Synthesis and Structural Features of α-Fluorocarbonyl Systems

FISHER, CRAIG,AARON

Appendix 3 - X-ray Crystallography Information

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A3.1. Dimethyl 2-fluoro-2-methylmalonate 232 (15srv211)

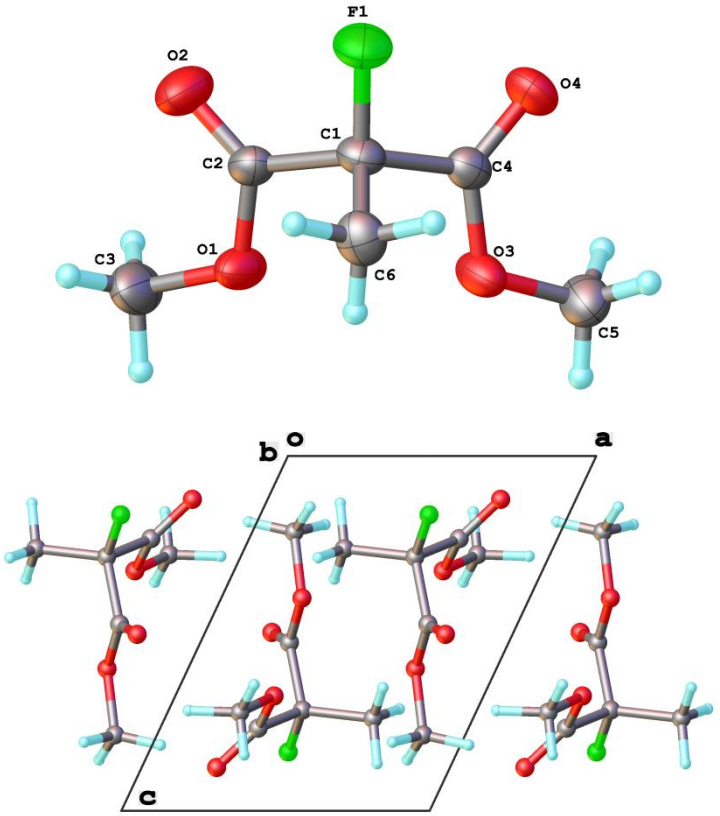


Table 1 Crystal data and structure refinement for 15srv211.	
Identification code	15srv211
Empirical formula	$C_6H_9O_4F$
Formula weight	164.13
Temperature/K	250.0
Crystal system	triclinic
Space group	P-1
a/Å	6.701(2)
b/Å	7.893(2)
c/Å	8.182(3)
α/°	93.71(6)
β/°	112.89(5)
γ/°	106.42(9)
Volume/Å^3	374.87(19)
Z	2
ρ_{calc}g/cm^3	1.454

μ/mm^{-1}	0.137
F(000)	172.0
Crystal size/mm^3	0.3 × 0.3 × 0.3
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.52 to 55.98
Index ranges	$-8 \le h \le 8$, $-9 \le k \le 9$, $-10 \le l \le 10$
Reflections collected	2783
Independent reflections	1425 [R_{int} = 0.0301, R_{sigma} = 0.0439]
Data/restraints/parameters	1425/0/136
Goodness-of-fit on F^2	1.094
Final R indexes [I>=2σ (I)]	R_1 = 0.0643, wR_2 = 0.1588
Final R indexes [all data]	R_1 = 0.0800, wR_2 = 0.1710
Largest diff. peak/hole / e $Å^{-3}$	0.43/-0.34

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 15srv211. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	4522(3)	6250(2)	8341(2)	46.9(5)
O1	2622(4)	7218(3)	4003(2)	45.9(6)
O2	2170(5)	4635(3)	4972(3)	56.3(7)
O3	3211(3)	9939(3)	6764(2)	40.9(5)
O4	2435(3)	8419(3)	8778(2)	43.7(6)
C1	4343(4)	7420(4)	7160(3)	32.1(6)
C2	2893(4)	6237(4)	5257(3)	32.2(6)
C3	1363(7)	6219(5)	2141(4)	53.1(9)
C4	3182(4)	8638(4)	7670(3)	29.3(6)
C5	2347(6)	11285(5)	7223(4)	44.2(7)
C6	6755(5)	8507(5)	7390(4)	40.3(7)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 15srv211. 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}**
F1	62(1)	46.5(13)	38.7(8)	22.4(7)	21.9(7)	23.9(10)
O1	65.1(13)	30.3(14)	28.8(9)	6.3(8)	15.6(8)	2.7(11)
O2	80.0(17)	31.9(19)	47.7(12)	10.4(10)	23.6(11)	10.1(14)
O3	56.1(12)	45.5(14)	40.4(9)	20.0(8)	30.8(9)	27.5(11)
O4	51.0(11)	49.1(15)	41.8(10)	12.5(8)	31.7(9)	14.4(10)

C1	36.5(12)	33.8(18)	29.1(11)	13(1)	15.9(9)	12.2(13)
C2	36.7(13)	28(2)	35.6(12)	9(1)	20.5(10)	8.3(14)
C3	72(2)	44(2)	30.5(13)	4.2(12)	18.0(13)	8(2)
C4	26.5(10)	31.5(17)	24.7(10)	4.4(9)	9.3(8)	4.5(11)
C5	55.8(17)	43(2)	43.4(15)	13.5(13)	24.1(13)	25.3(16)
C6	33.4(13)	46(2)	41.4(14)	5.5(12)	16.9(11)	13.1(14)

Table 4 Bond Lengths for 15srv211.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C1	1.375(3)		O3	C5	1.439(3)
O1	C2	1.313(3)		O4	C4	1.195(3)
O1	C3	1.457(4)		C1	C2	1.537(4)
O2	C2	1.190(4)		C1	C4	1.525(4)
O3	C4	1.305(3)		C1	C6	1.530(4)

Table 5 Bond Angles for 15srv211.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C2	O1	C3	115.7(3)		C6	C1	C2	111.3(2)
C4	O3	C5	115.93(19)		O1	C2	C1	111.3(2)
F1	C1	C2	105.8(2)		O2	C2	O1	124.9(2)
F1	C1	C4	105.91(17)		O2	C2	C1	123.8(2)
F1	C1	C6	108.9(2)		O3	C4	C1	109.80(18)
C4	C1	C2	113.22(19)		O4	C4	O3	126.7(2)
C4	C1	C6	111.2(2)		O4	C4	C1	123.5(2)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15srv211.

Atom	***x***	***y***	***z***	**U(eq)**
H3A	2250(70)	5410(60)	1940(50)	70(11)
H3B	1500(80)	7180(60)	1440(60)	82(13)
H3C	-270(80)	5560(60)	1850(60)	83(13)
H5A	3130(60)	11840(50)	8420(50)	54(9)
H5B	2390(70)	12070(60)	6450(50)	66(11)
H5C	650(90)	10780(70)	7170(60)	96(14)
H6A	7470(60)	7680(50)	7130(40)	45(8)
H6B	6620(50)	9240(40)	6530(40)	38(7)
H6C	7580(60)	9180(50)	8670(50)	59(10)

A3.2. Dimethyl 2-fluoro-2-(4-nitrobenzyl)malonate 236 (15srv034)

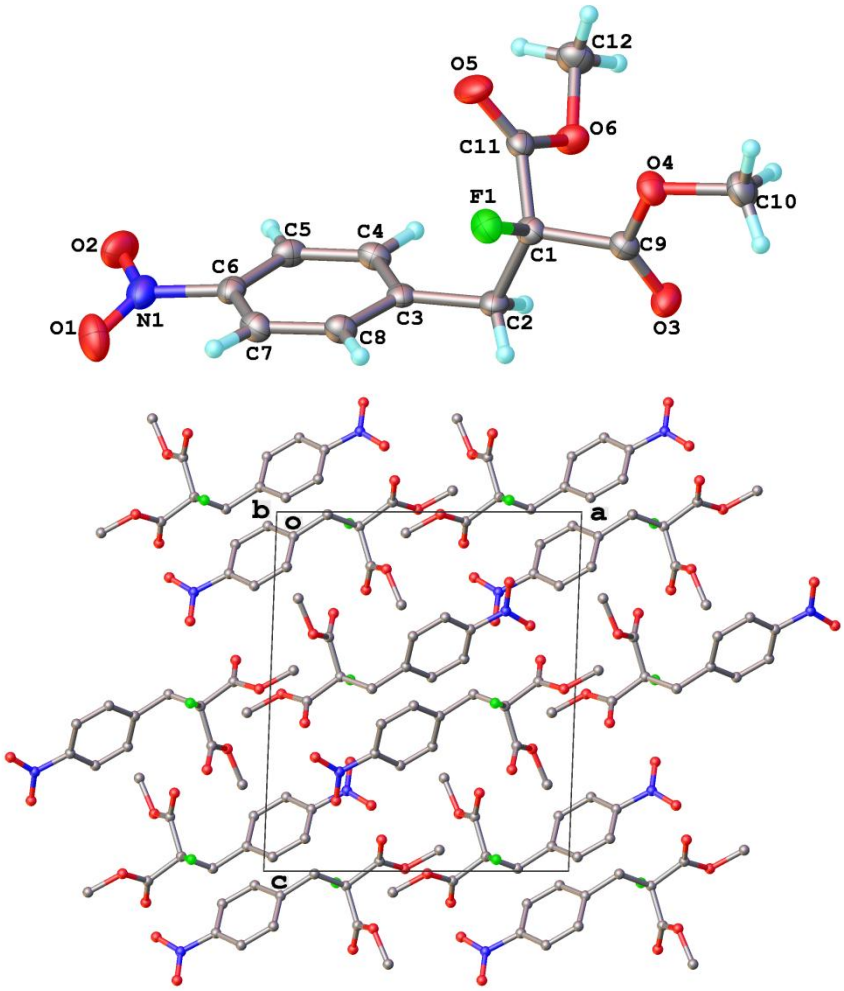


Table 1 Crystal data and structure refinement for 15srv034.	
Identification code	15srv034
Empirical formula	$C_{12}H_{12}FNO_6$
Formula weight	285.23
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	11.2398(3)
b/Å	8.5962(2)
c/Å	13.2556(3)
α/°	90.00
β/°	92.089(2)

γ/°	90.00
Volume/Å^3	1279.90(5)
Z	4
ρ_{calc}g/cm^3	1.480
μ/mm^{-1}	0.129
F(000)	592.0
Crystal size/mm^3	0.21 × 0.2 × 0.11
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.66 to 57.98
Index ranges	-15 ≤ h ≤ 15, -11 ≤ k ≤ 11, -18 ≤ l ≤ 18
Reflections collected	18054
Independent reflections	3404 [R_{int} = 0.0515, R_{sigma} = 0.0433]
Data/restraints/parameters	3404/0/229
Goodness-of-fit on F^2	1.033
Final R indexes [I>=2σ (I)]	R_1 = 0.0427, wR_2 = 0.0929
Final R indexes [all data]	R_1 = 0.0676, wR_2 = 0.1071
Largest diff. peak/hole / e Å^{-3}	0.30/-0.20

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 15srv034. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	2610.2(7)	6066.1(9)	4676.3(7)	26.9(2)
O1	8447.3(10)	7290.2(14)	3169.8(11)	42.4(3)
O2	7714.0(11)	8735.3(16)	1960.4(9)	43.1(3)
O3	1170.3(10)	8952.2(13)	5870.3(9)	33.2(3)
O4	369.2(9)	6865.7(12)	5070.1(9)	29.1(3)
O5	1979.9(10)	6955.3(13)	2825.0(9)	37.5(3)
O6	1339.2(9)	9256.7(12)	3401.1(8)	27.6(3)
N1	7629.1(12)	8034.8(15)	2762.5(11)	30.7(3)
C1	2274.5(12)	7623.6(16)	4592.0(11)	21.7(3)
C2	3328.6(13)	8642.9(18)	4921.5(12)	23.5(3)
C3	4420.3(12)	8407.9(16)	4313.4(11)	20.7(3)
C4	4613.5(13)	9326.4(17)	3466.2(12)	24.2(3)
C5	5649.5(13)	9182.1(18)	2940.9(12)	25.4(3)
C6	6493.1(13)	8111.6(17)	3274.9(11)	23.7(3)
C7	6317.5(14)	7146.3(17)	4091.5(12)	25.7(3)
C8	5276.3(13)	7300.8(18)	4604.0(12)	24.6(3)
C9	1215.3(12)	7909.9(17)	5272.2(11)	23.6(3)

C10	-737.0(14)	7094(2)	5586.5(15)	32.6(4)
C11	1855.1(12)	7876.7(17)	3488.3(12)	23.5(3)
C12	805.2(17)	9625(2)	2410.9(13)	34.0(4)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 15srv034. 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}**
F1	27.5(5)	18.2(4)	35.0(5)	0.1(4)	1.0(4)	1.4(3)
O1	30.6(6)	34.4(7)	62.7(9)	-0.3(6)	10.7(6)	6.6(5)
O2	40.7(7)	60.2(9)	29.0(7)	-1.3(6)	10.8(5)	-10.9(6)
O3	28.7(6)	34.7(6)	36.7(7)	-12.3(5)	6.3(5)	-3.7(5)
O4	22.3(5)	26.8(6)	38.4(7)	-3.5(5)	3.2(5)	-4.6(4)
O5	43.7(7)	36.1(7)	31.9(7)	-14.7(5)	-8.7(5)	7.5(5)
O6	33.4(6)	25.0(6)	24.1(6)	0.5(4)	-2.4(4)	3.1(4)
N1	30.1(7)	28.4(7)	33.9(8)	-8.8(6)	6.6(6)	-5.7(6)
C1	23.6(7)	15.5(7)	25.8(8)	-2.0(6)	-0.9(6)	0.1(5)
C2	22.6(7)	23.8(8)	23.9(8)	-5.0(6)	-0.8(6)	-3.6(6)
C3	20.6(7)	19.5(7)	22.0(7)	-4.8(6)	-2.1(5)	-3.8(5)
C4	25.1(7)	20.3(7)	26.9(8)	2.2(6)	-4.9(6)	-1.8(6)
C5	30.5(8)	24.6(8)	20.9(8)	2.5(6)	-0.9(6)	-6.0(6)
C6	24.1(7)	23.1(7)	23.9(8)	-6.2(6)	2.3(6)	-4.0(6)
C7	24.8(8)	20.9(7)	31.2(9)	-1.3(6)	-2.7(6)	1.5(6)
C8	25.8(7)	23.9(7)	23.9(8)	3.0(6)	-2.0(6)	-2.2(6)
C9	22.2(7)	22.8(7)	25.5(8)	1.3(6)	-1.4(6)	-1.3(5)
C10	22.2(8)	36.2(10)	39.7(11)	0.2(8)	3.5(7)	-3.7(7)
C11	19.3(7)	23.2(7)	27.8(8)	-4.0(6)	-0.3(6)	-2.5(5)
C12	35.5(9)	37.3(10)	28.8(9)	5.8(8)	-4.1(7)	0.8(8)

Table 4 Bond Lengths for 15srv034.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C1	1.3945(15)		C1	C2	1.5246(19)
O1	N1	1.2291(17)		C1	C9	1.539(2)
O2	N1	1.2286(18)		C1	C11	1.536(2)
O3	C9	1.1987(18)		C2	C3	1.506(2)
O4	C9	1.3279(17)		C3	C4	1.396(2)
O4	C10	1.4543(19)		C3	C8	1.397(2)
O5	C11	1.1955(18)		C4	C5	1.384(2)
O6	C11	1.3236(17)		C5	C6	1.382(2)
O6	C12	1.4580(19)		C6	C7	1.384(2)
N1	C6	1.4694(19)		C7	C8	1.381(2)

Table 5 Bond Angles for 15srv034.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**

C9	O4	C10	115.61(12)		C8	C3	C2	120.67(14)
C11	O6	C12	115.98(13)		C5	C4	C3	120.82(14)
O1	N1	C6	117.98(14)		C6	C5	C4	118.58(14)
O2	N1	O1	123.70(14)		C5	C6	N1	118.64(14)
O2	N1	C6	118.31(13)		C5	C6	C7	122.28(14)
F1	C1	C2	108.88(11)		C7	C6	N1	119.06(13)
F1	C1	C9	108.63(11)		C8	C7	C6	118.34(14)
F1	C1	C11	106.56(11)		C7	C8	C3	121.15(14)
C2	C1	C9	110.48(12)		O3	C9	O4	126.27(14)
C2	C1	C11	113.56(12)		O3	C9	C1	123.99(13)
C11	C1	C9	108.56(11)		O4	C9	C1	109.71(12)
C3	C2	C1	114.17(12)		O5	C11	O6	126.32(15)
C4	C3	C2	120.54(13)		O5	C11	C1	124.51(14)
C4	C3	C8	118.76(14)		O6	C11	C1	109.17(12)

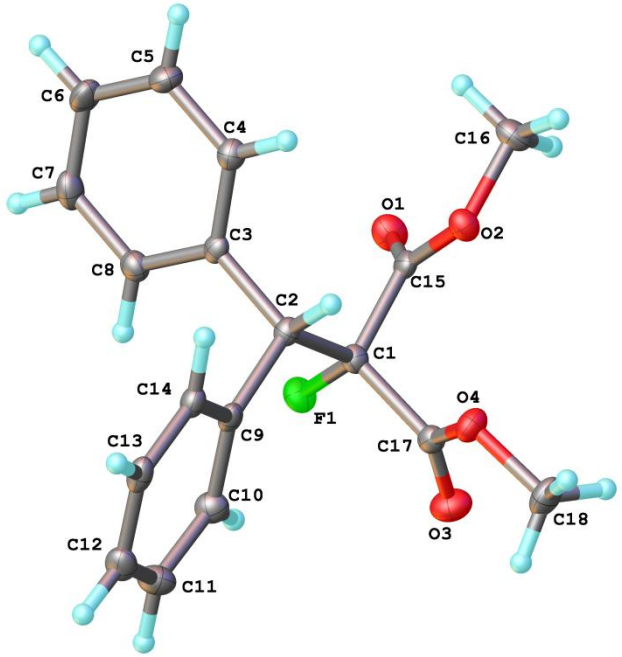
Table 6 Selected Torsion Angles for 15srv034.

