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# *Synthesis and Structural Features of -Fluorocarbonyl Systems*

CRAIG AARON FISHER

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## Appendix 3 - X-ray Crystallography Information

### Table of Contents

|   |    |
|---|----|
| A3.1. Dimethyl 2-fluoro-2-methylmalonate <b>232</b> (15srv211).....                                       | 2  |
| A3.2. Dimethyl 2-fluoro-2-(4-nitrobenzyl)malonate <b>236</b> (15srv034).....                              | 5  |
| A3.3. Dimethyl 2-diphenylmethyl-2-fluoromalonate <b>238</b> (15srv036).....                               | 9  |
| A3.4. Methyl 2-fluoro-3-(benzylamino)-3-oxopropanoate <b>240</b> (15srv201).....                          | 14 |
| A3.5. Dimethyl 2-(3-aminopropyl)-2-fluoromalonate, hydrochloride salt <b>247</b><br>(15srv065) .....      | 18 |
| A3.6. Racemic methyl 3-fluoro-2-oxo-3-piperidinecarboxylate <b>248</b> (14srv042) .....                   | 22 |
| A3.7. ( <i>S</i> )-Methyl 3-fluoro-2-oxo-3-piperidinecarboxylate ( <i>S</i> )- <b>248</b> (14srv257)..... | 28 |
| A3.8. ( <i>R</i> )-Methyl 3-fluoro-2-oxo-3-piperidinecarboxylate ( <i>R</i> )- <b>248</b> (14srv258)..... | 34 |
| A3.9. Dimethyl 2-(2-aminoethyl)-2-fluoromalonate, hydrochloride <b>257</b> (14srv104).                    | 39 |
| A3.10. Methyl 3-fluoro-2-oxo-3-pyrrolidinecarboxylate <b>250</b> (14srv109) .....                         | 43 |
| A3.11. Dimethyl 2-(4-aminobutyl)-2-fluoromalonate hydrochloride <b>259</b> (16srv100)                     | 46 |
| A3.12. Methyl 3-fluoro-2-oxo-3-azepanecarboxylate <b>251</b> (16srv117) .....                             | 52 |

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

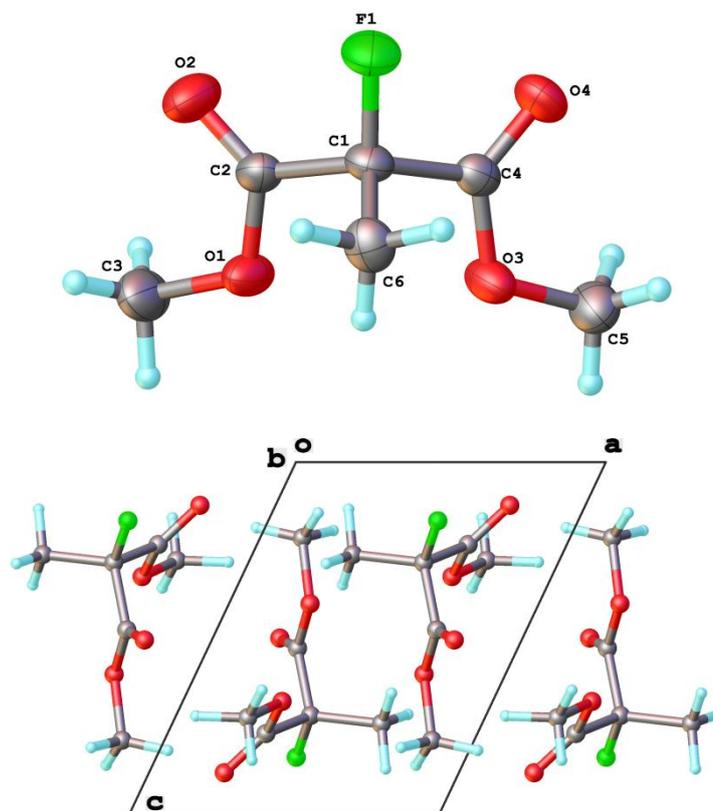


Table 1 Crystal data and structure refinement for 15srv211.

|                                       |  |
|---------------------------------------|--|
| Identification code                   | 15srv211                                       |
| Empirical formula                     | C <sub>6</sub> H <sub>9</sub> O <sub>4</sub> F |
| 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)                                       |
| $\alpha$ /°                           | 93.71(6)                                       |
| $\beta$ /°                            | 112.89(5)                                      |
| $\gamma$ /°                           | 106.42(9)                                      |
| Volume/Å <sup>3</sup>                 | 374.87(19)                                     |
| Z                                     | 2  |
| $\rho_{\text{calc}}$ /cm <sup>3</sup> | 1.454  |

|  |   |
|--|---|
| $\mu/\text{mm}^{-1}$                           | 0.137   |
| F(000)   | 172.0   |
| Crystal size/ $\text{mm}^3$                    | $0.3 \times 0.3 \times 0.3$                                   |
| Radiation                                      | MoK $\alpha$ ( $\lambda = 0.71073$ )                          |
| 2 $\Theta$ range for data collection/ $^\circ$ | 5.52 to 55.98   |
| Index ranges                                   | $-8 \leq h \leq 8, -9 \leq k \leq 9, -10 \leq l \leq 10$      |
| Reflections collected                          | 2783  |
| Independent reflections                        | 1425 [ $R_{\text{int}} = 0.0301, R_{\text{sigma}} = 0.0439$ ] |
| Data/restraints/parameters                     | 1425/0/136  |
| Goodness-of-fit on $F^2$                       | 1.094   |
| Final R indexes [ $I \geq 2\sigma(I)$ ]        | $R_1 = 0.0643, wR_2 = 0.1588$                                 |
| Final R indexes [all data]                     | $R_1 = 0.0800, wR_2 = 0.1710$                                 |
| Largest diff. peak/hole / $e \text{ \AA}^{-3}$ | 0.43/-0.34  |

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

| Atom | x       | y        | z       | $U(\text{eq})$ |
|------|---------|----------|---------|----------------|
| 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 ( $\text{\AA}^2 \times 10^3$ ) for 15srv211. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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) |

| 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) |

| 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)   |

| Atom | <i>x</i> | <i>y</i>  | <i>z</i> | 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)

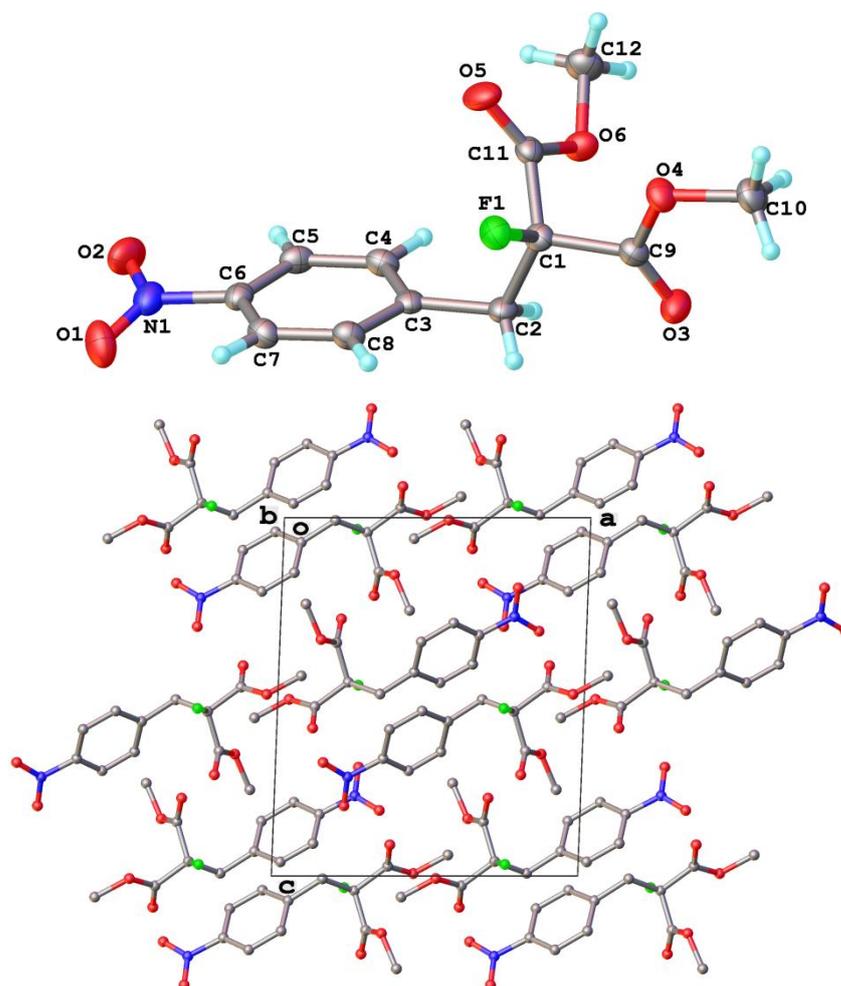


Table 1 Crystal data and structure refinement for 15srv034.

