Synthetic, Reactivity and Structural Studies of Pyridyl-N-Phosphinoimines

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Appendix 9 - Full X-Ray Crystallographic Reports

A9.1 – X-Ray Crystallographic Parameters for Compound 1

Parameter	
Empirical formula	$C_{30}H_{41}N_4P$
Formula weight	488.64
Temperature/K	120.0
Crystal system	triclinic
Space group	P-1
a/Å	9.3610(6)
b/Å	10.8701(6)
c/Å	13.5108(8)
α/°	98.648(2)
β/°	91.869(2)
γ/°	90.976(2)
Volume/Å^3	1358.10(14)
Z	2
ρ_{calc}mg/mm^3	1.195
m/mm^{-1}	0.126
F(000)	528.0
Crystal size/mm^3	0.205 × 0.175 × 0.072
2Θ range for data collection	4.494 to 50°
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16
Reflections collected	14945
Independent reflections	4749[R(int) = 0.0689]
Data/restraints/parameters	4749/0/332
Goodness-of-fit on F^2	1.024
Final R indexes [I>=2σ (I)]	R_1 = 0.0581, wR_2 = 0.1481
Final R indexes [all data]	R_1 = 0.0715, wR_2 = 0.1586
Largest diff. peak/hole / e Å^{-3}	0.39/-0.31

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14srv158. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
P	2178.8(5)	6701.1(5)	2454.3(4)	21.87(19)
N1	3303.0(18)	7412.2(16)	3245.8(13)	24.8(4)
N2	2972.0(18)	5317.7(16)	2297.7(13)	23.0(4)
N3	550.8(18)	6646.8(16)	2868.0(13)	22.7(4)
N4	1944.5(18)	7166.3(16)	1350.2(13)	24.5(4)
C1	4357(2)	6597.2(19)	3491.2(15)	24.0(5)
C2	4221(2)	5413.9(19)	2969.0(15)	23.1(5)
C3	5087(2)	4341.3(19)	2899.4(15)	24.1(5)
C4	4719(2)	3248.3(19)	2320.3(15)	23.9(5)
C5	3386(2)	3169(2)	1730.2(16)	26.9(5)
C6	2574(2)	4174.7(19)	1736.8(16)	26.3(5)
C7	-578(2)	5844.1(19)	2283.9(16)	24.9(5)
C8	-846(2)	4654(2)	2726.3(18)	30.8(5)
C9	-1963(2)	6532(2)	2172.3(18)	30.9(5)
C10	234(2)	7117(2)	3933.5(15)	26.4(5)
C11	1088(3)	6505(2)	4697.1(16)	33.1(5)
C12	358(3)	8532(2)	4149.4(18)	33.5(6)
C13	3119(2)	7062(2)	619.2(16)	29.2(5)
C14	4619(2)	7284(2)	1084.0(18)	35.6(6)
C15	3008(3)	5853(2)	-107.9(18)	38.8(6)
C16	954(2)	8206(2)	1257.5(17)	32.0(5)
C17	1718(3)	9479(2)	1467(2)	45.7(7)
C18	137(3)	8007(3)	259.4(19)	45.4(7)
C19	5449(2)	7102.6(19)	4252.5(16)	24.4(5)
C20	5716(2)	8385(2)	4428.1(18)	31.7(5)
C21	6687(3)	8911(2)	5172.9(19)	38.7(6)
C22	7428(2)	8181(2)	5753.9(18)	35.3(6)
C23	7190(2)	6912(2)	5583.6(16)	30.0(5)
C24	6216(2)	6368(2)	4846.8(16)	26.7(5)
C25	5644(2)	2152.3(19)	2276.6(17)	26.6(5)
C26	6467(3)	1955(2)	3113.5(18)	35.9(6)
C27	7374(3)	960(2)	3068(2)	43.9(7)
C28	7475(3)	139(2)	2197(2)	42.0(6)
C29	6648(3)	295(2)	1368.0(19)	37.3(6)
C30	5733(2)	1289(2)	1410.5(17)	28.7(5)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv158. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P	18.5(3)	23.5(3)	24.3(3)	5.1(2)	1.8(2)	2.7(2)
N1	21.1(9)	25.8(9)	27.5(10)	4.4(8)	-0.9(8)	3.1(8)
N2	20.0(9)	23.7(9)	25.4(9)	4.3(7)	-0.5(7)	1.7(7)
N3	18.9(9)	25.1(9)	24.1(9)	3.5(7)	0.9(7)	0.9(7)
N4	21.1(9)	28.3(10)	25.2(10)	7.4(8)	3.2(8)	4.2(8)
C1	21.3(11)	26.8(11)	25.0(11)	6.2(9)	4.5(9)	3.2(9)
C2	19.5(11)	27.5(11)	23.2(11)	6.9(9)	0.0(9)	0.6(9)
C3	20.1(11)	29.0(12)	24.2(11)	6.9(9)	2.8(9)	1.6(9)
C4	24.6(11)	26.8(11)	21.2(11)	5.7(9)	4.7(9)	0.7(9)
C5	26.9(12)	27.1(12)	25.7(11)	0.9(9)	0.9(9)	-0.1(9)
C6	23.8(11)	28.2(12)	25.5(11)	-0.1(9)	-1.3(9)	-0.4(9)
C7	19.3(11)	29.9(12)	24.5(11)	1.1(9)	-1.6(9)	-0.4(9)
C8	24.7(12)	29.7(12)	37.4(13)	3.9(10)	0.4(10)	-3.4(9)
C9	20.4(11)	37.1(13)	34.9(13)	4.5(10)	0.1(10)	3(1)
C10	23.2(11)	32.3(12)	22.5(11)	-1.0(9)	4.2(9)	0.7(9)
C11	33.6(13)	41.2(14)	24.9(12)	6.5(10)	1.2(10)	-0.3(11)
C12	32.6(13)	31.9(13)	34.4(13)	-0.8(10)	4.2(10)	5(1)
C13	24.6(11)	35.9(13)	29.3(12)	10.9(10)	4.6(10)	2(1)
C14	24.3(12)	48.0(15)	37.2(13)	13.6(11)	6.4(10)	0.4(11)
C15	39.4(14)	47.0(15)	29.6(13)	3.0(11)	11.3(11)	0.2(12)
C16	29.3(12)	35.0(13)	34.5(13)	12.2(10)	3.8(10)	10.7(10)
C17	55.6(17)	31.5(14)	53.5(17)	14.5(12)	13.0(14)	9.7(12)
C18	35.9(14)	66.2(19)	38.6(15)	20.4(13)	1.0(12)	15.7(13)
C19	19.1(11)	28.3(11)	24.8(11)	0.1(9)	5.5(9)	1.5(9)
C20	25.9(12)	31.7(12)	37.0(13)	3.6(10)	-0.8(10)	3.1(10)
C21	32.4(13)	31.0(13)	49.2(16)	-4.3(11)	-2.7(12)	-5.2(11)
C22	25.6(12)	46.2(15)	30.6(13)	-4.6(11)	-3(1)	-3.9(11)
C23	23.0(11)	42.3(14)	24.8(11)	3.9(10)	3.3(9)	3(1)
C24	23.8(11)	31.2(12)	24.7(11)	2.8(9)	3.0(9)	2.1(9)
C25	25.6(11)	24.3(11)	30.9(12)	7.0(9)	5.5(9)	-0.9(9)
C26	41.6(14)	32.3(13)	33.0(13)	2.2(10)	-2.7(11)	8.3(11)
C27	50.6(16)	37.9(14)	43.3(15)	7.7(12)	-11.5(13)	14.4(12)
C28	43.3(15)	33.5(14)	50.0(16)	7.4(12)	0.9(13)	15.8(12)
C29	45.1(15)	25.7(12)	40.6(14)	1.4(10)	8.9(12)	5.5(11)
C30	30.9(12)	25.5(11)	30.1(12)	4.6(9)	3.3(10)	3.3(10)

Table 4 Bond Lengths for 14srv158.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
P	N1	1.5808(18)	C7	C9	1.522(3)
P	N2	1.6763(17)	C10	C11	1.522(3)
P	N3	1.6433(17)	C10	C12	1.523(3)
P	N4	1.6547(17)	C13	C14	1.520(3)
N1	C1	1.402(3)	C13	C15	1.518(3)
N2	C2	1.449(3)	C16	C17	1.530(4)
N2	C6	1.393(3)	C16	C18	1.513(3)
N3	C7	1.486(3)	C19	C20	1.395(3)
N3	C10	1.495(3)	C19	C24	1.405(3)
N4	C13	1.496(3)	C20	C21	1.384(3)
N4	C16	1.491(3)	C21	C22	1.378(3)
C1	C2	1.373(3)	C22	C23	1.377(3)
C1	C19	1.465(3)	C23	C24	1.385(3)
C2	C3	1.425(3)	C25	C26	1.393(3)
C3	C4	1.353(3)	C25	C30	1.391(3)
C4	C5	1.452(3)	C26	C27	1.381(3)
C4	C25	1.480(3)	C27	C28	1.373(4)
C5	C6	1.341(3)	C28	C29	1.374(3)
C7	C8	1.525(3)	C29	C30	1.386(3)

Table 5 Bond Angles for 14srv158.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N1	P	N2	97.42(9)	N3	C7	C8	111.62(17)
N1	P	N3	113.89(9)	N3	C7	C9	112.36(17)
N1	P	N4	119.56(9)	C9	C7	C8	110.95(18)
N3	P	N2	113.51(9)	N3	C10	C11	114.25(18)
N3	P	N4	103.67(9)	N3	C10	C12	111.14(17)
N4	P	N2	109.07(9)	C11	C10	C12	112.06(19)
C1	N1	P	110.32(14)	N4	C13	C14	115.08(18)
C2	N2	P	107.79(13)	N4	C13	C15	111.95(18)
C6	N2	P	132.05(15)	C15	C13	C14	111.42(19)
C6	N2	C2	119.91(17)	N4	C16	C17	112.23(19)
C7	N3	P	120.61(13)	N4	C16	C18	111.49(19)
C7	N3	C10	115.97(16)	C18	C16	C17	112.3(2)
C10	N3	P	121.58(14)	C20	C19	C1	118.86(19)
C13	N4	P	120.62(13)	C20	C19	C24	117.7(2)
C16	N4	P	118.86(14)	C24	C19	C1	123.43(19)
C16	N4	C13	113.98(16)	C21	C20	C19	120.9(2)
N1	C1	C19	117.07(18)	C22	C21	C20	120.8(2)

C2	C1	N1	114.35(19)	C23	C22	C21	119.0(2)
C2	C1	C19	128.58(19)	C22	C23	C24	121.1(2)
C1	C2	N2	110.02(18)	C23	C24	C19	120.5(2)
C1	C2	C3	133.82(19)	C26	C25	C4	120.5(2)
C3	C2	N2	115.93(18)	C30	C25	C4	122.0(2)
C4	C3	C2	123.1(2)	C30	C25	C26	117.6(2)
C3	C4	C5	118.5(2)	C27	C26	C25	120.8(2)
C3	C4	C25	121.4(2)	C28	C27	C26	120.6(2)
C5	C4	C25	120.10(19)	C27	C28	C29	119.7(2)
C6	C5	C4	120.2(2)	C28	C29	C30	119.9(2)
C5	C6	N2	121.8(2)	C29	C30	C25	121.3(2)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv158.

Atom	*x*	*y*	*z*	U(eq)
H3	5966	4396	3277	29
H5	3089	2402	1338	32
H6	1704	4105	1348	32
H7	-218	5593	1596	30
H8A	60	4234	2799	34(4)
H8B	-1507	4103	2279	34(4)
H8C	-1265	4861	3384	34(4)
H9A	-2281	6889	2836	46(4)
H9B	-2698	5950	1835	46(4)
H9C	-1800	7201	1774	46(4)
H10	-793	6898	4015	32
H11A	992	5598	4526	35(4)
H11B	723	6766	5366	35(4)
H11C	2098	6756	4691	35(4)
H12A	1347	8796	4067	41(4)
H12B	82	8819	4838	41(4)
H12C	-277	8890	3682	41(4)
H13	2963	7740	205	35
H14A	4660	8075	1542	46(4)
H14B	5303	7317	554	46(4)
H14C	4862	6604	1456	46(4)
H15A	3293	5161	239	57(5)
H15B	3639	5902	-666	57(5)
H15C	2018	5713	-366	57(5)
H16	233	8193	1786	38
H17A	2270	9550	2103	51(5)

H17B	1010	10137	1509	51(5)
H17C	2364	9566	924	51(5)
H18A	805	8028	-281	47(4)
H18B	-560	8668	244	47(4)
H18C	-365	7197	170	47(4)
H20	5223	8903	4031	38
H21	6844	9788	5285	46
H22	8093	8547	6264	42
H23	7702	6401	5978	36
H24	6065	5490	4742	32
H26	6405	2512	3723	43
H27	7931	842	3645	53
H28	8116	-533	2167	50
H29	6702	-279	767	45
H30	5156	1384	836	34

Experimental

Single crystals of $C_{30}H_{41}N_4P$ **[14srv158]** were **[]**. A suitable crystal was selected and **[]** on a **D8V** diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Crystal structure determination of [14srv158]

Crystal Data for $C_{30}H_{41}N_4P$ (M =488.64 g/mol): triclinic, space group P-1 (no. 2), a = 9.3610(6) Å, b = 10.8701(6) Å, c = 13.5108(8) Å, α = 98.648(2)°, β = 91.869(2)°, γ = 90.976(2)°, V = 1358.10(14) Å^3, Z = 2, T = 120 K, μ(MoKα) = 0.126 mm^{-1}, *Dcalc* = 1.195 g/cm^3, 14945 reflections measured (4.494° ≤ 2Θ ≤ 49.996°), 4749 unique (R_{int} = 0.0689, R_{sigma} = 0.0727) which were used in all calculations. The final R_1 was 0.0581 (I > 2σ(I)) and wR_2 was 0.1586 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2. Others
 Uiso(H8A)=Uiso(H8B)=Uiso(H8C)=FVAR(1)
 Uiso(H9A)=Uiso(H9B)=Uiso(H9C)=FVAR(2)
 Uiso(H11A)=Uiso(H11B)=Uiso(H11C)=FVAR(3)
 Uiso(H12A)=Uiso(H12B)=Uiso(H12C)=FVAR(4)
 Uiso(H14A)=Uiso(H14B)=Uiso(H14C)=FVAR(5)
 Uiso(H15A)=Uiso(H15B)=Uiso(H15C)=FVAR(6)
 Uiso(H17A)=Uiso(H17B)=Uiso(H17C)=FVAR(7)
 Uiso(H18A)=Uiso(H18B)=Uiso(H18C)=FVAR(8)
3.a Riding coordinates:
 C3(H3), C5(H5), C6(H6), C7(H7), C10(H10), C13(H13), C16(H16), C20(H20),
 C21(H21), C22(H22), C23(H23), C24(H24), C26(H26), C27(H27), C28(H28), C29(H29),
  C30(H30)
3.b Rotating group:

C8(H8A,H8B,H8C), C9(H9A,H9B,H9C), C11(H11A,H11B,H11C), C12(H12A,H12B,H12C), C14(H14A,H14B,H14C), C15(H15A,H15B,H15C), C17(H17A,H17B,H17C), C18(H18A,H18B, H18C)

A9.2 – X-Ray Crystallographic Parameters for Compound 2

Parameter	
Empirical formula	$C_{30}H_{41}N_4P$
Formula weight	488.64
Temperature/K	120
Crystal system	triclinic
Space group	P-1
a/Å	9.5302(3)
b/Å	11.0544(4)
c/Å	13.8558(5)
α/°	88.3900(15)
β/°	70.2419(14)
γ/°	84.0851(14)
Volume/Å^3	1366.43(8)
Z	2
ρ_{calc}g/cm^3	1.188
μ/mm^{-1}	0.126
F(000)	528.0
Crystal size/mm^3	0.332 × 0.243 × 0.106
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.564 to 60.186
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -18 ≤ l ≤ 19
Reflections collected	25700
Independent reflections	8051 [R_{int} = 0.0346, R_{sigma} = 0.0464]
Data/restraints/parameters	8051/0/332
Goodness-of-fit on F^2	1.021
Final R indexes [I>=2σ (I)]	R_1 = 0.0466, wR_2 = 0.1118
Final R indexes [all data]	R_1 = 0.0724, wR_2 = 0.1232
Largest diff. peak/hole / e Å^{-3}	0.47/-0.34

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16srv085. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
P	6949.0(4)	2174.1(3)	2205.0(3)	15.02(9)
N1	6877.5(12)	692.5(10)	2510.2(8)	16.5(2)
N2	5277.1(12)	2628.1(10)	2861.9(8)	15.9(2)
N3	7513.4(13)	2275.2(10)	941.8(8)	18.8(2)
N4	8230.9(12)	2820.5(10)	2511.1(9)	18.9(2)
C1	4568.3(14)	1656.0(11)	3441.5(10)	15.0(2)
C2	5442.4(14)	552.0(12)	3278.4(10)	15.7(2)
C3	5264.3(15)	-612.6(12)	3748.7(10)	19.1(3)
C4	6362.5(16)	-1543.0(12)	3459.6(11)	21.5(3)
C5	7765.7(16)	-1371.6(13)	2677.4(11)	22.7(3)
C6	7995.3(15)	-278.8(12)	2240.5(11)	20.7(3)
C7	6843.1(16)	1579.0(13)	325.4(11)	22.5(3)
C8	7701.0(18)	332.2(15)	-13.8(13)	30.8(3)
C9	5165.5(17)	1484.9(16)	818.2(12)	29.2(3)
C10	8083.6(16)	3421.7(13)	441.1(11)	23.9(3)
C11	9406.1(18)	3150.1(15)	-545.9(13)	34.4(4)
C12	6848.4(19)	4300.0(15)	264.1(13)	33.3(4)
C13	9854.1(15)	2411.8(13)	2023.4(12)	23.4(3)
C14	10458.4(18)	1579.5(15)	2718.7(14)	31.0(4)
C15	10810.2(18)	3475.5(16)	1680.8(16)	40.5(4)
C16	7808.2(18)	3513.6(14)	3498.4(12)	28.3(3)
C17	7039(2)	2784(2)	4442.2(13)	39.7(4)
C18	6929(2)	4727.1(16)	3447.7(17)	42.4(5)
C19	3070.2(14)	1920.7(11)	4187(1)	14.5(2)
C20	2555.9(15)	3116.5(12)	4537(1)	17.5(3)
C21	1171.4(15)	3399.4(12)	5283.2(10)	18.4(3)
C22	204.5(14)	2512.1(11)	5717.2(10)	15.0(2)
C23	702.6(15)	1323.6(12)	5343.6(10)	16.9(3)
C24	2077.9(15)	1033.2(12)	4600(1)	17.1(3)
C25	-1285.4(14)	2797.6(12)	6511.4(10)	15.9(2)
C26	-1987.1(16)	3983.4(12)	6678.1(11)	20.5(3)
C27	-3422.1(16)	4241.0(13)	7373.4(11)	24.8(3)
C28	-4203.7(16)	3309.2(14)	7919.4(11)	25.9(3)
C29	-3520.2(16)	2126.7(13)	7782.8(11)	24.2(3)
C30	-2079.1(16)	1878.0(13)	7101.5(11)	21.1(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16srv085. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P	13.54(16)	15.33(16)	14.51(16)	0.37(12)	-2.75(12)	-0.75(12)
N1	14.3(5)	15.5(5)	16.6(5)	0.2(4)	-1.8(4)	0.8(4)
N2	14.1(5)	16.2(5)	15.4(5)	0.0(4)	-2.5(4)	-0.8(4)
N3	18.5(5)	20.9(6)	14.5(5)	1.4(4)	-2.4(4)	-2.4(4)
N4	14.6(5)	20.2(5)	21.9(6)	-0.7(4)	-5.8(5)	-2.3(4)
C1	14.7(6)	15.1(6)	14.2(6)	-0.5(5)	-3.4(5)	-1.5(5)
C2	14.1(6)	17.5(6)	13.8(6)	0.5(5)	-2.3(5)	-1.9(5)
C3	20.2(7)	18.4(6)	18.0(6)	1.5(5)	-5.6(5)	-2.0(5)
C4	27.0(7)	15.6(6)	22.3(7)	1.8(5)	-9.4(6)	-0.3(5)
C5	23.8(7)	19.6(6)	22.3(7)	-3.5(5)	-6.9(6)	7.1(5)
C6	17.4(6)	20.6(6)	20.3(7)	-3.0(5)	-3.1(5)	3.9(5)
C7	22.2(7)	27.7(7)	15.9(6)	-2.9(5)	-4.8(6)	0.6(6)
C8	29.6(8)	34.2(8)	27.1(8)	-11.1(6)	-8.2(7)	2.4(7)
C9	21.7(7)	40.5(9)	26.9(8)	-8.1(7)	-10.2(6)	-1.0(6)
C10	22.7(7)	21.9(7)	20.5(7)	4.5(5)	0.5(6)	-0.9(5)
C11	28.6(8)	33.5(9)	27.6(8)	7.4(7)	6.9(7)	-0.1(7)
C12	36.5(9)	27.5(8)	26.3(8)	5.0(6)	-1.5(7)	7.5(7)
C13	15.6(6)	25.1(7)	28.5(8)	3.3(6)	-6.6(6)	-1.7(5)
C14	24.5(8)	30.5(8)	41.5(9)	4.3(7)	-16.9(7)	0.3(6)
C15	22.0(8)	38.7(10)	60.0(12)	16.1(9)	-11.7(8)	-11.4(7)
C16	26.1(8)	31.2(8)	31.0(8)	-9.7(6)	-13.1(7)	-3.6(6)
C17	31.6(9)	68.4(13)	21.1(8)	-7.2(8)	-10.2(7)	-7.8(9)
C18	37.4(10)	30.0(9)	63.2(13)	-21.4(8)	-21.2(9)	1.2(7)
C19	14.3(6)	15.8(6)	13.5(6)	-0.7(4)	-5.0(5)	-0.1(5)
C20	15.9(6)	14.7(6)	19.7(6)	1.4(5)	-3.3(5)	-1.7(5)
C21	18.3(6)	13.4(6)	21.1(7)	-1.3(5)	-4.2(5)	1.2(5)
C22	14.8(6)	16.1(6)	14.0(6)	-0.6(5)	-5.0(5)	0.1(5)
C23	16.1(6)	15.9(6)	17.9(6)	-0.7(5)	-4.0(5)	-3.0(5)
C24	17.2(6)	15.4(6)	18.0(6)	-3.6(5)	-4.8(5)	-0.6(5)
C25	15.7(6)	18.9(6)	13.0(6)	-1.8(5)	-5.1(5)	0.3(5)
C26	20.4(7)	18.2(6)	20.0(7)	-0.6(5)	-3.8(5)	0.3(5)
C27	22.3(7)	22.9(7)	24.2(7)	-3.2(6)	-3.1(6)	4.7(6)
C28	18.1(7)	30.4(8)	23.2(7)	-4.5(6)	0.1(6)	0.9(6)
C29	21.9(7)	25.4(7)	20.2(7)	0.1(6)	0.4(6)	-5.1(6)
C30	21.6(7)	19.2(6)	19.2(7)	-0.6(5)	-3.1(6)	-0.5(5)

Table 4 Bond Lengths for 16srv085.

Atom	Atom	Length/Å	Atom	Atom	Length/Å

P	N1	1.6825(11)	C10	C11	1.526(2)
P	N2	1.5810(11)	C10	C12	1.528(2)
P	N3	1.6524(12)	C13	C14	1.524(2)
P	N4	1.6522(12)	C13	C15	1.528(2)
N1	C2	1.4415(16)	C16	C17	1.521(2)
N1	C6	1.3946(16)	C16	C18	1.520(2)
N2	C1	1.4108(16)	C19	C20	1.4017(17)
N3	C7	1.4956(18)	C19	C24	1.4065(18)
N3	C10	1.4900(17)	C20	C21	1.3851(18)
N4	C13	1.4900(17)	C21	C22	1.3969(18)
N4	C16	1.4978(19)	C22	C23	1.4011(17)
C1	C2	1.3832(17)	C22	C25	1.4823(17)
C1	C19	1.4576(17)	C23	C24	1.3789(18)
C2	C3	1.4262(18)	C25	C26	1.3967(18)
C3	C4	1.3533(19)	C25	C30	1.4027(18)
C4	C5	1.435(2)	C26	C27	1.3870(19)
C5	C6	1.3407(19)	C27	C28	1.386(2)
C7	C8	1.528(2)	C28	C29	1.387(2)
C7	C9	1.525(2)	C29	C30	1.3832(19)

Table 5 Bond Angles for 16srv085.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N2	P	N1	97.51(6)	C9	C7	C8	111.37(13)
N2	P	N3	119.80(6)	N3	C10	C11	110.91(12)
N2	P	N4	114.53(6)	N3	C10	C12	112.33(12)
N3	P	N1	107.86(6)	C11	C10	C12	111.98(13)
N3	P	N4	103.37(6)	N4	C13	C14	112.24(12)
N4	P	N1	114.02(6)	N4	C13	C15	112.51(12)
C2	N1	P	108.23(8)	C14	C13	C15	109.64(13)
C6	N1	P	130.41(10)	N4	C16	C17	113.88(13)
C6	N1	C2	120.76(11)	N4	C16	C18	111.10(14)
C1	N2	P	109.73(8)	C18	C16	C17	112.73(15)
C7	N3	P	121.01(9)	C20	C19	C1	119.80(11)
C10	N3	P	119.50(9)	C20	C19	C24	116.42(11)
C10	N3	C7	113.23(11)	C24	C19	C1	123.78(11)
C13	N4	P	120.82(10)	C21	C20	C19	121.73(12)
C13	N4	C16	115.97(11)	C20	C21	C22	121.85(12)
C16	N4	P	120.23(9)	C21	C22	C23	116.28(12)
N2	C1	C19	118.12(11)	C21	C22	C25	122.67(11)
C2	C1	N2	114.75(11)	C23	C22	C25	121.03(11)
C2	C1	C19	126.97(11)	C24	C23	C22	122.29(12)

C1	C2	N1	109.42(11)	C23	C24	C19	121.36(12)
C1	C2	C3	134.22(12)	C26	C25	C22	121.66(12)
C3	C2	N1	116.15(11)	C26	C25	C30	117.03(12)
C4	C3	C2	121.69(13)	C30	C25	C22	121.26(12)
C3	C4	C5	120.37(13)	C27	C26	C25	121.73(13)
C6	C5	C4	119.75(13)	C28	C27	C26	120.07(13)
C5	C6	N1	121.23(13)	C27	C28	C29	119.31(13)
N3	C7	C8	112.51(12)	C30	C29	C28	120.35(13)
N3	C7	C9	114.72(11)	C29	C30	C25	121.44(13)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16srv085.

Atom	*x*	*y*	*z*	U(eq)
H3	4351	-738	4279	23
H4	6204	-2314	3776	26
H5	8529	-2030	2470	27
H6	8937	-164	1738	25
H7	6965	2052	-319	27
H8A	8780	404	-238	39(3)
H8B	7443	26	-583	39(3)
H8C	7430	-234	562	39(3)
H9A	4986	951	1415	37(3)
H9B	4784	1148	320	37(3)
H9C	4648	2295	1036	37(3)
H10	8466	3834	922	29
H11A	10160	2569	-404	45(3)
H11B	9849	3905	-811	45(3)
H11C	9058	2801	-1057	45(3)
H12A	6532	3971	-271	46(3)
H12B	7230	5089	47	46(3)
H12C	5991	4403	902	46(3)
H13	9967	1932	1396	28
H14A	10422	2038	3323	38(3)
H14B	11497	1268	2344	38(3)
H14C	9845	897	2936	38(3)
H15A	10420	4008	1233	58(4)
H15B	11849	3164	1306	58(4)
H15C	10775	3937	2284	58(4)
H16	8770	3710	3570	34
H17A	6042	2635	4444	51(3)
H17B	6941	3242	5061	51(3)

H17C	7638	2006	4430	51(3)
H18A	7508	5188	2858	52(4)
H18B	6739	5191	4080	52(4)
H18C	5972	4586	3373	52(4)
H20	3173	3750	4255	21
H21	871	4220	5506	22
H23	71	696	5612	20
H24	2361	217	4362	21
H26	-1468	4630	6305	25
H27	-3870	5057	7475	30
H28	-5197	3479	8382	31
H29	-4045	1484	8159	29
H30	-1617	1067	7032	25

Experimental

Single crystals of $C_{30}H_{41}N_4P$ **[16srv085]** were **[]**. A suitable crystal was selected and **[]** on a **Bruker D8 Venture** diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the SIR2004 [2] structure solution program using Direct Methods and refined with the XH [3] refinement package using CGLS minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2.
3.

