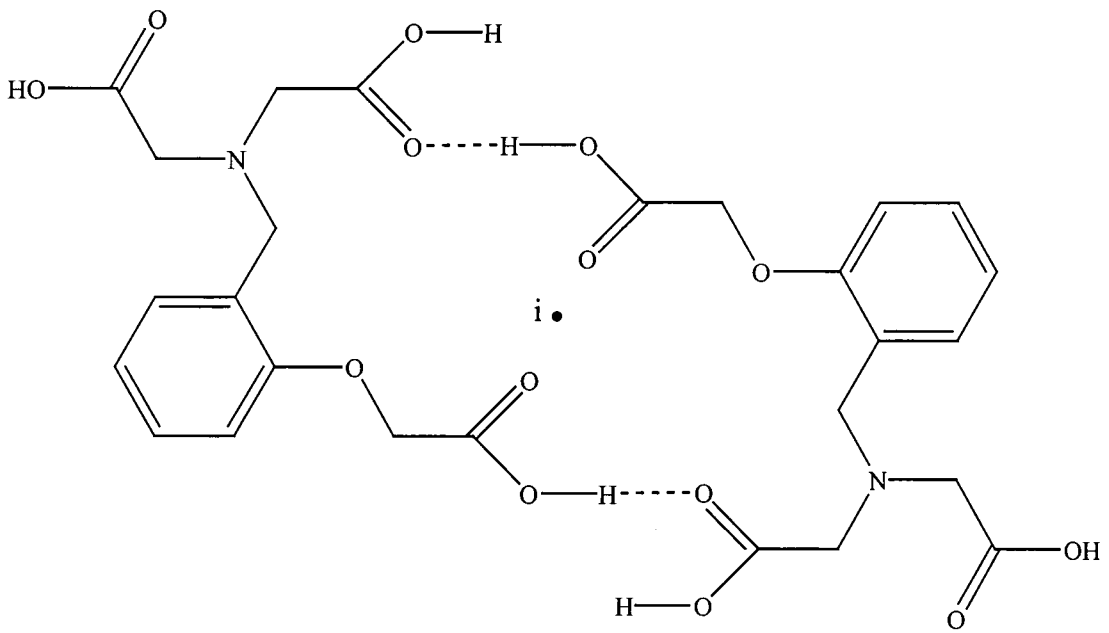


Diagram 4.9: hydrogen bonding between two molecules about an inversion centre.

From this diagram it looks as if the molecules could easily shift in relation to each other to give two of the stable synthons shown above, resulting in a very securely bonded dimer as shown below:

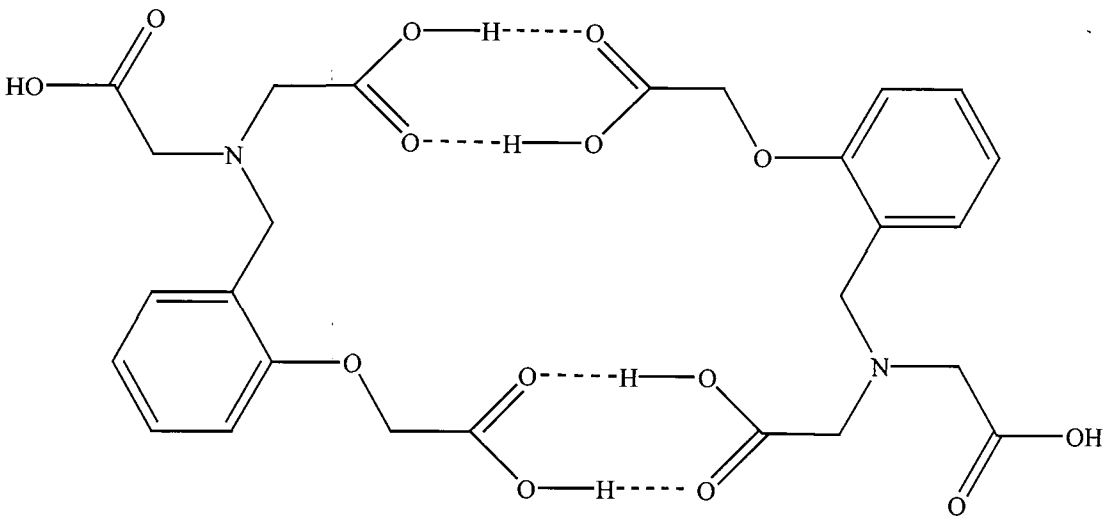


Diagram 7.10: Possible hydrogen bonding to give a dimer linked by four hydrogen bonds. Not exhibited by the crystal.

But instead the two OH groups from the acids in question form hydrogen bonds to the, as yet, unused acid group on another dimer, that is related by a translation. Thus the hydrogen bonding forms a chain running through the whole crystal rather than creating stable dimers that are than only weakly bonded together. This is advantageous since the whole crystal is then held together by hydrogen bonds running the length of the crystal, instead of just creating larger molecules that still have to find a way of packing by some other weak interactions, and have the disadvantage of having already lost/weakened four possible interaction sites. It is perhaps clearer to see this in the actual structure than in schematic drawings.

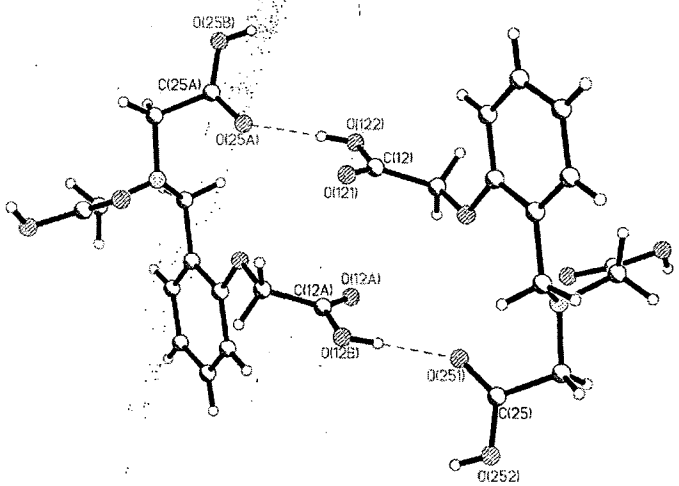


Diagram 7.11: dimer.

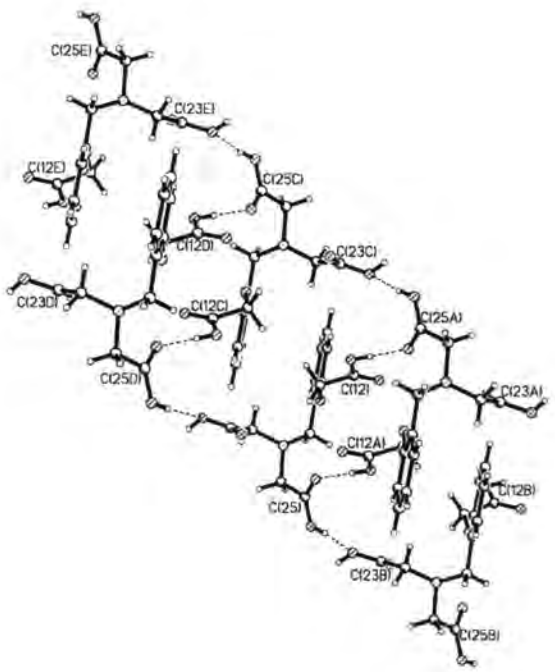


Diagram 7.12: extended chain.

The hydrogen bond sites as yet unused are all hydrogen bond acceptors (either =O of the acids or donation site on the oxygen of an OH group already used in bonding). These form links to any nearby CH groups (either secondary CH_2 attached to an electronegative N or an aromatic). i.e. form C-H..O hydrogen bonds. These weaker hydrogen bonds hold the chains together.

Table 7.2: Hydrogen bond distances.

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
O...H...O	O(251)	H(12)	O(122)	2.677(6)	1.872(10)	166.80(3.72)‡
O...H...O	O(232)	H(25)	O(252)	2.469(6)	1.655(69)‡	171.44(2.26)‡
O...H...C	O(121)	H(24B)	C(24)	3.271(7)	2.322(7)	166.02(0.18)
O...H...C	O(231)	H(24A)	C(24)	3.426(8)	2.466(8)	170.33(0.16)
O...H...C	O(251)	H(22B)	C(22)	3.319(7)	2.374(7)	164.70(0.16)
O...H...C	O(252)	H(5)	C(5)	3.251(8)	2.564(8)	131.16(0.18)

‡ The exceptionally large error in these angles and distance is probably due to a wrong assumption in the model. The hydrogen atoms were generated in the ideal positions and the thermal motion, U, only allowed to refine. In-fact the hydrogen atoms were probably not in these ideal positions. But, given the way the model is fixed, the only way it can allow for a wrongly positioned hydrogen atom, is to increase the value of U for this hydrogen atom, and thus increase the error. A better model would be to allow these hydrogen atoms to refine freely.

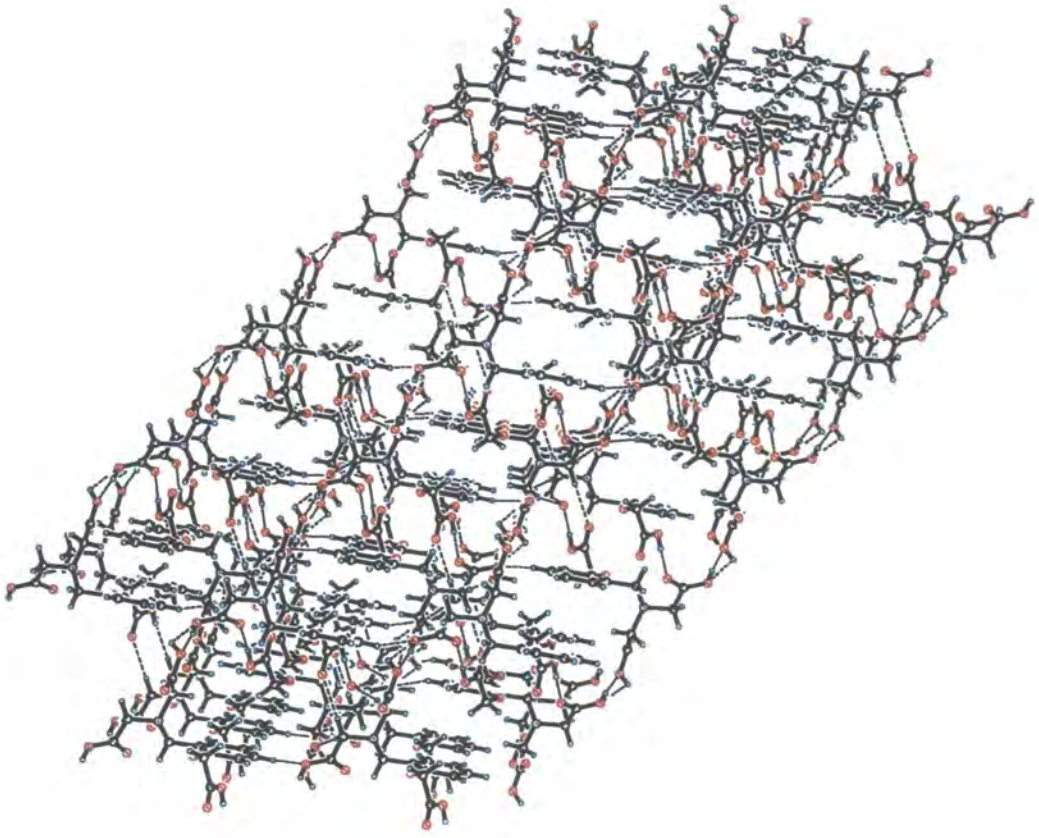


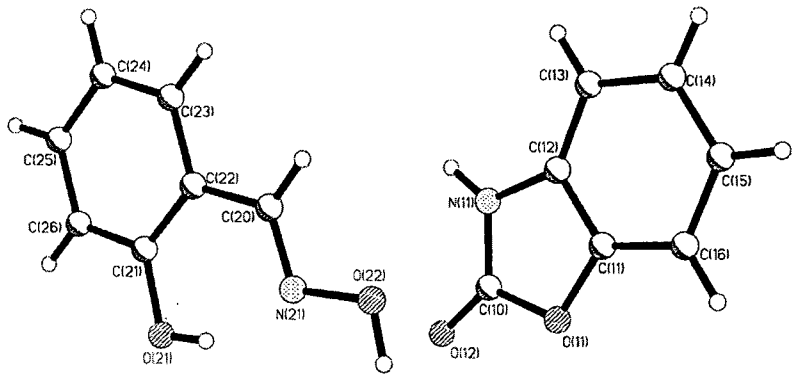
Diagram 7.13: Full structure.

Salicylaldoxime and Benoxazolin-2-one

Crystal Data:

Name:	Salicylaldoxime and Benoxazolin-2-one
Formula	C7 H7 N1 O2 / C7 H3 N1 O2
Colour	Colourless
Morphology	Prism
Size	0.35 × 0.12 × 0.06 mm
Instrument	Rigaku 4-circle diffractometer[3]
Temp/K	150 (2)
a	10.494(2)Å
b	6.435(1)Å
c	19.604(4)Å
α	90°
β	103.36(3)°
γ	90°
Volume / z	1287.9(4) $Å^3$ / 4
Crystal system	Monoclinic
Space group	P2(1)/n
R(int)	0.0209
Absorption correction	None
Mu mm^{-1}	0.105
Total data unique	2254
Data I>2σ	1770
R1 (all data)	0.0618
See also Appendix B:	Compound 14, page 191

Crystal structure and packing:



Diagam 7.14: Benzoxazolin-2-one and Salicylaldoxime

This co-crystal contains three classical hydrogen bond donors and five classical hydrogen bond acceptors. The crystal packs in such as to utilise all of the classical hydrogen bond donors and all, bar one, of the classical hydrogen bond acceptors in either inter or intra molecular bonding. In addition there is one occurrence of a non-classical hydrogen bond. While the over all structure is very complexed it can easily be understood if considered one step at a time. First the benzoxazolin-2-one forms a dimer:

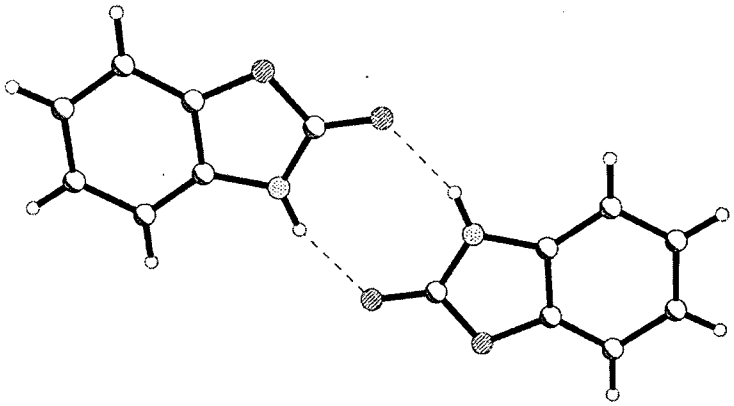


Diagram 7.15: the benzoxazolin-2-one forms a dimer.

Each of these dimers is surrounded by four salicylaldoxime molecules: two forming classical hydrogen bonds from O(22)-H to O(12) the hydrogen bond acceptor already used in the dimer formation, and two forming non-classical bonds from O(22) to a CH of the phenyl ring.

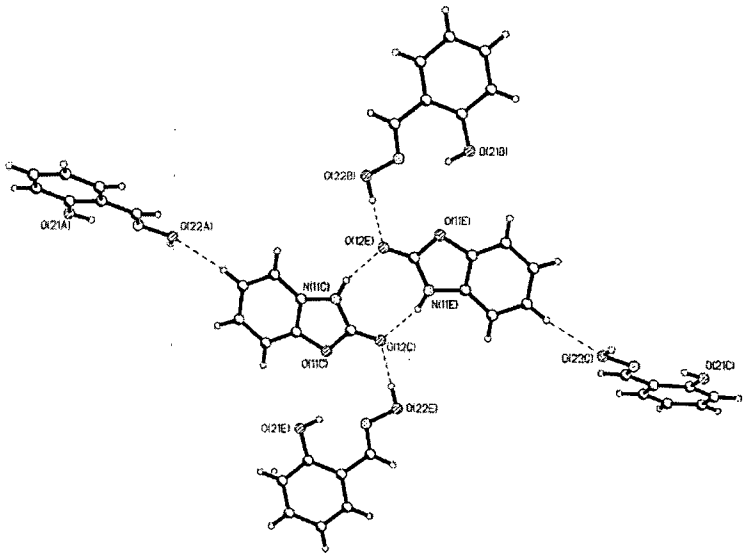


Diagram 7.16: Benzoxazolin-2-one dimer surrounded by four salicylaldoxime molecules.

When considered from the point of view of the salicylaldoxime molecule instead of the benzoxazolin-2-one dimer it is seen that each salicylaldoxime molecule is hydrogen bonded to two benzoxazolin-2-one dimers through O(22) with the remaining hydrogen bond donor and acceptor involved in an intramolecular interaction.

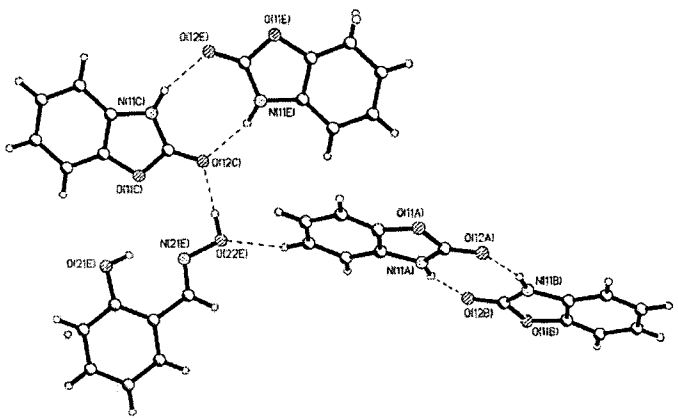


Diagram 7.17:A salicylaldoxime molecule hydrogen bonded to two benzoxazolin-2-one dimers.

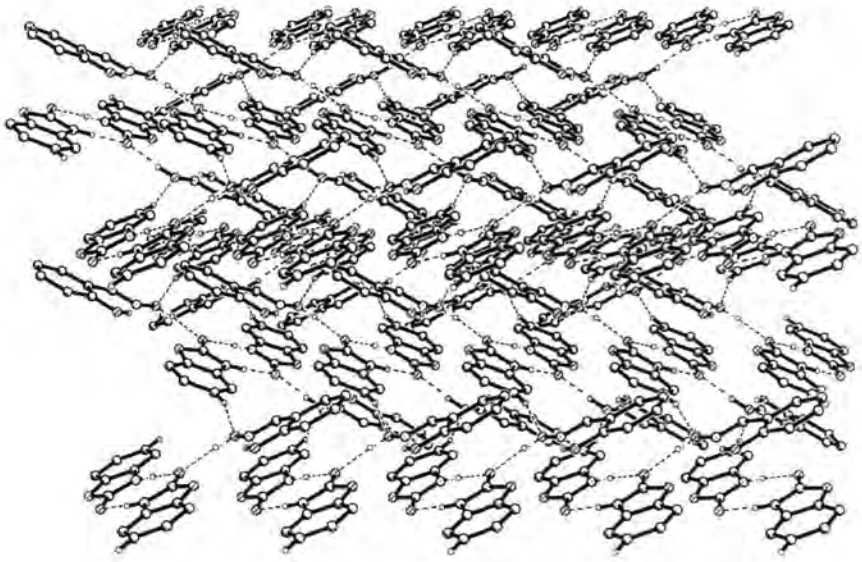


Diagram 7.18: Full structure.

Table 7.3: Hydrogen bond distances.

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
N...H...O	N(11)	H(11)	O(12)	2.827(2)	1.97(3)	161(2)
O...H...O	O(22)	H(22)	O(12)	2.725(2)	1.81(3)	175(3)
O...H...C	O(22)	H(14)	C(14)	3.375(3)	2.45(1)	163(1)
N...H...O	N(21)	H(21)	O(21)	2.648(2)	1.86(3)	150(2)

Conclusions:

The compounds studied in this chapter are three very varied organic molecules. It is clear that the molecules have packed both to optimise the hydrogen bonding and especially to optimise the formation of extended hydrogen bonded networks.

In all the cases weaker non-classical hydrogen bonds of the O..H-C type are utilised, as well as the classical O..H-O type and O..H-N, to form the extended network.

The third compound, the salicylaldoxime benoxazolin-2-one co-crystal, forms CONH dimers analogous to the COOH dimers that are conspicuously lacking in the (2-methyl (diethanoic acid)amine)phenoxyethanoic acid structure.

Only one of the hydrogen bonds in (2-methyl(diethanoic acid)amine)phenoxyethanoic acid and none in [5,6'(5',8'-dihydroxy-1',4'-naphthoquinone)]-2-methylpent-2-ene phenyl acrylate or in the salicylaldoxime benoxazolin-2-one co-crystal are short enough to be classed as strong hydrogen bonds as defined by Emsley[7] (0.3Å shorter than the sum of the Van der Waals radii of atoms A and B of the hydrogen bond A..H-B). Indeed the weaker O..H-C bonds are in many cases longer than the sum of the Van der Waals radii.

The A..H-B angles show a linear tendency but are not fully linear. They are closer to linear in (2-methyl(diethanoic acid)amine)phenoxyethanoic acid and the salicylaldoxime benoxazolin-2-one co-crystal than in [5,6'(5',8'-dihydroxy-1',4'-naphthoquinone)]-2-methylpent-2-ene phenyl acrylate where all the hydrogen bonds are either internal or of the weak non-classical type.

References:

1) SMART Version 4.050. Seines Analytical X-ray Instruments, Madison, U.S.A., 1995

2) G.M.Sheldrick, (1994) SHELXTL-Plus Release 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, U.S.A.

3) a) MSC/AFC. Molecular Structure Corporation (1991).Molecular Structure Corporation (1991). MSC/AFC Diffractometer Control Software. (1991). MSC, 3200 Research Forest Drive, Woodlands, TX77381, U.S.A.

4) F.H.Allen, O.Kennard. *Chem. Des. Autom. News.* 1993, **8**, 1.

5) G.M.Sheldrick, (1996), SADABS, Program for the correction of area detector data, University of Göttingen, Germany.

6) G.R.Desiraju. Angew. *Chem. Int. Ed. Engl.*1995, **34,** 2311.

7) J.Emsley. *Chem. Soc. Rev.* 1980, **9**, 91.

Chapter 8

Crystals Studied: Miscellaneous Organometallics

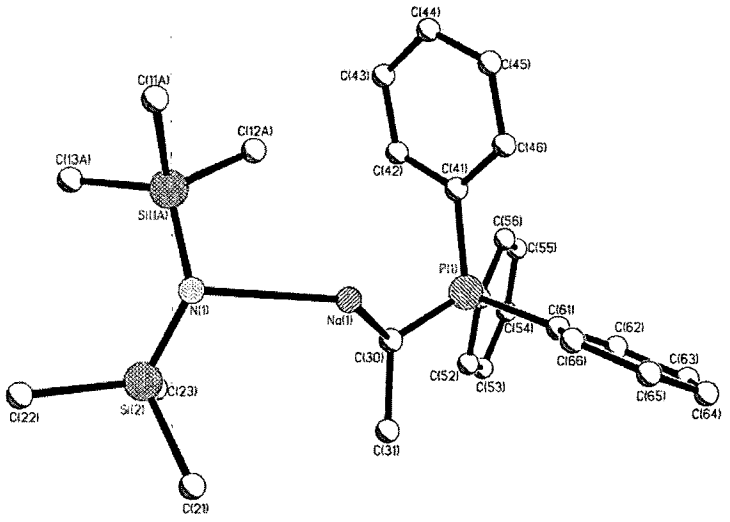
Sodium (hexamethyldisilylamide) (triphenylethylphosphonium)

Crystal Data:

Name:	Sodium(hexamethyldisilylamide)(triphenylethylphosphonium)
Formula	C26 H37 P1 N1 Si2 Na
Colour	Yellow
Morphology	Block
Size	0.42 × 0.38 × 0.10 mm
Instrument	Smart CCD[1]
Temp/K	150 (2)
a	10.0510(2)Å
b	10.3864(2)Å
c	14.3005(3)Å
α	105.765(1)°
β	99.254(1)°
γ	102.194(1)°
Volume / z	1366.02(7)$Å^3$ / 2
Crystal system	Triclinic
Space group	P-1
R(int)	0.0334
Absorption correction	Multiscan[2]
Mu mm^{-1}	0.218
Total data unique	7130
Data I>2σ	5105
R1 (all data)	0.0887
See also Appendix B:	Compound 15, page 197

Crystal structure and Packing

A:



B:

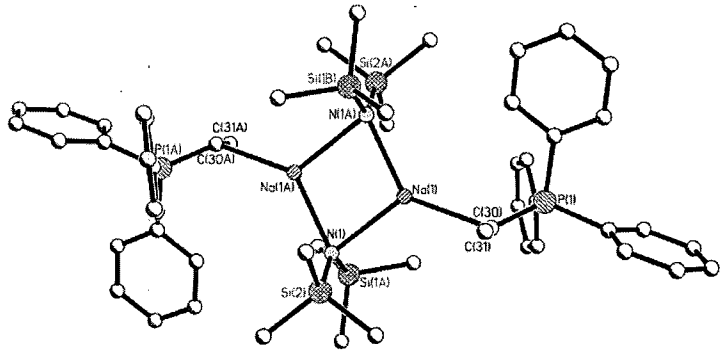


Diagram 8.1: A) sodium (hexamethydilsilylamide)(triphenyethylphosphonium). B) dimer of sodium (hexamethydilsilylamide)(triphenyethylphosphonium).

There are no classical hydrogen bond donors or acceptors in this molecule, the only hydrogen bonding interaction possible is the weak non-classical C-H…π bond of the type seen in the very similar alkali earth compounds discussed in chapter 5, however this interaction is not seen here.

The molecule forms a dimer about an inversion centre, with the nitrogen and silicon acting as a bridge between the two molecules. The sodium – sodium distance in the dimer is 3.135(2)Å The dimers align in columns such that the columns are parallel to the b axis. The columns pack in a regular array.

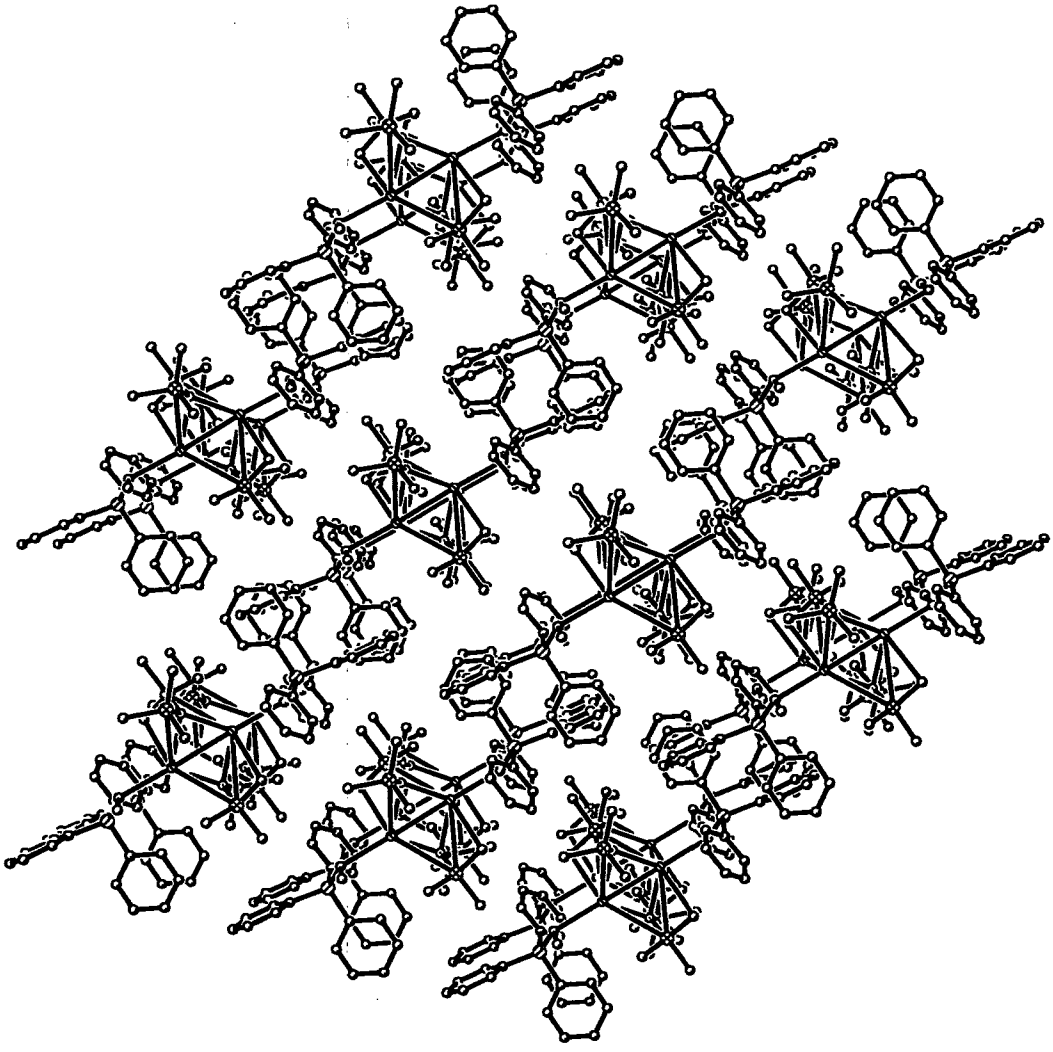


Diagram 8.2: packing of the dimers viewed down the a axis.

Like the alkali earth compounds discussed in chapter 6, this compound exhibits agostic bonds[3].

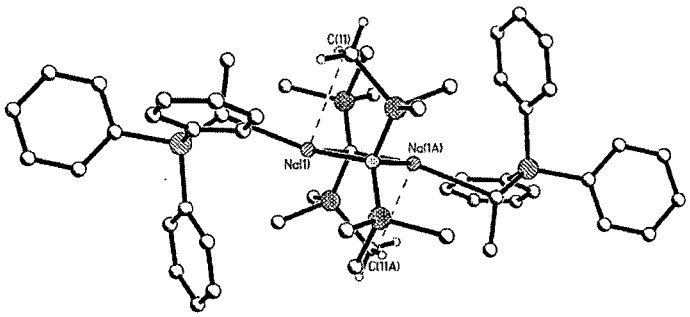


Diagram 8.3: Agostic bonds in Sodium(hexamethyldisilylamide)(triphenylethylphosphonium).

Table 8.1: Agostic bond distances.

C...M	Distance /Å	H...M	Distance /Å	σ...M	Distance /Å
C(12)...Na	3.04(1)	H(121)...Na	2.97(12)	C(12)-H(121) | Na	2.96(2)
		H(122)...Na	2.69(12)	C(12)-H(122) | Na	2.83(2)

5-ferrocenyl-4-nitrile-7-acetonitrile-2-(4'-bromophenyl)indole

Crystal Data:

Name:	5-ferrocenyl-4-nitrile-7-acetonitrile-2-(4'-bromophenyl)indole
Formula	C27 H17 Br Fe N3
Colour	Red
Morphology	Plate
Size	0.25 × 0.15 × 0.06 mm
Instrument	Smart CCD[1]
Temp/K	150 (2)
a	7.9608(1) Å
b	10.6844(1) Å
c	12.8215(3) Å
α	93.577(1)°
β	97.737(1)°
γ	99.046(1)°
Volume / z	1063.28(3) $Å^3$ / 2
Crystal system	Triclinic
Space group	P-1
R(int)	0.0403
Absorption correction	Psi scan[4]
Mu mm^{-1}	2.609
Total data unique	4823
Data I>2σ	3953
R1 (all data)	0.0713
See also Appendix B:	Compound 16, page 202

Data solution and refinement:

The structure of the molecule was unknown, only the molecular formula (C27 H17 N3 Fe Br) was given. Direct methods identified the iron and bromine atoms but the rest of the structure was analysed by fitting the q peak patterns to known chemical groups. For instance, the ferrocene was easy to locate, as was the benzene ring, the indole group was at first input as an indene group (i.e. the nitrogen was input as a carbon) until careful analysis of the bond lengths proved otherwise. The structure was checked by careful study of all bond lengths and angles, and comparison to known bond lengths for identical chemical environments, taken from the Cambridge database[5], and also by careful checking of the position of the residual q peaks. It is worth carrying out similar checks even if the structure modelled is identical to the expected structure.

Crystal structure and Packing:

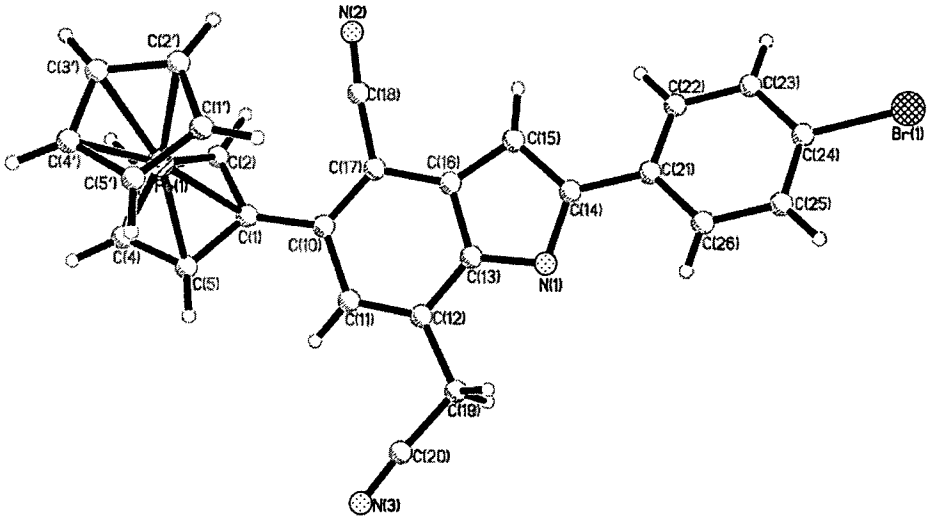


Diagram 8.4: : 5-ferrocenyl-4-nitrile-7-acetonitrile-2-(4'-bromophenyl)indole

In this compound there are no classical (N-H, O-H) hydrogen bond donors although there are plenty of strong hydrogen bond acceptors. The hydrogen bonds therefore form from the C-H, to the strong acceptors.

The packing of the molecule can be analysed in terms of dimers, which link to form chains, which link to form sheets, which stack to give the 3-Dimensional crystal structure. The dimers are made up of two molecules related by an inversion centre. Hydrogen bonds link N(2) on one molecule to H(15)-C(15) and H(22)-C(22) on the second, inversion related, molecule. Also, since related by an inversion centre, the N(2) on the second molecule is hydrogen bonded to H(15)-C(15) and H(22)-C(22) on the first. So although there are no N-H groups to form strong classical hydrogen bonds, the dimer is held together by four N..H-C hydrogen bonds where the H-C is part of an aromatic group, thus giving a strongly bound system.

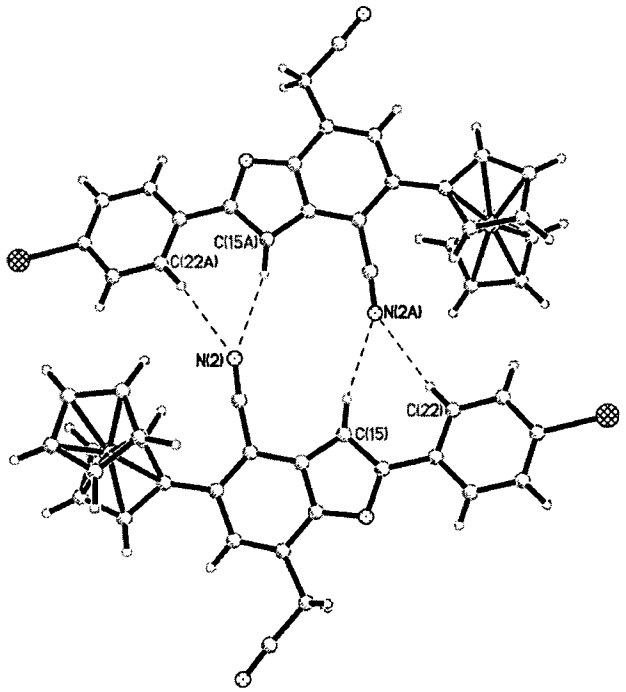


Diagram 8.5: the hydrogen bonded dimer

The dimers are linked to the next dimer along by an interaction between the bromine (Br1) and H(19A)-C(19) on the next dimer. The two dimers are, again, related by an inversion centre, so the interaction is again reciprocal. This forms chains of dimers.

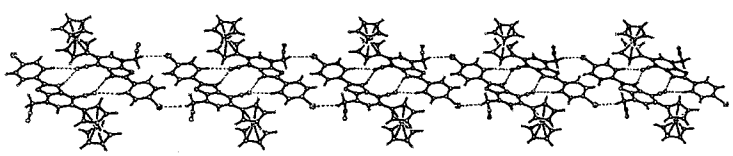


Diagram 8.6: a chain of dimers.

The sheets of molecules are built up by a hydrogen bond between N(4) and H(1')-C(1') (of the ferrocene) on the molecule directly adjacent to it in the a-direction (related by translation in a)

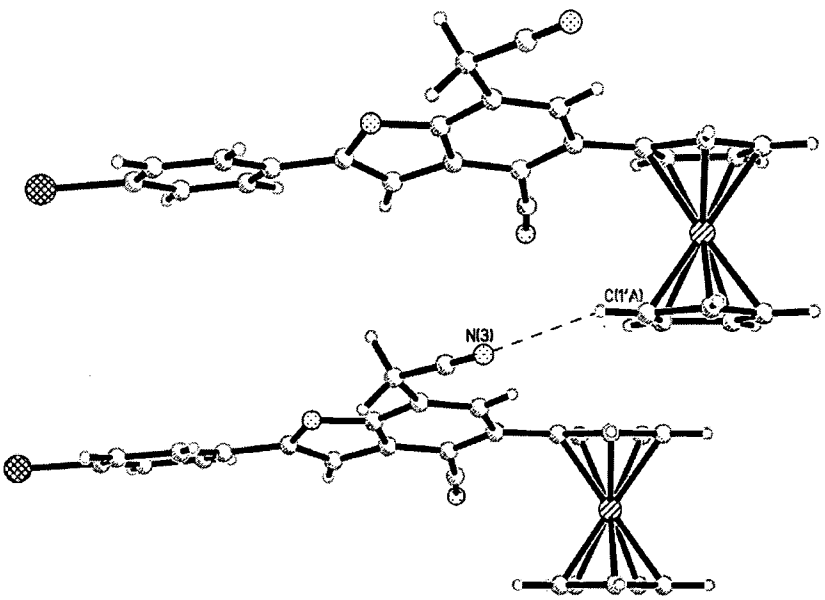


Diagram 8.7: the hydrogen bond that holds the chain together to give sheets.

The sheets stack together to give a 3-Dimensional lattice, but there is no direct bonding between the sheets.

Table 8.2: Hydrogen bond distance.

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
N...H...C	N(2)	H(15)	C(15)	3.440(6)	2.510(6)	161.76(14)
N...H...C	N(2)	H(22)	C(22)	3.426(6)	2.510(6)	161.85(15)
N...H...C	N(3)	H(1')	C(1')	3.356(6)	2.547(6)	137.87(16)
Br...H...C	Br(1)	H(19A)	C(19)	3.459(4)	3.044(4)	106.62(8)

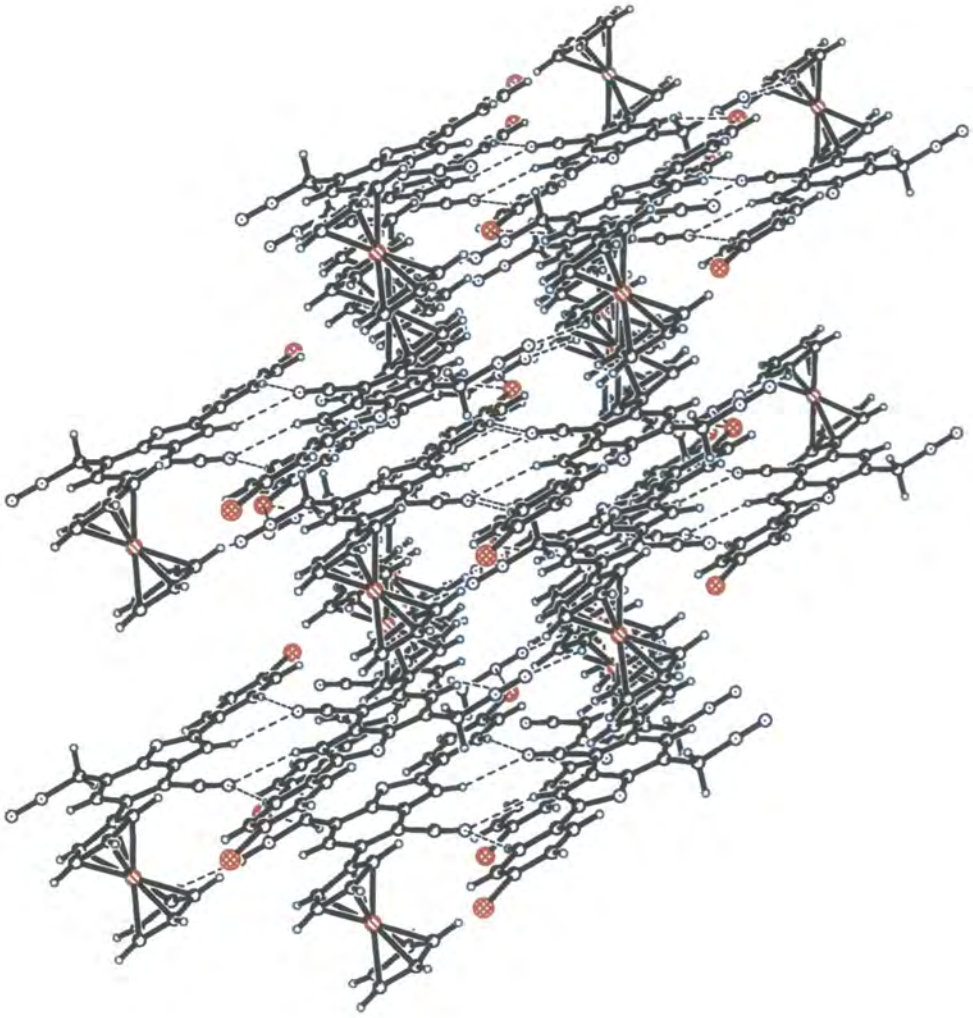


Diagram 8.8: the packing of sheets in the crystal.

Carbazol and triphenylethylphosphonium

Crystal Data:

Name:	Carbazol and triphenylethylphosphonium
Formula	C12 H9 N / C20 H19 P1
Colour	Yellow
Morphology	Block
Size	0.50 × 0.50 × 0.45 mm
Instrument	Smart CCD[1]
Temp/K	150
a	18.9682(4)Å
b	13.4146(9)Å
c	19.4322(10)Å
α	90°
β	95.246(3)°
γ	90°
Volume / z	1063.28(3)Å^3 / 4
Crystal system	Monoclinic
Space group	C2/c
R(int)	0.0627
Absorption correction	Multiscan[2]
Mu mm^{-1}	0.258
Total data unique	11282
Data I>2σ	9429
R1 (all data)	0.1154
Other	The crystal was a very good diffractor giving sharp well defined peaks. Since the data were collected using an area detector this meant that smaller steps between each area scan (0.25 instead of 0.3 steps in ω) had to be used. This gave a better coverage of space and therefore each peak appears on more scans, giving more data per peak than would have been obtained otherwise.
See also Appendix B:	Compound 17, page 207

Crystal structure and Packing:

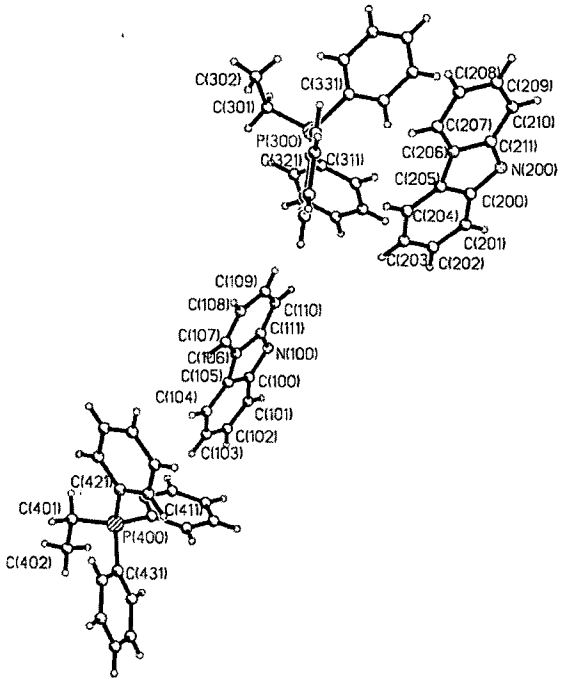


Diagram 8.9: Carbazol and triphenylethylphosphonum

The unit cell contains two crystallographically independent carbazol molecules and two crystallographically independent triphenylethylphosphonium molecules. For simplicity these are named carbazol (1), carbazol (2), triphenylethylphosphonium (3), and triphenylethylphosphonium (4) this matches with the numbering scheme used in the refinement.

The carbazol molecules align in layers with each carbazol slightly tilted out of the plane of the layer. There appears to be no hydrogen bonding interaction between adjacent carbazols.

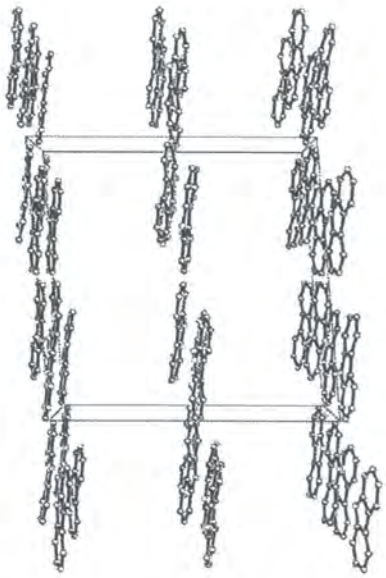


Diagram 8.10: Alignment of the carbazol molecules.

The triphenylethylphosphonium molecules pack in between these carbazol layers, such that the triphenylethylphosphonium molecules stack in columns. There equally appears to be no hydrogen bonding interaction between the triphenylethylphosphonium molecules.

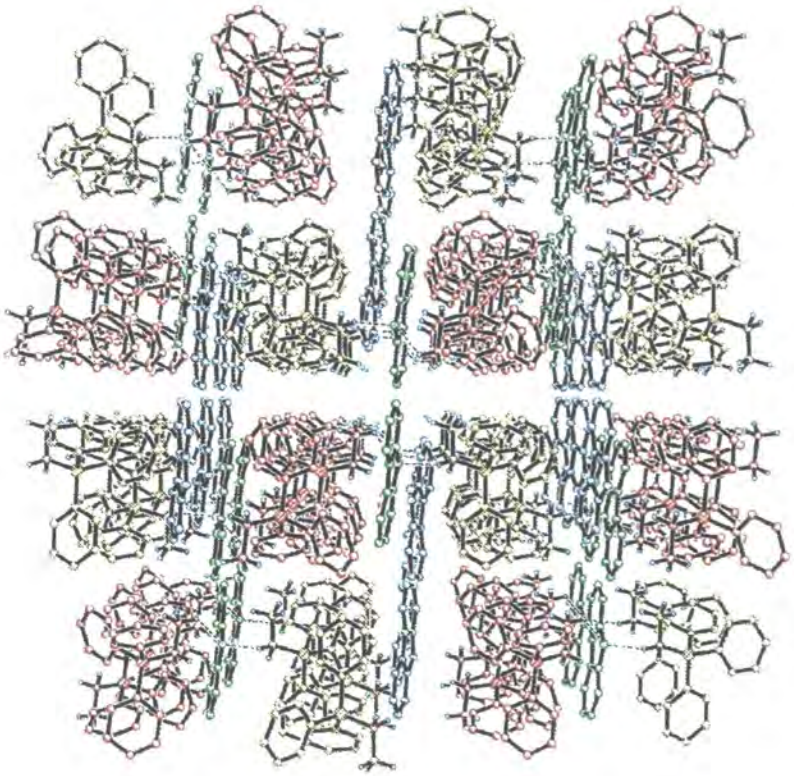


Diagram 8.11: packing within the crystal. For clarity carbazol (1) = Green, carbazol (2) = Blue, triphenylethylphosphonium (3) = Yellow, and triphenylethylphosphonium (4) = Red

Looking closely at this diagram it is perhaps possible to see that only carbazol (1) (Green) is involved in hydrogen bonds to the two triphenylethylphosphonium molecules, the second carbazol (carbazol (2) (blue)) is not involved in any hydrogen bonding interactions at all.

A closer look at the hydrogen bonds reveals that each carbazol (1) molecule is involved in two hydrogen bonds one from the nitrogen to the CH_2 of the ethyl group on triphenylethylphosphonium (3) and one from the nitrogen to the CH_3 of the ethyl group on triphenylethylphosphonium (4). Thus each carbazol (1) molecule joins two of the triphenylethylphosphonium stacks.

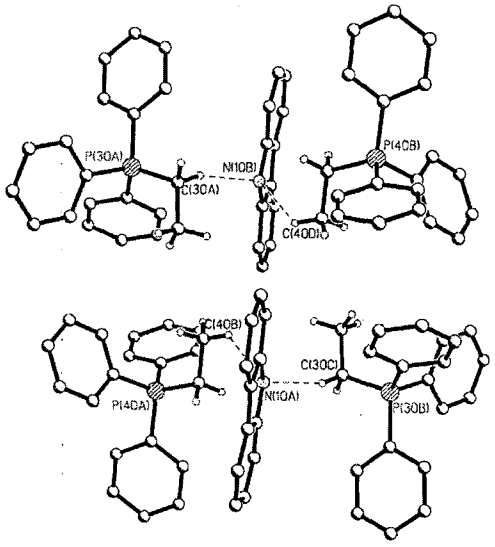


Diagram 8.12: the hydrogen bonding interactions between carbazol (1) and triphenylethylphosphonium molecules.

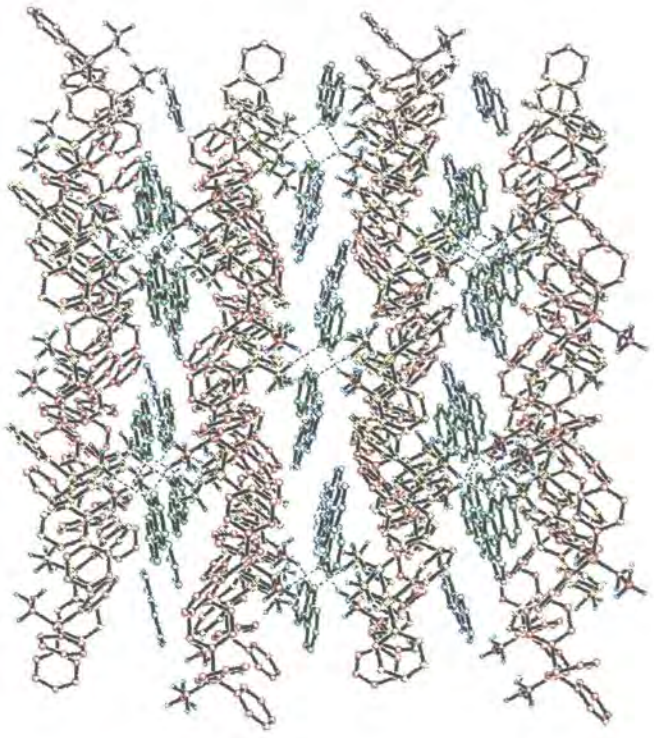


Diagram 8.13: the hydrogen bonding interactions between triphenylethylphosphonium stacks, viewed along the stacks. For clarity carbazol (1) = Green, carbazol (2) = Blue, triphenylethylphosphonium (3) = Yellow, and triphenylethylphosphonium (4) = Red.

Table 8.3 Hydrogen bonding distances.

TYPE: A...H...B	ATOM A	ATOM H	ATOM B	DISTANCE A...B /Å	DISTANCE A...H /Å	ANGLE A-H-B /°
N...H...C	N(100)	H(40C)	C(402)	3.218(2)	2.49(2)	133(1)
N...H...C	N(100)	H(30A)	C(301)	3.223(2)	2.30(2)	161(1)

Conclusions:

The three compounds studied analysed for hydrogen bonding in this chapter exhibit some very interesting hydrogen bonding patterns:

The complete lack of hydrogen bonds in sodium (triphenylethylphosphonium) (hexamethyldisilylamide), where the only possible hydrogen bond (the weak non-classical C-H...π interaction, see chapter 6) is not seen, and metal-metal and other intermolecular interactions hold the crystal together.

5-ferrocenyl-4-nitrile-7-acetonitrile-2-(4'-bromophenyl)indole, shows a more extended hydrogen bonded network, though still only of 2- dimensional sheets, not a full 3-dimensional lattice network. This compound has no classical hydrogen donors, so by definition all the hydrogen bonds must be of a weak non-classical type. Of especial interest in this crystal is the Br..H-C bond.

The final compound the carbazol triphenylethylphosphonium co-crystal also some hydrogen bonding though not such an extended network as seen in the ferrocene compound above. Again there are no classical hydrogen bond donors, the only classical hydrogen bond acceptors are the nitrogens on the carbazol molecules. Of the two chemically identical carbazol molecules only one is involved in hydrogen bonds the other shows no hydrogen bonding interactions.

References:

1) SMART Version 4.050. Seines Analytical X-ray Instruments, Madison, U.S.A., 1995

2) G.M.Sheldrick, (1996), SADABS, Program for the correction of area detector data, University of Göttingen, Germany.

3) a)M.Brookhart, M.L.H.Green. *J. Organomet. Chem.* 1983, **250,** 395.
b)P.L.A.Popelier, G.Logothetis. *J. Organomet. Chem.* 1998, **555,** 101.
c)D.Braga, F.Grepioni, K,Biradha, G.R.Desiraju. *J. Chem. Soc., Dalton Trans.* 1996, **20,** 3925

4) G.M.Sheldrick. SHELXL 93. Program for Refinement of Crystal Structures. University of Göttingen, Germany.

5) F.H.Allen, O.Kennard. *Chem. Des. Autom. News*. 1993, **8**, 1.

Appendix A

Lecture courses

Code	Title	Given By
PG4	Diffraction and Scattering methods	Dr. C.W.Lehmann
PG8	Molecular Modelling	Dr. C.W.Lehmann
PG6	Advanced Mass Spectrometry	Dr. M.Jones and Dr.C.A.Woodward

Passed all Courses

Conferences

Date	Title	Location
12th November 1997	BCA Autumn Meeting	Bristol
15th November 1997	Scottish Protein Structure Group	Edinburgh
5th – 8th April 1998	BCA Spring Meeting	St Andrews
9th April 1998 (last day only)	RSC Meeting	Durham
13th May 1998	MSI (Life Sciences)	MSI Cambridge

Seminars

Date	Title	Speaker
8th October 1997	Advances in Control Of Architecture for Polyamides	Professor E. Atkins
15th October 1997	Studying Catalysts in Action	Dr. R.M.Ormerod
22rd October 1997	Organoplatinum Chemistry and Catalysis	Professor R.J.Puddephatt
23rd October 1997	New Tetrathiafulvalene Derivatives in Molecular, Supramolecular and Macromolecular Chemistry	Professor M.R.Bryce
27th October 1997	Silyl Complexes of Rutheium and Osminum	Professor W.Roper
20th November 1997	Polynuclear Metal Complexes	Dr. L Spiccia
26th November 1997	A Random Walk in Polymer Science	Professor R.W.Richards
3rd December 1997	Steroid-based frameworks for Supramolecular Chemistry	Professor A.P.Davis
8th January 1997	Control of Structure and Dimensionality in mixed organic-inorganic solids	Ian Williams
21st January 1998	Aspects of Metal and Carbon Based Chemistry	Professor D.Cardin
4th February 1998	Classical and Non-Classical Fullerenes	Professor P.Fowler
24th February 1998	Synthesis and folding of Proteins	Professor R.Ramage
18th March 1998	Negative thermal expansion	Dr. J.Evans
20th March 1998	Chemical Information Hidden in Charge Density	Dr. Paul Popelier
20th March 1998	Buster Program	Dr. Pietro Roversi
15th May 1998	Recrystallisation using gel methods	M. Leech
9th October 1998	Carboranes exploitation of their unusual geometry and reactivities	Professor M.F.Hawthorn

Appendix B

Data files

1) 1,3-dimethyl-2-imidazolidinone methane-diphenol-3,5,3',5'-tetra^{t}butyl

Table 1. Crystal data and structure refinement for 1.

Identification code	1 (99srv011)
Empirical formula	C34 H54 N2 O3
Formula weight	538.79
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 13.4162(2) A alpha = 105.2990(10) deg. b = 14.10960(10) A beta = 102.3080(10) deg. c = 19.2269(3) A gamma = 94.5770(10) deg.
Volume, Z	3394.00(8) A^3, 4
Density (calculated)	1.054 Mg/m^3
Absorption coefficient	0.066 mm^-1
F(000)	1184
Crystal size	0.40 x 0.36 x 0.24 mm
Theta range for data collection	1.13 to 27.48 deg.
Limiting indices	-17<=h<=13, -18<=k<=18, -24<=l<=24
Reflections collected	28988
Independent reflections	15361 [R(int) = 0.0454]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1534 / 0 / 747
Goodness-of-fit on F^2	1.014
Final R indices [I>2sigma(I)]	R1 = 0.0596, wR2 = 0.1314
R indices (all data)	R1 = 0.1088, wR2 = 0.1697
Extinction coefficient	not refined
Largest diff. peak and hole	0.356 and -0.269 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	-2637(1)	8599(1)	2330(1)	45(1)
N(1)	-2605(1)	8377(1)	3485(1)	44(1)
N(2)	-2947(1)	7051(1)	2510(1)	41(1)
C(1)	-2721(2)	8061(2)	2731(1)	35(1)
C(2)	-2659(2)	9397(2)	3868(1)	64(1)
C(3)	-2912(2)	7558(2)	3758(1)	50(1)
C(4)	-2802(2)	6666(2)	3150(1)	46(1)
C(5)	-2845(2)	6462(2)	1793(1)	55(1)
O(2)	2127(1)	6956(1)	3173(1)	41(1)
N(3)	1649(2)	6877(2)	1931(1)	54(1)
N(4)	2670(2)	8175(2)	2687(1)	59(1)
C(6)	2142(1)	7304(2)	2649(1)	31(1)
C(7)	862(2)	6017(2)	1695(1)	66(1)
C(8)	1747(2)	7515(2)	1465(1)	41(1)
C(9)	2595(2)	8342(2)	1970(1)	47(1)
C(10)	3375(2)	8800(2)	3358(2)	76(1)
O(3)	5501(1)	8242(1)	1094(1)	44(1)
O(4)	-1569(1)	8177(1)	1163(1)	45(1)
C(11)	4596(1)	8590(2)	858(1)	31(1)
C(12)	4487(1)	9604(2)	1059(1)	29(1)
C(13)	3523(1)	9856(2)	781(1)	32(1)
C(14)	2706(1)	9155(2)	312(1)	33(1)
C(15)	2855(2)	8167(2)	120(1)	33(1)
C(16)	3782(2)	7849(2)	386(1)	32(1)
C(17)	5381(2)	10426(2)	1542(1)	37(1)
C(18)	6272(2)	10422(2)	1153(1)	56(1)
C(19)	5743(2)	10302(2)	2323(1)	51(1)
C(20)	5054(2)	11468(2)	1662(2)	57(1)
C(21)	3913(2)	6736(2)	177(1)	38(1)
C(22)	4778(2)	6559(2)	-239(1)	52(1)
C(23)	2930(2)	6083(2)	-342(1)	51(1)
C(24)	4151(2)	6380(2)	877(1)	46(1)
C(25)	1683(2)	9467(2)	0(1)	38(1)
C(26)	812(1)	9148(2)	318(1)	32(1)
C(27)	752(2)	9639(2)	1032(1)	34(1)
C(28)	-14(1)	9355(2)	1357(1)	32(1)
C(29)	-768(1)	8539(2)	917(1)	31(1)
C(30)	-743(1)	8023(1)	182(1)	30(1)
C(31)	60(1)	8351(2)	-92(1)	31(1)
C(32)	-1563(2)	7133(2)	-304(1)	37(1)
C(33)	-1369(2)	6716(2)	-1081(1)	49(1)
C(34)	-1545(2)	6283(2)	55(1)	54(1)
C(35)	-2640(2)	7458(2)	-426(2)	60(1)
C(36)	-22(2)	9913(2)	2167(1)	39(1)
C(37)	903(2)	10749(2)	2526(1)	58(1)
C(38)	-999(2)	10425(2)	2180(1)	47(1)
C(39)	50(2)	9208(2)	2660(1)	48(1)
O(5)	10488(1)	5742(1)	3365(1)	43(1)
O(6)	3977(1)	6981(1)	4222(1)	43(1)
C(40)	9792(1)	5673(2)	3791(1)	31(1)
C(41)	9131(1)	4757(2)	3552(1)	31(1)
C(42)	8425(1)	4633(2)	3972(1)	31(1)
C(43)	8351(1)	5367(2)	4596(1)	30(1)
C(44)	9000(1)	6264(2)	4797(1)	31(1)
C(45)	9727(1)	6452(1)	4407(1)	30(1)
C(46)	9164(2)	3925(2)	2853(1)	37(1)
C(47)	10219(2)	3548(2)	2950(1)	48(1)
C(48)	8939(2)	4302(2)	2165(1)	51(1)
C(49)	8338(2)	3024(2)	2705(2)	52(1)
C(50)	10403(2)	7478(2)	4648(1)	36(1)
C(51)	11554(2)	7385(2)	4894(1)	40(1)
C(52)	10142(2)	8194(2)	5321(1)	49(1)
C(53)	10218(2)	7975(2)	4017(1)	45(1)
C(54)	7552(1)	5212(2)	5023(1)	33(1)

C(55)	6590(1)	5669(2)	4800(1)	30(1)
C(56)	5891(1)	5246(2)	4117(1)	30(1)
C(57)	5005(1)	5646(2)	3882(1)	29(1)
C(58)	4830(1)	6514(2)	4379(1)	30(1)
C(59)	5515(1)	6974(2)	5075(1)	30(1)
C(60)	6393(1)	6533(2)	5263(1)	30(1)
C(61)	5312(2)	7911(2)	5623(1)	37(1)
C(62)	4301(2)	7679(2)	5848(1)	51(1)
C(63)	5255(2)	8777(2)	5276(1)	55(1)
C(64)	6177(2)	8265(2)	6344(1)	43(1)
C(65)	4262(2)	5147(2)	3108(1)	34(1)
C(66)	4642(2)	4218(2)	2677(1)	47(1)
C(67)	4190(2)	5862(2)	2620(1)	41(1)
C(68)	3187(2)	4794(2)	3195(1)	40(1)

Table 3. Bond lengths [A] and angles [deg] for 1.

