

[3-Methoxy-5-(methoxycarbonyl)isoxazol-4-yl](4-methoxyphenyl)iodonium 2,2,2-trifluoroacetate

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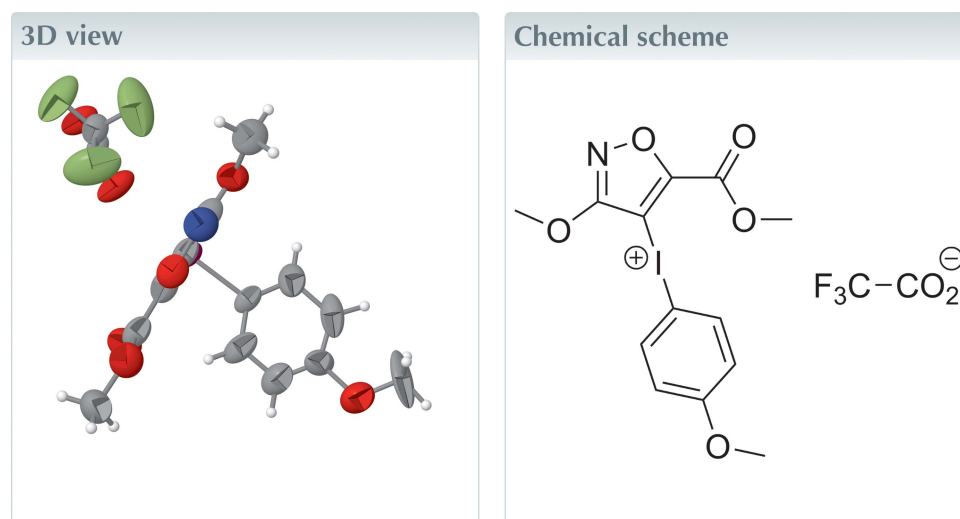
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Structural data: full structural data are available from iucrdata.iucr.org

A new isoxazole-based iodonium salt, $C_{13}H_{13}INO_5^+ \cdot C_2F_3O_2^-$, has been synthesized and structurally characterized. In the crystal, ions are linked by short $I \cdots O$ contacts to form a neutral tetra-ion aggregate. These combine with $C-H \cdots F$ and $C-H \cdots O$ interactions to form double-layered two-dimensional sheets in the (001) plane.



Structure description

Hypervalent iodine compounds exhibit attractive features of low cost, mild and selective reagents in organic synthesis (Wirth, 2005; Richardson & Wirth, 2006). These reagents serve as environmentally benign alternatives to toxic heavy-metal-based oxidants and expensive organometallic catalysts (Satam *et al.*, 2010; Wirth, 2001). The application of iodonium reagents in organic transformation encompasses areas such as C–C, C–heteroatom and heteroatom–heteroatom bond formation, oxidations, rearrangements and radical reactions (Frigerio & Santagostino, 1994; Zhdankin & Stang, 2008; Zhdankin, 2009, 2011).

A particularly important application is the reaction of diaryliodonium salts with fluorine anions, allowing the introduction of fluorine into chemical compounds of interest (Tredwell & Gouverneur 2012; Tredwell *et al.*, 2008). Furthermore, by using diaryliodonium salts, both electron-deficient and electron-rich rings can be fluorinated, allowing access to all regioisomers of a particular arene over standard S_NAr chemistry (Shah *et al.*, 1998). Moreover, these types of reaction typically require milder conditions than standard S_NAr reactions, and they can even take place in wet solvents (Chun *et al.*, 2013). Features which are privileged for the incorporation of radioactive [^{18}F]-fluoride into radiotracer molecules established for Positron Emission Tomography.

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C8-H8C\cdots O12^i$	0.98	2.58	3.475 (14)	152
$C11-H11\cdots F16^{ii}$	0.95	2.56	3.405 (17)	148
$C15-H15C\cdots O6^{iii}$	0.98	2.62	3.503 (14)	151

Symmetry codes: (i) $x, y + 1, z$; (ii) $x, y - 1, z$; (iii) $x + 1, y, z$.