|                     |  |
|---------------------|--|
| Identification code | 15srv034   |
| Empirical formula   | C <sub>12</sub> H <sub>12</sub> FNO <sub>6</sub> |
| Formula weight      | 285.23   |
| Temperature/K       | 120.0  |
| Crystal system      | monoclinic                                       |
| Space group         | P2 <sub>1</sub> /n                               |
| a/Å                 | 11.2398(3)                                       |
| b/Å                 | 8.5962(2)  |
| c/Å                 | 13.2556(3)                                       |
| α/°                 | 90.00  |
| β/°                 | 92.089(2)  |

|  |   |
|--|---|
| $\gamma/^\circ$                                | 90.00   |
| Volume/ $\text{\AA}^3$                         | 1279.90(5)  |
| Z  | 4   |
| $\rho_{\text{calc}}/\text{g/cm}^3$             | 1.480   |
| $\mu/\text{mm}^{-1}$                           | 0.129   |
| F(000)   | 592.0   |
| Crystal size/ $\text{mm}^3$                    | $0.21 \times 0.2 \times 0.11$                                 |
| Radiation                                      | MoK $\alpha$ ( $\lambda = 0.71073$ )                          |
| 2 $\Theta$ range for data collection/ $^\circ$ | 4.66 to 57.98   |
| Index ranges                                   | $-15 \leq h \leq 15, -11 \leq k \leq 11, -18 \leq l \leq 18$  |
| Reflections collected                          | 18054   |
| Independent reflections                        | 3404 [ $R_{\text{int}} = 0.0515, R_{\text{sigma}} = 0.0433$ ] |
| Data/restraints/parameters                     | 3404/0/229  |
| Goodness-of-fit on $F^2$                       | 1.033   |
| Final R indexes [ $I \geq 2\sigma(I)$ ]        | $R_1 = 0.0427, wR_2 = 0.0929$                                 |
| Final R indexes [all data]                     | $R_1 = 0.0676, wR_2 = 0.1071$                                 |
| Largest diff. peak/hole / $e \text{\AA}^{-3}$  | 0.30/-0.20  |

Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 15srv034.  $U_{\text{eq}}$  is defined as 1/3 of the trace of the orthogonalised  $U_{\text{II}}$  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 ( $\text{\AA}^2 \times 10^3$ ) for 15srv034. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/ $\text{\AA}$ | Atom | Atom | Length/ $\text{\AA}$ |
|------|------|----------------------|------|------|----------------------|
| 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/ $^\circ$ | Atom | Atom | Atom | Angle/ $^\circ$ |
|------|------|------|-----------------|------|------|------|-----------------|
|------|------|------|-----------------|------|------|------|-----------------|

|     |    |     |            |    |     |    |            |
|-----|----|-----|------------|----|-----|----|------------|
| 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.

| <b>A</b> | <b>B</b> | <b>C</b> | <b>D</b> | <b>Angle/°</b> | <b>A</b> | <b>B</b> | <b>C</b> | <b>D</b> | <b>Angle/°</b> |
|----------|----------|----------|----------|----------------|----------|----------|----------|----------|----------------|
| 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 ( $\text{\AA} \times 10^4$ ) and Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 15srv034.

| <b>Atom</b> | <b>x</b>  | <b>y</b>  | <b>z</b> | <b>U(eq)</b> |
|-------------|-----------|-----------|----------|--------------|
| 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)

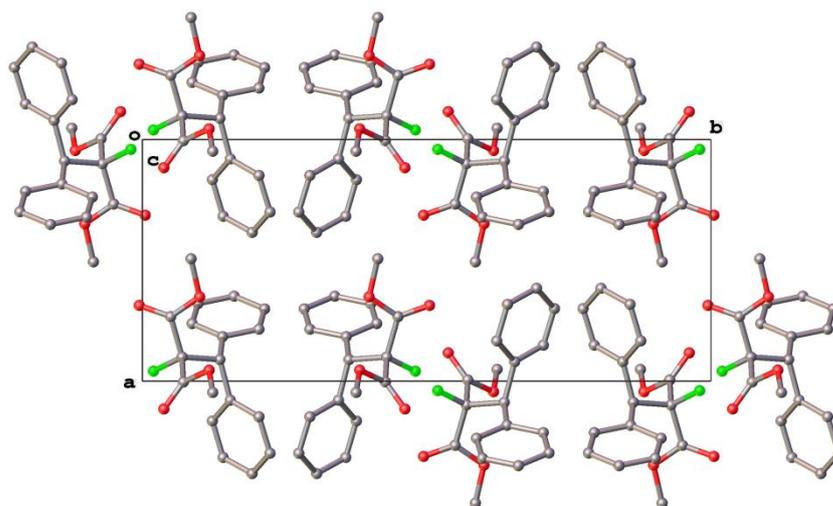
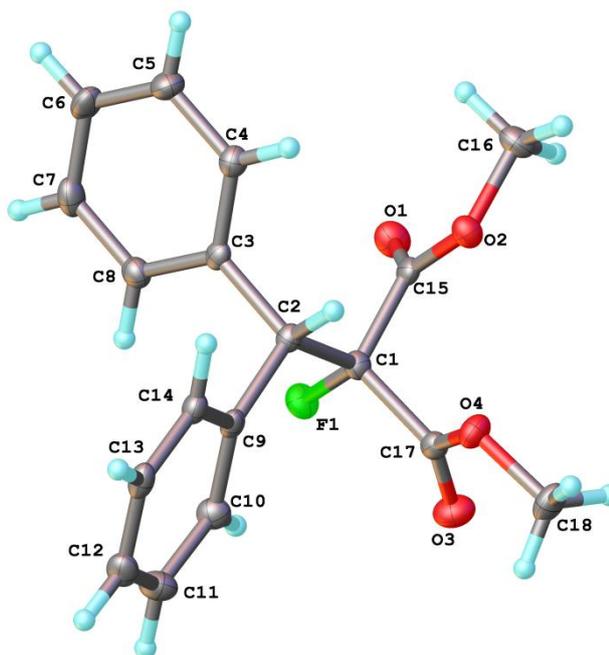


Table 1 Crystal data and structure refinement for 15srv036.

|                     |   |
|---------------------|---|
| Identification code | 15srv036  |
| Empirical formula   | C <sub>18</sub> H <sub>17</sub> FO <sub>4</sub> |
| Formula weight      | 316.32  |
| Temperature/K       | 120.0   |
| Crystal system      | monoclinic                                      |
| Space group         | P2 <sub>1</sub> /c                              |
| a/Å                 | 8.6393(2)                                       |
| b/Å                 | 20.2165(4)                                      |

|  |   |
|--|---|
| c/Å  | 8.7948(2)   |
| $\alpha/^\circ$                                | 90.00   |
| $\beta/^\circ$                                 | 91.495(4)   |
| $\gamma/^\circ$                                | 90.00   |
| Volume/Å <sup>3</sup>                          | 1535.55(6)  |
| Z  | 4   |
| $\rho_{\text{calc}}/\text{g}/\text{cm}^3$      | 1.368   |
| $\mu/\text{mm}^{-1}$                           | 0.104   |
| F(000)   | 664.0   |
| Crystal size/mm <sup>3</sup>                   | 0.23 × 0.23 × 0.1   |
| Radiation                                      | MoK $\alpha$ ( $\lambda = 0.71073$ )                          |
| 2 $\theta$ range for data collection/ $^\circ$ | 4.72 to 58  |
| Index ranges                                   | -11 ≤ h ≤ 11, -27 ≤ k ≤ 27, -11 ≤ l ≤ 11                      |
| Reflections collected                          | 31358   |
| Independent reflections                        | 4072 [R <sub>int</sub> = 0.0492, R <sub>sigma</sub> = 0.0339] |
| Data/restraints/parameters                     | 4072/0/273  |
| Goodness-of-fit on F <sup>2</sup>              | 1.082   |
| Final R indexes [I ≥ 2 $\sigma$ (I)]           | R <sub>1</sub> = 0.0508, wR <sub>2</sub> = 0.1228             |
| Final R indexes [all data]                     | R <sub>1</sub> = 0.0688, wR <sub>2</sub> = 0.1329             |
| Largest diff. peak/hole / e Å <sup>-3</sup>    | 0.65/-0.30  |

| Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 15srv036. $U_{\text{eq}}$ is defined as 1/3 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 ( $\text{\AA}^2 \times 10^3$ ) for 15srv036. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/ $\text{\AA}$ | Atom | Atom | Length/ $\text{\AA}$ |
|------|------|----------------------|------|------|----------------------|
| 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) |

| Atom | <i>x</i>  | <i>y</i> | <i>z</i>  | 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:

1.a Ternary CH refined with riding coordinates:  
C2 (H2)



|   |   |
|---|---|
| Identification code                         | 15srv201  |
| Empirical formula                           | C <sub>11</sub> H <sub>12</sub> FNO <sub>3</sub>              |
| 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 <sup>3</sup>                       | 2099.4(3)   |
| Z   | 8   |
| ρ <sub>calc</sub> /cm <sup>3</sup>          | 1.425   |
| μ/mm <sup>-1</sup>                          | 0.115   |
| F(000)                                      | 944.0   |
| Crystal size/mm <sup>3</sup>                | 0.38 × 0.2 × 0.12   |
| Radiation                                   | MoKα (λ = 0.71073)  |
| 2θ range for data collection/°              | 3.42 to 59  |
| Index ranges                                | -11 ≤ h ≤ 11, -14 ≤ k ≤ 14, -33 ≤ l ≤ 33                      |
| Reflections collected                       | 25266   |
| Independent reflections                     | 2925 [R <sub>int</sub> = 0.0534, R <sub>sigma</sub> = 0.0518] |
| Data/restraints/parameters                  | 2925/0/193  |
| Goodness-of-fit on F <sup>2</sup>           | 1.045   |
| Final R indexes [I ≥ 2σ (I)]                | R <sub>1</sub> = 0.0462, wR <sub>2</sub> = 0.1096             |
| Final R indexes [all data]                  | R <sub>1</sub> = 0.0711, wR <sub>2</sub> = 0.1315             |
| Largest diff. peak/hole / e Å <sup>-3</sup> | 0.30/-0.26  |

Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 15srv201.  $U_{\text{eq}}$  is defined as 1/3 of the trace of the orthogonalised  $U_{\text{ij}}$  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 ( $\text{\AA}^2 \times 10^3$ ) for 15srv201. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/\AA | Atom | Atom | Length/\AA |
|------|------|------------|------|------|------------|
| 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 <sup>1</sup> | 0.86(2)  | 2.11(2)  | 2.9077(19) | 155(2)  |

<sup>1</sup>1/2+X,1/2-Y,1-Z

Table 7 Selected Torsion Angles for 15srv201.

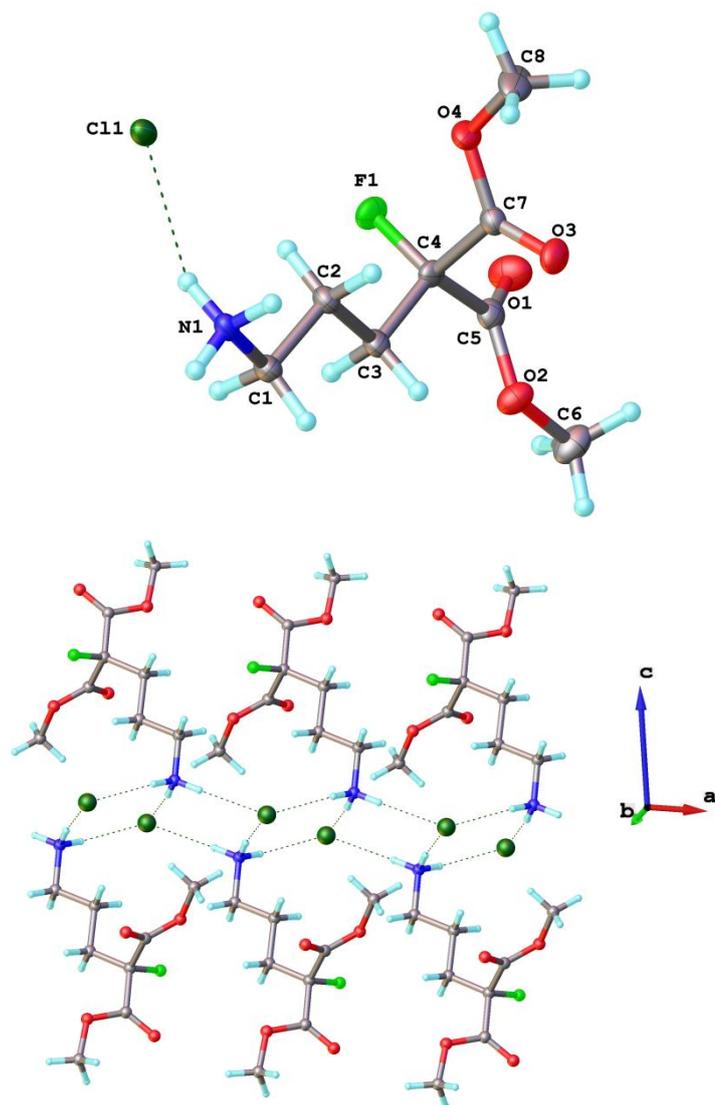
| A  | B  | C  | D  | Angle/°     | A   | B  | C  | D  | Angle/°     |
|----|----|----|----|-------------|-----|----|----|----|-------------|
| O1 | C1 | C2 | F1 | -41.90(19)  | C3  | C2 | C1 | O1 | 74.89(18)   |
| O2 | C3 | C2 | F1 | -165.68(13) | C3  | C2 | C1 | N1 | -102.43(16) |
| O3 | C3 | C2 | 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) |     |    |    |    |             |

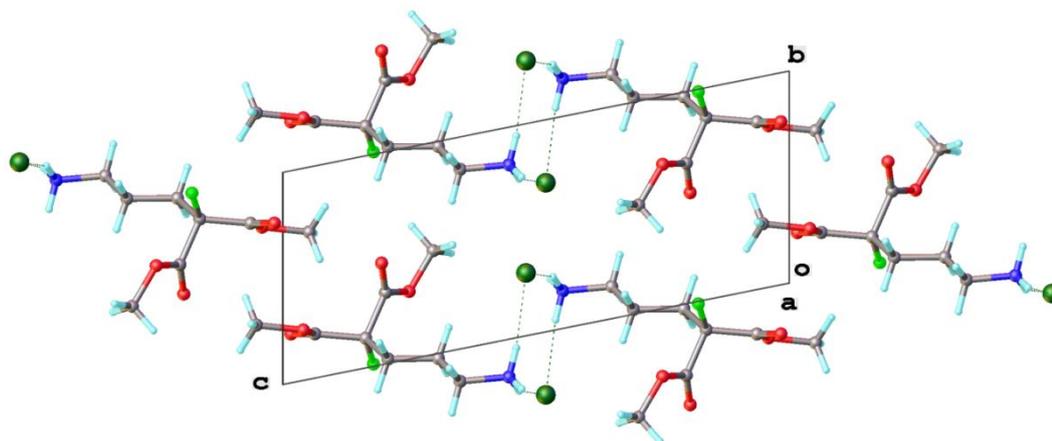
Table 8 Hydrogen Atom Coordinates (Å×10<sup>4</sup>) and Isotropic Displacement Parameters (Å<sup>2</sup>×10<sup>3</sup>) for 15srv201.

| 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)**





|  |  |
|--|--|
| Identification code                            | 15srv065   |
| Empirical formula                              | $C_8H_{15}FNO_4^+ \times Cl^-$                           |
| 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)  |
| $\alpha/^\circ$                                | 100.738(7)   |
| $\beta/^\circ$                                 | 96.625(6)  |
| $\gamma/^\circ$                                | 94.612(6)  |
| Volume/Å <sup>3</sup>                          | 577.23(7)  |
| Z  | 2  |
| $\rho_{calc}/cm^3$                             | 1.402  |
| $\mu/mm^{-1}$                                  | 0.340  |
| F(000)   | 256.0  |
| Crystal size/mm <sup>3</sup>                   | 0.36 × 0.21 × 0.05                                       |
| Radiation                                      | MoK $\alpha$ ( $\lambda = 0.71073$ )                     |
| 2 $\theta$ range for data collection/ $^\circ$ | 5.32 to 56   |
| Index ranges                                   | $-7 \leq h \leq 7, -8 \leq k \leq 8, -20 \leq l \leq 20$ |
| 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 [ $I \geq 2\sigma(I)$ ]        | $R_1 = 0.0521, wR_2 = 0.1240$                            |
| Final R indexes [all data]                     | $R_1 = 0.0626, wR_2 = 0.1289$                            |
| Largest diff. peak/hole / e Å <sup>-3</sup>    | 0.56/-0.27   |

Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) 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 ( $\text{\AA}^2 \times 10^3$ ) for 15srv065. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^*U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/ $\text{\AA}$ | Atom | Atom | Length/ $\text{\AA}$ |
|------|------|----------------------|------|------|----------------------|
| 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) |  |    |    |          |

| 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) |      |      |      |          |

| D  | H   | A                | d(D-H)/Å | d(H-A)/Å | d(D-A)/Å | D-H-A/° |
|----|-----|------------------|----------|----------|----------|---------|
| N1 | H1A | Cl1 <sup>1</sup> | 0.91     | 2.32     | 3.226(2) | 172.2   |
| N1 | H1B | Cl1 <sup>2</sup> | 0.91     | 2.30     | 3.169(2) | 158.9   |
| N1 | H1C | Cl1              | 0.91     | 2.31     | 3.160(2) | 154.7   |

<sup>1</sup>1-X,2-Y,1-Z; <sup>2</sup>-1+X,+Y,+Z

| 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)  |    |    |    |    |           |

| 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

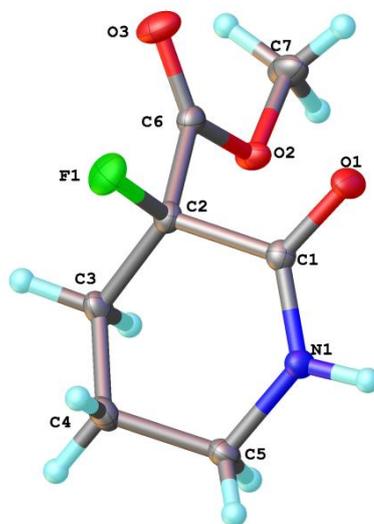
2.a Secondary CH2 refined with riding coordinates:

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)



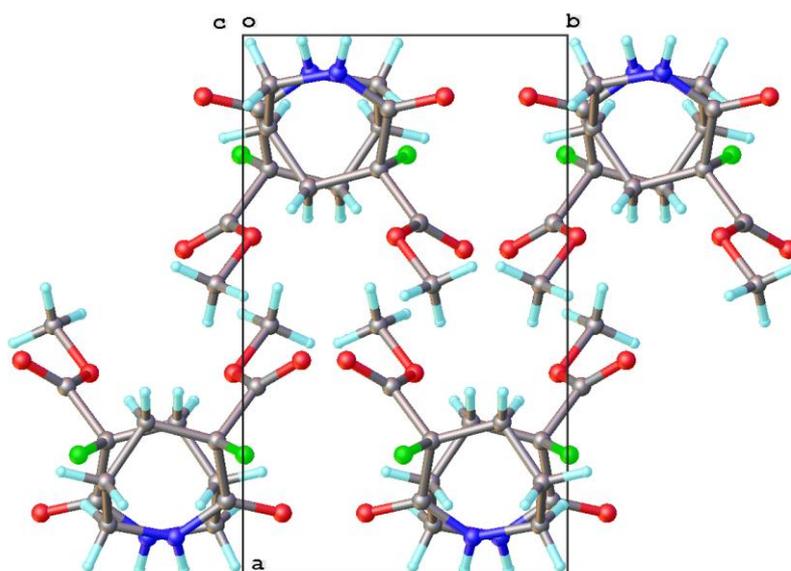
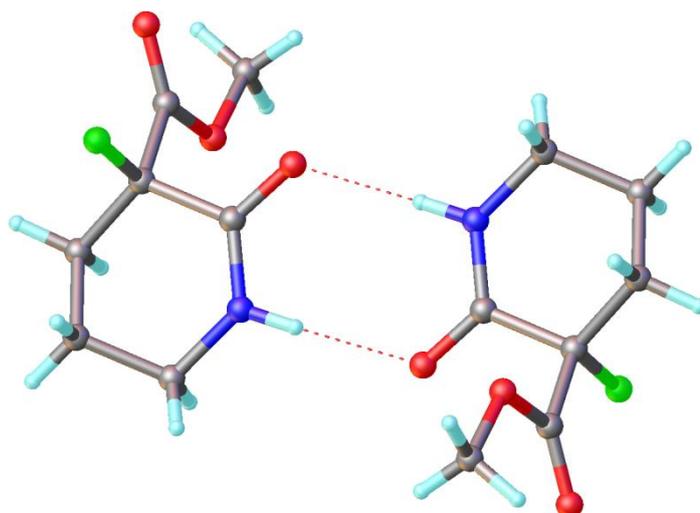


Table 1 Crystal data and structure refinement for 14srv042.

|                     |                  |
|---------------------|------------------|
| 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)  |
| $\alpha/^\circ$                             | 90.00   |
| $\beta/^\circ$                              | 113.193(4)  |
| $\gamma/^\circ$                             | 90.00   |
| Volume/Å <sup>3</sup>                       | 793.93(4)   |
| Z   | 4   |
| $\rho_{\text{calc}}/\text{mg}/\text{mm}^3$  | 1.465   |
| m/mm <sup>-1</sup>                          | 0.129   |
| F(000)                                      | 368.0   |
| Crystal size/mm <sup>3</sup>                | 0.31 × 0.26 × 0.17  |
| Radiation                                   | MoK $\alpha$ ( $\lambda$ = 0.71073)                           |
| 2 $\theta$ 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 <sub>int</sub> = 0.0385, R <sub>sigma</sub> = 0.0236] |
| Data/restraints/parameters                  | 2324/0/149  |
| Goodness-of-fit on F <sup>2</sup>           | 1.028   |
| Final R indexes [I >= 2 $\sigma$ (I)]       | R <sub>1</sub> = 0.0346, wR <sub>2</sub> = 0.0902             |
| Final R indexes [all data]                  | R <sub>1</sub> = 0.0410, wR <sub>2</sub> = 0.0942             |
| Largest diff. peak/hole / e Å <sup>-3</sup> | 0.47/-0.23  |

Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{Å}^2 \times 10^3$ ) for 14srv042.  $U_{\text{eq}}$  is defined as 1/3 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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv042. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*U_{11}+2hka^*b^*U_{12}+\dots]$ . |          |          |          |          |          |          |
|---|----------|----------|----------|----------|----------|----------|
| 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 <sup>1</sup> | 0.881(16) | 1.998(16) | 2.8770(11) | 175.7(14) |

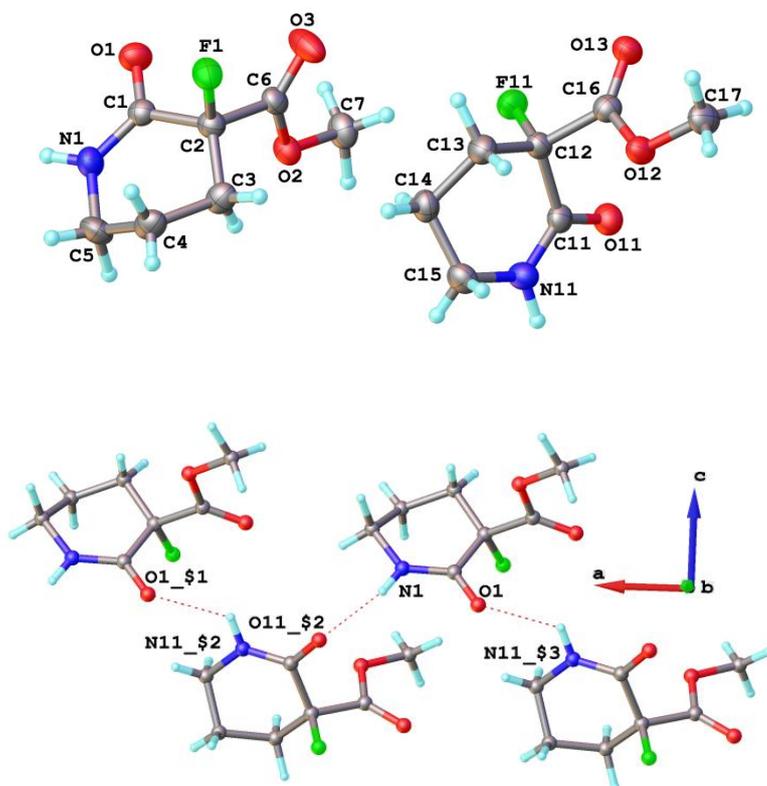
<sup>1</sup>2-X,2-Y,1-Z

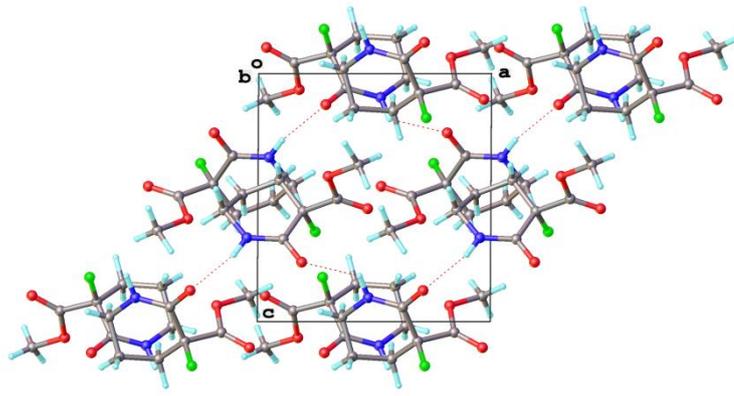
| Table 7 Torsion Angles for 14srv042. |    |    |    |            |  |    |    |    |    |             |
|--------------------------------------|----|----|----|------------|--|----|----|----|----|-------------|
| A                                    | B  | C  | D  | Angle/°    |  | A  | B  | C  | D  | 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 <sup>4</sup> ) and Isotropic Displacement Parameters (Å <sup>2</sup> ×10 <sup>3</sup> ) 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)**