A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
O3	C9	C1	F1	129.90(15)		C3	C2	C1	C9	178.43(12)
O4	C9	C1	F1	-51.87(15)		C3	C2	C1	C11	-59.33(17)
O5	C11	C1	F1	-10.20(19)		C5	C6	N1	O1	167.22(13)
O6	C11	C1	F1	169.55(11)		C5	C6	N1	O2	-11.7(2)
C1	C2	C3	C8	-89.91(16)		C7	C6	N1	O1	-10.9(2)
C2	C1	C9	O3	10.5(2)		C7	C6	N1	O2	170.18(14)
C2	C1	C9	O4	-171.24(12)		C9	C1	C11	O5	-127.03(16)
C2	C1	C11	O5	109.67(17)		C9	C1	C11	O6	52.72(15)
C2	C1	C11	O6	-70.58(15)		C11	C1	C9	O3	-114.61(16)
C3	C2	C1	F1	59.21(16)		C11	C1	C9	O4	63.63(15)

Table 7 Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 15srv034.

Atom	***x***	***y***	***z***	**U(eq)**
H2A	3496(14)	8396(19)	5627(14)	30(4)
H2B	3088(15)	9740(20)	4835(13)	33(5)
H4	4022(14)	10083(19)	3233(12)	28(4)
H5	5801(14)	9810(20)	2379(13)	33(5)
H7	6898(15)	6390(20)	4274(13)	35(5)
H8	5134(14)	6659(19)	5197(13)	27(4)
H10A	-1225(16)	6190(20)	5416(14)	40(5)
H10B	-601(17)	7160(20)	6302(16)	48(6)
H10C	-1117(16)	8090(20)	5319(15)	48(6)
H12A	251(17)	8800(20)	2221(14)	44(5)
H12B	1437(16)	9690(20)	1929(14)	39(5)
H12C	421(19)	10630(30)	2474(17)	66(7)

A3.3. Dimethyl 2-diphenylmethyl-2-fluoromalonate 238 (15srv036)



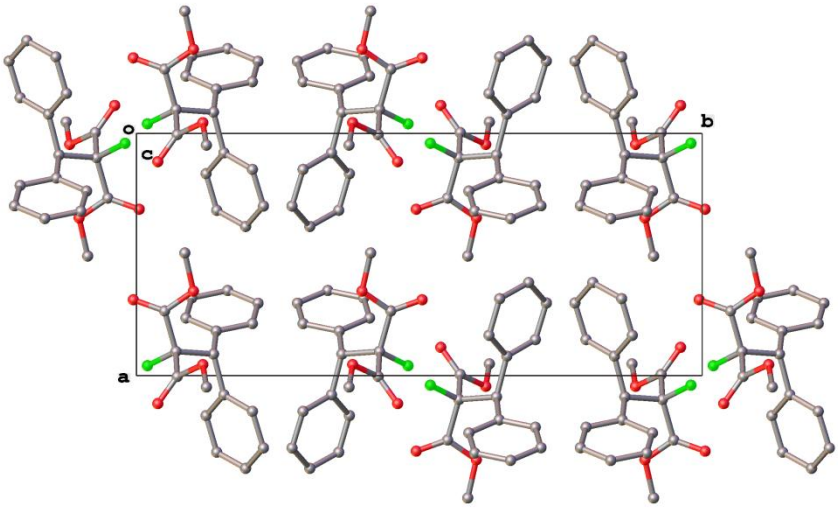


Table 1 Crystal data and structure refinement for 15srv036.	
Identification code	15srv036
Empirical formula	$C_{18}H_{17}FO_4$
Formula weight	316.32
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	8.6393(2)
b/Å	20.2165(4)

c/Å	8.7948(2)
α/°	90.00
β/°	91.495(4)
γ/°	90.00
Volume/Å^3	1535.55(6)
Z	4
ρ_{calc}g/cm^3	1.368
μ/mm^{-1}	0.104
F(000)	664.0
Crystal size/mm^3	0.23 × 0.23 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.72 to 58
Index ranges	-11 ≤ h ≤ 11, -27 ≤ k ≤ 27, -11 ≤ l ≤ 11
Reflections collected	31358
Independent reflections	4072 [R_{int} = 0.0492, R_{sigma} = 0.0339]
Data/restraints/parameters	4072/0/273
Goodness-of-fit on F^2	1.082
Final R indexes [I>=2σ (I)]	R_1 = 0.0508, wR_2 = 0.1228
Final R indexes [all data]	R_1 = 0.0688, wR_2 = 0.1329
Largest diff. peak/hole / e Å^{-3}	0.65/-0.30

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 15srv036. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	402.4(11)	5197.1(5)	3476.3(11)	21.4(2)
O1	-1162.8(14)	5381.1(6)	980.1(14)	24.0(3)
O2	449.8(13)	6176.6(6)	183.5(12)	19.2(2)
O3	3129.5(14)	4947.6(6)	2382.1(15)	27.7(3)
O4	3485.9(13)	6009.9(6)	1746.9(14)	21.4(3)
C1	993.4(18)	5701.5(8)	2601.8(17)	16.2(3)
C2	884.7(17)	6365.7(8)	3444.1(17)	15.7(3)
C3	-799.8(17)	6549.1(7)	3711.6(17)	15.3(3)
C4	-1484.0(18)	7052.7(8)	2857.0(18)	18.3(3)
C5	-2997(2)	7247.8(9)	3117(2)	23.8(4)
C6	-3840.7(19)	6933.1(10)	4219(2)	25.9(4)
C7	-3173(2)	6421.9(9)	5063(2)	24.3(4)
C8	-1658.3(19)	6233.8(8)	4818.8(18)	19.4(3)

C9	1836.9(17)	6413.0(8)	4925.5(17)	16.3(3)
C10	2376.7(19)	5877.4(9)	5776.3(19)	21.4(3)
C11	3207(2)	5981.3(9)	7143(2)	25.2(4)
C12	3513.1(19)	6615.0(9)	7650(2)	23.6(4)
C13	2987.8(18)	7151.9(9)	6803.4(19)	20.0(3)
C14	2142.3(18)	7051.9(8)	5465.4(18)	17.9(3)
C15	-44.6(17)	5723.5(8)	1150.3(17)	16.2(3)
C16	-478(2)	6239.6(9)	-1209(2)	23.8(4)
C17	2668.0(18)	5501.5(8)	2237.9(17)	17.5(3)
C18	5064(2)	5858(1)	1343(2)	27.4(4)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 15srv036. 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}
F1	26.7(5)	16.7(5)	20.9(5)	3.1(4)	2.7(4)	-3.6(4)
O1	24.0(6)	24.6(6)	23.4(6)	0.3(5)	-0.9(5)	-6.2(5)
O2	20.8(6)	20.1(6)	16.7(5)	-0.2(4)	-0.5(4)	-3.2(4)
O3	26.9(6)	21.0(6)	35.2(7)	0.0(5)	-2.6(5)	4.1(5)
O4	18.0(5)	20.3(6)	26.2(6)	-0.8(5)	5.4(5)	1.3(4)
C1	17.4(7)	16.2(7)	14.9(7)	0.4(5)	1.2(6)	-0.9(6)
C2	15.2(7)	15.4(7)	16.4(7)	-2.0(5)	1.7(5)	0.1(6)
C3	14.7(7)	16.6(7)	14.4(7)	-5.6(5)	-0.1(5)	0.0(5)
C4	18.0(7)	18.5(7)	18.4(7)	-1.4(6)	2.3(6)	-1.0(6)
C5	21.0(8)	26.4(8)	23.9(8)	0.5(7)	-0.6(6)	7.2(7)
C6	13.5(7)	39.7(10)	24.7(9)	-5.0(7)	1.2(6)	3.2(7)
C7	22.3(8)	33.1(9)	17.6(8)	-2.5(7)	4.9(6)	-6.4(7)
C8	21.7(8)	21.2(8)	15.3(7)	-1.1(6)	-0.2(6)	0.1(6)
C9	11.7(6)	21.4(8)	15.9(7)	-3.7(6)	2.1(5)	0.3(6)
C10	20.3(8)	21.1(8)	22.6(8)	-1.4(6)	-1.7(6)	-0.6(6)
C11	23.3(8)	27.4(9)	24.7(9)	3.3(7)	-5.2(7)	1.3(7)
C12	18.2(7)	33.6(9)	18.8(8)	-4.3(7)	-2.4(6)	-1.4(7)
C13	15.5(7)	24.1(8)	20.6(8)	-7.8(6)	4.3(6)	-2.6(6)
C14	16.1(7)	20.7(8)	17.2(7)	-3.1(6)	3.4(6)	0.8(6)
C15	16.9(7)	17.7(7)	14.0(7)	-4.0(5)	2.1(5)	3.8(6)
C16	28.1(9)	25.0(9)	18.0(8)	4.0(7)	-3.8(7)	-0.4(7)
C17	17.4(7)	20.4(7)	14.8(7)	-3.0(6)	-1.1(6)	2.0(6)
C18	16.8(8)	33.3(10)	32.3(10)	-6.7(8)	6.0(7)	0.9(7)

Table 4 Bond Lengths for 15srv036.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
F1	C1	1.3830(18)		C3	C4	1.389(2)
O1	C15	1.1947(19)		C3	C8	1.394(2)
O2	C15	1.3279(19)		C4	C5	1.390(2)
O2	C16	1.452(2)		C5	C6	1.383(3)

O3	C17	1.194(2)		C6	C7	1.389(3)
O4	C17	1.326(2)		C7	C8	1.385(2)
O4	C18	1.450(2)		C9	C10	1.390(2)
C1	C2	1.537(2)		C9	C14	1.399(2)
C1	C15	1.541(2)		C10	C11	1.399(2)
C1	C17	1.544(2)		C11	C12	1.380(3)
C2	C3	1.526(2)		C12	C13	1.386(3)
C2	C9	1.526(2)		C13	C14	1.384(2)

Table 5 Bond Angles for 15srv036.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C15	O2	C16	114.90(13)		C8	C7	C6	120.14(16)
C17	O4	C18	115.33(13)		C7	C8	C3	120.34(16)
F1	C1	C2	110.36(12)		C10	C9	C2	125.22(14)
F1	C1	C15	105.46(12)		C10	C9	C14	118.62(14)
F1	C1	C17	106.48(12)		C14	C9	C2	116.14(14)
C2	C1	C15	109.41(12)		C9	C10	C11	120.18(16)
C2	C1	C17	113.45(12)		C12	C11	C10	120.46(16)
C15	C1	C17	111.34(12)		C11	C12	C13	119.72(16)
C3	C2	C1	110.91(12)		C14	C13	C12	120.05(15)
C9	C2	C1	115.34(13)		C13	C14	C9	120.96(15)
C9	C2	C3	110.38(12)		O1	C15	O2	126.30(14)
C4	C3	C2	119.27(14)		O1	C15	C1	122.56(14)
C4	C3	C8	119.13(14)		O2	C15	C1	111.12(13)
C8	C3	C2	121.58(14)		O3	C17	O4	125.60(15)
C3	C4	C5	120.52(15)		O3	C17	C1	122.39(15)
C6	C5	C4	120.00(16)		O4	C17	C1	112.01(13)
C5	C6	C7	119.86(16)					

Table 6 Selected Torsion Angles for 15srv036.

A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
O1	C15	C1	F1	4.3(2)		C3	C2	C1	C15	52.65(16)
O2	C15	C1	F1	-177.53(12)		C3	C2	C1	C17	177.64(12)
O3	C17	C1	F1	16.8(2)		C3	C2	C9	C10	106.18(17)
O4	C17	C1	F1	-163.86(12)		C3	C2	C9	C14	-72.15(17)
C1	C2	C3	C4	-106.80(16)		C4	C3	C2	C9	124.10(15)
C1	C2	C3	C8	74.75(18)		C8	C3	C2	C9	-54.35(19)
C1	C2	C9	C10	-20.5(2)		C9	C2	C1	F1	63.44(16)
C1	C2	C9	C14	161.18(13)		C9	C2	C1	C15	179.05(12)
C2	C1	C15	O1	-114.38(16)		C9	C2	C1	C17	-55.96(17)
C2	C1	C15	O2	63.77(16)		C15	C1	C17	O3	-97.70(18)
C2	C1	C17	O3	138.35(16)		C15	C1	C17	O4	81.68(16)
C2	C1	C17	O4	-42.27(17)		C17	C1	C15	O1	119.41(16)
C3	C2	C1	F1	-62.96(16)		C17	C1	C15	O2	-62.44(16)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15srv036.				
Atom	***x***	***y***	***z***	**U(eq)**
H2	1301	6710	2748	20(5)
H4	-920(20)	7277(11)	2090(20)	27(5)
H5	-3420(20)	7606(11)	2510(20)	26(5)
H6	-4850(30)	7075(11)	4420(20)	29(5)
H7	-3710(30)	6207(13)	5800(30)	43(7)
H8	-1190(20)	5879(10)	5430(20)	25(5)
H10	2150(20)	5432(10)	5470(20)	19(5)
H11	3560(30)	5597(11)	7730(30)	34(6)
H12	4090(20)	6688(11)	8600(30)	31(6)
H13	3210(20)	7590(11)	7140(20)	30(6)
H14	1730(20)	7432(10)	4880(20)	22(5)
H16A	90(30)	6560(12)	-1830(30)	37(6)
H16B	-1520(30)	6380(12)	-920(30)	46(7)
H16C	-540(30)	5819(13)	-1720(30)	44(7)
H18A	5590(20)	5635(11)	2170(30)	28(5)
H18B	5550(30)	6275(11)	1060(30)	31(5)
H18C	5070(30)	5574(12)	430(30)	34(6)

Refinement model description

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

Details:

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1.a Ternary CH refined with riding coordinates:
 C2(H2)
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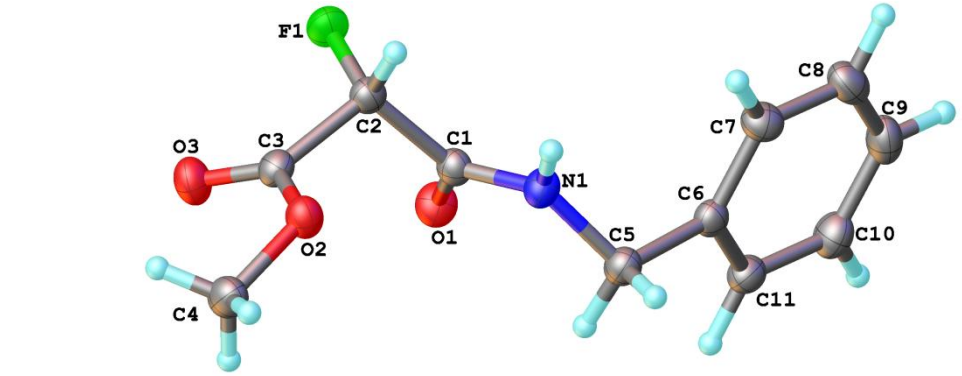
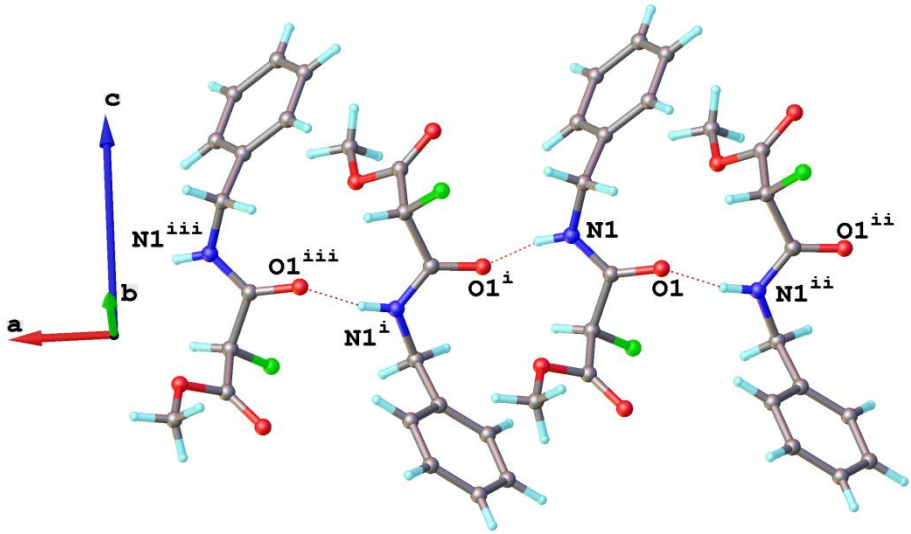
A3.4. Methyl 2-fluoro-3-(benzylamino)-3-oxopropanoate 240 (15srv201)





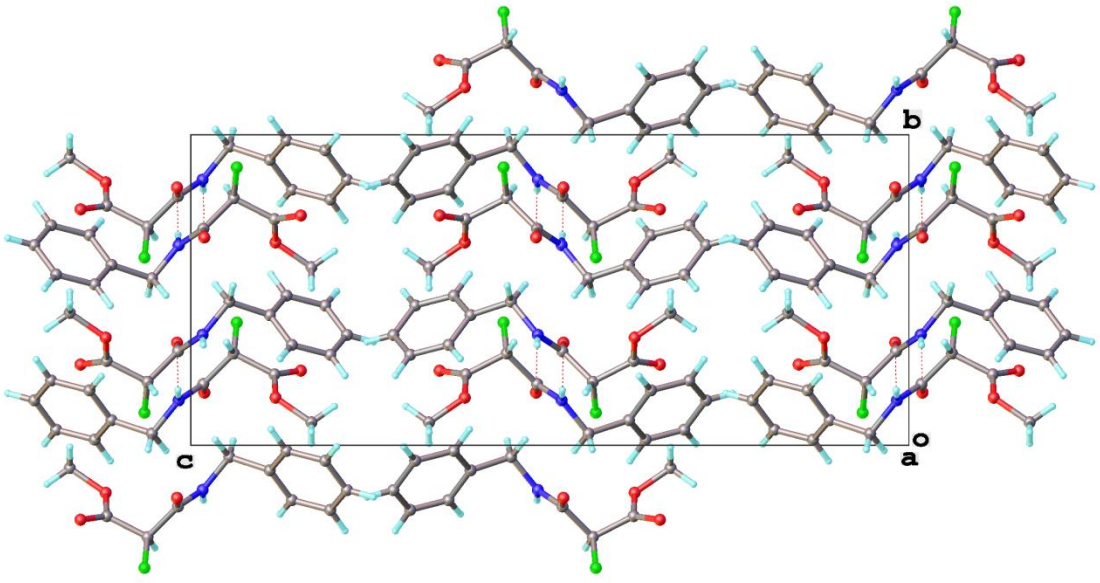


Table 1 Crystal data and structure refinement for 15srv201.