Crystal structure determination of [16srv085]

Crystal Data for $C_{30}H_{41}N_4P$ (M =488.64 g/mol): triclinic, space group P-1 (no. 2), a = 9.5302(3) Å, b = 11.0544(4) Å, c = 13.8558(5) Å, α = 88.3900(15)°, β = 70.2419(14)°, γ = 84.0851(14)°, V = 1366.43(8) Å^3, Z = 2, T = 120 K, μ(MoKα) = 0.126 mm^{-1}, *Dcalc* = 1.188 g/cm^3, 25700 reflections measured (4.564° ≤ 2Θ ≤ 60.186°), 8051 unique (R_{int} = 0.0346, R_{sigma} = 0.0464) which were used in all calculations. The final R_1 was 0.0466 (I > 2σ(I)) and wR_2 was 0.1232 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2. Others
 Uiso(H8A)=Uiso(H8B)=Uiso(H8C)=FVAR(1)
 Uiso(H9A)=Uiso(H9B)=Uiso(H9C)=FVAR(2)
 Uiso(H11A)=Uiso(H11B)=Uiso(H11C)=FVAR(3)
 Uiso(H12A)=Uiso(H12B)=Uiso(H12C)=FVAR(4)
 Uiso(H14A)=Uiso(H14B)=Uiso(H14C)=FVAR(5)
 Uiso(H15A)=Uiso(H15B)=Uiso(H15C)=FVAR(6)
 Uiso(H17A)=Uiso(H17B)=Uiso(H17C)=FVAR(7)
 Uiso(H18A)=Uiso(H18B)=Uiso(H18C)=FVAR(8)
3.a Ternary CH refined with riding coordinates:
 C7(H7), C10(H10), C13(H13), C16(H16)
3.b Aromatic/amide H refined with riding coordinates:
 C3(H3), C4(H4), C5(H5), C6(H6), C20(H20), C21(H21), C23(H23), C24(H24),
 C26(H26), C27(H27), C28(H28), C29(H29), C30(H30)
3.c Idealised Me refined as rotating group:
 C8(H8A,H8B,H8C), C9(H9A,H9B,H9C), C11(H11A,H11B,H11C), C12(H12A,H12B,H12C),

C14(H14A,H14B,H14C), C15(H15A,H15B,H15C), C17(H17A,H17B,H17C), C18(H18A,H18B, H18C)

A9.3 – X-Ray Crystallographic Parameters for Compound 4

Parameter	
Empirical formula	$C_{27}H_{43}N_4P$
Formula weight	454.62
Temperature/K	120
Crystal system	monoclinic
Space group	C2/c
a/Å	24.8627(11)
b/Å	9.2747(4)
c/Å	25.1947(11)
α/°	90
β/°	110.6439(15)
γ/°	90
Volume/Å^3	5436.7(4)
Z	8
ρ_{calc}g/cm^3	1.111
μ/mm^{-1}	0.122
F(000)	1984.0
Crystal size/mm^3	0.238 × 0.235 × 0.218
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.728 to 60.312
Index ranges	-35 ≤ h ≤ 34, -13 ≤ k ≤ 13, -35 ≤ l ≤ 35
Reflections collected	58568
Independent reflections	8027 [R_{int} = 0.0460, R_{sigma} = 0.0316]
Data/restraints/parameters	8027/0/320
Goodness-of-fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0430, wR_2 = 0.1035
Final R indexes [all data]	R_1 = 0.0588, wR_2 = 0.1110
Largest diff. peak/hole / e Å^{-3}	0.44/-0.34

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 15srv176. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
P	1403.9(2)	3731.9(3)	1233.3(2)	12.23(7)
N(1)	1356.1(4)	4338.1(11)	588.8(4)	13.12(19)
N(2)	2066.9(4)	3345.0(11)	1456.5(4)	14.75(19)
N(3)	929.8(4)	2412.7(11)	1154.1(4)	15.32(19)
N(4)	1212.2(4)	4924.3(11)	1618.2(4)	14.50(19)
C(1)	2301.2(5)	3689.1(13)	1038.9(5)	13.1(2)
C(2)	1920.8(5)	4285.5(12)	550.3(5)	12.7(2)
C(3)	1986.6(5)	4966.6(12)	67.5(5)	13.5(2)
C(4)	1551.0(5)	5664.1(13)	-334.8(5)	14.2(2)
C(5)	994.5(5)	5702.5(14)	-264.1(5)	16.3(2)
C(6)	915.0(5)	5074.5(13)	181.9(5)	16.0(2)
C(7)	1615.6(5)	6440.3(14)	-842.2(5)	17.3(2)
C(8)	1491.6(6)	8052.4(15)	-804.8(6)	26.6(3)
C(9)	1182.8(6)	5827.9(16)	-1395.6(5)	23.9(3)
C(10)	2217.4(6)	6269.8(18)	-864.5(6)	28.8(3)
C(11)	2921.1(5)	3481.2(12)	1180.3(5)	13.0(2)
C(12)	3281.5(5)	3567.8(13)	1748.8(5)	16.8(2)
C(13)	3874.1(5)	3463.4(14)	1901.9(5)	20.0(2)
C(14)	4119.6(5)	3249.7(14)	1493.1(6)	20.7(2)
C(15)	3767.2(5)	3100.3(14)	931.1(6)	20.5(2)
C(16)	3174.6(5)	3206.3(13)	775.3(5)	16.5(2)
C(17)	843.2(5)	1288.9(14)	706.5(6)	20.8(2)
C(18)	380.4(6)	1741.7(16)	151.3(6)	25.5(3)
C(19)	1389.8(6)	785.3(16)	615.4(7)	27.7(3)
C(20)	871.9(5)	1852.8(15)	1686.3(5)	21.5(3)
C(21)	258.6(6)	1376.0(17)	1592.0(7)	30.1(3)
C(22)	1310.1(6)	683.3(19)	1965.7(8)	37.5(4)
C(23)	1643.0(5)	5958.7(14)	1987.8(5)	20.0(2)
C(24)	2037.5(6)	5241.5(18)	2526.4(6)	29.2(3)
C(25)	1976.2(6)	6777.5(16)	1676.8(7)	29.3(3)
C(26)	601.0(5)	5308.8(14)	1508.0(5)	18.3(2)
C(27)	442.6(6)	6774.7(17)	1220.1(6)	28.7(3)
C(28)	451.6(6)	5271.9(17)	2048.1(6)	25.5(3)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for 15srv176. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}

P	9.61(12)	14.73(14)	13.27(13)	2.36(10)	5.19(10)	0.14(10)
N(1)	9.9(4)	16.6(5)	13.8(4)	2.9(4)	5.3(3)	0.3(3)
N(2)	10.4(4)	20.0(5)	15.0(4)	3.8(4)	5.9(4)	1.0(4)
N(3)	12.7(4)	16.0(5)	18.3(5)	3.5(4)	6.8(4)	-1.8(4)
N(4)	9.6(4)	19.7(5)	15.0(4)	-0.7(4)	5.3(3)	-0.4(4)
C(1)	10.7(5)	14.7(5)	15.2(5)	0.3(4)	5.9(4)	-0.2(4)
C(2)	10.7(5)	14.1(5)	14.9(5)	-0.6(4)	6.4(4)	-0.3(4)
C(3)	12.6(5)	14.4(5)	15.0(5)	-1.2(4)	6.8(4)	-2.2(4)
C(4)	15.1(5)	14.4(5)	14.1(5)	-0.7(4)	6.4(4)	-2.4(4)
C(5)	12.9(5)	19.9(6)	15.1(5)	2.5(4)	3.7(4)	1.8(4)
C(6)	10.9(5)	19.8(6)	16.8(5)	1.2(4)	4.4(4)	1.4(4)
C(7)	17.0(5)	20.3(6)	14.4(5)	2.5(4)	5.4(4)	-3.7(4)
C(8)	32.8(7)	20.2(7)	23.2(6)	4.7(5)	5.2(5)	-5.4(5)
C(9)	26.7(6)	29.4(7)	14.6(5)	-1.2(5)	6.1(5)	-5.8(5)
C(10)	20.5(6)	46.1(9)	22.8(6)	11.3(6)	11.3(5)	-1.8(6)
C(11)	11.0(5)	11.0(5)	17.1(5)	0.6(4)	5.2(4)	-0.1(4)
C(12)	14.3(5)	19.8(6)	17.1(5)	-1.0(4)	6.6(4)	1.5(4)
C(13)	13.9(5)	22.6(6)	21.1(6)	-2.5(5)	3.2(4)	0.6(4)
C(14)	11.8(5)	20.8(6)	30.1(7)	-0.2(5)	8.1(5)	2.3(4)
C(15)	18.0(6)	22.9(6)	25.3(6)	0.2(5)	13.4(5)	4.4(5)
C(16)	15.7(5)	17.1(6)	17.8(5)	-1.3(4)	7.1(4)	2.8(4)
C(17)	18.5(6)	16.2(6)	28.9(7)	-1.9(5)	10.0(5)	-3.9(5)
C(18)	22.8(6)	26.9(7)	25.2(6)	-5.1(5)	6.6(5)	-6.3(5)
C(19)	25.5(7)	21.3(7)	40.0(8)	-6.0(6)	16.0(6)	0.2(5)
C(20)	17.1(5)	26.4(7)	22.5(6)	10.2(5)	8.9(5)	-2.2(5)
C(21)	21.1(6)	33.6(8)	40.5(8)	11.1(6)	17.1(6)	-3.5(6)
C(22)	24.7(7)	41.5(9)	43.9(9)	27.2(8)	9.0(6)	4.5(6)
C(23)	15.8(5)	23.2(6)	23.0(6)	-7.2(5)	9.6(5)	-4.3(5)
C(24)	17.9(6)	47.4(9)	19.9(6)	-6.6(6)	3.8(5)	-2.5(6)
C(25)	29.4(7)	23.3(7)	43.6(8)	-7.6(6)	23.4(7)	-9.6(6)
C(26)	10.9(5)	26.4(6)	17.3(5)	-3.3(5)	4.9(4)	2.5(4)
C(27)	28.7(7)	33.1(8)	23.4(7)	0.0(6)	8.0(5)	14.3(6)
C(28)	18.1(6)	38.0(8)	25.5(6)	-6.1(6)	13.9(5)	-2.5(5)

Table 4 Bond Lengths for 15srv176.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
P	N(1)	1.6823(10)	C(7)	C(8)	1.5362(19)
P	N(2)	1.5837(10)	C(7)	C(9)	1.5384(17)
P	N(3)	1.6615(10)	C(7)	C(10)	1.5253(17)
P	N(4)	1.6484(10)	C(11)	C(12)	1.3986(16)
N(1)	C(2)	1.4419(13)	C(11)	C(16)	1.3990(16)

N(1)	C(6)	1.3884(14)	C(12)	C(13)	1.3886(16)
N(2)	C(1)	1.4071(14)	C(13)	C(14)	1.3841(18)
N(3)	C(17)	1.4944(16)	C(14)	C(15)	1.3846(19)
N(3)	C(20)	1.4915(15)	C(15)	C(16)	1.3881(16)
N(4)	C(23)	1.4936(15)	C(17)	C(18)	1.5239(19)
N(4)	C(26)	1.4886(14)	C(17)	C(19)	1.5289(18)
C(1)	C(2)	1.3765(16)	C(20)	C(21)	1.5238(17)
C(1)	C(11)	1.4671(15)	C(20)	C(22)	1.522(2)
C(2)	C(3)	1.4304(15)	C(23)	C(24)	1.519(2)
C(3)	C(4)	1.3582(16)	C(23)	C(25)	1.5281(18)
C(4)	C(5)	1.4564(15)	C(26)	C(27)	1.526(2)
C(4)	C(7)	1.5241(16)	C(26)	C(28)	1.5321(17)
C(5)	C(6)	1.3405(16)			

Table 5 Bond Angles for 15srv176.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N(2)	P	N(1)	97.41(5)	C(5)	C(6)	N(1)	121.42(10)
N(2)	P	N(3)	118.77(5)	C(4)	C(7)	C(8)	108.99(10)
N(2)	P	N(4)	115.07(5)	C(4)	C(7)	C(9)	109.82(10)
N(3)	P	N(1)	108.94(5)	C(4)	C(7)	C(10)	112.14(10)
N(4)	P	N(1)	114.01(5)	C(8)	C(7)	C(9)	108.88(11)
N(4)	P	N(3)	102.98(5)	C(10)	C(7)	C(8)	108.90(11)
C(2)	N(1)	P	108.33(7)	C(10)	C(7)	C(9)	108.05(11)
C(6)	N(1)	P	130.23(8)	C(12)	C(11)	C(1)	118.69(10)
C(6)	N(1)	C(2)	120.08(9)	C(12)	C(11)	C(16)	117.82(10)
C(1)	N(2)	P	109.66(8)	C(16)	C(11)	C(1)	123.49(10)
C(17)	N(3)	P	120.77(8)	C(13)	C(12)	C(11)	120.96(11)
C(20)	N(3)	P	115.96(8)	C(14)	C(13)	C(12)	120.43(12)
C(20)	N(3)	C(17)	113.69(10)	C(13)	C(14)	C(15)	119.29(11)
C(23)	N(4)	P	120.34(7)	C(14)	C(15)	C(16)	120.52(11)
C(26)	N(4)	P	122.61(8)	C(15)	C(16)	C(11)	120.86(11)
C(26)	N(4)	C(23)	115.02(9)	N(3)	C(17)	C(18)	111.21(11)
N(2)	C(1)	C(11)	117.71(10)	N(3)	C(17)	C(19)	115.29(10)
C(2)	C(1)	N(2)	115.14(9)	C(18)	C(17)	C(19)	111.71(12)
C(2)	C(1)	C(11)	126.98(10)	N(3)	C(20)	C(21)	111.67(11)
C(1)	C(2)	N(1)	109.36(9)	N(3)	C(20)	C(22)	112.55(11)
C(1)	C(2)	C(3)	133.60(10)	C(22)	C(20)	C(21)	112.27(12)
C(3)	C(2)	N(1)	116.64(10)	N(4)	C(23)	C(24)	111.77(11)
C(4)	C(3)	C(2)	123.00(10)	N(4)	C(23)	C(25)	113.51(11)
C(3)	C(4)	C(5)	117.55(10)	C(24)	C(23)	C(25)	112.31(11)
C(3)	C(4)	C(7)	123.96(10)	N(4)	C(26)	C(27)	112.18(11)

C(5)	C(4)	C(7)	118.47(10)	N(4)	C(26)	C(28)	112.34(10)
C(6)	C(5)	C(4)	121.26(11)	C(27)	C(26)	C(28)	109.84(11)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15srv176.