| Table 1 Crystal data and structure refinement for 14srv257 |   |
|--|---|
| Identification code  | 14srv257  |
| Empirical formula  | C <sub>7</sub> H <sub>10</sub> FNO <sub>3</sub>               |
| Formula weight   | 175.16  |
| Temperature/K  | 120.0   |
| Crystal system   | monoclinic  |
| Space group  | P2 <sub>1</sub>   |
| 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 <sup>3</sup>                                      | 803.06(6)   |
| Z  | 4   |
| ρ <sub>calc</sub> /g/cm <sup>3</sup>                       | 1.449   |
| μ/mm <sup>-1</sup>   | 1.105   |
| F(000)   | 368.0   |
| Crystal size/mm <sup>3</sup>                               | 0.53 × 0.35 × 0.29  |
| Radiation  | CuKα (λ = 1.54178)  |
| 2θ range for data collection/°                             | 9.68 to 144.92  |
| Index ranges   | -9 ≤ h ≤ 9, -12 ≤ k ≤ 12, -11 ≤ l ≤ 9                         |
| Reflections collected                                      | 7399  |
| Independent reflections                                    | 2833 [R <sub>int</sub> = 0.0612, R <sub>sigma</sub> = 0.0576] |
| Data/restraints/parameters                                 | 2833/1/297  |
| Goodness-of-fit on F <sup>2</sup>                          | 1.005   |
| Final R indexes [I >= 2σ (I)]                              | R <sub>1</sub> = 0.0547, wR <sub>2</sub> = 0.1520             |
| Final R indexes [all data]                                 | R <sub>1</sub> = 0.0547, wR <sub>2</sub> = 0.1520             |
| Largest diff. peak/hole / e Å <sup>-3</sup>                | 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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv257. $U_{eq}$ is defined as 1/3 of of the trace of the orthogonalised $U_{ij}$ tensor. |          |            |            |          |
|---|----------|------------|------------|----------|
| Atom  | <i>x</i> | <i>y</i>   | <i>z</i>   | $U_{eq}$ |
| 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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv257. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*^2U_{11}+2hka^*b^*U_{12}+\dots]$ . |          |          |          |           |          |          |
|---|----------|----------|----------|-----------|----------|----------|
| 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) |

| D   | H   | A                | d(D-H)/Å | d(H-A)/Å | d(D-A)/Å | D-H-A/° |
|-----|-----|------------------|----------|----------|----------|---------|
| N1  | H1  | O11 <sup>1</sup> | 0.86(5)  | 2.08(4)  | 2.857(4) | 150(4)  |
| N11 | H11 | O1 <sup>2</sup>  | 0.93(5)  | 2.37(4)  | 3.051(3) | 130(3)  |

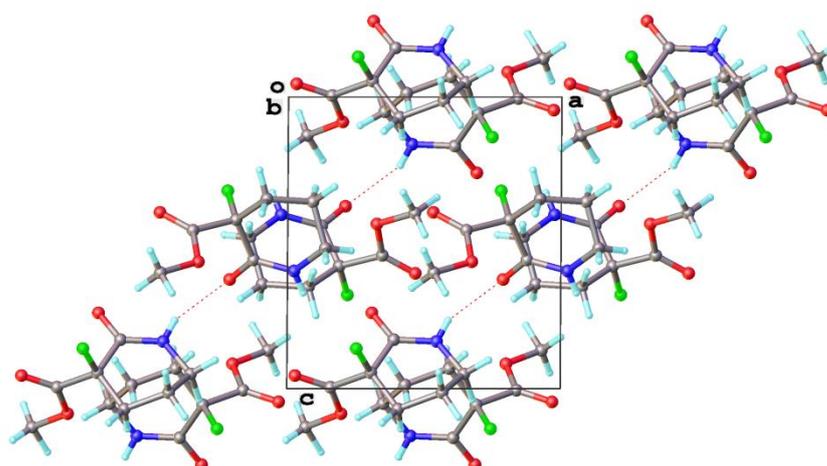
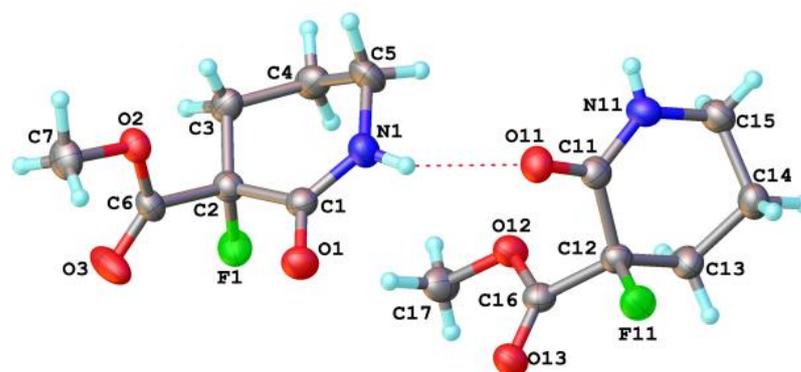
$${}^11+x,+y,-1+z; {}^2+x,+y,1+z$$

| 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) |

| 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)**



|                     |   |
|---------------------|---|
| Identification code | 14srv258  |
| Empirical formula   | C <sub>7</sub> H <sub>10</sub> FNO <sub>3</sub> |
| Formula weight      | 175.16  |
| Temperature/K       | 120.0   |
| Crystal system      | monoclinic                                      |

|   |  |
|---|--|
| Space group                                 | P2 <sub>1</sub>  |
| a/Å   | 8.5211(3)  |
| b/Å   | 10.3039(3)   |
| c/Å   | 9.1547(3)  |
| $\alpha$ /°                                 | 90.00  |
| $\beta$ /°                                  | 90.3650(8)   |
| $\gamma$ /°                                 | 90.00  |
| Volume/Å <sup>3</sup>                       | 803.77(5)  |
| Z   | 4  |
| $\rho_{\text{calc}}$ /cm <sup>3</sup>       | 1.447  |
| $\mu$ /mm <sup>-1</sup>                     | 1.104  |
| F(000)                                      | 368.0  |
| Crystal size/mm <sup>3</sup>                | 0.32 × 0.3 × 0.24  |
| Radiation                                   | CuK $\alpha$ ( $\lambda$ = 1.54178)                            |
| 2 $\theta$ range for data collection/°      | 9.66 to 144.94   |
| Index ranges                                | -9 ≤ h ≤ 10, -12 ≤ k ≤ 12, -11 ≤ l ≤ 11                        |
| Reflections collected                       | 6957   |
| Independent reflections                     | 2916 [ $R_{\text{int}}$ = 0.0579, $R_{\text{sigma}}$ = 0.0613] |
| Data/restraints/parameters                  | 2916/1/297   |
| Goodness-of-fit on F <sup>2</sup>           | 1.103  |
| Final R indexes [ $I \geq 2\sigma(I)$ ]     | $R_1$ = 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 <sup>-3</sup> | 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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv258.  $U_{\text{eq}}$  is defined as 1/3 of of the trace of the orthogonalised  $U_{\text{ij}}$  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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv258. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^*^2U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/\AA | Atom | Atom | Length/\AA |
|------|------|------------|------|------|------------|
| 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) |

| 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)   |

| 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 <sup>1</sup> | 0.79(4)  | 2.46(4)  | 3.042(3) | 132(3)  |

<sup>1</sup>-1+X,+Y,+Z

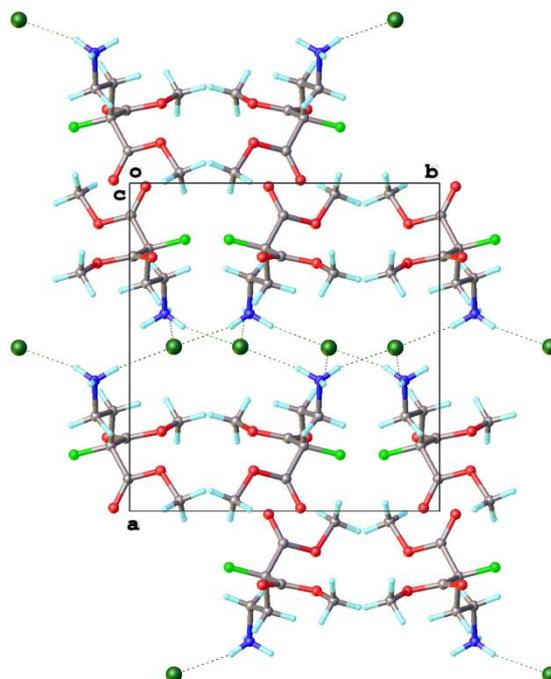
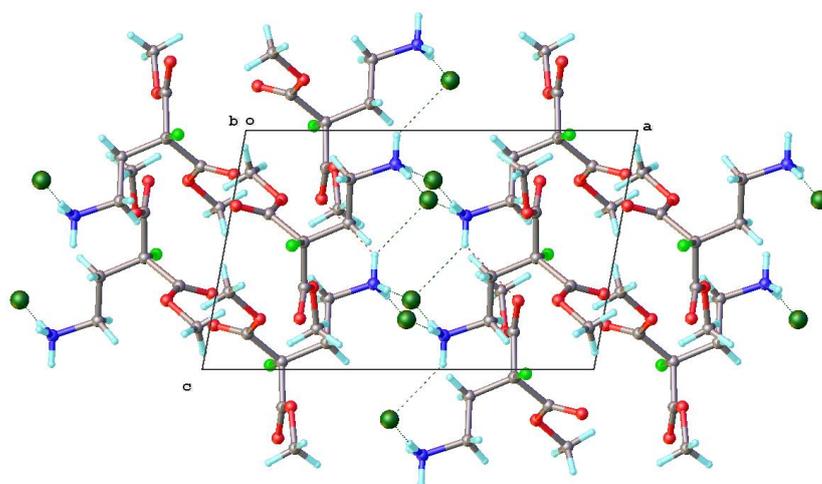
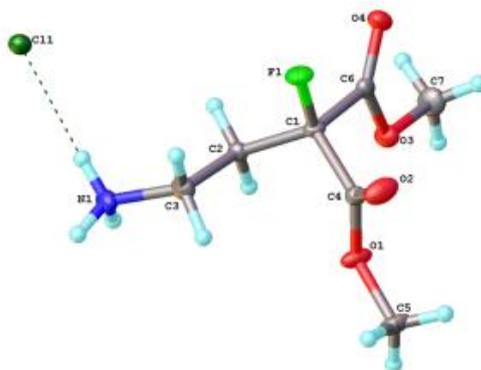
| A  | B  | C  | D  | Angle/°     | A   | B   | C   | D   | Angle/°    |
|----|----|----|----|-------------|-----|-----|-----|-----|------------|
| F1 | C2 | C3 | C4 | -77.0(3)    | F11 | C12 | C13 | C14 | -80.8(2)   |
| F1 | C2 | C6 | O2 | -174.02(19) | F11 | C12 | C16 | O12 | 172.25(19) |
| F1 | C2 | 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 ( $\text{\AA} \times 10^4$ ) and Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) 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)**