Identification code	15srv201
Empirical formula	$C_{11}H_{12}FNO_3$
Formula weight	225.22
Temperature/K	120.0
Crystal system	orthorhombic
Space group	Pbca
a/Å	8.5328(6)
b/Å	10.3079(8)
c/Å	23.8695(18)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	2099.4(3)
Z	8
ρ_{calc}g/cm^3	1.425
μ/mm^{-1}	0.115
F(000)	944.0
Crystal size/mm^3	0.38 × 0.2 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.42 to 59
Index ranges	$-11 \leq h \leq 11$, $-14 \leq k \leq 14$, $-33 \leq l \leq 33$
Reflections collected	25266
Independent reflections	2925 [R_{int} = 0.0534, R_{sigma} = 0.0518]
Data/restraints/parameters	2925/0/193
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0462, wR_2 = 0.1096
Final R indexes [all data]	R_1 = 0.0711, wR_2 = 0.1315
Largest diff. peak/hole / e Å^{-3}	0.30/-0.26

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 15srv201. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**

F1	1668.1(12)	1046.9(10)	4357.1(4)	30.9(3)
O1	703.3(13)	3305.1(12)	4809.6(5)	28.6(3)
O2	4115.6(14)	3468.5(12)	3817.5(5)	27.0(3)
O3	1902.7(14)	2598.6(13)	3466.4(5)	31.7(3)
N1	3144.5(17)	3557.1(14)	5166.5(6)	24.0(3)
C1	2109.7(18)	3026.7(16)	4823.2(6)	22.5(3)
C2	2746.1(19)	2044.8(16)	4399.3(6)	23.3(3)
C3	2857.0(18)	2716.1(15)	3832.6(6)	22.9(3)
C4	4282(2)	4262.6(19)	3318.0(7)	31.5(4)
C5	2708(2)	4623.8(17)	5538.9(7)	26.0(3)
C6	2097.6(18)	4192.3(15)	6102.7(6)	23.0(3)
C7	2856(2)	3232.5(17)	6409.3(7)	28.1(3)
C8	2337(2)	2915.3(19)	6943.9(8)	33.1(4)
C9	1068(2)	3553.1(19)	7177.0(7)	33.0(4)
C10	300(2)	4501.3(19)	6872.7(7)	32.1(4)
C11	810(2)	4818.7(16)	6335.5(7)	26.4(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 15srv201. 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}
F1	37.5(6)	24.9(5)	30.3(5)	-0.1(4)	-1.8(4)	-8.1(4)
O1	21.1(5)	34.0(7)	30.7(6)	-1.9(5)	-0.2(4)	0.1(5)
O2	27.9(6)	29.6(6)	23.5(6)	0.8(4)	-0.5(4)	-4.6(5)
O3	31.8(6)	38.9(7)	24.5(6)	1.9(5)	-4.9(5)	-4.3(5)
N1	22.2(7)	26.5(7)	23.4(6)	-3.5(5)	-0.4(5)	0.5(5)
C1	23.7(7)	23.8(7)	19.9(7)	1.1(6)	1.3(5)	-1.6(6)
C2	24.5(7)	22.1(7)	23.2(7)	-0.3(6)	-1.0(6)	-2.1(6)
C3	23.4(7)	22.9(7)	22.4(7)	-3.0(6)	1.2(6)	0.8(6)
C4	40.8(10)	28.9(9)	24.8(8)	1.3(7)	3.9(7)	-2.7(8)
C5	29.6(8)	24.3(8)	23.9(7)	-1.4(6)	1.3(6)	-1.6(6)
C6	24.5(7)	22.5(7)	22.0(7)	-1.8(5)	-1.8(6)	-2.7(6)
C7	25.0(8)	28.1(9)	31.1(8)	2.0(7)	-1.8(6)	1.5(6)
C8	36.0(9)	33.3(9)	29.9(8)	6.8(7)	-8.0(7)	-2.8(7)
C9	38.6(10)	38.7(10)	21.9(8)	-0.1(7)	-0.3(7)	-7.5(8)
C10	33.5(9)	34.5(9)	28.2(8)	-5.7(7)	4.0(7)	-0.8(7)
C11	27.9(8)	24.4(8)	26.8(8)	-2.8(6)	-1.0(6)	1.4(6)

Table 4 Bond Lengths for 15srv201.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
F1	C2	1.3836(18)		C2	C3	1.522(2)
O1	C1	1.2344(19)		C5	C6	1.510(2)
O2	C3	1.3252(19)		C6	C7	1.390(2)
O2	C4	1.453(2)		C6	C11	1.390(2)
O3	C3	1.2007(19)		C7	C8	1.390(3)

N1	C1	1.323(2)		C8	C9	1.383(3)
N1	C5	1.462(2)		C9	C10	1.383(3)
C1	C2	1.531(2)		C10	C11	1.393(2)

Table 5 Bond Angles for 15srv201.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C3	O2	C4	115.55(13)		O3	C3	C2	123.97(15)
C1	N1	C5	121.14(14)		N1	C5	C6	114.09(14)
O1	C1	N1	124.70(15)		C7	C6	C5	121.22(15)
O1	C1	C2	118.76(14)		C7	C6	C11	119.21(15)
N1	C1	C2	116.48(14)		C11	C6	C5	119.46(15)
F1	C2	C1	107.68(12)		C8	C7	C6	120.15(16)
F1	C2	C3	108.33(12)		C9	C8	C7	120.46(17)
C3	C2	C1	107.99(13)		C10	C9	C8	119.71(17)
O2	C3	C2	109.90(13)		C9	C10	C11	120.08(17)
O3	C3	O2	126.08(15)		C6	C11	C10	120.38(16)

Table 6 Hydrogen Bonds for 15srv201.

D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1	O1[1]	0.86(2)	2.11(2)	2.9077(19)	155(2)

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

Table 7 Selected Torsion Angles for 15srv201.

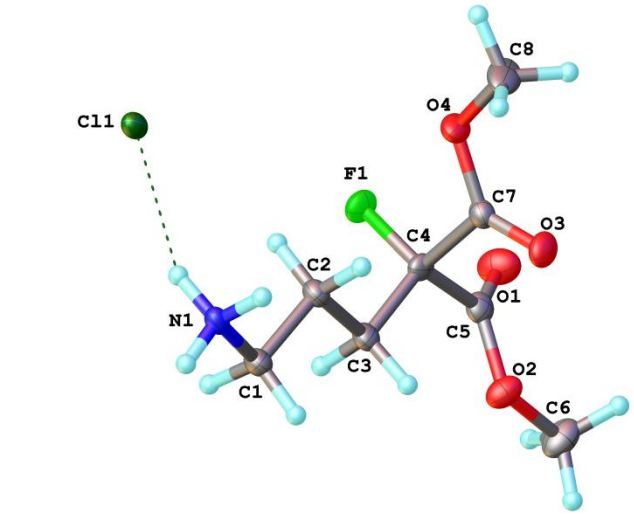
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
O1	C1	C2	F1	-41.90(19)		C3	C2	C1	O1	74.89(18)
O2	C3	C2	F1	-165.68(13)		C3	C2	C1	N1	-102.43(16)
O3	C3	C2	F1	16.5(2)		C4	O2	C3	O3	3.3(2)
N1	C1	C2	F1	140.78(14)		C5	N1	C1	O1	-5.0(3)
C1	C2	C3	O2	77.95(16)		C6	C5	N1	C1	89.20(19)
C1	C2	C3	O3	-99.84(18)		C7	C6	C5	N1	45.7(2)
C2	C1	N1	C5	172.14(14)		C11	C6	C5	N1	-138.24(16)
C2	C3	O2	C4	-174.42(14)						

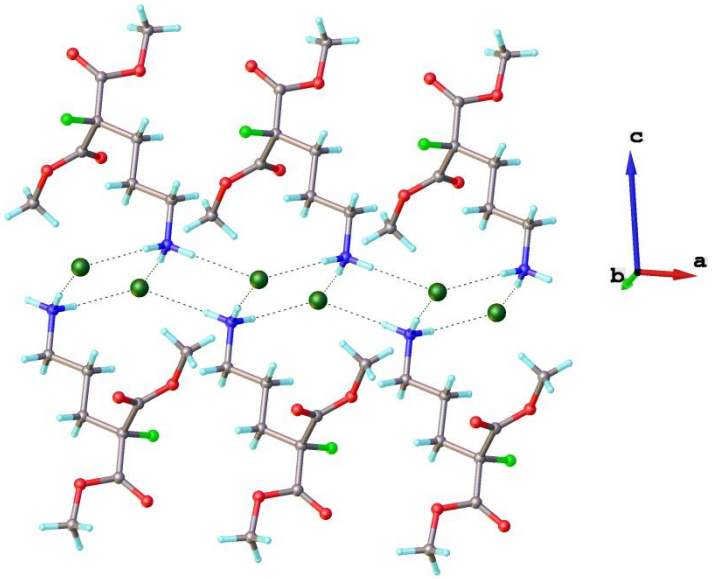
Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15srv201.

Atom	***x***	***y***	***z***	**U(eq)**
H1	4070(30)	3230(20)	5176(9)	38(6)
H2	3740(20)	1691(19)	4500(8)	23(5)
H4A	4100(30)	3750(20)	2999(10)	36(6)
H4B	5360(30)	4590(30)	3319(10)	54(7)
H4C	3540(30)	4940(30)	3318(10)	49(7)

H5A	3640(20)	5140(20)	5592(8)	30(5)
H5B	1890(20)	5170(20)	5345(9)	31(5)
H7	3750(30)	2780(20)	6246(9)	38(6)
H8	2920(30)	2240(30)	7149(10)	49(7)
H9	720(30)	3330(20)	7544(10)	41(6)
H10	-610(30)	4950(20)	7033(9)	36(6)
H11	250(20)	5510(20)	6119(9)	36(6)

A3.5. Dimethyl 2-(3-aminopropyl)-2-fluoromalonate, hydrochloride salt 247 (15srv065)





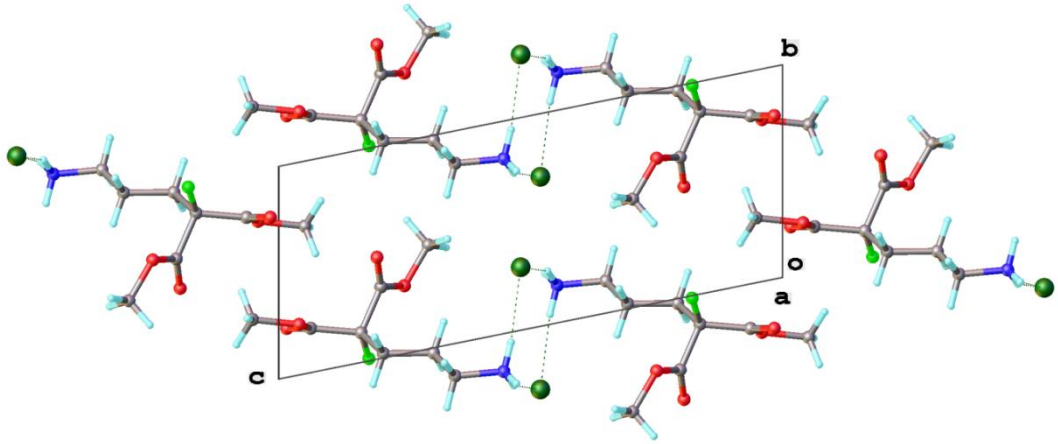


Table 1 Crystal data and structure refinement for 15srv065.	
Identification code	15srv065
Empirical formula	$C_8H_{15}FNO_4^+$ x Cl^-
Formula weight	243.66
Temperature/K	120.0
Crystal system	triclinic
Space group	P-1
a/Å	5.8210(4)
b/Å	6.4875(5)
c/Å	15.7485(12)
α/°	100.738(7)
β/°	96.625(6)
γ/°	94.612(6)
Volume/Å^3	577.23(7)
Z	2
ρ_{calc}g/cm^3	1.402
μ/mm^{-1}	0.340
F(000)	256.0
Crystal size/mm^3	0.36 × 0.21 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.32 to 56
Index ranges	$-7 \le h \le 7$, $-8 \le k \le 8$, $-20 \le l \le 20$
Reflections collected	8623
Independent reflections	2785 [R_{int} = 0.0419, R_{sigma} = 0.0444]
Data/restraints/parameters	2785/0/139
Goodness-of-fit on F^2	1.165
Final R indexes [I>=2σ (I)]	R_1 = 0.0521, wR_2 = 0.1240
Final R indexes [all data]	R_1 = 0.0626, wR_2 = 0.1289
Largest diff. peak/hole / e Å^{-3}	0.56/-0.27

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 15srv065. U_{eq} is defined as 1/3 of of the trace of the

orthogonalised U_{IJ} tensor.				
Atom	***x***	***y***	***z***	**U(eq)**
Cl1	7541.8(10)	12967.3(9)	5196.0(4)	20.11(16)
F1	6831(3)	9887(2)	1791.7(10)	24.8(4)
O1	5595(4)	7691(4)	162.9(14)	35.3(5)
O2	1871(3)	7620(3)	407.2(12)	25.6(4)
O3	3597(3)	5205(3)	1952.9(13)	27.3(4)
O4	7177(3)	6800(3)	2497.0(13)	28.6(5)
N1	2217(4)	11772(3)	4469.5(14)	19.5(4)
C1	1745(5)	11523(4)	3504.7(17)	22.7(5)
C2	3501(4)	10233(4)	3072.4(17)	20.3(5)
C3	2916(5)	9785(4)	2079.7(17)	20.6(5)
C4	4737(4)	8589(4)	1636.2(17)	18.8(5)
C5	4151(5)	7910(4)	635.4(17)	21.5(5)
C6	1074(6)	6948(5)	-520.2(18)	32.0(7)
C7	5104(4)	6631(4)	2030.0(16)	19.6(5)
C8	7546(7)	5106(5)	2970(2)	45.0(9)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 15srv065. 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}**
Cl1	16.0(3)	14.3(3)	30.2(3)	5.0(2)	2.8(2)	1.2(2)
F1	21.1(8)	21.0(8)	31.8(9)	8.6(6)	1.5(6)	-6.4(6)
O1	29.5(11)	48.4(13)	28.8(11)	5.6(10)	7.7(9)	7.1(10)
O2	22.7(10)	31.9(10)	21.5(10)	8.1(8)	0.0(7)	-3.8(8)
O3	31.4(11)	16.1(9)	33.1(11)	7.9(8)	-2.0(8)	-3.8(8)
O4	29.0(11)	17.2(9)	37.2(11)	7.1(8)	-8.9(9)	3.7(8)
N1	17.2(10)	13.9(9)	27.2(11)	2.0(8)	4.3(8)	2.9(8)
C1	21.9(13)	19.2(12)	26.3(13)	2.5(10)	0.4(10)	6(1)
C2	21.4(13)	16.7(11)	22.9(13)	3.8(10)	1.7(10)	5.3(10)
C3	22.9(13)	15.9(11)	23.2(13)	4.8(10)	0.7(10)	4.7(10)
C4	16.5(12)	13.9(11)	25.5(13)	6.4(10)	0.5(10)	-2.7(9)
C5	25.9(14)	15.9(11)	23.5(13)	6.6(10)	2.2(10)	1.6(10)
C6	34.8(16)	36.9(16)	21.6(14)	8.2(12)	-3.7(12)	-7.5(13)
C7	22.1(13)	16.2(11)	20.3(12)	2.6(9)	2.3(10)	3.5(10)
C8	55(2)	21.5(14)	53(2)	12.1(14)	-27.2(17)	6.9(14)

Table 4 Bond Lengths for 15srv065.						
Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C4	1.394(3)		N1	C1	1.488(3)
O1	C5	1.185(3)		C1	C2	1.515(3)
O2	C5	1.323(3)		C2	C3	1.528(4)
O2	C6	1.453(3)		C3	C4	1.521(4)
O3	C7	1.202(3)		C4	C5	1.545(4)

O4	C7	1.324(3)		C4	C7	1.533(3)
O4	C8	1.454(3)				

Table 5 Bond Angles for 15srv065.								
Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C5	O2	C6	116.1(2)		C3	C4	C7	109.2(2)
C7	O4	C8	114.7(2)		C7	C4	C5	109.1(2)
N1	C1	C2	110.3(2)		O1	C5	O2	126.8(3)
C1	C2	C3	111.2(2)		O1	C5	C4	122.9(3)
C4	C3	C2	111.7(2)		O2	C5	C4	110.3(2)
F1	C4	C3	108.8(2)		O3	C7	O4	125.8(2)
F1	C4	C5	106.4(2)		O3	C7	C4	122.2(2)
F1	C4	C7	108.5(2)		O4	C7	C4	111.8(2)
C3	C4	C5	114.6(2)					