Atom	*x*	*y*	*z*	U(eq)
H(3)	2352	4929	26	16
H(5)	682	6182	-539	20
H(6)	549	5135	220	19
H(8A)	1099	8175	-809	38(3)
H(8B)	1533	8560	-1129	38(3)
H(8C)	1764	8451	-452	38(3)
H(9A)	1270	4810	-1432	32(3)
H(9B)	1211	6369	-1719	32(3)
H(9C)	793	5915	-1388	32(3)
H(10A)	2499	6701	-525	39(3)
H(10B)	2236	6756	-1203	39(3)
H(10C)	2303	5243	-881	39(3)
H(12)	3119	3700	2034	20
H(13)	4113	3539	2290	24
H(14)	4526	3206	1597	25
H(15)	3932	2924	650	25
H(16)	2938	3091	389	20
H(17)	688	423	841	25
H(18A)	29	1996	222	33(3)
H(18B)	300	942	-120	33(3)
H(18C)	514	2578	-5	33(3)
H(19A)	1545	1576	454	35(3)
H(19B)	1299	-36	354	35(3)
H(19C)	1676	493	980	35(3)
H(20)	956	2679	1958	26
H(21A)	-7	2167	1420	43(3)
H(21B)	226	1117	1957	43(3)
H(21C)	162	538	1339	43(3)
H(22A)	1223	-183	1727	44(3)
H(22B)	1293	452	2339	44(3)
H(22C)	1696	1028	2010	44(3)
H(23)	1419	6700	2111	24
H(24A)	1806	4744	2714	38(3)
H(24B)	2277	5975	2782	38(3)
H(24C)	2285	4542	2431	38(3)

H(25A)	2223	6105	1568	36(3)
H(25B)	2214	7522	1927	36(3)
H(25C)	1705	7231	1336	36(3)
H(26)	357	4569	1242	22
H(27A)	612	7540	1497	37(3)
H(27B)	24	6880	1070	37(3)
H(27C)	591	6845	908	37(3)
H(28A)	510	4294	2206	36(3)
H(28B)	49	5552	1957	36(3)
H(28C)	700	5945	2327	36(3)

Experimental

Single crystals of $C_{27}H_{43}N_4P$ **[15srv176]** were **[]**. A suitable crystal was selected and **[]** on a **D8V_Mo** diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Crystal structure determination of [15srv176]

Crystal Data for $C_{27}H_{43}N_4P$ (M =454.62 g/mol): monoclinic, space group C2/c (no. 15), a = 24.8627(11) Å, b = 9.2747(4) Å, c = 25.1947(11) Å, β = 110.6439(15)°, V = 5436.7(4) Å^3, Z = 8, T = 120 K, μ(MoKα) = 0.122 mm^{-1}, *Dcalc* = 1.111 g/cm^3, 58568 reflections measured (4.728° ≤ 2Θ ≤ 60.312°), 8027 unique (R_{int} = 0.0460, R_{sigma} = 0.0316) which were used in all calculations. The final R_1 was 0.0430 (I > 2σ(I)) and wR_2 was 0.1110 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2. Others
 Uiso(H8A)=Uiso(H8B)=Uiso(H8C)=FVAR(1)
 Uiso(H9A)=Uiso(H9B)=Uiso(H9C)=FVAR(2)
 Uiso(H10A)=Uiso(H10B)=Uiso(H10C)=FVAR(3)
 Uiso(H18A)=Uiso(H18B)=Uiso(H18C)=FVAR(4)
 Uiso(H19A)=Uiso(H19B)=Uiso(H19C)=FVAR(5)
 Uiso(H21A)=Uiso(H21B)=Uiso(H21C)=FVAR(6)
 Uiso(H22A)=Uiso(H22B)=Uiso(H22C)=FVAR(7)
 Uiso(H24A)=Uiso(H24B)=Uiso(H24C)=FVAR(8)
 Uiso(H25A)=Uiso(H25B)=Uiso(H25C)=FVAR(9)
 Uiso(H27A)=Uiso(H27B)=Uiso(H27C)=FVAR(10)
 Uiso(H28A)=Uiso(H28B)=Uiso(H28C)=FVAR(11)
3.a Ternary CH refined with riding coordinates:
 C17(H17), C20(H20), C23(H23), C26(H26)
3.b Aromatic/amide H refined with riding coordinates:
 C3(H3), C5(H5), C6(H6), C12(H12), C13(H13), C14(H14), C15(H15), C16(H16)
3.c Idealised Me refined as rotating group:
 C8(H8A,H8B,H8C), C9(H9A,H9B,H9C), C10(H10A,H10B,H10C), C18(H18A,H18B,H18C),
 C19(H19A,H19B,H19C), C21(H21A,H21B,H21C), C22(H22A,H22B,H22C), C24(H24A,H24B,
 H24C), C25(H25A,H25B,H25C), C27(H27A,H27B,H27C), C28(H28A,H28B,H28C)

A9.4 – X-Ray Crystallographic Parameters for Compound 12

Parameter	
Empirical formula	$C_{30}H_{30}Cl_2N_4O_2P_2$
Formula weight	611.42
Temperature/K	120
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	8.5770(6)
b/Å	18.4771(13)
c/Å	17.9413(13)
α/°	90
β/°	100.438(2)
γ/°	90
Volume/Å^3	2796.3(3)
Z	4
ρ_{calc}g/cm^3	1.452
μ/mm^{-1}	0.384
F(000)	1272.0
Crystal size/mm^3	0.364 × 0.268 × 0.086
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.618 to 60
Index ranges	-12 ≤ h ≤ 12, -25 ≤ k ≤ 25, -25 ≤ l ≤ 25
Reflections collected	60245
Independent reflections	8128 [R_{int} = 0.0361, R_{sigma} = 0.0230]
Data/restraints/parameters	8128/6/387
Goodness-of-fit on F^2	1.071
Final R indexes [I>=2σ (I)]	R_1 = 0.0390, wR_2 = 0.1035
Final R indexes [all data]	R_1 = 0.0500, wR_2 = 0.1103
Largest diff. peak/hole / e Å^{-3}	0.55/-0.29

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for Henrietta cif. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	1516.7(4)	4002.0(2)	769.6(2)	16.18(9)
P1	3872.4(4)	4713.4(2)	848.3(2)	11.19(8)
N1	5852.2(14)	5345.2(6)	1009.5(7)	9.9(2)
N2	3271.5(15)	5264.6(7)	1506.1(7)	12.0(2)
C1	2934.5(16)	5212.2(8)	-31.1(8)	10.2(2)
C2	5872.1(17)	5736.0(8)	1648.1(8)	11.1(3)
C3	7258.4(17)	6041.0(8)	2040.4(8)	14.5(3)
C4	8634.2(18)	5956.8(9)	1740.4(9)	16.4(3)
C5	8601.2(17)	5555.0(8)	1082.7(8)	14.2(3)
C6	7186.1(16)	5240.9(8)	726.7(8)	10.3(2)
C7	4290.7(17)	5715.4(8)	1863.9(8)	11.3(3)
C8	3825.7(17)	6186.3(8)	2454.3(8)	11.6(3)
C9	4446.1(18)	6881.2(9)	2610.2(9)	15.9(3)
C10	3857.6(19)	7322.1(9)	3122.8(9)	18.4(3)
C11	2668.8(19)	7075.0(9)	3487.4(9)	18.3(3)
C12	2060.4(18)	6382.1(9)	3343.5(8)	16.5(3)
C13	2626.3(17)	5942.2(8)	2825.9(8)	13.3(3)
Cl2	8557.9(4)	5973.5(2)	4279.4(2)	16.97(9)
P2	6266.6(4)	5282.6(2)	4181.3(2)	11.65(9)
N3	4213.3(14)	4631.8(7)	3995.9(7)	10.5(2)
N4	6885.0(15)	4705.8(7)	3547.3(7)	12.7(2)
C14	7112.8(16)	4791.5(8)	5078.9(8)	10.6(2)
C15	4255.7(17)	4244.7(8)	3359.8(8)	11.4(3)
C16	2893.4(18)	3957.0(8)	2927.2(8)	14.2(3)
C17	1467.3(18)	4058.8(8)	3177.2(9)	15.8(3)
C18	1431.1(17)	4451.3(8)	3833.9(8)	13.9(3)
C19	2829.6(17)	4748.7(8)	4233.2(8)	11.0(3)
C20	5871.9(17)	4255.7(8)	3180.8(8)	11.8(3)
C22	5729.2(18)	3089.6(8)	2445.1(9)	16.1(3)
C23	6212.8(19)	2671.3(9)	1883.7(9)	17.6(3)
C24	7286.2(19)	2943.0(9)	1460.9(9)	17.3(3)
C25	7910.0(18)	3632.9(9)	1608.3(8)	16.1(3)
C26	7442.3(17)	4053.3(8)	2171.1(8)	12.9(3)
C27	6332.2(17)	3788.6(8)	2586.8(8)	12.2(3)
O1	448.5(17)	7457.0(8)	992.0(8)	24.4(3)
O2	4172.4(18)	6886.3(8)	497.7(9)	24.1(3)
C28	-475(3)	7823.2(13)	375.0(14)	28.8(4)
C29	1584(2)	6990.7(10)	758.5(10)	19.8(3)
C30	3084(2)	7396.2(10)	693.7(10)	18.8(3)

C31	5618(3)	7218.2(12)	400.6(14)	29.5(4)
O1B	5510(30)	7545(12)	1007(13)	71(6)
O2B	9180(30)	8100(15)	497(16)	76(8)
C28B	4600(40)	7167(17)	402(15)	47(7)
C29B	6610(30)	7991(15)	752(17)	53(6)
C30B	8110(30)	7586(15)	682(18)	57(7)
C31B	10620(30)	7773(18)	437(19)	62(8)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for Henrietta cif. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	16.53(17)	17.71(17)	15.14(17)	-1.38(13)	5.07(13)	-6.52(13)
P1	13.35(17)	12.76(17)	7.81(16)	-0.39(12)	2.89(13)	0.27(13)
N1	10.6(5)	11.4(5)	7.6(5)	0.3(4)	1.3(4)	-0.8(4)
N2	13.8(5)	14.1(6)	8.7(5)	-1.3(4)	4.1(4)	-1.3(4)
C1	9.9(6)	12.4(6)	8.3(6)	0.0(5)	1.8(5)	1.1(5)
C2	11.9(6)	12.9(6)	8.5(6)	-0.8(5)	1.7(5)	-0.2(5)
C3	14.2(7)	17.1(7)	11.4(6)	-3.6(5)	0.3(5)	-1.3(5)
C4	12.0(6)	21.0(8)	15.0(7)	-3.3(6)	-0.7(5)	-2.8(5)
C5	10.5(6)	19.0(7)	13.0(6)	-1.0(5)	2.1(5)	-1.1(5)
C6	9.9(6)	12.4(6)	8.5(6)	0.6(5)	1.7(5)	0.5(5)
C7	12.8(6)	13.5(6)	8.3(6)	-0.1(5)	3.4(5)	0.0(5)
C8	11.9(6)	14.9(6)	7.9(6)	-1.4(5)	1.4(5)	0.5(5)
C9	13.8(7)	17.7(7)	16.7(7)	-4.0(6)	4.0(5)	-1.9(5)
C10	16.9(7)	17.5(7)	20.0(7)	-7.5(6)	1.6(6)	-0.4(6)
C11	17.6(7)	24.7(8)	12.9(7)	-5.5(6)	3.2(6)	4.5(6)
C12	15.1(7)	23.5(8)	11.9(6)	1.0(6)	5.2(5)	3.8(6)
C13	13.4(6)	16.6(7)	10.1(6)	1.7(5)	2.4(5)	1.4(5)
Cl2	17.31(17)	18.15(17)	16.72(17)	-1.71(13)	6.42(13)	-6.57(13)
P2	13.44(17)	14.01(18)	8.00(16)	-0.02(13)	3.27(13)	0.65(13)
N3	10.2(5)	13.0(5)	8.2(5)	1.0(4)	1.1(4)	-0.3(4)
N4	14.0(6)	16.0(6)	9.0(5)	-1.1(4)	5.0(4)	-1.3(5)
C14	11.1(6)	12.1(6)	8.8(6)	0.1(5)	2.1(5)	1.4(5)
C15	12.4(6)	13.0(6)	8.9(6)	0.5(5)	2.7(5)	-0.5(5)
C16	15.7(7)	16.0(7)	10.0(6)	-2.1(5)	0.1(5)	-0.7(5)
C17	13.2(7)	18.2(7)	14.5(7)	-1.3(5)	-1.5(5)	-2.2(5)
C18	10.2(6)	17.8(7)	13.5(6)	0.6(5)	1.7(5)	-0.2(5)
C19	11.8(6)	12.9(6)	8.4(6)	1.6(5)	2.3(5)	0.5(5)
C20	13.4(6)	13.7(6)	8.7(6)	0.9(5)	3.2(5)	0.3(5)
C22	16.0(7)	16.5(7)	16.7(7)	-1.6(6)	5.7(5)	-0.8(5)
C23	17.2(7)	15.9(7)	19.2(7)	-5.0(6)	1.8(6)	0.8(6)

C24	16.8(7)	22.5(8)	12.8(7)	-5.0(6)	2.9(5)	4.6(6)
C25	14.3(7)	22.6(8)	11.9(6)	1.5(6)	3.9(5)	3.9(6)
C26	11.9(6)	16.4(7)	10.2(6)	1.0(5)	1.7(5)	2.0(5)
C27	12.2(6)	15.3(7)	9.2(6)	-0.8(5)	2.2(5)	1.1(5)
O1	24.4(7)	32.5(8)	16.1(6)	-2.9(5)	2.9(5)	5.7(6)
O2	22.3(7)	17.6(7)	34.1(8)	0.4(6)	9.5(6)	0.4(6)
C28	25.6(11)	23.6(10)	34.0(11)	-1.3(9)	-2.9(8)	5.3(8)
C29	21.1(8)	18.5(8)	18.9(8)	2.4(6)	1.4(7)	2.3(7)
C30	23.1(9)	15.2(8)	17.8(8)	-0.5(6)	2.6(7)	1.0(6)
C31	24(1)	27.9(10)	38.3(12)	6.2(9)	9.8(9)	0.4(8)

Table 4 Bond Lengths for Henrietta cif.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	P1	2.3931(5)	N3	C19	1.3488(18)
P1	N1	2.0376(12)	N4	C20	1.2930(19)
P1	N2	1.7076(13)	C14	C19[2]	1.4918(19)
P1	C1	1.8777(14)	C15	C16	1.386(2)
N1	C2	1.3518(18)	C15	C20	1.479(2)
N1	C6	1.3468(18)	C16	C17	1.390(2)
N2	C7	1.2910(19)	C17	C18	1.389(2)
C1	C6[1]	1.4908(19)	C18	C19	1.394(2)
C2	C3	1.387(2)	C19	C14[2]	1.4918(19)
C2	C7	1.4770(19)	C20	C27	1.480(2)
C3	C4	1.392(2)	C22	C23	1.391(2)
C4	C5	1.390(2)	C22	C27	1.397(2)
C5	C6	1.3918(19)	C23	C24	1.389(2)
C6	C1[1]	1.4907(19)	C24	C25	1.389(2)
C7	C8	1.4802(19)	C25	C26	1.389(2)
C8	C9	1.399(2)	C26	C27	1.400(2)
C8	C13	1.398(2)	O1	C28	1.412(3)
C9	C10	1.390(2)	O1	C29	1.419(2)
C10	C11	1.385(2)	O2	C30	1.415(2)
C11	C12	1.389(2)	O2	C31	1.422(3)
C12	C13	1.386(2)	C29	C30	1.511(3)
Cl2	P2	2.3233(5)	O1B	C28B	1.40(2)
P2	N3	2.1086(13)	O1B	C29B	1.40(2)
P2	N4	1.7117(13)	O2B	C30B	1.40(2)
P2	C14	1.8762(14)	O2B	C31B	1.40(2)
N3	C15	1.3530(18)	C29B	C30B	1.51(4)