O(1)-C(1)	1.230(3)
N(1)-C(1)	1.370(3)
N(1)-C(2)	1.448(3)
N(1)-C(3)	1.454(3)
N(2)-C(1)	1.364(3)
N(2)-C(5)	1.451(3)
N(2)-C(4)	1.454(3)
C(2)-H(2A)	0.98
C(2)-H(2B)	0.98
C(2)-H(2C)	0.98
C(3)-C(4)	1.516(3)
C(3)-H(3A)	0.99
C(3)-H(3B)	0.99
C(4)-H(4A)	0.99
C(4)-H(4B)	0.99
C(5)-H(5A)	0.98
C(5)-H(5B)	0.98
C(5)-H(5C)	0.98
O(2)-C(6)	1.235(2)
N(3)-C(6)	1.347(3)
N(3)-C(8)	1.445(3)
N(3)-C(7)	1.447(3)
N(4)-C(6)	1.344(3)
N(4)-C(10)	1.443(3)
N(4)-C(9)	1.444(3)
C(7)-H(7A)	0.98
C(7)-H(7B)	0.98
C(7)-H(7C)	0.98
C(8)-C(9)	1.523(3)
C(8)-H(8A)	0.99
C(8)-H(8B)	0.99
C(9)-H(9A)	0.99
C(9)-H(9B)	0.99
C(10)-H(10A)	0.98
C(10)-H(10B)	0.98
C(10)-H(10C)	0.98
O(3)-C(11)	1.378(2)
O(3)-H(3)	0.83(3)
O(4)-C(29)	1.377(2)
O(4)-H(4)	0.84(3)
C(11)-C(12)	1.408(3)
C(11)-C(16)	1.416(3)
C(12)-C(13)	1.401(3)
C(12)-C(17)	1.543(3)
C(13)-C(14)	1.388(3)
C(13)-H(13A)	0.95
C(14)-C(15)	1.387(3)
C(14)-C(25)	1.524(3)
C(15)-C(16)	1.395(3)
C(15)-H(15A)	0.95
C(16)-C(21)	1.549(3)
C(17)-C(19)	1.537(3)
C(17)-C(18)	1.539(3)
C(17)-C(20)	1.541(3)
C(18)-H(18A)	0.98
C(18)-H(18B)	0.98
C(18)-H(18C)	0.98
C(19)-H(19A)	0.98
C(19)-H(19B)	0.98
C(19)-H(19C)	0.98
C(20)-H(20A)	0.98
C(20)-H(20B)	0.98
C(20)-H(20C)	0.98
C(21)-C(23)	1.531(3)
C(21)-C(24)	1.538(3)
C(21)-C(22)	1.545(3)
C(22)-H(22A)	0.98
C(22)-H(22B)	0.98
C(22)-H(22C)	0.98
C(23)-H(23A)	0.98
C(23)-H(23B)	0.98
C(23)-H(23C)	0.98
C(24)-H(24A)	0.98
C(24)-H(24B)	0.98
C(24)-H(24C)	0.98
C(25)-C(26)	1.520(3)
C(25)-H(25A)	0.99
C(25)-H(25B)	0.99
C(26)-C(31)	1.385(3)
C(26)-C(27)	1.388(3)
C(27)-C(28)	1.394(3)
C(27)-H(27A)	0.95
C(28)-C(29)	1.414(3)
C(28)-C(36)	1.553(3)
C(29)-C(30)	1.418(3)
C(30)-C(31)	1.394(3)
C(30)-C(32)	1.543(3)
C(31)-H(31A)	0.95
C(32)-C(34)	1.533(3)
C(32)-C(33)	1.540(3)
C(32)-C(35)	1.541(3)
C(33)-H(33A)	0.98
C(33)-H(33B)	0.98
C(33)-H(33C)	0.98
C(34)-H(34A)	0.98
C(34)-H(34B)	0.98
C(34)-H(34C)	0.98
C(35)-H(35A)	0.98
C(35)-H(35B)	0.98
C(35)-H(35C)	0.98
C(36)-C(39)	1.539(3)
C(36)-C(37)	1.543(3)
C(36)-C(38)	1.547(3)
C(37)-H(37A)	0.98
C(37)-H(37B)	0.98
C(37)-H(37C)	0.98
C(38)-H(38A)	0.98
C(38)-H(38B)	0.98
C(38)-H(38C)	0.98

C(39)-H(39A)	0.98
C(39)-H(39B)	0.98
C(39)-H(39C)	0.98
O(5)-C(40)	1.380(2)
O(5)-H(5)	0.85(3)
O(6)-C(58)	1.383(2)
O(6)-H(6)	0.85(3)
C(40)-C(45)	1.410(3)
C(40)-C(41)	1.414(3)
C(41)-C(42)	1.398(3)
C(41)-C(46)	1.545(3)
C(42)-C(43)	1.390(3)
C(42)-H(42A)	0.95
C(43)-C(44)	1.391(3)
C(43)-C(54)	1.518(3)
C(44)-C(45)	1.400(3)
C(44)-H(44A)	0.95
C(45)-C(50)	1.546(3)
C(46)-C(48)	1.532(3)
C(46)-C(49)	1.542(3)
C(46)-C(47)	1.543(3)
C(47)-H(47A)	0.98
C(47)-H(47B)	0.98
C(47)-H(47C)	0.98
C(48)-H(48A)	0.98
C(48)-H(48B)	0.98
C(48)-H(48C)	0.98
C(49)-H(49A)	0.98
C(49)-H(49B)	0.98
C(49)-H(49C)	0.98
C(50)-C(53)	1.540(3)
C(50)-C(52)	1.542(3)
C(50)-C(51)	1.543(3)
C(51)-H(51A)	0.98
C(51)-H(51B)	0.98
C(51)-H(51C)	0.98
C(52)-H(52A)	0.98
C(52)-H(52B)	0.98
C(52)-H(52C)	0.98
C(53)-H(53A)	0.98
C(53)-H(53B)	0.98
C(53)-H(53C)	0.98
C(54)-C(55)	1.520(3)
C(54)-H(54A)	0.99
C(54)-H(54B)	0.99
C(55)-C(60)	1.387(3)
C(55)-C(56)	1.390(3)
C(56)-C(57)	1.396(3)
C(56)-H(56A)	0.95
C(57)-C(58)	1.412(3)
C(57)-C(65)	1.551(3)
C(58)-C(59)	1.407(3)
C(59)-C(60)	1.399(3)
C(59)-C(61)	1.541(3)
C(60)-H(60A)	0.95
C(61)-C(63)	1.538(3)
C(61)-C(64)	1.541(3)
C(61)-C(62)	1.547(3)
C(62)-H(62A)	0.98
C(62)-H(62B)	0.98
C(62)-H(62C)	0.98
C(63)-H(63A)	0.98
C(63)-H(63B)	0.98
C(63)-H(63C)	0.98
C(64)-H(64A)	0.98
C(64)-H(64B)	0.98
C(64)-H(64C)	0.98
C(65)-C(66)	1.541(3)
C(65)-C(67)	1.544(3)
C(65)-C(68)	1.546(3)
C(66)-H(66A)	0.98
C(66)-H(66B)	0.98
C(66)-H(66C)	0.98
C(67)-H(67A)	0.98
C(67)-H(67B)	0.98
C(67)-H(67C)	0.98
C(68)-H(68A)	0.98
C(68)-H(68B)	0.98
C(68)-H(68C)	0.98

C(1)-N(1)-C(2)	121.8(2)
C(1)-N(1)-C(3)	110.6(2)
C(2)-N(1)-C(3)	121.7(2)
C(1)-N(2)-C(5)	121.5(2)
C(1)-N(2)-C(4)	110.7(2)
C(5)-N(2)-C(4)	121.6(2)
O(1)-C(1)-N(2)	126.6(2)
O(1)-C(1)-N(1)	125.7(2)
N(2)-C(1)-N(1)	107.6(2)
N(1)-C(2)-H(2A)	109.47(14)
N(1)-C(2)-H(2B)	109.47(13)
H(2A)-C(2)-H(2B)	109.5
N(1)-C(2)-H(2C)	109.47(14)
H(2A)-C(2)-H(2C)	109.5
H(2B)-C(2)-H(2C)	109.5
N(1)-C(3)-C(4)	101.9(2)
N(1)-C(3)-H(3A)	111.39(12)
C(4)-C(3)-H(3A)	111.39(13)
N(1)-C(3)-H(3B)	111.39(12)
C(4)-C(3)-H(3B)	111.39(13)
H(3A)-C(3)-H(3B)	109.3
N(2)-C(4)-C(3)	101.7(2)
N(2)-C(4)-H(4A)	111.43(11)
C(3)-C(4)-H(4A)	111.43(13)
N(2)-C(4)-H(4B)	111.43(12)
C(3)-C(4)-H(4B)	111.43(13)
H(4A)-C(4)-H(4B)	109.3
N(2)-C(5)-H(5A)	109.47(13)
N(2)-C(5)-H(5B)	109.47(13)
H(5A)-C(5)-H(5B)	109.5
N(2)-C(5)-H(5C)	109.47(13)
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(6)-N(3)-C(8)	112.9(2)
C(6)-N(3)-C(7)	122.8(2)
C(8)-N(3)-C(7)	121.9(2)
C(6)-N(4)-C(10)	123.2(2)
C(6)-N(4)-C(9)	113.2(2)
C(10)-N(4)-C(9)	122.8(2)
O(2)-C(6)-N(4)	126.3(2)
O(2)-C(6)-N(3)	126.5(2)
N(4)-C(6)-N(3)	107.2(2)
N(3)-C(7)-H(7A)	109.47(14)
N(3)-C(7)-H(7B)	109.5(2)
H(7A)-C(7)-H(7B)	109.5
N(3)-C(7)-H(7C)	109.5(2)
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
N(3)-C(8)-C(9)	102.4(2)
N(3)-C(8)-H(8A)	111.29(12)
C(9)-C(8)-H(8A)	111.29(12)
N(3)-C(8)-H(8B)	111.29(12)
C(9)-C(8)-H(8B)	111.29(12)
H(8A)-C(8)-H(8B)	109.2
N(4)-C(9)-C(8)	102.3(2)
N(4)-C(9)-H(9A)	111.30(14)
C(8)-C(9)-H(9A)	111.30(13)
N(4)-C(9)-H(9B)	111.30(14)
C(8)-C(9)-H(9B)	111.30(12)
H(9A)-C(9)-H(9B)	109.2
N(4)-C(10)-H(10A)	109.5(2)
N(4)-C(10)-H(10B)	109.5(2)
H(10A)-C(10)-H(10B)	109.5
N(4)-C(10)-H(10C)	109.5(2)
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(11)-O(3)-H(3)	114(2)
C(29)-O(4)-H(4)	114(2)

O(3)-C(11)-C(12)	123.0(2)
O(3)-C(11)-C(16)	114.8(2)
C(12)-C(11)-C(16)	122.2(2)
C(13)-C(12)-C(11)	117.0(2)
C(13)-C(12)-C(17)	120.0(2)
C(11)-C(12)-C(17)	122.9(2)
C(14)-C(13)-C(12)	122.7(2)
C(14)-C(13)-H(13A)	118.67(12)
C(12)-C(13)-H(13A)	118.67(11)
C(15)-C(14)-C(13)	118.2(2)
C(15)-C(14)-C(25)	120.9(2)
C(13)-C(14)-C(25)	120.9(2)
C(14)-C(15)-C(16)	122.9(2)
C(14)-C(15)-H(15A)	118.57(11)
C(16)-C(15)-H(15A)	118.57(12)
C(15)-C(16)-C(11)	117.0(2)
C(15)-C(16)-C(21)	121.5(2)
C(11)-C(16)-C(21)	121.5(2)
C(19)-C(17)-C(18)	111.4(2)
C(19)-C(17)-C(20)	105.7(2)
C(18)-C(17)-C(20)	106.8(2)
C(19)-C(17)-C(12)	111.0(2)
C(18)-C(17)-C(12)	109.9(2)
C(20)-C(17)-C(12)	111.9(2)
C(17)-C(18)-H(18A)	109.47(12)
C(17)-C(18)-H(18B)	109.47(13)
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.47(13)
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(17)-C(19)-H(19A)	109.47(13)
C(17)-C(19)-H(19B)	109.47(12)
H(19A)-C(19)-H(19B)	109.5
C(17)-C(19)-H(19C)	109.47(12)
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(17)-C(20)-H(20A)	109.47(11)
C(17)-C(20)-H(20B)	109.47(13)
H(20A)-C(20)-H(20B)	109.5
C(17)-C(20)-H(20C)	109.47(14)
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(23)-C(21)-C(24)	107.0(2)
C(23)-C(21)-C(22)	106.7(2)
C(24)-C(21)-C(22)	110.2(2)
C(23)-C(21)-C(16)	111.7(2)
C(24)-C(21)-C(16)	110.5(2)
C(22)-C(21)-C(16)	110.5(2)
C(21)-C(22)-H(22A)	109.47(12)
C(21)-C(22)-H(22B)	109.47(12)
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.47(13)
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(21)-C(23)-H(23A)	109.47(12)
C(21)-C(23)-H(23B)	109.47(13)
H(23A)-C(23)-H(23B)	109.5
C(21)-C(23)-H(23C)	109.47(12)
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
C(21)-C(24)-H(24A)	109.47(12)
C(21)-C(24)-H(24B)	109.47(12)
H(24A)-C(24)-H(24B)	109.5
C(21)-C(24)-H(24C)	109.47(12)
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(26)-C(25)-C(14)	113.4(2)
C(26)-C(25)-H(25A)	108.90(11)
C(14)-C(25)-H(25A)	108.90(11)
C(26)-C(25)-H(25B)	108.90(11)
C(14)-C(25)-H(25B)	108.90(12)
H(25A)-C(25)-H(25B)	107.7
C(31)-C(26)-C(27)	117.9(2)
C(31)-C(26)-C(25)	121.0(2)
C(27)-C(26)-C(25)	121.1(2)
C(26)-C(27)-C(28)	123.2(2)
C(26)-C(27)-H(27A)	118.42(12)
C(28)-C(27)-H(27A)	118.42(11)
C(27)-C(28)-C(29)	117.0(2)
C(27)-C(28)-C(36)	120.8(2)
C(29)-C(28)-C(36)	122.1(2)
O(4)-C(29)-C(28)	123.4(2)
O(4)-C(29)-C(30)	114.9(2)
C(28)-C(29)-C(30)	121.7(2)
C(31)-C(30)-C(29)	117.3(2)
C(31)-C(30)-C(32)	120.6(2)
C(29)-C(30)-C(32)	122.1(2)
C(26)-C(31)-C(30)	122.9(2)
C(26)-C(31)-H(31A)	118.54(12)
C(30)-C(31)-H(31A)	118.54(11)
C(34)-C(32)-C(33)	107.0(2)
C(34)-C(32)-C(35)	110.6(2)
C(33)-C(32)-C(35)	106.5(2)
C(34)-C(32)-C(30)	110.6(2)
C(33)-C(32)-C(30)	111.8(2)
C(35)-C(32)-C(30)	110.2(2)
C(32)-C(33)-H(33A)	109.47(12)
C(32)-C(33)-H(33B)	109.47(12)
H(33A)-C(33)-H(33B)	109.5
C(32)-C(33)-H(33C)	109.47(13)
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(32)-C(34)-H(34A)	109.47(12)
C(32)-C(34)-H(34B)	109.47(12)
H(34A)-C(34)-H(34B)	109.5
C(32)-C(34)-H(34C)	109.47(12)
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(32)-C(35)-H(35A)	109.47(12)
C(32)-C(35)-H(35B)	109.47(14)
H(35A)-C(35)-H(35B)	109.5
C(32)-C(35)-H(35C)	109.47(13)
H(35A)-C(35)-H(35C)	109.5
H(35B)-C(35)-H(35C)	109.5
C(39)-C(36)-C(37)	106.9(2)
C(39)-C(36)-C(38)	110.7(2)
C(37)-C(36)-C(38)	106.0(2)
C(39)-C(36)-C(28)	111.3(2)
C(37)-C(36)-C(28)	111.4(2)
C(38)-C(36)-C(28)	110.4(2)
C(36)-C(37)-H(37A)	109.47(13)
C(36)-C(37)-H(37B)	109.47(14)
H(37A)-C(37)-H(37B)	109.5
C(36)-C(37)-H(37C)	109.47(13)
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
C(36)-C(38)-H(38A)	109.47(12)
C(36)-C(38)-H(38B)	109.47(12)
H(38A)-C(38)-H(38B)	109.5
C(36)-C(38)-H(38C)	109.47(13)
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5
C(36)-C(39)-H(39A)	109.47(11)
C(36)-C(39)-H(39B)	109.47(12)
H(39A)-C(39)-H(39B)	109.5
C(36)-C(39)-H(39C)	109.47(12)
H(39A)-C(39)-H(39C)	109.5
H(39B)-C(39)-H(39C)	109.5
C(40)-O(5)-H(5)	116(2)
C(58)-O(6)-H(6)	113(2)
O(5)-C(40)-C(45)	123.3(2)
O(5)-C(40)-C(41)	114.3(2)
C(45)-C(40)-C(41)	122.3(2)
C(42)-C(41)-C(40)	117.1(2)
C(42)-C(41)-C(46)	120.6(2)
C(40)-C(41)-C(46)	122.3(2)
C(43)-C(42)-C(41)	122.8(2)
C(43)-C(42)-H(42A)	118.60(11)

C(41)-C(42)-H(42A)	118.60(11)
C(42)-C(43)-C(44)	117.9(2)
C(42)-C(43)-C(54)	121.1(2)
C(44)-C(43)-C(54)	121.0(2)
C(43)-C(44)-C(45)	123.0(2)
C(43)-C(44)-H(44A)	118.50(11)
C(45)-C(44)-H(44A)	118.50(11)
C(44)-C(45)-C(40)	116.9(2)
C(44)-C(45)-C(50)	120.2(2)
C(40)-C(45)-C(50)	122.9(2)
C(48)-C(46)-C(49)	106.8(2)
C(48)-C(46)-C(47)	110.5(2)
C(49)-C(46)-C(47)	107.0(2)
C(48)-C(46)-C(41)	110.1(2)
C(49)-C(46)-C(41)	111.5(2)
C(47)-C(46)-C(41)	110.7(2)
C(46)-C(47)-H(47A)	109.47(11)
C(46)-C(47)-H(47B)	109.47(13)
H(47A)-C(47)-H(47B)	109.5
C(46)-C(47)-H(47C)	109.47(13)
H(47A)-C(47)-H(47C)	109.5
H(47B)-C(47)-H(47C)	109.5
C(46)-C(48)-H(48A)	109.47(12)
C(46)-C(48)-H(48B)	109.47(12)
H(48A)-C(48)-H(48B)	109.5
C(46)-C(48)-H(48C)	109.47(12)
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(46)-C(49)-H(49A)	109.47(12)
C(46)-C(49)-H(49B)	109.47(12)
H(49A)-C(49)-H(49B)	109.5
C(46)-C(49)-H(49C)	109.47(13)
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
C(53)-C(50)-C(52)	106.4(2)
C(53)-C(50)-C(51)	110.8(2)
C(52)-C(50)-C(51)	106.2(2)
C(53)-C(50)-C(45)	110.7(2)
C(52)-C(50)-C(45)	111.7(2)
C(51)-C(50)-C(45)	110.9(2)
C(50)-C(51)-H(51A)	109.47(12)
C(50)-C(51)-H(51B)	109.47(11)
H(51A)-C(51)-H(51B)	109.5
C(50)-C(51)-H(51C)	109.47(11)
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
C(50)-C(52)-H(52A)	109.47(13)
C(50)-C(52)-H(52B)	109.47(11)
H(52A)-C(52)-H(52B)	109.5
C(50)-C(52)-H(52C)	109.47(12)
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5
C(50)-C(53)-H(53A)	109.47(11)
C(50)-C(53)-H(53B)	109.47(11)
H(53A)-C(53)-H(53B)	109.5
C(50)-C(53)-H(53C)	109.47(12)
H(53A)-C(53)-H(53C)	109.5
H(53B)-C(53)-H(53C)	109.5
C(43)-C(54)-C(55)	112.6(2)
C(43)-C(54)-H(54A)	109.08(10)
C(55)-C(54)-H(54A)	109.08(11)
C(43)-C(54)-H(54B)	109.08(11)
C(55)-C(54)-H(54B)	109.08(11)
H(54A)-C(54)-H(54B)	107.8
C(60)-C(55)-C(56)	117.9(2)
C(60)-C(55)-C(54)	121.2(2)
C(56)-C(55)-C(54)	120.9(2)
C(55)-C(56)-C(57)	123.1(2)
C(55)-C(56)-H(56A)	118.47(11)
C(57)-C(56)-H(56A)	118.47(11)
C(56)-C(57)-C(58)	116.8(2)
C(56)-C(57)-C(65)	120.6(2)
C(58)-C(57)-C(65)	122.6(2)
O(6)-C(58)-C(59)	114.7(2)
O(6)-C(58)-C(57)	122.9(2)
C(59)-C(58)-C(57)	122.4(2)
C(60)-C(59)-C(58)	117.1(2)
C(60)-C(59)-C(61)	120.6(2)
C(58)-C(59)-C(61)	122.3(2)
C(55)-C(60)-C(59)	122.7(2)
C(55)-C(60)-H(60A)	118.65(11)
C(59)-C(60)-H(60A)	118.65(11)
C(63)-C(61)-C(64)	106.8(2)
C(63)-C(61)-C(59)	110.8(2)
C(64)-C(61)-C(59)	112.0(2)
C(63)-C(61)-C(62)	110.5(2)
C(64)-C(61)-C(62)	107.0(2)
C(59)-C(61)-C(62)	109.6(2)
C(61)-C(62)-H(62A)	109.47(11)
C(61)-C(62)-H(62B)	109.47(11)
H(62A)-C(62)-H(62B)	109.5
C(61)-C(62)-H(62C)	109.47(13)
H(62A)-C(62)-H(62C)	109.5
H(62B)-C(62)-H(62C)	109.5
C(61)-C(63)-H(63A)	109.47(13)
C(61)-C(63)-H(63B)	109.47(12)
H(63A)-C(63)-H(63B)	109.5
C(61)-C(63)-H(63C)	109.47(12)
H(63A)-C(63)-H(63C)	109.5
H(63B)-C(63)-H(63C)	109.5
C(61)-C(64)-H(64A)	109.47(12)
C(61)-C(64)-H(64B)	109.47(12)
H(64A)-C(64)-H(64B)	109.5
C(61)-C(64)-H(64C)	109.47(11)
H(64A)-C(64)-H(64C)	109.5
H(64B)-C(64)-H(64C)	109.5
C(66)-C(65)-C(67)	106.3(2)
C(66)-C(65)-C(68)	106.5(2)
C(67)-C(65)-C(68)	111.2(2)
C(66)-C(65)-C(57)	111.8(2)
C(67)-C(65)-C(57)	110.7(2)
C(68)-C(65)-C(57)	110.3(2)
C(65)-C(66)-H(66A)	109.47(11)
C(65)-C(66)-H(66B)	109.47(12)
H(66A)-C(66)-H(66B)	109.5
C(65)-C(66)-H(66C)	109.47(12)
H(66A)-C(66)-H(66C)	109.5
H(66B)-C(66)-H(66C)	109.5
C(65)-C(67)-H(67A)	109.47(11)
C(65)-C(67)-H(67B)	109.47(11)
H(67A)-C(67)-H(67B)	109.5
C(65)-C(67)-H(67C)	109.47(11)
H(67A)-C(67)-H(67C)	109.5
H(67B)-C(67)-H(67C)	109.5
C(65)-C(68)-H(68A)	109.47(10)
C(65)-C(68)-H(68B)	109.47(11)
H(68A)-C(68)-H(68B)	109.5
C(65)-C(68)-H(68C)	109.47(12)
H(68A)-C(68)-H(68C)	109.5
H(68B)-C(68)-H(68C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 1.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	59(1)	44(1)	41(1)	22(1)	18(1)	14(1)
N(1)	51(1)	46(1)	35(1)	15(1)	10(1)	4(1)
N(2)	50(1)	39(1)	39(1)	17(1)	12(1)	9(1)
C(1)	31(1)	43(1)	35(1)	18(1)	7(1)	8(1)
C(2)	90(2)	54(2)	42(1)	6(1)	13(1)	6(1)
C(3)	56(2)	59(2)	43(1)	28(1)	13(1)	5(1)
C(4)	45(1)	48(1)	52(1)	29(1)	10(1)	3(1)
C(5)	69(2)	45(1)	51(2)	12(1)	21(1)	15(1)
O(2)	41(1)	49(1)	35(1)	24(1)	4(1)	-1(1)
N(3)	70(1)	53(1)	34(1)	22(1)	-1(1)	-18(1)
N(4)	70(1)	51(1)	47(1)	31(1)	-13(1)	-21(1)
C(6)	28(1)	36(1)	34(1)	18(1)	7(1)	8(1)
C(7)	84(2)	61(2)	42(1)	22(1)	-3(1)	-25(2)
C(8)	47(1)	45(1)	38(1)	22(1)	11(1)	12(1)
C(9)	52(1)	47(1)	50(1)	31(1)	10(1)	3(1)
C(10)	87(2)	60(2)	63(2)	32(2)	-24(2)	-24(2)
O(3)	32(1)	42(1)	48(1)	2(1)	-2(1)	11(1)
O(4)	43(1)	49(1)	41(1)	3(1)	19(1)	-5(1)
C(11)	28(1)	38(1)	31(1)	13(1)	8(1)	7(1)
C(12)	27(1)	35(1)	29(1)	14(1)	7(1)	2(1)
C(13)	33(1)	33(1)	35(1)	19(1)	10(1)	5(1)
C(14)	30(1)	40(1)	35(1)	23(1)	7(1)	4(1)
C(15)	31(1)	36(1)	32(1)	17(1)	3(1)	-3(1)
C(16)	35(1)	34(1)	30(1)	15(1)	10(1)	3(1)
C(17)	33(1)	36(1)	39(1)	7(1)	6(1)	2(1)
C(18)	37(1)	65(2)	57(2)	9(1)	12(1)	-14(1)
C(19)	55(1)	52(2)	35(1)	-1(1)	2(1)	15(1)
C(20)	49(1)	36(1)	73(2)	10(1)	-1(1)	-3(1)
C(21)	43(1)	34(1)	37(1)	12(1)	9(1)	6(1)
C(22)	59(2)	44(1)	54(2)	7(1)	23(1)	9(1)
C(23)	57(2)	37(1)	49(1)	8(1)	4(1)	-3(1)
C(24)	55(1)	39(1)	50(1)	20(1)	11(1)	14(1)
C(25)	31(1)	45(1)	46(1)	31(1)	5(1)	3(1)
C(26)	28(1)	36(1)	38(1)	23(1)	2(1)	8(1)
C(27)	30(1)	29(1)	40(1)	17(1)	-3(1)	2(1)
C(28)	32(1)	31(1)	32(1)	12(1)	1(1)	7(1)
C(29)	27(1)	33(1)	36(1)	13(1)	6(1)	6(1)
C(30)	27(1)	31(1)	32(1)	11(1)	4(1)	5(1)
C(31)	29(1)	36(1)	31(1)	17(1)	6(1)	8(1)
C(32)	31(1)	42(1)	35(1)	6(1)	6(1)	-2(1)
C(33)	50(1)	51(1)	37(1)	4(1)	8(1)	-6(1)
C(34)	71(2)	40(1)	46(1)	8(1)	17(1)	-9(1)
C(35)	30(1)	68(2)	61(2)	-3(1)	0(1)	-3(1)
C(36)	41(1)	37(1)	35(1)	8(1)	3(1)	8(1)
C(37)	57(2)	51(2)	49(1)	-3(1)	1(1)	-1(1)
C(38)	58(1)	40(1)	44(1)	11(1)	12(1)	19(1)
C(39)	53(1)	55(2)	35(1)	16(1)	6(1)	19(1)
O(5)	43(1)	42(1)	44(1)	10(1)	21(1)	-3(1)
O(6)	39(1)	47(1)	34(1)	3(1)	-5(1)	15(1)
C(40)	30(1)	36(1)	32(1)	16(1)	10(1)	6(1)
C(41)	29(1)	32(1)	32(1)	12(1)	6(1)	6(1)
C(42)	30(1)	29(1)	37(1)	15(1)	5(1)	3(1)
C(43)	28(1)	34(1)	33(1)	18(1)	7(1)	9(1)
C(44)	31(1)	33(1)	30(1)	11(1)	7(1)	8(1)
C(45)	28(1)	30(1)	33(1)	14(1)	4(1)	4(1)
C(46)	36(1)	34(1)	37(1)	6(1)	7(1)	5(1)
C(47)	45(1)	48(1)	50(1)	7(1)	14(1)	16(1)
C(48)	56(2)	52(2)	36(1)	8(1)	4(1)	3(1)
C(49)	54(1)	35(1)	59(2)	-1(1)	15(1)	0(1)
C(50)	32(1)	31(1)	41(1)	11(1)	4(1)	1(1)
C(51)	34(1)	40(1)	44(1)	14(1)	2(1)	0(1)
C(52)	47(1)	36(1)	56(2)	3(1)	11(1)	0(1)
C(53)	40(1)	39(1)	59(2)	25(1)	6(1)	3(1)
C(54)	32(1)	39(1)	35(1)	20(1)	10(1)	7(1)

C(55)	30(1)	36(1)	30(1)	18(1)	9(1)	4(1)
C(56)	36(1)	30(1)	29(1)	11(1)	12(1)	5(1)
C(57)	32(1)	34(1)	24(1)	12(1)	7(1)	1(1)
C(58)	29(1)	33(1)	28(1)	13(1)	4(1)	5(1)
C(59)	30(1)	33(1)	27(1)	10(1)	6(1)	2(1)
C(60)	28(1)	37(1)	25(1)	13(1)	3(1)	1(1)
C(61)	37(1)	37(1)	30(1)	4(1)	1(1)	6(1)
C(62)	40(1)	60(2)	40(1)	-5(1)	7(1)	9(1)
C(63)	72(2)	38(1)	44(1)	5(1)	-5(1)	11(1)
C(64)	44(1)	44(1)	31(1)	2(1)	1(1)	6(1)
C(65)	39(1)	37(1)	24(1)	10(1)	5(1)	3(1)
C(66)	57(1)	47(1)	29(1)	1(1)	3(1)	8(1)
C(67)	42(1)	55(1)	28(1)	18(1)	6(1)	5(1)
C(68)	40(1)	43(1)	32(1)	11(1)	3(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 1.

	x	y	z	U(eq)
H(2A)	-2442(14)	9835(2)	3592(6)	96
H(2B)	-2204(11)	9581(5)	4369(4)	96
H(2C)	-3370(4)	9464(4)	3903(9)	96
H(3A)	-3631(2)	7547(2)	3808(1)	60
H(3B)	-2449(2)	7593(2)	4242(1)	60
H(4A)	-2112(2)	6460(2)	3265(1)	55
H(4B)	-3337(2)	6097(2)	3071(1)	55
H(5A)	-2992(13)	6840(5)	1431(3)	82
H(5B)	-3334(10)	5846(6)	1625(5)	82
H(5C)	-2141(4)	6302(11)	1838(3)	82
H(7A)	956(9)	5655(8)	2069(5)	99
H(7B)	917(10)	5583(7)	1218(6)	99
H(7C)	180(2)	6232(2)	1637(10)	99
H(8A)	1095(2)	7777(2)	1320(1)	49
H(8B)	1951(2)	7161(2)	1011(1)	49
H(9A)	3253(2)	8283(2)	1818(1)	56
H(9B)	2400(2)	9005(2)	1972(1)	56
H(10A)	3277(12)	8576(10)	3783(2)	114
H(10B)	3242(12)	9488(3)	3434(7)	114
H(10C)	4086(2)	8760(13)	3312(5)	114
H(3)	5911(19)	8646(19)	1459(14)	51(7)
H(4)	-1633(19)	8543(20)	1571(15)	57(8)
H(13A)	3425(1)	10533(2)	918(1)	38
H(15A)	2301(2)	7685(2)	-206(1)	39
H(18A)	6832(6)	10953(9)	1459(5)	83
H(18B)	6025(4)	10531(13)	667(4)	83
H(18C)	6526(9)	9780(5)	1087(9)	83
H(19A)	6276(9)	10857(7)	2630(3)	76
H(19B)	6027(12)	9676(6)	2285(1)	76
H(19C)	5157(3)	10292(12)	2551(4)	76
H(20A)	5633(5)	11965(2)	1985(8)	86
H(20B)	4471(9)	11496(4)	1897(9)	86
H(20C)	4848(13)	11604(5)	1182(2)	86
H(22A)	4832(9)	5849(2)	-384(8)	78
H(22B)	5434(3)	6929(10)	88(4)	78
H(22C)	4616(7)	6787(11)	-684(5)	78
H(23A)	3033(5)	5384(2)	-449(8)	76
H(23B)	2772(7)	6265(8)	-807(4)	76
H(23C)	2355(3)	6181(9)	-102(4)	76
H(24A)	4211(12)	5671(3)	734(2)	70
H(24B)	3592(6)	6487(11)	1132(5)	70
H(24C)	4800(6)	6754(8)	1211(4)	70
H(25A)	1773(2)	10198(2)	106(1)	46
H(25B)	1488(2)	9175(2)	-547(1)	46
H(27A)	1257(2)	10194(2)	1314(1)	41
H(31A)	93(1)	8012(2)	-583(1)	37
H(33A)	-1897(8)	6146(8)	-1369(3)	73
H(33B)	-686(6)	6504(11)	-1031(1)	73

H(33C)	-1402(13)	7232(4)	-1337(4)	73
H(34A)	-2067(9)	5724(5)	-261(5)	81
H(34B)	-1694(13)	6513(4)	545(4)	81
H(34C)	-862(4)	6070(8)	112(8)	81
H(35A)	-3157(2)	6883(3)	-721(9)	89
H(35B)	-2653(5)	7964(10)	-692(9)	89
H(35C)	-2796(6)	7735(13)	56(2)	89
H(37A)	874(8)	11090(9)	3035(4)	87
H(37B)	876(8)	11225(7)	2234(6)	87
H(37C)	1547(2)	10464(2)	2536(9)	87
H(38A)	-984(6)	10788(10)	2693(2)	71
H(38B)	-1614(2)	9921(2)	1976(8)	71
H(38C)	-1017(7)	10889(9)	1879(7)	71
H(39A)	1(12)	9570(3)	3159(3)	71
H(39B)	712(5)	8952(9)	2692(7)	71
H(39C)	-515(7)	8653(6)	2442(5)	71
H(5)	10876(23)	6299(24)	3489(17)	82(10)
H(6)	3553(19)	6663(19)	3812(15)	54(7)
H(42A)	7977(1)	4021(2)	3825(1)	38
H(44A)	8948(1)	6774(2)	5218(1)	37
H(47A)	10218(5)	3011(9)	2506(4)	72
H(47B)	10345(6)	3300(11)	3386(6)	72
H(47C)	10763(2)	4095(4)	3018(9)	72
H(48A)	8959(12)	3768(4)	1725(2)	76
H(48B)	9459(7)	4867(8)	2231(4)	76
H(48C)	8254(5)	4511(11)	2100(5)	76
H(49A)	8364(9)	2520(5)	2251(5)	78
H(49B)	7654(2)	3235(3)	2646(9)	78
H(49C)	8470(7)	2743(7)	3126(4)	78
H(51A)	11659(2)	7092(10)	5308(6)	60
H(51B)	11965(2)	8044(2)	5052(8)	60
H(51C)	11768(3)	6958(9)	4476(3)	60
H(52A)	10271(12)	7919(6)	5744(3)	74
H(52B)	9414(4)	8280(10)	5193(3)	74
H(52C)	10575(9)	8839(4)	5453(6)	74
H(53A)	10672(8)	8612(5)	4174(3)	68
H(53B)	9498(3)	8088(10)	3904(6)	68
H(53C)	10367(11)	7542(5)	3573(3)	68
H(54A)	7861(1)	5510(2)	5562(1)	40
H(54B)	7356(1)	4491(2)	4935(1)	40
H(56A)	6022(1)	4657(2)	3796(1)	36
H(60A)	6874(1)	6837(2)	5726(1)	36
H(62A)	3730(3)	7447(12)	5402(2)	76
H(62B)	4161(7)	8282(3)	6183(7)	76
H(62C)	4371(5)	7162(9)	6103(8)	76
H(63A)	5908(5)	8925(8)	5149(9)	83
H(63B)	5127(13)	9364(4)	5633(4)	83
H(63C)	4692(9)	8592(5)	4825(5)	83
H(64A)	6835(3)	8414(11)	6223(1)	64
H(64B)	6223(8)	7742(5)	6593(4)	64
H(64C)	6024(6)	8865(7)	6673(4)	64
H(66A)	4155(7)	3924(7)	2193(4)	71
H(66B)	4691(12)	3733(5)	2962(4)	71
H(66C)	5323(6)	4407(3)	2604(8)	71
H(67A)	3665(8)	5560(5)	2155(4)	62
H(67B)	4859(3)	5992(8)	2509(7)	62
H(67C)	4000(11)	6488(4)	2887(3)	62
H(68A)	2732(4)	4460(10)	2704(1)	60
H(68B)	2894(5)	5367(2)	3441(7)	60
H(68C)	3252(2)	4329(8)	3496(7)	60

2) 1,3-dibutyl-2-imidazolidinone methane-diphenol-3,5,3',5'-tetra^{t}butyl

Table 1. Crystal data and structure refinement for 2.

Identification code	2 (99SRV019)
Empirical formula	C40 H66 N2 O3
Formula weight	622.95
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 14.0667(3) A alpha = 90 deg. b = 13.4124(3) A beta = 94.71 deg. c = 20.9124(5) A gamma = 90 deg.
Volume, Z	3932.2(2) A^3, 4
Density (calculated)	1.052 Mg/m^3
Absorption coefficient	0.065 mm^-1
F(000)	1376
Crystal size	0.30 x 0.20 x 0.15 mm
Theta range for data collection	1.68 to 27.48 deg.
Limiting indices	-18<=h<=18, -16<=k<=17, -26<=l<=27
Reflections collected	28333
Independent reflections	9002 [R(int) = 0.1523]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8873 / 6 / 453
Goodness-of-fit on F^2	1.233
Final R indices [I>2sigma(I)]	R1 = 0.1014, wR2 = 0.1520
R indices (all data)	R1 = 0.2219, wR2 = 0.2282
Extinction coefficient	0.0019(2)
Largest diff. peak and hole	0.343 and -0.279 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 2. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	3288(2)	2616(2)	2005(1)	35(1)
C(1)	2794(3)	2631(3)	2469(2)	28(1)
N(1)	1859(3)	2396(3)	2450(2)	46(1)
N(2)	3094(3)	2890(3)	3078(2)	44(1)
C(2)	1506(3)	2420(3)	3083(2)	36(1)
C(3)	2313(3)	2920(3)	3493(2)	39(1)
C(4)	1340(3)	1929(5)	1911(2)	63(2)
C(5)	438(6)	2603(7)	1729(4)	48(3)
C(6)	-75(5)	2342(6)	1089(4)	54(3)
C(7)	-986(8)	2934(8)	926(6)	68(3)
C(5D)	283(9)	1953(16)	1702(10)	68(7)
C(6D)	243(16)	2976(13)	1419(12)	64(7)
C(7D)	-751(19)	3306(22)	1168(18)	97(11)
C(8)	3985(3)	3426(4)	3239(2)	49(1)
C(9)	4562(3)	2970(4)	3818(2)	50(1)
C(10)	4923(4)	1931(4)	3712(3)	60(2)
C(11)	5448(4)	1457(4)	4295(3)	69(2)
O(20)	4536(2)	-5787(2)	1646(1)	40(1)
C(21)	4556(3)	-4971(3)	1237(2)	27(1)
C(22)	5430(3)	-4462(3)	1260(2)	30(1)
C(23)	5484(3)	-3629(3)	860(2)	30(1)
C(24)	4726(3)	-3315(3)	444(2)	24(1)
C(25)	3875(3)	-3838(3)	439(2)	25(1)
C(26)	3756(3)	-4667(3)	836(2)	26(1)
C(27)	2782(3)	-5196(3)	828(2)	30(1)
C(28)	2393(3)	-5138(3)	1498(2)	37(1)
C(29)	2855(3)	-6285(3)	587(2)	34(1)
C(30)	2021(3)	-4688(3)	358(2)	41(1)
C(31)	6304(3)	-4798(4)	1705(2)	42(1)
C(32)	6082(4)	-4754(4)	2414(2)	59(2)
C(33)	6601(3)	-5864(4)	1536(3)	59(2)
C(34)	7173(3)	-4120(4)	1634(3)	65(2)
C(35)	4805(3)	-2410(3)	16(2)	28(1)
O(40)	3182(2)	1146(2)	953(1)	35(1)
C(41)	3602(3)	274(3)	757(2)	25(1)
C(42)	4404(3)	-159(3)	1100(2)	24(1)
C(43)	4767(3)	-1023(3)	839(2)	26(1)
C(44)	4372(3)	-1469(3)	276(2)	26(1)
C(45)	3568(3)	-1028(3)	-35(2)	27(1)
C(46)	3165(3)	-146(3)	192(2)	27(1)
C(47)	2278(3)	318(3)	-179(2)	29(1)
C(48)	1938(3)	-293(3)	-776(2)	44(1)
C(49)	2511(3)	1375(3)	-420(2)	39(1)
C(50)	1436(3)	368(4)	249(2)	44(1)
C(51)	4844(3)	252(3)	1747(2)	28(1)
C(52)	5242(3)	1319(3)	1667(2)	36(1)
C(53)	5690(3)	-385(3)	2025(2)	39(1)
C(54)	4092(3)	234(3)	2249(2)	32(1)

Table 3. Bond lengths [A] and angles [deg] for 2.

O(1)-C(1)	1.240(4)
C(1)-N(1)	1.351(5)
C(1)-N(2)	1.354(5)
N(1)-C(4)	1.436(6)
N(1)-C(2)	1.451(5)
N(2)-C(3)	1.455(5)
N(2)-C(8)	1.459(5)
C(2)-C(3)	1.521(6)
C(2)-H(2A)	0.99
C(2)-H(2B)	0.99
C(3)-H(3A)	0.99
C(3)-H(3B)	0.99
C(4)-C(5D)	1.515(13)
C(4)-C(5)	1.579(9)
C(4)-H(4A)	0.99
C(4)-H(4B)	0.99
C(5)-C(6)	1.508(9)
C(5)-H(5A)	0.99
C(5)-H(5A)	0.99
C(6)-C(7)	1.522(10)
C(6)-H(6A)	0.99
C(6)-H(6B)	0.99
C(7)-H(7A)	0.98
C(7)-H(7B)	0.98
C(7)-H(7C)	0.98
C(5D)-C(6D)	1.494(13)
C(5D)-H(5E)	0.99
C(5D)-H(5F)	0.99
C(6D)-C(7D)	1.517(14)
C(6D)-H(6E)	0.99
C(6D)-H(6F)	0.99
C(7D)-H(7E)	0.98
C(7D)-H(7F)	0.98
C(7D)-H(7G)	0.98
C(8)-C(9)	1.529(6)
C(8)-H(8A)	0.99
C(8)-H(8B)	0.99
C(9)-C(10)	1.506(7)
C(9)-H(9A)	0.99
C(9)-H(9B)	0.99
C(10)-C(11)	1.513(7)
C(10)-H(10A)	0.99
C(10)-H(10B)	0.99
C(11)-H(11A)	0.98
C(11)-H(11B)	0.98
C(11)-H(11C)	0.98
O(20)-C(21)	1.391(4)
O(20)-H(20A)	0.84
C(21)-C(22)	1.404(5)
C(21)-C(26)	1.408(5)
C(22)-C(23)	1.402(5)
C(22)-C(31)	1.546(5)
C(23)-C(24)	1.385(5)
C(23)-H(23A)	0.95
C(24)-C(25)	1.387(5)
C(24)-C(35)	1.518(5)
C(25)-C(26)	1.406(5)
C(25)-H(25A)	0.95
C(26)-C(27)	1.541(5)
C(27)-C(28)	1.548(5)
C(27)-C(29)	1.551(5)
C(27)-C(30)	1.551(5)
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(30)-H(30A)	0.98
C(30)-H(30B)	0.98
C(30)-H(30C)	0.98
C(31)-C(33)	1.539(7)
C(31)-C(34)	1.540(6)
C(31)-C(32)	1.542(6)
C(32)-H(32A)	0.98
C(32)-H(32B)	0.98
C(32)-H(32C)	0.98
C(33)-H(33A)	0.98
C(33)-H(33B)	0.98
C(33)-H(33C)	0.98
C(34)-H(34A)	0.98
C(34)-H(34B)	0.98
C(34)-H(34C)	0.98
C(35)-C(44)	1.521(5)
C(35)-H(35A)	0.99
C(35)-H(35B)	0.99
O(40)-C(41)	1.388(4)
O(40)-H(40A)	0.84
C(41)-C(46)	1.404(5)
C(41)-C(42)	1.410(5)
C(42)-C(43)	1.395(5)
C(42)-C(51)	1.543(5)
C(43)-C(44)	1.394(5)
C(43)-H(43A)	0.95
C(44)-C(45)	1.390(5)
C(45)-C(46)	1.411(5)
C(45)-H(45A)	0.95
C(46)-C(47)	1.545(5)
C(47)-C(48)	1.537(5)
C(47)-C(50)	1.544(6)
C(47)-C(49)	1.548(6)
C(48)-H(48A)	0.98
C(48)-H(48B)	0.98
C(48)-H(48C)	0.98
C(49)-H(49A)	0.98
C(49)-H(49B)	0.98
C(49)-H(49C)	0.98
C(50)-H(50A)	0.98
C(50)-H(50B)	0.98
C(50)-H(50C)	0.98
C(51)-C(53)	1.540(5)
C(51)-C(54)	1.550(5)
C(51)-C(52)	1.551(5)
C(52)-H(52A)	0.98
C(52)-H(52B)	0.98
C(52)-H(52C)	0.98
C(53)-H(53A)	0.98
C(53)-H(53B)	0.98
C(53)-H(53C)	0.98
C(54)-H(54A)	0.98
C(54)-H(54B)	0.98
C(54)-H(54C)	0.98

O(1)-C(1)-N(1)	125.5(4)
O(1)-C(1)-N(2)	126.2(4)
N(1)-C(1)-N(2)	108.4(4)
C(1)-N(1)-C(4)	123.8(4)
C(1)-N(1)-C(2)	111.9(3)
C(4)-N(1)-C(2)	122.4(4)
C(1)-N(2)-C(3)	112.0(4)
C(1)-N(2)-C(8)	122.5(4)
C(3)-N(2)-C(8)	121.7(4)
N(1)-C(2)-C(3)	103.2(3)
N(1)-C(2)-H(2A)	111.1(2)
C(3)-C(2)-H(2A)	111.1(2)
N(1)-C(2)-H(2B)	111.1(2)
C(3)-C(2)-H(2B)	111.1(2)
H(2A)-C(2)-H(2B)	109.1
N(2)-C(3)-C(2)	102.6(3)
N(2)-C(3)-H(3A)	111.3(2)
C(2)-C(3)-H(3A)	111.3(2)
N(2)-C(3)-H(3B)	111.3(2)

C(2)-C(3)-H(3B)	111.3(2)
H(3A)-C(3)-H(3B)	109.2
N(1)-C(4)-C(5D)	129.8(10)
N(1)-C(4)-C(5)	106.6(5)
N(1)-C(4)-H(4A)	110.4(3)
C(5)-C(4)-H(4A)	110.4(4)
N(1)-C(4)-H(4B)	110.4(2)
C(5)-C(4)-H(4B)	110.4(4)
H(4A)-C(4)-H(4B)	108.6
C(6)-C(5)-C(4)	113.4(7)
C(6)-C(5)-H(5A)	108.9(5)
C(4)-C(5)-H(5A)	108.9(4)
C(6)-C(5)-H(5A)	108.9(5)
C(4)-C(5)-H(5A)	108.9(4)
H(5A)-C(5)-H(5A)	107.7
C(5)-C(6)-C(7)	114.2(7)
C(5)-C(6)-H(6A)	108.7(5)
C(7)-C(6)-H(6A)	108.7(6)
C(5)-C(6)-H(6B)	108.7(5)
C(7)-C(6)-H(6B)	108.7(6)
H(6A)-C(6)-H(6B)	107.6
C(6)-C(7)-H(7A)	109.5(5)
C(6)-C(7)-H(7B)	109.5(6)
H(7A)-C(7)-H(7B)	109.5
C(6)-C(7)-H(7C)	109.5(6)
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(6D)-C(5D)-C(4)	98.0(13)
C(6D)-C(5D)-H(5E)	112.2(12)
C(4)-C(5D)-H(5E)	112.2(9)
C(6D)-C(5D)-H(5F)	112.2(14)
C(4)-C(5D)-H(5F)	112.2(9)
H(5E)-C(5D)-H(5F)	109.8
C(5D)-C(6D)-C(7D)	114(2)
C(5D)-C(6D)-H(6E)	108.7(14)
C(7D)-C(6D)-H(6E)	109(2)
C(5D)-C(6D)-H(6F)	108.7(12)
C(7D)-C(6D)-H(6F)	108.7(14)
H(6E)-C(6D)-H(6F)	107.6
C(6D)-C(7D)-H(7E)	110(2)
C(6D)-C(7D)-H(7F)	110(2)
H(7E)-C(7D)-H(7F)	109.5
C(6D)-C(7D)-H(7G)	110(2)
H(7E)-C(7D)-H(7G)	109.5
H(7F)-C(7D)-H(7G)	109.5
N(2)-C(8)-C(9)	112.0(4)
N(2)-C(8)-H(8A)	109.2(3)
C(9)-C(8)-H(8A)	109.2(3)
N(2)-C(8)-H(8B)	109.2(2)
C(9)-C(8)-H(8B)	109.2(3)
H(8A)-C(8)-H(8B)	107.9
C(10)-C(9)-C(8)	114.7(4)
C(10)-C(9)-H(9A)	108.6(3)
C(8)-C(9)-H(9A)	108.6(3)
C(10)-C(9)-H(9B)	108.6(3)
C(8)-C(9)-H(9B)	108.6(3)
H(9A)-C(9)-H(9B)	107.6
C(9)-C(10)-C(11)	114.8(5)
C(9)-C(10)-H(10A)	108.6(3)
C(11)-C(10)-H(10A)	108.6(3)
C(9)-C(10)-H(10B)	108.6(3)
C(11)-C(10)-H(10B)	108.6(3)
H(10A)-C(10)-H(10B)	107.5
C(10)-C(11)-H(11A)	109.5(3)
C(10)-C(11)-H(11B)	109.5(3)
H(11A)-C(11)-H(11B)	109.5
C(10)-C(11)-H(11C)	109.5(3)
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(21)-O(20)-H(20A)	109.5(2)
O(20)-C(21)-C(22)	115.1(3)
O(20)-C(21)-C(26)	122.6(3)
C(22)-C(21)-C(26)	122.4(4)
C(23)-C(22)-C(21)	117.2(4)
C(23)-C(22)-C(31)	120.8(4)
C(21)-C(22)-C(31)	122.0(4)
C(24)-C(23)-C(22)	122.7(4)
C(24)-C(23)-H(23A)	118.6(2)
C(22)-C(23)-H(23A)	118.6(2)
C(23)-C(24)-C(25)	118.1(4)
C(23)-C(24)-C(35)	121.6(4)
C(25)-C(24)-C(35)	120.3(3)
C(24)-C(25)-C(26)	122.7(4)
C(24)-C(25)-H(25A)	118.6(2)
C(26)-C(25)-H(25A)	118.6(2)
C(25)-C(26)-C(21)	116.9(4)
C(25)-C(26)-C(27)	120.5(3)
C(21)-C(26)-C(27)	122.6(3)
C(26)-C(27)-C(28)	110.3(3)
C(26)-C(27)-C(29)	110.8(3)
C(28)-C(27)-C(29)	112.4(3)
C(26)-C(27)-C(30)	111.7(3)
C(28)-C(27)-C(30)	106.0(3)
C(29)-C(27)-C(30)	105.5(3)
C(27)-C(28)-H(28A)	109.5(2)
C(27)-C(28)-H(28B)	109.5(2)
H(28A)-C(28)-H(28B)	109.5
C(27)-C(28)-H(28C)	109.5(2)
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(27)-C(29)-H(29A)	109.5(2)
C(27)-C(29)-H(29B)	109.5(2)
H(29A)-C(29)-H(29B)	109.5
C(27)-C(29)-H(29C)	109.5(2)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(27)-C(30)-H(30A)	109.5(2)
C(27)-C(30)-H(30B)	109.5(2)
H(30A)-C(30)-H(30B)	109.5
C(27)-C(30)-H(30C)	109.5(2)
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
C(33)-C(31)-C(34)	107.1(4)
C(33)-C(31)-C(32)	109.6(4)
C(34)-C(31)-C(32)	107.2(4)
C(33)-C(31)-C(22)	110.5(4)
C(34)-C(31)-C(22)	111.6(4)
C(32)-C(31)-C(22)	110.8(4)
C(31)-C(32)-H(32A)	109.5(3)
C(31)-C(32)-H(32B)	109.5(3)
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5(3)
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(31)-C(33)-H(33A)	109.5(3)
C(31)-C(33)-H(33B)	109.5(3)
H(33A)-C(33)-H(33B)	109.5
C(31)-C(33)-H(33C)	109.5(3)
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(31)-C(34)-H(34A)	109.5(3)
C(31)-C(34)-H(34B)	109.5(3)
H(34A)-C(34)-H(34B)	109.5
C(31)-C(34)-H(34C)	109.5(2)
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(24)-C(35)-C(44)	113.7(3)
C(24)-C(35)-H(35A)	108.8(2)
C(44)-C(35)-H(35A)	108.8(2)
C(24)-C(35)-H(35B)	108.8(2)
C(44)-C(35)-H(35B)	108.8(2)
H(35A)-C(35)-H(35B)	107.7
C(41)-O(40)-H(40A)	109.5(2)
O(40)-C(41)-C(46)	114.7(3)
O(40)-C(41)-C(42)	122.6(3)
C(46)-C(41)-C(42)	122.6(4)
C(43)-C(42)-C(41)	116.5(3)
C(43)-C(42)-C(51)	120.4(3)

C(41)-C(42)-C(51)	123.0(3)
C(44)-C(43)-C(42)	123.5(4)
C(44)-C(43)-H(43A)	118.2(2)
C(42)-C(43)-H(43A)	118.2(2)
C(45)-C(44)-C(43)	117.8(4)
C(45)-C(44)-C(35)	121.3(4)
C(43)-C(44)-C(35)	120.9(4)
C(44)-C(45)-C(46)	122.2(4)
C(44)-C(45)-H(45A)	118.9(2)
C(46)-C(45)-H(45A)	118.9(2)
C(41)-C(46)-C(45)	117.3(4)
C(41)-C(46)-C(47)	122.8(4)
C(45)-C(46)-C(47)	119.9(3)
C(48)-C(47)-C(50)	107.0(3)
C(48)-C(47)-C(46)	112.2(3)
C(50)-C(47)-C(46)	110.6(3)
C(48)-C(47)-C(49)	106.5(3)
C(50)-C(47)-C(49)	110.0(4)
C(46)-C(47)-C(49)	110.3(3)
C(47)-C(48)-H(48A)	109.5(2)
C(47)-C(48)-H(48B)	109.5(2)
H(48A)-C(48)-H(48B)	109.5
C(47)-C(48)-H(48C)	109.5(2)
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(47)-C(49)-H(49A)	109.5(2)
C(47)-C(49)-H(49B)	109.5(2)
H(49A)-C(49)-H(49B)	109.5
C(47)-C(49)-H(49C)	109.5(2)
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
C(47)-C(50)-H(50A)	109.5(2)
C(47)-C(50)-H(50B)	109.5(2)
H(50A)-C(50)-H(50B)	109.5
C(47)-C(50)-H(50C)	109.5(2)
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
C(53)-C(51)-C(42)	112.0(3)
C(53)-C(51)-C(54)	106.6(3)
C(42)-C(51)-C(54)	110.0(3)
C(53)-C(51)-C(52)	106.1(3)
C(42)-C(51)-C(52)	111.0(3)
C(54)-C(51)-C(52)	111.0(3)
C(51)-C(52)-H(52A)	109.5(2)
C(51)-C(52)-H(52B)	109.5(2)
H(52A)-C(52)-H(52B)	109.5
C(51)-C(52)-H(52C)	109.5(2)
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5
C(51)-C(53)-H(53A)	109.5(2)
C(51)-C(53)-H(53B)	109.5(2)
H(53A)-C(53)-H(53B)	109.5
C(51)-C(53)-H(53C)	109.5(2)
H(53A)-C(53)-H(53C)	109.5
H(53B)-C(53)-H(53C)	109.5
C(51)-C(54)-H(54A)	109.5(2)
C(51)-C(54)-H(54B)	109.5(2)
H(54A)-C(54)-H(54B)	109.5
C(51)-C(54)-H(54C)	109.5(2)
H(54A)-C(54)-H(54C)	109.5
H(54B)-C(54)-H(54C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 2.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	35(2)	36(2)	34(2)	3(1)	8(1)	3(1)
C(1)	30(2)	23(2)	31(2)	6(2)	4(2)	8(2)
N(1)	33(2)	70(3)	35(2)	-13(2)	7(2)	-2(2)
N(2)	40(2)	62(3)	31(2)	-5(2)	6(2)	-10(2)
C(2)	33(2)	40(3)	35(2)	6(2)	9(2)	7(2)
C(3)	41(3)	44(3)	34(3)	3(2)	12(2)	10(2)
C(4)	39(3)	107(5)	43(3)	-21(3)	6(2)	-16(3)
C(5)	33(4)	64(7)	47(6)	5(5)	-3(4)	-15(5)
C(6)	50(5)	55(6)	57(5)	5(4)	0(4)	4(4)
C(7)	71(7)	48(6)	81(8)	1(5)	-14(6)	10(5)
C(5D)	58(13)	68(16)	76(16)	-12(13)	-11(11)	-35(12)
C(6D)	84(18)	43(13)	64(17)	-24(12)	-4(14)	-19(12)
C(7D)	103(23)	59(19)	120(30)	-9(17)	-44(19)	18(16)
C(8)	56(3)	55(3)	36(3)	-7(3)	4(2)	-19(3)
C(9)	42(3)	52(3)	55(3)	-15(3)	-2(2)	-7(2)
C(10)	54(3)	62(4)	65(4)	-17(3)	8(3)	-9(3)
C(11)	47(3)	58(4)	101(5)	3(4)	-2(3)	-4(3)
O(20)	36(2)	41(2)	41(2)	18(2)	0(1)	-3(1)
C(21)	27(2)	26(2)	29(2)	5(2)	8(2)	2(2)
C(22)	25(2)	35(2)	28(2)	2(2)	0(2)	7(2)
C(23)	27(2)	32(2)	30(2)	0(2)	4(2)	-4(2)
C(24)	29(2)	22(2)	22(2)	-3(2)	8(2)	2(2)
C(25)	28(2)	26(2)	21(2)	-1(2)	2(2)	2(2)
C(26)	30(2)	24(2)	24(2)	-2(2)	6(2)	5(2)
C(27)	28(2)	24(2)	38(2)	4(2)	4(2)	2(2)
C(28)	36(2)	33(2)	45(3)	-2(2)	16(2)	-6(2)
C(29)	43(3)	26(2)	34(2)	-2(2)	3(2)	-2(2)
C(30)	25(2)	41(3)	55(3)	5(2)	1(2)	0(2)

C(31)	33(2)	51(3)	41(3)	12(2)	-6(2)	-2(2)
C(32)	63(3)	77(4)	35(3)	7(3)	-14(3)	-5(3)
C(33)	44(3)	73(4)	61(4)	19(3)	1(3)	19(3)
C(34)	34(3)	91(4)	67(4)	28(3)	-20(3)	-6(3)
C(35)	31(2)	24(2)	28(2)	-1(2)	5(2)	0(2)
O(40)	40(2)	31(2)	35(2)	-8(1)	-1(1)	8(1)
C(41)	29(2)	21(2)	26(2)	0(2)	7(2)	1(2)
C(42)	24(2)	26(2)	23(2)	0(2)	1(2)	-2(2)
C(43)	23(2)	31(2)	24(2)	6(2)	0(2)	0(2)
C(44)	30(2)	22(2)	26(2)	4(2)	6(2)	-3(2)
C(45)	31(2)	26(2)	25(2)	0(2)	2(2)	-5(2)
C(46)	29(2)	28(2)	26(2)	4(2)	2(2)	-2(2)
C(47)	28(2)	30(2)	28(2)	2(2)	-4(2)	0(2)
C(48)	41(3)	46(3)	40(3)	-3(2)	-14(2)	5(2)
C(49)	35(2)	34(3)	47(3)	10(2)	-2(2)	6(2)
C(50)	29(2)	56(3)	45(3)	6(3)	4(2)	2(2)
C(51)	33(2)	26(2)	24(2)	-2(2)	1(2)	-5(2)
C(52)	35(2)	40(3)	33(2)	-3(2)	6(2)	-9(2)
C(53)	36(2)	44(3)	35(3)	0(2)	-7(2)	-1(2)
C(54)	41(3)	33(2)	23(2)	0(2)	4(2)	-5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 2.

	x	y	z	U(eq)
H(2A)	1386(3)	1738(3)	3239(2)	43
H(2B)	910(3)	2813(3)	3080(2)	43
H(3A)	2147(3)	3615(3)	3599(2)	47
H(3B)	2473(3)	2546(3)	3895(2)	47
H(4A)	1141(3)	1248(5)	2025(2)	75
H(4B)	1742(3)	1883(5)	1545(2)	75
H(5A)	-12(6)	2535(7)	2067(4)	58
H(5A)	641(6)	3309(7)	1720(4)	58
H(6A)	-236(5)	1623(6)	1088(4)	65
H(6B)	363(5)	2455(6)	749(4)	65
H(7A)	-1288(23)	2708(30)	512(14)	81
H(7B)	-831(9)	3644(10)	899(26)	81
H(7C)	-1424(17)	2831(32)	1261(15)	81
H(5E)	99(9)	1433(16)	1380(10)	82
H(5F)	-114(9)	1893(16)	2070(10)	82
H(6E)	662(16)	2998(13)	1061(12)	77
H(6F)	497(16)	3457(13)	1749(12)	77
H(7E)	-740(28)	4014(42)	1055(76)	116
H(7F)	-1192(28)	3202(113)	1501(35)	116
H(7G)	-962(54)	2913(94)	788(52)	116
H(8A)	3840(3)	4131(4)	3333(2)	59
H(8B)	4372(3)	3415(4)	2864(2)	59
H(9A)	5114(3)	3408(4)	3938(2)	60
H(9B)	4158(3)	2957(4)	4184(2)	60
H(10A)	5356(4)	1951(4)	3362(3)	72
H(10B)	4374(4)	1502(4)	3568(3)	72
H(11A)	5643(23)	780(11)	4189(6)	103
H(11B)	5027(10)	1431(25)	4646(6)	103
H(11C)	6015(15)	1855(16)	4429(11)	103
H(20A)	3992(2)	-6051(2)	1605(1)	59
H(23A)	6064(3)	-3264(3)	875(2)	35
H(25A)	3351(3)	-3627(3)	155(2)	30
H(28A)	2871(8)	-5400(18)	1822(3)	56
H(28B)	1808(11)	-5534(16)	1499(4)	56
H(28C)	2253(18)	-4442(4)	1598(6)	56
H(29A)	3053(18)	-6283(3)	148(5)	51
H(29B)	2231(5)	-6610(7)	590(12)	51
H(29C)	3325(14)	-6650(6)	868(7)	51
H(30A)	2222(10)	-4723(18)	-79(3)	61
H(30B)	1951(14)	-3988(6)	481(8)	61
H(30C)	1409(6)	-5031(13)	375(10)	61
H(32A)	5539(16)	-5190(19)	2480(4)	89
H(32B)	5925(23)	-4068(6)	2527(5)	89

H(32C)	6641(9)	-4978(24)	2688(2)	89
H(33A)	6081(10)	-6327(5)	1607(15)	89
H(33B)	7172(14)	-6054(10)	1809(11)	89
H(33C)	6740(23)	-5890(7)	1085(5)	89
H(34A)	7008(9)	-3430(6)	1733(17)	98
H(34B)	7356(16)	-4157(21)	1192(5)	98
H(34C)	7707(9)	-4341(17)	1930(13)	98
H(35A)	4482(3)	-2557(3)	-412(2)	33
H(35B)	5487(3)	-2285(3)	-40(2)	33
H(40A)	3479(2)	1354(2)	1291(1)	53
H(43A)	5313(3)	-1324(3)	1057(2)	31
H(45A)	3279(3)	-1332(3)	-413(2)	33
H(48A)	1763(19)	-966(7)	-645(2)	65
H(48B)	2453(8)	-335(18)	-1064(7)	65
H(48C)	1382(13)	31(12)	-1000(8)	65
H(49A)	3042(13)	1335(4)	-695(11)	58
H(49B)	2691(19)	1806(6)	-51(2)	58
H(49C)	1949(7)	1651(9)	-665(11)	58
H(50A)	1267(13)	-308(4)	377(11)	65
H(50B)	886(7)	679(19)	10(5)	65
H(50C)	1622(7)	764(18)	633(7)	65
H(52A)	4727(5)	1760(5)	1495(12)	53
H(52B)	5746(13)	1302(4)	1370(10)	53
H(52C)	5505(17)	1569(8)	2085(3)	53
H(53A)	5478(5)	-1072(6)	2082(12)	58
H(53B)	5937(13)	-111(12)	2440(6)	58
H(53C)	6195(8)	-376(17)	1729(6)	58
H(54A)	3878(13)	-452(4)	2308(9)	48
H(54B)	3546(8)	650(15)	2098(6)	48
H(54C)	4378(6)	495(17)	2658(4)	48

3) *p-tert*-butylcalix[4]arene triphenylmethylphosphonium

Table 1. Crystal data and structure refinement for 3.

Identification code	3 (98srv035)
Empirical formula	C67 H80 N2 O4 P
Formula weight	1008.30
Temperature	150.2). K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	a = 18.3934(3) A alpha = 90 deg. b = 12.6138(2) A beta = 90.4220(10) deg. c = 25.3686(4) A gamma = 90 deg.
Volume, Z	5885.6(2) A^3, 4
Density (calculated)	1.138 Mg/m^3
Absorption coefficient	0.095 mm^-1
F(000)	2172
Crystal size	0.5 x 0.4 x 0.05 mm
Theta range for data collection	1.11 to 27.50 deg.
Limiting indices	-23<=h<=23, -16<=k<=14, -32<=l<=32
Reflections collected	48067
Independent reflections	13478 [R(int) = 0.1002]
Absorption correction	None
Max. and min. transmission	0.969 and 0.932
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13313 / 6 / 723
Goodness-of-fit on F^2	1.224
Final R indices [I>2sigma(I)]	R1 = 0.0734, wR2 = 0.1234
R indices (all data)	R1 = 0.1473, wR2 = 0.1907
Largest diff. peak and hole	0.294 and -0.408 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 3. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	6320(1)	2259(2)	1202(1)	26(1)
O(2)	6037(1)	1882(2)	2166(1)	32(1)
O(3)	5866(1)	-389(2)	1896(1)	31(1)
O(4)	6210(1)	347(2)	921(1)	30(1)
C(1)	6863(2)	3765(2)	1980(1)	28(1)
C(2)	6432(2)	245(2)	2904(1)	27(1)
C(3)	6620(2)	-1786(2)	1171(1)	26(1)
C(4)	7046(2)	1745(2)	245(1)	23(1)
C(11)	6962(2)	2753(2)	1110(1)	22(1)
C(12)	7251(2)	3507(2)	1468(1)	24(1)
C(13)	7930(2)	3948(2)	1370(1)	26(1)
C(14)	8352(2)	3691(2)	932(1)	26(1)
C(15)	8041(2)	2968(2)	575(1)	24(1)
C(16)	7354(2)	2517(2)	649(1)	23(1)
C(21)	6641(2)	2048(2)	2474(1)	25(1)
C(22)	6843(2)	1296(2)	2857(1)	24(1)
C(23)	7459(2)	1492(2)	3165(1)	27(1)
C(24)	7899(2)	2383(2)	3102(1)	26(1)
C(25)	7679(2)	3112(2)	2719(1)	25(1)
C(26)	7060(2)	2966(2)	2407(1)	23(1)
C(31)	6509(2)	-852(2)	2061(1)	23(1)
C(32)	6868(2)	-1583(2)	1737(1)	23(1)
C(33)	7481(2)	-2104(2)	1935(1)	26(1)
C(34)	7754(2)	-1920(2)	2440(1)	25(1)
C(35)	7396(2)	-1160(2)	2744(1)	24(1)
C(36)	6777(2)	-617(2)	2565(1)	24(1)
C(41)	6829(2)	-35(2)	687(1)	22(1)
C(42)	7252(2)	601(2)	360(1)	21(1)
C(43)	7886(2)	179(2)	142(1)	23(1)
C(44)	8107(2)	-864(2)	228(1)	23(1)
C(45)	7659(2)	-1485(2)	550(1)	24(1)
C(46)	7035(2)	-1095(2)	784(1)	23(1)
C(10)	9110(2)	4177(3)	863(1)	32(1)
C(17)	9591(2)	3863(4)	1337(2)	59(1)
C(18)	9476(2)	3801(3)	355(2)	47(1)
C(19)	9056(2)	5397(3)	845(2)	37(1)
C(20)	8590(2)	2517(3)	3441(1)	32(1)
C(27)	9100(2)	1569(3)	3348(2)	58(1)
C(28)	9004(2)	3540(3)	3307(2)	53(1)
C(29)	8384(2)	2567(4)	4025(1)	53(1)
C(30)	8431(2)	-2517(3)	2646(1)	32(1)
C(37)	8332(4)	-3713(4)	2557(3)	81(3)
C(38)	9098(3)	-2135(7)	2347(4)	97(4)
C(39)	8558(3)	-2371(6)	3232(2)	66(2)
C(37D)	8733(11)	-3346(16)	2266(8)	38(5)
C(38D)	9060(12)	-1691(18)	2716(11)	56(7)
C(39D)	8234(15)	-3108(22)	3166(9)	66(8)
C(40)	8805(2)	-1334(2)	0(1)	27(1)
C(47)	9181(2)	-567(3)	-385(2)	40(1)
C(48)	9334(2)	-1594(3)	455(1)	36(1)
C(49)	8619(2)	-2357(3)	-308(1)	37(1)
P(1)	4396(1)	2805(1)	658(1)	24(1)
C(8)	4868(2)	3585(3)	1140(2)	35(1)
C(51)	4075(2)	1608(2)	963(1)	25(1)
C(52)	4434(2)	1206(3)	1410(1)	31(1)
C(53)	4146(2)	345(3)	1676(1)	36(1)
C(54)	3507(2)	-117(3)	1504(1)	38(1)
C(55)	3157(2)	256(3)	1053(1)	35(1)
C(56)	3442(2)	1119(3)	779(1)	30(1)
C(61)	3620(2)	3557(2)	441(1)	23(1)
C(62)	3072(2)	3756(2)	810(1)	26(1)
C(63)	2496(2)	4423(2)	677(1)	28(1)
C(64)	2469(2)	4891(2)	184(1)	31(1)
C(65)	3000(2)	4679(3)	-188(1)	32(1)

C(66)	3578(2)	4007(3)	-62(1)	28(1)
C(71)	4967(2)	2575(2)	101(1)	26(1)
C(72)	5440(2)	3378(3)	-65(2)	36(1)
C(73)	5836(2)	3257(3)	-523(2)	40(1)
C(74)	5769(2)	2337(3)	-817(1)	39(1)
C(75)	5311(2)	1532(3)	-651(1)	38(1)
C(76)	4907(2)	1647(3)	-195(1)	31(1)
N(1X)	9671(3)	833(4)	1726(3)	112(2)
C(11X)	9053(3)	813(3)	1715(2)	57(1)
C(12X)	8262(2)	799(3)	1704(2)	53(1)
N(2X)	4291(2)	4216(3)	-1338(2)	60(1)
C(21X)	4424(2)	3688(3)	-1689(2)	42(1)
C(22X)	4595(2)	2981(4)	-2128(2)	59(1)

Table 3. Bond lengths [A] and angles [deg] for 3.