Table 6 Hydrogen Bonds for 15srv065.						
D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1A	Cl1[1]	0.91	2.32	3.226(2)	172.2
N1	H1B	Cl1[2]	0.91	2.30	3.169(2)	158.9
N1	H1C	Cl1	0.91	2.31	3.160(2)	154.7

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

Table 7 Torsion Angles for 15srv065.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C4	C5	O1	31.3(3)		C3	C4	C7	O3	65.0(3)
F1	C4	C5	O2	-148.9(2)		C3	C4	C7	O4	-110.8(2)
F1	C4	C7	O3	-176.5(2)		C5	C4	C7	O3	-60.9(3)
F1	C4	C7	O4	7.7(3)		C5	C4	C7	O4	123.2(2)
N1	C1	C2	C3	174.8(2)		C6	O2	C5	O1	0.5(4)
C1	C2	C3	C4	177.0(2)		C6	O2	C5	C4	-179.3(2)
C2	C3	C4	F1	-66.2(3)		C7	C4	C5	O1	-85.7(3)
C2	C3	C4	C5	174.9(2)		C7	C4	C5	O2	94.1(2)
C2	C3	C4	C7	52.2(3)		C8	O4	C7	O3	-2.2(4)
C3	C4	C5	O1	151.6(3)		C8	O4	C7	C4	173.4(2)
C3	C4	C5	O2	-28.6(3)						

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15srv065.				
Atom	***x***	***y***	***z***	**U(eq)**
H1A	2229	10478	4613	23
H1B	1086	12461	4719	23
H1C	3623	12530	4666	23

H1D	1831	12930	3345	27
H1E	157	10812	3295	27
H2A	3518	8882	3278	24
H2B	5072	11009	3242	24
H3A	1380	8946	1910	25
H3B	2815	11136	1878	25
H6A	1722	7978	-835	48
H6B	-628	6848	-620	48
H6C	1590	5566	-731	48
H8A	7465	3767	2554	68
H8B	6342	5019	3352	68
H8C	9080	5391	3322	68