|   |   |
|---|---|
| Identification code                         | 14srv104  |
| Empirical formula                           | C <sub>7</sub> H <sub>13</sub> NO <sub>4</sub> FCl            |
| Formula weight                              | 229.63  |
| Temperature/K                               | 120.0   |
| Crystal system                              | monoclinic  |
| Space group                                 | P2 <sub>1</sub> /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 <sup>3</sup>                       | 1040.12(10)   |
| Z   | 4   |
| ρ <sub>calc</sub> /mg/mm <sup>3</sup>       | 1.466   |
| m/mm <sup>-1</sup>                          | 0.372   |
| F(000)                                      | 480.0   |
| Crystal size/mm <sup>3</sup>                | 0.23 × 0.22 × 0.12  |
| Radiation                                   | MoKα (λ = 0.71073)  |
| 2θ range for data collection                | 4.94 to 60°   |
| Index ranges                                | -17 ≤ h ≤ 17, -15 ≤ k ≤ 15, -10 ≤ l ≤ 10                      |
| Reflections collected                       | 21745   |
| Independent reflections                     | 3038 [R <sub>int</sub> = 0.0617, R <sub>sigma</sub> = 0.0295] |
| Data/restraints/parameters                  | 3038/0/179  |
| Goodness-of-fit on F <sup>2</sup>           | 1.097   |
| Final R indexes [I >= 2σ (I)]               | R <sub>1</sub> = 0.0341, wR <sub>2</sub> = 0.0846             |
| Final R indexes [all data]                  | R <sub>1</sub> = 0.0384, wR <sub>2</sub> = 0.0873             |
| Largest diff. peak/hole / e Å <sup>-3</sup> | 0.59/-0.24  |

| 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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv104. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^*^2U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/ $\text{\AA}$ | Atom | Atom | Length/ $\text{\AA}$ |
|------|------|----------------------|------|------|----------------------|
| 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/ $^\circ$ | Atom | Atom | Atom | Angle/ $^\circ$ |
|------|------|------|-----------------|------|------|------|-----------------|
| 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)       |

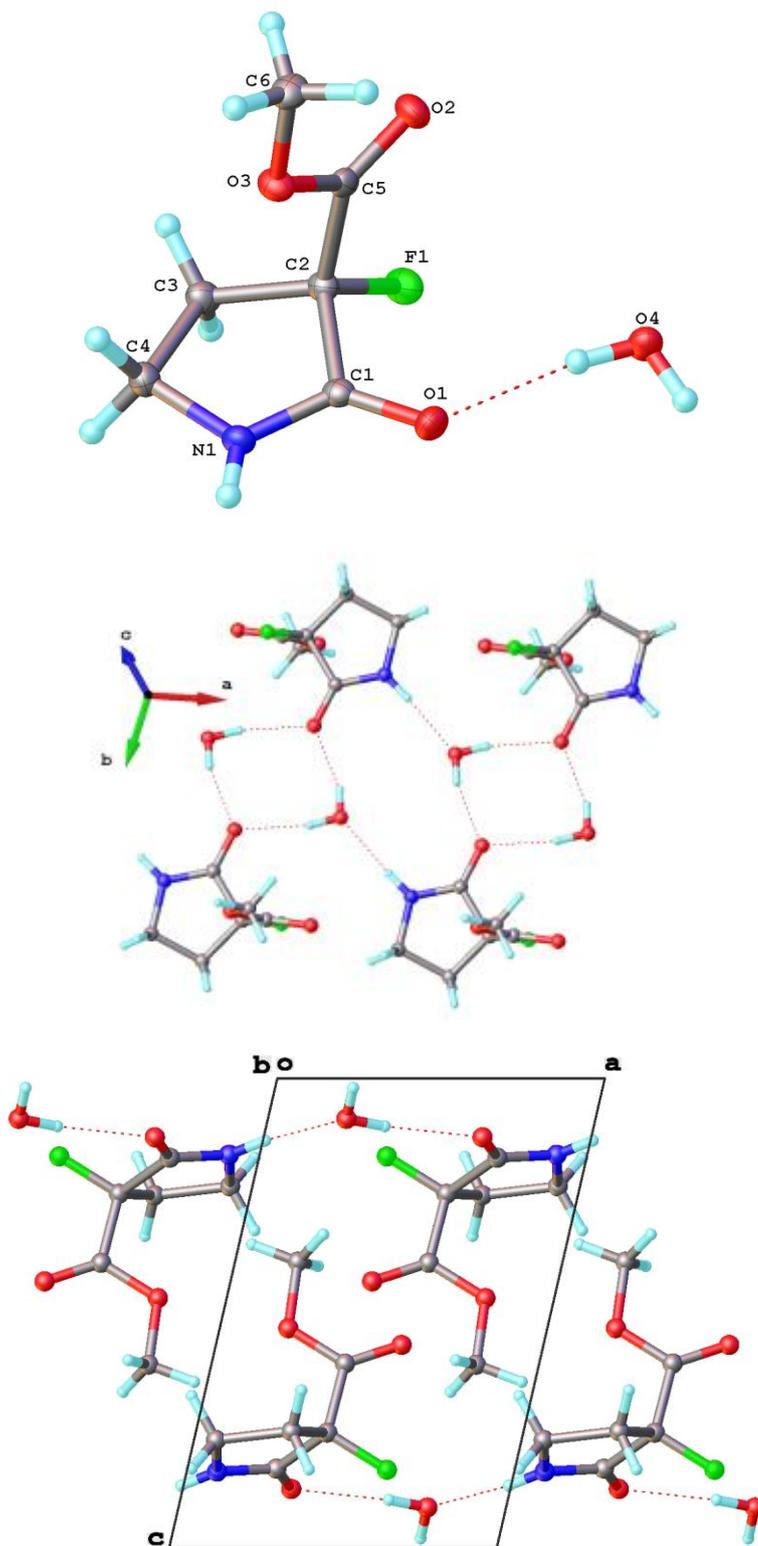
| D  | H   | A                | d(D-H)/Å  | d(H-A)/Å  | d(D-A)/Å   | D-H-A/°   |
|----|-----|------------------|-----------|-----------|------------|-----------|
| N1 | H1A | Cl1 <sup>1</sup> | 0.907(17) | 2.421(17) | 3.1605(9)  | 138.9(13) |
| N1 | H1A | O4 <sup>2</sup>  | 0.907(17) | 2.506(16) | 3.1602(13) | 129.3(13) |
| N1 | H1B | Cl1 <sup>3</sup> | 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) |

<sup>1</sup>+X,3/2-Y,1/2+Z; <sup>2</sup>+X,+Y,1+Z; <sup>3</sup>1-X,-1/2+Y,1/2-Z

| 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)   |

| 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   | $C_6H_8FNO_3 \times H_2O$ |
| 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)  |
| $\alpha$ /°                                 | 98.163(3)   |
| $\beta$ /°                                  | 100.929(3)  |
| $\gamma$ /°                                 | 101.047(3)  |
| Volume/Å <sup>3</sup>                       | 399.30(7)   |
| Z   | 2   |
| $\rho_{\text{calc}}$ mg/mm <sup>3</sup>     | 1.490   |
| m/mm <sup>-1</sup>                          | 0.138   |
| F(000)                                      | 188.0   |
| Crystal size/mm <sup>3</sup>                | 0.56 × 0.25 × 0.2   |
| Radiation                                   | MoK $\alpha$ ( $\lambda$ = 0.71073)                           |
| 2 $\theta$ range for data collection        | 6.32 to 60°   |
| Index ranges                                | -9 ≤ h ≤ 9, -9 ≤ k ≤ 9, -13 ≤ l ≤ 13                          |
| Reflections collected                       | 6012  |
| Independent reflections                     | 2317 [R <sub>int</sub> = 0.0666, R <sub>sigma</sub> = 0.0681] |
| Data/restraints/parameters                  | 2317/0/149  |
| Goodness-of-fit on F <sup>2</sup>           | 1.012   |
| Final R indexes [I >= 2 $\sigma$ (I)]       | R <sub>1</sub> = 0.0548, wR <sub>2</sub> = 0.1404             |
| Final R indexes [all data]                  | R <sub>1</sub> = 0.0671, wR <sub>2</sub> = 0.1501             |
| Largest diff. peak/hole / e Å <sup>-3</sup> | 0.54/-0.28  |

Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 14srv109.  $U_{\text{eq}}$  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 ( $\text{\AA}^2 \times 10^3$ ) for 14srv109. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| Atom | U <sub>11</sub> | U <sub>22</sub> | U <sub>33</sub> | U <sub>23</sub> | U <sub>13</sub> | U <sub>12</sub> |
|------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 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.