O(1)-C(11)	1.357(3)
O(1)-H(1A)	0.84
O(2)-C(21)	1.369(4)
O(2)-H(2A)	0.84
O(3)-C(31)	1.381(4)
O(3)-H(3A)	0.84
O(4)-C(41)	1.375(3)
O(4)-H(4A)	0.84
C(1)-C(12)	1.520(4)
C(1)-C(26)	1.522(4)
C(1)-H(1B)	0.99
C(1)-H(1C)	0.99
C(2)-C(36)	1.527(4)
C(2)-C(22)	1.530(4)
C(2)-H(2B)	0.99
C(2)-H(2C)	0.99
C(3)-C(46)	1.523(4)
C(3)-C(32)	1.525(4)
C(3)-H(3B)	0.99
C(3)-H(3C)	0.99
C(4)-C(42)	1.519(4)
C(4)-C(16)	1.520(4)
C(4)-H(4B)	0.99
C(4)-H(4C)	0.99
C(11)-C(16)	1.408(4)
C(11)-C(12)	1.417(4)
C(12)-C(13)	1.392(4)
C(13)-C(14)	1.398(4)
C(13)-H(13A)	0.95
C(14)-C(15)	1.404(4)
C(14)-C(10)	1.533(4)
C(15)-C(16)	1.401(4)
C(15)-H(15A)	0.95
C(21)-C(26)	1.402(4)
C(21)-C(22)	1.407(4)
C(22)-C(23)	1.393(4)
C(23)-C(24)	1.397(4)
C(23)-H(23A)	0.95
C(24)-C(25)	1.396(4)
C(24)-C(20)	1.538(4)
C(25)-C(26)	1.395(4)
C(25)-H(25A)	0.95
C(31)-C(36)	1.399(4)
C(31)-C(32)	1.403(4)
C(32)-C(33)	1.394(4)
C(33)-C(34)	1.392(4)
C(33)-H(33A)	0.95
C(34)-C(35)	1.399(4)
C(34)-C(30)	1.544(4)
C(35)-C(36)	1.401(4)
C(35)-H(35A)	0.95
C(41)-C(42)	1.396(4)
C(41)-C(46)	1.410(4)
C(42)-C(43)	1.399(4)
C(43)-C(44)	1.393(4)
C(43)-H(43A)	0.95
C(44)-C(45)	1.403(4)
C(44)-C(40)	1.533(4)
C(45)-C(46)	1.387(4)
C(45)-H(45A)	0.95
C(10)-C(18)	1.534(5)
C(10)-C(17)	1.540(5)
C(10)-C(19)	1.543(4)
C(17)-H(17A)	0.98
C(17)-H(17B)	0.98
C(17)-H(17C)	0.98
C(18)-H(18A)	0.98
C(18)-H(18B)	0.98
C(18)-H(18C)	0.98
C(19)-H(19A)	0.98
C(19)-H(19B)	0.98
C(19)-H(19C)	0.98
C(20)-C(29)	1.535(5)
C(20)-C(27)	1.538(5)
C(20)-C(28)	1.538(5)
C(27)-H(27A)	0.98
C(27)-H(27B)	0.98
C(27)-H(27C)	0.98
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(30)-C(39)	1.514(6)
C(30)-C(38)	1.525(6)
C(30)-C(37D)	1.53(2)
C(30)-C(37)	1.536(6)
C(30)-C(39D)	1.56(2)
C(30)-C(38D)	1.56(2)
C(37)-H(37A)	0.98
C(37)-H(37B)	0.98
C(37)-H(37C)	0.98
C(38)-H(38A)	0.98
C(38)-H(38B)	0.98
C(38)-H(38C)	0.98
C(39)-H(39A)	0.98
C(39)-H(39B)	0.98
C(39)-H(39C)	0.98
C(37D)-H(37E)	0.98
C(37D)-H(37F)	0.98
C(37D)-H(37G)	0.98
C(38D)-H(38E)	0.98
C(38D)-H(38F)	0.98
C(38D)-H(38G)	0.98

C(39D)-H(39E)	0.98
C(39D)-H(39F)	0.98
C(39D)-H(39G)	0.98
C(40)-C(48)	1.540(4)
C(40)-C(47)	1.541(5)
C(40)-C(49)	1.546(4)
C(47)-H(47A)	0.98
C(47)-H(47B)	0.98
C(47)-H(47C)	0.98
C(48)-H(48A)	0.98
C(48)-H(48B)	0.98
C(48)-H(48C)	0.98
C(49)-H(49A)	0.98
C(49)-H(49B)	0.98
C(49)-H(49C)	0.98
P(1)-C(71)	1.790(3)
P(1)-C(8)	1.791(3)
P(1)-C(61)	1.796(3)
P(1)-C(51)	1.800(3)
C(8)-H(8C)	0.98(4)
C(8)-H(8B)	0.97(4)
C(8)-H(8A)	1.01(4)
C(51)-C(56)	1.395(4)
C(51)-C(52)	1.402(4)
C(52)-C(53)	1.386(5)
C(52)-H(52A)	0.95
C(53)-C(54)	1.382(5)
C(53)-H(53A)	0.95
C(54)-C(55)	1.388(5)
C(54)-H(54A)	0.95
C(55)-C(56)	1.396(5)
C(55)-H(55A)	0.95
C(56)-H(56A)	0.95
C(61)-C(66)	1.399(4)
C(61)-C(62)	1.403(4)
C(62)-C(63)	1.391(4)
C(62)-H(62A)	0.95
C(63)-C(64)	1.385(4)
C(63)-H(63A)	0.95
C(64)-C(65)	1.388(5)
C(64)-H(64A)	0.95
C(65)-C(66)	1.394(4)
C(65)-H(65A)	0.95
C(66)-H(66A)	0.95
C(71)-C(76)	1.394(4)
C(71)-C(72)	1.403(4)
C(72)-C(73)	1.385(5)
C(72)-H(72A)	0.95
C(73)-C(74)	1.385(5)
C(73)-H(73A)	0.95
C(74)-C(75)	1.387(5)
C(74)-H(74A)	0.95
C(75)-C(76)	1.387(5)
C(75)-H(75A)	0.95
C(76)-H(76A)	0.95
N(1X)-C(11X)	1.138(6)
C(11X)-C(12X)	1.454(6)
C(12X)-H(1XA)	0.98
C(12X)-H(1XB)	0.98
C(12X)-H(1XC)	0.98
N(2X)-C(21X)	1.139(5)
C(21X)-C(22X)	1.463(6)
C(22X)-H(2XA)	0.98
C(22X)-H(2XB)	0.98
C(22X)-H(2XC)	0.98
C(11)-O(1)-H(1A)	109.5(2)
C(21)-O(2)-H(2A)	109.5(2)
C(31)-O(3)-H(3A)	109.5(2)
C(41)-O(4)-H(4A)	109.5(2)
C(12)-C(1)-C(26)	110.9(2)
C(12)-C(1)-H(1B)	109.5(2)
C(26)-C(1)-H(1B)	109.5(2)
C(12)-C(1)-H(1C)	109.5(2)
C(26)-C(1)-H(1C)	109.5(2)
H(1B)-C(1)-H(1C)	108.0
C(36)-C(2)-C(22)	111.5(2)
C(36)-C(2)-H(2B)	109.3(2)
C(22)-C(2)-H(2B)	109.3(2)
C(36)-C(2)-H(2C)	109.3(2)
C(22)-C(2)-H(2C)	109.3(2)
H(2B)-C(2)-H(2C)	108.0
C(46)-C(3)-C(32)	111.3(2)
C(46)-C(3)-H(3B)	109.4(2)
C(32)-C(3)-H(3B)	109.4(2)
C(46)-C(3)-H(3C)	109.4(2)
C(32)-C(3)-H(3C)	109.4(2)
H(3B)-C(3)-H(3C)	108.0
C(42)-C(4)-C(16)	112.8(2)
C(42)-C(4)-H(4B)	109.0(2)
C(16)-C(4)-H(4B)	109.0(2)
C(42)-C(4)-H(4C)	109.0(2)
C(16)-C(4)-H(4C)	109.0(2)
H(4B)-C(4)-H(4C)	107.8
O(1)-C(11)-C(16)	119.8(3)
O(1)-C(11)-C(12)	121.3(3)
C(16)-C(11)-C(12)	118.9(3)
C(13)-C(12)-C(11)	119.1(3)
C(13)-C(12)-C(1)	119.6(3)
C(11)-C(12)-C(1)	121.1(3)
C(12)-C(13)-C(14)	123.6(3)
C(12)-C(13)-H(13A)	118.2(2)
C(14)-C(13)-H(13A)	118.2(2)
C(13)-C(14)-C(15)	115.9(3)
C(13)-C(14)-C(10)	120.5(3)
C(15)-C(14)-C(10)	123.6(3)
C(16)-C(15)-C(14)	122.8(3)
C(16)-C(15)-H(15A)	118.6(2)
C(14)-C(15)-H(15A)	118.6(2)
C(15)-C(16)-C(11)	119.5(3)
C(15)-C(16)-C(4)	120.1(3)
C(11)-C(16)-C(4)	120.3(3)
O(2)-C(21)-C(26)	120.1(3)
O(2)-C(21)-C(22)	120.0(3)
C(26)-C(21)-C(22)	119.9(3)
C(23)-C(22)-C(21)	118.5(3)
C(23)-C(22)-C(2)	120.7(3)
C(21)-C(22)-C(2)	120.6(3)
C(22)-C(23)-C(24)	123.3(3)
C(22)-C(23)-H(23A)	118.3(2)
C(24)-C(23)-H(23A)	118.3(2)
C(25)-C(24)-C(23)	116.4(3)
C(25)-C(24)-C(20)	123.5(3)
C(23)-C(24)-C(20)	120.1(3)
C(26)-C(25)-C(24)	122.7(3)
C(26)-C(25)-H(25A)	118.7(2)
C(24)-C(25)-H(25A)	118.7(2)
C(25)-C(26)-C(21)	119.2(3)
C(25)-C(26)-C(1)	120.3(3)
C(21)-C(26)-C(1)	120.4(3)
O(3)-C(31)-C(36)	118.8(3)
O(3)-C(31)-C(32)	120.4(3)
C(36)-C(31)-C(32)	120.7(3)
C(33)-C(32)-C(31)	118.8(3)
C(33)-C(32)-C(3)	119.7(3)
C(31)-C(32)-C(3)	121.5(3)
C(34)-C(33)-C(32)	122.6(3)
C(34)-C(33)-H(33A)	118.7(2)
C(32)-C(33)-H(33A)	118.7(2)
C(33)-C(34)-C(35)	116.9(3)
C(33)-C(34)-C(30)	121.1(3)
C(35)-C(34)-C(30)	122.0(3)
C(34)-C(35)-C(36)	122.8(3)
C(34)-C(35)-H(35A)	118.6(2)
C(36)-C(35)-H(35A)	118.6(2)
C(31)-C(36)-C(35)	118.2(3)
C(31)-C(36)-C(2)	121.4(3)
C(35)-C(36)-C(2)	120.3(3)

O(4)-C(41)-C(42)	121.4(3)
O(4)-C(41)-C(46)	118.6(3)
C(42)-C(41)-C(46)	119.9(3)
C(41)-C(42)-C(43)	119.1(3)
C(41)-C(42)-C(4)	121.4(3)
C(43)-C(42)-C(4)	119.5(3)
C(44)-C(43)-C(42)	122.7(3)
C(44)-C(43)-H(43A)	118.7(2)
C(42)-C(43)-H(43A)	118.7(2)
C(43)-C(44)-C(45)	116.5(3)
C(43)-C(44)-C(40)	123.4(3)
C(45)-C(44)-C(40)	120.1(3)
C(46)-C(45)-C(44)	122.8(3)
C(46)-C(45)-H(45A)	118.6(2)
C(44)-C(45)-H(45A)	118.6(2)
C(45)-C(46)-C(41)	118.9(3)
C(45)-C(46)-C(3)	119.6(3)
C(41)-C(46)-C(3)	121.4(3)
C(14)-C(10)-C(18)	112.2(3)
C(14)-C(10)-C(17)	109.0(3)
C(18)-C(10)-C(17)	108.8(3)
C(14)-C(10)-C(19)	110.1(3)
C(18)-C(10)-C(19)	108.2(3)
C(17)-C(10)-C(19)	108.5(3)
C(10)-C(17)-H(17A)	109.5(2)
C(10)-C(17)-H(17B)	109.5(2)
H(17A)-C(17)-H(17B)	109.5
C(10)-C(17)-H(17C)	109.5(2)
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(10)-C(18)-H(18A)	109.5(2)
C(10)-C(18)-H(18B)	109.5(2)
H(18A)-C(18)-H(18B)	109.5
C(10)-C(18)-H(18C)	109.5(2)
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(10)-C(19)-H(19A)	109.5(2)
C(10)-C(19)-H(19B)	109.5(2)
H(19A)-C(19)-H(19B)	109.5
C(10)-C(19)-H(19C)	109.5(2)
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(29)-C(20)-C(27)	109.6(3)
C(29)-C(20)-C(28)	107.7(3)
C(27)-C(20)-C(28)	108.4(3)
C(29)-C(20)-C(24)	109.6(3)
C(27)-C(20)-C(24)	109.3(3)
C(28)-C(20)-C(24)	112.2(3)
C(20)-C(27)-H(27A)	109.5(2)
C(20)-C(27)-H(27B)	109.5(2)
H(27A)-C(27)-H(27B)	109.5
C(20)-C(27)-H(27C)	109.5(2)
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(20)-C(28)-H(28A)	109.5(2)
C(20)-C(28)-H(28B)	109.5(2)
H(28A)-C(28)-H(28B)	109.5
C(20)-C(28)-H(28C)	109.5(2)
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(20)-C(29)-H(29A)	109.5(2)
C(20)-C(29)-H(29B)	109.5(2)
H(29A)-C(29)-H(29B)	109.5
C(20)-C(29)-H(29C)	109.5(2)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(39)-C(30)-C(38)	109.4(5)
C(39)-C(30)-C(37)	106.3(4)
C(38)-C(30)-C(37)	109.4(5)
C(39)-C(30)-C(34)	113.1(3)
C(38)-C(30)-C(34)	109.0(3)
C(37D)-C(30)-C(34)	114.5(8)
C(37)-C(30)-C(34)	109.6(3)
C(37D)-C(30)-C(39D)	107.1(13)
C(34)-C(30)-C(39D)	109.2(10)
C(37D)-C(30)-C(38D)	104.7(12)
C(34)-C(30)-C(38D)	107.8(9)
C(39D)-C(30)-C(38D)	113.6(14)
C(30)-C(37)-H(37A)	109.5(3)
C(30)-C(37)-H(37B)	109.5(4)
H(37A)-C(37)-H(37B)	109.5
C(30)-C(37)-H(37C)	109.5(3)
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
C(30)-C(38)-H(38A)	109.5(4)
C(30)-C(38)-H(38B)	109.5(4)
H(38A)-C(38)-H(38B)	109.5
C(30)-C(38)-H(38C)	109.5(3)
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5
C(30)-C(39)-H(39A)	109.5(3)
C(30)-C(39)-H(39B)	109.5(2)
H(39A)-C(39)-H(39B)	109.5
C(30)-C(39)-H(39C)	109.5(3)
H(39A)-C(39)-H(39C)	109.5
H(39B)-C(39)-H(39C)	109.5
C(30)-C(37D)-H(37E)	109.5(8)
C(30)-C(37D)-H(37F)	109.5(8)
H(37E)-C(37D)-H(37F)	109.5
C(30)-C(37D)-H(37G)	109.5(9)
H(37E)-C(37D)-H(37G)	109.5
H(37F)-C(37D)-H(37G)	109.5
C(30)-C(38D)-H(38E)	109.5(11)
C(30)-C(38D)-H(38F)	109.5(10)
H(38E)-C(38D)-H(38F)	109.5
C(30)-C(38D)-H(38G)	109.5(9)
H(38E)-C(38D)-H(38G)	109.5
H(38F)-C(38D)-H(38G)	109.5
C(30)-C(39D)-H(39E)	109.5(10)
C(30)-C(39D)-H(39F)	109.5(12)
H(39E)-C(39D)-H(39F)	109.5
C(30)-C(39D)-H(39G)	109.5(11)
H(39E)-C(39D)-H(39G)	109.5
H(39F)-C(39D)-H(39G)	109.5
C(44)-C(40)-C(48)	109.0(3)
C(44)-C(40)-C(47)	112.1(3)
C(48)-C(40)-C(47)	109.0(3)
C(44)-C(40)-C(49)	109.3(3)
C(48)-C(40)-C(49)	109.8(3)
C(47)-C(40)-C(49)	107.6(3)
C(40)-C(47)-H(47A)	109.5(2)
C(40)-C(47)-H(47B)	109.5(2)
H(47A)-C(47)-H(47B)	109.5
C(40)-C(47)-H(47C)	109.5(2)
H(47A)-C(47)-H(47C)	109.5
H(47B)-C(47)-H(47C)	109.5
C(40)-C(48)-H(48A)	109.5(2)
C(40)-C(48)-H(48B)	109.5(2)
H(48A)-C(48)-H(48B)	109.5
C(40)-C(48)-H(48C)	109.5(2)
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(40)-C(49)-H(49A)	109.5(2)
C(40)-C(49)-H(49B)	109.5(2)
H(49A)-C(49)-H(49B)	109.5
C(40)-C(49)-H(49C)	109.5(2)
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
C(71)-P(1)-C(8)	110.2(2)
C(71)-P(1)-C(61)	108.33(14)
C(8)-P(1)-C(61)	107.4(2)
C(71)-P(1)-C(51)	113.57(14)
C(8)-P(1)-C(51)	109.0(2)
C(61)-P(1)-C(51)	108.24(14)
P(1)-C(8)-H(8C)	109(2)
P(1)-C(8)-H(8B)	110(2)
H(8C)-C(8)-H(8B)	110(3)
P(1)-C(8)-H(8A)	110(2)

H(8C)-C(8)-H(8A)	106(3)
H(8B)-C(8)-H(8A)	111(3)
C(56)-C(51)-C(52)	119.8(3)
C(56)-C(51)-P(1)	120.1(2)
C(52)-C(51)-P(1)	119.9(2)
C(53)-C(52)-C(51)	119.9(3)
C(53)-C(52)-H(52A)	120.1(2)
C(51)-C(52)-H(52A)	120.1(2)
C(54)-C(53)-C(52)	120.2(3)
C(54)-C(53)-H(53A)	119.9(2)
C(52)-C(53)-H(53A)	119.9(2)
C(53)-C(54)-C(55)	120.4(3)
C(53)-C(54)-H(54A)	119.8(2)
C(55)-C(54)-H(54A)	119.8(2)
C(54)-C(55)-C(56)	120.0(3)
C(54)-C(55)-H(55A)	120.0(2)
C(56)-C(55)-H(55A)	120.0(2)
C(51)-C(56)-C(55)	119.6(3)
C(51)-C(56)-H(56A)	120.2(2)
C(55)-C(56)-H(56A)	120.2(2)
C(66)-C(61)-C(62)	120.0(3)
C(66)-C(61)-P(1)	122.2(2)
C(62)-C(61)-P(1)	117.7(2)
C(63)-C(62)-C(61)	119.8(3)
C(63)-C(62)-H(62A)	120.1(2)
C(61)-C(62)-H(62A)	120.1(2)
C(64)-C(63)-C(62)	119.9(3)
C(64)-C(63)-H(63A)	120.0(2)
C(62)-C(63)-H(63A)	120.0(2)
C(63)-C(64)-C(65)	120.7(3)
C(63)-C(64)-H(64A)	119.6(2)
C(65)-C(64)-H(64A)	119.6(2)
C(64)-C(65)-C(66)	120.0(3)
C(64)-C(65)-H(65A)	120.0(2)
C(66)-C(65)-H(65A)	120.0(2)
C(65)-C(66)-C(61)	119.5(3)
C(65)-C(66)-H(66A)	120.2(2)
C(61)-C(66)-H(66A)	120.2(2)
C(76)-C(71)-C(72)	119.5(3)
C(76)-C(71)-P(1)	121.1(2)
C(72)-C(71)-P(1)	119.2(3)
C(73)-C(72)-C(71)	120.2(3)
C(73)-C(72)-H(72A)	119.9(2)
C(71)-C(72)-H(72A)	119.9(2)
C(72)-C(73)-C(74)	120.0(3)
C(72)-C(73)-H(73A)	120.0(2)
C(74)-C(73)-H(73A)	120.0(2)
C(73)-C(74)-C(75)	120.1(3)
C(73)-C(74)-H(74A)	119.9(2)
C(75)-C(74)-H(74A)	119.9(2)
C(74)-C(75)-C(76)	120.5(3)
C(74)-C(75)-H(75A)	119.8(2)
C(76)-C(75)-H(75A)	119.8(2)
C(75)-C(76)-C(71)	119.7(3)
C(75)-C(76)-H(76A)	120.1(2)
C(71)-C(76)-H(76A)	120.1(2)
N(1X)-C(11X)-C(12X)	179.3(5)
C(11X)-C(12X)-H(1XA)	109.5(3)
C(11X)-C(12X)-H(1XB)	109.5(2)
H(1XA)-C(12X)-H(1XB)	109.5
C(11X)-C(12X)-H(1XC)	109.5(3)
H(1XA)-C(12X)-H(1XC)	109.5
H(1XB)-C(12X)-H(1XC)	109.5
N(2X)-C(21X)-C(22X)	178.2(4)
C(21X)-C(22X)-H(2XA)	109.5(2)
C(21X)-C(22X)-H(2XB)	109.5(2)
H(2XA)-C(22X)-H(2XB)	109.5
C(21X)-C(22X)-H(2XC)	109.5(2)
H(2XA)-C(22X)-H(2XC)	109.5
H(2XB)-C(22X)-H(2XC)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4 Anisotropic displacement parameters (A^2 x 10^3) for 3.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	24(1)	30(1)	22(1)	2(1)	2(1)	2(1)
O(2)	29(1)	32(1)	35(1)	4(1)	-4(1)	-4(1)
O(3)	25(1)	39(1)	31(1)	3(1)	-3(1)	3(1)
O(4)	29(1)	26(1)	36(1)	4(1)	7(1)	1(1)
C(1)	35(2)	22(2)	26(2)	1(1)	-4(1)	4(1)
C(2)	31(2)	28(2)	22(2)	5(1)	5(1)	2(1)
C(3)	27(2)	27(2)	24(2)	1(1)	-1(1)	-6(1)
C(4)	25(2)	24(2)	19(2)	3(1)	-2(1)	0(1)
C(11)	22(2)	21(2)	22(2)	7(1)	-1(1)	4(1)
C(12)	30(2)	19(2)	23(2)	4(1)	-4(1)	4(1)
C(13)	33(2)	19(2)	26(2)	1(1)	-7(1)	2(1)
C(14)	24(2)	21(2)	31(2)	6(1)	-3(1)	2(1)
C(15)	26(2)	20(2)	25(2)	6(1)	1(1)	6(1)
C(16)	26(2)	18(2)	24(2)	5(1)	-3(1)	2(1)
C(21)	25(2)	29(2)	21(2)	-1(1)	5(1)	5(1)
C(22)	27(2)	25(2)	22(2)	0(1)	8(1)	3(1)
C(23)	34(2)	27(2)	20(2)	2(1)	2(1)	6(1)
C(24)	29(2)	26(2)	21(2)	-3(1)	-1(1)	3(1)
C(25)	32(2)	21(2)	23(2)	-2(1)	4(1)	1(1)
C(26)	29(2)	22(2)	18(2)	-2(1)	4(1)	8(1)
C(31)	21(2)	23(2)	25(2)	4(1)	1(1)	-5(1)
C(32)	24(2)	22(2)	23(2)	3(1)	-1(1)	-9(1)

C(33)	29(2)	22(2)	27(2)	1(1)	1(1)	-1(1)
C(34)	25(2)	24(2)	25(2)	4(1)	2(1)	-2(1)
C(35)	26(2)	29(2)	19(2)	2(1)	2(1)	-3(1)
C(36)	24(2)	23(2)	25(2)	4(1)	5(1)	-3(1)
C(41)	23(2)	24(2)	20(2)	-1(1)	-2(1)	-2(1)
C(42)	26(2)	21(2)	17(2)	0(1)	-7(1)	-2(1)
C(43)	27(2)	20(2)	20(2)	-1(1)	-2(1)	-4(1)
C(44)	25(2)	21(2)	23(2)	-2(1)	-5(1)	-1(1)
C(45)	27(2)	20(2)	25(2)	-1(1)	-5(1)	-2(1)
C(46)	25(2)	23(2)	20(2)	1(1)	-5(1)	-6(1)
C(10)	27(2)	25(2)	44(2)	5(2)	-5(2)	-3(1)
C(17)	37(2)	62(3)	76(3)	29(2)	-21(2)	-11(2)
C(18)	28(2)	43(2)	68(3)	-6(2)	13(2)	-5(2)
C(19)	39(2)	30(2)	43(2)	1(2)	-3(2)	-6(2)
C(20)	33(2)	32(2)	30(2)	2(2)	-9(1)	-1(2)
C(27)	43(2)	62(3)	69(3)	-8(2)	-18(2)	13(2)
C(28)	48(2)	59(3)	51(3)	10(2)	-17(2)	-18(2)
C(29)	61(3)	69(3)	28(2)	-3(2)	-9(2)	-12(2)
C(30)	31(2)	34(2)	32(2)	0(2)	-3(1)	8(2)
C(37)	106(6)	38(3)	99(6)	-5(3)	-54(5)	26(3)
C(38)	33(3)	143(8)	115(7)	73(6)	21(4)	33(4)
C(39)	63(4)	97(5)	37(3)	-13(3)	-24(3)	45(4)
C(40)	26(2)	24(2)	30(2)	-2(1)	-1(1)	0(1)
C(47)	30(2)	40(2)	49(2)	3(2)	10(2)	2(2)
C(48)	31(2)	37(2)	40(2)	-3(2)	-3(2)	3(2)
C(49)	36(2)	33(2)	41(2)	-11(2)	1(2)	0(2)
P(1)	23(1)	22(1)	27(1)	-3(1)	-3(1)	2(1)
C(8)	35(2)	27(2)	42(2)	-9(2)	-12(2)	3(2)
C(51)	26(2)	25(2)	24(2)	-3(1)	0(1)	5(1)
C(52)	27(2)	38(2)	29(2)	-1(2)	-4(1)	6(2)
C(53)	41(2)	41(2)	26(2)	7(2)	1(2)	7(2)
C(54)	43(2)	36(2)	35(2)	8(2)	10(2)	3(2)
C(55)	32(2)	33(2)	39(2)	-2(2)	1(2)	-6(2)
C(56)	30(2)	28(2)	30(2)	-2(1)	-6(1)	1(1)
C(61)	24(2)	20(2)	25(2)	0(1)	-5(1)	-1(1)
C(62)	28(2)	25(2)	25(2)	3(1)	-3(1)	0(1)
C(63)	28(2)	24(2)	32(2)	-6(1)	-2(1)	4(1)
C(64)	31(2)	23(2)	39(2)	-1(2)	-9(2)	6(1)
C(65)	38(2)	29(2)	28(2)	7(1)	-10(2)	0(2)
C(66)	29(2)	29(2)	27(2)	-2(1)	1(1)	-1(1)
C(71)	21(2)	26(2)	31(2)	2(1)	-1(1)	3(1)
C(72)	31(2)	27(2)	50(2)	4(2)	3(2)	0(2)
C(73)	33(2)	38(2)	48(2)	16(2)	6(2)	2(2)
C(74)	31(2)	56(2)	31(2)	6(2)	2(2)	7(2)
C(75)	34(2)	46(2)	33(2)	-10(2)	0(2)	2(2)
C(76)	25(2)	31(2)	38(2)	-4(2)	-3(2)	-2(1)
N(1X)	58(3)	92(4)	187(6)	1(4)	4(3)	-4(3)
C(11X)	52(3)	43(2)	75(3)	3(2)	1(2)	-1(2)
C(12X)	47(2)	55(3)	57(3)	8(2)	-9(2)	1(2)
N(2X)	73(3)	43(2)	64(3)	1(2)	8(2)	-19(2)
C(21X)	43(2)	40(2)	43(2)	10(2)	0(2)	-16(2)
C(22X)	57(3)	68(3)	53(3)	-4(2)	20(2)	-27(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 3.

	x	y	z	U(eq)
H(1A)	6270(9)	2169(25)	1527(1)	38
H(2A)	5976(12)	1228(2)	2122(12)	48
H(3A)	5900(7)	-214(27)	1578(4)	47
H(4A)	6259(8)	998(7)	980(13)	46
H(1B)	6331(2)	3757(2)	1919(1)	34
H(1C)	7002(2)	4486(2)	2098(1)	34
H(2B)	6433(2)	15(2)	3277(1)	32
H(2C)	5921(2)	350(2)	2792(1)	32
H(3B)	6699(2)	-2542(2)	1083(1)	31
H(3C)	6094(2)	-1636(2)	1139(1)	31
H(4B)	6510(2)	1807(2)	239(1)	27
H(4C)	7226(2)	1941(2)	-108(1)	27
H(13A)	8117(2)	4452(2)	1614(1)	31
H(15A)	8308(2)	2777(2)	271(1)	28
H(23A)	7585(2)	993(2)	3431(1)	32
H(25A)	7963(2)	3732(2)	2668(1)	30
H(33A)	7721(2)	-2603(2)	1717(1)	31
H(35A)	7580(2)	-1005(2)	3086(1)	29
H(43A)	8178(2)	622(2)	-73(1)	27
H(45A)	7790(2)	-2204(2)	610(1)	29
H(17A)	9371(8)	4122(20)	1663(2)	88
H(17B)	10075(5)	4177(19)	1297(6)	88
H(17C)	9634(13)	3089(4)	1352(7)	88
H(18A)	9962(6)	4115(16)	332(5)	70
H(18B)	9183(7)	4021(18)	50(2)	70
H(18C)	9518(13)	3027(3)	360(5)	70
H(19A)	8852(12)	5658(3)	1176(4)	56
H(19B)	8741(10)	5610(3)	551(6)	56
H(19C)	9542(3)	5699(3)	797(9)	56
H(27A)	9543(7)	1657(12)	3560(9)	87
H(27B)	9228(13)	1535(14)	2974(3)	87
H(27C)	8854(6)	912(4)	3450(11)	87
H(28A)	9438(8)	3596(12)	3533(8)	79
H(28B)	8688(6)	4153(4)	3367(10)	79
H(28C)	9150(13)	3524(10)	2937(3)	79
H(29A)	8825(3)	2646(23)	4241(2)	79
H(29B)	8132(14)	1913(10)	4124(3)	79
H(29C)	8063(12)	3175(14)	4084(2)	79
H(37A)	8260(27)	-3851(6)	2180(4)	122
H(37B)	7907(16)	-3961(8)	2752(16)	122
H(37C)	8767(12)	-4089(6)	2682(18)	122
H(38A)	9171(19)	-1376(13)	2413(18)	145
H(38B)	9025(14)	-2253(42)	1969(4)	145
H(38C)	9527(6)	-2529(34)	2468(17)	145
H(39A)	8683(22)	-1630(10)	3304(3)	99
H(39B)	8957(16)	-2831(26)	3348(4)	99
H(39C)	8115(8)	-2559(32)	3423(2)	99
H(37E)	8362(21)	-3886(45)	2197(30)	67(80)
H(37F)	9164(32)	-3680(54)	2423(17)	0(31)
H(37G)	8867(49)	-3001(21)	1934(15)	27(44)
H(38E)	8940(30)	-1202(58)	3002(30)	18(39)
H(38F)	9119(42)	-1292(62)	2387(17)	33(52)
H(38G)	9513(18)	-2063(19)	2801(46)	111(103)
H(39E)	8660(21)	-3498(65)	3296(22)	147(133)
H(39F)	7836(37)	-3607(53)	3097(12)	0(31)
H(39G)	8081(48)	-2592(22)	3432(14)	0(31)
H(47A)	9617(7)	-904(8)	-528(7)	59
H(47B)	9318(12)	83(8)	-198(3)	59
H(47C)	8845(5)	-392(15)	-674(5)	59
H(48A)	9779(5)	-1906(16)	312(2)	54
H(48B)	9105(5)	-2098(14)	696(5)	54
H(48C)	9455(10)	-942(4)	646(6)	54
H(49A)	9062(3)	-2643(10)	-467(8)	55
H(49B)	8263(9)	-2195(4)	-586(6)	55
H(49C)	8414(12)	-2882(7)	-67(2)	55

H(8C)	5324(22)	3232(30)	1237(15)	54(12)
H(8B)	4964(20)	4282(31)	999(14)	46(11)
H(8A)	4573(21)	3634(30)	1476(16)	54(12)
H(52A)	4873(2)	1523(3)	1530(1)	37
H(53A)	4390(2)	71(3)	1979(1)	43
H(54A)	3305(2)	-693(3)	1694(1)	46
H(55A)	2723(2)	-76(3)	932(1)	42
H(56A)	3207(2)	1372(3)	469(1)	36
H(62A)	3093(2)	3436(2)	1148(1)	31
H(63A)	2123(2)	4557(2)	925(1)	34
H(64A)	2083(2)	5361(2)	99(1)	37
H(65A)	2970(2)	4993(3)	-527(1)	38
H(66A)	3939(2)	3856(3)	-316(1)	34
H(72A)	5490(2)	4008(3)	138(2)	43
H(73A)	6154(2)	3804(3)	-636(2)	48
H(74A)	6037(2)	2256(3)	-1133(1)	47
H(75A)	5274(2)	897(3)	-850(1)	45
H(76A)	4591(2)	1096(3)	-84(1)	37
H(1XA)	8093(2)	345(17)	1414(7)	80
H(1XB)	8080(2)	1521(5)	1651(11)	80
H(1XC)	8081(2)	520(20)	2039(4)	80
H(2XA)	5006(11)	3269(13)	-2325(7)	89
H(2XB)	4171(6)	2922(18)	-2362(6)	89
H(2XC)	4722(15)	2278(8)	-1991(2)	89

4) *p-tert*-butylcalix[4]arene triphenylaminophosphonium

Table 1. Crystal data and structure refinement for 4.

Identification code	4 (98srv064)
Empirical formula	C66 H78 N3 O4 P
Formula weight	1008.28
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	a = 18.161(4) A alpha = 90 deg. b = 12.592(3) A beta = 90.63(3) deg. c = 25.652(5) A gamma = 90 deg.
Volume, Z	5866(2) A^3, 4
Density (calculated)	1.142 Mg/m^3
Absorption coefficient	0.096 mm^-1
F(000)	2168
Crystal size	0.65 x 0.15 x 0.15 mm
Theta range for data collection	1.12 to 27.46 deg.
Limiting indices	-21<=h<=23, -15<=k<=16, -19<=l<=33
Reflections collected	41742
Independent reflections	13400 [R(int) = 0.1332]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13302 / 0 / 707
Goodness-of-fit on F^2	1.272
Final R indices [I>2sigma(I)]	R1 = 0.0939, wR2 = 0.1714
R indices (all data)	R1 = 0.1680, wR2 = 0.2235
Extinction coefficient	not refined
Largest diff. peak and hole	0.464 and -0.565 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 4. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	6284(1)	2345(2)	1199(1)	22(1)
O(3)	5827(1)	-381(2)	1908(1)	27(1)
O(2)	6030(1)	1893(2)	2158(1)	28(1)
O(4)	6201(1)	382(2)	951(1)	25(1)
C(1)	6859(2)	3813(3)	1978(1)	24(1)
C(11)	6950(2)	2807(3)	1116(1)	20(1)
C(12)	7252(2)	3542(3)	1475(1)	22(1)
C(13)	7941(2)	3976(3)	1381(2)	25(1)
C(14)	8365(2)	3702(3)	949(2)	24(1)
C(15)	8055(2)	2978(3)	599(2)	23(1)
C(16)	7355(2)	2542(3)	671(1)	20(1)
C(10)	9131(2)	4206(3)	879(2)	31(1)
C(17)	9623(3)	3936(4)	1349(2)	57(2)
C(18)	9507(2)	3803(4)	383(2)	47(1)
C(19)	9060(2)	5422(3)	837(2)	37(1)
C(2)	6408(2)	295(3)	2894(1)	24(1)
C(21)	6633(2)	2094(3)	2465(1)	23(1)
C(22)	6825(2)	1340(3)	2852(1)	23(1)
C(23)	7436(2)	1546(3)	3165(1)	25(1)
C(24)	7882(2)	2449(3)	3108(1)	25(1)
C(25)	7668(2)	3171(3)	2722(1)	25(1)
C(26)	7048(2)	3016(3)	2403(1)	22(1)
C(20)	8574(2)	2597(3)	3456(2)	32(1)
C(27)	9099(3)	1659(4)	3373(2)	58(2)
C(28)	8985(3)	3635(4)	3332(2)	47(1)
C(29)	8339(3)	2635(4)	4029(2)	49(1)
C(3)	6602(2)	-1761(3)	1196(1)	23(1)
C(31)	6489(2)	-824(3)	2070(1)	21(1)
C(32)	6862(2)	-1546(3)	1750(1)	20(1)
C(33)	7485(2)	-2057(3)	1947(1)	22(1)
C(34)	7754(2)	-1873(3)	2451(2)	24(1)
C(35)	7389(2)	-1119(3)	2749(1)	23(1)
C(36)	6761(2)	-572(3)	2565(1)	22(1)
C(30)	8441(2)	-2463(3)	2657(2)	28(1)
C(37)	9122(3)	-1963(7)	2435(3)	107(3)
C(38)	8505(3)	-2427(6)	3249(2)	75(2)
C(39)	8411(4)	-3628(5)	2506(3)	114(3)
C(4)	7042(2)	1763(3)	271(1)	21(1)
C(41)	6819(2)	-18(3)	713(1)	19(1)
C(42)	7246(2)	608(3)	382(1)	19(1)
C(43)	7872(2)	175(3)	157(1)	20(1)
C(44)	8086(2)	-871(3)	241(1)	20(1)
C(45)	7640(2)	-1482(3)	566(1)	20(1)
C(46)	7019(2)	-1081(3)	809(1)	18(1)
C(40)	8782(2)	-1360(3)	1(2)	26(1)
C(47)	8563(2)	-2376(3)	-300(2)	33(1)
C(48)	9332(2)	-1639(3)	444(2)	34(1)
C(49)	9154(2)	-605(3)	-384(2)	37(1)
P(1)	4491(1)	2839(1)	603(1)	21(1)
N(1)	5028(2)	3489(3)	992(2)	33(1)
C(51)	3723(2)	3665(3)	425(1)	22(1)
C(52)	3648(2)	4118(3)	-70(2)	26(1)
C(53)	3057(2)	4775(3)	-183(2)	29(1)
C(54)	2532(2)	4976(3)	199(2)	28(1)
C(55)	2590(2)	4509(3)	683(2)	27(1)
C(56)	3184(2)	3857(3)	802(2)	26(1)
C(61)	4110(2)	1701(3)	930(2)	22(1)
C(62)	4417(2)	1371(3)	1403(2)	28(1)
C(63)	4085(2)	557(3)	1682(2)	34(1)
C(64)	3448(2)	82(3)	1491(2)	32(1)
C(65)	3152(2)	395(3)	1016(2)	32(1)
C(66)	3481(2)	1195(3)	728(2)	26(1)
C(71)	4977(2)	2478(3)	27(2)	23(1)
C(72)	5453(2)	3250(3)	-183(2)	30(1)

C(73)	5836(2)	3046(3)	-634(2)	36(1)
C(74)	5754(2)	2067(4)	-884(2)	35(1)
C(75)	5297(2)	1297(3)	-674(2)	32(1)
C(76)	4906(2)	1498(3)	-222(2)	25(1)
N(1X)	9682(3)	887(5)	1706(3)	118(3)
C(11X)	9054(3)	868(4)	1714(2)	59(2)
C(12X)	8253(3)	829(5)	1727(2)	50(1)
N(2X)	5459(3)	-4351(3)	1375(2)	62(1)
C(21X)	5430(2)	-3740(4)	1700(2)	39(1)
C(22X)	5383(3)	-2945(4)	2111(2)	44(1)

Table 3. Bond lengths [A] and angles [deg] for 4.

O(1)-C(11)	1.361(4)
O(3)-C(31)	1.385(4)
O(3)-H(3)	0.88(5)
O(2)-C(21)	1.365(4)
O(2)-H(2)	0.91(5)
O(4)-C(41)	1.380(4)
O(4)-H(4)	0.92(6)
C(1)-C(12)	1.520(5)
C(1)-C(26)	1.520(5)
C(1)-H(1C)	0.99
C(1)-H(1D)	0.99
C(11)-C(16)	1.405(5)
C(11)-C(12)	1.412(5)
C(12)-C(13)	1.389(5)
C(13)-C(14)	1.400(5)
C(13)-H(13)	0.95
C(14)-C(15)	1.394(5)
C(14)-C(10)	1.541(5)
C(15)-C(16)	1.399(5)
C(15)-H(15)	0.95
C(16)-C(4)	1.524(5)
C(10)-C(17)	1.532(6)
C(10)-C(18)	1.537(6)
C(10)-C(19)	1.540(6)
C(17)-H(17A)	0.98
C(17)-H(17B)	0.98
C(17)-H(17C)	0.98
C(18)-H(18A)	0.98
C(18)-H(18B)	0.98
C(18)-H(18C)	0.98
C(19)-H(19A)	0.98
C(19)-H(19B)	0.98
C(19)-H(19C)	0.98
C(2)-C(22)	1.522(5)
C(2)-C(36)	1.525(5)
C(2)-H(2A)	0.99
C(2)-H(2B)	0.99
C(21)-C(26)	1.395(5)
C(21)-C(22)	1.414(5)
C(22)-C(23)	1.388(5)
C(23)-C(24)	1.403(5)
C(23)-H(23A)	0.95
C(24)-C(25)	1.397(5)
C(24)-C(20)	1.545(5)
C(25)-C(26)	1.397(5)
C(25)-H(25A)	0.95
C(20)-C(27)	1.535(6)
C(20)-C(29)	1.536(6)
C(20)-C(28)	1.541(6)
C(27)-H(27A)	0.98
C(27)-H(27B)	0.98
C(27)-H(27C)	0.98
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(3)-C(32)	1.518(5)
C(3)-C(46)	1.519(5)
C(3)-H(3A)	0.99
C(3)-H(3B)	0.99
C(31)-C(36)	1.393(5)
C(31)-C(32)	1.405(5)
C(32)-C(33)	1.391(5)
C(33)-C(34)	1.398(5)
C(33)-H(33A)	0.95
C(34)-C(35)	1.391(5)
C(34)-C(30)	1.540(5)
C(35)-C(36)	1.410(5)
C(35)-H(35A)	0.95
C(30)-C(37)	1.506(7)
C(30)-C(39)	1.519(7)
C(30)-C(38)	1.522(6)
C(37)-H(37A)	0.98
C(37)-H(37B)	0.98
C(37)-H(37C)	0.98
C(38)-H(38A)	0.98
C(38)-H(38B)	0.98
C(38)-H(38C)	0.98
C(39)-H(39A)	0.98
C(39)-H(39B)	0.98
C(39)-H(39C)	0.98
C(4)-C(42)	1.527(5)
C(4)-H(4A)	0.99
C(4)-H(4B)	0.99
C(41)-C(42)	1.400(5)
C(41)-C(46)	1.407(5)
C(42)-C(43)	1.391(5)
C(43)-C(44)	1.390(5)
C(43)-H(43A)	0.95
C(44)-C(45)	1.399(5)
C(44)-C(40)	1.540(5)
C(45)-C(46)	1.389(5)
C(45)-H(45A)	0.95
C(40)-C(49)	1.535(5)
C(40)-C(47)	1.544(5)
C(40)-C(48)	1.546(5)
C(47)-H(47A)	0.98
C(47)-H(47B)	0.98
C(47)-H(47C)	0.98
C(48)-H(48A)	0.98
C(48)-H(48B)	0.98
C(48)-H(48C)	0.98
C(49)-H(49A)	0.98
C(49)-H(49B)	0.98
C(49)-H(49C)	0.98
P(1)-N(1)	1.611(4)
P(1)-C(71)	1.787(4)
P(1)-C(51)	1.795(4)
P(1)-C(61)	1.802(4)
N(1)-H(1A)	0.83(5)
N(1)-H(1B)	0.89(5)

C(51)-C(52)	1.397(5)
C(51)-C(56)	1.406(5)
C(52)-C(53)	1.385(5)
C(52)-H(52A)	0.95
C(53)-C(54)	1.397(6)
C(53)-H(53A)	0.95
C(54)-C(55)	1.379(5)
C(54)-H(54A)	0.95
C(55)-C(56)	1.386(5)
C(55)-H(55A)	0.95
C(56)-H(56A)	0.95
C(61)-C(62)	1.395(5)
C(61)-C(66)	1.402(5)
C(62)-C(63)	1.391(6)
C(62)-H(62A)	0.95
C(63)-C(64)	1.387(6)
C(63)-H(63A)	0.95
C(64)-C(65)	1.384(6)
C(64)-H(64A)	0.95
C(65)-C(66)	1.388(5)
C(65)-H(65A)	0.95
C(66)-H(66A)	0.95
C(71)-C(76)	1.396(5)
C(71)-C(72)	1.411(5)
C(72)-C(73)	1.381(6)
C(72)-H(72A)	0.95
C(73)-C(74)	1.397(6)
C(73)-H(73A)	0.95
C(74)-C(75)	1.388(6)
C(74)-H(74A)	0.95
C(75)-C(76)	1.390(5)
C(75)-H(75A)	0.95
C(76)-H(76A)	0.95
N(1X)-C(11X)	1.142(7)
C(11X)-C(12X)	1.456(7)
C(12X)-H(1AX)	0.98
C(12X)-H(1BX)	0.98
C(12X)-H(1CX)	0.98
N(2X)-C(21X)	1.138(6)
C(21X)-C(22X)	1.457(7)
C(22X)-H(2AX)	0.98
C(22X)-H(2BX)	0.98
C(22X)-H(2CX)	0.98
C(31)-O(3)-H(3)	102(3)
C(21)-O(2)-H(2)	110(3)
C(41)-O(4)-H(4)	117(4)
C(12)-C(1)-C(26)	110.9(3)
C(12)-C(1)-H(1C)	109.5(2)
C(26)-C(1)-H(1C)	109.5(2)
C(12)-C(1)-H(1D)	109.5(2)
C(26)-C(1)-H(1D)	109.5(2)
H(1C)-C(1)-H(1D)	108.0
O(1)-C(11)-C(16)	120.0(3)
O(1)-C(11)-C(12)	121.2(3)
C(16)-C(11)-C(12)	118.9(3)
C(13)-C(12)-C(11)	119.3(3)
C(13)-C(12)-C(1)	119.4(3)
C(11)-C(12)-C(1)	121.2(3)
C(12)-C(13)-C(14)	123.0(4)
C(12)-C(13)-H(13)	118.5(2)
C(14)-C(13)-H(13)	118.5(2)
C(15)-C(14)-C(13)	116.6(3)
C(15)-C(14)-C(10)	123.7(4)
C(13)-C(14)-C(10)	119.7(4)
C(14)-C(15)-C(16)	122.3(3)
C(14)-C(15)-H(15)	118.9(2)
C(16)-C(15)-H(15)	118.9(2)
C(15)-C(16)-C(11)	119.9(3)
C(15)-C(16)-C(4)	119.7(3)
C(11)-C(16)-C(4)	120.4(3)
C(17)-C(10)-C(18)	108.5(4)
C(17)-C(10)-C(19)	108.9(4)
C(18)-C(10)-C(19)	107.9(4)
C(17)-C(10)-C(14)	109.7(3)
C(18)-C(10)-C(14)	111.7(3)
C(19)-C(10)-C(14)	110.1(3)
C(10)-C(17)-H(17A)	109.5(3)
C(10)-C(17)-H(17B)	109.5(2)
H(17A)-C(17)-H(17B)	109.5
C(10)-C(17)-H(17C)	109.5(3)
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(10)-C(18)-H(18A)	109.5(3)
C(10)-C(18)-H(18B)	109.5(2)
H(18A)-C(18)-H(18B)	109.5
C(10)-C(18)-H(18C)	109.5(2)
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(10)-C(19)-H(19A)	109.5(2)
C(10)-C(19)-H(19B)	109.5(2)
H(19A)-C(19)-H(19B)	109.5
C(10)-C(19)-H(19C)	109.5(2)
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(22)-C(2)-C(36)	111.6(3)
C(22)-C(2)-H(2A)	109.3(2)
C(36)-C(2)-H(2A)	109.3(2)
C(22)-C(2)-H(2B)	109.3(2)
C(36)-C(2)-H(2B)	109.3(2)
H(2A)-C(2)-H(2B)	108.0
O(2)-C(21)-C(26)	121.3(3)
O(2)-C(21)-C(22)	118.1(3)
C(26)-C(21)-C(22)	120.5(3)
C(23)-C(22)-C(21)	118.1(3)
C(23)-C(22)-C(2)	121.1(3)
C(21)-C(22)-C(2)	120.6(3)
C(22)-C(23)-C(24)	123.3(3)
C(22)-C(23)-H(23A)	118.3(2)
C(24)-C(23)-H(23A)	118.3(2)
C(25)-C(24)-C(23)	116.4(4)
C(25)-C(24)-C(20)	123.3(4)
C(23)-C(24)-C(20)	120.3(3)
C(24)-C(25)-C(26)	122.7(4)
C(24)-C(25)-H(25A)	118.7(2)
C(26)-C(25)-H(25A)	118.7(2)
C(21)-C(26)-C(25)	118.9(3)
C(21)-C(26)-C(1)	120.8(3)
C(25)-C(26)-C(1)	120.2(3)
C(27)-C(20)-C(29)	109.7(4)
C(27)-C(20)-C(28)	108.7(4)
C(29)-C(20)-C(28)	108.3(4)
C(27)-C(20)-C(24)	109.2(3)
C(29)-C(20)-C(24)	108.9(3)
C(28)-C(20)-C(24)	112.1(3)
C(20)-C(27)-H(27A)	109.5(3)
C(20)-C(27)-H(27B)	109.5(2)
H(27A)-C(27)-H(27B)	109.5
C(20)-C(27)-H(27C)	109.5(3)
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(20)-C(28)-H(28A)	109.5(2)
C(20)-C(28)-H(28B)	109.5(2)
H(28A)-C(28)-H(28B)	109.5
C(20)-C(28)-H(28C)	109.5(3)
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(20)-C(29)-H(29A)	109.5(2)
C(20)-C(29)-H(29B)	109.5(3)
H(29A)-C(29)-H(29B)	109.5
C(20)-C(29)-H(29C)	109.5(2)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(32)-C(3)-C(46)	111.1(3)
C(32)-C(3)-H(3A)	109.4(2)
C(46)-C(3)-H(3A)	109.4(2)
C(32)-C(3)-H(3B)	109.4(2)
C(46)-C(3)-H(3B)	109.4(2)

H(3A)-C(3)-H(3B)	108.0
O(3)-C(31)-C(36)	118.7(3)
O(3)-C(31)-C(32)	120.5(3)
C(36)-C(31)-C(32)	120.8(3)
C(33)-C(32)-C(31)	118.8(3)
C(33)-C(32)-C(3)	120.1(3)
C(31)-C(32)-C(3)	121.1(3)
C(32)-C(33)-C(34)	122.4(3)
C(32)-C(33)-H(33A)	118.8(2)
C(34)-C(33)-H(33A)	118.8(2)
C(35)-C(34)-C(33)	117.2(3)
C(35)-C(34)-C(30)	122.0(3)
C(33)-C(34)-C(30)	120.8(3)
C(34)-C(35)-C(36)	122.6(3)
C(34)-C(35)-H(35A)	118.7(2)
C(36)-C(35)-H(35A)	118.7(2)
C(31)-C(36)-C(35)	118.1(3)
C(31)-C(36)-C(2)	121.3(3)
C(35)-C(36)-C(2)	120.6(3)
C(37)-C(30)-C(39)	109.5(6)
C(37)-C(30)-C(38)	108.1(5)
C(39)-C(30)-C(38)	106.6(5)
C(37)-C(30)-C(34)	109.5(4)
C(39)-C(30)-C(34)	110.6(4)
C(38)-C(30)-C(34)	112.4(3)
C(30)-C(37)-H(37A)	109.5(4)
C(30)-C(37)-H(37B)	109.5(3)
H(37A)-C(37)-H(37B)	109.5
C(30)-C(37)-H(37C)	109.5(4)
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
C(30)-C(38)-H(38A)	109.5(3)
C(30)-C(38)-H(38B)	109.5(3)
H(38A)-C(38)-H(38B)	109.5
C(30)-C(38)-H(38C)	109.5(2)
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5
C(30)-C(39)-H(39A)	109.5(4)
C(30)-C(39)-H(39B)	109.5(4)
H(39A)-C(39)-H(39B)	109.5
C(30)-C(39)-H(39C)	109.5(3)
H(39A)-C(39)-H(39C)	109.5
H(39B)-C(39)-H(39C)	109.5
C(16)-C(4)-C(42)	113.5(3)
C(16)-C(4)-H(4A)	108.9(2)
C(42)-C(4)-H(4A)	108.9(2)
C(16)-C(4)-H(4B)	108.9(2)
C(42)-C(4)-H(4B)	108.9(2)
H(4A)-C(4)-H(4B)	107.7
O(4)-C(41)-C(42)	121.4(3)
O(4)-C(41)-C(46)	118.7(3)
C(42)-C(41)-C(46)	119.9(3)
C(43)-C(42)-C(41)	119.4(3)
C(43)-C(42)-C(4)	119.6(3)
C(41)-C(42)-C(4)	121.0(3)
C(44)-C(43)-C(42)	122.4(3)
C(44)-C(43)-H(43A)	118.8(2)
C(42)-C(43)-H(43A)	118.8(2)
C(43)-C(44)-C(45)	116.8(3)
C(43)-C(44)-C(40)	123.1(3)
C(45)-C(44)-C(40)	120.0(3)
C(46)-C(45)-C(44)	123.0(3)
C(46)-C(45)-H(45A)	118.5(2)
C(44)-C(45)-H(45A)	118.5(2)
C(45)-C(46)-C(41)	118.5(3)
C(45)-C(46)-C(3)	120.0(3)
C(41)-C(46)-C(3)	121.4(3)
C(49)-C(40)-C(44)	112.3(3)
C(49)-C(40)-C(47)	107.7(3)
C(44)-C(40)-C(47)	108.9(3)
C(49)-C(40)-C(48)	109.2(3)
C(44)-C(40)-C(48)	108.9(3)
C(47)-C(40)-C(48)	109.9(3)
C(40)-C(47)-H(47A)	109.5(2)
C(40)-C(47)-H(47B)	109.5(2)
H(47A)-C(47)-H(47B)	109.5
C(40)-C(47)-H(47C)	109.5(2)
H(47A)-C(47)-H(47C)	109.5
H(47B)-C(47)-H(47C)	109.5
C(40)-C(48)-H(48A)	109.5(2)
C(40)-C(48)-H(48B)	109.5(2)
H(48A)-C(48)-H(48B)	109.5
C(40)-C(48)-H(48C)	109.5(2)
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(40)-C(49)-H(49A)	109.5(2)
C(40)-C(49)-H(49B)	109.5(2)
H(49A)-C(49)-H(49B)	109.5
C(40)-C(49)-H(49C)	109.5(2)
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
N(1)-P(1)-C(71)	109.9(2)
N(1)-P(1)-C(51)	109.1(2)
C(71)-P(1)-C(51)	109.1(2)
N(1)-P(1)-C(61)	110.4(2)
C(71)-P(1)-C(61)	112.2(2)
C(51)-P(1)-C(61)	106.1(2)
P(1)-N(1)-H(1A)	122(4)
P(1)-N(1)-H(1B)	114(3)
H(1A)-N(1)-H(1B)	122(5)
C(52)-C(51)-C(56)	119.6(3)
C(52)-C(51)-P(1)	122.3(3)
C(56)-C(51)-P(1)	118.0(3)
C(53)-C(52)-C(51)	120.1(4)
C(53)-C(52)-H(52A)	119.9(2)
C(51)-C(52)-H(52A)	119.9(2)
C(52)-C(53)-C(54)	119.6(4)
C(52)-C(53)-H(53A)	120.2(2)
C(54)-C(53)-H(53A)	120.2(2)
C(55)-C(54)-C(53)	120.6(4)
C(55)-C(54)-H(54A)	119.7(2)
C(53)-C(54)-H(54A)	119.7(2)
C(54)-C(55)-C(56)	120.2(4)
C(54)-C(55)-H(55A)	119.9(2)
C(56)-C(55)-H(55A)	119.9(2)
C(55)-C(56)-C(51)	119.7(4)
C(55)-C(56)-H(56A)	120.1(2)
C(51)-C(56)-H(56A)	120.1(2)
C(62)-C(61)-C(66)	120.1(4)
C(62)-C(61)-P(1)	119.4(3)
C(66)-C(61)-P(1)	120.4(3)
C(63)-C(62)-C(61)	119.8(4)
C(63)-C(62)-H(62A)	120.1(2)
C(61)-C(62)-H(62A)	120.1(2)
C(64)-C(63)-C(62)	120.0(4)
C(64)-C(63)-H(63A)	120.0(2)
C(62)-C(63)-H(63A)	120.0(2)
C(65)-C(64)-C(63)	120.3(4)
C(65)-C(64)-H(64A)	119.9(2)
C(63)-C(64)-H(64A)	119.9(2)
C(64)-C(65)-C(66)	120.6(4)
C(64)-C(65)-H(65A)	119.7(2)
C(66)-C(65)-H(65A)	119.7(2)
C(65)-C(66)-C(61)	119.2(4)
C(65)-C(66)-H(66A)	120.4(2)
C(61)-C(66)-H(66A)	120.4(2)
C(76)-C(71)-C(72)	119.2(4)
C(76)-C(71)-P(1)	124.1(3)
C(72)-C(71)-P(1)	116.8(3)
C(73)-C(72)-C(71)	120.6(4)
C(73)-C(72)-H(72A)	119.7(3)
C(71)-C(72)-H(72A)	119.7(2)
C(72)-C(73)-C(74)	119.7(4)
C(72)-C(73)-H(73A)	120.1(3)
C(74)-C(73)-H(73A)	120.1(2)
C(75)-C(74)-C(73)	120.0(4)
C(75)-C(74)-H(74A)	120.0(2)
C(73)-C(74)-H(74A)	120.0(2)

C(74)-C(75)-C(76)	120.6(4)	C(21X)-C(22X)-H(2AX)	109.5(2)
C(74)-C(75)-H(75A)	119.7(2)	C(21X)-C(22X)-H(2BX)	109.5(3)
C(76)-C(75)-H(75A)	119.7(2)	H(2AX)-C(22X)-H(2BX)	109.5
C(75)-C(76)-C(71)	119.9(4)	C(21X)-C(22X)-H(2CX)	109.5(3)
C(75)-C(76)-H(76A)	120.0(2)	H(2AX)-C(22X)-H(2CX)	109.5
C(71)-C(76)-H(76A)	120.0(2)	H(2BX)-C(22X)-H(2CX)	109.5
N(1X)-C(11X)-C(12X)	179.3(7)		
C(11X)-C(12X)-H(1AX)	109.5(3)		
C(11X)-C(12X)-H(1BX)	109.5(3)		
H(1AX)-C(12X)-H(1BX)	109.5		
C(11X)-C(12X)-H(1CX)	109.5(3)		
H(1AX)-C(12X)-H(1CX)	109.5		
H(1BX)-C(12X)-H(1CX)	109.5		
N(2X)-C(21X)-C(22X)	178.9(5)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 4. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	16(1)	25(1)	25(1)	1(1)	0(1)	0(1)
O(3)	18(1)	35(2)	27(2)	3(1)	-2(1)	1(1)
O(2)	23(1)	39(2)	23(2)	4(1)	-5(1)	-2(1)
O(4)	20(1)	25(1)	29(2)	1(1)	3(1)	-1(1)
C(1)	27(2)	22(2)	23(2)	-3(2)	-4(2)	4(2)
C(11)	18(2)	20(2)	22(2)	5(2)	-4(1)	3(2)
C(12)	25(2)	21(2)	21(2)	3(2)	-4(2)	5(2)
C(13)	28(2)	22(2)	25(2)	0(2)	-10(2)	2(2)
C(14)	17(2)	20(2)	35(2)	9(2)	-5(2)	1(2)
C(15)	21(2)	20(2)	27(2)	5(2)	0(2)	5(2)
C(16)	20(2)	17(2)	22(2)	3(2)	-6(2)	3(2)
C(10)	22(2)	25(2)	46(3)	5(2)	-1(2)	-3(2)
C(17)	30(3)	64(3)	76(4)	31(3)	-28(2)	-13(2)
C(18)	22(2)	39(3)	81(4)	-5(2)	16(2)	-9(2)
C(19)	32(2)	28(2)	50(3)	4(2)	-2(2)	-7(2)
C(2)	24(2)	30(2)	18(2)	0(2)	4(2)	3(2)
C(21)	22(2)	29(2)	17(2)	1(2)	2(2)	6(2)
C(22)	23(2)	26(2)	19(2)	-3(2)	3(2)	3(2)
C(23)	31(2)	25(2)	19(2)	0(2)	3(2)	6(2)
C(24)	29(2)	27(2)	20(2)	-4(2)	-2(2)	8(2)
C(25)	31(2)	22(2)	23(2)	-4(2)	2(2)	2(2)
C(26)	25(2)	24(2)	17(2)	-3(2)	1(2)	7(2)
C(20)	33(2)	37(2)	27(2)	-2(2)	-8(2)	0(2)
C(27)	38(3)	57(3)	77(4)	-9(3)	-24(3)	13(3)
C(28)	43(3)	54(3)	43(3)	8(2)	-20(2)	-16(2)
C(29)	58(3)	61(3)	28(2)	0(2)	-16(2)	-11(3)
C(3)	26(2)	21(2)	21(2)	0(2)	-1(2)	-3(2)
C(31)	13(2)	25(2)	26(2)	4(2)	0(1)	-6(2)
C(32)	17(2)	22(2)	19(2)	2(2)	0(1)	-5(2)
C(33)	20(2)	22(2)	25(2)	-1(2)	3(2)	-1(2)
C(34)	21(2)	24(2)	26(2)	2(2)	-4(2)	-2(2)
C(35)	25(2)	25(2)	17(2)	2(2)	-2(2)	1(2)
C(36)	21(2)	22(2)	24(2)	3(2)	5(2)	-3(2)
C(30)	26(2)	32(2)	27(2)	0(2)	-4(2)	6(2)
C(37)	22(3)	152(7)	147(7)	93(6)	4(3)	18(4)
C(38)	67(4)	115(5)	42(3)	-2(3)	-15(3)	54(4)
C(39)	138(7)	59(4)	143(7)	-39(4)	-105(6)	55(4)
C(4)	18(2)	26(2)	17(2)	3(2)	-2(1)	2(2)
C(41)	15(2)	25(2)	17(2)	-1(2)	-4(1)	-1(2)
C(42)	20(2)	21(2)	16(2)	2(1)	-8(1)	-2(2)
C(43)	18(2)	25(2)	18(2)	1(2)	-2(1)	-4(2)
C(44)	17(2)	24(2)	18(2)	-2(2)	-4(1)	0(2)
C(45)	21(2)	20(2)	20(2)	-2(2)	-7(2)	-1(2)
C(46)	20(2)	20(2)	15(2)	-2(1)	-4(1)	-4(2)
C(40)	22(2)	26(2)	29(2)	-2(2)	-2(2)	0(2)
C(47)	31(2)	35(2)	32(2)	-9(2)	0(2)	3(2)
C(48)	25(2)	39(2)	37(2)	0(2)	-6(2)	4(2)
C(49)	32(2)	40(2)	39(3)	4(2)	13(2)	4(2)

P(1)	17(1)	22(1)	24(1)	-1(1)	-6(1)	2(1)
N(1)	26(2)	25(2)	49(2)	-8(2)	-15(2)	6(2)
C(51)	18(2)	20(2)	28(2)	-3(2)	-9(2)	1(2)
C(52)	23(2)	28(2)	27(2)	1(2)	-2(2)	-1(2)
C(53)	29(2)	29(2)	31(2)	6(2)	-8(2)	0(2)
C(54)	24(2)	24(2)	36(2)	-2(2)	-13(2)	6(2)
C(55)	19(2)	30(2)	33(2)	-5(2)	-5(2)	6(2)
C(56)	26(2)	29(2)	22(2)	-1(2)	-4(2)	2(2)
C(61)	16(2)	24(2)	27(2)	-3(2)	-5(2)	5(2)
C(62)	25(2)	34(2)	25(2)	-2(2)	-7(2)	1(2)
C(63)	40(2)	40(2)	21(2)	4(2)	-2(2)	4(2)
C(64)	36(2)	31(2)	30(2)	3(2)	10(2)	-2(2)
C(65)	23(2)	36(2)	37(2)	1(2)	0(2)	-6(2)
C(66)	23(2)	32(2)	22(2)	1(2)	-3(2)	2(2)
C(71)	17(2)	24(2)	28(2)	0(2)	-6(2)	1(2)
C(72)	25(2)	26(2)	38(2)	4(2)	-3(2)	3(2)
C(73)	28(2)	36(2)	45(3)	13(2)	5(2)	1(2)
C(74)	29(2)	48(3)	28(2)	4(2)	3(2)	9(2)
C(75)	26(2)	37(2)	31(2)	-7(2)	-3(2)	4(2)
C(76)	19(2)	31(2)	26(2)	0(2)	-6(2)	-3(2)
N(1X)	51(4)	89(5)	215(8)	3(5)	7(4)	-9(3)
C(11X)	54(4)	44(3)	79(4)	4(3)	1(3)	-2(3)
C(12X)	37(3)	59(3)	54(3)	12(3)	-10(2)	3(3)
N(2X)	77(3)	36(2)	72(3)	-9(2)	-13(3)	1(2)
C(21X)	35(2)	33(2)	47(3)	10(2)	-5(2)	-5(2)
C(22X)	44(3)	47(3)	42(3)	-2(2)	14(2)	-18(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 4.

	x	y	z	U(eq)
H(3)	5922(28)	-191(41)	1584(21)	59(16)
H(2)	6133(27)	2051(38)	1818(20)	54(15)
H(4)	6186(32)	1104(49)	1008(22)	81(20)
H(1C)	6320(2)	3813(3)	1915(1)	29
H(1D)	7005(2)	4534(3)	2093(1)	29
H(13)	8133(2)	4482(3)	1621(2)	30
H(15)	8328(2)	2774(3)	302(2)	27
H(17A)	9393(10)	4193(25)	1669(3)	86
H(17B)	10104(7)	4277(24)	1309(7)	86
H(17C)	9688(16)	3164(5)	1371(8)	86
H(18A)	9203(9)	3984(23)	78(2)	71
H(18B)	9566(16)	3030(5)	404(6)	71
H(18C)	9992(8)	4137(20)	352(7)	71
H(19A)	8749(13)	5603(3)	535(7)	55
H(19B)	9550(3)	5735(4)	794(11)	55
H(19C)	8837(15)	5701(4)	1155(5)	55
H(2A)	5893(2)	403(3)	2776(1)	29
H(2B)	6400(2)	67(3)	3263(1)	29
H(23A)	7559(2)	1052(3)	3432(1)	30
H(25A)	7955(2)	3793(3)	2675(1)	30
H(27A)	8858(8)	999(6)	3480(14)	87
H(27B)	9548(9)	1765(15)	3583(11)	87
H(27C)	9227(16)	1615(18)	3004(3)	87
H(28A)	9158(16)	3613(11)	2971(4)	70
H(28B)	9408(11)	3713(13)	3569(8)	70
H(28C)	8651(6)	4240(4)	3375(12)	70
H(29A)	8107(17)	1959(11)	4123(4)	73
H(29B)	7986(14)	3215(17)	4078(3)	73
H(29C)	8772(4)	2754(27)	4252(2)	73
H(3A)	6069(2)	-1607(3)	1165(1)	27
H(3B)	6678(2)	-2520(3)	1112(1)	27
H(33A)	7735(2)	-2548(3)	1731(1)	27
H(35A)	7571(2)	-967(3)	3090(1)	27
H(37A)	9103(16)	-2007(42)	2054(3)	160
H(37B)	9559(3)	-2339(31)	2565(19)	160
H(37C)	9148(17)	-1216(15)	2541(19)	160

H(38A)	8052(10)	-2705(32)	3402(2)	112
H(38B)	8580(24)	-1692(7)	3363(2)	112
H(38C)	8924(15)	-2861(29)	3364(2)	112
H(39A)	8477(35)	-3697(6)	2128(5)	171
H(39B)	7933(14)	-3926(13)	2602(22)	171
H(39C)	8805(23)	-4014(11)	2688(20)	171
H(4A)	7224(2)	1959(3)	-78(1)	25
H(4B)	6499(2)	1829(3)	263(1)	25
H(43A)	8163(2)	610(3)	-62(1)	24
H(45A)	7768(2)	-2205(3)	624(1)	24
H(47A)	9001(3)	-2682(12)	-463(9)	49
H(47B)	8199(11)	-2199(5)	-571(7)	49
H(47C)	8352(14)	-2893(9)	-58(2)	49
H(48A)	9472(11)	-991(4)	632(7)	50
H(48B)	9772(7)	-1967(20)	296(2)	50
H(48C)	9101(6)	-2137(17)	686(6)	50
H(49A)	9319(14)	35(11)	-200(3)	55
H(49B)	8802(5)	-408(18)	-660(6)	55
H(49C)	9579(10)	-960(9)	-539(9)	55
H(1A)	4967(28)	4128(43)	1053(19)	55(16)
H(1B)	5456(25)	3170(35)	1059(16)	35(12)
H(52A)	4004(2)	3973(3)	-329(2)	31
H(53A)	3008(2)	5089(3)	-519(2)	35
H(54A)	2132(2)	5438(3)	123(2)	34
H(55A)	2222(2)	4634(3)	937(2)	33
H(56A)	3226(2)	3541(3)	1138(2)	31
H(62A)	4851(2)	1700(3)	1535(2)	34
H(63A)	4295(2)	326(3)	2003(2)	41
H(64A)	3214(2)	-459(3)	1687(2)	39
H(65A)	2719(2)	59(3)	886(2)	38
H(66A)	3283(2)	1397(3)	399(2)	31
H(72A)	5510(2)	3915(3)	-13(2)	35
H(73A)	6155(2)	3569(3)	-774(2)	43
H(74A)	6010(2)	1928(4)	-1198(2)	42
H(75A)	5252(2)	627(3)	-842(2)	38
H(76A)	4591(2)	969(3)	-83(2)	30
H(1AX)	8096(3)	521(39)	2059(9)	131(29)
H(1BX)	8069(3)	390(34)	1439(12)	185(42)
H(1CX)	8054(3)	1550(6)	1693(21)	140(31)
H(2AX)	4995(12)	-3146(13)	2355(7)	56(15)
H(2BX)	5267(17)	-2252(6)	1957(2)	82(20)
H(2CX)	5855(6)	-2904(18)	2299(8)	62(16)

5) *p-tert*-butylcalix[4]arene triphenylethylphosphonium

Table 1. Crystal data and structure refinement for 5.