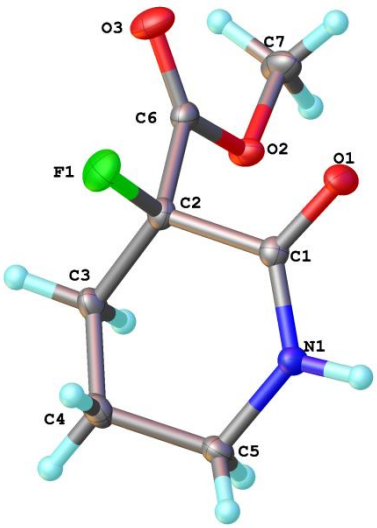
Refinement model description

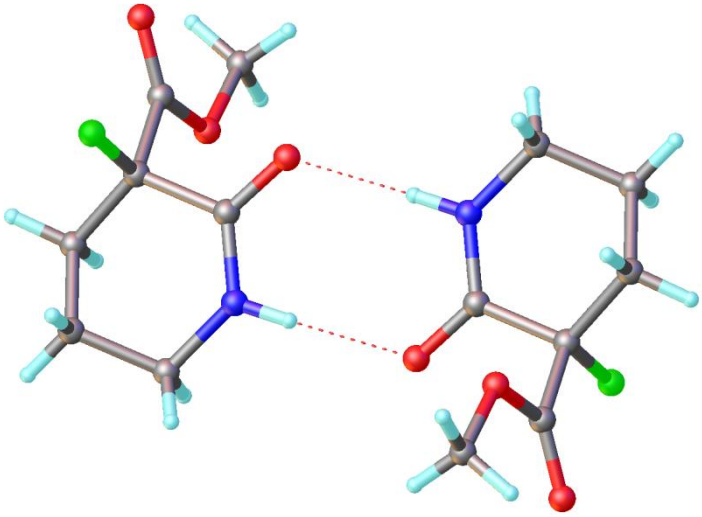
Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H,H) groups, All N(H,H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 C1(H1D,H1E), C2(H2A,H2B), C3(H3A,H3B)
2.b Idealised Me refined as rotating group:
 N1(H1A,H1B,H1C), C6(H6A,H6B,H6C), C8(H8A,H8B,H8C)
```

A3.6. Racemic methyl 3-fluoro-2-oxo-3-piperidinecarboxylate 248 (14srv042)





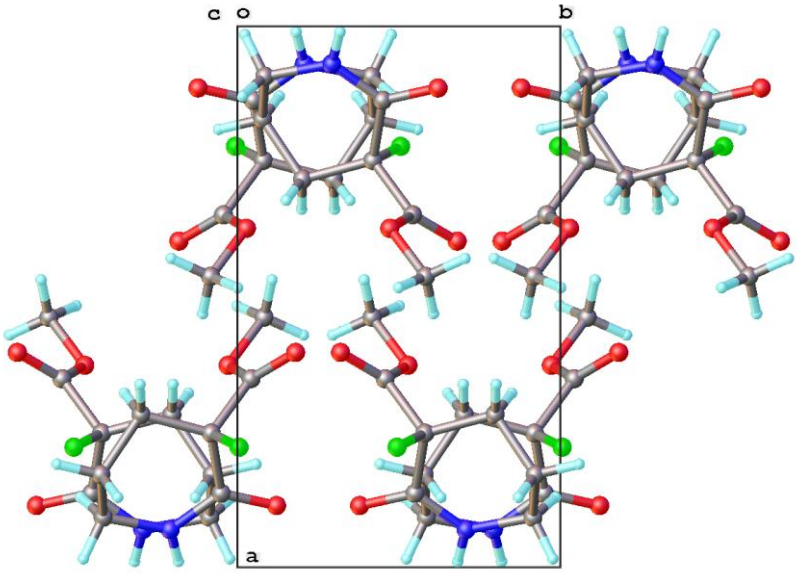


Table 1 Crystal data and structure refinement for 14srv042.	
Identification code	14srv042
Empirical formula	$C_7H_{10}NO_3F$
Formula weight	175.16
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.2560(4)
b/Å	6.72544(17)

c/Å	10.4787(4)
α/°	90.00
β/°	113.193(4)
γ/°	90.00
Volume/Å^3	793.93(4)
Z	4
ρ_{calc}mg/mm^3	1.465
m/mm^{-1}	0.129
F(000)	368.0
Crystal size/mm^3	0.31 × 0.26 × 0.17
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	7.06 to 59.98°
Index ranges	-17 ≤ h ≤ 17, -9 ≤ k ≤ 9, -14 ≤ l ≤ 14
Reflections collected	14038
Independent reflections	2324 [R_{int} = 0.0385, R_{sigma} = 0.0236]
Data/restraints/parameters	2324/0/149
Goodness-of-fit on F^2	1.028
Final R indexes [I>=2σ (I)]	R_1 = 0.0346, wR_2 = 0.0902
Final R indexes [all data]	R_1 = 0.0410, wR_2 = 0.0942
Largest diff. peak/hole / e Å^{-3}	0.47/-0.23

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14srv042. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**

F1	7779.8(6)	10055.9(10)	7218.2(6)	23.13(16)
O1	8858.9(7)	11213.8(10)	5334.4(8)	20.62(17)
O2	6266.6(6)	9718.6(10)	3585.0(7)	17.04(16)
O3	6028.8(7)	11804.4(12)	5143.8(8)	25.13(19)
N1	9301.7(7)	7922.9(12)	5640.6(9)	15.82(17)
C1	8632.1(8)	9499.9(14)	5596(1)	14.08(18)
C2	7518.3(8)	9193.5(14)	5912.6(9)	14.46(18)
C3	7177.6(9)	7025.3(15)	5968.8(11)	19.2(2)
C4	8284.1(9)	5778.4(16)	6731.3(11)	18.9(2)
C5	9075.2(9)	5851.9(14)	5928.8(11)	17.4(2)
C6	6513.9(8)	10424.6(14)	4859.7(10)	15.09(19)
C7	5379.2(10)	10827.8(18)	2466.8(11)	23.5(2)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv042. 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}**
F1	24.4(3)	31.2(3)	14.1(3)	-5.1(2)	7.9(2)	0.9(3)
O1	20.5(4)	12.5(3)	32.8(4)	0.6(3)	14.7(3)	-1.6(3)
O2	18.7(3)	16.9(3)	14.5(3)	1.1(2)	5.4(3)	5.5(2)
O3	26.9(4)	24.5(4)	24.9(4)	-3.6(3)	11.2(3)	8.7(3)
N1	14.9(4)	13.2(4)	21.3(4)	1.1(3)	9.2(3)	-0.3(3)
C1	13.2(4)	14.5(4)	14.1(4)	-1.4(3)	4.9(3)	-2.2(3)
C2	14.6(4)	16.3(4)	13.2(4)	-0.7(3)	6.2(3)	-1.1(3)
C3	16.2(4)	18.0(4)	24.4(5)	5.6(4)	9.1(4)	-1.5(3)
C4	20.1(5)	18.1(4)	18.6(5)	6.0(4)	7.7(4)	0.4(4)

C5	18.9(4)	12.9(4)	20.7(5)	2.9(3)	8.2(4)	0.9(3)
C6	14.8(4)	14.6(4)	17.5(4)	-0.1(3)	8.1(3)	-0.7(3)
C7	25.7(5)	25.5(5)	18.0(5)	6.0(4)	7.4(4)	11.5(4)

Table 4 Bond Lengths for 14srv042.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C2	1.4020(11)		N1	C5	1.4746(12)
O1	C1	1.2414(12)		C1	C2	1.5403(13)
O2	C6	1.3352(12)		C2	C3	1.5243(13)
O2	C7	1.4521(12)		C2	C6	1.5325(13)
O3	C6	1.2011(12)		C3	C4	1.5255(14)
N1	C1	1.3305(12)		C4	C5	1.5139(15)

Table 5 Bond Angles for 14srv042.

Atom	**Atom**	**Atom**	**Angle/˚**		**Atom**	**Atom**	**Atom**	**Angle/˚**
C6	O2	C7	115.30(8)		C3	C2	C6	112.96(8)
C1	N1	C5	126.65(8)		C6	C2	C1	107.54(8)
O1	C1	N1	124.00(9)		C2	C3	C4	110.22(8)
O1	C1	C2	117.79(8)		C5	C4	C3	108.83(8)
N1	C1	C2	118.19(8)		N1	C5	C4	110.97(8)
F1	C2	C1	105.87(7)		O2	C6	C2	109.25(8)
F1	C2	C3	108.60(8)		O3	C6	O2	125.68(9)
F1	C2	C6	106.80(8)		O3	C6	C2	125.08(9)
C3	C2	C1	114.57(8)					

Table 6 Hydrogen Bonds for 14srv042.						
D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1	O1[1]	0.881(16)	1.998(16)	2.8770(11)	175.7(14)

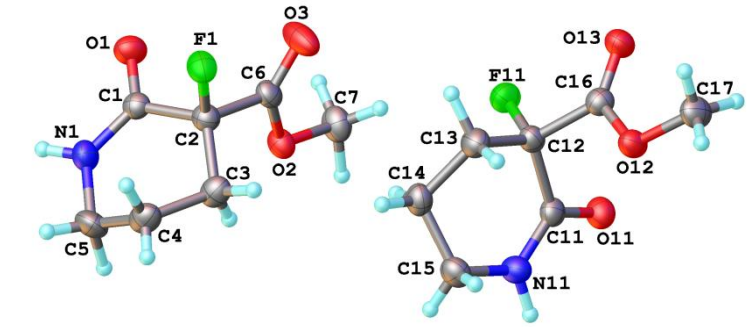
[1]2-X,2-Y,1-Z

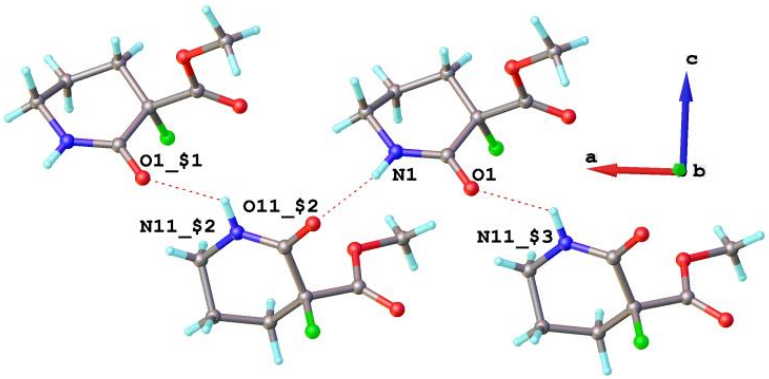
Table 7 Torsion Angles for 14srv042.										
A	**B**	**C**	**D**	**Angle/˚**		**A**	**B**	**C**	**D**	**Angle/˚**
F1	C2	C3	C4	-76.45(10)		C1	C2	C6	O2	63.90(10)
F1	C2	C6	O2	177.17(7)		C1	C2	C6	O3	-116.16(11)
F1	C2	C6	O3	-2.89(13)		C2	C3	C4	C5	-62.56(11)
O1	C1	C2	F1	-70.70(11)		C3	C2	C6	O2	-63.50(10)
O1	C1	C2	C3	169.66(9)		C3	C2	C6	O3	116.43(11)
O1	C1	C2	C6	43.19(11)		C3	C4	C5	N1	52.18(11)
N1	C1	C2	F1	108.25(9)		C5	N1	C1	O1	-179.27(9)
N1	C1	C2	C3	-11.39(12)		C5	N1	C1	C2	1.85(14)
N1	C1	C2	C6	-137.85(9)		C6	C2	C3	C4	165.27(8)
C1	N1	C5	C4	-23.07(14)		C7	O2	C6	O3	3.87(15)
C1	C2	C3	C4	41.65(11)		C7	O2	C6	C2	-176.19(8)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv042.				
Atom	***x***	***y***	***z***	**U(eq)**
H1	9892(14)	8180(20)	5387(15)	28(4)

H3A	6781(13)	6510(20)	5013(15)	23(3)
H3B	6619(14)	6990(20)	6431(16)	31(4)
H4A	8094(13)	4420(20)	6855(15)	25(3)
H4B	8707(13)	6300(20)	7655(16)	27(4)
H5A	8708(13)	5150(20)	5032(15)	23(3)
H5B	9843(14)	5240(20)	6432(16)	26(4)
H7A	4688(14)	11070(20)	2659(15)	30(4)
H7B	5707(15)	12050(30)	2357(17)	37(4)
H7C	5167(15)	9990(20)	1637(17)	33(4)

A3.7. (*S*)-Methyl 3-fluoro-2-oxo-3-piperidinecarboxylate (*S*)-248 (14srv257)





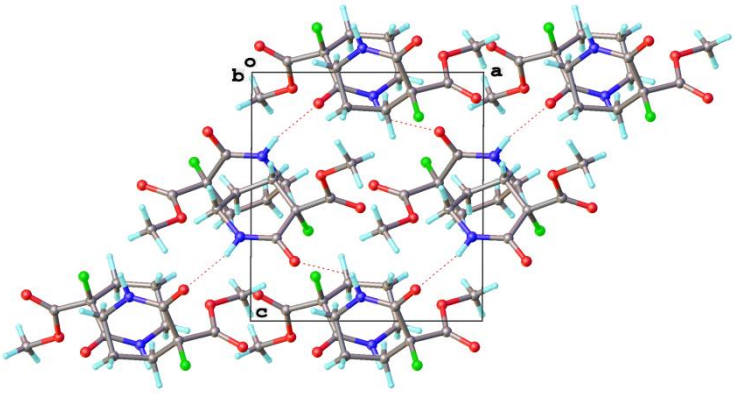

b
o
a
c

Table 1 Crystal data and structure refinement for 14srv257	
Identification code	14srv257
Empirical formula	$C_7H_{10}FNO_3$
Formula weight	175.16
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1$
a/Å	8.5224(4)
b/Å	10.3050(5)
c/Å	9.1442(4)
α/°	90.00
β/°	90.3404(11)
γ/°	90.00
Volume/Å^3	803.06(6)
Z	4
ρ_{calc}g/cm^3	1.449
μ/mm^{-1}	1.105
F(000)	368.0
Crystal size/mm^3	0.53 × 0.35 × 0.29
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	9.68 to 144.92
Index ranges	$-9 \leq h \leq 9$, $-12 \leq k \leq 12$, $-11 \leq l \leq 9$
Reflections collected	7399
Independent reflections	2833 [R_{int} = 0.0612, R_{sigma} = 0.0576]
Data/restraints/parameters	2833/1/297
Goodness-of-fit on F^2	1.005
Final R indexes [I>=2σ (I)]	R_1 = 0.0547, wR_2 = 0.1520
Final R indexes [all data]	R_1 = 0.0547, wR_2 = 0.1520
Largest diff. peak/hole / e Å^{-3}	0.24/-0.24
Flack parameter	0.1(2)
Hooft parameter	0.02(7)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv257. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
F1	7533(2)	4806.2(18)	3623(2)	36.2(4)
O1	8108(3)	7298(2)	2396(2)	37.7(5)
O2	6992(2)	7552(2)	5858(2)	30.9(5)
O3	5292(3)	6351(3)	4548(3)	51.6(7)
N1	10467(3)	6772(3)	3379(3)	36.2(6)
C1	8905(4)	6734(3)	3319(3)	27.9(6)
C2	8069(3)	5869(3)	4447(3)	29.9(6)
C3	9074(4)	5363(4)	5693(4)	35.6(7)
C4	10664(4)	4980(4)	5137(4)	38.8(7)
C5	11465(4)	6158(4)	4497(4)	44.5(9)
C6	6594(3)	6595(3)	4946(3)	29.1(6)
C7	5692(4)	8382(3)	6321(4)	39.3(8)
F11	2797(2)	6620.1(19)	8214.7(19)	37.3(4)
O11	2890(3)	7040(2)	11254(2)	36.4(5)
O12	1743(2)	4135(2)	10648(2)	33.2(5)
O13	326(3)	5222(2)	8952(2)	36.6(5)
N11	5238(3)	6050(3)	10965(3)	34.0(6)
C11	3759(3)	6296(3)	10590(3)	28.4(6)
C12	3111(3)	5620(3)	9204(3)	28.0(6)
C13	4223(4)	4644(4)	8494(4)	36.0(7)
C14	5908(4)	5122(3)	8589(3)	35.3(7)
C15	6367(4)	5246(4)	10182(4)	36.0(7)
C16	1539(4)	4991(3)	9564(3)	30.2(6)
C17	352(4)	3397(4)	11030(4)	38.9(8)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv257. 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}
F1	35.3(10)	33(1)	40.3(9)	-8.5(8)	4.2(7)	-4.8(7)
O1	37.3(12)	39.1(13)	36.6(11)	5.8(10)	-5.3(9)	0.2(10)
O2	25.9(10)	28.6(11)	38.2(11)	-3.6(9)	3.1(8)	1.0(8)
O3	27.9(13)	60.1(17)	66.7(17)	-28.0(14)	-1.7(10)	2.5(11)
N1	29.9(14)	44.1(16)	34.5(13)	3.2(12)	4.3(11)	-3.4(11)
C1	29.4(15)	26.9(14)	27.2(13)	-3.4(12)	-0.1(10)	2.0(11)
C2	28.7(15)	29.8(15)	31.1(15)	-2.2(12)	-0.3(11)	1.4(12)
C3	39.5(18)	34.9(18)	32.5(16)	0.7(14)	1.3(13)	9.5(13)
C4	38.2(18)	44.0(19)	34.0(16)	1.7(16)	-2.7(13)	14.0(15)
C5	24.8(16)	63(2)	45.4(18)	-0.4(18)	-6.9(13)	1.3(15)
C6	24.0(14)	29.7(14)	33.5(14)	2.3(12)	1.2(10)	-3.6(11)
C7	35.3(17)	34.1(17)	49(2)	-3.9(16)	8.3(14)	5.1(13)
F11	37.5(10)	41.5(10)	32.9(9)	8.4(8)	-1.0(7)	-2.8(8)
O11	34.1(11)	37.3(12)	37.7(11)	-5.1(9)	4.0(9)	0.3(9)

O12	29.9(11)	36.2(12)	33.7(11)	6.8(9)	-1.9(8)	-3.3(9)
O13	28.1(12)	44.9(13)	36.8(11)	3.5(10)	-2.3(9)	0.1(9)
N11	30.8(14)	37.7(15)	33.4(14)	-4.4(11)	-3.2(10)	2.9(11)
C11	28.8(14)	27.3(14)	29.1(14)	1.7(11)	3.9(11)	-3.3(11)
C12	25.3(15)	30.1(15)	28.8(14)	3.4(11)	0.6(10)	0.8(11)
C13	34.4(17)	41.6(18)	32.2(15)	-5.1(14)	1.3(11)	1.5(13)
C14	30.9(17)	38.6(17)	36.6(16)	2.2(14)	6.7(12)	3.6(13)
C15	28.9(17)	37.4(19)	41.8(18)	1.4(15)	-1.1(12)	3.9(13)
C16	31.9(15)	28.9(15)	29.9(14)	-0.8(12)	-0.4(11)	-1.4(11)
C17	37.9(19)	37.0(18)	41.8(19)	7.3(15)	-0.2(14)	-9.0(15)

Table 4 Bond Lengths for 14srv257.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C2	1.404(4)		F11	C12	1.396(3)
O1	C1	1.226(4)		O11	C11	1.229(4)
O2	C6	1.333(4)		O12	C16	1.337(4)
O2	C7	1.465(4)		O12	C17	1.454(4)
O3	C6	1.193(4)		O13	C16	1.196(4)
N1	C1	1.333(4)		N11	C11	1.328(4)
N1	C5	1.469(5)		N11	C15	1.461(4)
C1	C2	1.541(4)		C11	C12	1.546(4)
C2	C3	1.514(4)		C12	C13	1.529(4)
C2	C6	1.535(4)		C12	C16	1.526(4)
C3	C4	1.503(5)		C13	C14	1.521(5)
C4	C5	1.513(6)		C14	C15	1.511(5)

Table 5 Bond Angles for 14srv257.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C6	O2	C7	115.0(2)		C16	O12	C17	114.9(2)
C1	N1	C5	126.2(3)		C11	N11	C15	127.4(3)
O1	C1	N1	124.3(3)		O11	C11	N11	124.4(3)
O1	C1	C2	118.6(3)		O11	C11	C12	118.2(3)
N1	C1	C2	117.1(3)		N11	C11	C12	117.4(3)
F1	C2	C1	104.1(2)		F11	C12	C11	105.3(2)
F1	C2	C3	108.4(3)		F11	C12	C13	109.1(2)
F1	C2	C6	106.0(2)		F11	C12	C16	106.7(2)
C3	C2	C1	116.2(3)		C13	C12	C11	115.2(2)
C3	C2	C6	113.8(2)		C16	C12	C11	108.9(2)
C6	C2	C1	107.4(2)		C16	C12	C13	111.1(3)
C4	C3	C2	110.1(3)		C14	C13	C12	110.5(3)
C3	C4	C5	109.2(3)		C15	C14	C13	108.7(3)
N1	C5	C4	110.8(3)		N11	C15	C14	110.7(3)
O2	C6	C2	109.9(2)		O12	C16	C12	109.2(2)
O3	C6	O2	125.4(3)		O13	C16	O12	125.9(3)
O3	C6	C2	124.6(3)		O13	C16	C12	124.9(3)

Table 6 Hydrogen Bonds for 14srv257.						
D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1	O11[1]	0.86(5)	2.08(4)	2.857(4)	150(4)
N11	H11	O1[2]	0.93(5)	2.37(4)	3.051(3)	130(3)

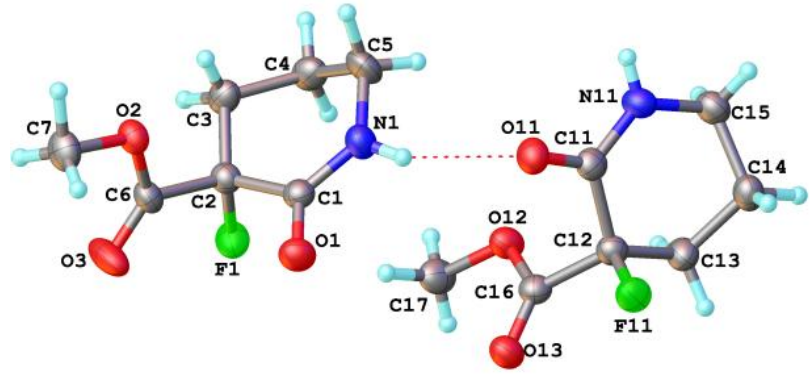
[1]1+X,+Y,-1+Z; [2]+X,+Y,1+Z

Table 7 Torsion Angles for 14srv257.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C2	C3	C4	76.8(3)		F11	C12	C13	C14	80.9(3)
F1	C2	C6	O2	174.0(2)		F11	C12	C16	O12	-172.2(2)
F1	C2	C6	O3	-7.5(4)		F11	C12	C16	O13	8.8(4)
O1	C1	C2	F1	69.8(3)		O11	C11	C12	F11	63.5(3)
O1	C1	C2	C3	-171.0(3)		O11	C11	C12	C13	-176.2(3)
O1	C1	C2	C6	-42.3(3)		O11	C11	C12	C16	-50.7(4)
N1	C1	C2	F1	-107.3(3)		N11	C11	C12	F11	-115.2(3)
N1	C1	C2	C3	11.9(4)		N11	C11	C12	C13	5.1(4)
N1	C1	C2	C6	140.6(3)		N11	C11	C12	C16	130.6(3)
C1	N1	C5	C4	27.5(5)		C11	N11	C15	C14	22.3(5)
C1	C2	C3	C4	-39.9(4)		C11	C12	C13	C14	-37.3(4)
C1	C2	C6	O2	-75.3(3)		C11	C12	C16	O12	-58.9(3)
C1	C2	C6	O3	103.3(4)		C11	C12	C16	O13	122.0(3)
C2	C3	C4	C5	61.3(4)		C12	C13	C14	C15	61.8(4)
C3	C2	C6	O2	54.9(3)		C13	C12	C16	O12	69.0(3)
C3	C2	C6	O3	-126.6(4)		C13	C12	C16	O13	-110.1(4)
C3	C4	C5	N1	-54.4(4)		C13	C14	C15	N11	-53.6(4)
C5	N1	C1	O1	177.4(3)		C15	N11	C11	O11	-175.8(3)
C5	N1	C1	C2	-5.7(5)		C15	N11	C11	C12	2.8(5)
C6	C2	C3	C4	-165.5(3)		C16	C12	C13	C14	-161.7(3)
C7	O2	C6	O3	-3.2(5)		C17	O12	C16	O13	3.2(5)
C7	O2	C6	C2	175.4(2)		C17	O12	C16	C12	-175.8(3)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv257.				
Atom	***x***	***y***	***z***	**U(eq)**