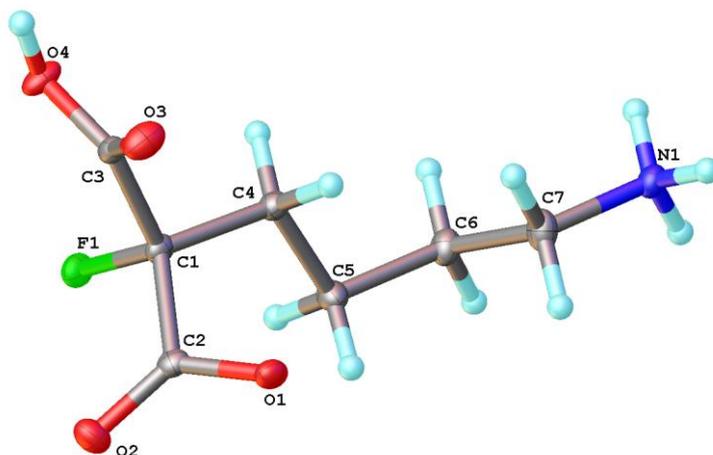
| D  | H   | A               | d(D-H)/Å | d(H-A)/Å | d(D-A)/Å   | D-H-A/°   |
|----|-----|-----------------|----------|----------|------------|-----------|
| N1 | H1  | O4 <sup>1</sup> | 0.85(2)  | 1.96(2)  | 2.8076(16) | 171.8(17) |
| O4 | H4C | O1              | 0.86(2)  | 2.02(2)  | 2.8635(15) | 170(2)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

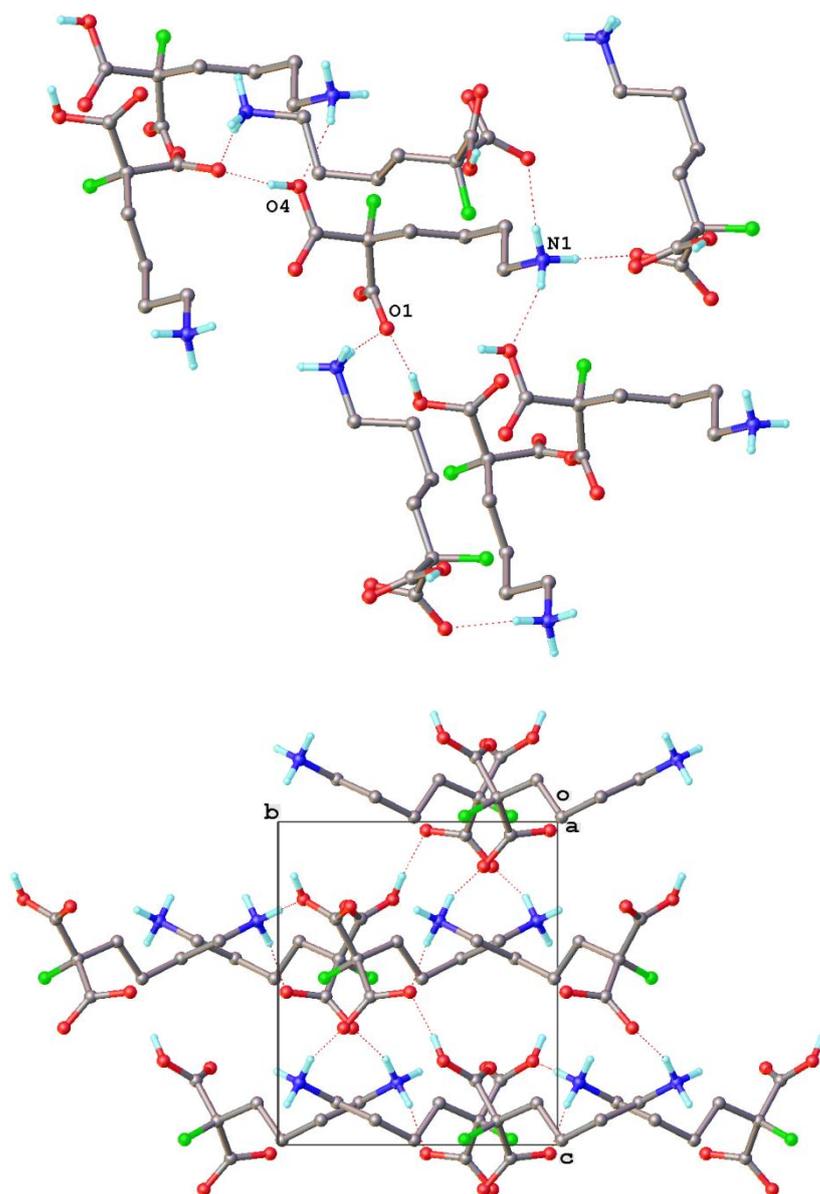
<sup>1</sup>1+X,+Y,+Z; <sup>2</sup>1-X,2-Y,-Z

| 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)  |    |    |    |    |             |

| Atom | <i>x</i>  | <i>y</i> | <i>z</i> | 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)





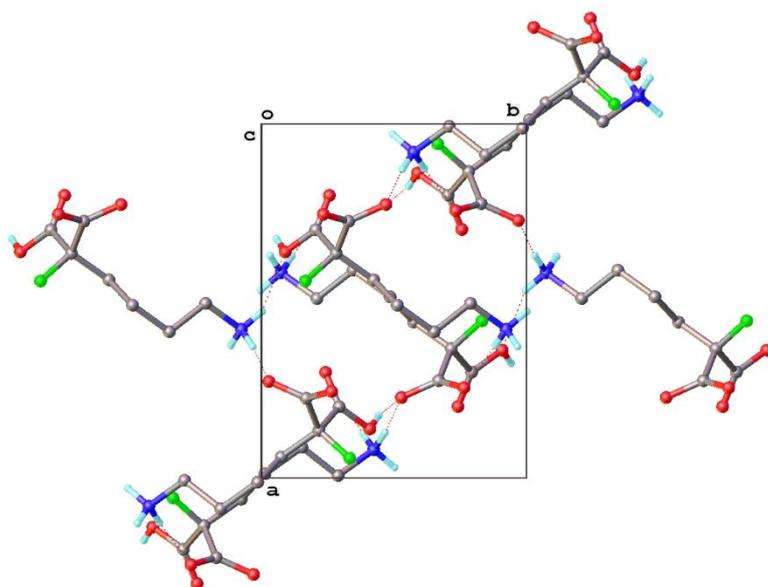
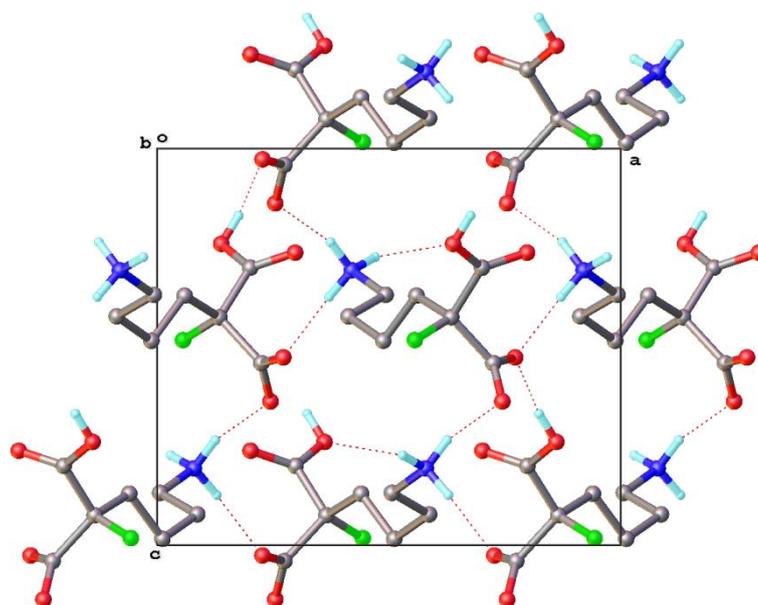


Table 1 Crystal data and structure refinement for 16srv100.