Identification code	5 (98srv036)
Empirical formula	C68 H81 N2 O4 P
Formula weight	1021.32
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 12.8544(2) A alpha = 90 deg. b = 21.84100(10) A beta = 90.2100(10) deg. c = 21.02710(10) A gamma = 90 deg.
Volume	5903.38(10) A^3
Z, Calculated density	4, 1.149 Mg/m^3
Absorption coefficient	0.096 mm^-1
F(000)	2200
Crystal size	0.5 x 0.5 x 0.3 mm
Theta range for data collection	1.34 to 27.49 deg.
Limiting indices	-16<=h<=15, -28<=k<=28, -27<=l<=27
Reflections collected / unique	58007 / 13525 [R(int) = 0.0926]
Completeness to theta = 27.49	99.8 %
Absorption correction	Multiscan
Max. and min. transmission	0.72614 and 1.00000
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13525 / 6 / 743
Goodness-of-fit on F^2	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0767, wR2 = 0.1776
R indices (all data)	R1 = 0.1397, wR2 = 0.2154
Extinction coefficient	not refined
Largest diff. peak and hole	1.029 and -0.455 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 5.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	4908(2)	-1390(1)	472(1)	29(1)
C(11)	5510(2)	-1393(1)	1006(1)	26(1)
C(12)	5130(3)	-1137(1)	1578(1)	26(1)
C(13)	5716(3)	-1207(1)	2133(2)	29(1)
C(14)	6669(3)	-1514(2)	2156(2)	31(1)
C(15)	7056(3)	-1721(2)	1574(2)	33(1)
C(16)	6501(3)	-1661(2)	1003(2)	30(1)
C(10)	7221(3)	-1635(2)	2795(2)	40(1)
C(17)	6505(5)	-2032(3)	3205(2)	53(2)
C(18)	8241(4)	-2003(3)	2700(3)	56(2)
C(19)	7478(5)	-1037(3)	3116(3)	57(2)
C(17D)	8239(19)	-1259(16)	2813(12)	86(11)
C(18D)	7340(40)	-2308(9)	2925(13)	110(17)
C(19D)	6621(18)	-1349(13)	3382(8)	68(9)
C(1)	4059(3)	-847(1)	1605(2)	27(1)
O(2)	2908(2)	-1464(1)	618(1)	32(1)
C(21)	2717(2)	-1626(1)	1239(1)	24(1)
C(22)	2009(2)	-2095(1)	1366(1)	26(1)
C(23)	1820(3)	-2251(1)	2001(2)	27(1)
C(24)	2326(2)	-1967(1)	2511(1)	25(1)
C(25)	3028(2)	-1502(1)	2365(1)	25(1)
C(26)	3232(2)	-1324(1)	1737(1)	24(1)
C(20)	2099(3)	-2167(2)	3198(2)	34(1)
C(27)	2743(3)	-1825(2)	3686(2)	48(1)
C(28)	2278(5)	-2849(2)	3268(2)	70(2)
C(29)	936(4)	-2021(3)	3334(2)	64(1)
C(2)	1492(3)	-2460(2)	834(2)	29(1)
O(3)	2974(2)	-2587(1)	-173(1)	36(1)
C(31)	2856(3)	-3076(1)	235(1)	29(1)
C(32)	3486(3)	-3593(2)	177(1)	31(1)
C(33)	3321(3)	-4090(2)	588(2)	32(1)
C(34)	2541(3)	-4087(1)	1048(1)	28(1)
C(35)	1940(3)	-3558(1)	1094(1)	29(1)
C(36)	2086(3)	-3047(1)	707(1)	27(1)
C(30)	2349(3)	-4637(2)	1492(2)	33(1)
C(37)	3233(3)	-5108(2)	1458(2)	48(1)
C(38)	2274(4)	-4417(2)	2187(2)	61(1)
C(39)	1333(3)	-4949(2)	1304(2)	51(1)
C(3)	4389(3)	-3607(2)	-287(2)	34(1)
O(4)	4999(2)	-2348(1)	-169(1)	33(1)
C(41)	5662(3)	-2780(2)	62(1)	29(1)
C(42)	6604(3)	-2614(2)	343(2)	33(1)
C(43)	7236(3)	-3066(2)	615(2)	44(1)
C(44)	6953(4)	-3685(2)	615(2)	52(1)
C(45)	6054(3)	-3839(2)	293(2)	45(1)
C(46)	5394(3)	-3408(2)	16(2)	34(1)
C(40)	7581(5)	-4175(2)	973(3)	76(2)
C(47)	6905(6)	-4429(4)	1544(4)	149(4)
C(48)	7875(6)	-4694(3)	568(3)	105(2)
C(49)	8512(5)	-3912(3)	1332(2)	81(2)
C(4)	6930(3)	-1946(2)	402(2)	33(1)
P(1)	8091(1)	393(1)	638(1)	23(1)
C(51)	8873(2)	-129(1)	1097(1)	26(1)
C(52)	9329(3)	75(2)	1666(2)	32(1)
C(53)	10003(3)	-304(2)	1996(2)	40(1)
C(54)	10232(3)	-888(2)	1769(2)	40(1)
C(55)	9754(3)	-1098(2)	1220(2)	35(1)
C(56)	9079(3)	-723(1)	880(2)	29(1)
C(61)	7560(2)	-5(1)	-37(1)	24(1)
C(62)	8189(3)	-99(2)	-573(2)	34(1)
C(63)	7796(3)	-433(2)	-1083(2)	36(1)
C(64)	6802(3)	-661(2)	-1068(2)	32(1)
C(65)	6176(3)	-570(1)	-539(2)	30(1)

C(66)	6561(3)	-242(1)	-21(1)	27(1)
C(71)	7068(2)	678(1)	1131(1)	24(1)
C(72)	6663(3)	312(2)	1619(2)	30(1)
C(73)	5881(3)	541(2)	2003(2)	36(1)
C(74)	5509(3)	1129(2)	1909(2)	36(1)
C(75)	5891(3)	1489(2)	1412(2)	37(1)
C(76)	6673(3)	1265(2)	1025(2)	31(1)
C(81)	8883(3)	1018(2)	355(2)	30(1)
C(82)	9389(3)	1427(2)	853(2)	44(1)
N(1X)	140(5)	1294(2)	2485(2)	110(2)
C(11X)	221(4)	1673(2)	2854(2)	56(1)
C(12X)	338(3)	2153(2)	3314(2)	43(1)
N(2X)	9614(4)	-2824(3)	-126(3)	99(2)
C(21X)	10026(5)	-3298(3)	-227(3)	82(2)
C(22X)	10539(6)	-3877(3)	-353(3)	98(2)

Table 3. Bond lengths [A] and angles [deg] for 5.

O(1)-C(11)	1.360(4)
O(1)-H(4)	1.33(5)
C(11)-C(16)	1.402(5)
C(11)-C(12)	1.416(4)
C(12)-C(13)	1.395(4)
C(12)-C(1)	1.517(4)
C(13)-C(14)	1.397(5)
C(13)-H(14)	0.9500
C(14)-C(15)	1.398(5)
C(14)-C(10)	1.541(5)
C(15)-C(16)	1.401(5)
C(15)-H(15)	0.9500
C(16)-C(4)	1.515(4)
C(10)-C(18D)	1.502(18)
C(10)-C(19)	1.506(6)
C(10)-C(17)	1.532(6)
C(10)-C(17D)	1.545(18)
C(10)-C(18)	1.553(6)
C(10)-C(19D)	1.585(17)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800
C(17D)-H(17D)	0.9800
C(17D)-H(17E)	0.9800
C(17D)-H(17F)	0.9800
C(18D)-H(18D)	0.9800
C(18D)-H(18E)	0.9800
C(18D)-H(18F)	0.9800
C(19D)-H(19D)	0.9800
C(19D)-H(19E)	0.9800
C(19D)-H(19F)	0.9800
C(1)-C(26)	1.514(4)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
O(2)-C(21)	1.376(4)
O(2)-H(2)	0.91(5)
C(21)-C(22)	1.397(4)
C(21)-C(26)	1.403(4)
C(22)-C(23)	1.399(4)
C(22)-C(2)	1.525(4)
C(23)-C(24)	1.398(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.393(4)
C(24)-C(20)	1.539(4)
C(25)-C(26)	1.402(4)
C(25)-H(25)	0.9500
C(20)-C(28)	1.513(5)
C(20)-C(27)	1.513(5)
C(20)-C(29)	1.556(6)
C(27)-H(27A)	0.9800
C(27)-H(27B)	0.9800
C(27)-H(27C)	0.9800
C(28)-H(28A)	0.9800
C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800
C(29)-H(29A)	0.9800
C(29)-H(29B)	0.9800
C(29)-H(29C)	0.9800
C(2)-C(36)	1.516(4)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
O(3)-C(31)	1.379(4)
O(3)-H(3)	1.03(5)
C(31)-C(32)	1.395(5)
C(31)-C(36)	1.406(4)
C(32)-C(33)	1.403(4)
C(32)-C(3)	1.518(5)
C(33)-C(34)	1.397(5)
C(33)-H(33)	0.9500
C(34)-C(35)	1.395(5)
C(34)-C(30)	1.541(4)
C(35)-C(36)	1.395(4)
C(35)-H(35)	0.9500
C(30)-C(39)	1.525(5)
C(30)-C(37)	1.533(5)
C(30)-C(38)	1.541(5)
C(37)-H(37A)	0.9800
C(37)-H(37B)	0.9800
C(37)-H(37C)	0.9800
C(38)-H(38A)	0.9800
C(38)-H(38B)	0.9800
C(38)-H(38C)	0.9800
C(39)-H(39A)	0.9800
C(39)-H(39B)	0.9800
C(39)-H(39C)	0.9800
C(3)-C(46)	1.503(5)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
O(4)-C(41)	1.359(4)
O(4)-H(4)	1.18(5)
C(41)-C(42)	1.393(5)
C(41)-C(46)	1.418(5)
C(42)-C(43)	1.400(5)
C(42)-C(4)	1.522(5)
C(43)-C(44)	1.400(6)
C(43)-H(43)	0.9500
C(44)-C(45)	1.379(6)
C(44)-C(40)	1.536(6)

C(45)-C(46)	1.394(5)
C(45)-H(45)	0.9500
C(40)-C(48)	1.467(7)
C(40)-C(49)	1.524(8)
C(40)-C(47)	1.585(8)
C(47)-H(47A)	0.9800
C(47)-H(47B)	0.9800
C(47)-H(47C)	0.9800
C(48)-H(48A)	0.9800
C(48)-H(48B)	0.9800
C(48)-H(48C)	0.9800
C(49)-H(49A)	0.9800
C(49)-H(49B)	0.9800
C(49)-H(49C)	0.9800
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
P(1)-C(71)	1.789(3)
P(1)-C(51)	1.796(3)
P(1)-C(61)	1.797(3)
P(1)-C(81)	1.805(3)
C(51)-C(56)	1.401(4)
C(51)-C(52)	1.403(4)
C(52)-C(53)	1.384(5)
C(52)-H(52)	0.9500
C(53)-C(54)	1.394(5)
C(53)-H(53)	0.9500
C(54)-C(55)	1.384(5)
C(54)-H(54)	0.9500
C(55)-C(56)	1.390(5)
C(55)-H(55)	0.9500
C(56)-H(56)	0.9500
C(61)-C(66)	1.385(4)
C(61)-C(62)	1.404(4)
C(62)-C(63)	1.391(5)
C(62)-H(62)	0.9500
C(63)-C(64)	1.372(5)
C(63)-H(63)	0.9500
C(64)-C(65)	1.389(5)
C(64)-H(64)	0.9500
C(65)-C(66)	1.393(4)
C(65)-H(65)	0.9500
C(66)-H(66)	0.9500
C(71)-C(76)	1.397(4)
C(71)-C(72)	1.402(4)
C(72)-C(73)	1.386(5)
C(72)-H(72)	0.9500
C(73)-C(74)	1.384(5)
C(73)-H(73)	0.9500
C(74)-C(75)	1.397(5)
C(74)-H(74)	0.9500
C(75)-C(76)	1.386(5)
C(75)-H(75)	0.9500
C(76)-H(76)	0.9500
C(81)-C(82)	1.521(5)
C(81)-H(81A)	0.95(4)
C(81)-H(81B)	0.90(4)
C(82)-H(82C)	0.9800
C(82)-H(82B)	0.9800
C(82)-H(82A)	0.9800
N(1X)-C(11X)	1.139(5)
C(11X)-C(12X)	1.434(5)
C(12X)-H(1AX)	0.9800
C(12X)-H(1BX)	0.9800
C(12X)-H(1CX)	0.9800
N(2X)-C(21X)	1.184(8)
C(21X)-C(22X)	1.452(9)
C(22X)-H(2AX)	0.9800
C(22X)-H(2BX)	0.9800
C(22X)-H(2CX)	0.9800

C(11)-O(1)-H(4)	110(2)
O(1)-C(11)-C(16)	120.8(3)
O(1)-C(11)-C(12)	120.2(3)
C(16)-C(11)-C(12)	119.0(3)
C(13)-C(12)-C(11)	118.8(3)
C(13)-C(12)-C(1)	120.1(3)
C(11)-C(12)-C(1)	120.8(3)
C(12)-C(13)-C(14)	123.4(3)
C(12)-C(13)-H(14)	118.3
C(14)-C(13)-H(14)	118.3
C(13)-C(14)-C(15)	116.2(3)
C(13)-C(14)-C(10)	120.8(3)
C(15)-C(14)-C(10)	122.9(3)
C(14)-C(15)-C(16)	122.5(3)
C(14)-C(15)-H(15)	118.8
C(16)-C(15)-H(15)	118.8
C(15)-C(16)-C(11)	119.8(3)
C(15)-C(16)-C(4)	119.4(3)
C(11)-C(16)-C(4)	120.6(3)
C(18D)-C(10)-C(19)	138.2(11)
C(18D)-C(10)-C(17)	53.4(18)
C(19)-C(10)-C(17)	111.8(4)
C(18D)-C(10)-C(14)	111.8(11)
C(19)-C(10)-C(14)	110.0(3)
C(17)-C(10)-C(14)	108.2(3)
C(18D)-C(10)-C(17D)	115(2)
C(19)-C(10)-C(17D)	49.0(13)
C(17)-C(10)-C(17D)	142.9(9)
C(14)-C(10)-C(17D)	108.5(9)
C(18D)-C(10)-C(18)	55.3(18)
C(19)-C(10)-C(18)	108.9(4)
C(17)-C(10)-C(18)	106.7(4)
C(14)-C(10)-C(18)	111.3(3)
C(17D)-C(10)-C(18)	64.0(14)
C(18D)-C(10)-C(19D)	107.1(18)
C(19)-C(10)-C(19D)	54.3(11)
C(17)-C(10)-C(19D)	59.4(11)
C(14)-C(10)-C(19D)	112.8(8)
C(17D)-C(10)-C(19D)	100.8(16)
C(18)-C(10)-C(19D)	135.9(8)
C(10)-C(17)-H(17A)	109.5
C(10)-C(17)-H(17B)	109.5
C(10)-C(17)-H(17C)	109.5
C(10)-C(18)-H(18A)	109.5
C(10)-C(18)-H(18B)	109.5
C(10)-C(18)-H(18C)	109.5
C(10)-C(19)-H(19A)	109.5
C(10)-C(19)-H(19B)	109.5
C(10)-C(19)-H(19C)	109.5
C(10)-C(17D)-H(17D)	109.5
C(10)-C(17D)-H(17E)	109.5
H(17D)-C(17D)-H(17E)	109.5
C(10)-C(17D)-H(17F)	109.5
H(17D)-C(17D)-H(17F)	109.5
H(17E)-C(17D)-H(17F)	109.5
C(10)-C(18D)-H(18D)	109.5
C(10)-C(18D)-H(18E)	109.5
H(18D)-C(18D)-H(18E)	109.5
C(10)-C(18D)-H(18F)	109.5
H(18D)-C(18D)-H(18F)	109.5
H(18E)-C(18D)-H(18F)	109.5
C(10)-C(19D)-H(19D)	109.5
C(10)-C(19D)-H(19E)	109.5
H(19D)-C(19D)-H(19E)	109.5
C(10)-C(19D)-H(19F)	109.5
H(19D)-C(19D)-H(19F)	109.5
H(19E)-C(19D)-H(19F)	109.5
C(26)-C(1)-C(12)	111.0(2)
C(26)-C(1)-H(1A)	109.4
C(12)-C(1)-H(1A)	109.4
C(26)-C(1)-H(1B)	109.4
C(12)-C(1)-H(1B)	109.4
H(1A)-C(1)-H(1B)	108.0
C(21)-O(2)-H(2)	108(3)
O(2)-C(21)-C(22)	119.2(3)
O(2)-C(21)-C(26)	120.2(3)
C(22)-C(21)-C(26)	120.6(3)
C(21)-C(22)-C(23)	118.5(3)

C(21)-C(22)-C(2)	121.7(3)
C(23)-C(22)-C(2)	119.7(3)
C(24)-C(23)-C(22)	122.8(3)
C(24)-C(23)-H(23)	118.6
C(22)-C(23)-H(23)	118.6
C(25)-C(24)-C(23)	117.0(3)
C(25)-C(24)-C(20)	122.6(3)
C(23)-C(24)-C(20)	120.3(3)
C(24)-C(25)-C(26)	122.3(3)
C(24)-C(25)-H(25)	118.9
C(26)-C(25)-H(25)	118.9
C(25)-C(26)-C(21)	118.8(3)
C(25)-C(26)-C(1)	119.9(3)
C(21)-C(26)-C(1)	121.1(3)
C(28)-C(20)-C(27)	109.7(3)
C(28)-C(20)-C(24)	110.0(3)
C(27)-C(20)-C(24)	113.0(3)
C(28)-C(20)-C(29)	109.3(4)
C(27)-C(20)-C(29)	107.3(3)
C(24)-C(20)-C(29)	107.5(3)
C(20)-C(27)-H(27A)	109.5
C(20)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(20)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(20)-C(28)-H(28A)	109.5
C(20)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(20)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(20)-C(29)-H(29A)	109.5
C(20)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
C(20)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(36)-C(2)-C(22)	110.7(3)
C(36)-C(2)-H(2A)	109.5
C(22)-C(2)-H(2A)	109.5
C(36)-C(2)-H(2B)	109.5
C(22)-C(2)-H(2B)	109.5
H(2A)-C(2)-H(2B)	108.1
C(31)-O(3)-H(3)	101(3)
O(3)-C(31)-C(32)	120.6(3)
O(3)-C(31)-C(36)	118.8(3)
C(32)-C(31)-C(36)	120.6(3)
C(31)-C(32)-C(33)	118.8(3)
C(31)-C(32)-C(3)	121.2(3)
C(33)-C(32)-C(3)	119.8(3)
C(34)-C(33)-C(32)	122.4(3)
C(34)-C(33)-H(33)	118.8
C(32)-C(33)-H(33)	118.8
C(35)-C(34)-C(33)	116.7(3)
C(35)-C(34)-C(30)	121.0(3)
C(33)-C(34)-C(30)	122.3(3)
C(36)-C(35)-C(34)	123.2(3)
C(36)-C(35)-H(35)	118.4
C(34)-C(35)-H(35)	118.4
C(35)-C(36)-C(31)	118.2(3)
C(35)-C(36)-C(2)	120.3(3)
C(31)-C(36)-C(2)	121.3(3)
C(39)-C(30)-C(37)	108.8(3)
C(39)-C(30)-C(38)	109.3(3)
C(37)-C(30)-C(38)	107.6(3)
C(39)-C(30)-C(34)	109.2(3)
C(37)-C(30)-C(34)	111.9(3)
C(38)-C(30)-C(34)	110.0(3)
C(30)-C(37)-H(37A)	109.5
C(30)-C(37)-H(37B)	109.5
H(37A)-C(37)-H(37B)	109.5
C(30)-C(37)-H(37C)	109.5
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
C(30)-C(38)-H(38A)	109.5
C(30)-C(38)-H(38B)	109.5
H(38A)-C(38)-H(38B)	109.5
C(30)-C(38)-H(38C)	109.5
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5
C(30)-C(39)-H(39A)	109.5
C(30)-C(39)-H(39B)	109.5
H(39A)-C(39)-H(39B)	109.5
C(30)-C(39)-H(39C)	109.5
H(39A)-C(39)-H(39C)	109.5
H(39B)-C(39)-H(39C)	109.5
C(46)-C(3)-C(32)	112.3(3)
C(46)-C(3)-H(3A)	109.1
C(32)-C(3)-H(3A)	109.1
C(46)-C(3)-H(3B)	109.1
C(32)-C(3)-H(3B)	109.1
H(3A)-C(3)-H(3B)	107.9
C(41)-O(4)-H(4)	110(2)
O(4)-C(41)-C(42)	120.9(3)
O(4)-C(41)-C(46)	119.7(3)
C(42)-C(41)-C(46)	119.4(3)
C(41)-C(42)-C(43)	119.5(3)
C(41)-C(42)-C(4)	121.5(3)
C(43)-C(42)-C(4)	118.9(3)
C(44)-C(43)-C(42)	122.1(4)
C(44)-C(43)-H(43)	119.0
C(42)-C(43)-H(43)	119.0
C(45)-C(44)-C(43)	117.0(4)
C(45)-C(44)-C(40)	120.5(4)
C(43)-C(44)-C(40)	122.5(4)
C(44)-C(45)-C(46)	123.1(4)
C(44)-C(45)-H(45)	118.4
C(46)-C(45)-H(45)	118.4
C(45)-C(46)-C(41)	118.6(3)
C(45)-C(46)-C(3)	120.1(3)
C(41)-C(46)-C(3)	121.2(3)
C(48)-C(40)-C(49)	112.0(5)
C(48)-C(40)-C(44)	112.9(4)
C(49)-C(40)-C(44)	113.0(4)
C(48)-C(40)-C(47)	108.2(6)
C(49)-C(40)-C(47)	100.8(5)
C(44)-C(40)-C(47)	109.1(5)
C(40)-C(47)-H(47A)	109.5
C(40)-C(47)-H(47B)	109.5
H(47A)-C(47)-H(47B)	109.5
C(40)-C(47)-H(47C)	109.5
H(47A)-C(47)-H(47C)	109.5
H(47B)-C(47)-H(47C)	109.5
C(40)-C(48)-H(48A)	109.5
C(40)-C(48)-H(48B)	109.5
H(48A)-C(48)-H(48B)	109.5
C(40)-C(48)-H(48C)	109.5
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(40)-C(49)-H(49A)	109.5
C(40)-C(49)-H(49B)	109.5
H(49A)-C(49)-H(49B)	109.5
C(40)-C(49)-H(49C)	109.5
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
C(16)-C(4)-C(42)	111.1(3)
C(16)-C(4)-H(4A)	109.4
C(42)-C(4)-H(4A)	109.4
C(16)-C(4)-H(4B)	109.4
C(42)-C(4)-H(4B)	109.4
H(4A)-C(4)-H(4B)	108.0
C(71)-P(1)-C(51)	108.72(14)
C(71)-P(1)-C(61)	110.36(14)
C(51)-P(1)-C(61)	109.09(14)
C(71)-P(1)-C(81)	110.12(16)
C(51)-P(1)-C(81)	110.00(16)
C(61)-P(1)-C(81)	108.54(15)

C(56)-C(51)-C(52)	119.5(3)
C(56)-C(51)-P(1)	121.3(2)
C(52)-C(51)-P(1)	119.1(2)
C(53)-C(52)-C(51)	119.8(3)
C(53)-C(52)-H(52)	120.1
C(51)-C(52)-H(52)	120.1
C(52)-C(53)-C(54)	120.6(3)
C(52)-C(53)-H(53)	119.7
C(54)-C(53)-H(53)	119.7
C(55)-C(54)-C(53)	119.7(3)
C(55)-C(54)-H(54)	120.2
C(53)-C(54)-H(54)	120.2
C(54)-C(55)-C(56)	120.6(3)
C(54)-C(55)-H(55)	119.7
C(56)-C(55)-H(55)	119.7
C(55)-C(56)-C(51)	119.8(3)
C(55)-C(56)-H(56)	120.1
C(51)-C(56)-H(56)	120.1
C(66)-C(61)-C(62)	120.1(3)
C(66)-C(61)-P(1)	120.7(2)
C(62)-C(61)-P(1)	119.1(2)
C(63)-C(62)-C(61)	119.1(3)
C(63)-C(62)-H(62)	120.4
C(61)-C(62)-H(62)	120.4
C(64)-C(63)-C(62)	120.5(3)
C(64)-C(63)-H(63)	119.8
C(62)-C(63)-H(63)	119.8
C(63)-C(64)-C(65)	120.6(3)
C(63)-C(64)-H(64)	119.7
C(65)-C(64)-H(64)	119.7
C(64)-C(65)-C(66)	119.6(3)
C(64)-C(65)-H(65)	120.2
C(66)-C(65)-H(65)	120.2
C(61)-C(66)-C(65)	120.0(3)
C(61)-C(66)-H(66)	120.0
C(65)-C(66)-H(66)	120.0
C(76)-C(71)-C(72)	120.3(3)
C(76)-C(71)-P(1)	119.7(2)
C(72)-C(71)-P(1)	120.1(2)
C(73)-C(72)-C(71)	119.5(3)
C(73)-C(72)-H(72)	120.3
C(71)-C(72)-H(72)	120.3
C(74)-C(73)-C(72)	120.2(3)
C(74)-C(73)-H(73)	119.9
C(72)-C(73)-H(73)	119.9
C(73)-C(74)-C(75)	120.4(3)
C(73)-C(74)-H(74)	119.8
C(75)-C(74)-H(74)	119.8
C(76)-C(75)-C(74)	119.8(3)
C(76)-C(75)-H(75)	120.1
C(74)-C(75)-H(75)	120.1
C(75)-C(76)-C(71)	119.7(3)
C(75)-C(76)-H(76)	120.2
C(71)-C(76)-H(76)	120.2
C(82)-C(81)-P(1)	117.2(2)
C(82)-C(81)-H(81A)	109(2)
P(1)-C(81)-H(81A)	104(2)
C(82)-C(81)-H(81B)	113(2)
P(1)-C(81)-H(81B)	102(2)
H(81A)-C(81)-H(81B)	111(3)
C(81)-C(82)-H(82C)	109.5
C(81)-C(82)-H(82B)	109.5
H(82C)-C(82)-H(82B)	109.5
C(81)-C(82)-H(82A)	109.5
H(82C)-C(82)-H(82A)	109.5
H(82B)-C(82)-H(82A)	109.5
N(1X)-C(11X)-C(12X)	179.2(6)
C(11X)-C(12X)-H(1AX)	109.5
C(11X)-C(12X)-H(1BX)	109.5
H(1AX)-C(12X)-H(1BX)	109.5
C(11X)-C(12X)-H(1CX)	109.5
H(1AX)-C(12X)-H(1CX)	109.5
H(1BX)-C(12X)-H(1CX)	109.5
N(2X)-C(21X)-C(22X)	179.5(8)
C(21X)-C(22X)-H(2AX)	109.5
C(21X)-C(22X)-H(2BX)	109.5
H(2AX)-C(22X)-H(2BX)	109.5
C(21X)-C(22X)-H(2CX)	109.5
H(2AX)-C(22X)-H(2CX)	109.5
H(2BX)-C(22X)-H(2CX)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 5.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	32(1)	34(1)	21(1)	6(1)	3(1)	1(1)
C(11)	31(2)	23(2)	24(2)	7(1)	2(1)	-4(1)
C(12)	32(2)	22(1)	24(2)	4(1)	4(1)	-6(1)
C(13)	36(2)	28(2)	24(2)	2(1)	3(1)	-7(1)
C(14)	33(2)	33(2)	29(2)	6(1)	-3(1)	-7(1)
C(15)	31(2)	35(2)	34(2)	8(2)	1(2)	-1(1)
C(16)	32(2)	29(2)	28(2)	4(1)	5(1)	-6(1)
C(10)	41(2)	47(2)	32(2)	5(2)	-7(2)	-3(2)
C(17)	64(4)	67(4)	28(2)	17(3)	-8(2)	-6(3)
C(18)	51(3)	74(4)	42(3)	10(3)	-11(3)	11(3)
C(19)	66(4)	58(3)	46(3)	-7(3)	-19(3)	-5(3)
C(17D)	70(20)	150(30)	39(14)	3(16)	-26(13)	-20(20)
C(18D)	210(50)	70(20)	45(16)	5(14)	-40(20)	50(30)
C(19D)	56(15)	120(20)	25(10)	15(12)	-9(10)	7(15)
C(1)	38(2)	21(2)	23(2)	3(1)	4(1)	-2(1)
O(2)	35(1)	40(1)	20(1)	9(1)	0(1)	-2(1)
C(21)	26(2)	24(2)	22(2)	7(1)	1(1)	4(1)
C(22)	28(2)	25(2)	24(2)	1(1)	-1(1)	6(1)
C(23)	29(2)	22(2)	31(2)	4(1)	4(1)	1(1)

C(24)	29(2)	26(2)	20(1)	2(1)	6(1)	2(1)
C(25)	32(2)	20(1)	22(2)	-1(1)	3(1)	1(1)
C(26)	27(2)	21(1)	25(2)	3(1)	4(1)	4(1)
C(20)	46(2)	36(2)	21(2)	5(1)	6(2)	-8(2)
C(27)	65(3)	51(2)	27(2)	9(2)	0(2)	-15(2)
C(28)	139(5)	38(2)	33(2)	13(2)	2(3)	-9(3)
C(29)	57(3)	103(4)	32(2)	-1(2)	12(2)	-12(3)
C(2)	26(2)	33(2)	26(2)	6(1)	-3(1)	1(1)
O(3)	47(2)	35(1)	28(1)	13(1)	2(1)	1(1)
C(31)	40(2)	29(2)	17(1)	5(1)	-2(1)	-5(1)
C(32)	47(2)	31(2)	13(1)	-2(1)	1(1)	-3(2)
C(33)	48(2)	26(2)	23(2)	-3(1)	1(2)	-1(1)
C(34)	42(2)	25(2)	18(1)	0(1)	-1(1)	-6(1)
C(35)	37(2)	31(2)	19(1)	-1(1)	-1(1)	-5(1)
C(36)	32(2)	29(2)	21(2)	1(1)	-5(1)	-3(1)
C(30)	51(2)	25(2)	22(2)	3(1)	1(1)	-5(2)
C(37)	61(3)	40(2)	42(2)	18(2)	-2(2)	-3(2)
C(38)	125(4)	36(2)	23(2)	8(2)	12(2)	-6(2)
C(39)	56(3)	41(2)	55(2)	14(2)	2(2)	-11(2)
C(3)	51(2)	31(2)	21(2)	-1(1)	9(2)	2(2)
O(4)	38(1)	32(1)	29(1)	7(1)	4(1)	3(1)
C(41)	38(2)	33(2)	17(1)	4(1)	9(1)	8(1)
C(42)	41(2)	36(2)	21(2)	1(1)	9(1)	7(2)
C(43)	53(2)	46(2)	33(2)	-5(2)	-4(2)	16(2)
C(44)	73(3)	43(2)	40(2)	-4(2)	-6(2)	20(2)
C(45)	69(3)	33(2)	34(2)	-2(2)	0(2)	11(2)
C(46)	47(2)	36(2)	18(2)	1(1)	9(1)	5(2)
C(40)	104(4)	54(3)	70(3)	-2(2)	-19(3)	33(3)
C(47)	150(7)	173(8)	125(6)	105(6)	38(5)	65(6)
C(48)	159(6)	76(4)	79(4)	-16(3)	-32(4)	69(4)
C(49)	106(4)	78(4)	59(3)	-1(3)	-27(3)	38(3)
C(4)	29(2)	41(2)	29(2)	5(2)	8(1)	2(2)
P(1)	28(1)	23(1)	18(1)	1(1)	0(1)	1(1)
C(51)	30(2)	25(2)	22(2)	1(1)	2(1)	2(1)
C(52)	40(2)	31(2)	24(2)	-4(1)	-5(1)	2(1)
C(53)	45(2)	48(2)	28(2)	2(2)	-10(2)	5(2)
C(54)	40(2)	43(2)	35(2)	13(2)	1(2)	11(2)
C(55)	42(2)	28(2)	35(2)	4(1)	6(2)	6(2)
C(56)	35(2)	26(2)	25(2)	1(1)	2(1)	-1(1)
C(61)	31(2)	23(2)	19(1)	2(1)	-3(1)	3(1)
C(62)	30(2)	47(2)	24(2)	-6(2)	2(1)	-1(2)
C(63)	39(2)	48(2)	21(2)	-6(2)	3(1)	3(2)
C(64)	47(2)	28(2)	21(2)	-3(1)	-6(1)	1(2)
C(65)	38(2)	27(2)	27(2)	4(1)	-3(1)	-5(1)
C(66)	32(2)	29(2)	20(1)	2(1)	2(1)	-4(1)
C(71)	27(2)	26(2)	18(1)	-1(1)	-1(1)	-1(1)
C(72)	35(2)	29(2)	26(2)	2(1)	0(1)	-1(1)
C(73)	40(2)	46(2)	20(2)	-2(1)	5(1)	-4(2)
C(74)	34(2)	47(2)	27(2)	-11(2)	2(2)	2(2)
C(75)	42(2)	32(2)	36(2)	-4(2)	-6(2)	9(2)
C(76)	39(2)	30(2)	23(2)	-1(1)	-2(1)	4(1)
C(81)	33(2)	30(2)	27(2)	4(1)	0(2)	0(2)
C(82)	51(2)	36(2)	45(2)	6(2)	-7(2)	-14(2)
N(1X)	175(6)	71(3)	83(3)	-37(3)	-39(4)	-10(3)
C(11X)	79(3)	43(2)	47(2)	-8(2)	-12(2)	-4(2)
C(12X)	57(2)	37(2)	34(2)	-7(2)	3(2)	-4(2)
N(2X)	91(4)	111(4)	94(4)	8(4)	-25(3)	-16(3)
C(21X)	86(4)	97(5)	62(3)	6(3)	-16(3)	-26(4)
C(22X)	123(5)	94(5)	76(4)	-21(4)	-11(4)	-19(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 5.

	x	y	z	U(eq)
H(14)	5454	-1036	2516	35
H(15)	7722	-1911	1566	40
H(17A)	5860	-1809	3293	80
H(17B)	6856	-2130	3607	80
H(17C)	6341	-2412	2977	80
H(18A)	8565	-2082	3115	84
H(18B)	8721	-1767	2435	84
H(18C)	8082	-2394	2491	84
H(19A)	6838	-801	3180	86
H(19B)	7956	-802	2849	86
H(19C)	7807	-1118	3529	86
H(17D)	8076	-823	2773	129
H(17E)	8687	-1385	2461	129
H(17F)	8600	-1332	3218	129
H(18D)	7588	-2514	2540	166
H(18E)	6664	-2479	3047	166
H(18F)	7840	-2369	3271	166
H(19D)	6544	-906	3319	101
H(19E)	7016	-1425	3773	101
H(19F)	5932	-1538	3416	101
H(1A)	4047	-532	1944	32
H(1B)	3906	-643	1195	32
H(23)	1327	-2564	2088	33
H(25)	3380	-1299	2702	30
H(27A)	2612	-1385	3646	71
H(27B)	3483	-1907	3613	71
H(27C)	2553	-1961	4114	71
H(28A)	1869	-3068	2947	105
H(28B)	2063	-2980	3694	105
H(28C)	3018	-2939	3210	105
H(29A)	828	-1577	3318	96
H(29B)	752	-2173	3757	96
H(29C)	497	-2220	3013	96
H(2A)	768	-2560	954	34
H(2B)	1469	-2209	441	34
H(33)	3755	-4440	551	39
H(35)	1404	-3545	1404	35
H(37A)	3897	-4905	1548	71
H(37B)	3252	-5288	1032	71
H(37C)	3113	-5431	1773	71
H(38A)	1678	-4141	2231	92
H(38B)	2914	-4199	2303	92
H(38C)	2184	-4771	2468	92
H(39A)	760	-4654	1333	76
H(39B)	1200	-5293	1591	76
H(39C)	1384	-5100	866	76
H(3A)	4470	-4028	-453	41
H(3B)	4230	-3335	-650	41
H(43)	7877	-2949	806	52
H(45)	5876	-4260	258	54
H(47A)	6343	-4686	1377	224
H(47B)	6607	-4085	1781	224
H(47C)	7347	-4672	1828	224
H(48A)	8281	-4545	206	157
H(48B)	7246	-4899	412	157
H(48C)	8294	-4983	816	157
H(49A)	8982	-3712	1031	122
H(49B)	8881	-4244	1551	122
H(49C)	8270	-3612	1644	122
H(4A)	7699	-1920	408	39
H(4B)	6674	-1715	28	39
H(52)	9175	473	1823	38
H(53)	10313	-165	2380	49
H(54)	10713	-1141	1991	48
H(55)	9889	-1502	1074	42

H(56)	8758	-869	501	34
H(62)	8873	65	-587	40
H(63)	8220	-504	-1445	43
H(64)	6539	-882	-1422	39
H(65)	5490	-731	-530	36
H(66)	6138	-180	343	32
H(72)	6923	-91	1685	36
H(73)	5599	294	2331	43
H(74)	4990	1289	2184	43
H(75)	5614	1887	1340	44
H(76)	6940	1509	689	37
H(81A)	9410(30)	819(16)	113(16)	31(9)
H(81B)	8430(30)	1219(18)	99(18)	42(11)
H(82C)	9746	1766	642	53
H(82B)	9893	1188	1101	53
H(82A)	8853	1590	1137	53
H(1AX)	-39	2043	3702	64
H(1BX)	54	2534	3141	64
H(1CX)	1077	2208	3414	64
H(2AX)	10179	-4089	-702	147
H(2BX)	10520	-4133	30	147
H(2CX)	11264	-3802	-473	147
H(2)	3600(40)	-1410(20)	580(20)	62(14)
H(3)	3770(40)	-2520(20)	-160(20)	74(15)
H(4)	5070(40)	-1900(20)	130(20)	86(16)

6) Calcium bis(2,6-ditbutyl-4-methylphenoxy)bis(triphenylaminophosphonium)

Table 1. Crystal data and structure refinement for 6.

Identification code	6 (99srv007)
Empirical formula	C66 H78 Ca N2 O2 P2
Formula weight	1033.32
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Orthorombic
Space group	Pca2(1)
Unit cell dimensions	a = 25.6442(2) A alpha = 90 deg. b = 11.53990(10) A beta = 90 deg. c = 19.818 A gamma = 90 deg.
Volume, Z	5864.86(7) A^3, 4
Density (calculated)	1.170 Mg/m^3
Absorption coefficient	0.206 mm^-1
F(000)	2216
Crystal size	0.30 x 0.15 x 0.10 mm
Theta range for data collection	1.59 to 27.49 deg.
Limiting indices	-33<=h<=33, -14<=k<=14, -25<=l<=25
Reflections collected	66148
Independent reflections	13450 [R(int) = 0.1140]
Absorption correction	Multiscan
Max. and min. transmission	0.8942 and 0.9697
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13442 / 1 / 672
Goodness-of-fit on F^2	1.095
Final R indices [I>2sigma(I)]	R1 = 0.0544, wR2 = 0.0867
R indices (all data)	R1 = 0.1075, wR2 = 0.1071
Absolute structure parameter	-0.01(3)
Extinction coefficient	not refined
Largest diff. peak and hole	0.274 and -0.267 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 6. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ca(1)	8844(1)	9210(1)	-1884(1)	20(1)
O(1)	8147(1)	8376(2)	-2249(1)	23(1)
C(1)	7770(1)	7656(3)	-2443(2)	21(1)
C(2)	7319(1)	7487(3)	-2029(2)	24(1)
C(3)	6931(1)	6725(3)	-2249(2)	28(1)
C(4)	6949(1)	6159(3)	-2869(2)	29(1)
C(5)	7385(1)	6352(3)	-3268(2)	26(1)
C(6)	7802(1)	7063(3)	-3080(2)	22(1)
C(7)	8274(1)	7211(3)	-3548(2)	25(1)
C(8)	8776(1)	6808(3)	-3198(2)	33(1)
C(9)	8228(2)	6482(3)	-4191(2)	37(1)
C(10)	8322(1)	8487(3)	-3781(2)	36(1)
C(11)	7252(1)	8141(3)	-1355(2)	27(1)
C(12)	6727(1)	7894(3)	-1008(2)	38(1)
C(13)	7258(1)	9460(3)	-1488(2)	31(1)
C(14)	7681(1)	7788(3)	-855(2)	36(1)
C(0)	6503(1)	5407(3)	-3101(2)	42(1)
O(2)	9480(1)	8071(2)	-1669(1)	24(1)
C(21)	9869(1)	7370(3)	-1501(2)	22(1)
C(22)	10376(1)	7525(3)	-1786(2)	24(1)
C(24)	10728(1)	6018(3)	-1047(2)	32(1)
C(25)	10227(1)	5808(3)	-811(2)	32(1)
C(26)	10787(1)	6852(3)	-1539(2)	31(1)
C(26)	9791(1)	6432(3)	-1031(2)	26(1)
C(27)	9243(1)	6125(3)	-767(2)	29(1)
C(28)	9045(1)	7102(3)	-305(2)	40(1)
C(29)	8863(1)	5911(3)	-1352(2)	34(1)
C(30)	9238(2)	4998(3)	-342(2)	46(1)
C(31)	10477(1)	8452(3)	-2329(2)	26(1)
C(32)	10106(1)	8276(3)	-2935(2)	30(1)
C(33)	11035(1)	8400(3)	-2615(2)	39(1)
C(34)	10401(1)	9675(3)	-2028(2)	30(1)
C(20)	11193(1)	5366(3)	-764(2)	51(1)
N(1)	9159(1)	10657(2)	-2655(1)	27(1)
P(1)	8989(1)	11736(1)	-3102(1)	22(1)
C(41)	8889(1)	11434(3)	-3988(2)	24(1)
C(42)	8459(1)	11807(3)	-4342(2)	42(1)
C(43)	8406(2)	11594(4)	-5031(2)	52(1)
C(44)	8796(2)	11017(4)	-5366(2)	49(1)
C(45)	9226(2)	10614(4)	-5017(2)	54(1)
C(46)	9271(2)	10819(4)	-4334(2)	45(1)
C(51)	8375(1)	12289(3)	-2794(2)	23(1)
C(52)	7955(1)	11529(3)	-2727(2)	34(1)
C(53)	7472(1)	11940(3)	-2531(2)	41(1)
C(54)	7404(1)	13109(3)	-2388(2)	38(1)
C(55)	7818(1)	13868(3)	-2443(2)	36(1)
C(56)	8304(1)	13454(3)	-2647(2)	29(1)
C(61)	9440(1)	12946(3)	-3090(2)	28(1)
C(62)	9561(1)	13587(3)	-3663(2)	43(1)
C(63)	9893(2)	14533(4)	-3616(3)	63(1)
C(64)	10097(2)	14845(4)	-3005(3)	53(1)
C(65)	9983(2)	14240(4)	-2437(3)	62(1)
C(66)	9651(2)	13268(4)	-2479(2)	48(1)
N(2)	8576(1)	10328(2)	-935(1)	27(1)
P(2)	8698(1)	10984(1)	-254(1)	24(1)
C(71)	9348(1)	11587(3)	-295(2)	23(1)
C(72)	9570(1)	12100(3)	273(2)	29(1)
C(73)	10068(1)	12542(3)	255(2)	33(1)
C(74)	10352(1)	12499(3)	-336(2)	33(1)
C(75)	10129(1)	12033(3)	-912(2)	39(1)
C(76)	9630(1)	11577(3)	-895(2)	32(1)
C(81)	8699(1)	10085(3)	502(2)	30(1)
C(82)	9161(2)	9542(3)	697(2)	36(1)
C(83)	9167(2)	8788(3)	1245(2)	47(1)

C(84)	8713(2)	8574(4)	1597(2)	57(1)
C(85)	8251(2)	9081(4)	1396(2)	55(1)
C(86)	8240(2)	9829(3)	845(2)	42(1)
C(91)	8259(1)	12187(3)	-99(2)	26(1)
C(92)	8134(1)	12601(3)	545(2)	34(1)
C(93)	7802(1)	13541(4)	613(2)	43(1)
C(94)	7599(1)	14086(4)	45(2)	40(1)
C(95)	7728(1)	13702(3)	-590(2)	39(1)
C(96)	8050(1)	12751(3)	-666(2)	33(1)

Table 3. Bond lengths [A] and angles [deg] for 6.

Ca(1)-O(2)	2.138(2)
Ca(1)-O(1)	2.155(2)
Ca(1)-N(2)	2.382(3)
Ca(1)-N(1)	2.403(3)
O(1)-C(1)	1.330(3)
C(1)-C(2)	1.433(4)
C(1)-C(6)	1.437(4)
C(2)-C(3)	1.398(4)
C(2)-C(11)	1.543(4)
C(3)-C(4)	1.391(5)
C(3)-H(3A)	0.95
C(4)-C(5)	1.388(4)
C(4)-C(0)	1.508(5)
C(5)-C(6)	1.397(4)
C(5)-H(5A)	0.95
C(6)-C(7)	1.536(4)
C(7)-C(9)	1.532(5)
C(7)-C(8)	1.534(4)
C(7)-C(10)	1.548(5)
C(8)-H(8A)	0.98
C(8)-H(8B)	0.98
C(8)-H(8C)	0.98
C(9)-H(9A)	0.98
C(9)-H(9B)	0.98
C(9)-H(9C)	0.98
C(10)-H(10A)	0.98
C(10)-H(10B)	0.98
C(10)-H(10C)	0.98
C(11)-C(14)	1.536(4)
C(11)-C(12)	1.538(4)
C(11)-C(13)	1.545(5)
C(12)-H(12A)	0.98
C(12)-H(12B)	0.98
C(12)-H(12C)	0.98
C(13)-H(13A)	0.98
C(13)-H(13B)	0.98
C(13)-H(13C)	0.98
C(14)-H(14A)	0.98
C(14)-H(14B)	0.98
C(14)-H(14C)	0.98
C(0)-H(0A)	0.98
C(0)-H(0B)	0.98
C(0)-H(0C)	0.98
O(2)-C(21)	1.326(4)
C(21)-C(22)	1.428(4)
C(21)-C(26)	1.443(5)
C(22)-C(26)	1.398(4)
C(22)-C(31)	1.541(4)
C(24)-C(26)	1.378(5)
C(24)-C(25)	1.388(5)
C(24)-C(20)	1.518(5)
C(25)-C(26)	1.398(4)
C(25)-H(25A)	0.95
C(26)-H(26A)	0.95
C(26)-C(27)	1.542(4)
C(27)-C(29)	1.536(5)
C(27)-C(28)	1.538(5)
C(27)-C(30)	1.549(5)
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(30)-H(30A)	0.98
C(30)-H(30B)	0.98
C(30)-H(30C)	0.98
C(31)-C(33)	1.540(4)
C(31)-C(34)	1.543(5)
C(31)-C(32)	1.547(4)
C(32)-H(32A)	0.98
C(32)-H(32B)	0.98
C(32)-H(32C)	0.98
C(33)-H(33A)	0.98
C(33)-H(33B)	0.98
C(33)-H(33C)	0.98
C(34)-H(34A)	0.98
C(34)-H(34B)	0.98
C(34)-H(34C)	0.98
C(20)-H(20A)	0.98
C(20)-H(20B)	0.98
C(20)-H(20C)	0.98
N(1)-P(1)	1.589(3)
N(1)-H(1A)	0.88
P(1)-C(51)	1.806(3)
P(1)-C(41)	1.809(3)
P(1)-C(61)	1.813(3)
C(41)-C(42)	1.375(5)
C(41)-C(46)	1.389(5)
C(42)-C(43)	1.395(5)
C(42)-H(42A)	0.95
C(43)-C(44)	1.371(5)
C(43)-H(43A)	0.95
C(44)-C(45)	1.383(6)
C(44)-H(44A)	0.95
C(45)-C(46)	1.379(5)
C(45)-H(45A)	0.95
C(46)-H(46A)	0.95
C(51)-C(56)	1.387(5)
C(51)-C(52)	1.395(5)
C(52)-C(53)	1.382(5)
C(52)-H(52A)	0.95
C(53)-C(54)	1.389(5)
C(53)-H(53A)	0.95
C(54)-C(55)	1.380(5)
C(54)-H(54A)	0.95
C(55)-C(56)	1.396(4)
C(55)-H(55A)	0.95
C(56)-H(56A)	0.95
C(61)-C(66)	1.378(5)
C(61)-C(62)	1.390(5)
C(62)-C(63)	1.388(5)
C(62)-H(62A)	0.95
C(63)-C(64)	1.366(7)
C(63)-H(63A)	0.95
C(64)-C(65)	1.357(6)
C(64)-H(64A)	0.95
C(65)-C(66)	1.410(6)
C(65)-H(65A)	0.95

C(66)-H(66A)	0.95
N(2)-P(2)	1.579(3)
N(2)-H(2A)	0.88
P(2)-C(71)	1.809(3)
P(2)-C(91)	1.814(3)
P(2)-C(81)	1.822(4)
C(71)-C(76)	1.391(4)
C(71)-C(72)	1.393(5)
C(72)-C(73)	1.378(4)
C(72)-H(72A)	0.95
C(73)-C(74)	1.379(5)
C(73)-H(73A)	0.95
C(74)-C(75)	1.385(5)
C(74)-H(74A)	0.95
C(75)-C(76)	1.384(5)
C(75)-H(75A)	0.95
C(76)-H(76A)	0.95
C(81)-C(86)	1.392(5)
C(81)-C(82)	1.395(5)
C(82)-C(83)	1.391(5)
C(82)-H(82A)	0.95
C(83)-C(84)	1.380(6)
C(83)-H(83A)	0.95
C(84)-C(85)	1.382(6)
C(84)-H(84A)	0.95
C(85)-C(86)	1.393(6)
C(85)-H(85A)	0.95
C(86)-H(86A)	0.95
C(91)-C(92)	1.400(5)
C(91)-C(96)	1.405(5)
C(92)-C(93)	1.385(5)
C(92)-H(92A)	0.95
C(93)-C(94)	1.389(5)
C(93)-H(93A)	0.95
C(94)-C(95)	1.376(5)
C(94)-H(94A)	0.95
C(95)-C(96)	1.381(5)
C(95)-H(95A)	0.95
C(96)-H(96A)	0.95

O(2)-Ca(1)-O(1)	115.14(9)
O(2)-Ca(1)-N(2)	113.36(9)
O(1)-Ca(1)-N(2)	105.52(9)
O(2)-Ca(1)-N(1)	107.30(9)
O(1)-Ca(1)-N(1)	112.04(9)
N(2)-Ca(1)-N(1)	102.88(10)
C(1)-O(1)-Ca(1)	168.0(2)
O(1)-C(1)-C(2)	120.4(3)
O(1)-C(1)-C(6)	120.7(3)
C(2)-C(1)-C(6)	118.9(3)
C(3)-C(2)-C(1)	118.7(3)
C(3)-C(2)-C(11)	120.0(3)
C(1)-C(2)-C(11)	121.3(3)
C(4)-C(3)-C(2)	123.3(3)
C(4)-C(3)-H(3A)	118.4(2)
C(2)-C(3)-H(3A)	118.4(2)
C(5)-C(4)-C(3)	117.0(3)
C(5)-C(4)-C(0)	122.0(3)
C(3)-C(4)-C(0)	121.0(3)
C(4)-C(5)-C(6)	123.9(3)
C(4)-C(5)-H(5A)	118.0(2)
C(6)-C(5)-H(5A)	118.0(2)
C(5)-C(6)-C(1)	118.1(3)
C(5)-C(6)-C(7)	120.4(3)
C(1)-C(6)-C(7)	121.4(3)
C(9)-C(7)-C(8)	105.9(3)
C(9)-C(7)-C(6)	112.4(3)
C(8)-C(7)-C(6)	110.8(3)
C(9)-C(7)-C(10)	106.3(3)
C(8)-C(7)-C(10)	110.8(3)
C(6)-C(7)-C(10)	110.4(3)
C(7)-C(8)-H(8A)	109.5(2)
C(7)-C(8)-H(8B)	109.5(2)
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5(2)
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(7)-C(9)-H(9A)	109.5(2)
C(7)-C(9)-H(9B)	109.5(2)
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5(2)
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(7)-C(10)-H(10A)	109.5(2)
C(7)-C(10)-H(10B)	109.5(2)
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.5(2)
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(14)-C(11)-C(12)	106.8(3)
C(14)-C(11)-C(2)	110.4(3)
C(12)-C(11)-C(2)	113.2(3)
C(14)-C(11)-C(13)	111.3(3)
C(12)-C(11)-C(13)	105.6(3)
C(2)-C(11)-C(13)	109.4(3)
C(11)-C(12)-H(12A)	109.5(2)
C(11)-C(12)-H(12B)	109.5(2)
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5(2)
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5(2)
C(11)-C(13)-H(13B)	109.5(2)
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5(2)
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5(2)
C(11)-C(14)-H(14B)	109.5(2)
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5(2)
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(4)-C(0)-H(0A)	109.5(2)
C(4)-C(0)-H(0B)	109.5(2)
H(0A)-C(0)-H(0B)	109.5
C(4)-C(0)-H(0C)	109.5(2)
H(0A)-C(0)-H(0C)	109.5
H(0B)-C(0)-H(0C)	109.5
C(21)-O(2)-Ca(1)	177.0(2)
O(2)-C(21)-C(22)	120.6(3)
O(2)-C(21)-C(26)	121.1(3)
C(22)-C(21)-C(26)	118.3(3)
C(26)-C(22)-C(21)	118.6(3)
C(26)-C(22)-C(31)	120.2(3)
C(21)-C(22)-C(31)	121.1(3)
C(26)-C(24)-C(25)	117.5(3)
C(26)-C(24)-C(20)	121.4(3)
C(25)-C(24)-C(20)	121.2(3)
C(24)-C(25)-C(26)	123.0(3)
C(24)-C(25)-H(25A)	118.5(2)
C(26)-C(25)-H(25A)	118.5(2)
C(24)-C(26)-C(22)	123.6(3)
C(24)-C(26)-H(26A)	118.2(2)
C(22)-C(26)-H(26A)	118.2(2)
C(25)-C(26)-C(21)	118.5(3)
C(25)-C(26)-C(27)	120.3(3)
C(21)-C(26)-C(27)	121.2(3)
C(29)-C(27)-C(28)	111.0(3)
C(29)-C(27)-C(26)	111.1(3)
C(28)-C(27)-C(26)	109.5(3)
C(29)-C(27)-C(30)	105.7(3)
C(28)-C(27)-C(30)	106.8(3)
C(26)-C(27)-C(30)	112.6(3)
C(27)-C(28)-H(28A)	109.5(2)
C(27)-C(28)-H(28B)	109.5(2)
H(28A)-C(28)-H(28B)	109.5
C(27)-C(28)-H(28C)	109.5(2)

H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(27)-C(29)-H(29A)	109.5(2)
C(27)-C(29)-H(29B)	109.5(2)
H(29A)-C(29)-H(29B)	109.5
C(27)-C(29)-H(29C)	109.5(2)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(27)-C(30)-H(30A)	109.5(2)
C(27)-C(30)-H(30B)	109.5(2)
H(30A)-C(30)-H(30B)	109.5
C(27)-C(30)-H(30C)	109.5(2)
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
C(33)-C(31)-C(22)	112.8(3)
C(33)-C(31)-C(34)	107.2(3)
C(22)-C(31)-C(34)	110.1(3)
C(33)-C(31)-C(32)	106.4(3)
C(22)-C(31)-C(32)	110.3(3)
C(34)-C(31)-C(32)	110.0(3)
C(31)-C(32)-H(32A)	109.5(2)
C(31)-C(32)-H(32B)	109.5(2)
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5(2)
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(31)-C(33)-H(33A)	109.5(2)
C(31)-C(33)-H(33B)	109.5(2)
H(33A)-C(33)-H(33B)	109.5
C(31)-C(33)-H(33C)	109.5(2)
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(31)-C(34)-H(34A)	109.5(2)
C(31)-C(34)-H(34B)	109.5(2)
H(34A)-C(34)-H(34B)	109.5
C(31)-C(34)-H(34C)	109.5(2)
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(24)-C(20)-H(20A)	109.5(2)
C(24)-C(20)-H(20B)	109.5(2)
H(20A)-C(20)-H(20B)	109.5
C(24)-C(20)-H(20C)	109.5(2)
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
P(1)-N(1)-Ca(1)	143.8(2)
P(1)-N(1)-H(1A)	108.08(10)
Ca(1)-N(1)-H(1A)	108.08(6)
N(1)-P(1)-C(51)	109.1(2)
N(1)-P(1)-C(41)	115.4(2)
C(51)-P(1)-C(41)	105.8(2)
N(1)-P(1)-C(61)	115.0(2)
C(51)-P(1)-C(61)	106.2(2)
C(41)-P(1)-C(61)	104.6(2)
C(42)-C(41)-C(46)	118.2(3)
C(42)-C(41)-P(1)	123.3(3)
C(46)-C(41)-P(1)	118.5(3)
C(41)-C(42)-C(43)	121.5(3)
C(41)-C(42)-H(42A)	119.2(2)
C(43)-C(42)-H(42A)	119.2(2)
C(44)-C(43)-C(42)	119.2(4)
C(44)-C(43)-H(43A)	120.4(2)
C(42)-C(43)-H(43A)	120.4(2)
C(43)-C(44)-C(45)	120.2(4)
C(43)-C(44)-H(44A)	119.9(2)
C(45)-C(44)-H(44A)	119.9(2)
C(46)-C(45)-C(44)	120.0(4)
C(46)-C(45)-H(45A)	120.0(2)
C(44)-C(45)-H(45A)	120.0(2)
C(45)-C(46)-C(41)	120.9(4)
C(45)-C(46)-H(46A)	119.6(2)
C(41)-C(46)-H(46A)	119.6(2)
C(56)-C(51)-C(52)	119.2(3)
C(56)-C(51)-P(1)	121.9(3)
C(52)-C(51)-P(1)	118.9(3)
C(53)-C(52)-C(51)	120.2(3)
C(53)-C(52)-H(52A)	119.9(2)
C(51)-C(52)-H(52A)	119.9(2)
C(52)-C(53)-C(54)	120.2(4)
C(52)-C(53)-H(53A)	119.9(2)
C(54)-C(53)-H(53A)	119.9(2)
C(55)-C(54)-C(53)	120.3(3)
C(55)-C(54)-H(54A)	119.9(2)
C(53)-C(54)-H(54A)	119.9(2)
C(54)-C(55)-C(56)	119.4(3)
C(54)-C(55)-H(55A)	120.3(2)
C(56)-C(55)-H(55A)	120.3(2)
C(51)-C(56)-C(55)	120.7(3)
C(51)-C(56)-H(56A)	119.6(2)
C(55)-C(56)-H(56A)	119.6(2)
C(66)-C(61)-C(62)	119.2(3)
C(66)-C(61)-P(1)	118.0(3)
C(62)-C(61)-P(1)	122.7(3)
C(63)-C(62)-C(61)	120.0(4)
C(63)-C(62)-H(62A)	120.0(3)
C(61)-C(62)-H(62A)	120.0(2)
C(64)-C(63)-C(62)	120.1(5)
C(64)-C(63)-H(63A)	119.9(3)
C(62)-C(63)-H(63A)	119.9(3)
C(65)-C(64)-C(63)	121.2(4)
C(65)-C(64)-H(64A)	119.4(3)
C(63)-C(64)-H(64A)	119.4(3)
C(64)-C(65)-C(66)	119.3(4)
C(64)-C(65)-H(65A)	120.4(3)
C(66)-C(65)-H(65A)	120.4(3)
C(61)-C(66)-C(65)	120.3(4)
C(61)-C(66)-H(66A)	119.9(2)
C(65)-C(66)-H(66A)	119.9(3)
P(2)-N(2)-Ca(1)	151.2(2)
P(2)-N(2)-H(2A)	104.38(10)
Ca(1)-N(2)-H(2A)	104.38(6)
N(2)-P(2)-C(71)	109.3(2)
N(2)-P(2)-C(91)	112.9(2)
C(71)-P(2)-C(91)	106.6(2)
N(2)-P(2)-C(81)	115.5(2)
C(71)-P(2)-C(81)	104.7(2)
C(91)-P(2)-C(81)	107.3(2)
C(76)-C(71)-C(72)	118.9(3)
C(76)-C(71)-P(2)	120.9(3)
C(72)-C(71)-P(2)	120.2(3)
C(73)-C(72)-C(71)	120.9(3)
C(73)-C(72)-H(72A)	119.5(2)
C(71)-C(72)-H(72A)	119.5(2)
C(72)-C(73)-C(74)	119.9(3)
C(72)-C(73)-H(73A)	120.1(2)
C(74)-C(73)-H(73A)	120.1(2)
C(73)-C(74)-C(75)	119.8(3)
C(73)-C(74)-H(74A)	120.1(2)
C(75)-C(74)-H(74A)	120.1(2)
C(76)-C(75)-C(74)	120.6(3)
C(76)-C(75)-H(75A)	119.7(2)
C(74)-C(75)-H(75A)	119.7(2)
C(75)-C(76)-C(71)	119.8(3)
C(75)-C(76)-H(76A)	120.1(2)
C(71)-C(76)-H(76A)	120.1(2)
C(86)-C(81)-C(82)	119.2(3)
C(86)-C(81)-P(2)	121.4(3)
C(82)-C(81)-P(2)	119.0(3)
C(83)-C(82)-C(81)	120.5(4)
C(83)-C(82)-H(82A)	119.8(3)
C(81)-C(82)-H(82A)	119.8(2)
C(84)-C(83)-C(82)	119.8(4)
C(84)-C(83)-H(83A)	120.1(3)
C(82)-C(83)-H(83A)	120.1(3)
C(83)-C(84)-C(85)	120.2(4)
C(83)-C(84)-H(84A)	119.9(3)
C(85)-C(84)-H(84A)	119.9(2)
C(84)-C(85)-C(86)	120.4(4)
C(84)-C(85)-H(85A)	119.8(2)