H1	11020(50)	7120(40)	2700(50)	36(10)
H3A	9170(50)	5960(40)	6290(50)	36(10)
H3B	8520(50)	4690(50)	6190(50)	50(12)
H4A	10590(40)	4250(40)	4390(40)	29(9)
H4B	11250(60)	4650(60)	5750(60)	63(14)
H5A	11800(50)	6900(40)	5330(40)	38(10)
H5B	12330(50)	5900(40)	4010(40)	37(10)
H7A	5310(50)	8840(50)	5450(50)	41(10)
H7B	4820(50)	7890(40)	6670(40)	37(10)
H7C	6280(50)	8980(40)	7160(50)	44(11)
H11	5620(50)	6390(50)	11830(50)	44(11)

H13A	4240(40)	3910(40)	9060(40)	29(9)
H13B	3910(50)	4450(50)	7420(50)	48(11)
H14A	6670(50)	4520(50)	8160(50)	52(12)
H14B	6070(40)	6080(30)	8180(40)	22(7)
H15A	7270(50)	5670(40)	10390(40)	40(10)
H15B	6340(40)	4460(40)	10620(40)	25(8)
H17A	-540(60)	3920(60)	11410(60)	62(14)
H17B	800(60)	2740(60)	11570(60)	60(13)
H17C	10(60)	2870(50)	10260(60)	53(13)

A3.8. (*R*)-Methyl 3-fluoro-2-oxo-3-piperidinecarboxylate (*R*)-248 (14srv258)



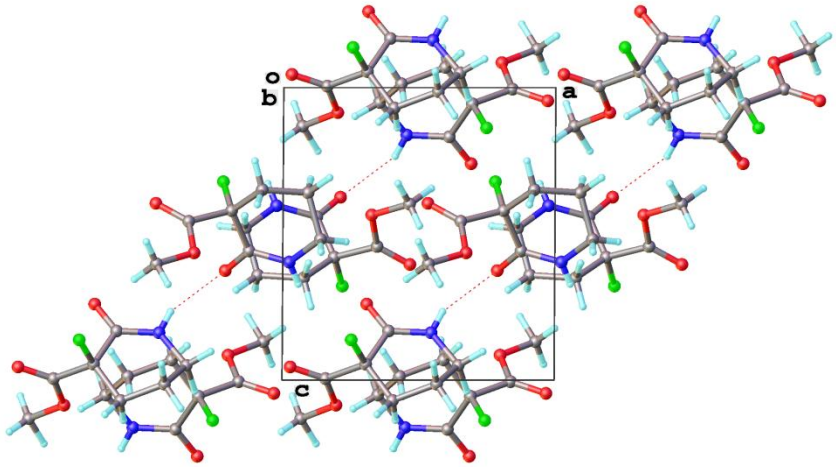


Table 1 Crystal data and structure refinement for 14srv258	
Identification code	14srv258
Empirical formula	$C_7H_{10}FNO_3$
Formula weight	175.16
Temperature/K	120.0
Crystal system	monoclinic

Space group	$P2_1$
a/Å	8.5211(3)
b/Å	10.3039(3)
c/Å	9.1547(3)
α/°	90.00
β/°	90.3650(8)
γ/°	90.00
Volume/$Å^3$	803.77(5)
Z	4
$\rho_{calc}g/cm^3$	1.447
μ/mm^{-1}	1.104
F(000)	368.0
Crystal size/mm^3	0.32 × 0.3 × 0.24
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	9.66 to 144.94
Index ranges	$-9 \leq h \leq 10$, $-12 \leq k \leq 12$, $-11 \leq l \leq 11$
Reflections collected	6957
Independent reflections	2916 [R_{int} = 0.0579, R_{sigma} = 0.0613]
Data/restraints/parameters	2916/1/297
Goodness-of-fit on F^2	1.103
Final R indexes [I>=2σ (I)]	R_1 = 0.0513, wR_2 = 0.1451
Final R indexes [all data]	R_1 = 0.0515, wR_2 = 0.1455
Largest diff. peak/hole / e $Å^{-3}$	0.33/-0.26
Flack parameter	-0.01(16)
Hooft parameter	0.00(7)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv258. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	7463.4(17)	5472.8(15)	1375.6(16)	36.8(4)
O1	6903(2)	2983.0(19)	2600(2)	37.3(4)
O2	8010.5(19)	2734.7(17)	-854.1(19)	31.1(4)
O3	9704(2)	3938(3)	451(3)	52.8(6)
N1	4532(3)	3510(2)	1621(2)	35.8(5)
C1	6094(3)	3546(2)	1680(2)	28.6(5)
C2	6929(3)	4414(2)	549(3)	29.0(5)
C3	5934(3)	4920(3)	-694(3)	34.6(6)
C4	4317(3)	5305(3)	-134(3)	37.9(6)
C5	3524(3)	4125(3)	507(3)	44.0(7)
C6	8403(3)	3682(2)	53(3)	29.9(5)
C7	9305(3)	1902(3)	-1316(3)	39.6(6)
F11	2198.4(18)	3662.2(16)	6783.9(16)	37.5(4)
O11	2110(2)	3242.7(19)	3745(2)	36.0(4)
O12	3261(2)	6149.0(19)	4352.9(19)	34.2(4)
O13	4675(2)	5059.7(19)	6046(2)	35.5(4)
N11	-241(2)	4225(2)	4037(2)	33.8(5)

C11	1242(3)	3988(2)	4416(3)	29.8(5)
C12	1891(3)	4664(2)	5796(3)	28.9(5)
C13	769(3)	5633(3)	6508(3)	35.4(5)
C14	-918(3)	5154(3)	6408(3)	35.4(5)
C15	-1381(3)	5033(3)	4817(3)	36.1(6)
C16	3466(3)	5293(2)	5431(2)	29.7(5)
C17	4646(3)	6888(3)	3979(3)	39.1(6)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv258. 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}**
F1	36.6(8)	33.2(8)	40.6(8)	-8.9(6)	4.4(6)	-3.4(6)
O1	36.4(9)	39.5(10)	35.9(9)	5.0(8)	-4.7(7)	1.1(8)
O2	26.1(8)	28.5(8)	38.8(9)	-2.8(7)	2.7(6)	2.2(7)
O3	25.2(9)	62.1(14)	71.1(15)	-27.7(12)	-6.0(8)	2.6(9)
N1	28(1)	43.6(12)	35.8(11)	3.9(10)	1.9(8)	-0.2(9)
C1	29.3(11)	29.7(11)	26.9(10)	-2.4(9)	-1.8(8)	1.4(9)
C2	26.7(11)	29.5(11)	30.8(11)	-1.4(9)	-0.1(8)	1.2(10)
C3	33.8(13)	39.0(14)	30.8(12)	0.8(11)	1.4(10)	10.4(10)
C4	34.7(13)	43.7(14)	35.2(12)	0.6(12)	-2.7(10)	12.5(11)
C5	22.4(11)	60.0(19)	49.5(15)	1.0(14)	-6.3(10)	3.0(12)
C6	21.6(10)	32.5(11)	35.6(11)	1.9(10)	1.0(8)	-0.4(9)
C7	34.6(13)	34.4(13)	49.9(15)	-3.8(13)	7.7(11)	7.4(11)
F11	37.6(7)	40.8(8)	34.2(7)	10.3(6)	-1.2(6)	-1.5(6)
O11	33.9(9)	36.4(9)	37.9(9)	-4.6(7)	4.5(7)	1.5(7)
O12	31.7(9)	37.0(9)	33.9(9)	6.7(7)	-3.3(7)	-4.5(7)
O13	29.2(9)	40(1)	37.4(9)	4.2(8)	-2.8(7)	-1.3(7)
N11	30.4(10)	37.1(12)	33.8(11)	-3.0(9)	-5.2(8)	0.9(9)
C11	30.0(11)	27.9(11)	31.5(11)	1.6(9)	1.8(9)	-3.9(9)
C12	28.5(12)	29.5(12)	28.6(11)	2.6(9)	-2.3(8)	0.5(9)
C13	31.9(12)	40.6(14)	33.8(12)	-6.9(11)	1.7(9)	-1(1)
C14	30.7(12)	39.1(14)	36.5(12)	3.2(11)	4.1(9)	3.8(10)
C15	29.5(12)	39.6(14)	39.3(13)	0.3(11)	-3.0(9)	3.0(11)
C16	31.0(11)	29.4(11)	28.7(10)	-2.4(9)	-2.6(8)	-1.3(9)
C17	39.8(14)	38.5(14)	39.1(14)	6.3(12)	-1.5(11)	-11.9(12)

Table 4 Bond Lengths for 14srv258.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C2	1.402(3)		F11	C12	1.396(3)
O1	C1	1.230(3)		O11	C11	1.234(3)
O2	C6	1.323(3)		O12	C16	1.334(3)
O2	C7	1.462(3)		O12	C17	1.447(3)
O3	C6	1.195(3)		O13	C16	1.195(3)
N1	C1	1.332(3)		N11	C11	1.331(3)

N1	C5	1.472(4)		N11	C15	1.468(3)
C1	C2	1.545(3)		C11	C12	1.542(3)
C2	C3	1.507(3)		C12	C13	1.530(3)
C2	C6	1.536(3)		C12	C16	1.529(3)
C3	C4	1.525(4)		C13	C14	1.522(4)
C4	C5	1.512(4)		C14	C15	1.512(4)

Table 5 Bond Angles for 14srv258.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C6	O2	C7	115.23(19)		C16	O12	C17	114.81(19)
C1	N1	C5	126.5(2)		C11	N11	C15	127.4(2)
O1	C1	N1	124.8(2)		O11	C11	N11	123.7(2)
O1	C1	C2	118.3(2)		O11	C11	C12	118.5(2)
N1	C1	C2	116.9(2)		N11	C11	C12	117.8(2)
F1	C2	C1	103.74(18)		F11	C12	C11	105.15(19)
F1	C2	C3	108.5(2)		F11	C12	C13	108.76(19)
F1	C2	C6	106.14(18)		F11	C12	C16	107.08(18)
C3	C2	C1	116.5(2)		C13	C12	C11	114.9(2)
C3	C2	C6	113.84(19)		C16	C12	C11	108.84(19)
C6	C2	C1	107.14(19)		C16	C12	C13	111.6(2)
C2	C3	C4	109.9(2)		C14	C13	C12	110.8(2)
C5	C4	C3	109.2(2)		C15	C14	C13	109.0(2)
N1	C5	C4	110.7(2)		N11	C15	C14	110.3(2)
O2	C6	C2	110.10(19)		O12	C16	C12	109.32(19)
O3	C6	O2	125.8(2)		O13	C16	O12	126.1(2)
O3	C6	C2	124.1(2)		O13	C16	C12	124.5(2)

Table 6 Hydrogen Bonds for 14srv258.

D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1	O11	0.82(4)	2.14(4)	2.858(3)	145(3)
N11	H11	O1[1]	0.79(4)	2.46(4)	3.042(3)	132(3)

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

Table 7 Torsion Angles for 14srv258.

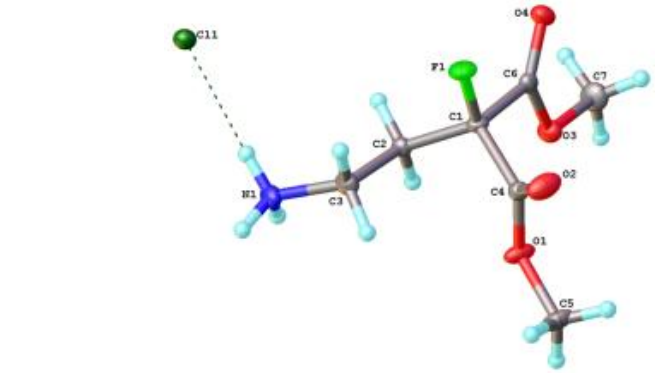
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C2	C3	C4	-77.0(3)		F11	C12	C13	C14	-80.8(2)
F1	C2	C6	O2	-174.02(19)		F11	C12	C16	O12	172.25(19)
F1	C2	C6	O3	6.6(3)		F11	C12	C16	O13	-9.0(3)
O1	C1	C2	F1	-69.9(3)		O11	C11	C12	F11	-63.9(3)
O1	C1	C2	C3	170.9(2)		O11	C11	C12	C13	176.5(2)
O1	C1	C2	C6	42.1(3)		O11	C11	C12	C16	50.5(3)
N1	C1	C2	F1	107.3(2)		N11	C11	C12	F11	114.7(2)
N1	C1	C2	C3	-11.9(3)		N11	C11	C12	C13	-4.9(3)

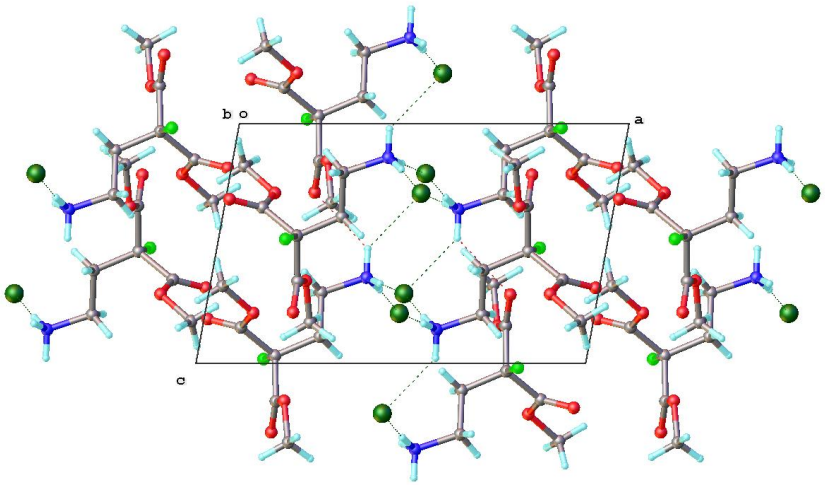
N1	C1	C2	C6	-140.7(2)		N11	C11	C12	C16	-130.9(2)
C1	N1	C5	C4	-27.6(4)		C11	N11	C15	C14	-22.7(4)
C1	C2	C3	C4	39.6(3)		C11	C12	C13	C14	36.7(3)
C1	C2	C6	O2	75.6(2)		C11	C12	C16	O12	59.1(3)
C1	C2	C6	O3	-103.8(3)		C11	C12	C16	O13	-122.2(3)
C2	C3	C4	C5	-60.9(3)		C12	C13	C14	C15	-61.7(3)
C3	C2	C6	O2	-54.7(3)		C13	C12	C16	O12	-68.8(2)
C3	C2	C6	O3	125.9(3)		C13	C12	C16	O13	109.9(3)
C3	C4	C5	N1	54.0(3)		C13	C14	C15	N11	53.6(3)
C5	N1	C1	O1	-177.3(3)		C15	N11	C11	O11	176.1(2)
C5	N1	C1	C2	5.7(4)		C15	N11	C11	C12	-2.4(4)
C6	C2	C3	C4	165.1(2)		C16	C12	C13	C14	161.3(2)
C7	O2	C6	O3	3.9(4)		C17	O12	C16	O13	-3.3(4)
C7	O2	C6	C2	-175.51(19)		C17	O12	C16	C12	175.4(2)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv258.

Atom	***x***	***y***	***z***	**U(eq)**
H1	4110(40)	3190(40)	2340(40)	36(8)
H3A	5820(50)	4320(40)	-1370(40)	52(10)
H3B	6500(50)	5630(40)	-1170(40)	51(10)
H4A	4440(40)	5990(30)	550(40)	31(7)
H4B	3710(30)	5600(30)	-970(30)	19(6)
H5A	2630(40)	4440(40)	980(40)	38(8)
H5B	3210(40)	3440(40)	-370(40)	46(9)
H7A	8740(40)	1370(30)	-2170(40)	35(8)
H7B	9610(50)	1390(50)	-550(50)	56(11)
H7C	10010(50)	2440(40)	-1770(40)	47(9)
H11	-570(40)	3960(40)	3290(40)	45(9)
H13A	950(40)	6470(40)	6000(40)	36(8)
H13B	1040(50)	5840(40)	7490(50)	57(11)
H14A	-1700(40)	5760(40)	6920(40)	40(8)
H14B	-1010(40)	4330(40)	6870(40)	38(8)
H15A	-1410(30)	5910(30)	4380(30)	19(6)
H15B	-2310(40)	4610(40)	4730(40)	37(8)
H17A	4220(40)	7420(50)	3280(40)	50(10)
H17B	5440(60)	6250(60)	3450(60)	83(15)
H17C	5020(40)	7260(40)	4720(40)	39(9)

A3.9. Dimethyl 2-(2-aminoethyl)-2-fluoromalonate, hydrochloride 257 (14srv104)





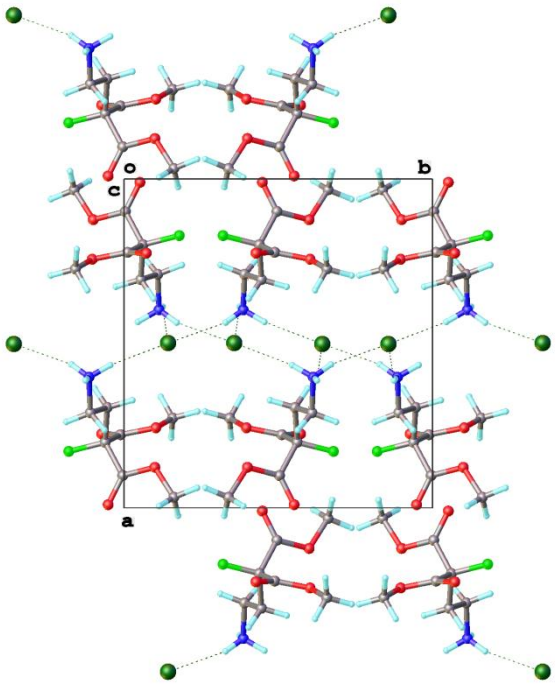


Table 1 Crystal data and structure refinement for 14srv104.	
Identification code	14srv104
Empirical formula	$C_7H_{13}NO_4FCl$
Formula weight	229.63
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.2350(7)
b/Å	11.3068(7)
c/Å	7.6415(4)
α/°	90.00
β/°	100.2891(17)
γ/°	90.00
Volume/Å^3	1040.12(10)
Z	4
ρ_{calc}mg/mm^3	1.466
m/mm^{-1}	0.372
F(000)	480.0
Crystal size/mm^3	0.23 × 0.22 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	4.94 to 60°
Index ranges	$-17 \leq h \leq 17$, $-15 \leq k \leq 15$, $-10 \leq l \leq 10$
Reflections collected	21745
Independent reflections	3038 [R_{int} = 0.0617, R_{sigma} = 0.0295]
Data/restraints/parameters	3038/0/179
Goodness-of-fit on F^2	1.097
Final R indexes [I>=2σ (I)]	R_1 = 0.0341, wR_2 = 0.0846
Final R indexes [all data]	R_1 = 0.0384, wR_2 = 0.0873
Largest diff. peak/hole / e Å^{-3}	0.59/-0.24

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14srv104. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
Cl1	5014.76(19)	8572.1(2)	2087.3(3)	17.50(9)
F1	8283.4(5)	6790.9(5)	181.8(8)	18.78(14)
O1	8765.9(6)	3999.8(7)	2109.7(11)	21.17(17)
O2	9902.8(7)	5514.5(7)	1847.1(12)	26.71(19)
O3	7564.1(6)	3968.7(7)	-1557.2(9)	17.85(16)
O4	7748.1(6)	5724.2(7)	-2865.8(10)	19.28(16)
N1	6023.8(9)	6134.4(9)	3582.4(13)	20.04(19)
C1	8005.1(8)	5614.2(8)	328.9(12)	13.01(18)
C2	6946.7(8)	5543.1(9)	1118.3(12)	13.87(18)
C3	7107.9(9)	6094.2(10)	2960.1(13)	18.8(2)
C4	9016.8(8)	5042.2(9)	1504.5(12)	13.93(18)
C5	9677.7(10)	3385.2(11)	3231.3(18)	25.7(2)
C6	7774.2(8)	5115.1(9)	-1573.6(13)	13.72(18)

C7	7268.4(11)	3423.4(11)	-3298.1(16)	25.1(2)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 14srv104. 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}
Cl1	18.23(13)	19.42(14)	14.70(13)	2.47(8)	2.56(9)	1.98(8)
F1	23.3(3)	11.4(3)	20.4(3)	3.3(2)	0.3(2)	-2.7(2)
O1	16.0(3)	16.7(4)	28.0(4)	9.3(3)	-3.6(3)	-0.9(3)
O2	15.9(4)	20.8(4)	40.1(5)	6.1(3)	-4.1(3)	-3.8(3)
O3	22.3(4)	16.6(3)	13.8(3)	-2.5(3)	0.9(3)	-1.1(3)
O4	19.3(4)	24.4(4)	14.0(3)	4.5(3)	2.8(3)	1.7(3)
N1	29.1(5)	15.9(4)	17.2(4)	-0.2(3)	9.6(4)	3.1(4)
C1	15.0(4)	10.6(4)	12.8(4)	1.5(3)	0.5(3)	-1.0(3)
C2	15.4(4)	15.1(4)	10.8(4)	-0.6(3)	1.3(3)	0.9(3)
C3	22.3(5)	20.4(5)	13.2(4)	-3.3(4)	2.2(4)	-0.5(4)
C4	15.0(4)	13.6(4)	12.9(4)	0.0(3)	1.5(3)	0.5(3)
C5	21.4(5)	20.1(5)	31.9(6)	9.7(5)	-5.5(5)	3.7(4)
C6	10.5(4)	17.0(4)	13.5(4)	0.1(3)	1.7(3)	1.3(3)
C7	30.1(6)	25.7(6)	17.7(5)	-7.7(4)	-0.5(4)	-0.1(4)

Table 4 Bond Lengths for 14srv104.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
F1	C1	1.3829(11)		O4	C6	1.1996(12)
O1	C4	1.3222(12)		N1	C3	1.4877(14)
O1	C5	1.4562(13)		C1	C2	1.5252(13)
O2	C4	1.1948(12)		C1	C4	1.5362(13)
O3	C6	1.3219(12)		C1	C6	1.5379(13)
O3	C7	1.4522(13)		C2	C3	1.5194(13)

Table 5 Bond Angles for 14srv104.

Atom	Atom	Atom	Angle/°		Atom	Atom	Atom	Angle/°
C4	O1	C5	115.46(8)		C3	C2	C1	111.46(8)
C6	O3	C7	115.13(8)		N1	C3	C2	109.45(9)
F1	C1	C2	108.76(8)		O1	C4	C1	111.51(8)
F1	C1	C4	105.56(7)		O2	C4	O1	125.46(9)
F1	C1	C6	106.41(7)		O2	C4	C1	123.01(9)
C2	C1	C4	113.51(8)		O3	C6	C1	110.73(8)