The versatility of isoxazoles core components in biologically active compounds, natural products and functional materials (Abdul Manan *et al.*, 2017; Frolund *et al.*, 2002; Lee *et al.*, 2009) led us to examine the synthesis of iodonium salts bearing an isoxazole motif possessing novel structural features with the possibly of some interest as a precursor to fluoroisoxazole.

The title salt, $C_{13}H_{13}INO_5^+ \cdot C_2F_3O_2^-$, crystallizes in the space group $P\bar{1}$ with one ion pair in the asymmetric-unit (Fig. 1). In the crystal, the ring of the isoxazole group is inclined to the methoxyphenyl ring at an angle of $84.4(3)^\circ$ and the $C-I-C$ bond angle is $90.8(3)^\circ$. Short $I\cdots O$ contacts of 2.555(6) and 2.823(7) Å are observed due to the strong electrostatic interaction between two iodonium cations and two trifluoroacetate counter-ions (Fig. 2). There are also $C-H\cdots F$ and $C-H\cdots O$ interactions present (Table 1). The $C-H\cdots O$ interactions give rise to two-dimensional sheets in the (001) plane, with the $C-H\cdots F$ interactions holding the trifluoroacetate anion in place within the sheets (Fig. 3). The combination of the weak hydrogen bonds with the $I\cdots O$

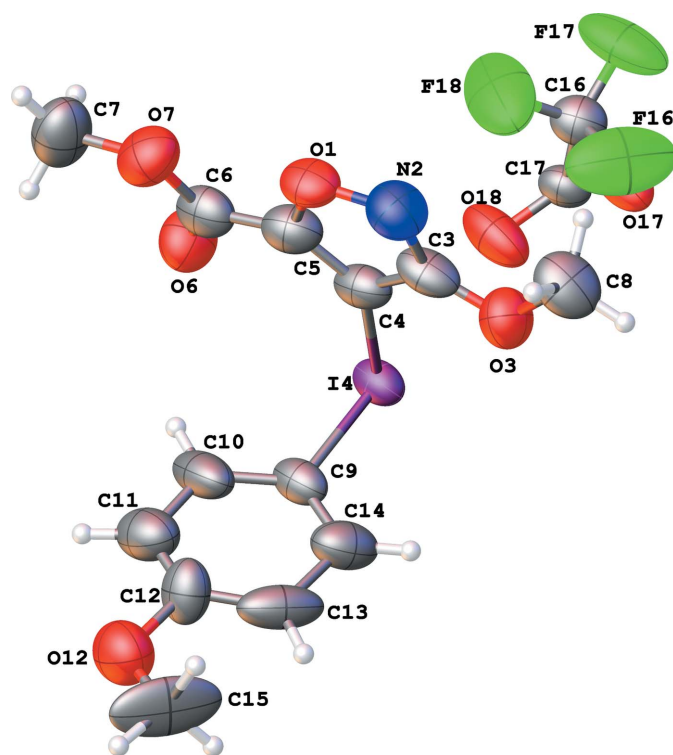


Figure 1
Molecular structures of the constituents of the asymmetric unit of the title compound, showing the atom-labelling scheme and displacement ellipsoids drawn at the 50% probability level.

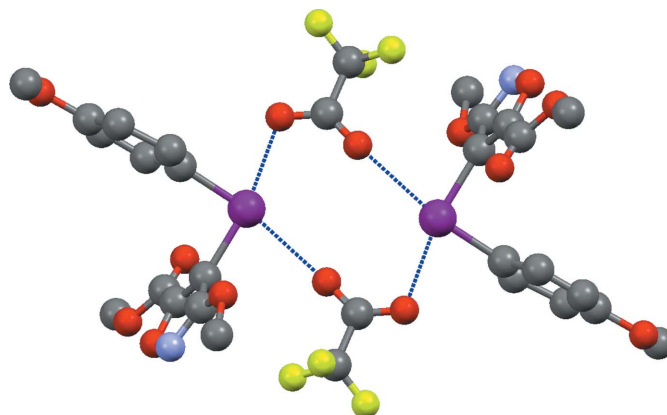


Figure 2
View down the [110] axis of the neutral, tetra-ion aggregate formed by $I\cdots O$ interactions, shown as dashed lines. Hydrogen atoms are omitted for clarity.

interactions gives rise to double-layered sheets, also in the (001) plane. These interactions are comparable to those observed in phenyl(phenylethynyl)iodonium tosylate and phenyl(phenylethynyl)iodonium trifluoroacetate salts (Dixon *et al.*, 2013).