C(86)-C(85)-H(85A)	119.8(3)	C(95)-C(94)-C(93)	120.3(3)
C(81)-C(86)-C(85)	119.9(4)	C(95)-C(94)-H(94A)	119.9(2)
C(81)-C(86)-H(86A)	120.1(2)	C(93)-C(94)-H(94A)	119.9(2)
C(85)-C(86)-H(86A)	120.1(3)	C(94)-C(95)-C(96)	120.0(4)
C(92)-C(91)-C(96)	118.9(3)	C(94)-C(95)-H(95A)	120.0(2)
C(92)-C(91)-P(2)	124.0(3)	C(96)-C(95)-H(95A)	120.0(2)
C(96)-C(91)-P(2)	117.1(2)	C(95)-C(96)-C(91)	120.6(3)
C(93)-C(92)-C(91)	119.7(4)	C(95)-C(96)-H(96A)	119.7(2)
C(93)-C(92)-H(92A)	120.1(2)	C(91)-C(96)-H(96A)	119.7(2)
C(91)-C(92)-H(92A)	120.1(2)		
C(92)-C(93)-C(94)	120.5(4)		
C(92)-C(93)-H(93A)	119.8(2)		
C(94)-C(93)-H(93A)	119.8(2)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 6. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ca(1)	19(1)	19(1)	22(1)	-1(1)	-2(1)	0(1)
O(1)	18(1)	28(1)	24(1)	-3(1)	2(1)	-5(1)
C(1)	18(2)	18(2)	25(2)	3(2)	-4(1)	-1(1)
C(2)	22(2)	22(2)	27(2)	3(1)	-3(1)	0(1)
C(3)	20(2)	26(2)	37(2)	5(2)	4(2)	-2(2)
C(4)	23(2)	22(2)	42(2)	2(2)	-5(2)	0(2)
C(5)	29(2)	20(2)	28(2)	-4(2)	-7(2)	0(1)
C(6)	24(2)	18(2)	25(2)	2(2)	-2(2)	-1(1)
C(7)	26(2)	25(2)	24(2)	-5(2)	2(2)	-8(2)
C(8)	27(2)	36(2)	35(2)	-6(2)	4(2)	2(2)
C(9)	42(2)	38(2)	31(2)	-11(2)	5(2)	-7(2)
C(10)	45(2)	31(2)	31(2)	-1(2)	7(2)	-10(2)
C(11)	23(2)	30(2)	28(2)	0(2)	4(2)	-3(2)
C(12)	33(2)	42(2)	39(2)	-2(2)	13(2)	-3(2)
C(13)	27(2)	31(2)	36(2)	-1(2)	9(2)	3(2)
C(14)	38(2)	43(2)	27(2)	5(2)	1(2)	0(2)
C(0)	24(2)	36(2)	66(3)	-6(2)	-1(2)	-9(2)
O(2)	23(1)	22(1)	27(1)	6(1)	0(1)	5(1)
C(21)	20(2)	23(2)	23(2)	-4(2)	-5(1)	2(1)
C(22)	24(2)	20(2)	26(2)	-3(2)	-3(2)	3(1)
C(24)	27(2)	23(2)	44(2)	6(2)	-9(2)	4(2)
C(25)	36(2)	24(2)	36(2)	5(2)	-7(2)	-1(2)
C(26)	24(2)	24(2)	44(2)	-2(2)	-2(2)	-2(2)
C(26)	26(2)	24(2)	29(2)	-3(2)	0(2)	2(2)
C(27)	31(2)	24(2)	32(2)	7(2)	4(2)	3(2)
C(28)	43(2)	48(2)	30(2)	4(2)	2(2)	8(2)
C(29)	28(2)	28(2)	46(2)	1(2)	2(2)	-2(2)
C(30)	41(2)	46(2)	51(3)	24(2)	5(2)	2(2)
C(31)	24(2)	24(2)	31(2)	3(2)	-1(2)	-1(2)
C(32)	36(2)	28(2)	25(2)	3(2)	4(2)	1(2)
C(33)	28(2)	38(2)	51(2)	9(2)	7(2)	2(2)
C(34)	30(2)	24(2)	37(2)	2(2)	-8(2)	-3(2)
C(20)	38(2)	38(2)	78(3)	19(2)	-12(2)	5(2)
N(1)	19(1)	32(2)	30(2)	7(1)	0(1)	4(1)
P(1)	21(1)	21(1)	24(1)	2(1)	1(1)	0(1)
C(41)	27(2)	23(2)	23(2)	1(2)	3(2)	-3(2)
C(42)	39(2)	52(3)	33(2)	-6(2)	-5(2)	18(2)
C(43)	60(3)	60(3)	35(2)	-6(2)	-15(2)	14(2)
C(44)	68(3)	52(3)	26(2)	-9(2)	1(2)	-4(2)
C(45)	54(3)	69(3)	40(3)	-17(2)	6(2)	9(2)
C(46)	39(2)	60(3)	34(2)	-7(2)	0(2)	17(2)
C(51)	23(2)	24(2)	21(2)	1(2)	2(1)	1(1)
C(52)	30(2)	31(2)	42(2)	6(2)	5(2)	3(2)
C(53)	20(2)	44(2)	58(3)	15(2)	11(2)	1(2)
C(54)	29(2)	48(3)	36(2)	9(2)	10(2)	14(2)
C(55)	41(2)	33(2)	34(2)	0(2)	4(2)	17(2)
C(56)	31(2)	27(2)	28(2)	3(2)	0(2)	-2(2)
C(61)	22(2)	24(2)	37(2)	-6(2)	5(2)	1(1)

C(62)	42(2)	36(2)	50(3)	5(2)	8(2)	-8(2)
C(63)	53(3)	43(3)	92(4)	16(3)	20(3)	-11(2)
C(64)	34(2)	30(2)	96(4)	-9(3)	11(3)	-10(2)
C(65)	47(3)	67(3)	71(3)	-30(3)	-8(2)	-14(3)
C(66)	44(2)	52(3)	49(3)	-2(2)	-3(2)	-21(2)
N(2)	22(1)	31(2)	30(2)	-10(1)	0(1)	2(1)
P(2)	23(1)	26(1)	23(1)	-4(1)	2(1)	-3(1)
C(71)	21(2)	22(2)	26(2)	0(2)	0(2)	2(1)
C(72)	29(2)	35(2)	23(2)	0(2)	2(2)	-3(2)
C(73)	33(2)	33(2)	34(2)	-1(2)	-11(2)	-6(2)
C(74)	21(2)	37(2)	40(2)	-2(2)	2(2)	-5(2)
C(75)	36(2)	42(2)	40(2)	-6(2)	14(2)	-2(2)
C(76)	32(2)	34(2)	29(2)	-9(2)	5(2)	-4(2)
C(81)	38(2)	27(2)	26(2)	-3(2)	4(2)	-12(2)
C(82)	43(2)	31(2)	34(2)	0(2)	1(2)	-6(2)
C(83)	71(3)	32(2)	38(2)	6(2)	-10(2)	-11(2)
C(84)	92(4)	38(3)	41(3)	8(2)	-6(3)	-31(3)
C(85)	67(3)	57(3)	40(3)	2(2)	14(2)	-38(3)
C(86)	45(2)	44(2)	36(2)	-1(2)	1(2)	-21(2)
C(91)	21(2)	30(2)	27(2)	-7(2)	2(1)	-5(2)
C(92)	30(2)	40(2)	31(2)	-12(2)	-2(2)	-1(2)
C(93)	41(2)	49(3)	37(2)	-22(2)	2(2)	1(2)
C(94)	29(2)	44(2)	48(2)	-16(2)	4(2)	9(2)
C(95)	31(2)	44(2)	43(2)	-1(2)	-1(2)	13(2)
C(96)	32(2)	41(2)	27(2)	-10(2)	2(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 6.

	x	y	z	U(eq)
H(3A)	6641(1)	6586(3)	-1962(2)	33
H(5A)	7402(1)	5979(3)	-3695(2)	31
H(8A)	8747(4)	5984(6)	-3085(10)	49
H(8B)	8828(5)	7258(14)	-2784(6)	49
H(8C)	9073(2)	6926(18)	-3501(5)	49
H(9A)	8192(9)	5662(4)	-4070(2)	56
H(9B)	8542(4)	6587(15)	-4467(6)	56
H(9C)	7921(5)	6730(13)	-4448(6)	56
H(10A)	8628(5)	8570(5)	-4074(9)	53
H(10B)	8362(9)	8990(4)	-3385(2)	53
H(10C)	8008(4)	8709(7)	-4030(10)	53
H(12A)	6711(4)	8314(16)	-579(6)	56
H(12B)	6694(4)	7061(4)	-924(11)	56
H(12C)	6441(1)	8150(19)	-1301(5)	56
H(13A)	7237(8)	9876(3)	-1058(2)	47
H(13B)	6959(5)	9671(4)	-1771(8)	47
H(13C)	7582(4)	9671(4)	-1721(9)	47
H(14A)	7648(5)	8249(14)	-441(5)	53
H(14B)	8024(1)	7926(18)	-1058(5)	53
H(14C)	7645(5)	6964(6)	-745(9)	53
H(0A)	6641(1)	4703(10)	-3312(11)	63
H(0B)	6291(5)	5834(8)	-3430(9)	63
H(0C)	6286(5)	5195(17)	-2713(3)	63
H(25A)	10178(1)	5213(3)	-486(2)	39
H(26A)	11125(1)	6975(3)	-1720(2)	37
H(28A)	9054(9)	7839(5)	-551(4)	60
H(28B)	9269(6)	7155(14)	94(6)	60
H(28C)	8687(3)	6935(10)	-165(10)	60
H(29A)	8817(6)	6629(6)	-1610(7)	51
H(29B)	8525(3)	5661(18)	-1172(2)	51
H(29C)	9003(4)	5307(13)	-1648(6)	51
H(30A)	9380(9)	4359(6)	-610(5)	69
H(30B)	8879(2)	4816(13)	-209(11)	69
H(30C)	9452(8)	5107(8)	63(7)	69
H(32A)	10166(6)	7510(8)	-3134(7)	45
H(32B)	10172(6)	8876(11)	-3275(5)	45
H(32C)	9744(1)	8331(18)	-2781(2)	45
H(33A)	11097(3)	7634(8)	-2814(10)	59
H(33B)	11286(1)	8534(20)	-2250(3)	59

H(33C)	11077(3)	8997(13)	-2962(8)	59
H(34A)	10656(6)	9800(7)	-1667(7)	45
H(34B)	10048(3)	9743(7)	-1845(10)	45
H(34C)	10453(8)	10257(3)	-2382(3)	45
H(20A)	11124(4)	4532(4)	-780(13)	76
H(20B)	11253(6)	5604(17)	-296(5)	76
H(20C)	11503(3)	5543(18)	-1034(8)	76
H(1A)	9495(1)	10547(2)	-2712(1)	33
H(42A)	8193(1)	12218(3)	-4111(2)	50
H(43A)	8103(2)	11845(4)	-5266(2)	62
H(44A)	8770(2)	10895(4)	-5839(2)	58
H(45A)	9491(2)	10196(4)	-5248(2)	65
H(46A)	9566(2)	10537(4)	-4097(2)	54
H(52A)	8002(1)	10727(3)	-2817(2)	41
H(53A)	7186(1)	11422(3)	-2494(2)	49
H(54A)	7071(1)	13386(3)	-2253(2)	45
H(55A)	7772(1)	14667(3)	-2342(2)	43
H(56A)	8589(1)	13974(3)	-2686(2)	34
H(62A)	9415(1)	13379(3)	-4087(2)	51
H(63A)	9979(2)	14964(4)	-4009(3)	75
H(64A)	10322(2)	15497(4)	-2978(3)	64
H(65A)	10125(2)	14469(4)	-2015(3)	74
H(66A)	9573(2)	12833(4)	-2085(2)	58
H(2A)	8238(1)	10425(2)	-994(1)	33
H(72A)	9374(1)	12145(3)	679(2)	35
H(73A)	10217(1)	12876(3)	649(2)	40
H(74A)	10698(1)	12787(3)	-348(2)	39
H(75A)	10320(1)	12027(3)	-1323(2)	47
H(76A)	9480(1)	11258(3)	-1292(2)	38
H(82A)	9473(2)	9687(3)	454(2)	43
H(83A)	9483(2)	8423(3)	1376(2)	56
H(84A)	8719(2)	8075(4)	1979(2)	68
H(85A)	7938(2)	8920(4)	1636(2)	66
H(86A)	7919(2)	10163(3)	703(2)	50
H(92A)	8275(1)	12238(3)	934(2)	40
H(93A)	7713(1)	13815(4)	1050(2)	51
H(94A)	7370(1)	14727(4)	97(2)	48
H(95A)	7596(1)	14090(3)	-977(2)	47
H(96A)	8131(1)	12475(3)	-1105(2)	40

7) Strontium bis(2,6-ditbutyl-4-methylphenoxy)bis(triphenylaminophosphonium)

Table 1. Crystal data and structure refinement for 7.

Identification code	7 (99srv006)
Empirical formula	C66 H78 N2 O2 P2 Sr
Formula weight	1080.86
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Orthorombic
Space group	Pca2(1)
Unit cell dimensions	a = 25.6929(2) A alpha = 90 deg. b = 11.69650(10) A beta = 90 deg. c = 19.9288(3) A gamma = 90 deg.
Volume, Z	5988.94(11) A^3, 4
Density (calculated)	1.199 Mg/m^3
Absorption coefficient	0.997 mm^-1
F(000)	2288
Crystal size	0.35 x 0.25 x 0.20 mm
Theta range for data collection	1.59 to 27.49 deg.
Limiting indices	-33<=h<=33, -15<=k<=15, -25<=l<=25
Reflections collected	66948
Independent reflections	13720 [R(int) = 0.0931]
Absorption correction	Multiscan
Max. and min. transmission	0.8215 and 0.7380
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13707 / 1 / 672
Goodness-of-fit on F^2	1.062
Final R indices [I>2sigma(I)]	R1 = 0.0460, wR2 = 0.0729
R indices (all data)	R1 = 0.0864, wR2 = 0.0876
Absolute structure parameter	-0.006(4)
Extinction coefficient	not refined
Largest diff. peak and hole	0.375 and -0.269 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 7. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Sr(1)	3857(1)	752(1)	7511(1)	23(1)
O(1)	4520(1)	1969(2)	7265(1)	29(1)
C(1)	4906(1)	2667(3)	7103(2)	26(1)
C(2)	5412(1)	2503(3)	7385(2)	26(1)
C(3)	5822(1)	3183(3)	7156(2)	33(1)
C(4)	5761(1)	4006(3)	6669(2)	34(1)
C(5)	5261(1)	4208(3)	6426(2)	34(1)
C(6)	4827(1)	3588(3)	6632(2)	30(1)
C(7)	4280(1)	3875(3)	6368(2)	35(1)
C(8)	4082(2)	2895(4)	5918(2)	51(1)
C(9)	4271(2)	4973(3)	5938(2)	57(1)
C(10)	3899(1)	4090(3)	6956(2)	42(1)
C(11)	5506(1)	1577(3)	7928(2)	29(1)
C(12)	6063(1)	1605(3)	8204(2)	45(1)
C(13)	5131(1)	1744(3)	8523(2)	34(1)
C(14)	5425(1)	376(3)	7621(2)	35(1)
C(0)	6228(2)	4664(3)	6396(2)	56(1)
O(2)	3118(1)	1620(2)	7900(1)	26(1)
C(21)	2746(1)	2337(3)	8083(2)	24(1)
C(22)	2298(1)	2517(3)	7661(2)	26(1)
C(23)	1914(1)	3280(3)	7879(2)	30(1)
C(24)	1932(1)	3841(3)	8485(2)	33(1)
C(25)	2367(1)	3651(3)	8891(2)	30(1)
C(26)	2776(1)	2932(2)	8711(2)	23(1)
C(27)	3252(1)	2772(3)	9179(2)	29(1)
C(28)	3209(2)	3501(3)	9820(2)	42(1)
C(29)	3749(1)	3149(3)	8821(2)	36(1)
C(30)	3296(2)	1512(3)	9409(2)	40(1)
C(31)	2238(1)	1864(3)	6994(2)	30(1)
C(32)	2672(1)	2209(3)	6503(2)	40(1)
C(33)	2244(1)	562(3)	7130(2)	34(1)
C(34)	1717(1)	2115(3)	6644(2)	42(1)
C(20)	1491(1)	4610(3)	8714(2)	48(1)
N(1)	3560(1)	-415(2)	6520(1)	36(1)
P(1)	3690(1)	-1057(1)	5843(1)	31(1)
C(41)	3685(2)	-155(3)	5095(2)	40(1)
C(42)	3226(2)	70(4)	4757(2)	58(1)
C(43)	3228(2)	839(5)	4217(2)	78(2)
C(44)	3681(3)	1369(4)	4025(3)	82(2)
C(45)	4136(2)	1171(4)	4370(2)	64(1)
C(46)	4142(2)	406(3)	4911(2)	47(1)
C(51)	3260(1)	-2259(3)	5688(2)	33(1)
C(52)	3133(1)	-2671(3)	5052(2)	41(1)
C(53)	2802(2)	-3601(4)	4989(2)	52(1)
C(54)	2613(2)	-4146(4)	5554(2)	51(1)
C(55)	2747(2)	-3768(3)	6182(2)	49(1)
C(56)	3067(2)	-2824(3)	6253(2)	41(1)
C(61)	4344(1)	-1620(3)	5890(2)	26(1)
C(62)	4617(2)	-1612(3)	6493(2)	39(1)
C(63)	5123(2)	-2027(3)	6512(2)	46(1)
C(64)	5356(1)	-2461(3)	5943(2)	38(1)
C(65)	5079(2)	-2504(3)	5346(2)	38(1)
C(66)	4578(1)	-2093(3)	5326(2)	34(1)
N(2)	4191(1)	-771(2)	8319(1)	35(1)
P(2)	4013(1)	-1840(1)	8747(1)	26(1)
C(71)	3902(1)	-1559(3)	9630(2)	31(1)
C(72)	4271(2)	-940(4)	9977(2)	71(2)
C(73)	4209(2)	-723(5)	10654(2)	90(2)
C(74)	3771(2)	-1096(4)	10987(2)	55(1)
C(75)	3397(2)	-1655(4)	10640(2)	62(1)
C(76)	3463(2)	-1896(3)	9964(2)	50(1)
C(81)	4456(1)	-3042(3)	8727(2)	32(1)
C(82)	4564(2)	-3722(3)	9273(2)	50(1)
C(83)	4892(2)	-4650(4)	9224(3)	76(2)
C(84)	5102(2)	-4916(4)	8610(4)	71(2)

C(85)	5001(2)	-4283(5)	8054(3)	75(2)
C(86)	4676(2)	-3328(4)	8114(2)	59(1)
C(91)	3402(1)	-2357(3)	8422(2)	26(1)
C(92)	2991(1)	-1592(3)	8338(2)	34(1)
C(93)	2508(1)	-1974(3)	8124(2)	43(1)
C(94)	2432(2)	-3120(3)	7984(2)	43(1)
C(95)	2835(2)	-3892(3)	8058(2)	39(1)
C(96)	3321(1)	-3504(3)	8271(2)	33(1)

Table 3. Bond lengths [A] and angles [deg] for 7.

Sr(1)-O(1)	2.272(2)
Sr(1)-O(2)	2.288(2)
Sr(1)-N(1)	2.520(3)
Sr(1)-N(2)	2.551(3)
O(1)-C(1)	1.326(4)
C(1)-C(2)	1.428(4)
C(1)-C(6)	1.443(5)
C(2)-C(3)	1.398(4)
C(2)-C(11)	1.550(4)
C(3)-C(4)	1.376(5)
C(3)-H(3)	0.95
C(4)-C(5)	1.394(5)
C(4)-C(0)	1.525(5)
C(5)-C(6)	1.392(5)
C(5)-H(5)	0.95
C(6)-C(7)	1.539(5)
C(7)-C(8)	1.543(5)
C(7)-C(9)	1.544(5)
C(7)-C(10)	1.547(5)
C(8)-H(8A)	0.98
C(8)-H(8B)	0.98
C(8)-H(8C)	0.98
C(9)-H(9A)	0.98
C(9)-H(9B)	0.98
C(9)-H(9C)	0.98
C(10)-H(10A)	0.98
C(10)-H(10B)	0.98
C(10)-H(10C)	0.98
C(11)-C(12)	1.533(5)
C(11)-C(13)	1.539(5)
C(11)-C(14)	1.547(4)
C(12)-H(12A)	0.98
C(12)-H(12B)	0.98
C(12)-H(12C)	0.98
C(13)-H(13A)	0.98
C(13)-H(13B)	0.98
C(13)-H(13C)	0.98
C(14)-H(14A)	0.98
C(14)-H(14B)	0.98
C(14)-H(14C)	0.98
C(0)-H(0A)	0.98
C(0)-H(0B)	0.98
C(0)-H(0C)	0.98
O(2)-C(21)	1.324(4)
C(21)-C(26)	1.434(4)
C(21)-C(22)	1.440(4)
C(22)-C(23)	1.398(4)
C(22)-C(31)	1.542(4)
C(23)-C(24)	1.376(5)
C(23)-H(23)	0.95
C(24)-C(25)	1.397(5)
C(24)-C(20)	1.518(5)
C(25)-C(26)	1.393(4)
C(25)-H(25)	0.95
C(26)-C(27)	1.549(4)
C(27)-C(29)	1.529(5)
C(27)-C(28)	1.539(5)
C(27)-C(30)	1.547(4)
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(30)-H(30A)	0.98
C(30)-H(30B)	0.98
C(30)-H(30C)	0.98
C(31)-C(34)	1.537(5)
C(31)-C(32)	1.539(5)
C(31)-C(33)	1.547(5)
C(32)-H(32A)	0.98
C(32)-H(32B)	0.98
C(32)-H(32C)	0.98
C(33)-H(33A)	0.98
C(33)-H(33B)	0.98
C(33)-H(33C)	0.98
C(34)-H(34A)	0.98
C(34)-H(34B)	0.98
C(34)-H(34C)	0.98
C(20)-H(20A)	0.98
C(20)-H(20B)	0.98
C(20)-H(20C)	0.98
N(1)-P(1)	1.581(3)
N(1)-H(1)	0.88
P(1)-C(61)	1.808(3)
P(1)-C(51)	1.814(4)
P(1)-C(41)	1.826(4)
C(41)-C(42)	1.382(5)
C(41)-C(46)	1.395(5)
C(42)-C(43)	1.402(7)
C(42)-H(42)	0.95
C(43)-C(44)	1.375(7)
C(43)-H(43)	0.95
C(44)-C(45)	1.374(7)
C(44)-H(44)	0.95
C(45)-C(46)	1.401(6)
C(45)-H(45)	0.95
C(46)-H(46)	0.95
C(51)-C(52)	1.394(5)
C(51)-C(56)	1.396(5)
C(52)-C(53)	1.387(5)
C(52)-H(52)	0.95
C(53)-C(54)	1.381(6)
C(53)-H(53)	0.95
C(54)-C(55)	1.371(5)
C(54)-H(54)	0.95
C(55)-C(56)	1.383(5)
C(55)-H(55)	0.95
C(56)-H(56)	0.95
C(61)-C(66)	1.389(5)
C(61)-C(62)	1.391(5)
C(62)-C(63)	1.390(5)
C(62)-H(62)	0.95
C(63)-C(64)	1.379(5)
C(63)-H(63)	0.95
C(64)-C(65)	1.387(5)

C(64)-H(64)	0.95
C(65)-C(66)	1.375(5)
C(65)-H(65)	0.95
C(66)-H(66)	0.95
N(2)-P(2)	1.582(3)
N(2)-H(2)	0.88
P(2)-C(91)	1.805(3)
P(2)-C(81)	1.808(3)
P(2)-C(71)	1.813(3)
C(71)-C(76)	1.368(5)
C(71)-C(72)	1.377(5)
C(72)-C(73)	1.382(6)
C(72)-H(72)	0.95
C(73)-C(74)	1.376(6)
C(73)-H(73)	0.95
C(74)-C(75)	1.353(6)
C(74)-H(74)	0.95
C(75)-C(76)	1.386(5)
C(75)-H(75)	0.95
C(76)-H(76)	0.95
C(81)-C(82)	1.376(5)
C(81)-C(86)	1.388(5)
C(82)-C(83)	1.378(6)
C(82)-H(82)	0.95
C(83)-C(84)	1.373(7)
C(83)-H(83)	0.95
C(84)-C(85)	1.358(7)
C(84)-H(84)	0.95
C(85)-C(86)	1.400(6)
C(85)-H(85)	0.95
C(86)-H(86)	0.95
C(91)-C(96)	1.390(4)
C(91)-C(92)	1.395(5)
C(92)-C(93)	1.386(5)
C(92)-H(92)	0.95
C(93)-C(94)	1.383(5)
C(93)-H(93)	0.95
C(94)-C(95)	1.381(5)
C(94)-H(94)	0.95
C(95)-C(96)	1.395(5)
C(95)-H(95)	0.95
C(96)-H(96)	0.95

O(1)-Sr(1)-O(2)	114.66(8)
O(1)-Sr(1)-N(1)	113.42(9)
O(2)-Sr(1)-N(1)	104.71(8)
O(1)-Sr(1)-N(2)	108.74(9)
O(2)-Sr(1)-N(2)	112.06(8)
N(1)-Sr(1)-N(2)	102.60(9)
C(1)-O(1)-Sr(1)	178.3(2)
O(1)-C(1)-C(2)	120.1(3)
O(1)-C(1)-C(6)	120.9(3)
C(2)-C(1)-C(6)	119.0(3)
C(3)-C(2)-C(1)	118.7(3)
C(3)-C(2)-C(11)	120.5(3)
C(1)-C(2)-C(11)	120.8(3)
C(4)-C(3)-C(2)	122.9(3)
C(4)-C(3)-H(3)	118.6(2)
C(2)-C(3)-H(3)	118.6(2)
C(3)-C(4)-C(5)	117.9(3)
C(3)-C(4)-C(0)	121.0(3)
C(5)-C(4)-C(0)	121.1(3)
C(6)-C(5)-C(4)	123.2(3)
C(6)-C(5)-H(5)	118.4(2)
C(4)-C(5)-H(5)	118.4(2)
C(5)-C(6)-C(1)	118.0(3)
C(5)-C(6)-C(7)	121.2(3)
C(1)-C(6)-C(7)	120.9(3)
C(6)-C(7)-C(8)	109.8(3)
C(6)-C(7)-C(9)	112.6(3)
C(8)-C(7)-C(9)	106.9(3)
C(6)-C(7)-C(10)	110.8(3)
C(8)-C(7)-C(10)	110.7(3)
C(9)-C(7)-C(10)	106.1(3)
C(7)-C(8)-H(8A)	109.5(2)
C(7)-C(8)-H(8B)	109.5(2)
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5(2)
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(7)-C(9)-H(9A)	109.5(2)
C(7)-C(9)-H(9B)	109.5(2)
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5(2)
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(7)-C(10)-H(10A)	109.5(2)
C(7)-C(10)-H(10B)	109.5(2)
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.5(2)
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(12)-C(11)-C(13)	107.7(3)
C(12)-C(11)-C(14)	106.7(3)
C(13)-C(11)-C(14)	109.6(3)
C(12)-C(11)-C(2)	112.4(3)
C(13)-C(11)-C(2)	110.6(3)
C(14)-C(11)-C(2)	109.7(3)
C(11)-C(12)-H(12A)	109.5(2)
C(11)-C(12)-H(12B)	109.5(2)
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5(2)
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5(2)
C(11)-C(13)-H(13B)	109.5(2)
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5(2)
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5(2)
C(11)-C(14)-H(14B)	109.5(2)
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5(2)
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(4)-C(0)-H(0A)	109.5(2)
C(4)-C(0)-H(0B)	109.5(2)
H(0A)-C(0)-H(0B)	109.5
C(4)-C(0)-H(0C)	109.5(2)
H(0A)-C(0)-H(0C)	109.5
H(0B)-C(0)-H(0C)	109.5
C(21)-O(2)-Sr(1)	167.0(2)
O(2)-C(21)-C(26)	120.6(3)
O(2)-C(21)-C(22)	120.6(3)
C(26)-C(21)-C(22)	118.8(3)
C(23)-C(22)-C(21)	118.4(3)
C(23)-C(22)-C(31)	120.9(3)
C(21)-C(22)-C(31)	120.7(3)
C(24)-C(23)-C(22)	123.6(3)
C(24)-C(23)-H(23)	118.2(2)
C(22)-C(23)-H(23)	118.2(2)
C(23)-C(24)-C(25)	117.3(3)
C(23)-C(24)-C(20)	121.5(3)
C(25)-C(24)-C(20)	121.2(3)
C(26)-C(25)-C(24)	123.4(3)
C(26)-C(25)-H(25)	118.3(2)
C(24)-C(25)-H(25)	118.3(2)
C(25)-C(26)-C(21)	118.5(3)
C(25)-C(26)-C(27)	120.9(3)
C(21)-C(26)-C(27)	120.6(3)
C(29)-C(27)-C(28)	106.6(3)
C(29)-C(27)-C(30)	110.6(3)
C(28)-C(27)-C(30)	106.7(3)
C(29)-C(27)-C(26)	110.1(3)
C(28)-C(27)-C(26)	112.1(3)
C(30)-C(27)-C(26)	110.6(3)
C(27)-C(28)-H(28A)	109.5(2)

C(27)-C(28)-H(28B)	109.5(2)
H(28A)-C(28)-H(28B)	109.5
C(27)-C(28)-H(28C)	109.5(2)
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(27)-C(29)-H(29A)	109.5(2)
C(27)-C(29)-H(29B)	109.5(2)
H(29A)-C(29)-H(29B)	109.5
C(27)-C(29)-H(29C)	109.5(2)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(27)-C(30)-H(30A)	109.5(2)
C(27)-C(30)-H(30B)	109.5(2)
H(30A)-C(30)-H(30B)	109.5
C(27)-C(30)-H(30C)	109.5(2)
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
C(34)-C(31)-C(32)	107.0(3)
C(34)-C(31)-C(22)	112.6(3)
C(32)-C(31)-C(22)	110.2(3)
C(34)-C(31)-C(33)	106.1(3)
C(32)-C(31)-C(33)	111.2(3)
C(22)-C(31)-C(33)	109.6(3)
C(31)-C(32)-H(32A)	109.5(2)
C(31)-C(32)-H(32B)	109.5(2)
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5(2)
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(31)-C(33)-H(33A)	109.5(2)
C(31)-C(33)-H(33B)	109.5(2)
H(33A)-C(33)-H(33B)	109.5
C(31)-C(33)-H(33C)	109.5(2)
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(31)-C(34)-H(34A)	109.5(2)
C(31)-C(34)-H(34B)	109.5(2)
H(34A)-C(34)-H(34B)	109.5
C(31)-C(34)-H(34C)	109.5(2)
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(24)-C(20)-H(20A)	109.5(2)
C(24)-C(20)-H(20B)	109.5(2)
H(20A)-C(20)-H(20B)	109.5
C(24)-C(20)-H(20C)	109.5(2)
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
P(1)-N(1)-Sr(1)	149.6(2)
P(1)-N(1)-H(1)	105.18(11)
Sr(1)-N(1)-H(1)	105.18(7)
N(1)-P(1)-C(61)	109.0(2)
N(1)-P(1)-C(51)	112.6(2)
C(61)-P(1)-C(51)	107.1(2)
N(1)-P(1)-C(41)	114.9(2)
C(61)-P(1)-C(41)	105.0(2)
C(51)-P(1)-C(41)	107.8(2)
C(42)-C(41)-C(46)	120.0(4)
C(42)-C(41)-P(1)	120.9(4)
C(46)-C(41)-P(1)	118.7(3)
C(41)-C(42)-C(43)	119.6(5)
C(41)-C(42)-H(42)	120.2(3)
C(43)-C(42)-H(42)	120.2(3)
C(44)-C(43)-C(42)	120.3(5)
C(44)-C(43)-H(43)	119.8(3)
C(42)-C(43)-H(43)	119.8(3)
C(45)-C(44)-C(43)	120.3(5)
C(45)-C(44)-H(44)	119.9(3)
C(43)-C(44)-H(44)	119.9(3)
C(44)-C(45)-C(46)	120.2(5)
C(44)-C(45)-H(45)	119.9(3)
C(46)-C(45)-H(45)	119.9(3)
C(41)-C(46)-C(45)	119.5(4)
C(41)-C(46)-H(46)	120.2(2)
C(45)-C(46)-H(46)	120.2(3)
C(52)-C(51)-C(56)	119.1(3)
C(52)-C(51)-P(1)	124.3(3)
C(56)-C(51)-P(1)	116.5(3)
C(53)-C(52)-C(51)	119.8(4)
C(53)-C(52)-H(52)	120.1(2)
C(51)-C(52)-H(52)	120.1(2)
C(54)-C(53)-C(52)	120.2(4)
C(54)-C(53)-H(53)	119.9(2)
C(52)-C(53)-H(53)	119.9(3)
C(55)-C(54)-C(53)	120.5(4)
C(55)-C(54)-H(54)	119.8(3)
C(53)-C(54)-H(54)	119.8(2)
C(54)-C(55)-C(56)	120.0(4)
C(54)-C(55)-H(55)	120.0(3)
C(56)-C(55)-H(55)	120.0(2)
C(55)-C(56)-C(51)	120.4(4)
C(55)-C(56)-H(56)	119.8(2)
C(51)-C(56)-H(56)	119.8(2)
C(66)-C(61)-C(62)	118.9(3)
C(66)-C(61)-P(1)	120.3(3)
C(62)-C(61)-P(1)	120.7(3)
C(63)-C(62)-C(61)	119.5(3)
C(63)-C(62)-H(62)	120.2(2)
C(61)-C(62)-H(62)	120.2(2)
C(64)-C(63)-C(62)	120.8(4)
C(64)-C(63)-H(63)	119.6(2)
C(62)-C(63)-H(63)	119.6(2)
C(63)-C(64)-C(65)	119.7(3)
C(63)-C(64)-H(64)	120.2(2)
C(65)-C(64)-H(64)	120.2(2)
C(66)-C(65)-C(64)	119.6(3)
C(66)-C(65)-H(65)	120.2(2)
C(64)-C(65)-H(65)	120.2(2)
C(65)-C(66)-C(61)	121.4(3)
C(65)-C(66)-H(66)	119.3(2)
C(61)-C(66)-H(66)	119.3(2)
P(2)-N(2)-Sr(1)	142.7(2)
P(2)-N(2)-H(2)	108.65(11)
Sr(1)-N(2)-H(2)	108.64(6)
N(2)-P(2)-C(91)	108.8(2)
N(2)-P(2)-C(81)	114.9(2)
C(91)-P(2)-C(81)	106.2(2)
N(2)-P(2)-C(71)	115.2(2)
C(91)-P(2)-C(71)	105.8(2)
C(81)-P(2)-C(71)	105.2(2)
C(76)-C(71)-C(72)	118.3(4)
C(76)-C(71)-P(2)	123.3(3)
C(72)-C(71)-P(2)	118.3(3)
C(71)-C(72)-C(73)	120.5(4)
C(71)-C(72)-H(72)	119.7(2)
C(73)-C(72)-H(72)	119.7(3)
C(74)-C(73)-C(72)	120.3(4)
C(74)-C(73)-H(73)	119.8(3)
C(72)-C(73)-H(73)	119.8(3)
C(75)-C(74)-C(73)	119.2(4)
C(75)-C(74)-H(74)	120.4(3)
C(73)-C(74)-H(74)	120.4(3)
C(74)-C(75)-C(76)	120.4(4)
C(74)-C(75)-H(75)	119.8(3)
C(76)-C(75)-H(75)	119.8(3)
C(71)-C(76)-C(75)	121.0(4)
C(71)-C(76)-H(76)	119.5(2)
C(75)-C(76)-H(76)	119.5(3)
C(82)-C(81)-C(86)	118.3(3)
C(82)-C(81)-P(2)	124.0(3)
C(86)-C(81)-P(2)	117.6(3)
C(81)-C(82)-C(83)	121.5(5)
C(81)-C(82)-H(82)	119.3(2)
C(83)-C(82)-H(82)	119.3(3)
C(84)-C(83)-C(82)	118.9(5)
C(84)-C(83)-H(83)	120.6(3)
C(82)-C(83)-H(83)	120.6(3)
C(85)-C(84)-C(83)	121.9(5)
C(85)-C(84)-H(84)	119.0(3)

C(83)-C(84)-H(84)	119.0(3)	C(95)-C(94)-C(93)	120.5(4)
C(84)-C(85)-C(86)	118.7(5)	C(95)-C(94)-H(94)	119.8(2)
C(84)-C(85)-H(85)	120.7(3)	C(93)-C(94)-H(94)	119.8(2)
C(86)-C(85)-H(85)	120.7(3)	C(94)-C(95)-C(96)	119.4(4)
C(81)-C(86)-C(85)	120.7(4)	C(94)-C(95)-H(95)	120.3(2)
C(81)-C(86)-H(86)	119.6(2)	C(96)-C(95)-H(95)	120.3(2)
C(85)-C(86)-H(86)	119.6(3)	C(91)-C(96)-C(95)	120.9(3)
C(96)-C(91)-C(92)	118.7(3)	C(91)-C(96)-H(96)	119.6(2)
C(96)-C(91)-P(2)	122.1(3)	C(95)-C(96)-H(96)	119.6(2)
C(92)-C(91)-P(2)	119.2(3)		
C(93)-C(92)-C(91)	120.5(3)		
C(93)-C(92)-H(92)	119.7(2)		
C(91)-C(92)-H(92)	119.7(2)		
C(94)-C(93)-C(92)	120.0(3)		
C(94)-C(93)-H(93)	120.0(2)		
C(92)-C(93)-H(93)	120.0(2)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 7. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Sr(1)	23(1)	22(1)	25(1)	-1(1)	3(1)	-1(1)
O(1)	24(1)	30(1)	32(1)	4(1)	1(1)	-7(1)
C(1)	28(2)	27(2)	23(2)	-5(2)	6(2)	-3(2)
C(2)	25(2)	25(2)	29(2)	-1(2)	1(2)	0(1)
C(3)	27(2)	28(2)	45(2)	-2(2)	1(2)	1(2)
C(4)	34(2)	24(2)	46(2)	1(2)	11(2)	-3(2)
C(5)	38(2)	26(2)	38(2)	6(2)	5(2)	-4(2)
C(6)	31(2)	27(2)	31(2)	0(2)	-1(2)	-4(2)
C(7)	37(2)	32(2)	37(2)	10(2)	-6(2)	-6(2)
C(8)	52(3)	61(3)	41(2)	7(2)	-11(2)	-14(2)
C(9)	52(3)	54(3)	66(3)	32(2)	-10(2)	-3(2)
C(10)	33(2)	39(2)	53(2)	4(2)	-1(2)	5(2)
C(11)	25(2)	28(2)	34(2)	0(2)	-3(2)	1(2)
C(12)	38(3)	37(2)	61(3)	13(2)	-9(2)	-3(2)
C(13)	40(2)	35(2)	28(2)	0(2)	-4(2)	0(2)
C(14)	32(2)	28(2)	44(2)	0(2)	7(2)	2(1)
C(0)	36(3)	41(2)	91(4)	19(2)	17(2)	-8(2)
O(2)	22(1)	30(1)	27(1)	-3(1)	-1(1)	5(1)
C(21)	23(2)	21(2)	27(2)	2(2)	4(2)	1(1)
C(22)	22(2)	27(2)	28(2)	5(1)	0(1)	-1(1)
C(23)	21(2)	29(2)	40(2)	5(2)	-5(2)	3(2)
C(24)	21(2)	26(2)	50(2)	-1(2)	4(2)	1(2)
C(25)	30(2)	24(2)	34(2)	-3(2)	3(2)	-1(2)
C(26)	22(2)	20(2)	28(2)	0(2)	2(2)	0(1)
C(27)	26(2)	28(2)	31(2)	-8(2)	-3(2)	7(2)
C(28)	44(3)	48(2)	35(2)	-9(2)	-7(2)	10(2)
C(29)	29(2)	39(2)	40(2)	-13(2)	-7(2)	1(2)
C(30)	54(3)	36(2)	31(2)	4(2)	-5(2)	12(2)
C(31)	26(2)	34(2)	31(2)	-2(2)	-6(2)	1(2)
C(32)	39(2)	51(2)	28(2)	4(2)	-4(2)	-3(2)
C(33)	30(2)	34(2)	37(2)	-4(2)	-4(2)	-4(2)
C(34)	35(2)	54(3)	38(2)	2(2)	-14(2)	1(2)
C(20)	31(2)	45(2)	67(3)	-10(2)	3(2)	11(2)
N(1)	28(2)	42(2)	38(2)	-14(1)	1(1)	0(1)
P(1)	30(1)	35(1)	28(1)	-8(1)	-4(1)	6(1)
C(41)	51(3)	35(2)	35(2)	-10(2)	-7(2)	22(2)
C(42)	61(3)	71(3)	42(3)	-6(2)	-4(2)	36(3)
C(43)	103(5)	89(4)	43(3)	-7(3)	-18(3)	67(4)
C(44)	148(6)	51(3)	48(3)	7(2)	9(4)	56(4)
C(45)	105(4)	35(2)	53(3)	2(2)	9(3)	10(3)
C(46)	68(3)	33(2)	40(2)	0(2)	-4(2)	8(2)
C(51)	23(2)	40(2)	36(2)	-11(2)	-1(2)	6(2)
C(52)	33(2)	53(3)	36(2)	-18(2)	-2(2)	2(2)
C(53)	41(3)	65(3)	50(3)	-31(2)	-6(2)	-3(2)
C(54)	38(3)	54(3)	61(3)	-22(2)	0(2)	-6(2)
C(55)	44(3)	52(3)	51(3)	-8(2)	3(2)	-10(2)
C(56)	40(3)	44(2)	38(2)	-10(2)	-7(2)	-2(2)

C(61)	31(2)	25(2)	23(2)	-2(2)	1(2)	0(1)
C(62)	40(2)	43(2)	33(2)	-12(2)	-3(2)	13(2)
C(63)	37(2)	56(3)	45(3)	-9(2)	-13(2)	5(2)
C(64)	26(2)	36(2)	51(2)	-2(2)	-1(2)	6(2)
C(65)	36(2)	41(2)	36(2)	0(2)	7(2)	7(2)
C(66)	32(2)	40(2)	29(2)	1(2)	-1(2)	3(2)
N(2)	25(2)	38(2)	41(2)	12(2)	-3(1)	-2(1)
P(2)	25(1)	26(1)	28(1)	1(1)	0(1)	2(1)
C(71)	34(2)	31(2)	29(2)	-1(2)	-3(2)	4(2)
C(72)	45(3)	115(4)	53(3)	-31(3)	4(2)	-34(3)
C(73)	65(4)	148(5)	57(3)	-48(4)	-4(3)	-21(4)
C(74)	65(3)	67(3)	34(2)	-11(2)	0(2)	4(2)
C(75)	64(3)	81(3)	40(3)	-9(2)	20(2)	-17(3)
C(76)	49(3)	63(3)	38(2)	-6(2)	2(2)	-24(2)
C(81)	27(2)	31(2)	39(2)	-10(2)	-4(2)	2(2)
C(82)	46(3)	39(2)	64(3)	12(2)	-4(2)	7(2)
C(83)	59(3)	41(3)	128(5)	14(3)	-22(4)	8(2)
C(84)	39(3)	32(3)	140(6)	-22(3)	-20(3)	6(2)
C(85)	45(3)	96(4)	86(4)	-51(4)	-2(3)	17(3)
C(86)	48(3)	74(3)	54(3)	-7(2)	-2(2)	24(2)
C(91)	27(2)	26(2)	26(2)	5(2)	0(2)	0(2)
C(92)	25(2)	29(2)	46(2)	0(2)	0(2)	-1(2)
C(93)	25(2)	45(2)	60(3)	12(2)	-7(2)	1(2)
C(94)	29(2)	55(3)	46(2)	6(2)	-11(2)	-14(2)
C(95)	39(2)	40(2)	38(2)	0(2)	-5(2)	-12(2)
C(96)	33(2)	30(2)	36(2)	2(2)	-4(2)	2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 7.

	x	y	z	U(eq)
H(3)	6158(1)	3071(3)	7344(2)	40
H(5)	5214(1)	4798(3)	6103(2)	40
H(8A)	4074(10)	2182(6)	6176(4)	77
H(8B)	4315(6)	2806(15)	5533(7)	77
H(8C)	3730(4)	3074(11)	5759(11)	77
H(9A)	4405(10)	5615(6)	6203(5)	86
H(9B)	3914(2)	5135(14)	5797(12)	86
H(9C)	4491(9)	4864(9)	5540(7)	86
H(10A)	4028(5)	4725(13)	7231(7)	63
H(10B)	3874(7)	3399(7)	7232(7)	63
H(10C)	3555(3)	4283(18)	6778(2)	63
H(12A)	6310(2)	1479(20)	7837(3)	68
H(12B)	6105(3)	1003(14)	8542(9)	68
H(12C)	6130(3)	2352(8)	8410(11)	68
H(13A)	5208(5)	2472(9)	8745(6)	52
H(13B)	5177(6)	1116(10)	8844(5)	52
H(13C)	4772(2)	1749(18)	8360(2)	52
H(14A)	5686(5)	243(8)	7271(8)	52
H(14B)	5076(3)	328(7)	7425(10)	52
H(14C)	5461(8)	-205(3)	7972(3)	52
H(0A)	6110(2)	5384(12)	6197(13)	84
H(0B)	6404(7)	4203(11)	6054(10)	84
H(0C)	6471(5)	4826(21)	6763(3)	84
H(23)	1626(1)	3418(3)	7592(2)	36
H(25)	2384(1)	4031(3)	9312(2)	35
H(28A)	3514(5)	3364(15)	10105(6)	63
H(28B)	2892(5)	3293(14)	10064(6)	63
H(28C)	3195(9)	4312(3)	9697(2)	63
H(29A)	4051(1)	2974(18)	9104(5)	54
H(29B)	3735(4)	3974(5)	8737(10)	54
H(29C)	3780(5)	2741(15)	8394(6)	54
H(30A)	3607(5)	1419(5)	9688(10)	61
H(30B)	3321(9)	1014(3)	9015(2)	61
H(30C)	2987(4)	1305(7)	9670(10)	61
H(32A)	2649(6)	1739(14)	6097(5)	59
H(32B)	3012(2)	2091(18)	6716(4)	59
H(32C)	2633(5)	3017(6)	6383(9)	59

H(33A)	1942(5)	354(4)	7405(9)	50
H(33B)	2564(4)	357(4)	7368(9)	50
H(33C)	2229(8)	148(3)	6702(2)	50
H(34A)	1429(1)	1887(18)	6938(5)	63
H(34B)	1698(4)	1684(15)	6223(6)	63
H(34C)	1692(4)	2935(5)	6548(10)	63
H(20A)	1190(4)	4502(17)	8419(8)	71
H(20B)	1603(4)	5410(3)	8695(13)	71
H(20C)	1394(7)	4415(15)	9176(5)	71
H(1)	3223(1)	-512(2)	6580(1)	43
H(42)	2912(2)	-295(4)	4889(2)	69
H(43)	2913(2)	996(5)	3984(2)	94
H(44)	3681(3)	1873(4)	3651(3)	99
H(45)	4446(2)	1556(4)	4243(2)	77
H(46)	4456(2)	269(3)	5150(2)	57
H(52)	3274(1)	-2316(3)	4663(2)	49
H(53)	2704(2)	-3865(4)	4556(2)	62
H(54)	2389(2)	-4786(4)	5507(2)	61
H(55)	2621(2)	-4155(3)	6568(2)	59
H(56)	3155(2)	-2558(3)	6689(2)	49
H(62)	4458(2)	-1325(3)	6889(2)	47
H(63)	5312(2)	-2011(3)	6922(2)	55
H(64)	5705(1)	-2730(3)	5959(2)	45
H(65)	5234(2)	-2815(3)	4954(2)	45
H(66)	4387(1)	-2134(3)	4918(2)	40
H(2)	4527(1)	-663(2)	8383(1)	42
H(72)	4569(2)	-660(4)	9749(2)	85
H(73)	4471(2)	-315(5)	10891(2)	108
H(74)	3733(2)	-962(4)	11454(2)	66
H(75)	3086(2)	-1883(4)	10861(2)	74
H(76)	3200(2)	-2301(3)	9729(2)	60
H(82)	4410(2)	-3546(3)	9694(2)	60
H(83)	4971(2)	-5099(4)	9607(3)	91
H(84)	5325(2)	-5561(4)	8573(4)	85
H(85)	5148(2)	-4484(5)	7633(3)	91
H(86)	4606(2)	-2870(4)	7731(2)	70
H(92)	3042(1)	-802(3)	8428(2)	40
H(93)	2228(1)	-1449(3)	8074(2)	52
H(94)	2101(2)	-3378(3)	7835(2)	52
H(95)	2781(2)	-4680(3)	7965(2)	47
H(96)	3601(1)	-4030(3)	8313(2)	40

8) Barium bis(2,6-ditbutyl-4-methylphenoxy)bis(triphenylaminophosphonium)

Table 1. Crystal data and structure refinement for 8.

Identification code	8 (99srv008)
Empirical formula	C66 H78 Ba N2 O2 P2
Formula weight	1130.58
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 12.59520(10) A alpha = 80.2270(10) deg. b = 14.8787(2) A beta = 71.0410(10) deg. c = 18.6550(3) A gamma = 69.5260(10) deg.
Volume, Z	3091.23(7) A^3, 2
Density (calculated)	1.215 Mg/m^3
Absorption coefficient	0.736 mm^-1
F(000)	1180
Crystal size	0.5 x 0.3 x 0.1 mm
Theta range for data collection	1.16 to 27.51 deg.
Limiting indices	-12<=h<=16, -18<=k<=19, -23<=l<=24
Reflections collected	22792
Independent reflections	14074 [R(int) = 0.0263]
Absorption correction	Psi scan
Max. and min. transmission	0.664 and 0.753
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	14038 / 0 / 666
Goodness-of-fit on F^2	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0345, wR2 = 0.0650
R indices (all data)	R1 = 0.0487, wR2 = 0.0744
Extinction coefficient	not refined
Largest diff. peak and hole	0.441 and -0.403 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 8. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ba(1)	8314(1)	7564(1)	7425(1)	24(1)
O(1)	10172(1)	6250(1)	7278(1)	28(1)
C(1)	11172(2)	5538(2)	7232(1)	24(1)
C(2)	11256(2)	4774(2)	7818(1)	24(1)
C(3)	12336(2)	4044(2)	7750(1)	29(1)
C(4)	13331(2)	4012(2)	7136(2)	34(1)
C(5)	13233(2)	4747(2)	6566(2)	32(1)
C(6)	12192(2)	5507(2)	6591(1)	26(1)
C(7)	12141(2)	6302(2)	5938(2)	33(1)
C(8)	13325(2)	6118(2)	5303(2)	50(1)
C(9)	11191(2)	6346(2)	5574(2)	43(1)
C(10)	11873(3)	7283(2)	6244(2)	44(1)
C(11)	10156(2)	4751(2)	8491(1)	26(1)
C(12)	9737(2)	5626(2)	8966(1)	34(1)
C(13)	10403(2)	3842(2)	9029(1)	36(1)
C(14)	9139(2)	4748(2)	8200(1)	31(1)
C(0)	14488(2)	3199(2)	7070(2)	58(1)
O(2)	8508(1)	9028(1)	7688(1)	29(1)
C(21)	8546(2)	9811(2)	7916(1)	25(1)
C(22)	8814(2)	9782(2)	8608(1)	28(1)
C(23)	8749(2)	10637(2)	8860(1)	33(1)
C(24)	8451(2)	11518(2)	8456(2)	35(1)
C(23)	8254(2)	11529(2)	7765(2)	33(1)
C(26)	8313(2)	10704(2)	7470(1)	26(1)
C(27)	8105(2)	10748(2)	6695(1)	32(1)
C(28)	6965(2)	10501(2)	6812(2)	43(1)
C(29)	7938(3)	11755(2)	6281(2)	47(1)
C(30)	9170(3)	10042(2)	6166(2)	42(1)
C(31)	9161(2)	8820(2)	9074(2)	36(1)
C(32)	8107(3)	8424(2)	9384(2)	43(1)
C(33)	10244(2)	8087(2)	8585(2)	42(1)
C(34)	9502(3)	8928(2)	9771(2)	57(1)
C(20)	8312(3)	12434(2)	8782(2)	57(1)
N(1)	7598(2)	8091(2)	6147(1)	39(1)
P(1)	7131(1)	7632(1)	5656(1)	29(1)
C(41)	5642(2)	8281(2)	5605(2)	34(1)
C(42)	5206(3)	8108(2)	5064(2)	49(1)
C(43)	4042(3)	8588(3)	5069(2)	64(1)
C(44)	3322(3)	9243(3)	5595(3)	82(1)
C(45)	3744(4)	9435(3)	6115(3)	102(2)
C(46)	4908(2)	8952(1)	6135(1)	70(1)
C(51)	7113(2)	6443(1)	6077(1)	33(1)
C(52)	8182(2)	5753(1)	6107(1)	49(1)
C(53)	8191(3)	4836(2)	6443(2)	63(1)
C(54)	7152(3)	4621(2)	6739(2)	61(1)
C(55)	6094(3)	5301(2)	6706(2)	60(1)
C(56)	6073(3)	6211(2)	6378(2)	45(1)
C(61)	8008(2)	7488(2)	4677(1)	30(1)
C(62)	8093(2)	6752(2)	4266(2)	39(1)
C(63)	8692(3)	6722(2)	3498(2)	45(1)
C(64)	9201(2)	7427(2)	3134(2)	45(1)
C(65)	9132(2)	8154(2)	3538(2)	43(1)
C(66)	8551(2)	8176(2)	4308(2)	37(1)
N(2)	6165(2)	7293(2)	8082(1)	44(1)
P(2)	4887(1)	7670(1)	8658(1)	33(1)
C(71)	5038(2)	7821(2)	9553(2)	33(1)
C(72)	5987(3)	7174(2)	9792(2)	49(1)
C(73)	6111(3)	7269(3)	10487(2)	65(1)
C(74)	5300(3)	7999(3)	10939(2)	62(1)
C(75)	4362(3)	8632(3)	10712(2)	55(1)
C(76)	4234(2)	8541(2)	10023(2)	43(1)
C(81)	3974(2)	8845(2)	8391(1)	35(1)
C(82)	4525(3)	9538(2)	8060(2)	54(1)
C(83)	3867(3)	10459(3)	7864(2)	68(1)

C(84)	2662(3)	10689(2)	7986(2)	60(1)
C(85)	2108(3)	10009(2)	8316(2)	53(1)
C(86)	2760(2)	9093(2)	8519(2)	44(1)
C(91)	4037(2)	6857(2)	8814(2)	37(1)
C(92)	3894(2)	6257(2)	9469(2)	46(1)
C(93)	3352(3)	5557(2)	9538(2)	60(1)
C(94)	2959(3)	5471(2)	8950(2)	60(1)
C(95)	3089(3)	6065(2)	8301(2)	56(1)
C(96)	3622(3)	6761(2)	8230(2)	48(1)

Table 3. Bond lengths [A] and angles [deg] for 8.

Ba(1)-O(2)	2.421(2)
Ba(1)-O(1)	2.437(2)
Ba(1)-N(1)	2.718(2)
Ba(1)-N(2)	2.726(2)
O(1)-C(1)	1.321(3)
C(1)-C(6)	1.436(3)
C(1)-C(2)	1.438(3)
C(2)-C(3)	1.395(3)
C(2)-C(11)	1.544(3)
C(3)-C(4)	1.388(3)
C(3)-H(3A)	0.95
C(4)-C(5)	1.393(3)
C(4)-C(0)	1.518(3)
C(5)-C(6)	1.392(3)
C(5)-H(5A)	0.95
C(6)-C(7)	1.547(3)
C(7)-C(9)	1.535(4)
C(7)-C(8)	1.543(3)
C(7)-C(10)	1.544(4)
C(8)-H(8A)	0.98
C(8)-H(8B)	0.98
C(8)-H(8C)	0.98
C(9)-H(9A)	0.98
C(9)-H(9B)	0.98
C(9)-H(9C)	0.98
C(10)-H(10A)	0.98
C(10)-H(10B)	0.98
C(10)-H(10C)	0.98
C(11)-C(12)	1.537(3)
C(11)-C(13)	1.543(3)
C(11)-C(14)	1.547(3)
C(12)-H(12A)	0.98
C(12)-H(12B)	0.98
C(12)-H(12C)	0.98
C(13)-H(13A)	0.98
C(13)-H(13B)	0.98
C(13)-H(13C)	0.98
C(14)-H(14A)	0.98
C(14)-H(14B)	0.98
C(14)-H(14C)	0.98
C(0)-H(0A)	0.98
C(0)-H(0B)	0.98
C(0)-H(0C)	0.98
O(2)-C(21)	1.326(3)
C(21)-C(22)	1.428(3)
C(21)-C(26)	1.433(3)
C(22)-C(23)	1.396(3)
C(22)-C(31)	1.543(3)
C(23)-C(24)	1.390(4)
C(23)-H(23A)	0.95
C(24)-C(23)	1.385(4)
C(24)-C(20)	1.518(3)
C(23)-C(26)	1.399(3)
C(23)-H(23B)	0.95
C(26)-C(27)	1.539(3)
C(27)-C(29)	1.542(3)
C(27)-C(28)	1.542(3)
C(27)-C(30)	1.544(4)
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98
C(29)-H(29A)	0.98
C(29)-H(29B)	0.98
C(29)-H(29C)	0.98
C(30)-H(30A)	0.98
C(30)-H(30B)	0.98
C(30)-H(30C)	0.98
C(31)-C(33)	1.540(4)
C(31)-C(32)	1.541(4)
C(31)-C(34)	1.545(4)
C(32)-H(32A)	0.98
C(32)-H(32B)	0.98
C(32)-H(32C)	0.98
C(33)-H(33A)	0.98
C(33)-H(33B)	0.98
C(33)-H(33C)	0.98
C(34)-H(34A)	0.98
C(34)-H(34B)	0.98
C(34)-H(34C)	0.98
C(20)-H(20A)	0.98
C(20)-H(20B)	0.98
C(20)-H(20C)	0.98
N(1)-P(1)	1.578(2)
N(1)-H(1A)	0.88
P(1)-C(61)	1.805(2)
P(1)-C(41)	1.811(3)
P(1)-C(51)	1.814(2)
C(41)-C(46)	1.387(3)
C(41)-C(42)	1.389(4)
C(42)-C(43)	1.386(4)
C(42)-H(42A)	0.95
C(43)-C(44)	1.365(6)
C(43)-H(43A)	0.95
C(44)-C(45)	1.356(6)
C(44)-H(44A)	0.95
C(45)-C(46)	1.399(4)
C(45)-H(45A)	0.95
C(46)-H(46A)	0.95
C(51)-C(56)	1.387(3)
C(51)-C(52)	1.39
C(52)-C(53)	1.398(4)
C(52)-H(52A)	0.95
C(53)-C(54)	1.373(5)
C(53)-H(53A)	0.95
C(54)-C(55)	1.375(5)
C(54)-H(54A)	0.95
C(55)-C(56)	1.383(4)
C(55)-H(55A)	0.95
C(56)-H(56A)	0.95
C(61)-C(66)	1.391(3)
C(61)-C(62)	1.395(3)
C(62)-C(63)	1.385(4)
C(62)-H(62A)	0.95
C(63)-C(64)	1.385(4)
C(63)-H(63A)	0.95
C(64)-C(65)	1.383(4)
C(64)-H(64A)	0.95
C(65)-C(66)	1.385(4)

C(65)-H(65A)	0.95
C(66)-H(66A)	0.95
N(2)-P(2)	1.584(2)
N(2)-H(2A)	0.88
P(2)-C(71)	1.800(3)
P(2)-C(91)	1.810(3)
P(2)-C(81)	1.815(3)
C(71)-C(76)	1.388(4)
C(71)-C(72)	1.396(4)
C(72)-C(73)	1.390(5)
C(72)-H(72A)	0.95
C(73)-C(74)	1.382(5)
C(73)-H(73A)	0.95
C(74)-C(75)	1.369(5)
C(74)-H(74A)	0.95
C(75)-C(76)	1.380(4)
C(75)-H(75A)	0.95
C(76)-H(76A)	0.95
C(81)-C(86)	1.388(4)
C(81)-C(82)	1.390(4)
C(82)-C(83)	1.389(5)
C(82)-H(82A)	0.95
C(83)-C(84)	1.382(5)
C(83)-H(83A)	0.95
C(84)-C(85)	1.376(4)
C(84)-H(84A)	0.95
C(85)-C(86)	1.386(4)
C(85)-H(85A)	0.95
C(86)-H(86A)	0.95
C(91)-C(92)	1.387(4)
C(91)-C(96)	1.399(4)
C(92)-C(93)	1.401(4)
C(92)-H(92A)	0.95
C(93)-C(94)	1.382(5)
C(93)-H(93A)	0.95
C(94)-C(95)	1.371(5)
C(94)-H(94A)	0.95
C(95)-C(96)	1.386(4)
C(95)-H(95A)	0.95
C(96)-H(96A)	0.95

O(2)-Ba(1)-O(1)	111.06(5)
O(2)-Ba(1)-N(1)	102.76(6)
O(1)-Ba(1)-N(1)	114.12(6)
O(2)-Ba(1)-N(2)	116.58(6)
O(1)-Ba(1)-N(2)	122.56(6)
N(1)-Ba(1)-N(2)	84.95(7)
C(1)-O(1)-Ba(1)	177.3(2)
O(1)-C(1)-C(6)	120.6(2)
O(1)-C(1)-C(2)	120.6(2)
C(6)-C(1)-C(2)	118.8(2)
C(3)-C(2)-C(1)	118.5(2)
C(3)-C(2)-C(11)	121.2(2)
C(1)-C(2)-C(11)	120.2(2)
C(4)-C(3)-C(2)	123.1(2)
C(4)-C(3)-H(3A)	118.44(14)
C(2)-C(3)-H(3A)	118.44(13)
C(3)-C(4)-C(5)	117.8(2)
C(3)-C(4)-C(0)	121.9(2)
C(5)-C(4)-C(0)	120.3(2)
C(6)-C(5)-C(4)	122.9(2)
C(6)-C(5)-H(5A)	118.55(14)
C(4)-C(5)-H(5A)	118.55(14)
C(5)-C(6)-C(1)	118.8(2)
C(5)-C(6)-C(7)	120.4(2)
C(1)-C(6)-C(7)	120.8(2)
C(9)-C(7)-C(8)	107.2(2)
C(9)-C(7)-C(10)	110.2(2)
C(8)-C(7)-C(10)	107.0(2)
C(9)-C(7)-C(6)	110.3(2)
C(8)-C(7)-C(6)	112.2(2)
C(10)-C(7)-C(6)	109.9(2)
C(7)-C(8)-H(8A)	109.47(14)
C(7)-C(8)-H(8B)	109.5(2)
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5(2)
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(7)-C(9)-H(9A)	109.47(14)
C(7)-C(9)-H(9B)	109.5(2)
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.47(14)
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(7)-C(10)-H(10A)	109.47(14)
C(7)-C(10)-H(10B)	109.47(14)
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.47(14)
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(12)-C(11)-C(13)	107.4(2)
C(12)-C(11)-C(2)	110.6(2)
C(13)-C(11)-C(2)	112.4(2)
C(12)-C(11)-C(14)	109.4(2)
C(13)-C(11)-C(14)	106.6(2)
C(2)-C(11)-C(14)	110.4(2)
C(11)-C(12)-H(12A)	109.47(13)
C(11)-C(12)-H(12B)	109.47(13)
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.47(13)
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.47(13)
C(11)-C(13)-H(13B)	109.47(13)
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.47(13)
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.47(12)
C(11)-C(14)-H(14B)	109.47(13)
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.47(13)
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(4)-C(0)-H(0A)	109.5(2)
C(4)-C(0)-H(0B)	109.5(2)
H(0A)-C(0)-H(0B)	109.5
C(4)-C(0)-H(0C)	109.5(2)
H(0A)-C(0)-H(0C)	109.5
H(0B)-C(0)-H(0C)	109.5
C(21)-O(2)-Ba(1)	173.3(2)
O(2)-C(21)-C(22)	120.8(2)
O(2)-C(21)-C(26)	120.4(2)
C(22)-C(21)-C(26)	118.8(2)
C(23)-C(22)-C(21)	119.0(2)
C(23)-C(22)-C(31)	120.6(2)
C(21)-C(22)-C(31)	120.4(2)
C(24)-C(23)-C(22)	122.6(2)
C(24)-C(23)-H(23A)	118.70(14)
C(22)-C(23)-H(23A)	118.7(2)
C(23)-C(24)-C(23)	117.8(2)
C(23)-C(24)-C(20)	121.8(3)
C(23)-C(24)-C(20)	120.4(3)
C(24)-C(23)-C(26)	123.1(2)
C(24)-C(23)-H(23B)	118.45(14)
C(26)-C(23)-H(23B)	118.4(2)
C(23)-C(26)-C(21)	118.4(2)
C(23)-C(26)-C(27)	121.3(2)
C(21)-C(26)-C(27)	120.3(2)
C(26)-C(27)-C(29)	112.7(2)
C(26)-C(27)-C(28)	109.3(2)
C(29)-C(27)-C(28)	107.0(2)
C(26)-C(27)-C(30)	110.5(2)
C(29)-C(27)-C(30)	107.0(2)
C(28)-C(27)-C(30)	110.2(2)
C(27)-C(28)-H(28A)	109.5(2)
C(27)-C(28)-H(28B)	109.47(13)
H(28A)-C(28)-H(28B)	109.5

C(27)-C(28)-H(28C)	109.5(2)
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(27)-C(29)-H(29A)	109.5(2)
C(27)-C(29)-H(29B)	109.5(2)
H(29A)-C(29)-H(29B)	109.5
C(27)-C(29)-H(29C)	109.47(14)
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(27)-C(30)-H(30A)	109.47(14)
C(27)-C(30)-H(30B)	109.47(14)
H(30A)-C(30)-H(30B)	109.5
C(27)-C(30)-H(30C)	109.47(14)
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
C(33)-C(31)-C(32)	110.7(2)
C(33)-C(31)-C(22)	110.5(2)
C(32)-C(31)-C(22)	110.2(2)
C(33)-C(31)-C(34)	106.6(2)
C(32)-C(31)-C(34)	106.4(2)
C(22)-C(31)-C(34)	112.4(2)
C(31)-C(32)-H(32A)	109.47(14)
C(31)-C(32)-H(32B)	109.5(2)
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5(2)
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(31)-C(33)-H(33A)	109.47(14)
C(31)-C(33)-H(33B)	109.47(14)
H(33A)-C(33)-H(33B)	109.5
C(31)-C(33)-H(33C)	109.47(14)
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(31)-C(34)-H(34A)	109.5(2)
C(31)-C(34)-H(34B)	109.5(2)
H(34A)-C(34)-H(34B)	109.5
C(31)-C(34)-H(34C)	109.5(2)
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(24)-C(20)-H(20A)	109.5(2)
C(24)-C(20)-H(20B)	109.5(2)
H(20A)-C(20)-H(20B)	109.5
C(24)-C(20)-H(20C)	109.5(2)
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
P(1)-N(1)-Ba(1)	136.49(12)
P(1)-N(1)-H(1A)	111.76(8)
Ba(1)-N(1)-H(1A)	111.76(5)
N(1)-P(1)-C(61)	115.25(12)
N(1)-P(1)-C(41)	115.26(12)
C(61)-P(1)-C(41)	104.30(12)
N(1)-P(1)-C(51)	108.57(10)
C(61)-P(1)-C(51)	106.76(10)
C(41)-P(1)-C(51)	106.02(10)
C(46)-C(41)-C(42)	119.3(2)
C(46)-C(41)-P(1)	118.7(2)
C(42)-C(41)-P(1)	122.0(2)
C(43)-C(42)-C(41)	120.0(3)
C(43)-C(42)-H(42A)	120.0(2)
C(41)-C(42)-H(42A)	120.0(2)
C(44)-C(43)-C(42)	120.4(4)
C(44)-C(43)-H(43A)	119.8(2)
C(42)-C(43)-H(43A)	119.8(2)
C(45)-C(44)-C(43)	120.2(3)
C(45)-C(44)-H(44A)	119.9(2)
C(43)-C(44)-H(44A)	119.9(2)
C(44)-C(45)-C(46)	120.9(4)
C(44)-C(45)-H(45A)	119.5(2)
C(46)-C(45)-H(45A)	119.5(2)
C(41)-C(46)-C(45)	119.2(3)
C(41)-C(46)-H(46A)	120.42(13)
C(45)-C(46)-H(46A)	120.4(2)
C(56)-C(51)-C(52)	119.44(14)
C(56)-C(51)-P(1)	122.2(2)
C(52)-C(51)-P(1)	118.33(6)
C(51)-C(52)-C(53)	119.5(2)
C(51)-C(52)-H(52A)	120.2
C(53)-C(52)-H(52A)	120.2(2)
C(54)-C(53)-C(52)	120.2(3)
C(54)-C(53)-H(53A)	119.9(2)
C(52)-C(53)-H(53A)	119.9(2)
C(53)-C(54)-C(55)	120.4(3)
C(53)-C(54)-H(54A)	119.8(2)
C(55)-C(54)-H(54A)	119.8(2)
C(54)-C(55)-C(56)	119.9(3)
C(54)-C(55)-H(55A)	120.0(2)
C(56)-C(55)-H(55A)	120.0(2)
C(55)-C(56)-C(51)	120.5(3)
C(55)-C(56)-H(56A)	119.8(2)
C(51)-C(56)-H(56A)	119.75(14)
C(66)-C(61)-C(62)	119.1(2)
C(66)-C(61)-P(1)	118.3(2)
C(62)-C(61)-P(1)	122.4(2)
C(63)-C(62)-C(61)	120.2(3)
C(63)-C(62)-H(62A)	119.9(2)
C(61)-C(62)-H(62A)	119.9(2)
C(64)-C(63)-C(62)	120.1(3)
C(64)-C(63)-H(63A)	120.0(2)
C(62)-C(63)-H(63A)	120.0(2)
C(65)-C(64)-C(63)	120.2(3)
C(65)-C(64)-H(64A)	119.9(2)
C(63)-C(64)-H(64A)	119.9(2)
C(64)-C(65)-C(66)	119.7(3)
C(64)-C(65)-H(65A)	120.1(2)
C(66)-C(65)-H(65A)	120.1(2)
C(65)-C(66)-C(61)	120.7(2)
C(65)-C(66)-H(66A)	119.7(2)
C(61)-C(66)-H(66A)	119.7(2)
P(2)-N(2)-Ba(1)	146.46(13)
P(2)-N(2)-H(2A)	106.77(9)
Ba(1)-N(2)-H(2A)	106.77(5)
N(2)-P(2)-C(71)	108.77(12)
N(2)-P(2)-C(91)	112.73(12)
C(71)-P(2)-C(91)	108.81(12)
N(2)-P(2)-C(81)	115.03(13)
C(71)-P(2)-C(81)	104.36(12)
C(91)-P(2)-C(81)	106.68(12)
C(76)-C(71)-C(72)	118.7(3)
C(76)-C(71)-P(2)	122.6(2)
C(72)-C(71)-P(2)	118.7(2)
C(73)-C(72)-C(71)	119.7(3)
C(73)-C(72)-H(72A)	120.2(2)
C(71)-C(72)-H(72A)	120.2(2)
C(74)-C(73)-C(72)	120.2(3)
C(74)-C(73)-H(73A)	119.9(2)
C(72)-C(73)-H(73A)	119.9(2)
C(75)-C(74)-C(73)	120.5(3)
C(75)-C(74)-H(74A)	119.7(2)
C(73)-C(74)-H(74A)	119.7(2)
C(74)-C(75)-C(76)	119.5(3)
C(74)-C(75)-H(75A)	120.3(2)
C(76)-C(75)-H(75A)	120.3(2)
C(75)-C(76)-C(71)	121.4(3)
C(75)-C(76)-H(76A)	119.3(2)
C(71)-C(76)-H(76A)	119.3(2)
C(86)-C(81)-C(82)	118.6(3)
C(86)-C(81)-P(2)	123.7(2)
C(82)-C(81)-P(2)	117.6(2)
C(83)-C(82)-C(81)	120.3(3)
C(83)-C(82)-H(82A)	119.9(2)
C(81)-C(82)-H(82A)	119.9(2)
C(84)-C(83)-C(82)	120.3(3)
C(84)-C(83)-H(83A)	119.9(2)
C(82)-C(83)-H(83A)	119.9(2)
C(85)-C(84)-C(83)	119.9(3)
C(85)-C(84)-H(84A)	120.1(2)
C(83)-C(84)-H(84A)	120.1(2)
C(84)-C(85)-C(86)	119.9(3)