C2	C1	C6	109.20(7)		O4	C6	O3	126.45(9)
C4	C1	C6	113.02(8)		O4	C6	C1	122.74(9)

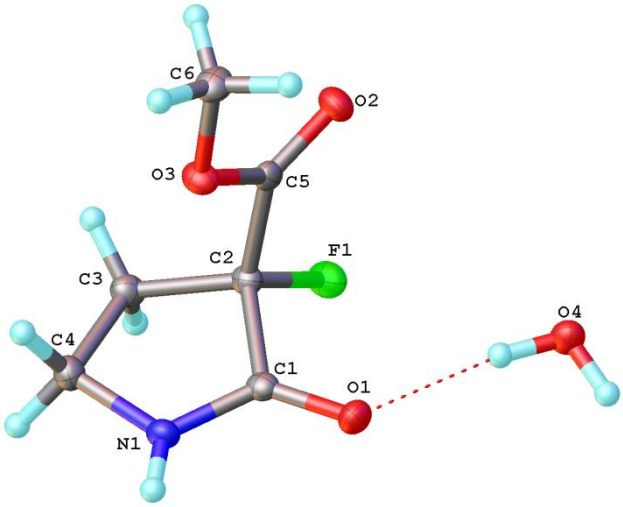
Table 6 Hydrogen Bonds for 14srv104.						
D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1A	Cl1[1]	0.907(17)	2.421(17)	3.1605(9)	138.9(13)
N1	H1A	O4[2]	0.907(17)	2.506(16)	3.1602(13)	129.3(13)
N1	H1B	Cl1[3]	0.899(16)	2.278(16)	3.1682(10)	170.8(13)
N1	H1C	Cl1	0.850(18)	2.324(18)	3.1499(10)	164.0(15)

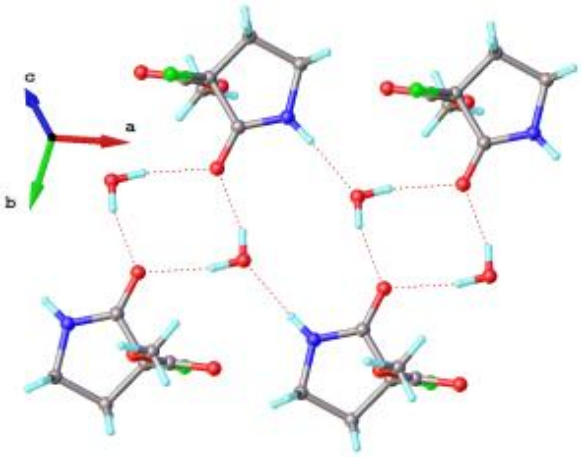
[1]+X,3/2-Y,1/2+Z; [2]+X,+Y,1+Z; [3]1-X,-1/2+Y,1/2-Z

Table 7 Torsion Angles for 14srv104.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C1	C2	C3	-60.80(10)		C4	C1	C2	C3	56.37(11)
F1	C1	C4	O1	164.51(8)		C4	C1	C6	O3	60.87(10)
F1	C1	C4	O2	-13.61(13)		C4	C1	C6	O4	-122.13(10)
F1	C1	C6	O3	176.27(7)		C5	O1	C4	O2	-2.31(16)
F1	C1	C6	O4	-6.73(12)		C5	O1	C4	C1	179.63(9)
C1	C2	C3	N1	173.26(8)		C6	C1	C2	C3	-176.53(8)
C2	C1	C4	O1	45.49(11)		C6	C1	C4	O1	-79.59(10)
C2	C1	C4	O2	-132.63(10)		C6	C1	C4	O2	102.30(11)
C2	C1	C6	O3	-66.50(10)		C7	O3	C6	O4	-0.41(14)
C2	C1	C6	O4	110.50(10)		C7	O3	C6	C1	176.45(9)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv104.				
Atom	***x***	***y***	***z***	**U(eq)**
H1A	6123(13)	6254(14)	4770(20)	30(4)
H1B	5657(13)	5449(14)	3330(20)	26(4)
H1C	5637(14)	6708(15)	3090(20)	32(4)
H2A	6726(11)	4728(12)	1148(17)	16(3)
H2B	6366(11)	5959(13)	339(18)	17(3)
H3A	7387(12)	6874(14)	2945(19)	24(4)
H3B	7635(12)	5633(13)	3807(19)	21(3)
H5A	9390(13)	2630(15)	3510(20)	32(4)
H5B	9948(16)	3867(17)	4270(20)	47(5)
H5C	10238(17)	3250(18)	2630(30)	48(5)
H7A	6624(14)	3808(14)	-3970(20)	30(4)
H7B	7068(14)	2623(15)	-3080(20)	33(4)
H7C	7868(15)	3470(14)	-3900(20)	32(4)

A3.10. Methyl 3-fluoro-2-oxo-3-pyrrolidinecarboxylate 250 (14srv109)





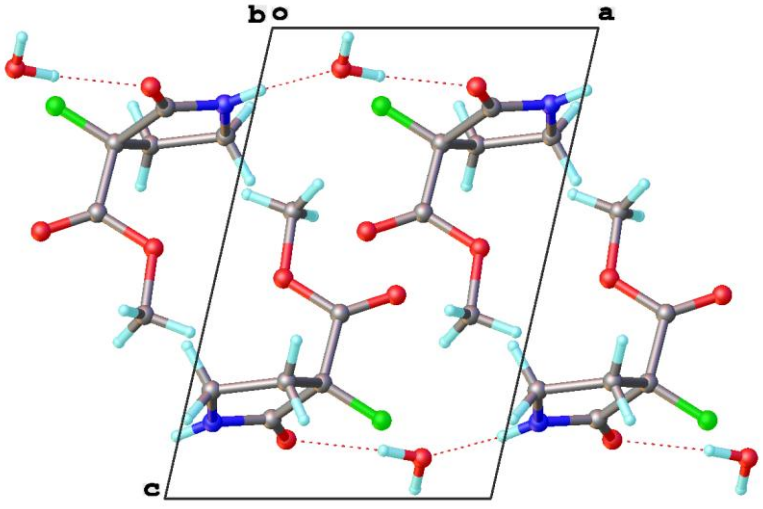


Table 1 Crystal data and structure refinement for 14srv109.	
Identification code	14srv109
Empirical formula	$C_6H_8FNO_3$ x H_2O
Formula weight	179.15

Temperature/K	120.0
Crystal system	triclinic
Space group	P-1
a/Å	6.5648(7)
b/Å	6.6759(7)
c/Å	9.6206(10)
α/°	98.163(3)
β/°	100.929(3)
γ/°	101.047(3)
Volume/Å^3	399.30(7)
Z	2
ρ_{calc}mg/mm^3	1.490
m/mm^{-1}	0.138
F(000)	188.0
Crystal size/mm^3	0.56 × 0.25 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	6.32 to 60°
Index ranges	$-9 \le h \le 9$, $-9 \le k \le 9$, $-13 \le l \le 13$
Reflections collected	6012
Independent reflections	2317 [R_{int} = 0.0666, R_{sigma} = 0.0681]
Data/restraints/parameters	2317/0/149
Goodness-of-fit on F^2	1.012
Final R indexes [I>=2σ (I)]	R_1 = 0.0548, wR_2 = 0.1404
Final R indexes [all data]	R_1 = 0.0671, wR_2 = 0.1501
Largest diff. peak/hole / e Å^{-3}	0.54/-0.28

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14srv109. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	3897.9(12)	5022.3(13)	1661.2(9)	20.8(2)
O1	6681.7(16)	8586.1(15)	1252.1(11)	21.0(3)
O2	4327.5(16)	6939.9(15)	4322.1(11)	20.9(3)
O3	7902.4(15)	7568.4(15)	4678.6(10)	18.5(2)
N1	9069.4(19)	6496.0(18)	1623.7(13)	16.7(3)
C1	7281(2)	7094(2)	1688.1(14)	15.1(3)
C2	5981(2)	5627(2)	2455.0(14)	15.0(3)
C3	7081(2)	3817(2)	2486.8(16)	18.0(3)
C4	9326(2)	4703(2)	2298.6(16)	18.4(3)
C5	5925(2)	6777.9(19)	3927.3(14)	14.6(3)
C6	8084(3)	8764(2)	6098.9(16)	21.0(3)
O4	2396.7(17)	9117.6(17)	860.2(13)	21.8(3)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for 14srv109. 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}
F1	13.0(4)	27.1(5)	18.5(4)	1.1(3)	0.7(3)	0.2(3)
O1	23.7(6)	22.7(5)	21.4(5)	10.6(4)	7.3(4)	9.4(4)
O2	18.1(5)	25.8(5)	21.3(5)	3.6(4)	7.7(4)	8.3(4)
O3	17.1(5)	21.2(5)	15.1(5)	-0.9(4)	2.9(4)	3.1(4)
N1	14.6(6)	20.0(6)	16.8(6)	5.1(4)	6.0(4)	3.4(4)
C1	15.0(6)	17.8(6)	11.5(6)	1.3(5)	2.3(5)	2.7(5)
C2	12.7(6)	17.4(6)	13.8(6)	2.2(5)	2.1(5)	2.1(4)
C3	20.2(7)	15.7(6)	19.0(7)	2.7(5)	6.5(5)	4.3(5)
C4	17.8(7)	19.1(6)	19.5(7)	3.7(5)	4.5(5)	6.4(5)
C5	17.2(6)	13.0(6)	15.6(6)	5.4(5)	5.0(5)	4.9(5)
C6	26.3(8)	19.7(7)	15.0(7)	-0.1(5)	1.9(6)	5.6(6)
O4	18.8(5)	26.2(5)	24.5(6)	12.1(4)	8.2(4)	6.4(4)

Table 4 Bond Lengths for 14srv109.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
F1	C2	1.3858(15)		N1	C4	1.4626(18)
O1	C1	1.2369(16)		C1	C2	1.5348(19)
O2	C5	1.1995(16)		C2	C3	1.5230(18)
O3	C5	1.3264(16)		C2	C5	1.5228(18)
O3	C6	1.4528(17)		C3	C4	1.534(2)
N1	C1	1.3196(17)				

Table 5 Bond Angles for 14srv109.

Atom	Atom	Atom	Angle/°		Atom	Atom	Atom	Angle/°
C5	O3	C6	115.04(11)		C3	C2	C1	103.90(10)
C1	N1	C4	114.51(12)		C5	C2	C1	109.85(10)
O1	C1	N1	128.40(13)		C5	C2	C3	115.00(11)
O1	C1	C2	123.06(12)		C2	C3	C4	104.54(11)
N1	C1	C2	108.53(11)		N1	C4	C3	103.39(11)
F1	C2	C1	108.65(10)		O2	C5	O3	126.21(12)
F1	C2	C3	112.29(10)		O2	C5	C2	124.53(12)
F1	C2	C5	107.01(10)		O3	C5	C2	109.25(11)

Table 6 Hydrogen Bonds for 14srv109.

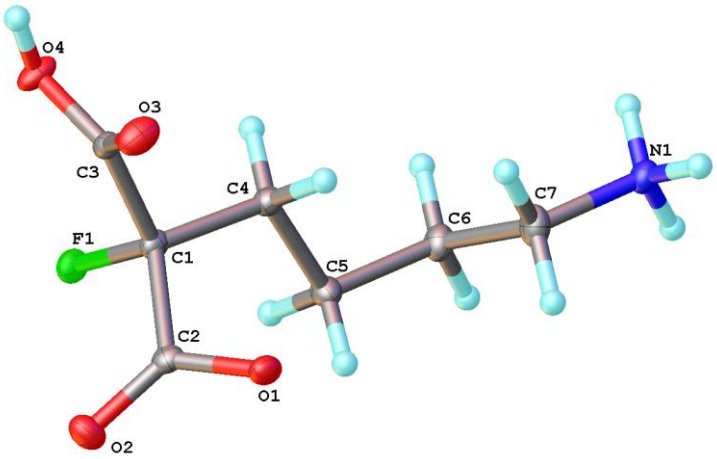
D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N1	H1	O4[1]	0.85(2)	1.96(2)	2.8076(16)	171.8(17)
O4	H4C	O1	0.86(2)	2.02(2)	2.8635(15)	170(2)
O4	H4D	O1[2]	0.84(3)	1.99(3)	2.8002(15)	162(2)

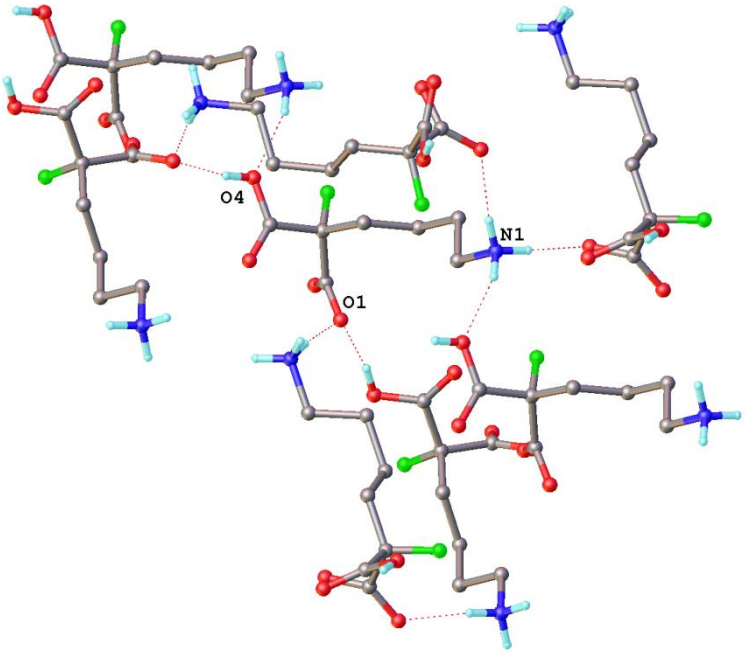
[1]1+X,+Y,+Z; [2]1-X,2-Y,-Z

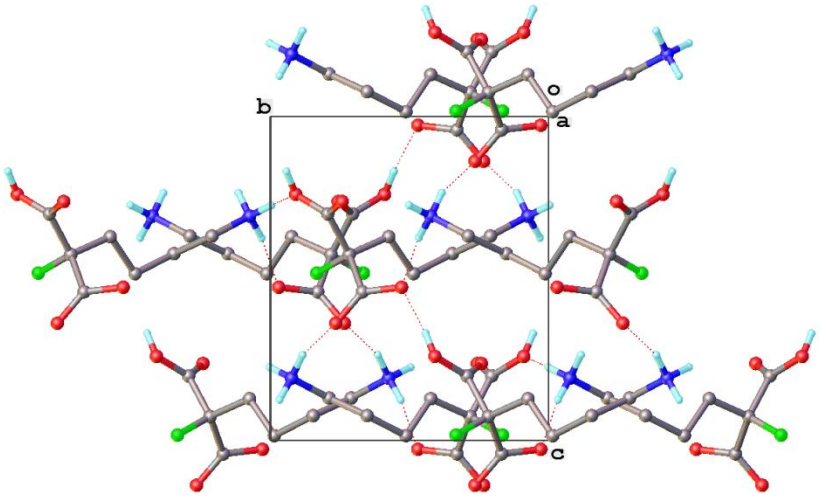
Table 7 Selected Torsion Angles for 14srv109.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
O1	C1	C2	F1	48.39(16)		C3	C2	C1	N1	-13.13(14)
O2	C5	C2	F1	5.66(17)		C3	C2	C5	O2	-119.81(14)
O3	C5	C2	F1	-173.84(9)		C3	C2	C5	O3	60.68(14)
N1	C1	C2	F1	-132.86(11)		C3	C4	N1	C1	14.78(15)
C1	C2	C3	C4	21.19(13)		C4	N1	C1	O1	177.53(13)
C1	C2	C5	O2	123.44(13)		C4	C3	C2	F1	138.42(11)
C1	C2	C5	O3	-56.07(13)		C4	C3	C2	C5	-98.90(13)
C2	C1	N1	C4	-1.14(15)		C5	C2	C1	O1	-68.36(15)
C2	C3	C4	N1	-21.72(14)		C5	C2	C1	N1	110.39(12)
C3	C2	C1	O1	168.12(12)						

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14srv109.				
Atom	***x***	***y***	***z***	**U(eq)**
H1	10070(30)	7190(30)	1320(20)	28(5)
H3A	7070(30)	3300(30)	3350(20)	36(5)
H3B	6280(30)	2760(30)	1670(20)	26(5)
H4A	10380(30)	5100(30)	3240(20)	23(4)
H4B	9870(30)	3690(30)	1690(20)	28(5)
H6A	7500(40)	7930(30)	6680(20)	36(5)
H6B	9600(30)	9210(30)	6460(20)	23(4)
H6C	7440(30)	9940(30)	6000(20)	26(5)
H4C	3620(40)	8820(30)	1020(30)	40(6)
H4D	2390(40)	9810(30)	200(30)	41(6)

A3.11. Dimethyl 2-(4-aminobutyl)-2-fluoromalonate hydrochloride 259 (16srv100)




O4
N1
O1


b
o
a
c

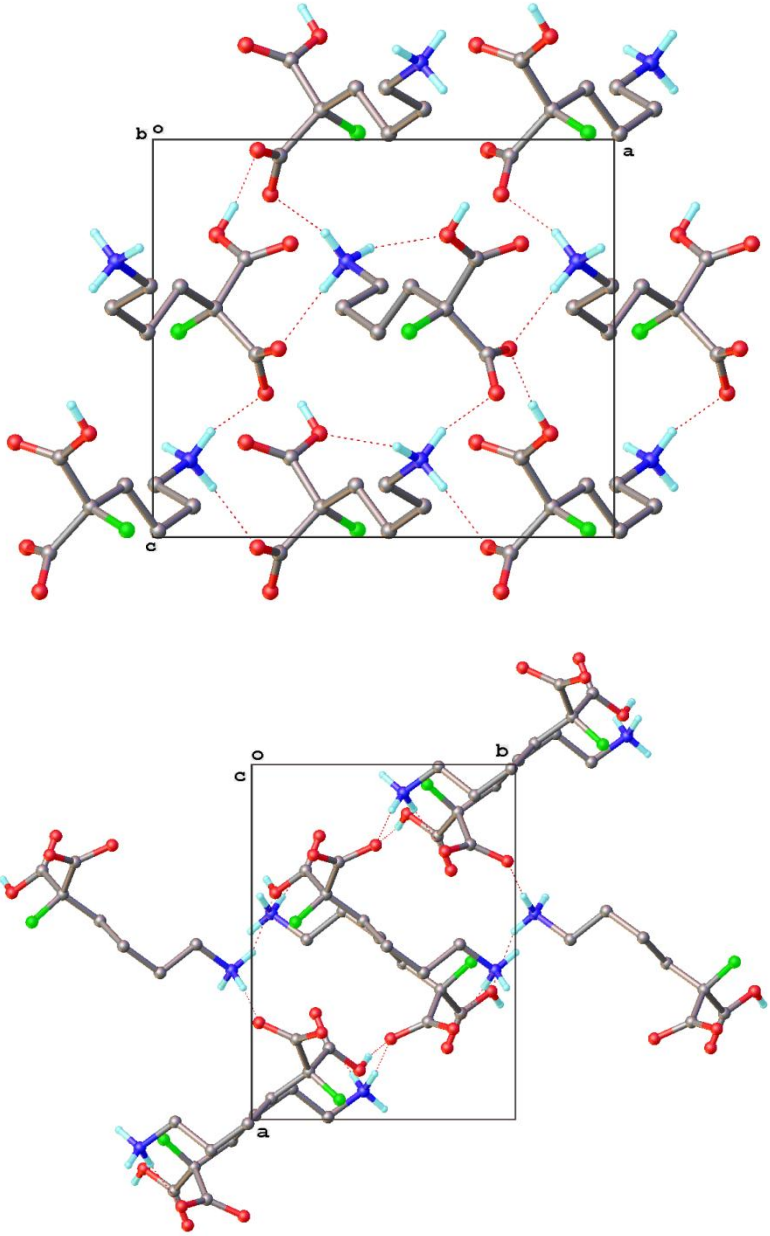


Table 1 Crystal data and structure refinement for 16srv100.	
Identification code	16srv100
Empirical formula	$C_7H_{12}FNO_4$
Formula weight	193.18
Temperature/K	120.0
Crystal system	orthorhombic
Space group	$Pna2_1$
a/Å	11.0907(3)
b/Å	8.2238(2)
c/Å	9.5589(3)

α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	871.85(4)
Z	4
ρ_{calc}g/cm^3	1.472
μ/mm^{-1}	0.133
F(000)	408.0
Crystal size/mm^3	0.37 × 0.25 × 0.08
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.16 to 60
Index ranges	-15 ≤ h ≤ 15, -11 ≤ k ≤ 11, -13 ≤ l ≤ 13
Reflections collected	13238
Independent reflections	2539 [R_{int} = 0.0469, R_{sigma} = 0.0356]
Data/restraints/parameters	2539/1/120
Goodness-of-fit on F^2	1.036
Final R indexes [I>=2σ (I)]	R_1 = 0.0339, wR_2 = 0.0781
Final R indexes [all data]	R_1 = 0.0400, wR_2 = 0.0823
Largest diff. peak/hole / e Å^{-3}	0.32/-0.17
Flack parameter	0.1(6)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16srv100. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	5561.4(7)	8337.8(10)	4829.6(8)	15.31(18)
O1	7725.1(9)	5292.0(12)	5267.1(11)	15.4(2)
O2	7448.9(10)	7655.7(13)	6378.6(12)	18.4(2)
O3	7980.7(10)	7428.1(13)	2622.6(11)	20.1(2)
O4	6387.1(9)	9095.5(12)	2401.3(11)	17.7(2)
N1	4201.9(12)	755.0(14)	3069.3(13)	15.0(2)
C1	6345.5(13)	7173.9(15)	4253.8(14)	12.1(3)
C2	7251.5(13)	6698.3(17)	5408.4(14)	12.6(3)
C3	7009.8(13)	7943.5(16)	3002.9(14)	12.4(3)
C4	5597.4(13)	5755.6(17)	3707.4(15)	13.3(3)
C5	4881.6(14)	4864.4(17)	4846.5(15)	15.5(3)
C6	4131.5(14)	3469.1(17)	4230.2(15)	16.4(3)

C7	4931.7(14)	2103.3(17)	3686.7(17)	17.9(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16srv100. 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}
F1	16.1(4)	13.3(4)	16.5(4)	0.3(3)	2.2(3)	4.6(3)
O1	18.5(5)	14.5(4)	13.2(4)	1.1(4)	-1.8(4)	4.3(4)
O2	18.5(5)	20.0(5)	16.7(5)	-4.4(4)	-2.7(4)	1.7(4)
O3	16.5(5)	22.3(5)	21.4(5)	5.6(4)	5.0(4)	4.8(4)
O4	17.3(5)	18.1(5)	17.9(5)	9.0(4)	5.4(4)	3.9(4)
N1	16.3(6)	12.2(5)	16.5(5)	1.6(5)	1.2(5)	-0.3(4)
C1	12.6(7)	11.0(6)	12.7(6)	0.7(5)	1.3(5)	2.5(5)
C2	11.6(6)	14.2(6)	12.0(6)	2.9(5)	1.8(5)	-1.1(5)
C3	14.7(7)	11.2(5)	11.4(6)	1.4(5)	-0.8(5)	-2.1(5)
C4	13.6(7)	13.4(6)	12.9(6)	1.6(5)	0.2(5)	-0.7(5)
C5	18.1(7)	15.2(6)	13.2(6)	-0.1(5)	1.6(6)	-3.3(5)
C6	16.1(7)	13.5(6)	19.6(7)	0.1(5)	3.2(5)	-1.8(5)
C7	14.1(7)	14.3(6)	25.4(7)	-1.2(6)	1.1(6)	-2.1(5)

Table 4 Bond Lengths for 16srv100.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
F1	C1	1.4055(15)		C1	C2	1.543(2)
O1	C2	1.2774(17)		C1	C3	1.5405(19)
O2	C2	1.2361(18)		C1	C4	1.5236(19)
O3	C3	1.2129(17)		C4	C5	1.534(2)
O4	C3	1.3058(16)		C5	C6	1.535(2)
N1	C7	1.4942(18)		C6	C7	1.523(2)

Table 5 Bond Angles for 16srv100.

Atom	Atom	Atom	Angle/°		Atom	Atom	Atom	Angle/°
F1	C1	C2	107.18(10)		O2	C2	C1	119.38(12)
F1	C1	C3	108.67(10)		O3	C3	O4	126.23(13)
F1	C1	C4	108.58(11)		O3	C3	C1	120.90(12)
C3	C1	C2	110.36(11)		O4	C3	C1	112.77(12)
C4	C1	C2	113.94(11)		C1	C4	C5	113.85(12)
C4	C1	C3	107.99(11)		C4	C5	C6	111.42(12)
O1	C2	C1	114.93(12)		C7	C6	C5	111.50(13)
O2	C2	O1	125.68(13)		N1	C7	C6	111.49(12)

Table 6 Hydrogen Bonds for 16srv100.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O4	H4	O1[1]	0.84	1.64	2.4697(14)	169.8
N1	H1A	O2[2]	0.91	1.89	2.7699(17)	160.9
N1	H1B	O1[3]	0.91	1.90	2.7996(16)	171.9
N1	H1C	O4[4]	0.91	2.03	2.8538(17)	150.5

[1]3/2-X,1/2+Y,-1/2+Z; [2]1-X,1-Y,-1/2+Z; [3]-1/2+X,1/2-Y,+Z; [4]+X,-1+Y,+Z

Table 7 Torsion Angles for 16srv100.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C1	C2	O1	155.43(11)		C3	C1	C2	O1	-86.40(14)
F1	C1	C2	O2	-23.59(17)		C3	C1	C2	O2	94.58(15)
F1	C1	C3	O3	155.59(13)		C3	C1	C4	C5	179.94(11)
F1	C1	C3	O4	-27.80(15)		C4	C1	C2	O1	35.29(17)
F1	C1	C4	C5	-62.40(14)		C4	C1	C2	O2	-143.74(14)
C1	C4	C5	C6	179.06(12)		C4	C1	C3	O3	-86.80(16)
C2	C1	C3	O3	38.34(17)		C4	C1	C3	O4	89.80(14)
C2	C1	C3	O4	-145.06(12)		C4	C5	C6	C7	67.95(15)
C2	C1	C4	C5	56.95(16)		C5	C6	C7	N1	-179.24(12)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16srv100.				
Atom	***x***	***y***	***z***	**U(eq)**
H4	6759	9430	1692	27
H1A	3743	1150	2357	18
H1B	3714	324	3739	18
H1C	4704	-30	2736	18
H4A	6142	4969	3242	16
H4B	5027	6168	2994	16
H5A	4339	5644	5322	19
H5B	5447	4424	5553	19
H6A	3585	3035	4959	20
H6B	3629	3890	3455	20
H7A	5486	2540	2967	22
H7B	5426	1672	4465	22

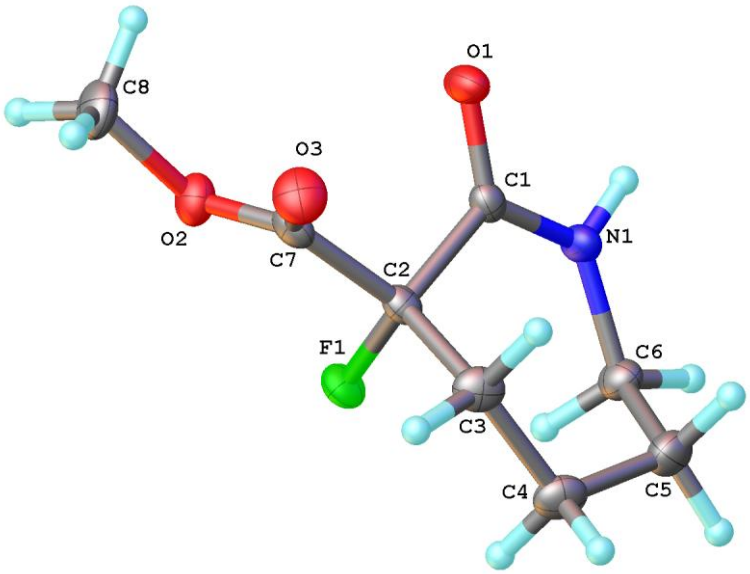
Refinement model description

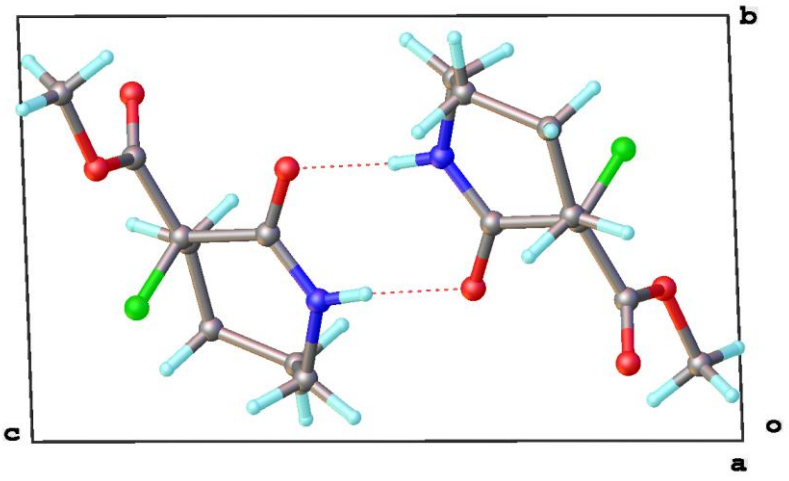
Number of restraints - 1, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H,H) groups, All N(H,H,H) groups
 At 1.5 times of:
  All O(H) groups
2.a Secondary CH2 refined with riding coordinates:
 C4(H4A,H4B), C5(H5A,H5B), C6(H6A,H6B), C7(H7A,H7B)
2.b Idealised Me refined as rotating group:
 N1(H1A,H1B,H1C)
2.c Idealised tetrahedral OH refined as rotating group:
 O4(H4)
```