[1]1-X,1-Y,-Z; [2]1-X,1-Y,1-Z

Table 5 Bond Angles for Henrietta cif.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N1	P1	Cl1	174.92(4)	C14	P2	Cl2	90.59(5)
N2	P1	Cl1	90.63(5)	C14	P2	N3	92.39(6)
N2	P1	N1	84.74(6)	C15	N3	P2	106.57(9)
N2	P1	C1	98.69(6)	C19	N3	P2	128.06(10)
C1	P1	Cl1	89.51(5)	C19	N3	C15	120.70(12)
C1	P1	N1	93.28(6)	C20	N4	P2	118.54(10)
C2	N1	P1	107.97(9)	C19[2]	C14	P2	112.61(10)
C6	N1	P1	127.41(10)	N3	C15	C16	121.75(13)
C6	N1	C2	120.97(12)	N3	C15	C20	110.21(12)
C7	N2	P1	117.81(10)	C16	C15	C20	127.73(13)
C6[1]	C1	P1	112.46(10)	C15	C16	C17	117.99(14)
N1	C2	C3	121.69(13)	C18	C17	C16	120.05(14)
N1	C2	C7	109.71(12)	C17	C18	C19	119.51(14)
C3	C2	C7	128.39(13)	N3	C19	C14[2]	116.95(12)
C2	C3	C4	117.86(13)	N3	C19	C18	119.95(13)
C5	C4	C3	119.99(14)	C18	C19	C14[2]	123.10(13)
C4	C5	C6	119.62(14)	N4	C20	C15	117.94(13)
N1	C6	C1[1]	117.44(12)	N4	C20	C27	119.93(13)
N1	C6	C5	119.82(13)	C15	C20	C27	122.10(13)
C5	C6	C1[1]	122.74(13)	C23	C22	C27	119.74(14)
N2	C7	C2	117.23(13)	C24	C23	C22	120.59(15)
N2	C7	C8	119.28(13)	C23	C24	C25	119.88(14)
C2	C7	C8	123.49(13)	C24	C25	C26	119.97(14)
C9	C8	C7	122.73(13)	C25	C26	C27	120.37(14)
C13	C8	C7	117.94(13)	C22	C27	C20	122.13(13)
C13	C8	C9	119.14(13)	C22	C27	C26	119.41(13)
C10	C9	C8	119.98(14)	C26	C27	C20	118.43(13)
C11	C10	C9	120.39(15)	C28	O1	C29	112.15(16)
C10	C11	C12	120.01(14)	C30	O2	C31	111.87(16)
C13	C12	C11	119.97(14)	O1	C29	C30	111.17(15)
C12	C13	C8	120.50(14)	O2	C30	C29	107.35(15)
N3	P2	Cl2	175.01(4)	C29B	O1B	C28B	111(2)
N4	P2	Cl2	91.88(5)	C31B	O2B	C30B	111(3)
N4	P2	N3	83.72(6)	O1B	C29B	C30B	112(2)
N4	P2	C14	98.69(6)	O2B	C30B	C29B	107(3)

[1]1-X,1-Y,-Z; [2]1-X,1-Y,1-Z

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for Henrietta cif.

Atom	*x*	*y*	*z*	U(eq)
H1A	1860	5372	24	12
H1B	3570	5649	-88	12
H3	7268	6299	2499	17
H4	9596	6174	1985	20
H5	9539	5495	877	17
H9	5269	7052	2366	19
H10	4273	7796	3224	22
H11	2269	7379	3836	22
H12	1257	6210	3600	20
H13	2196	5471	2723	16
H14A	6460	4357	5125	13
H14B	8199	4627	5051	13
H16	2933	3698	2474	17
H17	517	3860	2899	19
H18	460	4517	4010	17
H22	4992	2901	2731	19
H23	5805	2195	1789	21
H24	7593	2658	1071	21
H25	8656	3817	1325	19
H26	7878	4523	2274	15
H281	-1268	8124	558	43
H282	-1011	7469	9	43
H283	212	8131	129	43
H291	1130	6774	262	24
H292	1840	6593	1130	24
H301	2858	7775	299	23
H302	3529	7631	1182	23
H311	6155	7418	886	44
H312	5393	7608	27	44
H313	6302	6857	223	44
H284	3835	6857	592	71
H285	4041	7512	34	71
H286	5306	6867	156	71
H293	6883	8399	1110	64
H294	6139	8195	252	64
H303	8552	7345	1166	68
H304	7881	7214	280	68
H314	11352	8141	307	93
H315	11079	7548	922	93
H316	10443	7403	40	93

Table 7 Atomic Occupancy for Henrietta cif.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
O1	0.9	O2	0.9	C28	0.9
H281	0.9	H282	0.9	H283	0.9
C29	0.9	H291	0.9	H292	0.9
C30	0.9	H301	0.9	H302	0.9
C31	0.9	H311	0.9	H312	0.9
H313	0.9	O1B	0.1	O2B	0.1
C28B	0.1	H284	0.1	H285	0.1
H286	0.1	C29B	0.1	H293	0.1
H294	0.1	C30B	0.1	H303	0.1
H304	0.1	C31B	0.1	H314	0.1
H315	0.1	H316	0.1		

Experimental

Single crystals of $C_{30}H_{30}Cl_2N_4O_2P_2$ **[Henrietta cif]** were **[]**. A suitable crystal was selected and **[]** on a diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the SIR2004 [2] structure solution program using Direct Methods and refined with the XH [3] refinement package using CGLS minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2.
3.

Crystal structure determination of [Henrietta cif]

Crystal Data for $C_{30}H_{30}Cl_2N_4O_2P_2$ (M =611.42 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 8.5770(6) Å, b = 18.4771(13) Å, c = 17.9413(13) Å, β = 100.438(2)°, V = 2796.3(3) Å^3, Z = 4, T = 120 K, μ(MoKα) = 0.384 mm^{-1}, *Dcalc* = 1.452 g/cm^3, 60245 reflections measured (4.618° ≤ 2Θ ≤ 60°), 8128 unique (R_{int} = 0.0361, R_{sigma} = 0.0230) which were used in all calculations. The final R_1 was 0.0390 (I > 2σ(I)) and wR_2 was 0.1103 (all data).

Refinement model description

Number of restraints - 6, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2. Restrained distances
 O2B-C31B ≈ O1B-C28B ≈ O1B-C29B ≈ O2B-C30B
 with sigma of 0.02
3. Others
 Fixed Sof: O1(0.9) O2(0.9) C28(0.9) H281(0.9) H282(0.9) H283(0.9) C29(0.9)
 H291(0.9) H292(0.9) C30(0.9) H301(0.9) H302(0.9) C31(0.9) H311(0.9) H312(0.9)
 H313(0.9) O1B(0.1) O2B(0.1) C28B(0.1) H284(0.1) H285(0.1) H286(0.1) C29B(0.1)
 H293(0.1) H294(0.1) C30B(0.1) H303(0.1) H304(0.1) C31B(0.1) H314(0.1)
 H315(0.1) H316(0.1)
4.a Riding coordinates:
 C28B(H284,H285,H286), C29B(H293,H294), C30B(H303,H304), C31B(H314,H315,H316)
4.b Rotating group:

C28(H281,H282,H283), C31(H311,H312,H313)
4.c Secondary CH2 refined with riding coordinates:
C1(H1A,H1B), C14(H14A,H14B), C29(H291,H292), C30(H301,H302)
4.d Aromatic/amide H refined with riding coordinates:
C3(H3), C4(H4), C5(H5), C9(H9), C10(H10), C11(H11), C12(H12), C13(H13),
C16(H16), C17(H17), C18(H18), C22(H22), C23(H23), C24(H24), C25(H25), C26(H26)

A9.5 – X-Ray Crystallographic Parameters for Compound 21

Parameter	
Empirical formula	$C_{36}H_{35}BClF_{15}N_2$
Formula weight	826.92
Temperature/K	120.0
Crystal system	triclinic
Space group	P-1
a/Å	11.8076(11)
b/Å	11.9474(11)
c/Å	13.6967(13)
α/°	93.264(4)
β/°	99.151(4)
γ/°	99.959(4)
Volume/Å^3	1871.8(3)
Z	2
ρ_{calc}g/cm^3	1.467
μ/mm^{-1}	0.206
F(000)	844.0
Crystal size/mm^3	0.4 × 0.36 × 0.15
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.404 to 57.998
Index ranges	-16 ≤ h ≤ 16, -16 ≤ k ≤ 16, -18 ≤ l ≤ 18
Reflections collected	39004
Independent reflections	9954 [R_{int} = 0.0510, R_{sigma} = 0.0524]
Data/restraints/parameters	9954/0/609
Goodness-of-fit on F^2	1.007
Final R indexes [I>=2σ (I)]	R_1 = 0.0486, wR_2 = 0.1020
Final R indexes [all data]	R_1 = 0.0816, wR_2 = 0.1152
Largest diff. peak/hole / e Å^{-3}	0.41/-0.42

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 16srv383. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl(1)	1441.8(4)	5119.9(4)	6487.6(3)	23.99(11)
F(1)	4987.7(9)	4006.7(9)	8787.5(7)	26.7(2)
F(2)	5852.5(11)	6199.6(10)	8733.1(9)	41.3(3)
F(3)	5504.9(11)	7234.5(10)	7027.4(10)	43.4(3)
F(4)	4280.5(10)	5989.7(9)	5346.3(8)	29.6(3)
F(5)	3496.0(9)	3788.1(8)	5342.7(7)	23.0(2)
F(6)	6347.6(9)	2912.4(9)	7976.1(8)	29.2(3)
F(7)	7483.1(10)	1588.8(11)	9057.6(9)	39.0(3)
F(8)	6391.5(12)	-483.8(11)	9471.3(9)	45.5(3)
F(9)	4065.5(12)	-1188(1)	8753.3(9)	42.4(3)
F(10)	2879.4(10)	130.2(9)	7682.1(8)	31.6(3)
F(11)	5562.4(9)	2185.1(10)	5996.4(8)	28.8(2)
F(12)	5450.5(11)	1134.6(11)	4209.0(9)	38.1(3)
F(13)	3360.9(13)	24.4(11)	3161.4(9)	44.0(3)
F(14)	1386.5(11)	6.3(10)	3966.6(9)	39.1(3)
F(15)	1469.6(9)	1013.9(9)	5734.7(8)	26.2(2)
N(1)	2376.6(14)	4081.3(13)	8462.8(11)	23.3(3)
C(1)	2399.4(15)	2430.0(15)	7419.5(13)	18.0(3)
C(2)	2483.3(15)	3021.9(15)	8397.4(13)	21.3(4)
C(3)	2712(2)	2409.2(19)	9305.2(15)	31.0(5)
C(4)	2474(2)	4840.3(18)	9376.5(14)	34.0(5)
C(5)	2965(2)	6046.4(19)	9190.0(19)	40.8(5)
C(6)	1281(3)	4747(3)	9675(2)	56.8(7)
C(7)	4285.2(14)	3731.5(14)	7048.1(12)	17.5(3)
C(8)	4856.2(15)	4434.5(15)	7884.3(13)	21.5(4)
C(9)	5287.5(17)	5581.5(16)	7884.9(14)	27.3(4)
C(10)	5114.4(16)	6111.5(15)	7025.3(15)	27.6(4)
C(11)	4506.0(15)	5478.0(15)	6184.5(13)	22.9(4)
C(12)	4110.1(14)	4325.2(14)	6206.2(12)	18.0(3)
C(13)	4509.8(15)	1638.6(14)	7804.6(12)	19.5(4)
C(14)	5700.4(16)	1924.6(15)	8170.1(13)	22.6(4)
C(15)	6332.7(17)	1238.2(17)	8729.8(13)	27.2(4)
C(16)	5786.5(19)	193.1(17)	8934.8(14)	30.2(5)
C(17)	4618.9(19)	-160.7(16)	8570.5(14)	28.8(4)
C(18)	4022.4(17)	556.9(15)	8020.5(13)	23.8(4)
C(19)	3528.2(15)	1682.2(14)	5969.7(12)	18.4(3)
C(20)	4499.7(16)	1650.5(15)	5517.3(13)	22.5(4)
C(21)	4469.2(18)	1117.1(16)	4594.5(14)	26.9(4)

C(22)	3421.6(19)	558.8(16)	4061.3(14)	29.8(4)
C(23)	2430.8(18)	549.4(15)	4468.7(14)	26.7(4)
C(24)	2500.2(16)	1094.3(14)	5402.6(13)	21.0(4)
B(1)	3708.4(17)	2378.1(16)	7078.9(14)	16.8(4)
N(2)	9153.4(14)	3455.5(13)	5383.1(12)	22.6(3)
C(25)	8333.1(17)	3276.5(17)	6121.2(15)	28.7(4)
C(26)	8026(2)	4419.0(19)	6403.9(18)	34.0(5)
C(27)	8905(2)	2768(2)	7021.3(18)	39.2(5)
C(28)	9422.8(18)	2408.4(16)	4866.4(15)	27.7(4)
C(29)	10459(2)	2773.4(19)	4358.8(17)	33.4(5)
C(30)	8360(2)	1813(2)	4137.9(19)	39.1(5)
C(31)	10693(6)	8346(6)	8531(5)	49.9(16)
C(31A)	10582(7)	8093(6)	8774(5)	60.0(19)
C(32)	9842(3)	7284(3)	8307(2)	59.6(8)
C(33)	8675(2)	7336(2)	8127(2)	56.4(7)
C(34)	8312(2)	8330(2)	8270.0(19)	48.1(6)
C(35)	9061(5)	9304(5)	8333(4)	47.0(12)
C(35A)	9137(5)	9244(5)	8881(5)	54.2(14)
C(36)	10255(5)	9296(5)	8494(5)	60.7(15)
C(36A)	10295(6)	9130(5)	9103(5)	63.7(15)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16srv383. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl(1)	21.9(2)	24.8(2)	23.2(2)	7.42(16)	-3.92(17)	3.79(17)
F(1)	33.9(6)	26.1(6)	18.8(5)	6.8(4)	-3.3(4)	7.4(5)
F(2)	50.4(8)	27.6(6)	33.2(7)	1.0(5)	-17.4(6)	-4.9(5)
F(3)	52.0(8)	20.5(6)	46.5(8)	10.9(5)	-10.5(6)	-9.3(5)
F(4)	36.0(6)	24.4(6)	27.4(6)	15.0(4)	0.7(5)	3.5(5)
F(5)	27.9(6)	21.3(5)	18.3(5)	4.9(4)	-2.0(4)	4.4(4)
F(6)	20.8(5)	34.2(6)	33.7(6)	12.6(5)	2.1(5)	7.1(5)
F(7)	29.9(6)	58.4(8)	34.8(7)	12.6(6)	1.6(5)	25.6(6)
F(8)	62.3(9)	53.5(8)	37.4(7)	26.6(6)	14.6(6)	43.6(7)
F(9)	65.9(9)	23.6(6)	44.8(8)	17.9(5)	17.0(6)	15.8(6)
F(10)	36.0(6)	21.0(6)	35.8(6)	7.2(5)	2.3(5)	1.2(5)
F(11)	21.8(5)	38.3(6)	29.5(6)	10.7(5)	7.3(5)	8.7(5)
F(12)	47.7(7)	46.3(7)	34.2(7)	15.3(5)	24.0(6)	27.9(6)
F(13)	73.7(9)	39.8(7)	23.4(6)	-3.8(5)	13.6(6)	21.3(7)
F(14)	49.7(8)	31.9(6)	28.0(6)	-5.7(5)	-2.3(5)	-2.8(5)
F(15)	23.8(5)	28.5(6)	24.6(6)	1.6(4)	3.5(4)	0.9(4)
N(1)	29.2(8)	26.2(8)	16.6(8)	3.9(6)	1.9(6)	12.5(7)