C(84)-C(85)-H(85A)	120.0(2)
C(86)-C(85)-H(85A)	120.0(2)
C(85)-C(86)-C(81)	121.0(3)
C(85)-C(86)-H(86A)	119.5(2)
C(81)-C(86)-H(86A)	119.5(2)
C(92)-C(91)-C(96)	119.2(3)
C(92)-C(91)-P(2)	121.2(2)
C(96)-C(91)-P(2)	119.3(2)
C(91)-C(92)-C(93)	120.1(3)
C(91)-C(92)-H(92A)	119.9(2)
C(93)-C(92)-H(92A)	119.9(2)
C(94)-C(93)-C(92)	119.5(3)
C(94)-C(93)-H(93A)	120.2(2)
C(92)-C(93)-H(93A)	120.2(2)
C(95)-C(94)-C(93)	120.9(3)
C(95)-C(94)-H(94A)	119.6(2)
C(93)-C(94)-H(94A)	119.6(2)
C(94)-C(95)-C(96)	119.9(3)
C(94)-C(95)-H(95A)	120.0(2)
C(96)-C(95)-H(95A)	120.0(2)
C(95)-C(96)-C(91)	120.4(3)
C(95)-C(96)-H(96A)	119.8(2)
C(91)-C(96)-H(96A)	119.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 8.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ba(1)	21(1)	21(1)	33(1)	-7(1)	-10(1)	-5(1)
O(1)	24(1)	23(1)	32(1)	1(1)	-7(1)	-4(1)
C(1)	22(1)	23(1)	29(1)	-3(1)	-8(1)	-7(1)
C(2)	28(1)	23(1)	24(1)	-4(1)	-9(1)	-10(1)
C(3)	33(1)	24(1)	30(1)	4(1)	-12(1)	-8(1)
C(4)	26(1)	29(1)	42(2)	2(1)	-10(1)	-3(1)
C(5)	23(1)	32(1)	36(1)	0(1)	-3(1)	-7(1)
C(6)	27(1)	24(1)	28(1)	1(1)	-10(1)	-9(1)
C(7)	31(1)	30(1)	33(1)	6(1)	-5(1)	-9(1)
C(8)	42(2)	46(2)	42(2)	12(1)	3(1)	-11(1)
C(9)	47(2)	45(2)	33(2)	10(1)	-15(1)	-14(1)
C(10)	43(2)	33(2)	52(2)	6(1)	-8(1)	-19(1)
C(11)	29(1)	26(1)	26(1)	-3(1)	-8(1)	-11(1)
C(12)	37(1)	35(1)	31(1)	-10(1)	-6(1)	-10(1)
C(13)	42(2)	35(1)	29(1)	5(1)	-8(1)	-14(1)
C(14)	28(1)	32(1)	34(1)	-3(1)	-7(1)	-13(1)
C(0)	36(2)	46(2)	64(2)	17(2)	-8(2)	7(1)
O(2)	39(1)	22(1)	32(1)	-3(1)	-14(1)	-12(1)
C(21)	26(1)	23(1)	26(1)	-4(1)	-5(1)	-10(1)
C(22)	30(1)	27(1)	29(1)	-3(1)	-6(1)	-15(1)
C(23)	37(1)	38(1)	29(1)	-11(1)	-3(1)	-20(1)
C(24)	39(1)	28(1)	39(2)	-14(1)	2(1)	-18(1)
C(25)	33(1)	23(1)	38(1)	-1(1)	2(1)	-12(1)
C(26)	24(1)	24(1)	29(1)	-1(1)	-3(1)	-9(1)
C(27)	36(1)	29(1)	32(1)	7(1)	-14(1)	-13(1)
C(28)	44(2)	37(2)	59(2)	6(1)	-28(1)	-16(1)
C(29)	57(2)	38(2)	54(2)	17(1)	-28(2)	-22(1)
C(30)	51(2)	48(2)	28(1)	-3(1)	-9(1)	-17(1)
C(31)	51(2)	34(1)	33(1)	3(1)	-19(1)	-21(1)
C(32)	59(2)	37(2)	36(2)	7(1)	-11(1)	-26(1)
C(33)	45(2)	34(1)	53(2)	-1(1)	-26(1)	-11(1)
C(34)	90(3)	53(2)	46(2)	6(2)	-36(2)	-32(2)
C(20)	78(2)	40(2)	52(2)	-18(1)	3(2)	-33(2)
N(1)	59(2)	36(1)	35(1)	-2(1)	-22(1)	-22(1)
P(1)	34(1)	29(1)	27(1)	-5(1)	-11(1)	-10(1)
C(41)	33(1)	28(1)	37(1)	0(1)	-10(1)	-7(1)
C(42)	39(2)	58(2)	49(2)	0(2)	-20(1)	-11(1)
C(43)	50(2)	69(2)	80(3)	16(2)	-36(2)	-21(2)
C(44)	46(2)	46(2)	145(4)	7(2)	-38(3)	-1(2)
C(45)	54(2)	87(3)	146(5)	-60(3)	-17(3)	17(2)
C(46)	54(2)	66(2)	81(3)	-36(2)	-17(2)	5(2)
C(51)	40(1)	32(1)	33(1)	-3(1)	-18(1)	-12(1)
C(52)	47(2)	42(2)	66(2)	1(2)	-30(2)	-13(1)
C(53)	75(2)	40(2)	83(3)	5(2)	-49(2)	-6(2)
C(54)	89(3)	36(2)	68(2)	12(2)	-38(2)	-26(2)
C(55)	66(2)	46(2)	70(2)	7(2)	-18(2)	-29(2)
C(56)	44(2)	39(2)	51(2)	2(1)	-13(1)	-17(1)

C(61)	28(1)	32(1)	30(1)	-8(1)	-10(1)	-7(1)
C(62)	42(2)	39(2)	39(2)	-13(1)	-8(1)	-16(1)
C(63)	48(2)	50(2)	38(2)	-20(1)	-9(1)	-12(1)
C(64)	39(2)	52(2)	32(2)	-7(1)	-7(1)	-3(1)
C(65)	40(2)	39(2)	42(2)	1(1)	-5(1)	-10(1)
C(66)	41(1)	33(1)	38(2)	-8(1)	-9(1)	-13(1)
N(2)	31(1)	56(2)	49(1)	-18(1)	-1(1)	-20(1)
P(2)	27(1)	42(1)	34(1)	-6(1)	-5(1)	-16(1)
C(71)	30(1)	39(1)	37(1)	1(1)	-10(1)	-19(1)
C(72)	47(2)	44(2)	64(2)	4(2)	-29(2)	-17(1)
C(73)	67(2)	72(2)	74(3)	23(2)	-48(2)	-30(2)
C(74)	71(2)	96(3)	41(2)	9(2)	-27(2)	-48(2)
C(75)	51(2)	85(2)	36(2)	-11(2)	-9(1)	-30(2)
C(76)	35(1)	60(2)	35(2)	-3(1)	-12(1)	-15(1)
C(81)	37(1)	43(2)	30(1)	-1(1)	-11(1)	-18(1)
C(82)	48(2)	55(2)	60(2)	10(2)	-15(2)	-26(2)
C(83)	69(2)	56(2)	83(3)	21(2)	-24(2)	-34(2)
C(84)	68(2)	51(2)	63(2)	8(2)	-30(2)	-14(2)
C(85)	46(2)	57(2)	59(2)	0(2)	-24(2)	-13(2)
C(86)	40(2)	49(2)	47(2)	-1(1)	-15(1)	-18(1)
C(91)	28(1)	40(2)	45(2)	-14(1)	-5(1)	-12(1)
C(92)	43(2)	48(2)	52(2)	-9(1)	-9(1)	-22(1)
C(93)	59(2)	49(2)	79(3)	-2(2)	-13(2)	-33(2)
C(94)	45(2)	44(2)	102(3)	-21(2)	-20(2)	-20(2)
C(95)	46(2)	53(2)	81(3)	-26(2)	-24(2)	-16(2)
C(96)	44(2)	53(2)	56(2)	-15(2)	-16(1)	-18(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 8.

	x	y	z	U(eq)
H(3A)	12394(2)	3546(2)	8142(1)	35
H(5A)	13905(2)	4729(2)	6140(2)	39
H(8A)	13251(6)	6631(8)	4899(6)	75
H(8B)	13528(9)	5497(7)	5095(8)	75
H(8C)	13950(4)	6109(15)	5512(3)	75
H(9A)	10421(4)	6475(14)	5961(3)	64
H(9B)	11391(9)	5730(5)	5360(10)	64
H(9C)	11156(12)	6860(9)	5169(7)	64
H(10A)	11145(10)	7411(7)	6668(7)	65
H(10B)	11774(17)	7790(3)	5839(3)	65
H(10C)	12531(8)	7271(5)	6421(10)	65
H(12A)	10345(7)	5588(7)	9201(8)	51
H(12B)	9001(8)	5630(7)	9364(6)	51
H(12C)	9598(15)	6217(2)	8637(2)	51
H(13A)	10681(15)	3269(2)	8742(2)	54
H(13B)	9674(4)	3846(6)	9434(6)	54
H(13C)	11010(11)	3834(7)	9253(8)	54
H(14A)	9377(6)	4158(6)	7941(8)	46
H(14B)	8964(10)	5307(7)	7844(7)	46
H(14C)	8431(5)	4777(12)	8630(2)	46
H(0A)	14338(4)	2581(2)	7143(13)	87
H(0B)	14835(10)	3239(10)	7459(9)	87
H(0C)	15037(8)	3251(10)	6565(5)	87
H(23A)	8915(2)	10615(2)	9325(1)	40
H(23B)	8070(2)	12125(2)	7479(2)	40
H(28A)	6283(3)	10999(8)	7097(10)	65
H(28B)	7020(8)	9878(7)	7098(10)	65
H(28C)	6869(9)	10471(14)	6317(2)	65
H(29A)	7241(11)	12220(3)	6588(5)	71
H(29B)	7827(18)	11741(4)	5787(5)	71
H(29C)	8640(8)	11944(6)	6204(10)	71
H(30A)	9311(11)	9392(3)	6413(5)	64
H(30B)	9875(4)	10238(8)	6061(9)	64
H(30C)	9001(7)	10047(10)	5688(4)	64
H(32A)	7821(11)	8385(13)	8964(2)	65
H(32B)	7470(7)	8853(8)	9754(8)	65
H(32C)	8362(5)	7781(6)	9631(10)	65

H(33A)	10034(5)	7929(10)	8171(6)	62
H(33B)	10498(10)	7502(5)	8901(3)	62
H(33C)	10891(6)	8364(5)	8372(9)	62
H(34A)	8821(7)	9366(13)	10113(6)	86
H(34B)	10163(13)	9191(15)	9603(2)	86
H(34C)	9737(19)	8298(3)	10040(7)	86
H(20A)	9082(5)	12424(7)	8804(12)	85
H(20B)	7759(16)	12480(8)	9294(5)	85
H(20C)	8004(19)	12990(2)	8457(7)	85
H(1A)	7637(2)	8666(2)	5957(1)	47
H(42A)	5706(3)	7660(2)	4692(2)	58
H(43A)	3744(3)	8461(3)	4703(2)	77
H(44A)	2524(3)	9565(3)	5597(3)	98
H(45A)	3241(4)	9903(3)	6470(3)	123
H(46A)	5192(2)	9083(1)	6506(1)	84
H(52A)	8900(2)	5902(1)	5902(1)	59
H(53A)	8918(3)	4362(2)	6466(2)	76
H(54A)	7163(3)	3997(2)	6967(2)	73
H(55A)	5379(3)	5146(2)	6909(2)	71
H(56A)	5341(3)	6681(2)	6360(2)	54
H(62A)	7738(2)	6271(2)	4513(2)	46
H(63A)	8754(3)	6217(2)	3221(2)	54
H(64A)	9599(2)	7411(2)	2606(2)	54
H(65A)	9482(2)	8637(2)	3288(2)	52
H(66A)	8522(2)	8666(2)	4588(2)	44
H(2A)	6193(2)	6778(2)	7895(1)	53
H(72A)	6545(3)	6672(2)	9482(2)	58
H(73A)	6756(3)	6830(3)	10651(2)	78
H(74A)	5395(3)	8062(3)	11411(2)	75
H(75A)	3803(3)	9130(3)	11026(2)	66
H(76A)	3582(2)	8981(2)	9867(2)	51
H(82A)	5354(3)	9381(2)	7968(2)	64
H(83A)	4247(3)	10932(3)	7645(2)	82
H(84A)	2217(3)	11316(2)	7842(2)	72
H(85A)	1279(3)	10168(2)	8404(2)	64
H(86A)	2372(2)	8628(2)	8749(2)	53
H(92A)	4165(2)	6320(2)	9872(2)	55
H(93A)	3255(3)	5144(2)	9986(2)	72
H(94A)	2594(3)	4995(2)	8995(2)	72
H(95A)	2815(3)	5999(2)	7901(2)	67
H(96A)	3705(3)	7175(2)	7782(2)	58

9) Calcium bis(hexamethyldisilyamide)bis(triphenylmethylphosphonium)

Table 1. Crystal data and structure refinement for 9.

Identification code	9 (98srv136)
Empirical formula	C64 H86 Ca N2 P2 Si4
Formula weight	1097.73
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 30.598(6) A alpha = 90 deg. b = 12.393(3) A beta = 129.03(3) deg. c = 22.023(4) A gamma = 90 deg.
Volume, Z	6488(2) A^3, 4
Density (calculated)	1.124 Mg/m^3
Absorption coefficient	0.258 mm^-1
F(000)	2360
Crystal size	0.5 x 0.1 x 0.1 mm
Theta range for data collection	1.71 to 27.49 deg.
Limiting indices	-39<=h<=28, -16<=k<=15, -23<=l<=28
Reflections collected	24039
Independent reflections	7208 [R(int) = 0.0627]
Absorption correction	Multiscan
Max. and min. transmission	0.826542 and 0.972716
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7145 / 0 / 345
Goodness-of-fit on F^2	1.198
Final R indices [I>2sigma(I)]	R1 = 0.0610, wR2 = 0.0983
R indices (all data)	R1 = 0.1154, wR2 = 0.1347
Extinction coefficient	not refined
Largest diff. peak and hole	0.333 and -0.330 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 9. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ca(1)	0	7908(1)	7500	20(1)
N(1)	-99(1)	6971(2)	8340(2)	24(1)
Si(1)	511(1)	6319(1)	9038(1)	25(1)
C(41)	445(2)	4816(3)	9071(3)	45(1)
C(42)	883(2)	6837(3)	10060(2)	40(1)
C(43)	1030(1)	6524(3)	8855(2)	33(1)
Si(2)	-683(1)	6853(1)	8269(1)	24(1)
C(51)	-1043(2)	5497(3)	7926(2)	39(1)
C(52)	-549(2)	7097(3)	9220(2)	38(1)
C(53)	-1247(1)	7834(3)	7542(2)	29(1)
P(1)	-923(1)	10371(1)	6026(1)	21(1)
C(1)	-729(1)	9513(3)	6771(2)	25(1)
C(11)	-1214(1)	11671(3)	5998(2)	24(1)
C(12)	-951(2)	12211(3)	6700(2)	39(1)
C(13)	-1147(2)	13211(3)	6713(3)	46(1)
C(14)	-1612(2)	13672(3)	6036(3)	41(1)
C(15)	-1873(2)	13141(3)	5341(2)	43(1)
C(16)	-1674(1)	12143(3)	5316(2)	34(1)
C(21)	-331(1)	10716(3)	6063(2)	21(1)
C(22)	-23(1)	9881(3)	6057(2)	26(1)
C(23)	434(1)	10133(3)	6090(2)	33(1)
C(24)	591(1)	11194(3)	6139(2)	31(1)
C(25)	293(1)	12016(3)	6149(2)	31(1)
C(26)	-172(1)	11784(3)	6104(2)	26(1)
C(31)	-1457(1)	9756(3)	5085(2)	25(1)
C(32)	-1883(2)	9152(3)	4981(2)	42(1)
C(33)	-2309(2)	8717(4)	4254(3)	57(1)
C(34)	-2313(2)	8875(4)	3634(3)	55(1)
C(35)	-1897(2)	9483(4)	3727(2)	47(1)
C(36)	-1466(2)	9925(3)	4452(2)	36(1)
C(1X)	2685(2)	3014(4)	11948(2)	45(1)
C(2X)	2145(2)	3346(4)	11613(2)	48(1)
C(3X)	1959(2)	4360(4)	11295(3)	61(1)
C(4X)	2305(2)	5063(4)	11292(3)	61(1)
C(5X)	2836(2)	4751(4)	11612(3)	55(1)
C(6X)	3026(2)	3726(4)	11938(2)	47(1)
C(7X)	2893(2)	1918(4)	12321(3)	84(2)

Table 3. Bond lengths [A] and angles [deg] for 9.

Ca(1)-N(1)	2.360(3)
Ca(1)-N(1)#1	2.360(3)
Ca(1)-C(1)#1	2.646(4)
Ca(1)-C(1)	2.646(3)
Ca(1)-C(43)#1	3.151(4)
Ca(1)-Si(1)	3.3350(14)
Ca(1)-Si(1)#1	3.3350(14)
N(1)-Si(2)	1.699(3)
N(1)-Si(1)	1.699(3)
Si(1)-C(41)	1.881(4)
Si(1)-C(42)	1.884(4)
Si(1)-C(43)	1.884(3)
C(41)-H(41A)	0.98
C(41)-H(41B)	0.98
C(41)-H(41C)	0.98
C(42)-H(42A)	0.98
C(42)-H(42B)	0.98
C(42)-H(42C)	0.98
C(43)-H(43A)	0.98
C(43)-H(43B)	0.98
C(43)-H(43C)	0.98
Si(2)-C(53)	1.881(3)
Si(2)-C(51)	1.885(4)
Si(2)-C(52)	1.887(3)
C(51)-H(51A)	0.98
C(51)-H(51B)	0.98
C(51)-H(51C)	0.98
C(52)-H(52A)	0.98
C(52)-H(52B)	0.98
C(52)-H(52C)	0.98
C(53)-H(53A)	0.98
C(53)-H(53B)	0.98
C(53)-H(53C)	0.98
P(1)-C(1)	1.717(3)
P(1)-C(31)	1.811(3)
P(1)-C(21)	1.814(3)
P(1)-C(11)	1.823(3)
C(1)-H(1A)	0.99(4)
C(1)-H(1B)	0.96(4)

C(11)-C(12)	1.384(5)
C(11)-C(16)	1.386(5)
C(12)-C(13)	1.385(5)
C(12)-H(12A)	0.95
C(13)-C(14)	1.380(5)
C(13)-H(13A)	0.95
C(14)-C(15)	1.369(5)
C(14)-H(14A)	0.95
C(15)-C(16)	1.394(5)
C(15)-H(15A)	0.95
C(16)-H(16A)	0.95
C(21)-C(26)	1.394(4)
C(21)-C(22)	1.404(4)
C(22)-C(23)	1.388(4)
C(22)-H(22A)	0.95
C(23)-C(24)	1.382(5)
C(23)-H(23A)	0.95
C(24)-C(25)	1.378(5)
C(24)-H(24A)	0.95
C(25)-C(26)	1.390(4)
C(25)-H(25A)	0.95
C(26)-H(26A)	0.95
C(31)-C(36)	1.390(5)
C(31)-C(32)	1.392(5)
C(32)-C(33)	1.387(5)
C(32)-H(32A)	0.95
C(33)-C(34)	1.371(6)
C(33)-H(33A)	0.95
C(34)-C(35)	1.380(6)
C(34)-H(34A)	0.95
C(35)-C(36)	1.392(5)
C(35)-H(35A)	0.95
C(36)-H(36A)	0.95
C(1X)-C(6X)	1.376(6)
C(1X)-C(2X)	1.381(5)
C(1X)-C(7X)	1.506(6)
C(2X)-C(3X)	1.376(6)
C(2X)-H(2XA)	0.95
C(3X)-C(4X)	1.376(6)
C(3X)-H(3XA)	0.95
C(4X)-C(5X)	1.358(6)
C(4X)-H(4XA)	0.95
C(5X)-C(6X)	1.393(6)
C(5X)-H(5XA)	0.95
C(6X)-H(6XA)	0.95
C(7X)-H(7XA)	0.98
C(7X)-H(7XB)	0.98
C(7X)-H(7XC)	0.98

N(1)-Ca(1)-N(1)#1	121.06(14)
N(1)-Ca(1)-C(1)#1	111.71(10)
N(1)#1-Ca(1)-C(1)#1	111.71(9)
N(1)-Ca(1)-C(1)	111.71(9)
N(1)#1-Ca(1)-C(1)	111.71(10)
C(1)#1-Ca(1)-C(1)	82.5(2)
N(1)-Ca(1)-C(43)#1	85.92(10)
N(1)#1-Ca(1)-C(43)#1	62.33(9)
C(1)#1-Ca(1)-C(43)#1	160.38(11)
C(1)-Ca(1)-C(43)#1	82.91(11)
N(1)-Ca(1)-Si(1)	28.72(6)
N(1)#1-Ca(1)-Si(1)	107.22(7)
C(1)#1-Ca(1)-Si(1)	98.67(9)
C(1)-Ca(1)-Si(1)	137.47(7)
C(43)#1-Ca(1)-Si(1)	100.95(8)
N(1)-Ca(1)-Si(1)#1	107.22(7)
N(1)#1-Ca(1)-Si(1)#1	28.72(6)
C(1)#1-Ca(1)-Si(1)#1	137.47(7)
C(1)-Ca(1)-Si(1)#1	98.67(9)
C(43)#1-Ca(1)-Si(1)#1	33.63(6)
Si(1)-Ca(1)-Si(1)#1	107.64(5)
Si(2)-N(1)-Si(1)	122.4(2)
Si(2)-N(1)-Ca(1)	128.11(14)
Si(1)-N(1)-Ca(1)	109.41(12)
N(1)-Si(1)-C(41)	115.0(2)
N(1)-Si(1)-C(42)	114.3(2)
C(41)-Si(1)-C(42)	106.9(2)
N(1)-Si(1)-C(43)	109.66(14)
C(41)-Si(1)-C(43)	105.4(2)
C(42)-Si(1)-C(43)	104.7(2)
N(1)-Si(1)-Ca(1)	41.87(8)
C(41)-Si(1)-Ca(1)	129.11(14)
C(42)-Si(1)-Ca(1)	123.84(14)
C(43)-Si(1)-Ca(1)	67.84(11)
Si(1)-C(41)-H(41A)	109.47(14)
Si(1)-C(41)-H(41B)	109.47(12)
H(41A)-C(41)-H(41B)	109.5
Si(1)-C(41)-H(41C)	109.47(12)
H(41A)-C(41)-H(41C)	109.5
H(41B)-C(41)-H(41C)	109.5
Si(1)-C(42)-H(42A)	109.47(13)
Si(1)-C(42)-H(42B)	109.47(12)
H(42A)-C(42)-H(42B)	109.5
Si(1)-C(42)-H(42C)	109.47(12)
H(42A)-C(42)-H(42C)	109.5
H(42B)-C(42)-H(42C)	109.5
Si(1)-C(43)-H(43A)	109.47(12)
Si(1)-C(43)-H(43B)	109.47(11)
H(43A)-C(43)-H(43B)	109.5
Si(1)-C(43)-H(43C)	109.47(12)
H(43A)-C(43)-H(43C)	109.5
H(43B)-C(43)-H(43C)	109.5
N(1)-Si(2)-C(53)	111.75(14)
N(1)-Si(2)-C(51)	115.4(2)
C(53)-Si(2)-C(51)	104.1(2)
N(1)-Si(2)-C(52)	113.7(2)
C(53)-Si(2)-C(52)	106.4(2)
C(51)-Si(2)-C(52)	104.6(2)
Si(2)-C(51)-H(51A)	109.47(12)
Si(2)-C(51)-H(51B)	109.47(12)
H(51A)-C(51)-H(51B)	109.5
Si(2)-C(51)-H(51C)	109.47(11)
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
Si(2)-C(52)-H(52A)	109.47(12)
Si(2)-C(52)-H(52B)	109.47(13)
H(52A)-C(52)-H(52B)	109.5
Si(2)-C(52)-H(52C)	109.47(11)
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5
Si(2)-C(53)-H(53A)	109.47(11)
Si(2)-C(53)-H(53B)	109.47(10)
H(53A)-C(53)-H(53B)	109.5
Si(2)-C(53)-H(53C)	109.47(11)
H(53A)-C(53)-H(53C)	109.5
H(53B)-C(53)-H(53C)	109.5
C(1)-P(1)-C(31)	110.7(2)
C(1)-P(1)-C(21)	111.6(2)
C(31)-P(1)-C(21)	107.79(14)
C(1)-P(1)-C(11)	116.5(2)
C(31)-P(1)-C(11)	105.3(2)
C(21)-P(1)-C(11)	104.29(14)
P(1)-C(1)-Ca(1)	134.2(2)
P(1)-C(1)-H(1A)	107(2)
Ca(1)-C(1)-H(1A)	90(2)
P(1)-C(1)-H(1B)	109(2)
Ca(1)-C(1)-H(1B)	104(2)
H(1A)-C(1)-H(1B)	109(3)
C(12)-C(11)-C(16)	119.1(3)
C(12)-C(11)-P(1)	117.5(3)
C(16)-C(11)-P(1)	123.4(3)
C(11)-C(12)-C(13)	120.1(4)
C(11)-C(12)-H(12A)	120.0(2)
C(13)-C(12)-H(12A)	120.0(2)
C(14)-C(13)-C(12)	120.9(4)
C(14)-C(13)-H(13A)	119.6(2)
C(12)-C(13)-H(13A)	119.6(2)
C(15)-C(14)-C(13)	119.2(4)
C(15)-C(14)-H(14A)	120.4(2)

C(13)-C(14)-H(14A)	120.4(2)
C(14)-C(15)-C(16)	120.6(4)
C(14)-C(15)-H(15A)	119.7(2)
C(16)-C(15)-H(15A)	119.7(2)
C(11)-C(16)-C(15)	120.1(4)
C(11)-C(16)-H(16A)	119.9(2)
C(15)-C(16)-H(16A)	119.9(2)
C(26)-C(21)-C(22)	119.4(3)
C(26)-C(21)-P(1)	121.7(2)
C(22)-C(21)-P(1)	118.9(2)
C(23)-C(22)-C(21)	119.6(3)
C(23)-C(22)-H(22A)	120.2(2)
C(21)-C(22)-H(22A)	120.2(2)
C(24)-C(23)-C(22)	120.5(3)
C(24)-C(23)-H(23A)	119.7(2)
C(22)-C(23)-H(23A)	119.7(2)
C(25)-C(24)-C(23)	120.2(3)
C(25)-C(24)-H(24A)	119.9(2)
C(23)-C(24)-H(24A)	119.9(2)
C(24)-C(25)-C(26)	120.3(3)
C(24)-C(25)-H(25A)	119.9(2)
C(26)-C(25)-H(25A)	119.9(2)
C(25)-C(26)-C(21)	120.0(3)
C(25)-C(26)-H(26A)	120.0(2)
C(21)-C(26)-H(26A)	120.0(2)
C(36)-C(31)-C(32)	119.3(3)
C(36)-C(31)-P(1)	121.2(3)
C(32)-C(31)-P(1)	119.4(3)
C(33)-C(32)-C(31)	120.1(4)
C(33)-C(32)-H(32A)	120.0(3)
C(31)-C(32)-H(32A)	120.0(2)
C(34)-C(33)-C(32)	120.4(4)
C(34)-C(33)-H(33A)	119.8(3)
C(32)-C(33)-H(33A)	119.8(3)
C(33)-C(34)-C(35)	120.1(4)
C(33)-C(34)-H(34A)	119.9(3)
C(35)-C(34)-H(34A)	119.9(2)
C(34)-C(35)-C(36)	120.2(4)
C(34)-C(35)-H(35A)	119.9(3)
C(36)-C(35)-H(35A)	119.9(2)
C(31)-C(36)-C(35)	119.9(4)
C(31)-C(36)-H(36A)	120.0(2)
C(35)-C(36)-H(36A)	120.0(2)
C(6X)-C(1X)-C(2X)	117.6(4)
C(6X)-C(1X)-C(7X)	121.5(4)
C(2X)-C(1X)-C(7X)	120.9(4)
C(3X)-C(2X)-C(1X)	121.1(4)
C(3X)-C(2X)-H(2XA)	119.4(2)
C(1X)-C(2X)-H(2XA)	119.4(3)
C(4X)-C(3X)-C(2X)	120.5(4)
C(4X)-C(3X)-H(3XA)	119.8(3)
C(2X)-C(3X)-H(3XA)	119.8(2)
C(5X)-C(4X)-C(3X)	119.4(5)
C(5X)-C(4X)-H(4XA)	120.3(3)
C(3X)-C(4X)-H(4XA)	120.3(3)
C(4X)-C(5X)-C(6X)	120.0(4)
C(4X)-C(5X)-H(5XA)	120.0(3)
C(6X)-C(5X)-H(5XA)	120.0(2)
C(1X)-C(6X)-C(5X)	121.3(4)
C(1X)-C(6X)-H(6XA)	119.3(3)
C(5X)-C(6X)-H(6XA)	119.3(2)
C(1X)-C(7X)-H(7XA)	109.5(3)
C(1X)-C(7X)-H(7XB)	109.5(3)
H(7XA)-C(7X)-H(7XB)	109.5
C(1X)-C(7X)-H(7XC)	109.5(3)
H(7XA)-C(7X)-H(7XC)	109.5
H(7XB)-C(7X)-H(7XC)	109.5

Symmetry transformations used to generate equivalent atoms:
#1-x, y, -z+3/2

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 9.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ca(1)	22(1)	18(1)	20(1)	0	13(1)	0
N(1)	26(1)	22(2)	24(2)	1(1)	17(1)	0(1)
Si(1)	28(1)	23(1)	25(1)	4(1)	17(1)	4(1)
C(41)	45(2)	32(2)	58(3)	14(2)	33(2)	11(2)
C(42)	34(2)	50(3)	28(2)	4(2)	16(2)	13(2)
C(43)	30(2)	32(2)	36(2)	9(2)	20(2)	8(2)
Si(2)	25(1)	23(1)	24(1)	-1(1)	16(1)	-3(1)
C(51)	40(2)	31(2)	46(3)	-1(2)	27(2)	-7(2)
C(52)	34(2)	55(3)	30(2)	-2(2)	23(2)	-5(2)
C(53)	26(2)	29(2)	34(2)	2(2)	19(2)	1(2)
P(1)	19(1)	22(1)	18(1)	0(1)	11(1)	2(1)
C(1)	23(2)	28(2)	23(2)	3(2)	14(2)	2(2)
C(11)	21(2)	23(2)	27(2)	0(1)	15(2)	1(1)
C(12)	47(2)	35(2)	30(2)	0(2)	22(2)	8(2)
C(13)	65(3)	33(2)	45(3)	-6(2)	36(2)	7(2)
C(14)	52(2)	24(2)	63(3)	2(2)	44(3)	8(2)
C(15)	38(2)	31(2)	50(3)	9(2)	23(2)	13(2)
C(16)	31(2)	34(2)	29(2)	0(2)	14(2)	6(2)
C(21)	21(2)	26(2)	15(2)	3(1)	11(2)	3(1)
C(22)	30(2)	24(2)	27(2)	4(2)	20(2)	3(1)
C(23)	31(2)	37(2)	38(2)	11(2)	25(2)	12(2)
C(24)	26(2)	42(2)	27(2)	9(2)	18(2)	2(2)
C(25)	33(2)	29(2)	28(2)	3(2)	18(2)	-4(2)
C(26)	26(2)	26(2)	25(2)	5(2)	16(2)	6(1)

C(31)	20(2)	27(2)	22(2)	-3(2)	10(2)	4(1)
C(32)	33(2)	47(3)	32(2)	-10(2)	15(2)	-12(2)
C(33)	34(2)	64(3)	49(3)	-18(3)	15(2)	-19(2)
C(34)	33(2)	66(3)	31(2)	-18(2)	5(2)	1(2)
C(35)	41(2)	63(3)	25(2)	-8(2)	15(2)	9(2)
C(36)	28(2)	51(3)	23(2)	-3(2)	13(2)	4(2)
C(1X)	42(2)	53(3)	34(2)	-5(2)	20(2)	3(2)
C(2X)	36(2)	69(3)	41(3)	-6(2)	25(2)	-12(2)
C(3X)	42(3)	87(4)	58(3)	14(3)	33(3)	16(3)
C(4X)	67(3)	53(3)	74(4)	7(3)	50(3)	10(2)
C(5X)	59(3)	58(3)	61(3)	-22(3)	44(3)	-26(2)
C(6X)	28(2)	67(3)	42(3)	-16(2)	20(2)	-7(2)
C(7X)	107(5)	64(4)	78(4)	16(3)	57(4)	22(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 9.

	x	y	z	U(eq)
H(41A)	249(9)	4514(4)	8546(4)	67
H(41B)	231(9)	4654(3)	9253(14)	67
H(41C)	821(2)	4495(4)	9430(11)	67
H(42A)	898(9)	7627(3)	10061(3)	60
H(42B)	1266(4)	6546(16)	10408(3)	60
H(42C)	678(6)	6607(16)	10244(6)	60
H(43A)	872(4)	6236(17)	8339(6)	50
H(43B)	1381(4)	6146(16)	9257(8)	50
H(43C)	1106(7)	7296(3)	8872(13)	50
H(51A)	-783(4)	4939(4)	8297(7)	58
H(51B)	-1155(9)	5339(9)	7409(6)	58
H(51C)	-1377(6)	5510(6)	7895(13)	58
H(52A)	-328(9)	7760(11)	9462(7)	57
H(52B)	-339(9)	6486(9)	9573(6)	57
H(52C)	-908(2)	7172(20)	9119(3)	57
H(53A)	-1090(3)	8565(3)	7670(7)	44
H(53B)	-1557(4)	7805(13)	7563(9)	44
H(53C)	-1387(6)	7642(10)	7015(2)	44
H(1A)	-504(15)	9938(31)	7260(22)	41(11)
H(1B)	-1061(15)	9250(30)	6681(20)	43(11)
H(12A)	-635(2)	11896(3)	7172(2)	46
H(13A)	-958(2)	13584(3)	7195(3)	56
H(14A)	-1750(2)	14350(3)	6051(3)	49
H(15A)	-2192(2)	13454(3)	4872(2)	51
H(16A)	-1855(1)	11787(3)	4829(2)	41
H(22A)	-127(1)	9150(3)	6030(2)	31
H(23A)	639(1)	9570(3)	6078(2)	40
H(24A)	907(1)	11358(3)	6167(2)	37
H(25A)	404(1)	12744(3)	6187(2)	37
H(26A)	-380(1)	12354(3)	6102(2)	31
H(32A)	-1882(2)	9038(3)	5408(2)	50
H(33A)	-2599(2)	8306(4)	4186(3)	68
H(34A)	-2603(2)	8566(4)	3140(3)	65
H(35A)	-1904(2)	9601(4)	3294(2)	56
H(36A)	-1180(2)	10341(3)	4515(2)	44
H(2XA)	1898(2)	2865(4)	11601(2)	58
H(3XA)	1588(2)	4578(4)	11077(3)	74
H(4XA)	2174(2)	5760(4)	11067(3)	73
H(5XA)	3079(2)	5231(4)	11613(3)	66
H(6XA)	3397(2)	3515(4)	12159(2)	56
H(7XA)	2627(8)	1599(13)	12378(20)	126
H(7XB)	3263(7)	1990(6)	12837(9)	126
H(7XC)	2925(15)	1451(10)	11991(11)	126

10) Strontium bis(hexamethyldisilyamide)bis(triphenylmethylphosphonium)

Table 1. Crystal data and structure refinement for 10.

Identification code	10 (98srv119)
Empirical formula	C32 H43 N P Si2 Sr0.50
Formula weight	572.63
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 30.612(5) A alpha = 90 deg. b = 12.435(2) A beta = 129.028(6) deg. c = 22.039(3) A gamma = 90 deg.
Volume	6517.2(17) A^3
Z, Calculated density	8, 1.167 Mg/m^3
Absorption coefficient	0.987 mm^-1
F(000)	2432
Crystal size	0.30 x 0.30 x 0.30 mm
Theta range for data collection	1.71 to 27.45 deg.
Limiting indices	-39<=h<=26, -15<=k<=16, -27<=l<=23
Reflections collected / unique	17240 / 7139 [R(int) = 0.0980]
Absorption correction	Multiscan
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7125 / 0 / 337
Goodness-of-fit on F^2	0.990
Final R indices [I>2sigma(I)]	R1 = 0.0555, wR2 = 0.1127
R indices (all data)	R1 = 0.1257, wR2 = 0.1883
Extinction coefficient	not measured
Largest diff. peak and hole	0.689 and -0.731 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 10.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Sr(1)	0	7922(1)	7500	24(1)
P(1)	939(1)	10430(1)	8996(1)	26(1)
Si(1)	-685(1)	6828(1)	8299(1)	27(1)
Si(2)	517(1)	6289(1)	9080(1)	30(1)
N(1)	-99(1)	6918(2)	8392(2)	25(1)
C(7)	741(2)	9593(3)	8257(2)	30(1)
C(15)	1684(2)	12190(4)	9709(3)	36(1)
C(5)	-1233(2)	7810(3)	7552(2)	32(1)
C(13)	342(2)	10748(3)	8950(2)	24(1)
C(12)	165(2)	11791(3)	8894(2)	31(1)
C(11)	-308(2)	12002(4)	8830(2)	36(1)
C(20)	1469(2)	9806(3)	9928(2)	32(1)
C(3)	1032(2)	6535(4)	8892(2)	33(1)
C(14)	1224(2)	11728(3)	9028(2)	31(1)
C(25)	1487(2)	9965(4)	10565(2)	38(1)
C(2)	880(2)	6790(4)	10103(3)	49(1)
C(6)	-569(2)	7100(4)	9230(2)	43(1)
C(10)	-590(2)	11174(4)	8852(2)	38(1)
C(4)	-1045(2)	5481(4)	7955(3)	43(1)
C(9)	-419(2)	10126(4)	8919(2)	35(1)
C(1)	454(2)	4787(3)	9101(3)	48(1)
C(8)	43(2)	9908(3)	8963(2)	33(1)
C(21)	1880(2)	9183(4)	10023(3)	47(1)
C(19)	966(2)	12284(4)	8329(3)	43(1)
C(23)	2313(2)	8888(5)	11364(3)	61(2)
C(24)	1910(2)	9505(5)	11283(3)	55(2)
C(16)	1890(2)	13178(4)	9689(3)	51(1)
C(17)	1631(3)	13710(4)	9000(3)	57(2)
C(18)	1162(2)	13268(4)	8321(3)	53(1)
C(22)	2307(2)	8729(5)	10743(3)	59(2)
C(28)	2834(2)	8361(4)	13373(3)	51(1)
C(27)	2292(2)	8029(4)	13040(3)	50(1)
C(29)	3021(2)	9364(5)	13681(3)	62(2)
C(30)	2676(3)	10077(5)	13678(3)	63(2)
C(32)	1955(2)	8756(5)	13050(3)	50(1)
C(26)	2091(3)	6942(5)	12688(4)	88(2)
C(31)	2154(2)	9767(5)	13372(3)	56(2)

Table 3. Bond lengths [A] and angles [deg] for 10.

Sr(1)-N(1)#1	2.502(3)
Sr(1)-N(1)	2.502(3)
Sr(1)-C(7)#1	2.736(4)
Sr(1)-C(7)	2.736(4)
Sr(1)-C(3)#1	3.187(4)
Sr(1)-Si(2)	3.4365(12)
Sr(1)-Si(2)#1	3.4365(12)
Sr(1)-Si(1)	3.7405(13)
Sr(1)-Si(1)#1	3.7405(13)
P(1)-C(7)	1.691(4)
P(1)-C(20)	1.804(4)
P(1)-C(13)	1.809(4)
P(1)-C(14)	1.815(4)
Si(1)-N(1)	1.676(4)
Si(1)-C(6)	1.878(4)
Si(1)-C(5)	1.882(4)
Si(1)-C(4)	1.882(4)
Si(2)-N(1)	1.693(3)
Si(2)-C(1)	1.881(5)
Si(2)-C(2)	1.885(5)
Si(2)-C(3)	1.888(5)
C(7)-H(7A)	0.97
C(7)-H(7B)	0.97
C(15)-C(14)	1.380(6)
C(15)-C(16)	1.395(6)
C(15)-H(15A)	0.93
C(5)-H(5A)	0.96
C(5)-H(5B)	0.96
C(5)-H(5C)	0.96
C(13)-C(12)	1.380(5)
C(13)-C(8)	1.402(6)
C(12)-C(11)	1.390(6)
C(12)-H(12A)	0.93
C(11)-C(10)	1.364(6)
C(11)-H(11A)	0.93
C(20)-C(21)	1.376(6)
C(20)-C(25)	1.385(6)
C(3)-H(3A)	0.96
C(3)-H(3B)	0.96
C(3)-H(3C)	0.96
C(14)-C(19)	1.393(6)
C(25)-C(24)	1.390(6)
C(25)-H(25A)	0.93
C(2)-H(2A)	0.96
C(2)-H(2B)	0.96
C(2)-H(2C)	0.96
C(6)-H(6A)	0.96
C(6)-H(6B)	0.96
C(6)-H(6C)	0.96
C(10)-C(9)	1.377(6)
C(10)-H(10A)	0.93
C(4)-H(4A)	0.96
C(4)-H(4B)	0.96
C(4)-H(4C)	0.96
C(9)-C(8)	1.382(6)
C(9)-H(9A)	0.93
C(1)-H(1A)	0.96
C(1)-H(1B)	0.96
C(1)-H(1C)	0.96
C(8)-H(8A)	0.93
C(21)-C(22)	1.391(6)
C(21)-H(21A)	0.93
C(19)-C(18)	1.369(7)
C(19)-H(19A)	0.93
C(23)-C(24)	1.365(8)
C(23)-C(22)	1.372(8)
C(23)-H(23A)	0.93
C(24)-H(24A)	0.93
C(16)-C(17)	1.362(7)
C(16)-H(16A)	0.93
C(17)-C(18)	1.378(7)
C(17)-H(17A)	0.93
C(18)-H(18A)	0.93
C(22)-H(22A)	0.93
C(28)-C(29)	1.363(7)
C(28)-C(27)	1.387(8)
C(28)-H(28A)	0.93
C(27)-C(32)	1.382(7)
C(27)-C(26)	1.487(7)
C(29)-C(30)	1.374(8)
C(29)-H(29A)	0.93
C(30)-C(31)	1.342(8)
C(30)-H(30A)	0.93
C(32)-C(31)	1.382(7)
C(32)-H(32A)	0.93
C(26)-H(26A)	0.96
C(26)-H(26B)	0.96
C(26)-H(26C)	0.96
C(31)-H(31A)	0.93
N(1)#1-Sr(1)-N(1)	120.17(14)
N(1)#1-Sr(1)-C(7)#1	111.34(11)
N(1)-Sr(1)-C(7)#1	113.20(12)
N(1)#1-Sr(1)-C(7)	113.20(12)
N(1)-Sr(1)-C(7)	111.34(11)
C(7)#1-Sr(1)-C(7)	81.1(2)
N(1)#1-Sr(1)-C(3)#1	60.69(11)
N(1)-Sr(1)-C(3)#1	87.12(11)
C(7)#1-Sr(1)-C(3)#1	83.43(12)
C(7)-Sr(1)-C(3)#1	159.50(12)
N(1)#1-Sr(1)-Si(2)	107.14(7)
N(1)-Sr(1)-Si(2)	27.86(8)
C(7)#1-Sr(1)-Si(2)	137.97(9)
C(7)-Sr(1)-Si(2)	98.92(9)
C(3)#1-Sr(1)-Si(2)	101.58(8)
N(1)#1-Sr(1)-Si(2)#1	27.86(8)
N(1)-Sr(1)-Si(2)#1	107.14(8)
C(7)#1-Sr(1)-Si(2)#1	98.92(9)
C(7)-Sr(1)-Si(2)#1	137.97(9)
C(3)#1-Sr(1)-Si(2)#1	32.85(8)
Si(2)-Sr(1)-Si(2)#1	107.57(4)
N(1)#1-Sr(1)-Si(1)	124.70(8)
N(1)-Sr(1)-Si(1)	21.27(8)
C(7)#1-Sr(1)-Si(1)	93.90(9)
C(7)-Sr(1)-Si(1)	118.97(9)
C(3)#1-Sr(1)-Si(1)	75.37(8)
Si(2)-Sr(1)-Si(1)	49.11(3)
Si(2)#1-Sr(1)-Si(1)	103.01(3)
N(1)#1-Sr(1)-Si(1)#1	21.27(8)
N(1)-Sr(1)-Si(1)#1	124.70(8)
C(7)#1-Sr(1)-Si(1)#1	118.97(9)
C(7)-Sr(1)-Si(1)#1	93.90(9)
C(3)#1-Sr(1)-Si(1)#1	81.90(8)
Si(2)-Sr(1)-Si(1)#1	103.00(3)
Si(2)#1-Sr(1)-Si(1)#1	49.11(3)
Si(1)-Sr(1)-Si(1)#1	137.36(4)
C(7)-P(1)-C(20)	110.6(2)
C(7)-P(1)-C(13)	110.1(2)
C(20)-P(1)-C(13)	108.1(2)
C(7)-P(1)-C(14)	117.1(2)
C(20)-P(1)-C(14)	105.8(2)
C(13)-P(1)-C(14)	104.6(2)
N(1)-Si(1)-C(6)	113.8(2)
N(1)-Si(1)-C(5)	111.1(2)
C(6)-Si(1)-C(5)	106.8(2)
N(1)-Si(1)-C(4)	114.8(2)
C(6)-Si(1)-C(4)	105.1(2)
C(5)-Si(1)-C(4)	104.6(2)
N(1)-Si(1)-Sr(1)	32.77(10)
C(6)-Si(1)-Sr(1)	132.2(2)
C(5)-Si(1)-Sr(1)	78.92(15)

C(4)-Si(1)-Sr(1)	119.6(2)
N(1)-Si(2)-C(1)	114.1(2)
N(1)-Si(2)-C(2)	113.8(2)
C(1)-Si(2)-C(2)	106.9(2)
N(1)-Si(2)-C(3)	109.9(2)
C(1)-Si(2)-C(3)	106.2(2)
C(2)-Si(2)-C(3)	105.3(2)
N(1)-Si(2)-Sr(1)	43.66(12)
C(1)-Si(2)-Sr(1)	128.5(2)
C(2)-Si(2)-Sr(1)	124.4(2)
C(3)-Si(2)-Sr(1)	66.30(13)
Si(1)-N(1)-Si(2)	125.5(2)
Si(1)-N(1)-Sr(1)	126.0(2)
Si(2)-N(1)-Sr(1)	108.5(2)
P(1)-C(7)-Sr(1)	135.3(2)
P(1)-C(7)-H(7A)	103.34(15)
Sr(1)-C(7)-H(7A)	103.34(9)
P(1)-C(7)-H(7B)	103.3(2)
Sr(1)-C(7)-H(7B)	103.34(9)
H(7A)-C(7)-H(7B)	105.2
C(14)-C(15)-C(16)	120.3(4)
C(14)-C(15)-H(15A)	119.9(3)
C(16)-C(15)-H(15A)	119.9(3)
Si(1)-C(5)-H(5A)	109.47(13)
Si(1)-C(5)-H(5B)	109.47(15)
H(5A)-C(5)-H(5B)	109.5
Si(1)-C(5)-H(5C)	109.47(14)
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(12)-C(13)-C(8)	118.5(4)
C(12)-C(13)-P(1)	122.3(3)
C(8)-C(13)-P(1)	119.1(3)
C(13)-C(12)-C(11)	120.8(4)
C(13)-C(12)-H(12A)	119.6(3)
C(11)-C(12)-H(12A)	119.6(3)
C(10)-C(11)-C(12)	119.8(4)
C(10)-C(11)-H(11A)	120.1(3)
C(12)-C(11)-H(11A)	120.1(3)
C(21)-C(20)-C(25)	118.5(4)
C(21)-C(20)-P(1)	119.8(4)
C(25)-C(20)-P(1)	121.7(4)
Si(2)-C(3)-H(3A)	109.47(13)
Si(2)-C(3)-H(3B)	109.47(13)
H(3A)-C(3)-H(3B)	109.5
Si(2)-C(3)-H(3C)	109.47(14)
H(3A)-C(3)-H(3C)	109.5
H(3B)-C(3)-H(3C)	109.5
C(15)-C(14)-C(19)	118.4(4)
C(15)-C(14)-P(1)	123.2(3)
C(19)-C(14)-P(1)	118.3(3)
C(20)-C(25)-C(24)	120.6(5)
C(20)-C(25)-H(25A)	119.7(3)
C(24)-C(25)-H(25A)	119.7(3)
Si(2)-C(2)-H(2A)	109.5(2)
Si(2)-C(2)-H(2B)	109.5(2)
H(2A)-C(2)-H(2B)	109.5
Si(2)-C(2)-H(2C)	109.5(2)
H(2A)-C(2)-H(2C)	109.5
H(2B)-C(2)-H(2C)	109.5
Si(1)-C(6)-H(6A)	109.47(15)
Si(1)-C(6)-H(6B)	109.5(2)
H(6A)-C(6)-H(6B)	109.5
Si(1)-C(6)-H(6C)	109.5(2)
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
C(11)-C(10)-C(9)	120.8(5)
C(11)-C(10)-H(10A)	119.6(3)
C(9)-C(10)-H(10A)	119.6(3)
Si(1)-C(4)-H(4A)	109.5(2)
Si(1)-C(4)-H(4B)	109.5(2)
H(4A)-C(4)-H(4B)	109.5
Si(1)-C(4)-H(4C)	109.5(2)
H(4A)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
C(10)-C(9)-C(8)	119.7(4)
C(10)-C(9)-H(9A)	120.1(3)
C(8)-C(9)-H(9A)	120.1(3)
Si(2)-C(1)-H(1A)	109.5(2)
Si(2)-C(1)-H(1B)	109.5(2)
H(1A)-C(1)-H(1B)	109.5
Si(2)-C(1)-H(1C)	109.5(2)
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
C(9)-C(8)-C(13)	120.4(4)
C(9)-C(8)-H(8A)	119.8(3)
C(13)-C(8)-H(8A)	119.8(3)
C(20)-C(21)-C(22)	121.1(5)
C(20)-C(21)-H(21A)	119.5(3)
C(22)-C(21)-H(21A)	119.5(3)
C(18)-C(19)-C(14)	120.7(5)
C(18)-C(19)-H(19A)	119.7(3)
C(14)-C(19)-H(19A)	119.7(3)
C(24)-C(23)-C(22)	120.5(5)
C(24)-C(23)-H(23A)	119.7(3)
C(22)-C(23)-H(23A)	119.7(3)
C(23)-C(24)-C(25)	119.9(5)
C(23)-C(24)-H(24A)	120.1(3)
C(25)-C(24)-H(24A)	120.1(3)
C(17)-C(16)-C(15)	120.4(5)
C(17)-C(16)-H(16A)	119.8(3)
C(15)-C(16)-H(16A)	119.8(3)
C(16)-C(17)-C(18)	119.6(5)
C(16)-C(17)-H(17A)	120.2(3)
C(18)-C(17)-H(17A)	120.2(3)
C(19)-C(18)-C(17)	120.5(5)
C(19)-C(18)-H(18A)	119.7(3)
C(17)-C(18)-H(18A)	119.7(3)
C(23)-C(22)-C(21)	119.4(5)
C(23)-C(22)-H(22A)	120.3(3)
C(21)-C(22)-H(22A)	120.3(3)
C(29)-C(28)-C(27)	121.5(6)
C(29)-C(28)-H(28A)	119.2(4)
C(27)-C(28)-H(28A)	119.2(3)
C(32)-C(27)-C(28)	116.9(5)
C(32)-C(27)-C(26)	122.3(6)
C(28)-C(27)-C(26)	120.8(6)
C(28)-C(29)-C(30)	120.5(6)
C(28)-C(29)-H(29A)	119.7(4)
C(30)-C(29)-H(29A)	119.7(4)
C(31)-C(30)-C(29)	119.0(6)
C(31)-C(30)-H(30A)	120.5(4)
C(29)-C(30)-H(30A)	120.5(4)
C(31)-C(32)-C(27)	120.8(5)
C(31)-C(32)-H(32A)	119.6(3)
C(27)-C(32)-H(32A)	119.6(3)
C(27)-C(26)-H(26A)	109.5(4)
C(27)-C(26)-H(26B)	109.5(3)
H(26A)-C(26)-H(26B)	109.5
C(27)-C(26)-H(26C)	109.5(4)
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(30)-C(31)-C(32)	121.2(6)
C(30)-C(31)-H(31A)	119.4(4)
C(32)-C(31)-H(31A)	119.4(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x, y, -z+3/2

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 10.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Sr(1)	25(1)	22(1)	20(1)	0	12(1)	0
P(1)	23(1)	29(1)	19(1)	-2(1)	10(1)	-1(1)
Si(1)	28(1)	26(1)	26(1)	-2(1)	17(1)	-4(1)
Si(2)	34(1)	25(1)	27(1)	5(1)	18(1)	6(1)
N(1)	25(2)	25(2)	23(2)	1(1)	14(2)	3(1)
C(7)	23(2)	31(2)	28(2)	-8(2)	12(2)	0(2)
C(15)	34(3)	40(3)	38(2)	-2(2)	24(2)	-5(2)
C(5)	27(2)	37(2)	34(2)	0(2)	20(2)	0(2)
C(13)	11(2)	34(2)	18(2)	-7(2)	6(2)	-3(2)
C(12)	32(3)	30(2)	26(2)	-4(2)	15(2)	-4(2)
C(11)	38(3)	32(2)	33(2)	-3(2)	21(2)	4(2)
C(20)	28(2)	29(2)	22(2)	1(2)	8(2)	-6(2)
C(3)	19(2)	42(3)	35(2)	6(2)	16(2)	6(2)
C(14)	38(3)	29(2)	29(2)	2(2)	23(2)	3(2)
C(25)	23(2)	59(3)	27(2)	4(2)	14(2)	-2(2)
C(2)	40(3)	63(3)	35(3)	6(2)	20(2)	16(2)
C(6)	34(3)	65(3)	35(2)	-3(2)	25(2)	-7(2)
C(10)	26(3)	50(3)	33(2)	-10(2)	17(2)	-3(2)
C(4)	52(3)	33(3)	53(3)	-4(2)	36(3)	-10(2)
C(9)	29(3)	44(3)	39(2)	-11(2)	24(2)	-13(2)
C(1)	52(3)	31(3)	56(3)	15(2)	31(3)	10(2)
C(8)	38(3)	31(2)	29(2)	-5(2)	20(2)	-5(2)
C(21)	32(3)	58(3)	38(3)	7(2)	16(2)	13(2)
C(19)	45(3)	46(3)	30(2)	5(2)	19(2)	-2(2)
C(23)	40(3)	64(4)	35(3)	21(3)	3(3)	-1(3)
C(24)	47(4)	72(4)	26(2)	12(2)	14(2)	-10(3)
C(16)	40(3)	39(3)	52(3)	-7(2)	19(3)	-8(2)
C(17)	84(4)	29(3)	76(4)	5(3)	58(4)	-1(3)
C(18)	69(4)	45(3)	61(3)	16(3)	50(3)	7(3)
C(22)	27(3)	74(4)	53(3)	19(3)	14(3)	21(3)
C(28)	46(3)	64(4)	39(3)	3(2)	25(3)	9(3)
C(27)	50(3)	52(3)	34(3)	7(2)	20(3)	-3(3)
C(29)	37(3)	86(5)	52(3)	-12(3)	23(3)	-10(3)
C(30)	67(4)	52(4)	69(4)	-5(3)	41(4)	-7(3)
C(32)	30(3)	73(4)	38(3)	14(2)	17(2)	1(3)
C(26)	101(6)	68(5)	68(4)	-10(3)	40(4)	-18(4)
C(31)	49(4)	58(4)	60(3)	18(3)	33(3)	21(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 10.

	x	y	z	U(eq)
H(7A)	1094(2)	9286(3)	8429(2)	36
H(7B)	627(2)	10081(3)	7837(2)	36
H(15A)	1857(2)	11841(4)	10183(3)	43
H(5A)	-1074(4)	8519(5)	7676(9)	48
H(5B)	-1546(5)	7804(16)	7554(11)	48
H(5C)	-1360(8)	7608(13)	7044(3)	48
H(12A)	366(2)	12358(3)	8900(2)	37
H(11A)	-433(2)	12706(4)	8772(2)	43
H(3A)	879(5)	6259(20)	8387(7)	49
H(3B)	1380(4)	6178(18)	9285(9)	49
H(3C)	1097(9)	7293(4)	8909(16)	49
H(25A)	1212(2)	10383(4)	10511(2)	45
H(2A)	685(9)	6531(23)	10284(8)	73
H(2B)	880(13)	7562(4)	10104(4)	73
H(2C)	1261(5)	6533(23)	10442(5)	73
H(6A)	-335(11)	7725(15)	9480(10)	64
H(6B)	-387(12)	6491(11)	9571(8)	64
H(6C)	-924(2)	7223(25)	9113(3)	64
H(10A)	-902(2)	11320(4)	8821(2)	45
H(4A)	-795(5)	4938(5)	8325(9)	65
H(4B)	-1145(12)	5318(12)	7456(9)	65
H(4C)	-1378(7)	5501(8)	7909(17)	65
H(9A)	-614(2)	9568(4)	8936(2)	42
H(1A)	240(12)	4507(5)	8580(4)	72
H(1B)	268(12)	4615(4)	9312(17)	72
H(1C)	823(2)	4471(4)	9422(15)	72
H(8A)	155(2)	9200(3)	9002(2)	39
H(21A)	1872(2)	9064(4)	9600(3)	56
H(19A)	656(2)	11984(4)	7864(3)	52
H(23A)	2593(2)	8572(5)	11844(3)	73
H(24A)	1920(2)	9618(5)	11709(3)	66
H(16A)	2206(2)	13476(4)	10149(3)	61
H(17A)	1770(3)	14368(4)	8988(3)	68
H(18A)	978(2)	13641(4)	7853(3)	63
H(22A)	2587(2)	8321(5)	10803(3)	71
H(28A)	3075(2)	7887(4)	13386(3)	61
H(29A)	3384(2)	9569(5)	13895(3)	74
H(30A)	2803(3)	10763(5)	13885(3)	76
H(32A)	1591(2)	8563(5)	12838(3)	60
H(26A)	1697(5)	6975(9)	12247(18)	132
H(26B)	2147(19)	6452(10)	13068(9)	132
H(26C)	2297(14)	6696(17)	12520(26)	132
H(31A)	1921(2)	10242(5)	13376(3)	67

11) Barium bis(hexamethyldisilyamide)bis(triphenylmethylphosphonium)

Table 1. Crystal data and structure refinement for 11.

Identification code	11 (98srv154)
Empirical formula	C50 H70 Ba N2 P2 Si4
Formula weight	1010.72
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 9.884(2) A alpha = 90 deg. b = 23.521(5) A beta = 98.91(3) deg. c = 24.193(5) A gamma = 90 deg.
Volume, Z	5556(2) A^3, 4
Density (calculated)	1.208 Mg/m^3
Absorption coefficient	0.890 mm^-1
F(000)	2104
Crystal size	0.25 x 0.10 x 0.10 mm
Theta range for data collection	1.21 to 27.49 deg.
Limiting indices	-12<=h<=12, -30<=k<=30, -31<=l<=31
Reflections collected	63342
Independent reflections	12724 [R(int) = 0.2068]
Absorption correction	Multiscan
Max. and min. transmission	0.8835 and 0.6322
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12691 / 0 / 544
Goodness-of-fit on F^2	1.085
Final R indices [I>2sigma(I)]	R1 = 0.0669, wR2 = 0.1030
R indices (all data)	R1 = 0.1822, wR2 = 0.1622
Extinction coefficient	not refined
Largest diff. peak and hole	0.949 and -0.950 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 11. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ba(1)	-2622(1)	7367(1)	7630(1)	31(1)
C(1)	-679(7)	8292(3)	8109(2)	39(2)
P(1)	-96(2)	8455(1)	8793(1)	33(1)
C(11)	855(6)	9115(3)	8964(3)	32(2)
C(12)	1684(8)	9300(3)	8596(3)	61(2)
C(13)	2479(9)	9782(4)	8715(4)	80(3)
C(14)	2461(9)	10082(3)	9206(4)	67(2)
C(15)	1643(8)	9891(3)	9573(3)	55(2)
C(16)	834(7)	9408(3)	9456(3)	45(2)
C(21)	1046(6)	7906(3)	9108(2)	32(2)
C(22)	2116(8)	8010(3)	9535(3)	67(2)
C(23)	2968(8)	7586(4)	9773(4)	78(3)
C(24)	2757(7)	7029(3)	9575(3)	50(2)
C(25)	1701(7)	6919(3)	9159(3)	50(2)
C(26)	854(6)	7353(3)	8925(3)	44(2)
C(31)	-1528(7)	8516(3)	9175(3)	36(2)
C(32)	-2640(8)	8855(4)	8967(3)	62(2)
C(33)	-3729(8)	8917(4)	9255(4)	76(3)
C(34)	-3709(9)	8638(4)	9757(4)	67(3)
C(35)	-2637(10)	8303(4)	9964(4)	68(2)
C(36)	-1537(8)	8241(3)	9682(3)	49(2)
C(2)	-2936(7)	6932(3)	8755(3)	41(2)
P(2)	-2980(2)	6267(1)	9035(1)	34(1)
C(51)	-4232(6)	5832(3)	8608(2)	33(2)
C(52)	-5514(7)	6059(3)	8415(3)	49(2)
C(53)	-6497(8)	5746(4)	8090(3)	63(2)
C(54)	-6253(8)	5183(4)	7967(3)	60(2)
C(55)	-5004(9)	4945(4)	8159(3)	57(2)
C(56)	-3997(7)	5260(3)	8483(3)	48(2)
C(61)	-1355(6)	5917(3)	9063(3)	32(2)
C(62)	-877(6)	5784(3)	8564(3)	35(2)
C(63)	351(7)	5499(3)	8579(3)	45(2)
C(64)	1147(7)	5361(3)	9087(3)	49(2)
C(65)	707(7)	5506(3)	9580(3)	46(2)
C(66)	-539(7)	5785(3)	9575(3)	39(2)
C(71)	-3466(6)	6196(3)	9726(3)	36(2)
C(72)	-3657(6)	6686(3)	10025(3)	38(2)
C(73)	-4100(7)	6653(3)	10538(3)	48(2)
C(74)	-4402(7)	6139(3)	10755(3)	48(2)
C(75)	-4206(7)	5639(3)	10468(3)	51(2)
C(76)	-3736(7)	5670(3)	9956(3)	44(2)
N(1)	-4327(5)	8093(2)	7072(2)	35(1)
Si(1)	-3879(2)	8278(1)	6450(1)	42(1)
Si(2)	-5600(2)	8337(1)	7390(1)	48(1)
C(41)	-3837(11)	9065(3)	6311(4)	96(3)
C(42)	-4984(8)	7961(4)	5832(3)	71(3)
C(43)	-2113(7)	8017(3)	6413(3)	55(2)
C(44)	-5641(8)	7931(3)	8061(3)	62(2)
C(45)	-5421(10)	9116(3)	7591(4)	85(3)
C(46)	-7344(8)	8264(5)	6976(4)	94(3)
N(2)	-2041(6)	6519(2)	6993(2)	39(1)
Si(3)	-3429(2)	6152(1)	6695(1)	49(1)
Si(4)	-406(2)	6436(1)	6893(1)	50(1)
C(81)	-3411(10)	5373(3)	6892(4)	87(3)
C(82)	-3740(9)	6170(4)	5901(3)	71(2)
C(83)	-5013(8)	6465(3)	6922(3)	61(2)
C(84)	240(9)	5680(3)	6940(3)	79(3)
C(85)	760(8)	6853(3)	7434(3)	66(2)
C(86)	-9(9)	6679(4)	6192(3)	84(3)

Table 3. Bond lengths [A] and angles [deg] for 11.