Synthesis and crystallization

m-CPBA (70% active oxidant, 791 mg, 3.21 mmol, 1.3 eq.) was added to a solution of methyl 4-iodo-3-methoxyisoxazole-5-carboxylate (700 mg, 2.47 mmol, 1.0 eq.) in AcOH (20 ml). After stirring at $55^\circ C$ for 96 h, water (30 ml) was added to the reaction mixture followed by extraction into DCM (3×20 ml). The combined organic layers were washed with a saturated aqueous solution of Na_2CO_3 (60 ml), dried over Na_2SO_4 , filtered and concentrated under reduced pressure to afford a colourless solid, which was used without further purification.

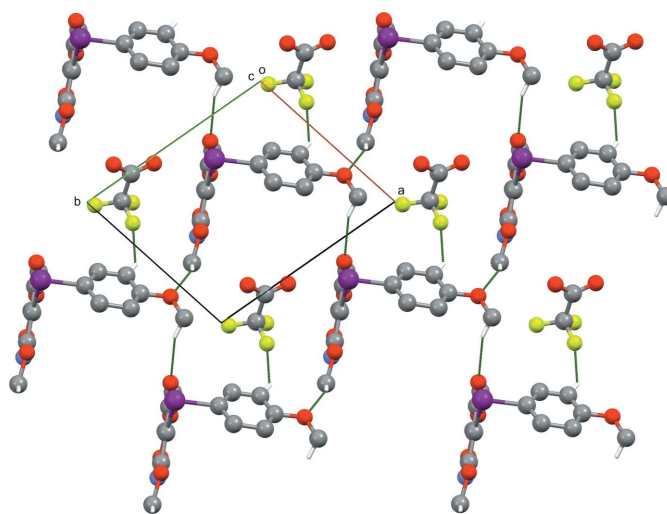


Figure 3
View down the [001] axis of the two-dimensional sheet formed by weak hydrogen-bonding interactions, shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding are omitted.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₃ H ₁₃ INO ₅ ⁺ ·C ₂ F ₃ O ₂ ⁻
<i>M_r</i>	503.17
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.436 (2), 10.750 (3), 11.338 (3)
α , β , γ (°)	113.913 (5), 97.392 (4), 98.975 (5)
<i>V</i> (Å ³)	907.2 (4)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.83
Crystal size (mm)	0.16 × 0.03 × 0.01
Data collection	
Diffractionmeter	Rigaku XtaLAB P200
Absorption correction	Multi-scan (<i>CrystalClear</i> ; Rigaku, 2014)
<i>T_{min}</i> , <i>T_{max}</i>	0.862, 0.982
No. of measured, independent and observed [<i>F</i> ² > 2.0 σ (<i>F</i> ²)] reflections	11079, 3270, 2423
<i>R_{int}</i>	0.053
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.603
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.060, 0.145, 1.04
No. of reflections	3270
No. of parameters	246
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	1.49, -0.65

Computer programs: *CrystalClear* (Rigaku, 2014), *DIRDIF99* (Beurskens *et al.*, 1999), *SHELXL2018/3* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2020), *CrystalStructure* (Rigaku, 2018), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2010).