|                     |                  |
|---------------------|------------------|
| Identification code | 16srv100         |
| Empirical formula   | $C_7H_{12}FNO_4$ |
| Formula weight      | 193.18           |
| Temperature/K       | 120.0            |
| Crystal system      | orthorhombic     |
| Space group         | $Pna2_1$         |
| $a/\text{\AA}$      | 11.0907(3)       |
| $b/\text{\AA}$      | 8.2238(2)        |
| $c/\text{\AA}$      | 9.5589(3)        |

|  |   |
|--|---|
| $\alpha/^\circ$                                | 90.00   |
| $\beta/^\circ$                                 | 90.00   |
| $\gamma/^\circ$                                | 90.00   |
| Volume/ $\text{\AA}^3$                         | 871.85(4)   |
| Z  | 4   |
| $\rho_{\text{calc}}/\text{g/cm}^3$             | 1.472   |
| $\mu/\text{mm}^{-1}$                           | 0.133   |
| F(000)   | 408.0   |
| Crystal size/ $\text{mm}^3$                    | $0.37 \times 0.25 \times 0.08$                                |
| Radiation                                      | MoK $\alpha$ ( $\lambda = 0.71073$ )                          |
| 2 $\theta$ range for data collection/ $^\circ$ | 6.16 to 60  |
| Index ranges                                   | $-15 \leq h \leq 15, -11 \leq k \leq 11, -13 \leq l \leq 13$  |
| Reflections collected                          | 13238   |
| Independent reflections                        | 2539 [ $R_{\text{int}} = 0.0469, R_{\text{sigma}} = 0.0356$ ] |
| Data/restraints/parameters                     | 2539/1/120  |
| Goodness-of-fit on $F^2$                       | 1.036   |
| Final R indexes [ $I \geq 2\sigma(I)$ ]        | $R_1 = 0.0339, wR_2 = 0.0781$                                 |
| Final R indexes [all data]                     | $R_1 = 0.0400, wR_2 = 0.0823$                                 |
| Largest diff. peak/hole / $e \text{\AA}^{-3}$  | 0.32/-0.17  |
| Flack parameter                                | 0.1(6)  |

| Table 2 Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for 16srv100. $U_{\text{eq}}$ is defined as 1/3 of the trace of the orthogonalised $U_{\text{II}}$ 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 ( $\text{\AA}^2 \times 10^3$ ) for 16srv100. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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/ $\text{\AA}$ | Atom | Atom | Length/ $\text{\AA}$ |
|------|------|----------------------|------|------|----------------------|
| 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/ $^\circ$ | Atom | Atom | Atom | Angle/ $^\circ$ |
|------|------|------|-----------------|------|------|------|-----------------|
| 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.

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

<sup>1</sup>3/2-X,1/2+Y,-1/2+Z; <sup>2</sup>1-X,1-Y,-1/2+Z; <sup>3</sup>-1/2+X,1/2-Y,+Z; <sup>4</sup>+X,-1+Y,+Z

| Table 7 Torsion Angles for 16srv100. |          |          |          |                |          |          |          |          |                |  |
|--------------------------------------|----------|----------|----------|----------------|----------|----------|----------|----------|----------------|--|
| <b>A</b>                             | <b>B</b> | <b>C</b> | <b>D</b> | <b>Angle/°</b> | <b>A</b> | <b>B</b> | <b>C</b> | <b>D</b> | <b>Angle/°</b> |  |
| 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 <sup>4</sup> ) and Isotropic Displacement Parameters (Å <sup>2</sup> ×10 <sup>3</sup> ) for 16srv100. |          |          |          |              |
|---|----------|----------|----------|--------------|
| <b>Atom</b>   | <b>x</b> | <b>y</b> | <b>z</b> | <b>U(eq)</b> |
| 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 CH<sub>2</sub> refined with riding coordinates:

C4(H4A,H4B), C5(H5A,H5B), C6(H6A,H6B), C7(H7A,H7B)

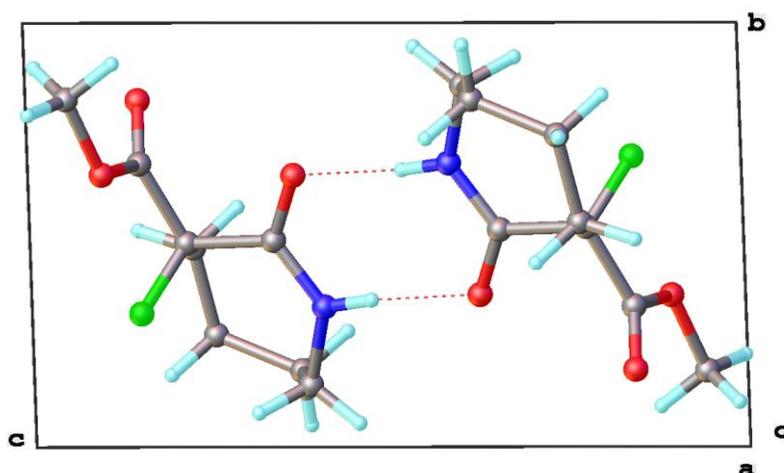
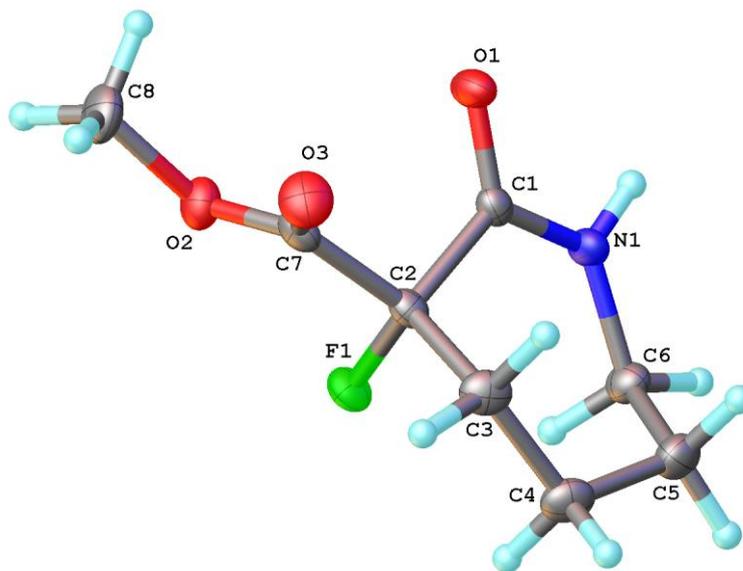
#### 2.b Idealised Me refined as rotating group:

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)



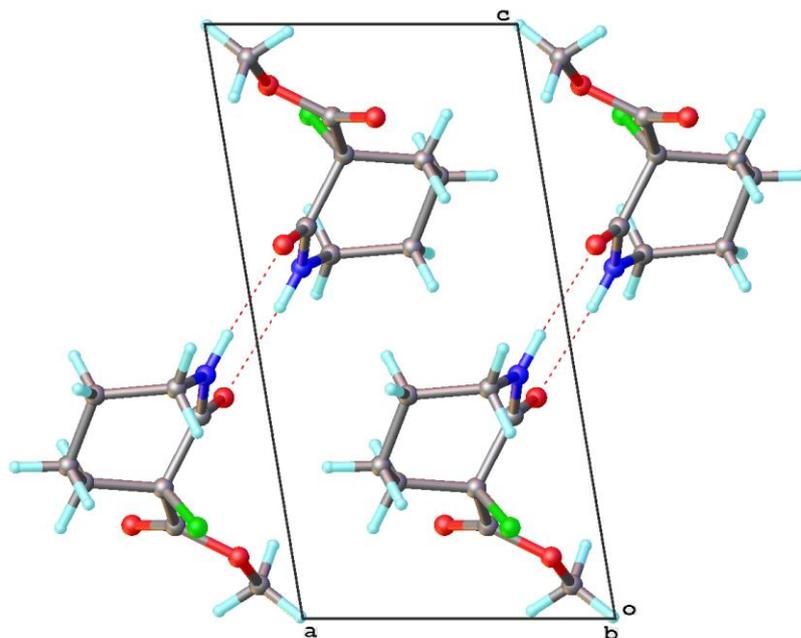


Table 1 Crystal data and structure refinement for 16srv117.

|                                    |                                      |
|------------------------------------|--------------------------------------|
| 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                                  |
| $a/\text{\AA}$                     | 5.9304(3)                            |
| $b/\text{\AA}$                     | 6.8227(3)                            |
| $c/\text{\AA}$                     | 11.3964(5)                           |
| $\alpha/^\circ$                    | 86.5454(18)                          |
| $\beta/^\circ$                     | 80.2800(17)                          |
| $\gamma/^\circ$                    | 80.4348(17)                          |
| Volume/ $\text{\AA}^3$             | 447.93(4)                            |
| Z                                  | 2                                    |
| $\rho_{\text{calc}}/\text{g/cm}^3$ | 1.403                                |
| $\mu/\text{mm}^{-1}$               | 0.120                                |
| F(000)                             | 200.0                                |
| Crystal size/ $\text{mm}^3$        | $0.25 \times 0.2 \times 0.08$        |
| Radiation                          | MoK $\alpha$ ( $\lambda = 0.71073$ ) |

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

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

| Atom | x          | y          | z          | $U_{\text{eq}}$ |
|------|------------|------------|------------|-----------------|
| 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 ( $\text{\AA}^2 \times 10^3$ ) for 16srv117. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

| 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)  |

| 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) |      |      |            |

| 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) |

| D  | H  | A               | d(D-H)/Å  | d(H-A)/Å  | d(D-A)/Å   | D-H-A/°   |
|----|----|-----------------|-----------|-----------|------------|-----------|
| N1 | H1 | O1 <sup>1</sup> | 0.858(17) | 2.020(17) | 2.8750(13) | 174.7(14) |

<sup>1</sup>-x,1-y,1-z

| 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)   |  |    |    |    |    |             |

| 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)