Ba(1)-N(1)	2.623(5)
Ba(1)-N(2)	2.637(5)
Ba(1)-C(2)	2.967(6)
Ba(1)-C(1)	3.013(6)
Ba(1)-Si(1)	3.632(2)
Ba(1)-Si(3)	3.654(2)
Ba(1)-Si(2)	3.699(2)
Ba(1)-Si(4)	3.739(2)
C(1)-P(1)	1.709(6)
C(1)-H(1A)	0.99
C(1)-H(1B)	0.99
P(1)-C(21)	1.805(7)
P(1)-C(31)	1.811(6)
P(1)-C(11)	1.828(6)
C(11)-C(12)	1.372(8)
C(11)-C(16)	1.379(8)
C(12)-C(13)	1.384(10)
C(12)-H(12A)	0.95
C(13)-C(14)	1.384(10)
C(13)-H(13A)	0.95
C(14)-C(15)	1.367(10)
C(14)-H(14A)	0.95
C(15)-C(16)	1.391(9)
C(15)-H(15A)	0.95
C(16)-H(16A)	0.95
C(21)-C(26)	1.378(8)
C(21)-C(22)	1.381(9)
C(22)-C(23)	1.373(10)
C(22)-H(22A)	0.95
C(23)-C(24)	1.399(10)
C(23)-H(23A)	0.95
C(24)-C(25)	1.359(9)
C(24)-H(24A)	0.95
C(25)-C(26)	1.385(9)
C(25)-H(25A)	0.95
C(26)-H(26A)	0.95
C(31)-C(32)	1.388(9)
C(31)-C(36)	1.389(9)
C(32)-C(33)	1.378(10)
C(32)-H(32A)	0.95
C(33)-C(34)	1.378(11)
C(33)-H(33A)	0.95
C(34)-C(35)	1.351(11)
C(34)-H(34A)	0.95
C(35)-C(36)	1.378(10)
C(35)-H(35A)	0.95
C(36)-H(36A)	0.95
C(2)-P(2)	1.709(6)
C(2)-H(2A)	0.99
C(2)-H(2B)	0.99
P(2)-C(61)	1.796(6)
P(2)-C(51)	1.802(7)
P(2)-C(71)	1.819(6)
C(51)-C(52)	1.388(8)
C(51)-C(56)	1.404(9)
C(52)-C(53)	1.367(10)
C(52)-H(52A)	0.95
C(53)-C(54)	1.385(11)
C(53)-H(53A)	0.95
C(54)-C(55)	1.369(11)
C(54)-H(54A)	0.95
C(55)-C(56)	1.384(9)
C(55)-H(55A)	0.95
C(56)-H(56A)	0.95
C(61)-C(62)	1.397(8)
C(61)-C(66)	1.404(8)
C(62)-C(63)	1.382(9)
C(62)-H(62A)	0.95
C(63)-C(64)	1.390(9)
C(63)-H(63A)	0.95
C(64)-C(65)	1.374(9)
C(64)-H(64A)	0.95
C(65)-C(66)	1.393(9)
C(65)-H(65A)	0.95
C(66)-H(66A)	0.95
C(71)-C(72)	1.389(8)
C(71)-C(76)	1.397(9)
C(72)-C(73)	1.382(8)
C(72)-H(72A)	0.95
C(73)-C(74)	1.369(9)
C(73)-H(73A)	0.95
C(74)-C(75)	1.393(10)
C(74)-H(74A)	0.95
C(75)-C(76)	1.391(9)
C(75)-H(75A)	0.95
C(76)-H(76A)	0.95
N(1)-Si(2)	1.675(5)
N(1)-Si(1)	1.688(5)
Si(1)-C(42)	1.865(7)
Si(1)-C(43)	1.866(7)
Si(1)-C(41)	1.884(7)
Si(2)-C(46)	1.862(8)
Si(2)-C(44)	1.890(7)
Si(2)-C(45)	1.897(8)
C(41)-H(41A)	0.98
C(41)-H(41B)	0.98
C(41)-H(41C)	0.98
C(42)-H(42A)	0.98
C(42)-H(42B)	0.98
C(42)-H(42C)	0.98
C(43)-H(43A)	0.98
C(43)-H(43B)	0.98
C(43)-H(43C)	0.98
C(44)-H(44A)	0.98
C(44)-H(44B)	0.98
C(44)-H(44C)	0.98
C(45)-H(45A)	0.98
C(45)-H(45B)	0.98
C(45)-H(45C)	0.98
C(46)-H(46A)	0.98
C(46)-H(46B)	0.98
C(46)-H(46C)	0.98
N(2)-Si(4)	1.682(6)
N(2)-Si(3)	1.686(6)
Si(3)-C(83)	1.887(8)
Si(3)-C(81)	1.893(8)
Si(3)-C(82)	1.900(7)
Si(4)-C(85)	1.879(8)
Si(4)-C(86)	1.887(7)
Si(4)-C(84)	1.887(7)
C(81)-H(81A)	0.98
C(81)-H(81B)	0.98
C(81)-H(81C)	0.98
C(82)-H(82A)	0.98
C(82)-H(82B)	0.98
C(82)-H(82C)	0.98
C(83)-H(83A)	0.98
C(83)-H(83B)	0.98
C(83)-H(83C)	0.98
C(84)-H(84A)	0.98
C(84)-H(84B)	0.98
C(84)-H(84C)	0.98
C(85)-H(85A)	0.98
C(85)-H(85B)	0.98
C(85)-H(85C)	0.98
C(86)-H(86A)	0.98
C(86)-H(86B)	0.98
C(86)-H(86C)	0.98
N(1)-Ba(1)-N(2)	111.8(2)
N(1)-Ba(1)-C(2)	123.1(2)
N(2)-Ba(1)-C(2)	109.8(2)
N(1)-Ba(1)-C(1)	92.7(2)
N(2)-Ba(1)-C(1)	126.0(2)

C(2)-Ba(1)-C(1)	92.7(2)
N(1)-Ba(1)-Si(1)	25.34(11)
N(2)-Ba(1)-Si(1)	93.75(12)
C(2)-Ba(1)-Si(1)	148.47(13)
C(1)-Ba(1)-Si(1)	89.77(13)
N(1)-Ba(1)-Si(3)	97.44(12)
N(2)-Ba(1)-Si(3)	25.01(13)
C(2)-Ba(1)-Si(3)	104.74(13)
C(1)-Ba(1)-Si(3)	150.21(13)
Si(1)-Ba(1)-Si(3)	87.70(5)
N(1)-Ba(1)-Si(2)	23.79(11)
N(2)-Ba(1)-Si(2)	127.93(13)
C(2)-Ba(1)-Si(2)	99.38(13)
C(1)-Ba(1)-Si(2)	93.37(13)
Si(1)-Ba(1)-Si(2)	49.09(5)
Si(3)-Ba(1)-Si(2)	107.05(6)
N(1)-Ba(1)-Si(4)	120.85(11)
N(2)-Ba(1)-Si(4)	23.35(13)
C(2)-Ba(1)-Si(4)	112.83(13)
C(1)-Ba(1)-Si(4)	102.75(13)
Si(1)-Ba(1)-Si(4)	97.18(5)
Si(3)-Ba(1)-Si(4)	48.32(5)
Si(2)-Ba(1)-Si(4)	142.85(5)
P(1)-C(1)-Ba(1)	129.3(3)
P(1)-C(1)-H(1A)	104.9(2)
Ba(1)-C(1)-H(1A)	104.95(13)
P(1)-C(1)-H(1B)	104.9(2)
Ba(1)-C(1)-H(1B)	104.95(12)
H(1A)-C(1)-H(1B)	105.8
C(1)-P(1)-C(21)	110.0(3)
C(1)-P(1)-C(31)	109.8(3)
C(21)-P(1)-C(31)	109.2(3)
C(1)-P(1)-C(11)	119.6(3)
C(21)-P(1)-C(11)	104.2(3)
C(31)-P(1)-C(11)	103.5(3)
C(12)-C(11)-C(16)	119.4(6)
C(12)-C(11)-P(1)	117.4(5)
C(16)-C(11)-P(1)	123.0(5)
C(11)-C(12)-C(13)	120.1(7)
C(11)-C(12)-H(12A)	119.9(4)
C(13)-C(12)-H(12A)	119.9(5)
C(12)-C(13)-C(14)	121.0(7)
C(12)-C(13)-H(13A)	119.5(5)
C(14)-C(13)-H(13A)	119.5(5)
C(15)-C(14)-C(13)	118.5(7)
C(15)-C(14)-H(14A)	120.8(5)
C(13)-C(14)-H(14A)	120.8(5)
C(14)-C(15)-C(16)	121.0(7)
C(14)-C(15)-H(15A)	119.5(5)
C(16)-C(15)-H(15A)	119.5(4)
C(11)-C(16)-C(15)	119.9(6)
C(11)-C(16)-H(16A)	120.0(4)
C(15)-C(16)-H(16A)	120.0(4)
C(26)-C(21)-C(22)	117.3(6)
C(26)-C(21)-P(1)	119.6(5)
C(22)-C(21)-P(1)	123.1(5)
C(23)-C(22)-C(21)	122.3(7)
C(23)-C(22)-H(22A)	118.8(5)
C(21)-C(22)-H(22A)	118.8(4)
C(22)-C(23)-C(24)	119.1(7)
C(22)-C(23)-H(23A)	120.4(5)
C(24)-C(23)-H(23A)	120.4(4)
C(25)-C(24)-C(23)	119.3(7)
C(25)-C(24)-H(24A)	120.3(4)
C(23)-C(24)-H(24A)	120.3(4)
C(24)-C(25)-C(26)	120.6(7)
C(24)-C(25)-H(25A)	119.7(4)
C(26)-C(25)-H(25A)	119.7(4)
C(21)-C(26)-C(25)	121.3(6)
C(21)-C(26)-H(26A)	119.3(4)
C(25)-C(26)-H(26A)	119.3(4)
C(32)-C(31)-C(36)	118.4(6)
C(32)-C(31)-P(1)	119.9(5)
C(36)-C(31)-P(1)	121.8(5)
C(33)-C(32)-C(31)	120.9(7)
C(33)-C(32)-H(32A)	119.5(5)
C(31)-C(32)-H(32A)	119.5(4)
C(32)-C(33)-C(34)	119.3(8)
C(32)-C(33)-H(33A)	120.3(5)
C(34)-C(33)-H(33A)	120.3(5)
C(35)-C(34)-C(33)	120.5(8)
C(35)-C(34)-H(34A)	119.8(5)
C(33)-C(34)-H(34A)	119.8(5)
C(34)-C(35)-C(36)	120.8(8)
C(34)-C(35)-H(35A)	119.6(5)
C(36)-C(35)-H(35A)	119.6(5)
C(35)-C(36)-C(31)	120.1(8)
C(35)-C(36)-H(36A)	120.0(5)
C(31)-C(36)-H(36A)	120.0(4)
P(2)-C(2)-Ba(1)	133.8(3)
P(2)-C(2)-H(2A)	103.8(2)
Ba(1)-C(2)-H(2A)	103.77(13)
P(2)-C(2)-H(2B)	103.8(2)
Ba(1)-C(2)-H(2B)	103.77(13)
H(2A)-C(2)-H(2B)	105.4
C(2)-P(2)-C(61)	110.9(3)
C(2)-P(2)-C(51)	110.5(3)
C(61)-P(2)-C(51)	107.0(3)
C(2)-P(2)-C(71)	118.4(3)
C(61)-P(2)-C(71)	106.6(3)
C(51)-P(2)-C(71)	102.7(3)
C(52)-C(51)-C(56)	118.0(6)
C(52)-C(51)-P(2)	118.9(5)
C(56)-C(51)-P(2)	123.0(5)
C(53)-C(52)-C(51)	121.0(7)
C(53)-C(52)-H(52A)	119.5(5)
C(51)-C(52)-H(52A)	119.5(4)
C(52)-C(53)-C(54)	120.4(7)
C(52)-C(53)-H(53A)	119.8(5)
C(54)-C(53)-H(53A)	119.8(5)
C(55)-C(54)-C(53)	119.8(7)
C(55)-C(54)-H(54A)	120.1(5)
C(53)-C(54)-H(54A)	120.1(5)
C(54)-C(55)-C(56)	120.2(8)
C(54)-C(55)-H(55A)	119.9(5)
C(56)-C(55)-H(55A)	119.9(5)
C(55)-C(56)-C(51)	120.4(7)
C(55)-C(56)-H(56A)	119.8(5)
C(51)-C(56)-H(56A)	119.8(4)
C(62)-C(61)-C(66)	119.1(6)
C(62)-C(61)-P(2)	119.3(5)
C(66)-C(61)-P(2)	121.5(5)
C(63)-C(62)-C(61)	119.9(6)
C(63)-C(62)-H(62A)	120.1(4)
C(61)-C(62)-H(62A)	120.1(4)
C(62)-C(63)-C(64)	120.7(7)
C(62)-C(63)-H(63A)	119.6(4)
C(64)-C(63)-H(63A)	119.6(4)
C(65)-C(64)-C(63)	119.8(7)
C(65)-C(64)-H(64A)	120.1(4)
C(63)-C(64)-H(64A)	120.1(4)
C(64)-C(65)-C(66)	120.4(6)
C(64)-C(65)-H(65A)	119.8(4)
C(66)-C(65)-H(65A)	119.8(4)
C(65)-C(66)-C(61)	119.9(6)
C(65)-C(66)-H(66A)	120.0(4)
C(61)-C(66)-H(66A)	120.0(4)
C(72)-C(71)-C(76)	118.6(6)
C(72)-C(71)-P(2)	118.6(5)
C(76)-C(71)-P(2)	122.7(5)
C(73)-C(72)-C(71)	120.5(6)
C(73)-C(72)-H(72A)	119.7(4)
C(71)-C(72)-H(72A)	119.7(4)
C(74)-C(73)-C(72)	120.8(7)
C(74)-C(73)-H(73A)	119.6(4)
C(72)-C(73)-H(73A)	119.6(4)
C(73)-C(74)-C(75)	119.9(7)
C(73)-C(74)-H(74A)	120.0(4)

C(75)-C(74)-H(74A)	120.0(4)
C(76)-C(75)-C(74)	119.4(7)
C(76)-C(75)-H(75A)	120.3(4)
C(74)-C(75)-H(75A)	120.3(4)
C(75)-C(76)-C(71)	120.7(7)
C(75)-C(76)-H(76A)	119.6(4)
C(71)-C(76)-H(76A)	119.6(4)
Si(2)-N(1)-Si(1)	129.8(3)
Si(2)-N(1)-Ba(1)	117.0(2)
Si(1)-N(1)-Ba(1)	113.0(2)
N(1)-Si(1)-C(42)	114.2(3)
N(1)-Si(1)-C(43)	109.6(3)
C(42)-Si(1)-C(43)	105.4(4)
N(1)-Si(1)-C(41)	115.4(3)
C(42)-Si(1)-C(41)	106.0(4)
C(43)-Si(1)-C(41)	105.5(4)
N(1)-Si(1)-Ba(1)	41.7(2)
C(42)-Si(1)-Ba(1)	118.7(3)
C(43)-Si(1)-Ba(1)	68.6(2)
C(41)-Si(1)-Ba(1)	135.0(3)
N(1)-Si(2)-C(46)	114.9(4)
N(1)-Si(2)-C(44)	109.8(3)
C(46)-Si(2)-C(44)	105.9(4)
N(1)-Si(2)-C(45)	113.7(3)
C(46)-Si(2)-C(45)	105.6(5)
C(44)-Si(2)-C(45)	106.3(4)
N(1)-Si(2)-Ba(1)	39.2(2)
C(46)-Si(2)-Ba(1)	132.5(3)
C(44)-Si(2)-Ba(1)	71.0(2)
C(45)-Si(2)-Ba(1)	121.0(3)
Si(1)-C(41)-H(41A)	109.5(3)
Si(1)-C(41)-H(41B)	109.5(3)
H(41A)-C(41)-H(41B)	109.5
Si(1)-C(41)-H(41C)	109.5(3)
H(41A)-C(41)-H(41C)	109.47(10)
H(41B)-C(41)-H(41C)	109.5
Si(1)-C(42)-H(42A)	109.5(3)
Si(1)-C(42)-H(42B)	109.5(3)
H(42A)-C(42)-H(42B)	109.5
Si(1)-C(42)-H(42C)	109.5(3)
H(42A)-C(42)-H(42C)	109.5
H(42B)-C(42)-H(42C)	109.5
Si(1)-C(43)-H(43A)	109.5(2)
Si(1)-C(43)-H(43B)	109.5(2)
H(43A)-C(43)-H(43B)	109.47(6)
Si(1)-C(43)-H(43C)	109.5(2)
H(43A)-C(43)-H(43C)	109.5
H(43B)-C(43)-H(43C)	109.5
Si(2)-C(44)-H(44A)	109.5(2)
Si(2)-C(44)-H(44B)	109.5(3)
H(44A)-C(44)-H(44B)	109.5
Si(2)-C(44)-H(44C)	109.5(2)
H(44A)-C(44)-H(44C)	109.5
H(44B)-C(44)-H(44C)	109.5
Si(2)-C(45)-H(45A)	109.5(3)
Si(2)-C(45)-H(45B)	109.5(3)
H(45A)-C(45)-H(45B)	109.5
Si(2)-C(45)-H(45C)	109.5(3)
H(45A)-C(45)-H(45C)	109.47(8)
H(45B)-C(45)-H(45C)	109.5
Si(2)-C(46)-H(46A)	109.5(3)
Si(2)-C(46)-H(46B)	109.5(3)
H(46A)-C(46)-H(46B)	109.5
Si(2)-C(46)-H(46C)	109.5(3)
H(46A)-C(46)-H(46C)	109.5
H(46B)-C(46)-H(46C)	109.5
Si(4)-N(2)-Si(3)	128.0(3)
Si(4)-N(2)-Ba(1)	118.2(3)
Si(3)-N(2)-Ba(1)	113.6(3)
N(2)-Si(3)-C(83)	109.5(3)
N(2)-Si(3)-C(81)	114.5(4)
C(83)-Si(3)-C(81)	106.3(4)
N(2)-Si(3)-C(82)	114.5(3)
C(83)-Si(3)-C(82)	105.9(4)
C(81)-Si(3)-C(82)	105.6(4)
N(2)-Si(3)-Ba(1)	41.4(2)
C(83)-Si(3)-Ba(1)	68.1(2)
C(81)-Si(3)-Ba(1)	127.5(3)
C(82)-Si(3)-Ba(1)	126.5(3)
N(2)-Si(4)-C(85)	109.8(3)
N(2)-Si(4)-C(86)	115.6(3)
C(85)-Si(4)-C(86)	106.1(4)
N(2)-Si(4)-C(84)	115.1(4)
C(85)-Si(4)-C(84)	106.5(4)
C(86)-Si(4)-C(84)	102.9(4)
N(2)-Si(4)-Ba(1)	38.4(2)
C(85)-Si(4)-Ba(1)	72.8(2)
C(86)-Si(4)-Ba(1)	118.0(3)
C(84)-Si(4)-Ba(1)	137.8(3)
Si(3)-C(81)-H(81A)	109.5(3)
Si(3)-C(81)-H(81B)	109.5(3)
H(81A)-C(81)-H(81B)	109.5
Si(3)-C(81)-H(81C)	109.5(3)
H(81A)-C(81)-H(81C)	109.5
H(81B)-C(81)-H(81C)	109.47(6)
Si(3)-C(82)-H(82A)	109.5(3)
Si(3)-C(82)-H(82B)	109.5(3)
H(82A)-C(82)-H(82B)	109.5
Si(3)-C(82)-H(82C)	109.5(3)
H(82A)-C(82)-H(82C)	109.5
H(82B)-C(82)-H(82C)	109.5
Si(3)-C(83)-H(83A)	109.5(2)
Si(3)-C(83)-H(83B)	109.5(2)
H(83A)-C(83)-H(83B)	109.5
Si(3)-C(83)-H(83C)	109.5(2)
H(83A)-C(83)-H(83C)	109.5
H(83B)-C(83)-H(83C)	109.5
Si(4)-C(84)-H(84A)	109.5(3)
Si(4)-C(84)-H(84B)	109.5(3)
H(84A)-C(84)-H(84B)	109.47(5)
Si(4)-C(84)-H(84C)	109.5(3)
H(84A)-C(84)-H(84C)	109.5
H(84B)-C(84)-H(84C)	109.5
Si(4)-C(85)-H(85A)	109.5(3)
Si(4)-C(85)-H(85B)	109.5(2)
H(85A)-C(85)-H(85B)	109.5
Si(4)-C(85)-H(85C)	109.5(2)
H(85A)-C(85)-H(85C)	109.5
H(85B)-C(85)-H(85C)	109.5
Si(4)-C(86)-H(86A)	109.5(3)
Si(4)-C(86)-H(86B)	109.5(3)
H(86A)-C(86)-H(86B)	109.5
Si(4)-C(86)-H(86C)	109.5(3)
H(86A)-C(86)-H(86C)	109.5
H(86B)-C(86)-H(86C)	109.47(6)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 11. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ba(1)	37(1)	25(1)	30(1)	1(1)	5(1)	3(1)
C(1)	44(4)	30(4)	40(4)	-4(3)	0(3)	-3(3)
P(1)	39(1)	28(1)	32(1)	-2(1)	2(1)	-3(1)
C(11)	38(4)	23(4)	33(4)	1(3)	0(3)	-1(3)
C(12)	84(6)	52(5)	53(5)	-21(4)	32(5)	-32(5)
C(13)	107(8)	74(7)	72(6)	-24(5)	50(6)	-47(6)
C(14)	74(6)	44(5)	84(7)	-7(5)	20(5)	-26(4)
C(15)	68(5)	41(5)	56(5)	-21(4)	9(4)	-8(4)
C(16)	58(5)	37(4)	40(4)	-3(3)	11(4)	-15(4)
C(21)	37(4)	28(4)	31(4)	-2(3)	2(3)	0(3)
C(22)	77(6)	39(5)	71(6)	-10(4)	-32(5)	5(4)
C(23)	66(5)	58(6)	91(6)	-11(5)	-46(5)	11(5)
C(24)	44(5)	44(5)	57(5)	7(4)	-6(4)	11(4)
C(25)	50(5)	28(4)	68(5)	0(4)	-2(4)	3(4)
C(26)	36(4)	38(4)	52(4)	-4(4)	-12(3)	-4(4)
C(31)	39(4)	32(4)	36(4)	-7(3)	7(3)	-9(3)
C(32)	47(5)	80(7)	58(5)	19(5)	5(4)	8(4)
C(33)	45(5)	112(8)	73(6)	-2(6)	13(5)	22(5)
C(34)	65(6)	77(7)	67(6)	-30(5)	33(5)	-23(5)
C(35)	89(7)	53(6)	67(6)	-4(5)	29(6)	-16(5)
C(36)	55(5)	42(5)	53(5)	-1(4)	22(4)	4(4)
C(2)	45(4)	34(4)	45(4)	7(3)	7(3)	1(3)
P(2)	39(1)	30(1)	34(1)	5(1)	5(1)	2(1)
C(51)	35(4)	42(4)	22(3)	8(3)	7(3)	-2(3)
C(52)	42(4)	47(5)	56(5)	11(4)	3(4)	4(4)
C(53)	31(4)	80(7)	70(6)	21(5)	-11(4)	-9(4)
C(54)	51(5)	90(7)	39(5)	2(5)	5(4)	-40(5)
C(55)	66(6)	64(6)	43(5)	-14(4)	15(4)	-27(5)
C(56)	48(5)	55(5)	42(4)	-7(4)	6(4)	-8(4)
C(61)	33(4)	28(4)	32(4)	-1(3)	1(3)	-4(3)
C(62)	37(4)	25(4)	43(4)	2(3)	3(3)	-4(3)
C(63)	44(4)	39(4)	53(5)	-6(4)	13(4)	-1(3)
C(64)	47(5)	30(4)	71(6)	3(4)	10(4)	5(3)
C(65)	43(4)	46(5)	47(5)	25(4)	-4(4)	6(4)
C(66)	41(4)	33(4)	41(4)	11(3)	4(3)	1(3)
C(71)	34(4)	38(4)	35(4)	6(3)	4(3)	5(3)
C(72)	40(4)	40(4)	32(4)	5(3)	2(3)	7(3)
C(73)	51(5)	59(5)	34(4)	-7(4)	4(4)	7(4)
C(74)	46(4)	64(6)	34(4)	0(4)	13(4)	5(4)
C(75)	53(5)	59(5)	41(4)	10(4)	5(4)	-10(4)
C(76)	55(5)	44(5)	35(4)	1(3)	10(4)	-4(4)
N(1)	37(3)	31(3)	37(3)	2(3)	1(3)	4(2)
Si(1)	53(1)	32(1)	42(1)	8(1)	8(1)	7(1)
Si(2)	49(1)	48(1)	49(1)	12(1)	10(1)	18(1)
C(41)	172(10)	32(5)	93(7)	27(5)	49(7)	13(6)
C(42)	72(6)	96(7)	40(5)	-2(5)	-10(4)	-2(5)
C(43)	52(5)	64(6)	53(5)	8(4)	20(4)	-11(4)
C(44)	67(5)	63(6)	61(5)	11(4)	27(4)	23(4)
C(45)	126(8)	47(5)	92(7)	-4(5)	48(6)	32(5)
C(46)	55(6)	147(10)	77(7)	37(6)	4(5)	6(6)
N(2)	58(4)	27(3)	37(3)	-2(3)	16(3)	6(3)
Si(3)	77(2)	34(1)	38(1)	-5(1)	18(1)	-8(1)
Si(4)	67(2)	45(1)	40(1)	0(1)	16(1)	22(1)
C(81)	140(9)	35(5)	90(7)	-3(5)	30(6)	-16(5)
C(82)	95(7)	72(6)	45(5)	-11(4)	7(5)	-12(5)
C(83)	69(6)	57(5)	61(5)	-5(4)	19(4)	-19(4)
C(84)	99(7)	62(6)	70(6)	-25(5)	-7(5)	43(5)
C(85)	64(6)	64(6)	73(6)	-14(5)	20(5)	3(4)
C(86)	81(7)	117(8)	63(6)	17(5)	40(5)	29(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 11.

	x	y	z	U(eq)
H(1A)	157(7)	8230(3)	7939(2)	47
H(1B)	-1092(7)	8648(3)	7943(2)	47
H(12A)	1712(8)	9097(3)	8258(3)	73
H(13A)	3045(9)	9908(4)	8456(4)	96
H(14A)	3005(9)	10414(3)	9286(4)	80
H(15A)	1626(8)	10090(3)	9914(3)	66
H(16A)	268(7)	9282(3)	9715(3)	54
H(22A)	2268(8)	8388(3)	9668(3)	80
H(23A)	3692(8)	7670(4)	10069(4)	93
H(24A)	3346(7)	6731(3)	9731(3)	60
H(25A)	1543(7)	6541(3)	9027(3)	60
H(26A)	125(6)	7267(3)	8632(3)	53
H(32A)	-2650(8)	9048(4)	8621(3)	74
H(33A)	-4485(8)	9150(4)	9109(4)	92
H(34A)	-4454(9)	8680(4)	9959(4)	81
H(35A)	-2643(10)	8109(4)	10308(4)	81
H(36A)	-784(8)	8009(3)	9835(3)	59
H(2A)	-2196(7)	7126(3)	9008(3)	50
H(2B)	-3797(7)	7110(3)	8830(3)	50
H(52A)	-5712(7)	6439(3)	8510(3)	59
H(53A)	-7353(8)	5915(4)	7947(3)	75
H(54A)	-6950(8)	4964(4)	7751(3)	72
H(55A)	-4829(9)	4562(4)	8068(3)	68
H(56A)	-3141(7)	5089(3)	8622(3)	58
H(62A)	-1397(6)	5888(3)	8215(3)	42
H(63A)	656(7)	5397(3)	8239(3)	54
H(64A)	1993(7)	5168(3)	9093(3)	59
H(65A)	1257(7)	5416(3)	9927(3)	56
H(66A)	-837(7)	5884(3)	9917(3)	46
H(72A)	-3481(6)	7047(3)	9875(3)	45
H(73A)	-4196(7)	6991(3)	10744(3)	58
H(74A)	-4744(7)	6122(3)	11100(3)	57
H(75A)	-4392(7)	5281(3)	10620(3)	62
H(76A)	-3596(7)	5331(3)	9760(3)	53
H(41A)	-3308(55)	9257(5)	6634(12)	144
H(41B)	-4773(11)	9215(6)	6248(26)	144
H(41C)	-3406(62)	9134(4)	5978(17)	144
H(42A)	-5921(15)	8106(17)	5811(13)	107
H(42B)	-4989(43)	7546(4)	5870(11)	107
H(42C)	-4624(32)	8064(19)	5490(4)	107
H(43A)	-1477(10)	8169(15)	6729(10)	83
H(43B)	-1833(18)	8144(16)	6061(9)	83
H(43C)	-2100(12)	7601(3)	6428(18)	83
H(44A)	-4777(21)	7989(16)	8314(8)	93
H(44B)	-5762(48)	7525(4)	7977(4)	93
H(44C)	-6404(31)	8067(14)	8239(11)	93
H(45A)	-4470(17)	9196(6)	7754(22)	128
H(45B)	-6024(47)	9203(7)	7866(19)	128
H(45C)	-5678(57)	9352(3)	7258(6)	128
H(46A)	-7444(24)	8530(19)	6660(16)	140
H(46B)	-8032(8)	8351(27)	7215(8)	140
H(46C)	-7474(24)	7874(8)	6836(22)	140
H(81A)	-2711(43)	5174(6)	6719(21)	131
H(81B)	-4311(21)	5205(7)	6759(22)	131
H(81C)	-3203(60)	5335(3)	7300(4)	131
H(82A)	-3616(53)	6559(5)	5772(3)	106
H(82B)	-4677(20)	6043(22)	5763(3)	106
H(82C)	-3088(36)	5916(17)	5757(3)	106
H(83A)	-5151(28)	6853(8)	6776(17)	92
H(83B)	-4901(21)	6474(19)	7331(3)	92
H(83C)	-5810(11)	6231(12)	6777(17)	92
H(84A)	-160(45)	5471(7)	6604(12)	119
H(84B)	-25(50)	5497(8)	7271(14)	119
H(84C)	1240(10)	5679(3)	6970(23)	119
H(85A)	503(33)	7255(4)	7407(14)	99
H(85B)	1709(9)	6810(17)	7369(13)	99

H(85C)	676(38)	6710(14)	7807(3)	99
H(86A)	-616(42)	6484(19)	5893(3)	126
H(86B)	946(19)	6589(23)	6164(10)	126
H(86C)	-150(59)	7091(5)	6156(11)	126

12) [5.6'(5',8'-dihydroxy-1'4'-naphthoquinone)]-2-methylpent-2-ene acrylate phenyl

Table 1. Crystal data and structure refinement for 12.

Identification code	12 (98ckb009)
Empirical formula	C25 H22 O6
Formula weight	418.43
Temperature	100(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)
Unit cell dimensions	a = 6.16770(10) A alpha = 90 deg. b = 15.7416(3) A beta = 90.7630(10) deg. c = 10.5141(2) A gamma = 90 deg.
Volume, Z	1020.72(3) A^3, 2
Density (calculated)	1.361 Mg/m^3
Absorption coefficient	0.097 mm^-1
F(000)	440
Crystal size	0.5 x 0.1 x 0.1 mm
Theta range for data collection	1.94 to 27.50 deg.
Limiting indices	-8<=h<=7, -20<=k<=20, -13<=l<=11
Reflections collected	7466
Independent reflections	4360 [R(int) = 0.0276]
Absorption correction	Psi-scan corrections
Max. and min. transmission	1.000 and 0.885
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4346 / 1 / 306
Goodness-of-fit on F^2	1.125
Final R indices [I>2sigma(I)]	R1 = 0.0403, wR2 = 0.0847
R indices (all data)	R1 = 0.0489, wR2 = 0.0965
Absolute structure parameter	0.3(9)
Largest diff. peak and hole	0.260 and -0.259 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 12. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	-13106(3)	12875(1)	5472(2)	27(1)
O(2)	-9628(2)	12750(1)	4834(1)	17(1)
C(1)	-7255(4)	14323(1)	7774(2)	17(1)
C(2)	-5254(4)	14714(1)	7579(2)	21(1)
C(3)	-4176(4)	15134(2)	8576(2)	25(1)
C(4)	-5095(4)	15152(2)	9777(2)	25(1)
C(5)	-7091(4)	14767(2)	9982(2)	22(1)
C(6)	-8186(4)	14356(1)	8995(2)	19(1)
C(7)	-8284(4)	13851(1)	6717(2)	18(1)
C(8)	-10314(4)	13560(2)	6664(2)	19(1)
C(9)	-11200(3)	13040(2)	5617(2)	18(1)
C(10)	-10309(3)	12192(1)	3801(2)	15(1)
C(11)	-10287(4)	11270(1)	4277(2)	18(1)
C(12)	-8100(4)	10963(1)	4753(2)	18(1)
C(13)	-7729(4)	10394(1)	5671(2)	18(1)
C(14)	-5448(4)	10139(2)	6043(2)	26(1)
C(15)	-9486(4)	9958(2)	6417(2)	24(1)
C(20)	-8784(3)	12354(1)	2715(2)	15(1)
C(21)	-9396(3)	12035(1)	1490(2)	14(1)
O(21)	-11281(2)	11604(1)	1399(2)	19(1)
C(22)	-8055(3)	12163(1)	442(2)	15(1)
C(23)	-8688(4)	11830(1)	-810(2)	18(1)
O(23)	-10414(3)	11429(1)	-989(2)	23(1)
C(24)	-7190(4)	11965(1)	-1875(2)	20(1)
C(25)	-5308(4)	12379(1)	-1722(2)	20(1)
C(26)	-4643(3)	12747(1)	-488(2)	16(1)
O(26)	-2906(2)	13154(1)	-385(2)	21(1)
C(27)	-6072(4)	12617(1)	599(2)	14(1)
C(28)	-5498(3)	12935(1)	1799(2)	15(1)
O(28)	-3659(2)	13386(1)	2004(1)	19(1)
C(29)	-6862(3)	12793(1)	2850(2)	15(1)

Table 3. Bond lengths [A] and angles [deg] for 12.

O(1)-C(9)	1.211(3)
O(2)-C(9)	1.359(3)
O(2)-C(10)	1.456(2)
C(1)-C(2)	1.397(3)
C(1)-C(6)	1.414(3)
C(1)-C(7)	1.473(3)
C(2)-C(3)	1.399(3)
C(2)-H(2)	0.95
C(3)-C(4)	1.391(3)
C(3)-H(3)	0.95
C(4)-C(5)	1.392(4)
C(4)-H(4)	0.95
C(5)-C(6)	1.390(3)
C(5)-H(5)	0.95
C(6)-H(6)	0.95
C(7)-C(8)	1.333(3)
C(7)-H(7)	0.95
C(8)-C(9)	1.472(3)
C(8)-H(8)	0.95
C(10)-C(20)	1.510(3)
C(10)-C(11)	1.536(3)
C(10)-H(10)	1.00
C(11)-C(12)	1.511(3)
C(11)-H(11A)	0.99
C(11)-H(11B)	0.99
C(12)-C(13)	1.334(3)
C(12)-H(12)	0.95
C(13)-C(14)	1.509(3)
C(13)-C(15)	1.511(3)
C(14)-H(14C)	0.98
C(14)-H(14B)	0.98
C(14)-H(14A)	0.98
C(15)-H(15C)	0.98
C(15)-H(15B)	0.98
C(15)-H(15A)	0.98
C(20)-C(29)	1.378(3)
C(20)-C(21)	1.429(3)
C(21)-O(21)	1.348(2)
C(21)-C(22)	1.401(3)
O(21)-H(21)	0.84
C(22)-C(27)	1.424(3)
C(22)-C(23)	1.465(3)
C(23)-O(23)	1.250(3)
C(23)-C(24)	1.476(3)
C(24)-C(25)	1.340(3)
C(24)-H(24)	0.95
C(25)-C(26)	1.474(3)
C(25)-H(25)	0.95
C(26)-O(26)	1.252(3)
C(26)-C(27)	1.467(3)
C(27)-C(28)	1.399(3)

C(28)-O(28)	1.354(2)
C(28)-C(29)	1.415(3)
O(28)-H(28)	0.84
C(29)-H(29)	0.95
C(9)-O(2)-C(10)	117.0(2)
C(2)-C(1)-C(6)	119.1(2)
C(2)-C(1)-C(7)	119.0(2)
C(6)-C(1)-C(7)	121.8(2)
C(1)-C(2)-C(3)	120.7(2)
C(1)-C(2)-H(2)	119.66(12)
C(3)-C(2)-H(2)	119.66(14)
C(4)-C(3)-C(2)	119.6(2)
C(4)-C(3)-H(3)	120.19(14)
C(2)-C(3)-H(3)	120.19(14)
C(3)-C(4)-C(5)	120.2(2)
C(3)-C(4)-H(4)	119.89(14)
C(5)-C(4)-H(4)	119.89(13)
C(6)-C(5)-C(4)	120.6(2)
C(6)-C(5)-H(5)	119.69(14)
C(4)-C(5)-H(5)	119.69(13)
C(5)-C(6)-C(1)	119.7(2)
C(5)-C(6)-H(6)	120.15(14)
C(1)-C(6)-H(6)	120.15(13)
C(8)-C(7)-C(1)	126.9(2)
C(8)-C(7)-H(7)	116.57(13)
C(1)-C(7)-H(7)	116.57(12)
C(7)-C(8)-C(9)	124.2(2)
C(7)-C(8)-H(8)	117.91(13)
C(9)-C(8)-H(8)	117.91(12)
O(1)-C(9)-O(2)	123.4(2)
O(1)-C(9)-C(8)	124.3(2)
O(2)-C(9)-C(8)	112.2(2)
O(2)-C(10)-C(20)	106.6(2)
O(2)-C(10)-C(11)	109.1(2)
C(20)-C(10)-C(11)	113.8(2)
O(2)-C(10)-H(10)	109.10(10)
C(20)-C(10)-H(10)	109.10(11)
C(11)-C(10)-H(10)	109.10(11)
C(12)-C(11)-C(10)	114.4(2)
C(12)-C(11)-H(11A)	108.65(12)
C(10)-C(11)-H(11A)	108.65(11)
C(12)-C(11)-H(11B)	108.65(12)
C(10)-C(11)-H(11B)	108.65(11)
H(11A)-C(11)-H(11B)	107.6
C(13)-C(12)-C(11)	126.7(2)
C(13)-C(12)-H(12)	116.65(13)
C(11)-C(12)-H(12)	116.65(12)
C(12)-C(13)-C(14)	121.1(2)
C(12)-C(13)-C(15)	124.3(2)
C(14)-C(13)-C(15)	114.7(2)
C(13)-C(14)-H(14C)	109.47(13)
C(13)-C(14)-H(14B)	109.47(13)
H(14C)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14A)	109.47(12)
H(14C)-C(14)-H(14A)	109.5
H(14B)-C(14)-H(14A)	109.5
C(13)-C(15)-H(15C)	109.47(13)
C(13)-C(15)-H(15B)	109.47(12)
H(15C)-C(15)-H(15B)	109.5
C(13)-C(15)-H(15A)	109.47(12)
H(15C)-C(15)-H(15A)	109.5
H(15B)-C(15)-H(15A)	109.5
C(29)-C(20)-C(21)	119.0(2)
C(29)-C(20)-C(10)	123.4(2)
C(21)-C(20)-C(10)	117.5(2)
O(21)-C(21)-C(22)	122.3(2)
O(21)-C(21)-C(20)	117.3(2)
C(22)-C(21)-C(20)	120.5(2)
C(21)-O(21)-H(21)	109.47(11)
C(21)-C(22)-C(27)	119.7(2)
C(21)-C(22)-C(23)	120.1(2)
C(27)-C(22)-C(23)	120.1(2)
O(23)-C(23)-C(22)	122.2(2)
O(23)-C(23)-C(24)	119.9(2)
C(22)-C(23)-C(24)	117.9(2)
C(25)-C(24)-C(23)	121.9(2)
C(25)-C(24)-H(24)	119.03(13)
C(23)-C(24)-H(24)	119.03(12)
C(24)-C(25)-C(26)	121.8(2)
C(24)-C(25)-H(25)	119.09(13)
C(26)-C(25)-H(25)	119.09(12)
O(26)-C(26)-C(27)	121.8(2)
O(26)-C(26)-C(25)	120.3(2)
C(27)-C(26)-C(25)	117.9(2)
C(28)-C(27)-C(22)	119.4(2)
C(28)-C(27)-C(26)	120.4(2)
C(22)-C(27)-C(26)	120.2(2)
O(28)-C(28)-C(27)	122.2(2)
O(28)-C(28)-C(29)	117.7(2)
C(27)-C(28)-C(29)	120.1(2)
C(28)-O(28)-H(28)	109.47(11)
C(20)-C(29)-C(28)	121.3(2)
C(20)-C(29)-H(29)	119.37(12)
C(28)-C(29)-H(29)	119.37(11)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 12. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	16(1)	46(1)	19(1)	-7(1)	2(1)	-2(1)
O(2)	15(1)	22(1)	14(1)	-3(1)	2(1)	-1(1)
C(1)	21(1)	14(1)	17(1)	-1(1)	1(1)	3(1)
C(2)	23(1)	20(1)	19(1)	0(1)	2(1)	1(1)
C(3)	24(1)	18(1)	32(1)	-1(1)	-2(1)	-3(1)
C(4)	32(1)	18(1)	25(1)	-5(1)	-5(1)	0(1)
C(5)	34(1)	18(1)	16(1)	-2(1)	2(1)	4(1)
C(6)	21(1)	17(1)	19(1)	1(1)	0(1)	2(1)
C(7)	22(1)	19(1)	12(1)	0(1)	4(1)	2(1)
C(8)	21(1)	23(1)	14(1)	-1(1)	2(1)	4(1)
C(9)	18(1)	24(1)	13(1)	3(1)	3(1)	2(1)
C(10)	14(1)	19(1)	11(1)	0(1)	-1(1)	-1(1)
C(11)	18(1)	19(1)	18(1)	4(1)	1(1)	-3(1)
C(12)	16(1)	22(1)	17(1)	1(1)	4(1)	0(1)
C(13)	17(1)	18(1)	18(1)	-4(1)	3(1)	1(1)
C(14)	18(1)	30(1)	31(1)	12(1)	0(1)	1(1)
C(15)	21(1)	26(1)	27(1)	9(1)	4(1)	0(1)
C(20)	14(1)	14(1)	15(1)	3(1)	0(1)	2(1)
C(21)	14(1)	13(1)	15(1)	-1(1)	-2(1)	1(1)
O(21)	18(1)	22(1)	18(1)	-4(1)	-1(1)	-4(1)
C(22)	16(1)	12(1)	16(1)	-1(1)	-1(1)	2(1)
C(23)	22(1)	14(1)	17(1)	1(1)	-2(1)	3(1)
O(23)	24(1)	26(1)	18(1)	-5(1)	-2(1)	-3(1)
C(24)	29(1)	18(1)	12(1)	0(1)	1(1)	4(1)
C(25)	26(1)	20(1)	14(1)	1(1)	6(1)	4(1)
C(26)	19(1)	14(1)	16(1)	3(1)	2(1)	3(1)
O(26)	20(1)	22(1)	21(1)	2(1)	4(1)	-2(1)
C(27)	18(1)	14(1)	11(1)	1(1)	2(1)	1(1)
C(28)	15(1)	11(1)	18(1)	1(1)	0(1)	1(1)
O(28)	17(1)	23(1)	16(1)	-1(1)	3(1)	-5(1)
C(29)	17(1)	15(1)	13(1)	-2(1)	0(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 12.

	x	y	z	U(eq)
H(2)	-4619(4)	14696(1)	6761(2)	12(6)
H(3)	-2824(4)	15404(2)	8434(2)	43(9)
H(4)	-4357(4)	15429(2)	10459(2)	36(8)
H(5)	-7711(4)	14785(2)	10803(2)	26(7)
H(6)	-9554(4)	14099(1)	9140(2)	27(7)
H(7)	-7410(4)	13742(1)	5998(2)	14(6)
H(8)	-11247(4)	13698(2)	7345(2)	28(7)
H(10)	-11817(3)	12345(1)	3527(2)	8(5)
H(11A)	-11341(4)	11215(1)	4974(2)	16(6)
H(11B)	-10779(4)	10894(1)	3575(2)	36(8)
H(12)	-6863(4)	11200(1)	4357(2)	38(8)
H(14C)	-5255(10)	9530(3)	5894(16)	32(8)
H(14B)	-5195(11)	10264(11)	6946(5)	40(8)
H(14A)	-4412(4)	10459(9)	5531(12)	46(9)
H(15C)	-9518(31)	10192(13)	7280(9)	73(12)
H(15B)	-9187(25)	9347(3)	6459(23)	84(14)
H(15A)	-10892(7)	10052(15)	5995(15)	134(20)
H(21)	-11405(27)	11391(19)	670(11)	50(10)
H(24)	-7572(4)	11751(1)	-2692(2)	24(7)
H(25)	-4376(4)	12438(1)	-2429(2)	23(7)
H(28)	-2970(24)	13424(16)	1323(7)	35(8)
H(29)	-6445(3)	13004(1)	3663(2)	11(6)

13) (2-methyl(diethanoic acid)amine)phenoxyethanoic acid

Table 1. Crystal data and structure refinement for 13.

Identification code	13 (98srv111)
Empirical formula	C13 H15 N O7
Formula weight	297.26
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 10.6417(3) A alpha = 90 deg. b = 9.6898(3) A beta = 94.031(2) deg. c = 13.1969(4) A gamma = 90 deg.
Volume, Z	1357.44(7) A^3, 4
Density (calculated)	1.455 Mg/m^3
Absorption coefficient	0.120 mm^-1
F(000)	624
Crystal size	0.25 x 0.20 x 0.05 mm
Theta range for data collection	2.38 to 27.49 deg.
Limiting indices	-13<=h<=13, -12<=k<=12, -15<=l<=17
Reflections collected	11644
Independent reflections	3110 [R(int) = 0.2209]
Absorption correction	Multi-scan
Max. and min. transmission	1.000000 and 0.511018
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3048 / 0 / 208
Goodness-of-fit on F^2	0.991
Final R indices [I>2sigma(I)]	R1 = 0.0923, wR2 = 0.1765
R indices (all data)	R1 = 0.2747, wR2 = 0.2724
Extinction coefficient	not refined
Largest diff. peak and hole	0.469 and -0.269 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 13. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	-1192(5)	3743(6)	8813(4)	39(2)
C(2)	-526(5)	4970(6)	8708(4)	39(2)
C(3)	-1171(6)	6215(7)	8705(4)	45(2)
C(4)	-2457(6)	6248(7)	8822(4)	50(2)
C(5)	-3102(6)	5024(7)	8927(5)	50(2)
C(6)	-2479(6)	3776(7)	8913(4)	47(2)
O(10)	-487(4)	2552(4)	8803(3)	47(1)
C(11)	-1086(6)	1241(6)	8714(5)	47(2)
C(12)	-1482(5)	678(7)	9699(5)	42(2)
O(121)	-1293(4)	1217(5)	10520(3)	65(1)
O(122)	-2066(5)	-514(5)	9540(4)	67(1)
C(20)	879(5)	4952(6)	8632(4)	38(2)
N(21)	1300(4)	4041(4)	7792(3)	34(1)
C(22)	705(6)	4427(6)	6760(4)	42(2)
C(23)	593(5)	3183(7)	6079(5)	45(2)
O(231)	807(4)	2030(5)	6417(3)	70(1)
O(232)	230(4)	3506(4)	5162(3)	62(1)
C(24)	2720(5)	4030(6)	7805(4)	39(2)
C(25)	3216(6)	2974(7)	8585(4)	40(2)
O(251)	2493(4)	2059(4)	8838(3)	47(1)
O(252)	4374(4)	3127(5)	8853(4)	67(1)

Table 3. Bond lengths [A] and angles [deg] for 13.

C(1)-O(10)	1.377(6)
C(1)-C(6)	1.385(8)
C(1)-C(2)	1.396(8)
C(2)-C(3)	1.388(8)
C(2)-C(20)	1.506(7)
C(3)-C(4)	1.388(8)
C(3)-H(3)	0.93
C(4)-C(5)	1.382(8)
C(4)-H(4)	0.93
C(5)-C(6)	1.380(8)
C(5)-H(5)	0.93
C(6)-H(6)	0.93
O(10)-C(11)	1.423(6)
C(11)-C(12)	1.497(8)
C(11)-H(11A)	0.97
C(11)-H(11B)	0.97
C(12)-O(121)	1.207(7)
C(12)-O(122)	1.321(7)
O(122)-H(12)	0.82
C(20)-N(21)	1.510(6)
C(20)-H(20A)	0.97
C(20)-H(20B)	0.97
N(21)-C(22)	1.508(6)
N(21)-C(24)	1.509(6)
C(22)-C(23)	1.503(8)
C(22)-H(22A)	0.97
C(22)-H(22B)	0.97
C(23)-O(231)	1.218(7)
C(23)-O(232)	1.283(7)
O(232)-H(23)	0.820(3)
C(24)-C(25)	1.519(8)
C(24)-H(24A)	0.97
C(24)-H(24B)	0.97
C(25)-O(251)	1.236(6)
C(25)-O(252)	1.266(6)
O(252)-H(25)	0.82
O(10)-C(1)-C(6)	124.3(5)
O(10)-C(1)-C(2)	115.6(5)
C(6)-C(1)-C(2)	120.1(6)
C(3)-C(2)-C(1)	119.1(6)
C(3)-C(2)-C(20)	120.2(5)
C(1)-C(2)-C(20)	120.7(5)
C(4)-C(3)-C(2)	120.7(6)
C(4)-C(3)-H(3)	119.6(4)
C(2)-C(3)-H(3)	119.6(4)
C(5)-C(4)-C(3)	119.5(6)
C(5)-C(4)-H(4)	120.3(4)
C(3)-C(4)-H(4)	120.3(4)
C(6)-C(5)-C(4)	120.5(6)
C(6)-C(5)-H(5)	119.8(4)
C(4)-C(5)-H(5)	119.8(4)
C(5)-C(6)-C(1)	120.1(6)
C(5)-C(6)-H(6)	120.0(4)
C(1)-C(6)-H(6)	120.0(4)
C(1)-O(10)-C(11)	120.5(4)
O(10)-C(11)-C(12)	113.9(5)
O(10)-C(11)-H(11A)	108.8(3)
C(12)-C(11)-H(11A)	108.8(3)
O(10)-C(11)-H(11B)	108.8(3)
C(12)-C(11)-H(11B)	108.8(3)
H(11A)-C(11)-H(11B)	107.7
O(121)-C(12)-O(122)	124.6(6)
O(121)-C(12)-C(11)	125.6(6)
O(122)-C(12)-C(11)	109.8(6)
C(12)-O(122)-H(12)	109.5(4)
C(2)-C(20)-N(21)	113.8(4)
C(2)-C(20)-H(20A)	108.8(3)
N(21)-C(20)-H(20A)	108.8(3)
C(2)-C(20)-H(20B)	108.8(3)

N(21)-C(20)-H(20B)	108.8(3)
H(20A)-C(20)-H(20B)	107.7
C(22)-N(21)-C(24)	111.7(4)
C(22)-N(21)-C(20)	113.0(4)
C(24)-N(21)-C(20)	110.1(4)
C(23)-C(22)-N(21)	110.6(5)
C(23)-C(22)-H(22A)	109.5(3)
N(21)-C(22)-H(22A)	109.5(3)
C(23)-C(22)-H(22B)	109.5(3)
N(21)-C(22)-H(22B)	109.5(3)
H(22A)-C(22)-H(22B)	108.1
O(231)-C(23)-O(232)	127.2(6)
O(231)-C(23)-C(22)	120.8(6)
O(232)-C(23)-C(22)	112.0(5)
C(23)-O(232)-H(23)	109.5(4)
N(21)-C(24)-C(25)	108.3(4)
N(21)-C(24)-H(24A)	110.0(3)
C(25)-C(24)-H(24A)	110.0(3)
N(21)-C(24)-H(24B)	110.0(3)
C(25)-C(24)-H(24B)	110.0(3)
H(24A)-C(24)-H(24B)	108.4
O(251)-C(25)-O(252)	128.3(6)
O(251)-C(25)-C(24)	118.2(5)
O(252)-C(25)-C(24)	113.3(5)
C(25)-O(252)-H(25)	109.5(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 13. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	35(4)	43(4)	40(4)	5(3)	2(3)	4(3)
C(2)	39(4)	42(4)	37(3)	2(3)	-3(3)	-8(3)
C(3)	53(4)	36(4)	45(4)	1(3)	-5(3)	-5(4)
C(4)	48(4)	46(5)	55(4)	-5(3)	-7(3)	8(4)
C(5)	34(4)	59(5)	56(4)	-1(4)	-7(3)	7(4)
C(6)	43(4)	42(4)	55(4)	4(3)	-3(3)	-6(3)
O(10)	41(2)	40(3)	60(3)	4(2)	6(2)	3(2)
C(11)	57(4)	37(4)	47(4)	4(3)	3(3)	-4(3)
C(12)	48(4)	37(4)	40(4)	10(3)	-5(3)	-7(3)
O(121)	83(4)	69(3)	44(3)	2(3)	1(2)	-17(3)
O(122)	93(4)	47(3)	59(3)	7(3)	3(3)	-20(3)
C(20)	42(3)	42(4)	28(3)	-6(3)	-3(3)	-5(3)
N(21)	31(3)	41(3)	28(3)	4(2)	-9(2)	0(2)
C(22)	45(4)	41(4)	38(3)	5(3)	-13(3)	-3(3)
C(23)	32(3)	51(4)	48(4)	3(4)	-13(3)	-10(3)
O(231)	100(4)	37(3)	67(3)	6(3)	-34(3)	-4(3)
O(232)	80(3)	54(3)	48(3)	2(2)	-29(2)	1(2)
C(24)	37(3)	46(4)	33(4)	8(3)	1(3)	-4(3)
C(25)	35(4)	48(4)	36(3)	3(3)	-2(3)	-3(3)
O(251)	47(3)	42(3)	53(3)	17(2)	-3(2)	0(2)
O(252)	42(3)	76(4)	79(4)	31(3)	-19(2)	-8(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 13.

	x	y	z	U(eq)
H(3)	-737(6)	7037(7)	8624(4)	51(18)
H(4)	-2881(6)	7087(7)	8830(4)	62(20)
H(5)	-3963(6)	5041(7)	9008(5)	28(14)
H(6)	-2923(6)	2957(7)	8971(4)	55(18)
H(11A)	-514(6)	591(6)	8430(5)	66(20)
H(11B)	-1824(6)	1318(6)	8242(5)	78(22)
H(12)	-2193(77)	-870(44)	10087(4)	133(41)
H(20A)	1279(5)	4636(6)	9274(4)	43(15)
H(20B)	1165(5)	5887(6)	8521(4)	38(16)
H(22A)	-125(6)	4814(6)	6833(4)	77(22)
H(22B)	1214(6)	5124(6)	6456(4)	49(18)
H(23)	637(828)	3064(965)	4768(18)	2000(1156)
H(24A)	3051(5)	4937(6)	7984(4)	41(16)
H(24B)	2982(5)	3788(6)	7139(4)	29(14)
H(25)	4586(22)	2557(65)	9292(53)	144(43)

14) Salicyldoxime benoxazolin-2-one

Table 1. Crystal data and structure refinement for 14.

Identification code	14 (99srv003)
Empirical formula	C14 H12 N2 O4
Formula weight	272.26
Temperature	153(2) K
Wavelength	1.54178 A
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 10.494(2) A alpha = 90 deg. b = 6.4345(13) A beta = 103.36(3) deg. c = 19.604(4) A gamma = 90 deg.
Volume, Z	1287.9(4) A^3, 4
Density (calculated)	1.404 Mg/m^3
Absorption coefficient	0.105 mm^-1
F(000)	568
Crystal size	0.35 x 0.12 x 0.06 mm
Theta range for data collection	2.03 to 26.44 deg.
Limiting indices	-1<=h<=13, -1<=k<=7, -24<=l<=24
Reflections collected	3192
Independent reflections	2254 [R(int) = 0.0209]
Absorption correction	None
Max. and min. transmission	1.0000 and 0.9075
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2247 / 0 / 205
Goodness-of-fit on F^2	1.102
Final R indices [I>2sigma(I)]	R1 = 0.0432, wR2 = 0.1118
R indices (all data)	R1 = 0.0618, wR2 = 0.1537
Extinction coefficient	not refined
Largest diff. peak and hole	0.282 and -0.261 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(11)	4545(1)	8815(2)	1063(1)	25(1)
O(12)	4249(2)	7555(2)	-42(1)	26(1)
N(11)	5265(2)	5644(3)	937(1)	23(1)
C(10)	4667(2)	7318(4)	591(1)	23(1)
C(11)	5101(2)	8015(4)	1727(1)	24(1)
C(12)	5554(2)	6034(4)	1657(1)	24(1)
C(13)	6133(2)	4876(4)	2239(1)	30(1)
C(14)	6199(2)	5809(5)	2890(1)	36(1)
C(15)	5721(2)	7807(4)	2946(1)	36(1)
C(16)	5157(2)	8968(4)	2360(1)	31(1)
O(21)	7061(2)	7198(3)	-1439(1)	30(1)
O(22)	7213(2)	9020(3)	522(1)	28(1)
N(21)	7310(2)	7930(3)	-85(1)	23(1)
C(20)	7915(2)	6218(4)	57(1)	24(1)
C(21)	7709(2)	5403(4)	-1219(1)	24(1)
C(22)	8138(2)	4874(4)	-506(1)	22(1)
C(23)	8792(2)	2989(4)	-331(1)	27(1)
C(24)	9045(2)	1654(4)	-839(1)	32(1)
C(25)	8616(2)	2203(4)	-1538(1)	31(1)
C(26)	7951(2)	4044(4)	-1728(1)	29(1)

Table 3. Bond lengths [A] and angles [deg] for 14.

O(11)-C(10)	1.362(3)
O(11)-C(11)	1.395(2)
O(12)-C(10)	1.226(2)
N(11)-C(10)	1.348(3)
N(11)-C(12)	1.396(3)
N(11)-H(11)	0.89(3)
C(11)-C(16)	1.373(3)
C(11)-C(12)	1.379(3)
C(12)-C(13)	1.380(3)
C(13)-C(14)	1.399(3)
C(13)-H(13)	0.95
C(14)-C(15)	1.394(4)
C(14)-H(14)	0.95
C(15)-C(16)	1.385(3)
C(15)-H(15)	0.95
C(16)-H(16)	0.95
O(21)-C(21)	1.359(3)
O(21)-H(21)	0.87(3)
O(22)-N(21)	1.405(2)
O(22)-H(22)	0.92(3)
N(21)-C(20)	1.270(3)
C(20)-C(22)	1.463(3)
C(20)-H(20)	0.97(2)
C(21)-C(26)	1.394(3)
C(21)-C(22)	1.408(3)
C(22)-C(23)	1.397(3)
C(23)-C(24)	1.387(3)
C(23)-H(23)	0.95
C(24)-C(25)	1.385(3)
C(24)-H(24)	0.95
C(25)-C(26)	1.382(4)
C(25)-H(25)	0.95
C(26)-H(26)	0.95
C(10)-O(11)-C(11)	106.7(2)
C(10)-N(11)-C(12)	109.4(2)
C(10)-N(11)-H(11)	126(2)
C(12)-N(11)-H(11)	125(2)
O(12)-C(10)-N(11)	128.6(2)
O(12)-C(10)-O(11)	122.1(2)
N(11)-C(10)-O(11)	109.3(2)
C(16)-C(11)-C(12)	124.0(2)
C(16)-C(11)-O(11)	126.8(2)
C(12)-C(11)-O(11)	109.1(2)
C(11)-C(12)-C(13)	120.9(2)
C(11)-C(12)-N(11)	105.5(2)
C(13)-C(12)-N(11)	133.6(2)
C(12)-C(13)-C(14)	116.3(2)
C(12)-C(13)-H(13)	121.85(14)
C(14)-C(13)-H(13)	121.9(2)
C(15)-C(14)-C(13)	121.7(2)
C(15)-C(14)-H(14)	119.17(13)
C(13)-C(14)-H(14)	119.2(2)
C(16)-C(15)-C(14)	121.7(2)
C(16)-C(15)-H(15)	119.2(2)
C(14)-C(15)-H(15)	119.16(13)
C(11)-C(16)-C(15)	115.5(2)
C(11)-C(16)-H(16)	122.3(2)
C(15)-C(16)-H(16)	122.3(2)
C(21)-O(21)-H(21)	105(2)
N(21)-O(22)-H(22)	106(2)
C(20)-N(21)-O(22)	112.2(2)
N(21)-C(20)-C(22)	120.4(2)
N(21)-C(20)-H(20)	119.6(14)
C(22)-C(20)-H(20)	120.0(14)
O(21)-C(21)-C(26)	117.9(2)
O(21)-C(21)-C(22)	122.8(2)
C(26)-C(21)-C(22)	119.3(2)
C(23)-C(22)-C(21)	118.7(2)
C(23)-C(22)-C(20)	118.9(2)
C(21)-C(22)-C(20)	122.4(2)
C(24)-C(23)-C(22)	121.7(2)
C(24)-C(23)-H(23)	119.14(14)
C(22)-C(23)-H(23)	119.14(12)
C(25)-C(24)-C(23)	118.7(2)
C(25)-C(24)-H(24)	120.7(2)
C(23)-C(24)-H(24)	120.66(14)

C(26)-C(25)-C(24)	121.0(2)
C(26)-C(25)-H(25)	119.50(13)
C(24)-C(25)-H(25)	119.5(2)
C(25)-C(26)-C(21)	120.5(2)
C(25)-C(26)-H(26)	119.73(13)
C(21)-C(26)-H(26)	119.73(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 14. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(11)	29(1)	23(1)	22(1)	-2(1)	7(1)	4(1)
O(12)	30(1)	25(1)	21(1)	-1(1)	6(1)	4(1)
N(11)	26(1)	20(1)	23(1)	1(1)	7(1)	4(1)
C(10)	22(1)	24(1)	24(1)	-1(1)	8(1)	1(1)
C(11)	23(1)	27(1)	25(1)	2(1)	8(1)	0(1)
C(12)	20(1)	29(1)	23(1)	0(1)	7(1)	-3(1)
C(13)	25(1)	33(1)	33(1)	7(1)	5(1)	-1(1)
C(14)	32(1)	49(2)	24(1)	9(1)	2(1)	-9(1)
C(15)	39(1)	45(2)	23(1)	-6(1)	7(1)	-10(1)
C(16)	32(1)	36(2)	28(1)	-5(1)	9(1)	-5(1)
O(21)	39(1)	26(1)	22(1)	3(1)	3(1)	5(1)
O(22)	34(1)	25(1)	23(1)	-3(1)	2(1)	5(1)
N(21)	24(1)	23(1)	21(1)	-2(1)	2(1)	0(1)
C(20)	25(1)	26(1)	21(1)	4(1)	3(1)	-1(1)
C(21)	21(1)	24(1)	26(1)	2(1)	5(1)	-2(1)
C(22)	19(1)	22(1)	24(1)	0(1)	4(1)	-3(1)
C(23)	25(1)	27(1)	27(1)	2(1)	2(1)	2(1)
C(24)	27(1)	26(1)	40(1)	-2(1)	5(1)	3(1)
C(25)	26(1)	31(1)	35(1)	-10(1)	10(1)	1(1)
C(26)	29(1)	35(1)	24(1)	-1(1)	5(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 14.

	x	y	z	U(eq)
H(11)	5452(26)	4471(46)	743(13)	36(7)
H(13)	6468(2)	3521(4)	2199(1)	37(7)
H(14)	6579(2)	5061(5)	3305(1)	55(9)
H(15)	5785(2)	8387(4)	3399(1)	37(7)
H(16)	4831(2)	10332(4)	2393(1)	35(7)
H(21)	6994(25)	7839(43)	-1057(14)	32(7)
H(22)	6763(27)	10218(48)	365(14)	40(8)
H(20)	8262(21)	5832(37)	542(11)	20(6)
H(23)	9070(2)	2611(4)	149(1)	23(6)
H(24)	9505(2)	389(4)	-710(1)	42(8)
H(25)	8781(2)	1301(4)	-1891(1)	36(7)
H(26)	7656(2)	4386(4)	-2211(1)	31(7)

15) Sodium bis(hexamethyldisilyamide)bis(triphenylethylphosphonium)

Table 1. Crystal data and structure refinement for 15.

Identification code	15 (97srv159)
Empirical formula	C26 H37 N Na P Si2
Formula weight	473.71
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Triclinic
Space group	P -1
Unit cell dimensions	a = 10.0623(8) A alpha = 105.774(4) deg. b = 10.3932(10) A beta = 99.236(4) deg. c = 14.2947(13) A gamma = 102.193(5) deg.
Volume, Z	1367.9(2) A^3, 2
Density (calculated)	1.150 Mg/m^3
Absorption coefficient	0.218 mm^-1
F(000)	508
Crystal size	1.00 x 0.95 x 0.25 mm
Theta range for data collection	1.52 to 30.32 deg.
Limiting indices	-10<=h<=13, -14<=k<=14, -20<=l<=19
Reflections collected	12314
Independent reflections	7130 [R(int) = 0.0334]
Absorption correction	Multiscan
Max. and min. transmission	1.0000 and 0.7896
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7092 / 0 / 428
Goodness-of-fit on F^2	1.117
Final R indices [I>2sigma(I)]	R1 = 0.0535, wR2 = 0.1131
R indices (all data)	R1 = 0.0887, wR2 = 0.1453
Largest diff. peak and hole	0.466 and -0.408 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 15. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
P(1)	2820(1)	5639(1)	1733(1)	18(1)
Si(1)	5870(1)	11709(1)	3694(1)	23(1)
Si(2)	7044(1)	8622(1)	6010(1)	24(1)
N(1)	5520(2)	9080(2)	5938(2)	23(1)
Na(1)	4471(1)	8614(1)	4102(1)	25(1)
C(11)	6720(3)	10462(3)	2948(2)	36(1)
C(12)	7307(3)	12905(3)	4800(2)	35(1)
C(13)	5530(4)	12837(3)	2896(2)	37(1)
C(21)	8265(4)	9602(4)	5423(3)	47(1)
C(22)	8098(3)	8965(4)	7319(2)	38(1)
C(23)	6835(3)	6725(3)	5337(2)	34(1)
C(30)	4293(2)	6299(2)	2681(2)	20(1)
C(31)	5724(3)	6462(3)	2428(2)	27(1)
C(41)	1317(2)	5675(2)	2278(2)	21(1)
C(42)	971(3)	4750(3)	2809(2)	28(1)
C(43)	-105(3)	4816(3)	3305(2)	36(1)
C(44)	-852(3)	5785(3)	3269(2)	39(1)
C(45)	-537(3)	6690(3)	2737(2)	38(1)
C(46)	547(3)	6638(3)	2234(2)	30(1)
C(51)	2375(2)	3866(2)	868(2)	20(1)
C(52)	3448(3)	3344(3)	532(2)	26(1)
C(53)	3139(3)	2049(3)	-186(2)	33(1)
C(54)	1759(3)	1246(3)	-574(2)	33(1)
C(55)	688(3)	1732(3)	-232(2)	31(1)
C(56)	986(3)	3039(3)	479(2)	26(1)
C(62)	2563(3)	6064(3)	-149(2)	25(1)
C(65)	3407(3)	8948(3)	660(2)	29(1)
C(63)	2684(3)	6888(3)	-768(2)	28(1)
C(66)	3301(3)	8131(3)	1289(2)	24(1)
C(64)	3108(3)	8320(3)	-367(2)	28(1)
C(61)	2879(2)	6677(2)	892(2)	20(1)

Table 3. Bond lengths [A] and angles [deg] for 15.