C(1)	20.1(8)	16.9(9)	17.8(9)	5.0(6)	4.5(7)	3.1(7)
C(2)	18.9(8)	24.4(9)	22.3(9)	5.8(7)	4.7(7)	6.6(7)
C(3)	47.0(13)	27.9(11)	22.1(10)	8.5(8)	8.3(9)	13.9(10)
C(4)	52.6(14)	35.4(11)	18.1(10)	-0.4(8)	2.9(9)	23.5(10)
C(5)	51.4(15)	33.0(12)	35.9(13)	-9.6(10)	-0.6(11)	13.6(11)
C(6)	81(2)	50.9(17)	53.4(18)	5.2(13)	41.0(16)	26.0(15)
C(7)	14.6(8)	18.8(8)	20.3(9)	6.0(6)	2.2(6)	5.3(6)
C(8)	20.8(9)	23.6(9)	19.9(9)	8.6(7)	-1.1(7)	5.8(7)
C(9)	25.6(10)	24.3(10)	25.7(10)	0.4(7)	-7.9(8)	-0.4(7)
C(10)	25.5(10)	18.1(9)	35.3(11)	8.5(8)	-1.9(8)	-1.9(7)
C(11)	21.3(9)	23.1(9)	25.1(9)	11.9(7)	1.4(7)	5.6(7)
C(12)	15.3(8)	19.6(8)	18.5(8)	3.4(6)	0.8(6)	3.0(6)
C(13)	26.1(9)	20.3(8)	14.8(8)	5.1(6)	4.6(7)	9.9(7)
C(14)	26.9(9)	26.5(9)	18.1(9)	7.3(7)	5.8(7)	11.9(8)
C(15)	28.9(10)	40.4(11)	18.3(9)	6.0(8)	5.0(8)	20.9(9)
C(16)	45.5(12)	36.6(11)	20.1(9)	13.1(8)	11.8(8)	30.3(10)
C(17)	47.9(12)	20.6(9)	24.9(10)	9.6(7)	15.6(9)	15.0(9)
C(18)	32.4(10)	22.6(9)	18.4(9)	3.2(7)	5.5(8)	9.0(8)
C(19)	24.4(9)	14.7(8)	18.2(8)	7.8(6)	4.0(7)	7.2(7)
C(20)	27.2(10)	19.8(9)	23.1(9)	9.4(7)	4.7(7)	8.8(7)
C(21)	37.6(11)	26.3(10)	26.3(10)	14.0(8)	15.4(8)	19.0(8)
C(22)	51.3(13)	22.3(9)	20.8(10)	5.4(7)	10.4(9)	15.4(9)
C(23)	39.1(11)	17.7(9)	21.0(9)	3.0(7)	0.4(8)	3.4(8)
C(24)	27.2(9)	16.4(8)	21.4(9)	6.9(7)	6.3(7)	6.1(7)
B(1)	19.0(9)	16.5(9)	15.9(9)	5.1(7)	3.0(7)	4.6(7)
N(2)	19.9(8)	21.2(8)	24.1(8)	6.6(6)	-1.8(6)	-0.1(6)
C(25)	21.7(9)	27.8(10)	35.1(11)	7.3(8)	5.6(8)	-1.8(8)
C(26)	27.2(11)	35.2(12)	42.6(13)	10.3(10)	10.5(10)	7.1(9)
C(27)	50.9(15)	37.3(13)	37.3(13)	17.7(10)	19.9(11)	14.4(11)
C(28)	33.5(11)	19.9(9)	28.8(10)	7.1(8)	2.1(8)	3.5(8)
C(29)	40.0(12)	30.1(11)	33.3(12)	8.1(9)	9.8(10)	10.1(10)
C(30)	45.1(14)	27.4(12)	38.2(13)	1.4(10)	-2.6(11)	-2.4(10)
C(32)	73(2)	63.9(18)	51.4(16)	5.8(14)	8.9(14)	38.6(16)
C(33)	55.3(17)	56.3(17)	56.6(17)	-7.1(13)	13.3(13)	8.1(13)
C(34)	38.7(13)	61.1(16)	49.9(15)	8.5(12)	14.3(11)	16.3(12)

Table 4 Bond Lengths for 16srv383.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
F(1)	C(8)	1.3617(19)	C(13)	C(18)	1.389(3)
F(2)	C(9)	1.351(2)	C(13)	B(1)	1.659(2)
F(3)	C(10)	1.341(2)	C(14)	C(15)	1.385(2)

F(4)	C(11)	1.3422(19)	C(15)	C(16)	1.369(3)
F(5)	C(12)	1.3477(19)	C(16)	C(17)	1.373(3)
F(6)	C(14)	1.355(2)	C(17)	C(18)	1.383(3)
F(7)	C(15)	1.345(2)	C(19)	C(20)	1.393(3)
F(8)	C(16)	1.343(2)	C(19)	C(24)	1.383(2)
F(9)	C(17)	1.344(2)	C(19)	B(1)	1.654(3)
F(10)	C(18)	1.354(2)	C(20)	C(21)	1.375(3)
F(11)	C(20)	1.351(2)	C(21)	C(22)	1.371(3)
F(12)	C(21)	1.345(2)	C(22)	C(23)	1.372(3)
F(13)	C(22)	1.341(2)	C(23)	C(24)	1.386(3)
F(14)	C(23)	1.348(2)	N(2)	C(25)	1.507(3)
F(15)	C(24)	1.356(2)	N(2)	C(28)	1.508(2)
N(1)	C(2)	1.293(2)	C(25)	C(26)	1.517(3)
N(1)	C(4)	1.480(2)	C(25)	C(27)	1.519(3)
C(1)	C(2)	1.459(2)	C(28)	C(29)	1.513(3)
C(1)	B(1)	1.694(3)	C(28)	C(30)	1.513(3)
C(2)	C(3)	1.490(3)	C(31)	C(32)	1.459(7)
C(4)	C(5)	1.509(3)	C(31)	C(36)	1.327(8)
C(4)	C(6)	1.516(4)	C(31A)	C(32)	1.252(8)
C(7)	C(8)	1.386(2)	C(31A)	C(36A)	1.408(9)
C(7)	C(12)	1.394(2)	C(32)	C(33)	1.373(4)
C(7)	B(1)	1.648(2)	C(33)	C(34)	1.345(4)
C(8)	C(9)	1.377(3)	C(34)	C(35)	1.323(6)
C(9)	C(10)	1.372(3)	C(34)	C(35A)	1.452(6)
C(10)	C(11)	1.366(3)	C(35)	C(36)	1.394(8)
C(11)	C(12)	1.379(2)	C(35A)	C(36A)	1.385(8)
C(13)	C(14)	1.391(3)			

Table 5 Bond Angles for 16srv383.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C(2)	N(1)	C(4)	127.64(16)	C(17)	C(18)	C(13)	125.06(18)
C(2)	C(1)	B(1)	113.91(14)	C(20)	C(19)	B(1)	118.97(15)
N(1)	C(2)	C(1)	119.24(16)	C(24)	C(19)	C(20)	113.15(16)
N(1)	C(2)	C(3)	120.89(17)	C(24)	C(19)	B(1)	127.87(15)
C(1)	C(2)	C(3)	119.86(16)	F(11)	C(20)	C(19)	119.19(16)
N(1)	C(4)	C(5)	109.18(18)	F(11)	C(20)	C(21)	115.96(16)
N(1)	C(4)	C(6)	109.0(2)	C(21)	C(20)	C(19)	124.84(18)
C(5)	C(4)	C(6)	111.8(2)	F(12)	C(21)	C(20)	120.99(18)
C(8)	C(7)	C(12)	112.74(15)	F(12)	C(21)	C(22)	119.64(17)
C(8)	C(7)	B(1)	123.44(15)	C(22)	C(21)	C(20)	119.37(18)
C(12)	C(7)	B(1)	123.27(15)	F(13)	C(22)	C(21)	120.94(19)

F(1)	C(8)	C(7)	119.96(15)	F(13)	C(22)	C(23)	120.33(19)
F(1)	C(8)	C(9)	115.56(16)	C(21)	C(22)	C(23)	118.72(17)
C(9)	C(8)	C(7)	124.45(16)	F(14)	C(23)	C(22)	120.32(17)
F(2)	C(9)	C(8)	120.52(17)	F(14)	C(23)	C(24)	119.60(18)
F(2)	C(9)	C(10)	119.51(17)	C(22)	C(23)	C(24)	120.08(18)
C(10)	C(9)	C(8)	119.94(17)	F(15)	C(24)	C(19)	121.32(15)
F(3)	C(10)	C(9)	120.20(17)	F(15)	C(24)	C(23)	114.86(16)
F(3)	C(10)	C(11)	121.26(17)	C(19)	C(24)	C(23)	123.82(17)
C(11)	C(10)	C(9)	118.51(17)	C(7)	B(1)	C(1)	103.48(13)
F(4)	C(11)	C(10)	119.49(16)	C(7)	B(1)	C(13)	115.60(14)
F(4)	C(11)	C(12)	120.54(16)	C(7)	B(1)	C(19)	110.77(14)
C(10)	C(11)	C(12)	119.95(16)	C(13)	B(1)	C(1)	113.16(14)
F(5)	C(12)	C(7)	120.67(15)	C(19)	B(1)	C(1)	110.51(14)
F(5)	C(12)	C(11)	115.04(14)	C(19)	B(1)	C(13)	103.49(13)
C(11)	C(12)	C(7)	124.27(16)	C(25)	N(2)	C(28)	117.54(15)
C(14)	C(13)	B(1)	127.13(15)	N(2)	C(25)	C(26)	108.11(16)
C(18)	C(13)	C(14)	112.64(15)	N(2)	C(25)	C(27)	109.82(17)
C(18)	C(13)	B(1)	119.91(15)	C(26)	C(25)	C(27)	111.60(19)
F(6)	C(14)	C(13)	121.43(15)	N(2)	C(28)	C(29)	108.31(16)
F(6)	C(14)	C(15)	114.24(16)	N(2)	C(28)	C(30)	109.85(17)
C(15)	C(14)	C(13)	124.31(18)	C(30)	C(28)	C(29)	111.91(19)
F(7)	C(15)	C(14)	120.19(18)	C(36)	C(31)	C(32)	115.7(5)
F(7)	C(15)	C(16)	120.06(16)	C(32)	C(31A)	C(36A)	123.0(7)
C(16)	C(15)	C(14)	119.74(18)	C(31A)	C(32)	C(33)	121.4(4)
F(8)	C(16)	C(15)	120.32(19)	C(33)	C(32)	C(31)	118.8(3)
F(8)	C(16)	C(17)	120.57(19)	C(34)	C(33)	C(32)	120.8(3)
C(15)	C(16)	C(17)	119.10(16)	C(33)	C(34)	C(35A)	116.9(3)
F(9)	C(17)	C(16)	120.22(17)	C(35)	C(34)	C(33)	119.8(3)
F(9)	C(17)	C(18)	120.69(19)	C(34)	C(35)	C(36)	119.5(5)
C(16)	C(17)	C(18)	119.08(18)	C(36A)	C(35A)	C(34)	118.5(5)
F(10)	C(18)	C(13)	120.05(15)	C(31)	C(36)	C(35)	123.2(6)
F(10)	C(18)	C(17)	114.89(17)	C(35A)	C(36A)	C(31A)	117.3(6)

Table 6 Hydrogen Bonds for 16srv383.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N(1)	H(1)	Cl(1)	0.93(2)	2.24(2)	3.1597(16)	171(2)
N(2)	H(2A)	Cl(1)[1]	0.92(2)	2.26(2)	3.1690(16)	174.4(18)
N(2)	H(2B)	Cl(1)[2]	0.92(2)	2.31(2)	3.2085(16)	166.6(17)

[1]1+X,+Y,+Z; [2]1-X,1-Y,1-Z

Table 7 Torsion Angles for 16srv383.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
C(1)	C(2)	N(1)	C(4)	-177.69(18)	C(13)	B(1)	C(7)	C(8)	-42.4(2)
C(1)	B(1)	C(7)	C(8)	81.90(18)	C(13)	B(1)	C(7)	C(12)	146.80(16)
C(1)	B(1)	C(7)	C(12)	-88.91(18)	C(13)	B(1)	C(19)	C(20)	-65.08(18)
C(1)	B(1)	C(13)	C(14)	-137.57(18)	C(13)	B(1)	C(19)	C(24)	114.56(18)
C(1)	B(1)	C(13)	C(18)	49.5(2)	C(19)	B(1)	C(1)	C(2)	179.42(14)
C(1)	B(1)	C(19)	C(20)	173.48(14)	C(19)	B(1)	C(7)	C(8)	-159.66(15)
C(1)	B(1)	C(19)	C(24)	-6.9(2)	C(19)	B(1)	C(7)	C(12)	29.5(2)
C(3)	C(2)	N(1)	C(4)	1.2(3)	C(19)	B(1)	C(13)	C(14)	102.80(19)
C(5)	C(4)	N(1)	C(2)	148.1(2)	C(19)	B(1)	C(13)	C(18)	-70.15(19)
C(6)	C(4)	N(1)	C(2)	-89.5(2)	B(1)	C(1)	C(2)	N(1)	97.18(19)
C(7)	B(1)	C(1)	C(2)	-61.97(17)	B(1)	C(1)	C(2)	C(3)	-81.7(2)
C(7)	B(1)	C(13)	C(14)	-18.5(2)	C(26)	C(25)	N(2)	C(28)	-170.11(16)
C(7)	B(1)	C(13)	C(18)	168.58(15)	C(27)	C(25)	N(2)	C(28)	67.9(2)
C(7)	B(1)	C(19)	C(20)	59.40(19)	C(29)	C(28)	N(2)	C(25)	-166.78(16)
C(7)	B(1)	C(19)	C(24)	-120.96(17)	C(30)	C(28)	N(2)	C(25)	70.7(2)
C(13)	B(1)	C(1)	C(2)	63.89(19)					

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16srv383.