Methyl (4-diacetoxyiodo)-3-methoxyisoxazole-5-carboxylate (281 mg, 0.7 mmol, 1.0 eq.), as a 40% mixture determined by ¹H-NMR, with methyl 4-iodo-3-methoxyisoxazole-5-carboxylate, was dissolved in DCM (10 ml) and cooled to -30°C, followed by dropwise addition of TFA (110 ml, 1.40 mmol, 2.0 eq.). The solution was stirred with the exclusion of light for 30 min, followed by 1 h at rt. The reaction mixture was re-cooled to -30°C and tributyl(4-methoxyphenyl)stannane (278 mg, 0.70 mmol, 1.0 eq.) added. The reaction was warmed to rt for the second time and left to stir overnight. The solvent was removed *in vacuo*. Upon the addition of Et₂O, the (3-methoxy-5-(methoxycarbonyl)isoxazol-4-yl)(4-methoxyphenyl)iodonium TFA salt (35 mg, 10%) crystallized. Crystals suitable for X-ray structure determination were obtained from the diffusion of diethyl ether into a dichloromethane solution of the title compound.

¹H (500 MHz, *d*₆-DMSO), δ : (p.p.m): 8.00 (2H, *d*, ³*J*_{HH} 7.2), 7.06 (2H, *d*, ³*J*_{HH} 7.2), 4.07 (3H, *s*), 4.01 (3H, *s*), 3.80 (3H, *s*); ¹³C (125 MHz, *d*₆-DMSO), δ : (p.p.m): 170.1, 162.0, 161.0, 155.3, 137.0, 117.5, 107.0, 83.9, 59.0, 55.7, 54.0; ¹⁹F (470 MHz, *d*₆-DMSO), δ : (p.p.m): -73.6 (3 F, *s*); HRMS *m/z* (ESI⁺), [*M* - TFA]⁺ calculated (C₁₃H₁₃NO₅¹²⁷I) 389.9833, found 389.9819.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The maximum residual electron density peak of 1.48 e Å⁻³ was located 1.01 Å from the I4 atom.

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References

- Abdul Manan, M. A. F., Cordes, D. B., Slawin, A. M., Bühl, M., Liao, V. W., Chua, H. C., Chebib, M. & O'Hagan, D. (2017). *Chem. Eur. J.* **23**, 10848–10852.
- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Beurskens, P. T., Beurskens, G., de Gelder, R., García-Granda, S., Israel, R., Gould, R. O. & Smits, J. M. M. (1999). *The DIRDIF99 Program System*. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- Chun, J. H., Telu, S., Lu, S. & Pike, V. W. (2013). *Org. Biomol. Chem.* **11**, 5094–5099.
- Dixon, L. I., Carroll, M. A., Gregson, T. J., Ellames, G. J., Harrington, R. W. & Clegg, W. (2013). *Eur. J. Org. Chem.* pp. 2334–2345.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Frigerio, M. & Santagostino, M. (1994). *Tetrahedron Lett.* **35**, 8019–8022.
- Frølund, B., Jørgensen, A. T., Tagmose, L., Stensbøl, T. B., Vestergaard, H. T., Engblom, C., Kristiansen, U., Sanchez, C., Krogsgaard-Larsen, P. & Liljefors, T. (2002). *J. Med. Chem.* **45**, 2454–2468.
- Lee, Y. G., Koyama, Y., Yonekawa, M. & Takata, T. (2009). *Macromolecules*, **42**, 7709–7717.
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Richardson, R. D. & Wirth, T. (2006). *Angew. Chem. Int. Ed.* **45**, 4402–4404.
- Rigaku (2014). *CrystalClear*. Rigaku Americas Corporation, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
- Rigaku (2018). *CrystalStructure*. Rigaku Americas Corporation, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
- Satam, V., Harad, A., Rajule, R. & Pati, H. (2010). *Tetrahedron*, **66**, 7659–7706.
- Shah, A., Pike, V. W. & Widdowson, D. A. (1998). *J. Chem. Soc. Perkin Trans. 1*, pp. 2043–2046.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Tredwell, M. & Gouverneur, V. (2012). *Angew. Chem. Int. Ed.* **51**, 11426–11437.
- Tredwell, M., Luft, J. A., Schuler, M., Tenza, K., Houk, K. N. & Gouverneur, V. (2008). *Angew. Chem. Int. Ed.* **47**, 357–360.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Wirth, T. (2001). *Angew. Chem. Int. Ed.* **40**, 2812–2814.
- Wirth, T. (2005). *Angew. Chem. Int. Ed.* **44**, 3656–3665.
- Zhdankin, V. V. (2009). *Arkivoc*, <https://doi.org/10.3998/ark.5550190.0010.101>.
- Zhdankin, V. V. (2011). *J. Org. Chem.* **76**, 1185–1197.
- Zhdankin, V. V. & Stang, P. J. (2008). *Chem. Rev.* **108**, 5299–5358.