P(1)-C(30)	1.710(2)	C(11)-H(111)	0.97(4)
P(1)-C(41)	1.812(2)	C(11)-H(112)	0.92(4)
P(1)-C(61)	1.820(2)	C(11)-H(113)	1.03(4)
P(1)-C(51)	1.833(2)	C(12)-Na(1)#1	3.035(3)
Si(1)-N(1)#1	1.701(2)	C(12)-H(121)	1.04(4)
Si(1)-C(11)	1.887(3)	C(12)-H(122)	0.92(3)
Si(1)-C(13)	1.887(3)	C(12)-H(123)	0.96(4)
Si(1)-C(12)	1.890(3)	C(13)-H(131)	0.96(4)
Si(1)-Na(1)#1	3.3207(12)	C(13)-H(132)	1.00(4)
Si(1)-Na(1)	3.4699(12)	C(13)-H(133)	0.95(4)
Si(2)-N(1)	1.695(2)	C(21)-H(211)	0.95(4)
Si(2)-C(21)	1.875(3)	C(21)-H(212)	0.95(5)
Si(2)-C(23)	1.894(3)	C(21)-H(213)	0.93(5)
Si(2)-C(22)	1.896(3)	C(22)-H(221)	0.94(3)
Si(2)-Na(1)	3.4430(12)	C(22)-H(222)	0.97(4)
N(1)-Si(1)#1	1.701(2)	C(22)-H(223)	0.95(4)
N(1)-Na(1)#1	2.411(2)	C(23)-H(231)	0.99(4)
N(1)-Na(1)	2.537(2)	C(23)-H(232)	1.00(4)
Na(1)-N(1)#1	2.411(2)	C(23)-H(233)	0.97(4)
Na(1)-C(30)	2.645(3)	C(30)-C(31)	1.527(3)
Na(1)-C(12)#1	3.035(3)	C(30)-H(30)	0.95(3)
Na(1)-Na(1)#1	3.135(2)	C(31)-H(311)	0.97(3)
Na(1)-Si(1)#1	3.3207(12)	C(31)-H(312)	0.99(3)
		C(31)-H(313)	0.97(3)
		C(41)-C(46)	1.397(3)
		C(41)-C(42)	1.400(3)
		C(42)-C(43)	1.389(4)
		C(42)-H(42)	0.97(3)

C(43)-C(44)	1.385(4)
C(43)-H(43)	0.99(4)
C(44)-C(45)	1.380(5)
C(44)-H(44)	0.96(3)
C(45)-C(46)	1.402(4)
C(45)-H(45)	0.97(4)
C(46)-H(46)	0.92(3)
C(51)-C(52)	1.402(3)
C(51)-C(56)	1.408(3)
C(52)-C(53)	1.389(4)
C(52)-H(52)	0.99(3)
C(53)-C(54)	1.394(4)
C(53)-H(53)	0.89(3)
C(54)-C(55)	1.386(4)
C(54)-H(54)	1.06(3)
C(55)-C(56)	1.396(3)
C(55)-H(55)	1.03(3)
C(56)-H(56)	0.92(3)
C(62)-C(63)	1.391(4)
C(62)-C(61)	1.402(3)
C(62)-H(62)	1.00(3)
C(65)-C(64)	1.388(4)
C(65)-C(66)	1.395(3)
C(65)-H(65)	0.98(3)
C(63)-C(64)	1.384(4)
C(63)-H(63)	0.93(3)
C(66)-C(61)	1.404(3)
C(66)-H(66)	0.96(3)
C(64)-H(64)	0.99(3)

C(30)-P(1)-C(41)	108.19(11)
C(30)-P(1)-C(61)	109.68(11)
C(41)-P(1)-C(61)	110.57(11)
C(30)-P(1)-C(51)	121.98(11)
C(41)-P(1)-C(51)	103.61(11)
C(61)-P(1)-C(51)	102.42(10)
N(1)#1-Si(1)-C(11)	113.77(13)
N(1)#1-Si(1)-C(13)	116.74(13)
C(11)-Si(1)-C(13)	104.1(2)
N(1)#1-Si(1)-C(12)	111.62(12)
C(11)-Si(1)-C(12)	105.3(2)
C(13)-Si(1)-C(12)	104.2(2)
N(1)#1-Si(1)-Na(1)#1	48.52(7)
C(11)-Si(1)-Na(1)#1	115.83(11)
C(13)-Si(1)-Na(1)#1	140.07(11)
C(12)-Si(1)-Na(1)#1	64.57(10)
N(1)#1-Si(1)-Na(1)	39.41(7)
C(11)-Si(1)-Na(1)	76.83(11)
C(13)-Si(1)-Na(1)	143.23(11)
C(12)-Si(1)-Na(1)	111.01(10)
Na(1)#1-Si(1)-Na(1)	54.94(3)
N(1)-Si(2)-C(21)	111.32(14)
N(1)-Si(2)-C(23)	113.98(13)
C(21)-Si(2)-C(23)	105.1(2)
N(1)-Si(2)-C(22)	115.73(13)
C(21)-Si(2)-C(22)	104.5(2)
C(23)-Si(2)-C(22)	105.26(14)
N(1)-Si(2)-Na(1)	44.75(7)
C(21)-Si(2)-Na(1)	84.66(12)
C(23)-Si(2)-Na(1)	88.69(10)
C(22)-Si(2)-Na(1)	160.30(11)
Si(2)-N(1)-Si(1)#1	123.52(12)
Si(2)-N(1)-Na(1)#1	118.78(10)
Si(1)#1-N(1)-Na(1)#1	113.99(10)
Si(2)-N(1)-Na(1)	107.20(10)
Si(1)#1-N(1)-Na(1)	101.34(9)
Na(1)#1-N(1)-Na(1)	78.58(7)
N(1)#1-Na(1)-N(1)	101.42(6)
N(1)#1-Na(1)-C(30)	132.05(8)
N(1)-Na(1)-C(30)	123.73(8)
N(1)#1-Na(1)-C(12)#1	123.57(9)
N(1)-Na(1)-C(12)#1	63.74(7)
C(30)-Na(1)-C(12)#1	93.00(8)
N(1)#1-Na(1)-Na(1)#1	52.50(5)
N(1)-Na(1)-Na(1)#1	48.93(5)
C(30)-Na(1)-Na(1)#1	164.70(8)
C(12)#1-Na(1)-Na(1)#1	93.85(7)
N(1)#1-Na(1)-Si(1)#1	111.08(6)
N(1)-Na(1)-Si(1)#1	30.14(5)
C(30)-Na(1)-Si(1)#1	116.09(6)
C(12)#1-Na(1)-Si(1)#1	34.23(5)
Na(1)#1-Na(1)-Si(1)#1	64.95(3)
N(1)#1-Na(1)-Si(2)	112.52(6)
N(1)-Na(1)-Si(2)	28.05(5)
C(30)-Na(1)-Si(2)	102.67(6)
C(12)#1-Na(1)-Si(2)	80.09(6)
Na(1)#1-Na(1)-Si(2)	65.18(3)
Si(1)#1-Na(1)-Si(2)	52.46(2)
N(1)#1-Na(1)-Si(1)	26.60(5)
N(1)-Na(1)-Si(1)	103.53(5)
C(30)-Na(1)-Si(1)	116.78(6)
C(12)#1-Na(1)-Si(1)	148.42(7)
Na(1)#1-Na(1)-Si(1)	60.11(3)
Si(1)#1-Na(1)-Si(1)	125.06(3)
Si(2)-Na(1)-Si(1)	101.76(3)
Si(1)-C(11)-H(111)	116(2)
Si(1)-C(11)-H(112)	113(2)
H(111)-C(11)-H(112)	108(3)
Si(1)-C(11)-H(113)	111(2)
H(111)-C(11)-H(113)	101(3)
H(112)-C(11)-H(113)	107(3)
Si(1)-C(12)-Na(1)#1	81.20(10)
Si(1)-C(12)-H(121)	111(2)
Na(1)#1-C(12)-H(121)	77(2)
Si(1)-C(12)-H(122)	112(2)
Na(1)#1-C(12)-H(122)	60(2)
H(121)-C(12)-H(122)	111(3)
Si(1)-C(12)-H(123)	110(2)
Na(1)#1-C(12)-H(123)	166(2)
H(121)-C(12)-H(123)	106(3)
H(122)-C(12)-H(123)	107(3)
Si(1)-C(13)-H(131)	115(2)
Si(1)-C(13)-H(132)	111(2)
H(131)-C(13)-H(132)	107(3)
Si(1)-C(13)-H(133)	115(2)
H(131)-C(13)-H(133)	103(3)
H(132)-C(13)-H(133)	105(3)
Si(2)-C(21)-H(211)	111(2)
Si(2)-C(21)-H(212)	116(3)
H(211)-C(21)-H(212)	104(3)
Si(2)-C(21)-H(213)	110(3)
H(211)-C(21)-H(213)	113(4)
H(212)-C(21)-H(213)	103(4)
Si(2)-C(22)-H(221)	109(2)
Si(2)-C(22)-H(222)	114(2)
H(221)-C(22)-H(222)	104(3)
Si(2)-C(22)-H(223)	107(2)
H(221)-C(22)-H(223)	113(3)
H(222)-C(22)-H(223)	110(3)
Si(2)-C(23)-H(231)	116(2)
Si(2)-C(23)-H(232)	114(2)
H(231)-C(23)-H(232)	105(3)
Si(2)-C(23)-H(233)	114(2)
H(231)-C(23)-H(233)	106(3)
H(232)-C(23)-H(233)	100(3)
C(31)-C(30)-P(1)	119.3(2)
C(31)-C(30)-Na(1)	106.1(2)
P(1)-C(30)-Na(1)	117.71(11)
C(31)-C(30)-H(30)	113(2)
P(1)-C(30)-H(30)	110(2)
Na(1)-C(30)-H(30)	86(2)
C(30)-C(31)-H(311)	110(2)
C(30)-C(31)-H(312)	112(2)
H(311)-C(31)-H(312)	105(3)
C(30)-C(31)-H(313)	116(2)
H(311)-C(31)-H(313)	108(3)
H(312)-C(31)-H(313)	106(2)
C(46)-C(41)-C(42)	119.3(2)

C(46)-C(41)-P(1)	122.4(2)
C(42)-C(41)-P(1)	118.3(2)
C(43)-C(42)-C(41)	120.1(3)
C(43)-C(42)-H(42)	121(2)
C(41)-C(42)-H(42)	119(2)
C(44)-C(43)-C(42)	120.3(3)
C(44)-C(43)-H(43)	119(2)
C(42)-C(43)-H(43)	121(2)
C(45)-C(44)-C(43)	120.2(3)
C(45)-C(44)-H(44)	119(2)
C(43)-C(44)-H(44)	121(2)
C(44)-C(45)-C(46)	120.1(3)
C(44)-C(45)-H(45)	119(2)
C(46)-C(45)-H(45)	121(2)
C(41)-C(46)-C(45)	119.9(3)
C(41)-C(46)-H(46)	121(2)
C(45)-C(46)-H(46)	119(2)
C(52)-C(51)-C(56)	118.6(2)
C(52)-C(51)-P(1)	118.8(2)
C(56)-C(51)-P(1)	122.5(2)
C(53)-C(52)-C(51)	120.4(2)
C(53)-C(52)-H(52)	118(2)
C(51)-C(52)-H(52)	121(2)
C(52)-C(53)-C(54)	120.5(3)
C(52)-C(53)-H(53)	122(2)
C(54)-C(53)-H(53)	118(2)
C(55)-C(54)-C(53)	119.9(2)
C(55)-C(54)-H(54)	121(2)
C(53)-C(54)-H(54)	119(2)
C(54)-C(55)-C(56)	120.0(2)
C(54)-C(55)-H(55)	122(2)
C(56)-C(55)-H(55)	118(2)
C(55)-C(56)-C(51)	120.5(2)
C(55)-C(56)-H(56)	121(2)
C(51)-C(56)-H(56)	118(2)
C(63)-C(62)-C(61)	120.3(2)
C(63)-C(62)-H(62)	119(2)
C(61)-C(62)-H(62)	120(2)
C(64)-C(65)-C(66)	119.8(2)
C(64)-C(65)-H(65)	120(2)
C(66)-C(65)-H(65)	120(2)
C(64)-C(63)-C(62)	120.5(2)
C(64)-C(63)-H(63)	119(2)
C(62)-C(63)-H(63)	120(2)
C(65)-C(66)-C(61)	120.6(2)
C(65)-C(66)-H(66)	118(2)
C(61)-C(66)-H(66)	121(2)
C(63)-C(64)-C(65)	120.1(2)
C(63)-C(64)-H(64)	120(2)
C(65)-C(64)-H(64)	120(2)
C(62)-C(61)-C(66)	118.6(2)
C(62)-C(61)-P(1)	121.7(2)
C(66)-C(61)-P(1)	119.6(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y+2, -z+1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 15. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
P(1)	16(1)	20(1)	14(1)	3(1)	0(1)	4(1)
Si(1)	25(1)	23(1)	17(1)	4(1)	2(1)	3(1)
Si(2)	23(1)	25(1)	20(1)	6(1)	0(1)	5(1)
N(1)	25(1)	22(1)	19(1)	7(1)	1(1)	4(1)
Na(1)	31(1)	22(1)	18(1)	3(1)	0(1)	5(1)
C(11)	35(2)	41(2)	28(2)	2(1)	7(1)	13(1)
C(12)	30(2)	37(2)	25(1)	4(1)	3(1)	-7(1)
C(13)	46(2)	36(2)	33(2)	18(1)	12(1)	8(1)
C(21)	32(2)	58(2)	60(2)	33(2)	12(2)	9(2)
C(22)	36(2)	42(2)	27(1)	3(1)	-7(1)	15(1)
C(23)	38(2)	34(2)	28(1)	3(1)	5(1)	15(1)
C(30)	17(1)	25(1)	16(1)	6(1)	-1(1)	3(1)
C(31)	19(1)	33(1)	25(1)	5(1)	-1(1)	5(1)
C(41)	16(1)	25(1)	15(1)	0(1)	0(1)	2(1)
C(42)	23(1)	35(1)	22(1)	8(1)	2(1)	6(1)
C(43)	24(1)	53(2)	22(1)	9(1)	3(1)	-2(1)
C(44)	19(1)	58(2)	27(1)	-4(1)	8(1)	3(1)
C(45)	22(1)	39(2)	45(2)	-1(1)	9(1)	10(1)
C(46)	21(1)	31(1)	34(1)	6(1)	4(1)	7(1)
C(51)	21(1)	22(1)	15(1)	3(1)	0(1)	6(1)
C(52)	23(1)	27(1)	24(1)	1(1)	1(1)	8(1)
C(53)	31(1)	33(1)	30(1)	-1(1)	4(1)	16(1)
C(54)	39(2)	27(1)	26(1)	-1(1)	-4(1)	13(1)
C(55)	26(1)	26(1)	30(1)	0(1)	-5(1)	3(1)
C(56)	21(1)	26(1)	25(1)	2(1)	0(1)	6(1)
C(62)	23(1)	28(1)	17(1)	3(1)	0(1)	6(1)
C(65)	32(1)	25(1)	30(1)	10(1)	5(1)	9(1)
C(63)	28(1)	38(1)	18(1)	8(1)	3(1)	10(1)
C(66)	25(1)	26(1)	19(1)	5(1)	1(1)	6(1)
C(64)	29(1)	37(1)	23(1)	15(1)	5(1)	10(1)
C(61)	15(1)	25(1)	17(1)	5(1)	1(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 15.

	x	y	z	U(eq)
H(111)	7545(44)	10872(41)	2750(30)	71(12)
H(112)	6938(39)	9844(39)	3262(29)	61(11)
H(113)	6059(43)	9883(42)	2262(33)	75(12)
H(121)	6942(37)	13676(38)	5232(28)	57(10)
H(122)	7679(35)	12419(35)	5178(26)	48(10)
H(123)	8059(38)	13384(36)	4576(26)	54(10)
H(131)	5126(36)	13566(37)	3187(26)	50(10)
H(132)	6413(44)	13288(41)	2742(30)	72(12)
H(133)	4908(44)	12370(43)	2266(33)	74(13)
H(211)	8452(38)	10577(42)	5730(29)	60(11)
H(212)	7926(47)	9475(45)	4734(36)	84(14)
H(213)	9071(50)	9298(45)	5429(33)	85(14)
H(221)	8926(37)	8687(34)	7278(25)	46(9)
H(222)	7643(38)	8426(37)	7690(28)	57(11)
H(223)	8276(40)	9926(42)	7656(29)	66(12)
H(231)	6292(36)	6047(36)	5596(26)	51(10)
H(232)	7739(37)	6481(34)	5321(25)	50(10)
H(233)	6419(37)	6437(36)	4630(29)	55(10)
H(30)	4207(29)	5856(28)	3178(21)	27(7)
H(311)	6430(34)	7135(34)	2987(25)	43(9)
H(312)	5762(30)	6834(30)	1866(23)	31(8)
H(313)	6027(31)	5615(32)	2249(22)	35(8)
H(42)	1504(30)	4084(30)	2838(22)	32(8)
H(43)	-326(37)	4198(37)	3713(28)	58(10)
H(44)	-1614(31)	5823(29)	3588(22)	32(8)
H(45)	-1084(38)	7349(37)	2717(27)	56(10)
H(46)	736(32)	7237(32)	1875(24)	39(9)
H(52)	4426(33)	3920(31)	744(23)	37(8)
H(53)	3804(30)	1705(29)	-419(22)	30(8)
H(54)	1542(31)	272(31)	-1137(23)	38(8)
H(55)	-342(35)	1149(34)	-483(25)	48(9)
H(56)	292(33)	3381(32)	714(23)	39(9)
H(62)	2299(32)	5036(33)	-449(24)	41(8)
H(65)	3703(28)	9962(29)	945(21)	27(7)
H(63)	2555(31)	6479(32)	-1450(24)	38(8)
H(66)	3494(30)	8589(30)	1998(23)	33(8)
H(64)	3277(30)	8898(30)	-807(22)	34(8)

16) 5-ferrocenyl-4-nitrile-7-acetonitrile-2-(4'-bromophenyl)indole

Table 1. Crystal data and structure refinement for 16.

Identification code	16 (98ckb008)
Empirical formula	C26 H17 Br Fe N4
Formula weight	521.20
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 7.96080(10) A alpha = 93.5770(10) deg. b = 10.68440(10) A beta = 97.7370(10) deg. c = 12.8215(3) A gamma = 99.0460(10) deg.
Volume, Z	1063.28(3) A^3, 2
Density (calculated)	1.628 Mg/m^3
Absorption coefficient	2.610 mm^-1
F(000)	524
Crystal size	0.25 x 0.15 x 0.06 mm
Theta range for data collection	1.61 to 27.49 deg.
Limiting indices	-10<=h<=9, -13<=k<=10, -16<=l<=16
Reflections collected	7790
Independent reflections	4823 [R(int) = 0.0403]
Absorption correction	Psi-scan corrections
Max. and min. transmission	0.59017 and 0.41751
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4783 / 0 / 289
Goodness-of-fit on F^2	1.195
Final R indices [I>2sigma(I)]	R1 = 0.0517, wR2 = 0.1024
R indices (all data)	R1 = 0.0722, wR2 = 0.1275
Largest diff. peak and hole	0.870 and -0.580 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 16. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Br(1)	4487(1)	2420(1)	3562(1)	33(1)
Fe(1)	10095(1)	9333(1)	12721(1)	24(1)
C(1)	11057(5)	7849(4)	12055(3)	22(1)
C(2)	10093(6)	7436(4)	12891(3)	28(1)
C(3)	10892(6)	8171(4)	13849(3)	32(1)
C(4)	12313(6)	9042(4)	13624(3)	29(1)
C(5)	12430(5)	8848(4)	12530(3)	24(1)
C(1')	8703(7)	10067(5)	11520(4)	41(1)
C(2')	7624(6)	9611(5)	12252(5)	45(1)
C(3')	8282(7)	10282(5)	13256(5)	42(1)
C(4')	9767(6)	11152(4)	13129(4)	33(1)
C(5')	10037(6)	11008(5)	12061(4)	34(1)
N(1)	10037(4)	5849(3)	7806(3)	21(1)
N(2)	6341(5)	6199(5)	11208(3)	42(1)
N(3)	14876(6)	8071(5)	9020(3)	44(1)
N(4)	16120(5)	8759(5)	9307(3)	46(1)
C(10)	10747(5)	7338(4)	10945(3)	22(1)
C(11)	12137(5)	7518(4)	10330(3)	22(1)
C(12)	11965(5)	7039(4)	9301(3)	22(1)
C(13)	10406(4)	6352(3)	8837(3)	11(1)
C(14)	8353(5)	5328(4)	7704(3)	23(1)
C(15)	7655(5)	5494(4)	8640(3)	21(1)
C(16)	8986(5)	6159(4)	9365(3)	20(1)
C(17)	9185(5)	6667(4)	10441(3)	21(1)
C(18)	7637(6)	6434(4)	10913(3)	27(1)
C(19)	13299(5)	7185(4)	8571(3)	26(1)
C(21)	7431(5)	4683(4)	6690(3)	23(1)
C(22)	5900(5)	3836(4)	6690(3)	26(1)
C(23)	5006(5)	3177(4)	5759(3)	27(1)
C(24)	5688(5)	3372(4)	4824(3)	25(1)
C(25)	7187(5)	4208(4)	4792(3)	27(1)
C(26)	8062(5)	4868(4)	5733(3)	26(1)

Table 3. Bond lengths [A] and angles [deg] for 16.

Br(1)-C(24)	1.908(4)
Fe(1)-C(5')	2.031(5)
Fe(1)-C(1')	2.044(5)
Fe(1)-C(4')	2.046(5)
Fe(1)-C(5)	2.048(4)
Fe(1)-C(2')	2.051(5)
Fe(1)-C(2)	2.051(4)
Fe(1)-C(1)	2.054(4)
Fe(1)-C(3')	2.054(5)
Fe(1)-C(4)	2.055(4)
Fe(1)-C(3)	2.064(5)
C(1)-C(5)	1.441(6)
C(1)-C(2)	1.451(6)
C(1)-C(10)	1.466(5)
C(2)-C(3)	1.429(6)
C(2)-H(2)	1.00
C(3)-C(4)	1.421(7)
C(3)-H(3)	1.00
C(4)-C(5)	1.423(6)
C(4)-H(4)	1.00
C(5)-H(5)	1.00
C(1')-C(2')	1.413(8)
C(1')-C(5')	1.415(7)
C(1')-H(1')	1.00
C(2')-C(3')	1.425(7)
C(2')-H(2')	1.00
C(3')-C(4')	1.418(7)
C(3')-H(3')	1.00
C(4')-C(5')	1.417(7)
C(4')-H(4')	1.00
C(5')-H(5')	1.00
N(1)-C(14)	1.354(5)
N(1)-C(13)	1.371(4)
N(2)-C(18)	1.143(6)
N(3)-N(4)	1.139(6)
N(3)-C(19)	1.472(6)
C(10)-C(17)	1.387(6)
C(10)-C(11)	1.439(6)
C(11)-C(12)	1.366(6)
C(11)-H(11)	0.95
C(12)-C(13)	1.376(5)
C(12)-C(19)	1.504(5)
C(13)-C(16)	1.389(5)
C(14)-C(15)	1.401(6)
C(14)-C(21)	1.476(5)
C(15)-C(16)	1.381(5)
C(15)-H(15)	0.95
C(16)-C(17)	1.430(5)
C(17)-C(18)	1.439(6)
C(19)-H(19B)	0.99
C(19)-H(19A)	0.99
C(21)-C(22)	1.399(6)
C(21)-C(26)	1.401(6)

C(22)-C(23)	1.391(6)
C(22)-H(22)	0.95
C(23)-C(24)	1.396(6)
C(23)-H(23)	0.95
C(24)-C(25)	1.381(6)
C(25)-C(26)	1.397(6)
C(25)-H(25)	0.95
C(26)-H(26)	0.95
C(5')-Fe(1)-C(1')	40.6(2)
C(5')-Fe(1)-C(4')	40.7(2)
C(1')-Fe(1)-C(4')	68.1(2)
C(5')-Fe(1)-C(5)	105.7(2)
C(1')-Fe(1)-C(5)	120.1(2)
C(4')-Fe(1)-C(5)	123.4(2)
C(5')-Fe(1)-C(2')	68.3(2)
C(1')-Fe(1)-C(2')	40.4(2)
C(4')-Fe(1)-C(2')	68.0(2)
C(5)-Fe(1)-C(2')	156.1(2)
C(5')-Fe(1)-C(2)	161.7(2)
C(1')-Fe(1)-C(2)	125.7(2)
C(4')-Fe(1)-C(2)	156.8(2)
C(5)-Fe(1)-C(2)	69.0(2)
C(2')-Fe(1)-C(2)	109.1(2)
C(5')-Fe(1)-C(1)	123.2(2)
C(1')-Fe(1)-C(1)	107.0(2)
C(4')-Fe(1)-C(1)	160.2(2)
C(5)-Fe(1)-C(1)	41.1(2)
C(2')-Fe(1)-C(1)	121.4(2)
C(2)-Fe(1)-C(1)	41.4(2)
C(5')-Fe(1)-C(3')	68.5(2)
C(1')-Fe(1)-C(3')	68.2(2)
C(4')-Fe(1)-C(3')	40.5(2)
C(5)-Fe(1)-C(3')	160.7(2)
C(2')-Fe(1)-C(3')	40.6(2)
C(2)-Fe(1)-C(3')	122.2(2)
C(1)-Fe(1)-C(3')	157.4(2)
C(5')-Fe(1)-C(4)	119.5(2)
C(1')-Fe(1)-C(4)	154.9(2)
C(4')-Fe(1)-C(4)	107.0(2)
C(5)-Fe(1)-C(4)	40.6(2)
C(2')-Fe(1)-C(4)	162.7(2)
C(2)-Fe(1)-C(4)	68.6(2)
C(1)-Fe(1)-C(4)	69.0(2)
C(3')-Fe(1)-C(4)	125.0(2)
C(5')-Fe(1)-C(3)	155.3(2)
C(1')-Fe(1)-C(3)	163.2(2)
C(4')-Fe(1)-C(3)	121.3(2)
C(5)-Fe(1)-C(3)	68.2(2)
C(2')-Fe(1)-C(3)	126.9(2)
C(2)-Fe(1)-C(3)	40.6(2)
C(1)-Fe(1)-C(3)	68.9(2)
C(3')-Fe(1)-C(3)	109.0(2)
C(4)-Fe(1)-C(3)	40.4(2)
C(5)-C(1)-C(2)	106.7(4)
C(5)-C(1)-C(10)	125.9(4)
C(2)-C(1)-C(10)	127.4(4)
C(5)-C(1)-Fe(1)	69.2(2)
C(2)-C(1)-Fe(1)	69.2(2)
C(10)-C(1)-Fe(1)	127.8(3)
C(3)-C(2)-C(1)	107.9(4)
C(3)-C(2)-Fe(1)	70.1(3)
C(1)-C(2)-Fe(1)	69.4(2)
C(3)-C(2)-H(2)	126.0(3)
C(1)-C(2)-H(2)	126.0(2)
Fe(1)-C(2)-H(2)	126.05(13)
C(4)-C(3)-C(2)	108.5(4)
C(4)-C(3)-Fe(1)	69.5(3)
C(2)-C(3)-Fe(1)	69.2(3)
C(4)-C(3)-H(3)	125.7(2)
C(2)-C(3)-H(3)	125.7(3)
Fe(1)-C(3)-H(3)	125.75(13)
C(3)-C(4)-C(5)	108.3(4)
C(3)-C(4)-Fe(1)	70.2(3)
C(5)-C(4)-Fe(1)	69.5(2)
C(3)-C(4)-H(4)	125.9(2)
C(5)-C(4)-H(4)	125.9(2)
Fe(1)-C(4)-H(4)	125.85(13)
C(4)-C(5)-C(1)	108.6(4)
C(4)-C(5)-Fe(1)	70.0(2)
C(1)-C(5)-Fe(1)	69.7(2)
C(4)-C(5)-H(5)	125.7(2)
C(1)-C(5)-H(5)	125.7(2)
Fe(1)-C(5)-H(5)	125.70(12)
C(2')-C(1')-C(5')	108.2(4)
C(2')-C(1')-Fe(1)	70.1(3)
C(5')-C(1')-Fe(1)	69.2(3)
C(2')-C(1')-H(1')	125.9(3)
C(5')-C(1')-H(1')	125.9(3)
Fe(1)-C(1')-H(1')	125.87(14)
C(1')-C(2')-C(3')	108.1(5)
C(1')-C(2')-Fe(1)	69.6(3)
C(3')-C(2')-Fe(1)	69.8(3)
C(1')-C(2')-H(2')	126.0(3)
C(3')-C(2')-H(2')	126.0(3)
Fe(1)-C(2')-H(2')	126.0(2)
C(4')-C(3')-C(2')	107.4(5)
C(4')-C(3')-Fe(1)	69.5(3)
C(2')-C(3')-Fe(1)	69.5(3)
C(4')-C(3')-H(3')	126.3(3)
C(2')-C(3')-H(3')	126.3(3)
Fe(1)-C(3')-H(3')	126.3(2)
C(5')-C(4')-C(3')	108.3(4)
C(5')-C(4')-Fe(1)	69.1(3)
C(3')-C(4')-Fe(1)	70.1(3)
C(5')-C(4')-H(4')	125.8(3)
C(3')-C(4')-H(4')	125.8(3)
Fe(1)-C(4')-H(4')	125.82(13)
C(1')-C(5')-C(4')	107.9(5)
C(1')-C(5')-Fe(1)	70.2(3)
C(4')-C(5')-Fe(1)	70.2(3)
C(1')-C(5')-H(5')	126.1(3)
C(4')-C(5')-H(5')	126.1(3)
Fe(1)-C(5')-H(5')	126.06(14)
C(14)-N(1)-C(13)	103.5(3)
N(4)-N(3)-C(19)	175.8(5)
C(17)-C(10)-C(11)	116.9(4)
C(17)-C(10)-C(1)	124.0(4)
C(11)-C(10)-C(1)	119.0(4)
C(12)-C(11)-C(10)	122.5(4)
C(12)-C(11)-H(11)	118.7(2)
C(10)-C(11)-H(11)	118.7(2)
C(11)-C(12)-C(13)	118.8(4)
C(11)-C(12)-C(19)	127.6(4)
C(13)-C(12)-C(19)	113.7(3)
N(1)-C(13)-C(12)	125.0(3)
N(1)-C(13)-C(16)	112.4(3)
C(12)-C(13)-C(16)	122.5(3)
N(1)-C(14)-C(15)	112.6(3)
N(1)-C(14)-C(21)	121.0(4)
C(15)-C(14)-C(21)	126.4(4)
C(16)-C(15)-C(14)	105.6(3)
C(16)-C(15)-H(15)	127.2(2)
C(14)-C(15)-H(15)	127.2(2)
C(15)-C(16)-C(13)	106.0(3)
C(15)-C(16)-C(17)	135.9(4)
C(13)-C(16)-C(17)	118.1(3)
C(10)-C(17)-C(16)	121.2(4)
C(10)-C(17)-C(18)	124.9(4)
C(16)-C(17)-C(18)	113.9(3)
N(2)-C(18)-C(17)	174.2(5)
N(3)-C(19)-C(12)	113.4(3)
N(3)-C(19)-H(19B)	108.9(3)
C(12)-C(19)-H(19B)	108.9(2)
N(3)-C(19)-H(19A)	108.9(3)
C(12)-C(19)-H(19A)	108.9(2)
H(19B)-C(19)-H(19A)	107.7
C(22)-C(21)-C(26)	119.1(4)

C(22)-C(21)-C(14)	118.8(4)	C(24)-C(25)-C(26)	118.7(4)
C(26)-C(21)-C(14)	122.0(4)	C(24)-C(25)-H(25)	120.6(2)
C(23)-C(22)-C(21)	121.1(4)	C(26)-C(25)-H(25)	120.6(2)
C(23)-C(22)-H(22)	119.4(3)	C(25)-C(26)-C(21)	120.6(4)
C(21)-C(22)-H(22)	119.4(2)	C(25)-C(26)-H(26)	119.7(2)
C(22)-C(23)-C(24)	118.2(4)	C(21)-C(26)-H(26)	119.7(2)
C(22)-C(23)-H(23)	120.9(3)		
C(24)-C(23)-H(23)	120.9(2)		
C(25)-C(24)-C(23)	122.3(4)		
C(25)-C(24)-Br(1)	120.0(3)		
C(23)-C(24)-Br(1)	117.7(3)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 16. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Br(1)	30(1)	42(1)	23(1)	-8(1)	2(1)	-3(1)
Fe(1)	25(1)	24(1)	20(1)	-3(1)	2(1)	3(1)
C(1)	25(2)	25(2)	17(2)	2(2)	2(2)	5(2)
C(2)	37(2)	25(2)	20(2)	1(2)	5(2)	2(2)
C(3)	44(3)	32(2)	19(2)	1(2)	3(2)	7(2)
C(4)	31(2)	32(2)	22(2)	-1(2)	-4(2)	6(2)
C(5)	24(2)	25(2)	23(2)	0(2)	0(2)	4(2)
C(1')	48(3)	40(3)	33(3)	-7(2)	-11(2)	22(2)
C(2')	24(2)	44(3)	64(4)	-17(3)	-5(2)	8(2)
C(3')	37(3)	39(3)	50(3)	-10(2)	14(2)	8(2)
C(4')	35(2)	26(2)	36(3)	-10(2)	3(2)	5(2)
C(5')	39(3)	32(3)	36(3)	7(2)	6(2)	17(2)
N(1)	24(2)	21(2)	16(2)	-3(1)	2(1)	-1(1)
N(2)	32(2)	60(3)	31(2)	-8(2)	12(2)	-9(2)
N(3)	46(3)	55(3)	33(2)	5(2)	11(2)	6(2)
N(4)	28(2)	65(3)	34(2)	-8(2)	5(2)	-20(2)
C(10)	26(2)	20(2)	20(2)	4(2)	2(2)	5(2)
C(11)	18(2)	25(2)	20(2)	1(2)	1(2)	-1(2)
C(12)	21(2)	22(2)	23(2)	-1(2)	3(2)	2(2)
C(13)	10(2)	13(2)	8(2)	0(1)	2(1)	1(1)
C(14)	21(2)	24(2)	21(2)	-1(2)	0(2)	1(2)
C(15)	19(2)	25(2)	18(2)	-1(2)	3(2)	2(2)
C(16)	20(2)	23(2)	18(2)	4(2)	4(2)	3(2)
C(17)	22(2)	21(2)	20(2)	2(2)	7(2)	3(2)
C(18)	28(2)	30(2)	20(2)	-4(2)	4(2)	-2(2)
C(19)	22(2)	32(2)	22(2)	-1(2)	7(2)	0(2)
C(21)	20(2)	25(2)	22(2)	0(2)	1(2)	7(2)
C(22)	25(2)	33(2)	20(2)	-1(2)	4(2)	1(2)
C(23)	21(2)	34(2)	25(2)	-2(2)	4(2)	0(2)
C(24)	28(2)	26(2)	19(2)	-4(2)	-1(2)	3(2)
C(25)	26(2)	35(2)	19(2)	0(2)	6(2)	1(2)
C(26)	26(2)	29(2)	23(2)	2(2)	4(2)	0(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 16.

	x	y	z	U(eq)
H(2)	9054(6)	6757(4)	12811(3)	33
H(3)	10496(6)	8101(4)	14554(3)	38
H(4)	13093(6)	9685(4)	14145(3)	34
H(5)	13309(5)	9331(4)	12152(3)	29
H(1')	8556(7)	9767(5)	10754(4)	49
H(2')	6585(6)	8931(5)	12094(5)	55
H(3')	7788(7)	10162(5)	13927(5)	50
H(4')	10507(6)	11754(4)	13699(4)	40
H(5')	10989(6)	11499(5)	11743(4)	41
H(11)	13215(5)	7988(4)	10651(3)	26
H(15)	6507(5)	5209(4)	8752(3)	25
H(19B)	13603(5)	6341(4)	8396(3)	31
H(19A)	12799(5)	7493(4)	7905(3)	31
H(22)	5463(5)	3709(4)	7336(3)	32
H(23)	3960(5)	2610(4)	5760(3)	33
H(25)	7614(5)	4333(4)	4143(3)	32
H(26)	9095(5)	5448(4)	5724(3)	31

17) Carbazol triphenylethylphosphonium

Table 1. Crystal data and structure refinement for 17.

Identification code	17 (97srv186)
Empirical formula	C32 H28 N P
Formula weight	457.52
Temperature	150(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 18.9883(2) A alpha = 90 deg. b = 13.422 A beta = 95.3080(10) deg. c = 19.4539(2) A gamma = 90 deg.
Volume, Z	4936.89(7) A^3, 8
Density (calculated)	1.231 Mg/m^3
Absorption coefficient	0.132 mm^-1
F(000)	1936
Crystal size	0.50 x 0.50 x 0.45 mm
Theta range for data collection	1.43 to 27.49 deg.
Limiting indices	-24<=h<=24, -17<=k<=17, -24<=l<=24
Reflections collected	53667
Independent reflections	11282 [R(int) = 0.0337]
Absorption correction	None
Max. and min. transmission	0.812 and 0.846
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11230 / 0 / 653
Goodness-of-fit on F^2	1.100
Final R indices [I>2sigma(I)]	R1 = 0.0386, wR2 = 0.0873
R indices (all data)	R1 = 0.0520, wR2 = 0.1026
Extinction coefficient	not refined
Largest diff. peak and hole	0.336 and -0.343 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 17. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(100)	1732(1)	749(1)	104(1)	30(1)
C(100)	1319(1)	1585(1)	127(1)	26(1)
C(101)	578(1)	1648(1)	-7(1)	31(1)
C(102)	253(1)	2566(1)	33(1)	34(1)
C(103)	644(1)	3434(1)	206(1)	36(1)
C(104)	1373(1)	3391(1)	347(1)	31(1)
C(105)	1718(1)	2473(1)	310(1)	25(1)
C(106)	2439(1)	2139(1)	425(1)	25(1)
C(107)	3089(1)	2593(1)	635(1)	30(1)
C(108)	3695(1)	2013(1)	721(1)	33(1)
C(109)	3662(1)	979(1)	590(1)	34(1)
C(110)	3031(1)	510(1)	378(1)	32(1)
C(111)	2406(1)	1085(1)	293(1)	26(1)
N(200)	7765(1)	-2862(1)	4847(1)	32(1)
C(200)	7382(1)	-2021(1)	4645(1)	31(1)
C(201)	6644(1)	-1924(1)	4524(1)	39(1)
C(202)	6358(1)	-1013(2)	4317(1)	47(1)
C(203)	6789(1)	-179(2)	4229(1)	49(1)
C(204)	7517(1)	-251(1)	4347(1)	41(1)
C(205)	7821(1)	-1169(1)	4548(1)	30(1)
C(206)	8532(1)	-1522(1)	4690(1)	28(1)
C(207)	9207(1)	-1099(1)	4673(1)	34(1)
C(208)	9799(1)	-1681(1)	4831(1)	36(1)
C(209)	9729(1)	-2691(1)	5010(1)	34(1)
C(210)	9072(1)	-3127(1)	5031(1)	31(1)
C(211)	8457(1)	-2553(1)	4866(1)	27(1)
P(300)	8062(1)	78(1)	2035(1)	24(1)
C(301)	8341(1)	625(1)	1260(1)	29(1)
C(302)	9131(1)	881(2)	1313(1)	39(1)
C(311)	7127(1)	-174(1)	1936(1)	27(1)
C(312)	6824(1)	-699(1)	2457(1)	33(1)
C(313)	6103(1)	-889(1)	2393(1)	38(1)
C(314)	5685(1)	-566(1)	1814(1)	39(1)
C(315)	5982(1)	-48(1)	1300(1)	39(1)
C(316)	6705(1)	158(1)	1355(1)	33(1)
C(321)	8242(1)	908(1)	2757(1)	26(1)
C(322)	7707(1)	1495(1)	2997(1)	29(1)
C(323)	7863(1)	2139(1)	3550(1)	34(1)
C(324)	8548(1)	2211(1)	3859(1)	34(1)
C(325)	9084(1)	1640(1)	3617(1)	34(1)
C(326)	8936(1)	987(1)	3070(1)	31(1)
C(331)	8547(1)	-1064(1)	2219(1)	26(1)
C(332)	8643(1)	-1444(1)	2892(1)	30(1)
C(333)	9003(1)	-2338(1)	3019(1)	34(1)
C(334)	9267(1)	-2854(1)	2479(1)	37(1)
C(335)	9171(1)	-2482(1)	1809(1)	37(1)
C(336)	8812(1)	-1591(1)	1677(1)	32(1)
P(400)	1997(1)	8193(1)	2028(1)	23(1)
C(401)	1829(1)	8894(1)	1238(1)	30(1)
C(402)	1045(1)	9172(2)	1094(1)	38(1)
C(411)	1537(1)	7018(1)	1955(1)	27(1)
C(412)	1422(1)	6474(1)	2547(1)	32(1)
C(413)	1085(1)	5554(1)	2481(1)	40(1)
C(414)	865(1)	5180(1)	1831(1)	48(1)
C(415)	993(1)	5713(1)	1246(1)	49(1)
C(416)	1329(1)	6629(1)	1304(1)	37(1)
C(421)	2925(1)	7943(1)	2210(1)	25(1)
C(422)	3184(1)	7619(1)	2870(1)	31(1)
C(423)	3891(1)	7348(1)	2998(1)	35(1)
C(424)	4336(1)	7396(1)	2470(1)	34(1)
C(425)	4079(1)	7713(1)	1817(1)	33(1)
C(426)	3374(1)	7987(1)	1680(1)	28(1)
C(431)	1675(1)	8909(1)	2715(1)	25(1)
C(432)	971(1)	8807(1)	2865(1)	28(1)
C(433)	709(1)	9427(1)	3356(1)	33(1)

C(434)	1140(1)	10144(1)	3688(1)	37(1)
C(435)	1837(1)	10249(1)	3542(1)	38(1)
C(436)	2108(1)	9635(1)	3052(1)	31(1)

Table 3. Bond lengths [A] and angles [deg] for 17.

N(100)-C(100)	1.372(2)
N(100)-C(111)	1.375(2)
C(100)-C(101)	1.410(2)
C(100)-C(105)	1.439(2)
C(101)-C(102)	1.384(2)
C(101)-H(101)	0.95
C(102)-C(103)	1.406(2)
C(102)-H(102)	0.95
C(103)-C(104)	1.388(2)
C(103)-H(103)	0.95
C(104)-C(105)	1.400(2)
C(104)-H(104)	0.95
C(105)-C(106)	1.439(2)
C(106)-C(107)	1.402(2)
C(106)-C(111)	1.437(2)
C(107)-C(108)	1.387(2)
C(107)-H(107)	0.95
C(108)-C(109)	1.410(2)
C(108)-H(108)	0.95
C(109)-C(110)	1.383(2)
C(109)-H(109)	0.95
C(110)-C(111)	1.413(2)
C(110)-H(110)	0.95
N(200)-C(211)	1.375(2)
N(200)-C(200)	1.380(2)
C(200)-C(201)	1.405(2)
C(200)-C(205)	1.438(2)
C(201)-C(202)	1.383(3)
C(201)-H(201)	0.95
C(202)-C(203)	1.406(3)
C(202)-H(202)	0.95
C(203)-C(204)	1.384(3)
C(203)-H(203)	0.95
C(204)-C(205)	1.400(2)
C(204)-H(204)	0.95
C(205)-C(206)	1.434(2)
C(206)-C(207)	1.404(2)
C(206)-C(211)	1.436(2)
C(207)-C(208)	1.380(2)
C(207)-H(207)	0.95
C(208)-C(209)	1.409(2)
C(208)-H(208)	0.95
C(209)-C(210)	1.383(2)
C(209)-H(209)	0.95
C(210)-C(211)	1.411(2)
C(210)-H(210)	0.95
P(300)-C(301)	1.800(2)
P(300)-C(321)	1.8001(14)
P(300)-C(311)	1.8005(14)
P(300)-C(331)	1.806(2)
C(301)-C(302)	1.533(2)
C(301)-H(31A)	0.96(2)
C(301)-H(31B)	0.98(2)
C(302)-H(32A)	0.97(2)
C(302)-H(32B)	1.00(2)
C(302)-H(32C)	0.96(2)
C(311)-C(316)	1.396(2)
C(311)-C(312)	1.402(2)
C(312)-C(313)	1.386(2)
C(312)-H(312)	0.95
C(313)-C(314)	1.387(2)
C(313)-H(313)	0.95
C(314)-C(315)	1.380(2)
C(314)-H(314)	0.95
C(315)-C(316)	1.394(2)
C(315)-H(315)	0.95
C(316)-H(316)	0.95
C(321)-C(322)	1.399(2)
C(321)-C(326)	1.404(2)
C(322)-C(323)	1.391(2)
C(322)-H(322)	0.95
C(323)-C(324)	1.383(2)
C(323)-H(323)	0.95
C(324)-C(325)	1.391(2)
C(324)-H(324)	0.95
C(325)-C(326)	1.388(2)
C(325)-H(325)	0.95
C(326)-H(326)	0.95
C(331)-C(332)	1.400(2)
C(331)-C(336)	1.402(2)
C(332)-C(333)	1.392(2)
C(332)-H(332)	0.95
C(333)-C(334)	1.391(2)
C(333)-H(333)	0.95
C(334)-C(335)	1.392(2)
C(334)-H(334)	0.95
C(335)-C(336)	1.389(2)
C(335)-H(335)	0.95
C(336)-H(336)	0.95
P(400)-C(421)	1.7970(14)
P(400)-C(431)	1.7982(14)
P(400)-C(411)	1.802(2)
P(400)-C(401)	1.804(2)
C(401)-C(402)	1.535(2)
C(401)-H(41A)	0.99(2)
C(401)-H(41B)	0.99(2)
C(402)-H(42A)	0.95(2)
C(402)-H(42B)	1.00(2)
C(402)-H(42C)	0.99(2)
C(411)-C(416)	1.393(2)
C(411)-C(412)	1.398(2)
C(412)-C(413)	1.391(2)
C(412)-H(412)	0.95
C(413)-C(414)	1.388(3)
C(413)-H(413)	0.95
C(414)-C(415)	1.384(3)
C(414)-H(414)	0.95
C(415)-C(416)	1.385(2)
C(415)-H(415)	0.95
C(416)-H(416)	0.95
C(421)-C(426)	1.399(2)
C(421)-C(422)	1.401(2)
C(422)-C(423)	1.390(2)
C(422)-H(422)	0.95
C(423)-C(424)	1.391(2)
C(423)-H(423)	0.95
C(424)-C(425)	1.386(2)
C(424)-H(424)	0.95
C(425)-C(426)	1.391(2)
C(425)-H(425)	0.95
C(426)-H(426)	0.95
C(431)-C(436)	1.399(2)
C(431)-C(432)	1.402(2)
C(432)-C(433)	1.393(2)
C(432)-H(432)	0.95
C(433)-C(434)	1.384(2)
C(433)-H(433)	0.95
C(434)-C(435)	1.387(2)
C(434)-H(434)	0.95

C(435)-C(436)	1.393(2)
C(435)-H(435)	0.95
C(436)-H(436)	0.95
C(100)-N(100)-C(111)	104.03(12)
N(100)-C(100)-C(101)	127.24(13)
N(100)-C(100)-C(105)	113.39(12)
C(101)-C(100)-C(105)	119.37(13)
C(102)-C(101)-C(100)	119.03(14)
C(102)-C(101)-H(101)	120.49(9)
C(100)-C(101)-H(101)	120.49(9)
C(101)-C(102)-C(103)	121.55(14)
C(101)-C(102)-H(102)	119.22(9)
C(103)-C(102)-H(102)	119.22(9)
C(104)-C(103)-C(102)	120.5(2)
C(104)-C(103)-H(103)	119.73(9)
C(102)-C(103)-H(103)	119.73(9)
C(103)-C(104)-C(105)	119.28(14)
C(103)-C(104)-H(104)	120.36(9)
C(105)-C(104)-H(104)	120.36(9)
C(104)-C(105)-C(100)	120.24(13)
C(104)-C(105)-C(106)	135.19(14)
C(100)-C(105)-C(106)	104.56(12)
C(107)-C(106)-C(111)	120.23(14)
C(107)-C(106)-C(105)	135.05(14)
C(111)-C(106)-C(105)	104.70(12)
C(108)-C(107)-C(106)	119.19(14)
C(108)-C(107)-H(107)	120.41(9)
C(106)-C(107)-H(107)	120.41(9)
C(107)-C(108)-C(109)	120.61(14)
C(107)-C(108)-H(108)	119.69(9)
C(109)-C(108)-H(108)	119.69(9)
C(110)-C(109)-C(108)	121.6(2)
C(110)-C(109)-H(109)	119.20(9)
C(108)-C(109)-H(109)	119.20(9)
C(109)-C(110)-C(111)	118.8(2)
C(109)-C(110)-H(110)	120.62(9)
C(111)-C(110)-H(110)	120.62(9)
N(100)-C(111)-C(110)	127.09(14)
N(100)-C(111)-C(106)	113.29(13)
C(110)-C(111)-C(106)	119.61(13)
C(211)-N(200)-C(200)	103.76(12)
N(200)-C(200)-C(201)	127.9(2)
N(200)-C(200)-C(205)	113.07(13)
C(201)-C(200)-C(205)	119.0(2)
C(202)-C(201)-C(200)	119.3(2)
C(202)-C(201)-H(201)	120.37(11)
C(200)-C(201)-H(201)	120.37(10)
C(201)-C(202)-C(203)	121.5(2)
C(201)-C(202)-H(202)	119.23(11)
C(203)-C(202)-H(202)	119.23(11)
C(204)-C(203)-C(202)	120.5(2)
C(204)-C(203)-H(203)	119.76(11)
C(202)-C(203)-H(203)	119.76(11)
C(203)-C(204)-C(205)	119.1(2)
C(203)-C(204)-H(204)	120.43(11)
C(205)-C(204)-H(204)	120.43(10)
C(204)-C(205)-C(206)	134.5(2)
C(204)-C(205)-C(200)	120.5(2)
C(206)-C(205)-C(200)	104.98(13)
C(207)-C(206)-C(205)	134.9(2)
C(207)-C(206)-C(211)	120.4(2)
C(205)-C(206)-C(211)	104.60(13)
C(208)-C(207)-C(206)	119.5(2)
C(208)-C(207)-H(207)	120.27(9)
C(206)-C(207)-H(207)	120.27(10)
C(207)-C(208)-C(209)	120.42(14)
C(207)-C(208)-H(208)	119.79(9)
C(209)-C(208)-H(208)	119.79(10)
C(210)-C(209)-C(208)	121.4(2)
C(210)-C(209)-H(209)	119.31(10)
C(208)-C(209)-H(209)	119.31(9)
C(209)-C(210)-C(211)	119.5(2)
C(209)-C(210)-H(210)	120.26(10)
C(211)-C(210)-H(210)	120.26(9)
N(200)-C(211)-C(210)	127.59(14)
N(200)-C(211)-C(206)	113.58(14)
C(210)-C(211)-C(206)	118.83(13)
C(301)-P(300)-C(321)	110.61(7)
C(301)-P(300)-C(311)	110.45(7)
C(321)-P(300)-C(311)	108.25(7)
C(301)-P(300)-C(331)	109.20(7)
C(321)-P(300)-C(331)	108.31(7)
C(311)-P(300)-C(331)	109.99(7)
C(302)-C(301)-P(300)	113.34(11)
C(302)-C(301)-H(31A)	111.5(10)
P(300)-C(301)-H(31A)	106.6(10)
C(302)-C(301)-H(31B)	112.5(11)
P(300)-C(301)-H(31B)	106.2(11)
H(31A)-C(301)-H(31B)	106.2(14)
C(301)-C(302)-H(32A)	108.8(11)
C(301)-C(302)-H(32B)	112.7(12)
H(32A)-C(302)-H(32B)	107(2)
C(301)-C(302)-H(32C)	111.9(12)
H(32A)-C(302)-H(32C)	108(2)
H(32B)-C(302)-H(32C)	108(2)
C(316)-C(311)-C(312)	120.12(14)
C(316)-C(311)-P(300)	120.95(11)
C(312)-C(311)-P(300)	118.93(11)
C(313)-C(312)-C(311)	119.8(2)
C(313)-C(312)-H(312)	120.11(10)
C(311)-C(312)-H(312)	120.11(9)
C(312)-C(313)-C(314)	120.0(2)
C(312)-C(313)-H(313)	119.99(10)
C(314)-C(313)-H(313)	119.99(10)
C(315)-C(314)-C(313)	120.3(2)
C(315)-C(314)-H(314)	119.84(10)
C(313)-C(314)-H(314)	119.84(10)
C(314)-C(315)-C(316)	120.7(2)
C(314)-C(315)-H(315)	119.66(10)
C(316)-C(315)-H(315)	119.66(10)
C(315)-C(316)-C(311)	119.1(2)
C(315)-C(316)-H(316)	120.47(10)
C(311)-C(316)-H(316)	120.47(9)
C(322)-C(321)-C(326)	119.59(13)
C(322)-C(321)-P(300)	121.21(11)
C(326)-C(321)-P(300)	119.17(11)
C(323)-C(322)-C(321)	119.89(14)
C(323)-C(322)-H(322)	120.06(9)
C(321)-C(322)-H(322)	120.06(8)
C(324)-C(323)-C(322)	120.3(2)
C(324)-C(323)-H(323)	119.87(9)
C(322)-C(323)-H(323)	119.87(9)
C(323)-C(324)-C(325)	120.24(14)
C(323)-C(324)-H(324)	119.88(9)
C(325)-C(324)-H(324)	119.88(9)
C(326)-C(325)-C(324)	120.21(14)
C(326)-C(325)-H(325)	119.90(9)
C(324)-C(325)-H(325)	119.90(9)
C(325)-C(326)-C(321)	119.8(2)
C(325)-C(326)-H(326)	120.10(9)
C(321)-C(326)-H(326)	120.10(9)
C(332)-C(331)-C(336)	119.66(14)
C(332)-C(331)-P(300)	120.96(11)
C(336)-C(331)-P(300)	119.36(11)
C(333)-C(332)-C(331)	119.98(14)
C(333)-C(332)-H(332)	120.01(9)
C(331)-C(332)-H(332)	120.01(9)
C(334)-C(333)-C(332)	120.0(2)
C(334)-C(333)-H(333)	119.98(10)
C(332)-C(333)-H(333)	119.98(9)
C(333)-C(334)-C(335)	120.2(2)
C(333)-C(334)-H(334)	119.88(10)
C(335)-C(334)-H(334)	119.88(10)
C(336)-C(335)-C(334)	120.1(2)
C(336)-C(335)-H(335)	119.95(9)
C(334)-C(335)-H(335)	119.95(10)
C(335)-C(336)-C(331)	119.97(14)

C(335)-C(336)-H(336)	120.01(9)
C(331)-C(336)-H(336)	120.01(9)
C(421)-P(400)-C(431)	110.35(7)
C(421)-P(400)-C(411)	108.09(7)
C(431)-P(400)-C(411)	109.33(7)
C(421)-P(400)-C(401)	110.98(7)
C(431)-P(400)-C(401)	108.02(7)
C(411)-P(400)-C(401)	110.07(7)
C(402)-C(401)-P(400)	111.96(11)
C(402)-C(401)-H(41A)	110.6(10)
P(400)-C(401)-H(41A)	106.0(10)
C(402)-C(401)-H(41B)	112.9(11)
P(400)-C(401)-H(41B)	107.4(11)
H(41A)-C(401)-H(41B)	108(2)
C(401)-C(402)-H(42A)	107.7(11)
C(401)-C(402)-H(42B)	110.2(11)
H(42A)-C(402)-H(42B)	108(2)
C(401)-C(402)-H(42C)	111.6(11)
H(42A)-C(402)-H(42C)	112(2)
H(42B)-C(402)-H(42C)	108(2)
C(416)-C(411)-C(412)	119.98(14)
C(416)-C(411)-P(400)	119.63(12)
C(412)-C(411)-P(400)	120.30(11)
C(413)-C(412)-C(411)	119.6(2)
C(413)-C(412)-H(412)	120.21(10)
C(411)-C(412)-H(412)	120.21(9)
C(414)-C(413)-C(412)	120.2(2)
C(414)-C(413)-H(413)	119.91(11)
C(412)-C(413)-H(413)	119.91(10)
C(415)-C(414)-C(413)	120.0(2)
C(415)-C(414)-H(414)	120.01(10)
C(413)-C(414)-H(414)	120.01(11)
C(414)-C(415)-C(416)	120.5(2)
C(414)-C(415)-H(415)	119.75(10)
C(416)-C(415)-H(415)	119.75(11)
C(415)-C(416)-C(411)	119.7(2)
C(415)-C(416)-H(416)	120.13(11)
C(411)-C(416)-H(416)	120.13(10)
C(426)-C(421)-C(422)	120.14(13)
C(426)-C(421)-P(400)	120.00(11)
C(422)-C(421)-P(400)	119.63(11)
C(423)-C(422)-C(421)	119.85(14)
C(423)-C(422)-H(422)	120.08(9)
C(421)-C(422)-H(422)	120.08(8)
C(422)-C(423)-C(424)	119.83(14)
C(422)-C(423)-H(423)	120.08(9)
C(424)-C(423)-H(423)	120.08(9)
C(425)-C(424)-C(423)	120.41(14)
C(425)-C(424)-H(424)	119.80(9)
C(423)-C(424)-H(424)	119.80(9)
C(424)-C(425)-C(426)	120.47(14)
C(424)-C(425)-H(425)	119.76(9)
C(426)-C(425)-H(425)	119.76(9)
C(425)-C(426)-C(421)	119.30(14)
C(425)-C(426)-H(426)	120.35(9)
C(421)-C(426)-H(426)	120.35(8)
C(436)-C(431)-C(432)	120.03(13)
C(436)-C(431)-P(400)	119.75(11)
C(432)-C(431)-P(400)	119.93(11)
C(433)-C(432)-C(431)	119.51(14)
C(433)-C(432)-H(432)	120.24(9)
C(431)-C(432)-H(432)	120.24(8)
C(434)-C(433)-C(432)	120.19(14)
C(434)-C(433)-H(433)	119.91(9)
C(432)-C(433)-H(433)	119.91(9)
C(433)-C(434)-C(435)	120.6(2)
C(433)-C(434)-H(434)	119.71(9)
C(435)-C(434)-H(434)	119.71(9)
C(434)-C(435)-C(436)	120.0(2)
C(434)-C(435)-H(435)	119.99(9)
C(436)-C(435)-H(435)	119.99(10)
C(435)-C(436)-C(431)	119.67(14)
C(435)-C(436)-H(436)	120.17(10)
C(431)-C(436)-H(436)	120.17(8)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 17.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
N(100)	30(1)	28(1)	32(1)	-4(1)	5(1)	-1(1)
C(100)	30(1)	29(1)	20(1)	-2(1)	6(1)	-2(1)
C(101)	28(1)	36(1)	28(1)	-4(1)	2(1)	-4(1)
C(102)	28(1)	44(1)	31(1)	-1(1)	2(1)	2(1)
C(103)	36(1)	33(1)	37(1)	-3(1)	2(1)	8(1)
C(104)	36(1)	29(1)	29(1)	-2(1)	2(1)	-2(1)
C(105)	28(1)	29(1)	19(1)	-1(1)	5(1)	-2(1)
C(106)	29(1)	30(1)	18(1)	-2(1)	6(1)	-1(1)
C(107)	32(1)	33(1)	26(1)	-5(1)	7(1)	-5(1)
C(108)	27(1)	46(1)	28(1)	-7(1)	7(1)	-5(1)
C(109)	28(1)	45(1)	29(1)	-4(1)	7(1)	6(1)
C(110)	33(1)	32(1)	31(1)	-4(1)	9(1)	4(1)
C(111)	29(1)	29(1)	21(1)	-4(1)	6(1)	-1(1)
N(200)	29(1)	32(1)	35(1)	0(1)	7(1)	-6(1)
C(200)	34(1)	35(1)	24(1)	-5(1)	7(1)	-2(1)
C(201)	33(1)	51(1)	33(1)	-7(1)	9(1)	1(1)
C(202)	41(1)	67(1)	33(1)	-6(1)	9(1)	16(1)
C(203)	63(1)	50(1)	35(1)	2(1)	12(1)	22(1)
C(204)	56(1)	37(1)	31(1)	1(1)	12(1)	5(1)
C(205)	40(1)	32(1)	20(1)	-2(1)	8(1)	-2(1)
C(206)	37(1)	30(1)	18(1)	-3(1)	6(1)	-6(1)
C(207)	44(1)	34(1)	23(1)	-3(1)	7(1)	-16(1)

C(208)	33(1)	50(1)	25(1)	-3(1)	5(1)	-16(1)
C(209)	30(1)	49(1)	24(1)	0(1)	4(1)	-3(1)
C(210)	34(1)	34(1)	27(1)	2(1)	6(1)	-4(1)
C(211)	31(1)	31(1)	21(1)	-2(1)	6(1)	-8(1)
P(300)	23(1)	28(1)	22(1)	-1(1)	1(1)	-2(1)
C(301)	30(1)	32(1)	25(1)	3(1)	3(1)	-3(1)
C(302)	31(1)	48(1)	39(1)	9(1)	6(1)	-7(1)
C(311)	24(1)	29(1)	27(1)	-6(1)	2(1)	-3(1)
C(312)	32(1)	39(1)	29(1)	-2(1)	4(1)	-5(1)
C(313)	35(1)	42(1)	40(1)	-5(1)	12(1)	-9(1)
C(314)	25(1)	42(1)	49(1)	-10(1)	5(1)	-5(1)
C(315)	29(1)	46(1)	41(1)	-2(1)	-5(1)	0(1)
C(316)	30(1)	38(1)	31(1)	-1(1)	1(1)	-3(1)
C(321)	28(1)	27(1)	22(1)	1(1)	0(1)	-3(1)
C(322)	30(1)	29(1)	27(1)	1(1)	-2(1)	1(1)
C(323)	41(1)	28(1)	32(1)	-1(1)	3(1)	4(1)
C(324)	47(1)	28(1)	26(1)	-1(1)	1(1)	-8(1)
C(325)	33(1)	40(1)	29(1)	1(1)	-4(1)	-11(1)
C(326)	27(1)	35(1)	29(1)	0(1)	1(1)	-3(1)
C(331)	25(1)	27(1)	26(1)	-1(1)	2(1)	-3(1)
C(332)	32(1)	31(1)	25(1)	-2(1)	4(1)	-2(1)
C(333)	37(1)	34(1)	30(1)	4(1)	1(1)	-1(1)
C(334)	35(1)	31(1)	44(1)	3(1)	6(1)	2(1)
C(335)	39(1)	35(1)	38(1)	-5(1)	13(1)	2(1)
C(336)	34(1)	35(1)	27(1)	-1(1)	7(1)	-2(1)
P(400)	22(1)	27(1)	22(1)	0(1)	4(1)	0(1)
C(401)	29(1)	35(1)	27(1)	5(1)	4(1)	1(1)
C(402)	31(1)	44(1)	40(1)	13(1)	-1(1)	2(1)
C(411)	23(1)	26(1)	31(1)	-3(1)	3(1)	2(1)
C(412)	32(1)	31(1)	34(1)	-2(1)	9(1)	-2(1)
C(413)	37(1)	30(1)	56(1)	3(1)	14(1)	0(1)
C(414)	39(1)	27(1)	76(1)	-8(1)	-3(1)	-1(1)
C(415)	55(1)	34(1)	54(1)	-14(1)	-13(1)	2(1)
C(416)	44(1)	32(1)	33(1)	-4(1)	-3(1)	5(1)
C(421)	22(1)	26(1)	26(1)	-1(1)	4(1)	0(1)
C(422)	29(1)	40(1)	24(1)	2(1)	6(1)	1(1)
C(423)	32(1)	43(1)	29(1)	2(1)	0(1)	6(1)
C(424)	24(1)	40(1)	38(1)	-2(1)	3(1)	6(1)
C(425)	27(1)	38(1)	34(1)	0(1)	10(1)	1(1)
C(426)	29(1)	30(1)	26(1)	1(1)	6(1)	1(1)
C(431)	26(1)	25(1)	24(1)	0(1)	5(1)	1(1)
C(432)	25(1)	30(1)	29(1)	1(1)	3(1)	1(1)
C(433)	31(1)	37(1)	33(1)	2(1)	10(1)	9(1)
C(434)	48(1)	34(1)	30(1)	-3(1)	11(1)	9(1)
C(435)	47(1)	30(1)	37(1)	-7(1)	4(1)	-4(1)
C(436)	32(1)	30(1)	33(1)	-2(1)	7(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 17.

	x	y	z	U(eq)
H(101)	306(1)	1068(1)	-124(1)	37
H(102)	-246(1)	2610(1)	-59(1)	41
H(103)	407(1)	4055(1)	226(1)	43
H(104)	1636(1)	3978(1)	467(1)	37
H(107)	3113(1)	3291(1)	717(1)	36
H(108)	4135(1)	2314(1)	869(1)	40
H(109)	4085(1)	597(1)	649(1)	41
H(110)	3018(1)	-187(1)	290(1)	38
H(201)	6345(1)	-2478(1)	4584(1)	46
H(202)	5860(1)	-949(2)	4233(1)	56
H(203)	6578(1)	439(2)	4088(1)	58
H(204)	7807(1)	314(1)	4291(1)	49
H(207)	9255(1)	-417(1)	4554(1)	40
H(208)	10256(1)	-1398(1)	4819(1)	43
H(209)	10142(1)	-3080(1)	5119(1)	41
H(210)	9034(1)	-3809(1)	5155(1)	38
H(31A)	8216(9)	159(13)	893(9)	33(4)

H(31B)	8040(10)	1210(15)	1163(9)	41(5)
H(32A)	9263(10)	1040(15)	858(10)	47(5)
H(32B)	9436(11)	315(16)	1490(10)	51(6)
H(32C)	9240(10)	1450(16)	1605(11)	52(6)
H(312)	7111(1)	-923(1)	2852(1)	40
H(313)	5895(1)	-1242(1)	2747(1)	46
H(314)	5192(1)	-702(1)	1770(1)	46
H(315)	5691(1)	171(1)	906(1)	47
H(316)	6907(1)	518(1)	1002(1)	40
H(322)	7238(1)	1454(1)	2782(1)	35
H(323)	7499(1)	2531(1)	3717(1)	41
H(324)	8651(1)	2651(1)	4237(1)	41
H(325)	9554(1)	1698(1)	3828(1)	41
H(326)	9302(1)	595(1)	2908(1)	37
H(332)	8463(1)	-1091(1)	3261(1)	35
H(333)	9068(1)	-2595(1)	3475(1)	40
H(334)	9513(1)	-3463(1)	2567(1)	44
H(335)	9352(1)	-2838(1)	1441(1)	44
H(336)	8745(1)	-1339(1)	1219(1)	38
H(41A)	2126(9)	9500(14)	1297(9)	39(5)
H(41B)	2007(10)	8492(14)	863(10)	44(5)
H(42A)	990(10)	9511(15)	663(10)	45(5)
H(42B)	903(10)	9638(16)	1456(11)	50(6)
H(42C)	737(10)	8579(15)	1094(10)	47(5)
H(412)	1573(1)	6730(1)	2991(1)	38
H(413)	1005(1)	5182(1)	2881(1)	48
H(414)	627(1)	4558(1)	1787(1)	57
H(415)	849(1)	5449(1)	802(1)	58
H(416)	1418(1)	6990(1)	901(1)	44
H(422)	2878(1)	7585(1)	3229(1)	37
H(423)	4069(1)	7131(1)	3445(1)	42
H(424)	4818(1)	7210(1)	2558(1)	41
H(425)	4388(1)	7743(1)	1459(1)	39
H(426)	3199(1)	8202(1)	1231(1)	34
H(432)	675(1)	8317(1)	2635(1)	34
H(433)	233(1)	9358(1)	3463(1)	40
H(434)	956(1)	10568(1)	4019(1)	44
H(435)	2130(1)	10739(1)	3775(1)	46
H(436)	2584(1)	9709(1)	2949(1)	38