Atom	*x*	*y*	*z*	U(eq)
H(1)	2190(20)	4410(20)	7874(18)	40(6)
H(1A)	2002(17)	1716(18)	7431(14)	21(5)
H(1B)	2006(16)	2810(16)	6920(14)	18(5)
H(3A)	2460(20)	1630(20)	9146(18)	48(7)
H(3B)	2320(20)	2640(20)	9830(20)	60(8)
H(3C)	3530(30)	2530(30)	9570(20)	74(9)
H(4)	3020(20)	4580(20)	9881(18)	47(7)
H(5A)	2490(20)	6290(20)	8613(19)	44(7)
H(5B)	2960(20)	6530(20)	9780(20)	63(8)
H(5C)	3780(30)	6110(20)	9060(20)	73(9)
H(6A)	970(30)	3910(30)	9780(20)	69(9)
H(6B)	730(20)	4970(20)	9090(20)	61(8)
H(6C)	1360(30)	5290(30)	10300(20)	83(10)
H(2A)	9836(19)	3892(17)	5713(15)	26(5)
H(2B)	8887(17)	3899(17)	4900(15)	23(5)
H(25)	7643(19)	2749(18)	5769(15)	29(5)
H(26A)	7704(19)	4791(19)	5823(17)	36(6)
H(26B)	8710(20)	4935(19)	6771(16)	33(6)
H(26C)	7450(20)	4310(20)	6818(18)	48(7)

H(27A)	9090(20)	2030(20)	6855(19)	54(7)
H(27B)	8390(20)	2700(20)	7470(20)	53(7)
H(27C)	9670(30)	3340(20)	7350(20)	66(8)
H(28)	9629(17)	1925(17)	5415(15)	24(5)
H(29A)	11160(20)	3180(20)	4839(18)	45(7)
H(29B)	10270(20)	3300(20)	3857(18)	47(7)
H(29C)	10640(20)	2080(20)	4025(17)	44(7)
H(30A)	8111(19)	2309(19)	3649(17)	34(6)
H(30B)	7690(20)	1571(19)	4459(17)	35(6)
H(30C)	8530(20)	1170(20)	3790(20)	57(8)
H(31)	11507	8356	8694	60
H(31A)	11374	8003	8913	72
H(32)	10092	6620	8073	72
H(32A)	10090	6569	8287	72
H(33)	8117	6662	7897	68
H(34)	7553	8430	7985	58
H(34A)	7521	8334	8326	58
H(35)	8786	10003	8270	56
H(35A)	8887	9905	9123	65
H(36)	10782	10006	8580	73
H(36A)	10873	9727	9463	76

Table 9 Atomic Occupancy for 16srv383.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
C(31)	0.5	H(31)	0.5	C(31A)	0.5
H(31A)	0.5	H(32)	0.5	H(32A)	0.5
H(34)	0.5	H(34A)	0.5	C(35)	0.5
H(35)	0.5	C(35A)	0.5	H(35A)	0.5
C(36)	0.5	H(36)	0.5	C(36A)	0.5
H(36A)	0.5				

Experimental

Single crystals of $C_{36}H_{35}BClF_{15}N_2$ **[16srv383]** were **[]**. A suitable crystal was selected and **[]** on a **Bruker D8 Venture** diffractometer. The crystal was kept at 120.0 K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2.
3.

Crystal structure determination of [16srv383]

Crystal Data for $C_{36}H_{35}BClF_{15}N_2$ (M =826.92 g/mol): triclinic, space group P-1 (no. 2), a = 11.8076(11) Å, b = 11.9474(11) Å, c = 13.6967(13) Å, α = 93.264(4)°, β = 99.151(4)°, γ = 99.959(4)°, V = 1871.8(3) Å^3, Z = 2, T =

120.0 K, μ(MoKα) = 0.206 mm^{-1}, *Dcalc* = 1.467 g/cm^3, 39004 reflections measured (4.404° ≤ 2Θ ≤ 57.998°), 9954 unique (R_{int} = 0.0510, R_{sigma} = 0.0524) which were used in all calculations. The final R_1 was 0.0486 (I > 2σ(I)) and wR_2 was 0.1152 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
 All C(H) groups, All C(H,H) groups
2. Others
 Fixed Sof: C31(0.5) H31(0.5) C31A(0.5) H31A(0.5) H32(0.5) H32A(0.5) H34(0.5)
 H34A(0.5) C35(0.5) H35(0.5) C35A(0.5) H35A(0.5) C36(0.5) H36(0.5) C36A(0.5)
 H36A(0.5)
3.a Aromatic/amide H refined with riding coordinates:
 C31(H31), C31A(H31A), C32(H32), C32(H32A), C33(H33), C34(H34), C34(H34A),
 C35(H35), C35A(H35A), C36(H36), C36A(H36A)

A9.6 – X-Ray Crystallographic Parameters for 4-Dimethylamino-2-Pyridinecarboxamide

Parameter	
Empirical formula	$C_8H_{11}N_3O$
Formula weight	165.20
Temperature/K	120
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	7.6315(5)
b/Å	16.9683(10)
c/Å	26.1099(16)
α/°	90
β/°	91.747(2)
γ/°	90
Volume/Å^3	3379.5(4)
Z	16
ρ_{calc}g/cm^3	1.299
μ/mm^{-1}	0.090
F(000)	1408.0
Crystal size/mm^3	0.159 × 0.141 × 0.058
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.938 to 50.154
Index ranges	-9 ≤ h ≤ 9, -20 ≤ k ≤ 20, -31 ≤ l ≤ 31
Reflections collected	42270
Independent reflections	5976 [R_{int} = 0.0764, R_{sigma} = 0.0588]
Data/restraints/parameters	5976/0/449
Goodness-of-fit on F^2	1.076
Final R indexes [I>=2σ (I)]	R_1 = 0.0790, wR_2 = 0.1854
Final R indexes [all data]	R_1 = 0.1414, wR_2 = 0.2137
Largest diff. peak/hole / e Å^{-3}	0.59/-0.38

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16srv014. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1A	2674(3)	795.7(15)	2001.2(10)	21.7(7)
N2A	4772(4)	1725(2)	2050.8(14)	26.3(9)
N1A	7309(4)	579.5(19)	2000.8(13)	19.1(8)
N3A	5827(5)	-1775.3(19)	1747.0(13)	24.4(9)
C7A	4003(5)	-1970(2)	1623.3(17)	26.7(10)
C2A	5612(5)	361(2)	1952.2(15)	18.6(9)
C1A	4228(5)	982(2)	2003.2(15)	17.1(9)
C4A	6307(5)	-1010(2)	1825.8(15)	18.0(9)
C5A	8081(5)	-782(2)	1884.0(15)	19.7(9)
C8A	7158(6)	-2387(2)	1710.9(17)	27.9(11)
C3A	5063(5)	-405(2)	1867.4(15)	18.8(9)
C6A	8488(5)	1(2)	1963.1(15)	18.8(9)
O1C	7349(3)	6474.0(15)	533.4(11)	21.1(7)
N2C	5248(4)	7381.7(18)	392.9(13)	20.3(8)
N1C	2718(4)	6238.6(18)	425.5(12)	17.0(8)
N3C	4154(4)	3889.6(18)	718.2(13)	20.8(8)
C3C	4938(5)	5255(2)	605.1(14)	14.5(9)
C2C	4398(5)	6019(2)	501.4(14)	13.1(9)
C7C	5951(5)	3699(2)	888.3(17)	26.1(10)
C8C	2843(6)	3277(2)	738.2(17)	26.8(11)
C1C	5780(5)	6646(2)	478.4(15)	15.1(9)
C5C	1932(5)	4869(2)	539.3(15)	17.9(9)
C4C	3694(5)	4648(2)	626.3(15)	15.4(9)
C6C	1529(5)	5650(2)	446.6(15)	19.7(9)
O1B	7655(3)	2833.8(15)	1992.4(11)	21.6(7)
N3B	10832(4)	5419.5(19)	1800.2(13)	21.0(8)
N1B	12304(4)	3074.6(18)	2082.2(12)	17.6(8)
N2B	9776(4)	1921.7(18)	2101.1(13)	22.2(8)
C6B	13477(5)	3663(2)	2068.7(15)	19.1(9)
C7B	9016(6)	5615(2)	1648.1(18)	30.8(11)
C5B	13080(5)	4442(2)	1981.6(15)	18.8(9)
C4B	11323(5)	4676(2)	1898.3(15)	16.6(9)
C2B	10617(5)	3290(2)	2012.4(15)	15.5(9)
C8B	12142(6)	6045(2)	1767.4(17)	26.3(11)
C3B	10077(5)	4059(2)	1918.2(15)	16.8(9)
C1B	9230(5)	2664(2)	2033.5(15)	17.9(9)
O1D	12338(3)	8507.4(15)	458.6(11)	20.9(7)
N1D	7705(4)	8726.5(18)	492.0(12)	17.2(8)

N2D	10225(4)	7586.0(18)	442.1(14)	25.2(9)
N3D	9176(5)	11081.9(19)	743.2(14)	24.6(8)
C3D	9960(5)	9717(2)	610.5(15)	15.7(9)
C1D	10769(5)	8332(2)	475.1(15)	17.6(9)
C2D	9391(5)	8952(2)	530.0(14)	14.5(9)
C8D	7875(6)	11694(2)	794.5(18)	28.9(11)
C6D	6528(5)	9311(2)	528.3(15)	21.8(10)
C7D	11030(6)	11284(2)	841.9(17)	28.6(11)
C4D	8703(5)	10316(2)	654.0(15)	18.6(9)
C5D	6941(5)	10090(2)	600.8(15)	20.1(10)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16srv014. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1A	11.5(16)	19.2(15)	34.5(18)	0.7(13)	1.5(12)	2.8(12)
N2A	6.2(18)	18.9(19)	54(3)	-2.9(17)	4.7(16)	1.2(15)
N1A	8.4(19)	22.0(18)	27(2)	1.6(15)	1.1(14)	-1.4(15)
N3A	29(2)	9.8(17)	35(2)	-0.8(15)	1.5(16)	-0.4(15)
C7A	28(3)	17(2)	35(3)	-4.2(19)	0(2)	-4.4(19)
C2A	16(2)	22(2)	17(2)	0.3(17)	3.1(17)	0.6(19)
C1A	14(2)	13(2)	24(2)	-1.9(17)	1.7(17)	-1.6(17)
C4A	21(2)	14(2)	19(2)	0.4(17)	1.7(17)	3.6(18)
C5A	18(2)	16(2)	26(2)	3.7(18)	2.2(17)	1.4(18)
C8A	38(3)	11(2)	34(3)	0.8(19)	4(2)	5(2)
C3A	14(2)	19(2)	24(2)	1.2(18)	2.7(17)	-3.0(18)
C6A	13(2)	17(2)	27(2)	1.8(18)	0.8(17)	3.2(17)
O1C	16.3(17)	12.2(14)	34.9(18)	0.5(12)	0.2(12)	1.0(12)
N2C	10.2(18)	10.6(17)	40(2)	-0.6(15)	1.3(15)	0.3(14)
N1C	9.7(18)	16.1(17)	25(2)	-0.4(15)	-0.2(14)	2.0(14)
N3C	23(2)	7.2(16)	32(2)	1.2(14)	1.7(15)	-1.1(15)
C3C	15(2)	6.6(19)	21(2)	-0.6(16)	0.3(17)	2.4(16)
C2C	13(2)	10.5(19)	15(2)	-1.8(16)	-0.3(16)	-1.1(16)
C7C	24(3)	17(2)	38(3)	0(2)	1.1(19)	5.3(19)
C8C	33(3)	10(2)	37(3)	0.7(19)	10(2)	-4.3(19)
C1C	13(2)	11(2)	22(2)	-2.4(16)	4.1(16)	3.6(17)
C5C	17(2)	13(2)	24(2)	-0.9(17)	1.0(17)	-4.5(17)
C4C	16(2)	11(2)	20(2)	-4.7(16)	3.0(16)	0.0(17)
C6C	13(2)	17(2)	29(2)	-2.6(18)	1.1(17)	-0.4(17)
O1B	10.2(16)	13.2(14)	41.4(19)	-0.1(13)	0.1(12)	5.1(11)
N3B	15.0(19)	14.4(18)	34(2)	2.5(15)	1.2(15)	-1.2(15)
N1B	17.3(19)	11.7(17)	23.7(19)	-3.9(14)	-1.1(14)	-2.7(14)

N2B	15.2(19)	10.4(17)	41(2)	2.9(15)	-1.5(15)	-1.8(14)
C6B	11(2)	19(2)	27(2)	-2.3(18)	0.3(17)	-3.0(17)
C7B	26(3)	17(2)	50(3)	6(2)	-2(2)	5.2(19)
C5B	11(2)	20(2)	25(2)	-2.2(18)	2.6(17)	-4.0(18)
C4B	16(2)	17(2)	17(2)	-0.6(17)	3.3(16)	1.1(18)
C2B	11(2)	19(2)	17(2)	-1.6(17)	1.1(16)	5.2(17)
C8B	26(3)	17(2)	37(3)	2.8(19)	5(2)	-3(2)
C3B	11(2)	20(2)	19(2)	-2.8(17)	-0.4(16)	2.9(17)
C1B	17(2)	18(2)	19(2)	-1.0(17)	1.1(17)	2.5(18)
O1D	11.8(16)	14.3(14)	36.4(18)	2.2(12)	-0.5(12)	1.8(12)
N1D	16.3(19)	10.1(16)	25(2)	0.2(14)	2.4(14)	4.5(15)
N2D	13.2(19)	6.8(17)	56(3)	-0.3(16)	3.5(17)	2.7(14)
N3D	25(2)	11.3(17)	37(2)	-2.8(16)	2.3(16)	0.5(15)
C3D	13(2)	12(2)	22(2)	2.2(17)	2.6(17)	1.1(17)
C1D	15(2)	15(2)	23(2)	1.2(17)	-0.3(17)	2.2(17)
C2D	14(2)	11(2)	17(2)	1.3(16)	-1.9(16)	1.6(17)
C8D	37(3)	9(2)	42(3)	-1.1(19)	6(2)	4(2)
C6D	15(2)	21(2)	30(2)	2.4(19)	-0.5(17)	1.2(18)
C7D	33(3)	17(2)	36(3)	-2(2)	1(2)	-6(2)
C4D	22(2)	13(2)	21(2)	1.7(17)	0.3(17)	0.7(18)
C5D	17(2)	16(2)	27(3)	3.7(18)	4.3(18)	4.8(18)

Table 4 Bond Lengths for 16srv014.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1A	C1A	1.227(4)	O1B	C1B	1.237(4)
N2A	C1A	1.332(5)	N3B	C7B	1.468(5)
N1A	C2A	1.349(5)	N3B	C4B	1.339(5)
N1A	C6A	1.337(5)	N3B	C8B	1.463(5)
N3A	C7A	1.458(5)	N1B	C6B	1.341(5)
N3A	C4A	1.363(5)	N1B	C2B	1.345(5)
N3A	C8A	1.457(5)	N2B	C1B	1.337(5)
C2A	C1A	1.501(5)	C6B	C5B	1.374(5)
C2A	C3A	1.382(5)	C5B	C4B	1.409(5)
C4A	C5A	1.412(5)	C4B	C3B	1.416(5)
C4A	C3A	1.405(5)	C2B	C3B	1.388(5)
C5A	C6A	1.379(5)	C2B	C1B	1.502(5)
O1C	C1C	1.236(4)	O1D	C1D	1.236(5)
N2C	C1C	1.330(5)	N1D	C2D	1.343(5)
N1C	C2C	1.344(5)	N1D	C6D	1.344(5)
N1C	C6C	1.352(5)	N2D	C1D	1.333(5)
N3C	C7C	1.465(5)	N3D	C8D	1.446(5)