A3.12. Methyl 3-fluoro-2-oxo-3-azepanecarboxylate 251 (16srv117)





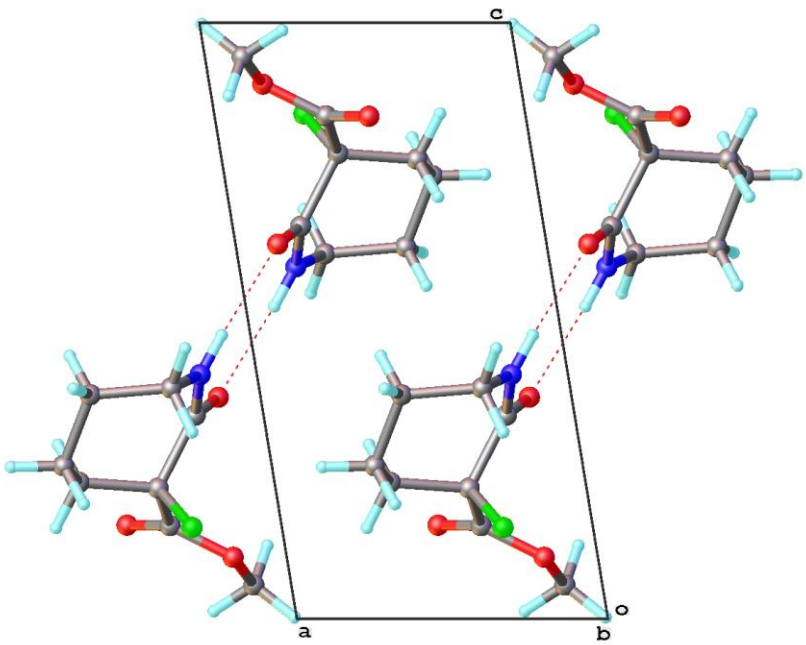


Table 1 Crystal data and structure refinement for 16srv117.	
Identification code	16srv117
Empirical formula	$C_8H_{12}NO_3F$
Formula weight	189.19
Temperature/K	120.0
Crystal system	triclinic
Space group	P-1
a/Å	5.9304(3)
b/Å	6.8227(3)
c/Å	11.3964(5)
α/°	86.5454(18)
β/°	80.2800(17)
γ/°	80.4348(17)
Volume/Å^3	447.93(4)
Z	2
ρ_{calc}g/cm^3	1.403
μ/mm^{-1}	0.120
F(000)	200.0
Crystal size/mm^3	0.25 × 0.2 × 0.08
Radiation	MoKα (λ = 0.71073)

2Θ range for data collection/°	6.06 to 60
Index ranges	$-8 \le h \le 8$, $-9 \le k \le 9$, $-15 \le l \le 15$
Reflections collected	9920
Independent reflections	2621 [R_{int} = 0.0271, R_{sigma} = 0.0273]
Data/restraints/parameters	2621/0/123
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0396, wR_2 = 0.0959
Final R indexes [all data]	R_1 = 0.0526, wR_2 = 0.1019
Largest diff. peak/hole / e $Å^{-3}$	0.34/-0.21

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 16srv117. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	2884.7(12)	6853.6(10)	1533.5(6)	21.26(16)
O1	1355.4(15)	3592.9(12)	3717.1(7)	23.8(2)
O2	1721.2(15)	3580.5(13)	1034.2(7)	24.3(2)
O3	4964.0(17)	1824.6(13)	1572.2(8)	30.2(2)
N1	1818.7(18)	6694.5(15)	4097.5(9)	20.4(2)
C1	2192.5(18)	5125.5(17)	3415.4(9)	17.5(2)
C2	3770.8(19)	5190.8(16)	2196.4(9)	17.3(2)
C3	6275(2)	5302.2(18)	2289.5(11)	23.9(2)
C4	6700(2)	7349.2(19)	2570.2(12)	25.9(3)
C5	5453(2)	8193(2)	3758.6(12)	26.9(3)
C6	2828(2)	8529.8(17)	3874.4(11)	22.8(2)
C7	3581(2)	3323.3(17)	1564.3(10)	20.0(2)
C8	1202(3)	1803(2)	568.4(12)	32.0(3)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 16srv117. 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}**
F1	23.9(3)	19.5(3)	19.0(3)	4.2(2)	-5.2(3)	0.8(3)
O1	30.4(5)	22.0(4)	18.8(4)	-1.6(3)	2.2(3)	-9.2(3)
O2	26.6(4)	24.1(4)	22.5(4)	-6.5(3)	-6.4(3)	-0.5(3)
O3	31.8(5)	22.2(4)	34.1(5)	-3.1(4)	-6.2(4)	4.8(4)
N1	22.8(5)	22.2(5)	16.0(4)	-2.7(3)	2.3(4)	-7.4(4)
C1	16.1(5)	21.6(5)	14.5(5)	0.4(4)	-2.8(4)	-2.7(4)
C2	17.4(5)	17.7(5)	15.4(5)	2.2(4)	-2.1(4)	0.2(4)
C3	15.8(5)	24.1(6)	29.8(6)	1.6(4)	-2.3(4)	0.1(4)
C4	15.9(5)	27.4(6)	34.2(7)	0.9(5)	-3.1(5)	-5.0(4)

C5	25.6(6)	30.6(7)	28.0(6)	0.3(5)	-8.2(5)	-10.6(5)
C6	25.5(6)	20.3(6)	23.3(6)	-3.1(4)	-2.0(4)	-7.2(4)
C7	22.1(5)	21.6(5)	14.0(5)	0.9(4)	1.0(4)	-1.1(4)
C8	40.8(8)	29.9(7)	27.5(6)	-11.8(5)	-9.2(6)	-4.8(6)

Table 4 Bond Lengths for 16srv117.

Atom	**Atom**	**Length/Å**		**Atom**	**Atom**	**Length/Å**
F1	C2	1.4024(12)		C1	C2	1.5412(15)
O1	C1	1.2368(14)		C2	C3	1.5213(16)
O2	C7	1.3264(14)		C2	C7	1.5300(16)
O2	C8	1.4506(15)		C3	C4	1.5212(18)
O3	C7	1.2006(14)		C4	C5	1.5253(18)
N1	C1	1.3276(14)		C5	C6	1.5195(17)
N1	C6	1.4663(15)				

Table 5 Bond Angles for 16srv117.

Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C7	O2	C8	115.54(10)		C3	C2	C7	111.44(9)
C1	N1	C6	128.08(10)		C7	C2	C1	106.17(9)
O1	C1	N1	123.10(10)		C4	C3	C2	114.42(9)
O1	C1	C2	118.04(10)		C3	C4	C5	116.80(10)
N1	C1	C2	118.86(10)		C6	C5	C4	113.82(10)
F1	C2	C1	108.55(8)		N1	C6	C5	113.59(10)
F1	C2	C3	109.04(9)		O2	C7	C2	111.71(9)
F1	C2	C7	108.05(8)		O3	C7	O2	125.57(11)
C3	C2	C1	113.40(9)		O3	C7	C2	122.72(11)

Table 6 Hydrogen Bonds for 16srv117.

D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
N1	H1	O1[1]	0.858(17)	2.020(17)	2.8750(13)	174.7(14)

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

Table 7 Torsion Angles for 16srv117.

A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
F1	C2	C3	C4	43.96(13)		C1	C2	C7	O3	-96.64(13)
F1	C2	C7	O2	-33.52(12)		C2	C3	C4	C5	62.38(15)
F1	C2	C7	O3	147.07(11)		C3	C2	C7	O2	-153.30(10)
O1	C1	C2	F1	124.60(10)		C3	C2	C7	O3	27.29(15)
O1	C1	C2	C3	-114.05(12)		C3	C4	C5	C6	-62.77(14)
O1	C1	C2	C7	8.65(13)		C4	C5	C6	N1	75.78(13)
N1	C1	C2	F1	-56.29(13)		C6	N1	C1	O1	175.62(11)
N1	C1	C2	C3	65.06(13)		C6	N1	C1	C2	-3.45(17)

N1	C1	C2	C7	-172.24(10)		C7	C2	C3	C4	163.16(10)
C1	N1	C6	C5	-60.50(16)		C8	O2	C7	O3	8.45(17)
C1	C2	C3	C4	-77.11(13)		C8	O2	C7	C2	-170.94(10)
C1	C2	C7	O2	82.77(11)						

Table 8 Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 16srv117.

Atom	***x***	***y***	***z***	**U(eq)**
H3A	6763	4339	2919	29
H3B	7263	4891	1527	29
H4A	6241	8296	1927	31
H4B	8386	7290	2548	31
H5A	5916	7266	4410	32
H5B	5964	9473	3860	32
H6A	2167	9437	4535	27
H6B	2381	9194	3132	27
H8A	66	2179	29	48
H8B	564	965	1229	48
H8C	2625	1064	132	48
H1	920(30)	6530(20)	4758(15)	27(4)

Refinement model description

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

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 C3(H3A,H3B), C4(H4A,H4B), C5(H5A,H5B), C6(H6A,H6B)
2.b Idealised Me refined as rotating group:
 C8(H8A,H8B,H8C)
```