N3C	C8C	1.445(5)	N3D	C7D	1.471(5)
N3C	C4C	1.353(5)	N3D	C4D	1.367(5)
C3C	C2C	1.384(5)	C3D	C2D	1.383(5)
C3C	C4C	1.403(5)	C3D	C4D	1.405(5)
C2C	C1C	1.500(5)	C1D	C2D	1.498(5)
C5C	C4C	1.408(5)	C6D	C5D	1.371(6)
C5C	C6C	1.380(5)	C4D	C5D	1.401(6)

Table 5 Bond Angles for 16srv014.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C6A	N1A	C2A	116.0(3)	C4B	N3B	C7B	121.4(3)
C4A	N3A	C7A	120.0(3)	C4B	N3B	C8B	120.5(3)
C4A	N3A	C8A	120.3(3)	C8B	N3B	C7B	117.5(3)
C8A	N3A	C7A	119.1(3)	C6B	N1B	C2B	115.5(3)
N1A	C2A	C1A	118.4(4)	N1B	C6B	C5B	125.2(4)
N1A	C2A	C3A	124.0(4)	C6B	C5B	C4B	120.1(4)
C3A	C2A	C1A	117.6(4)	N3B	C4B	C5B	123.7(4)
O1A	C1A	N2A	122.9(4)	N3B	C4B	C3B	121.3(4)
O1A	C1A	C2A	120.1(3)	C5B	C4B	C3B	115.0(4)
N2A	C1A	C2A	117.0(3)	N1B	C2B	C3B	123.8(4)
N3A	C4A	C5A	122.0(4)	N1B	C2B	C1B	118.4(3)
N3A	C4A	C3A	121.9(4)	C3B	C2B	C1B	117.7(3)
C3A	C4A	C5A	116.1(4)	C2B	C3B	C4B	120.4(4)
C6A	C5A	C4A	119.4(4)	O1B	C1B	N2B	121.9(4)
C2A	C3A	C4A	119.8(4)	O1B	C1B	C2B	121.1(3)
N1A	C6A	C5A	124.7(4)	N2B	C1B	C2B	117.0(3)
C2C	N1C	C6C	115.3(3)	C2D	N1D	C6D	115.2(3)
C8C	N3C	C7C	118.2(3)	C8D	N3D	C7D	118.4(3)
C4C	N3C	C7C	119.9(3)	C4D	N3D	C8D	121.4(4)
C4C	N3C	C8C	120.9(3)	C4D	N3D	C7D	120.0(3)
C2C	C3C	C4C	119.8(4)	C2D	C3D	C4D	118.6(4)
N1C	C2C	C3C	124.5(4)	O1D	C1D	N2D	121.8(4)
N1C	C2C	C1C	117.8(3)	O1D	C1D	C2D	121.1(3)
C3C	C2C	C1C	117.8(3)	N2D	C1D	C2D	117.1(3)
O1C	C1C	N2C	122.0(4)	N1D	C2D	C3D	125.1(4)
O1C	C1C	C2C	120.5(3)	N1D	C2D	C1D	117.8(3)
N2C	C1C	C2C	117.5(3)	C3D	C2D	C1D	117.1(3)
C6C	C5C	C4C	119.4(4)	N1D	C6D	C5D	124.8(4)
N3C	C4C	C3C	122.2(4)	N3D	C4D	C3D	121.6(4)
N3C	C4C	C5C	121.5(4)	N3D	C4D	C5D	121.7(4)
C3C	C4C	C5C	116.2(3)	C5D	C4D	C3D	116.7(4)

N1C	C6C	C5C	124.8(4)	C6D	C5D	C4D	119.6(4)

Table 6 Hydrogen Bonds for 16srv014.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N2A	H2AA	N1B[1]	0.88	2.09	2.968(5)	173.3
N2A	H2AB	O1B	0.88	2.17	2.903(4)	140.3
N2C	H2CA	N1D	0.88	2.09	2.960(4)	167.5
N2C	H2CB	O1D[1]	0.88	2.22	2.938(4)	137.9
N2B	H2BA	N1A	0.88	2.09	2.961(5)	168.3
N2B	H2BB	O1A[2]	0.88	2.23	2.941(4)	137.7
N2D	H2DA	N1C[2]	0.88	2.10	2.975(4)	172.1
N2D	H2DB	O1C	0.88	2.18	2.910(4)	140.5

[1]-1+X,+Y,+Z; [2]1+X,+Y,+Z

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16srv014.

Atom	*x*	*y*	*z*	U(eq)
H2AA	4004	2107	2083	32
H2AB	5899	1833	2050	32
H7AA	3305	-1905	1929	30(7)
H7AB	3925	-2518	1505	30(7)
H7AC	3553	-1619	1352	30(7)
H5A	8986	-1165	1869	24
H8AA	7837	-2301	1403	32(7)
H8AB	6589	-2905	1690	32(7)
H8AC	7942	-2368	2015	32(7)
H3A	3847	-523	1837	23
H6A	9693	140	1993	23
H2CA	6022	7764	374	24
H2CB	4122	7486	355	24
H3C	6148	5142	662	17
H7CA	6736	3760	601	47(8)
H7CB	5999	3154	1012	47(8)
H7CC	6320	4056	1166	47(8)
H8CA	2100	3368	1032	30(7)
H8CB	3420	2763	774	30(7)
H8CC	2119	3285	422	30(7)
H5C	1029	4484	544	22
H6C	328	5784	394	24

H2BA	9006	1537	2117	27
H2BB	10904	1818	2129	27
H6B	14677	3529	2124	23
H7BA	8635	5281	1359	61(10)
H7BB	8947	6170	1546	61(10)
H7BC	8254	5524	1938	61(10)
H5B	13991	4823	1977	23
H8BA	12872	6053	2083	39(8)
H8BB	11551	6554	1723	39(8)
H8BC	12882	5946	1474	39(8)
H3B	8865	4172	1867	20
H2DA	10991	7204	407	30
H2DB	9100	7476	455	30
H3D	11177	9835	636	19
H8DA	7092	11699	490	33(7)
H8DB	8461	12206	829	33(7)
H8DC	7191	11593	1099	33(7)
H6D	5321	9175	502	26
H7DA	11538	10925	1100	51(9)
H7DB	11117	11828	967	51(9)
H7DC	11670	11235	524	51(9)
H5D	6038	10474	615	24

Experimental

Single crystals of $C_8H_{11}N_3O$ **[16srv014]** were **[]**. A suitable crystal was selected and **[]** on a diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the SIR2004 [2] structure solution program using Direct Methods and refined with the XH [3] refinement package using CGLS minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2.
3.

Crystal structure determination of [16srv014]

Crystal Data for $C_8H_{11}N_3O$ (M =165.20 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 7.6315(5) Å, b = 16.9683(10) Å, c = 26.1099(16) Å, β = 91.747(2)°, V = 3379.5(4) Å^3, Z = 16, T = 120 K, μ(MoKα) = 0.090 mm^{-1}, $Dcalc$ = 1.299 g/cm^3, 42270 reflections measured (3.938° ≤ 2Θ ≤ 50.154°), 5976 unique (R_{int} = 0.0764, R_{sigma} = 0.0588) which were used in all calculations. The final R_1 was 0.0790 (I > 2σ(I)) and wR_2 was 0.2137 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All N(H,H) groups
2. Others
 Uiso(H7AA)=Uiso(H7AB)=Uiso(H7AC)=FVAR(1)
 Uiso(H8AA)=Uiso(H8AB)=Uiso(H8AC)=FVAR(2)
 Uiso(H7CA)=Uiso(H7CB)=Uiso(H7CC)=FVAR(3)

Uiso(H8CA)=Uiso(H8CB)=Uiso(H8CC)=FVAR(4)
Uiso(H7BA)=Uiso(H7BB)=Uiso(H7BC)=FVAR(5)
Uiso(H8BA)=Uiso(H8BB)=Uiso(H8BC)=FVAR(6)
Uiso(H8DA)=Uiso(H8DB)=Uiso(H8DC)=FVAR(7)
Uiso(H7DA)=Uiso(H7DB)=Uiso(H7DC)=FVAR(8)
3.a Aromatic/amide H refined with riding coordinates:
C5A(H5A), C3A(H3A), C6A(H6A), C3C(H3C), C5C(H5C), C6C(H6C), C6B(H6B),
C5B(H5B), C3B(H3B), C3D(H3D), C6D(H6D), C5D(H5D)
3.b X=CH2 refined with riding coordinates:
N2A(H2AA,H2AB), N2C(H2CA,H2CB), N2B(H2BA,H2BB), N2D(H2DA,H2DB)
3.c Idealised Me refined as rotating group:
C7A(H7AA,H7AB,H7AC), C8A(H8AA,H8AB,H8AC), C7C(H7CA,H7CB,H7CC), C8C(H8CA,H8CB,
H8CC), C7B(H7BA,H7BB,H7BC), C8B(H8BA,H8BB,H8BC), C8D(H8DA,H8DB,H8DC), C7D(H7DA,
H7DB,H7DC)

A9.7 – X-Ray Crystallographic Parameters for 4-Dimethylamino-2-Cyanopyridine

Parameter	
Empirical formula	$C_8H_9N_3$
Formula weight	147.18
Temperature/K	120
Crystal system	orthorhombic
Space group	Pnma
a/Å	10.5874(4)
b/Å	6.6852(3)
c/Å	10.9744(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	776.76(5)
Z	4
ρ_{calc}g/cm^3	1.259
μ/mm^{-1}	0.081
F(000)	312.0
Crystal size/mm^3	0.439 × 0.31 × 0.194
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.346 to 65.56
Index ranges	-16 ≤ h ≤ 16, -10 ≤ k ≤ 10, -16 ≤ l ≤ 16
Reflections collected	18939
Independent reflections	1542 [R_{int} = 0.0326, R_{sigma} = 0.0170]
Data/restraints/parameters	1542/0/81
Goodness-of-fit on F^2	1.077
Final R indexes [I>=2σ (I)]	R_1 = 0.0428, wR_2 = 0.1266
Final R indexes [all data]	R_1 = 0.0506, wR_2 = 0.1331
Largest diff. peak/hole / e Å^{-3}	0.45/-0.30

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16srv052. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	-687.0(9)	2500	6803.2(8)	23.9(2)
N2	-3147.2(9)	2500	4824.6(9)	26.6(2)
N3	2393.7(8)	2500	4368.5(9)	24.1(2)
C1	-2116.5(9)	2500	5162.6(9)	20.0(2)
C2	-819.9(9)	2500	5585.5(9)	17.2(2)
C3	141.1(9)	2500	4734.1(8)	16.6(2)
C4	1406.6(8)	2500	5146.5(9)	17.3(2)
C5	1571.3(9)	2500	6427.0(9)	21.9(2)
C6	527.3(10)	2500	7176.4(9)	26.2(2)
C7	2187.8(11)	2500	3059.1(11)	27.3(2)
C8	3685.1(10)	2500	4825.2(13)	30.2(3)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for 16srv052. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	18.5(4)	34.3(5)	18.9(4)	0	0.6(3)	0
N2	15.9(4)	34.6(5)	29.4(5)	0	-0.4(3)	0
N3	13.1(3)	34.8(5)	24.5(4)	0	2.1(3)	0
C1	15.6(4)	22.5(4)	21.9(5)	0	1.2(3)	0
C2	13.3(4)	18.5(4)	19.8(4)	0	-0.4(3)	0
C3	13.2(4)	18.2(4)	18.4(4)	0	-0.8(3)	0
C4	12.9(4)	17.8(4)	21.2(4)	0	-0.2(3)	0
C5	16.8(4)	27.2(5)	21.7(4)	0	-4.6(3)	0
C6	21.7(5)	39.0(6)	17.9(4)	0	-2.4(4)	0
C7	22.7(5)	35.2(6)	24.0(5)	0	7.4(4)	0
C8	12.4(4)	36.2(6)	42.0(6)	0	-1.0(4)	0

Table 4 Bond Lengths for 16srv052.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C2	1.3437(13)	C1	C2	1.4491(13)
N1	C6	1.3493(13)	C2	C3	1.3814(13)
N2	C1	1.1526(13)	C3	C4	1.4142(12)
N3	C4	1.3494(13)	C4	C5	1.4161(14)
N3	C7	1.4534(15)	C5	C6	1.3777(14)

N3	C8	1.4563(14)

Table 5 Bond Angles for 16srv052.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C2	N1	C6	113.68(9)	C3	C2	C1	118.75(9)
C4	N3	C7	120.62(9)	C2	C3	C4	118.77(8)
C4	N3	C8	120.62(9)	N3	C4	C3	122.09(9)
C7	N3	C8	118.75(9)	N3	C4	C5	122.18(9)
N2	C1	C2	179.91(11)	C3	C4	C5	115.73(8)
N1	C2	C1	114.69(8)	C6	C5	C4	119.58(9)
N1	C2	C3	126.55(9)	N1	C6	C5	125.68(9)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16srv052.

Atom	*x*	*y*	*z*	U(eq)
H3	-45	2500	3887	20
H5	2396	2500	6767	26
H6	675	2500	8030	31
H7A	1685(10)	1348(18)	2823(12)	42(3)
H7B	2945(19)	2500	2657(18)	54(6)
H8A	3859(12)	1338(17)	5281(10)	38(3)
H8B	4230(30)	2500	4210(20)	78(7)

Experimental

Single crystals of $C_8H_9N_3$ **[16srv052]** were **[]**. A suitable crystal was selected and **[]** on a **Bruker D8 Venture** diffractometer. The crystal was kept at 120 K during data collection. Using Olex2 [1], the structure was solved with the SIR2004 [2] structure solution program using Direct Methods and refined with the XH [3] refinement package using CGLS minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2.
3.

Crystal structure determination of [16srv052]

Crystal Data for $C_8H_9N_3$ (*M* =147.18 g/mol): orthorhombic, space group Pnma (no. 62), *a* = 10.5874(4) Å, *b* = 6.6852(3) Å, *c* = 10.9744(4) Å, *V* = 776.76(5) Å^3, *Z* = 4, *T* = 120 K, μ(MoKα) = 0.081 mm^{-1}, *Dcalc* = 1.259 g/cm^3, 18939 reflections measured (5.346° ≤ 2Θ ≤ 65.56°), 1542 unique (R_{int} = 0.0326, R_{sigma} = 0.0170) which were used in all calculations. The final R_1 was 0.0428 (I > 2σ(I)) and wR_2 was 0.1331 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups

2.a Aromatic/amide H refined with riding coordinates:
C3(H3), C5(H5), C6(H6)