full crystallographic data

IUCrData (2023). **8**, x230300 [https://doi.org/10.1107/S2414314623003000]

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[3-Methoxy-5-(methoxycarbonyl)isoxazol-4-yl](4-methoxyphenyl)iodonium 2,2,2-trifluoroacetate

Crystal data

$C_{13}H_{13}INO_5^+ \cdot C_2F_3O_2^-$

$M_r = 503.17$

Triclinic, $P\bar{1}$

$a = 8.436$ (2) Å

$b = 10.750$ (3) Å

$c = 11.338$ (3) Å

$\alpha = 113.913$ (5)°

$\beta = 97.392$ (4)°

$\gamma = 98.975$ (5)°

$V = 907.2$ (4) Å³

$Z = 2$

$F(000) = 492.00$

$D_x = 1.842$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1722 reflections

$\theta = 2.0$ – 25.3 °

$\mu = 1.83$ mm⁻¹

$T = 173$ K

Plate, colourless

$0.16 \times 0.03 \times 0.01$ mm

Data collection

Rigaku XtaLAB P200

diffractometer

Radiation source: Rotating Anode, Rigaku FR-

X

Rigaku Osmic Confocal Optical System

monochromator

Detector resolution: 5.814 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2014)

$T_{\min} = 0.862$, $T_{\max} = 0.982$

11079 measured reflections

3270 independent reflections

2423 reflections with $F^2 > 2.0\sigma(F^2)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.0$ °

$h = -10$ → 10

$k = -12$ → 12

$l = -13$ → 13

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.060$

$wR(F^2) = 0.145$

$S = 1.04$

3270 reflections

246 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 4.1326P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.49$ e Å⁻³

$\Delta\rho_{\min} = -0.65$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Carbon-bound H atoms were included in calculated positions (C—H = 0.95–0.98 Å) and refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ (sp^2) or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$ (sp^3).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I4	0.20970 (6)	0.42981 (5)	0.12013 (5)	0.0509 (2)
F16	0.2459 (9)	0.9270 (9)	0.2807 (8)	0.152 (4)
F17	0.0378 (8)	0.9854 (6)	0.3427 (6)	0.111 (3)
F18	0.1490 (12)	0.8548 (8)	0.4008 (6)	0.136 (3)
O1	0.4332 (8)	0.6834 (6)	0.5238 (5)	0.0632 (16)
O3	0.5054 (8)	0.7371 (7)	0.2570 (7)	0.0717 (18)
O6	0.1162 (9)	0.3812 (7)	0.3838 (7)	0.0777 (19)
O7	0.2682 (9)	0.4996 (8)	0.5853 (7)	0.081 (2)
O12	0.7116 (10)	0.0695 (8)	0.1381 (8)	0.094 (2)
O17	−0.0913 (8)	0.7775 (6)	0.1053 (6)	0.076 (2)
O18	0.0342 (9)	0.6423 (7)	0.1656 (7)	0.086 (2)
N2	0.5171 (10)	0.7647 (9)	0.4715 (8)	0.074 (2)
C3	0.4577 (11)	0.6993 (9)	0.3442 (9)	0.061 (3)
C4	0.3342 (10)	0.5742 (9)	0.3109 (8)	0.057 (2)
C5	0.3256 (11)	0.5714 (10)	0.4265 (9)	0.064 (2)
C6	0.2232 (12)	0.4716 (11)	0.4574 (9)	0.064 (3)
C7	0.1665 (14)	0.4074 (13)	0.6255 (13)	0.095 (4)
H7A	0.165550	0.309983	0.568918	0.143*
H7B	0.054106	0.421058	0.616962	0.143*
H7C	0.211381	0.429339	0.717581	0.143*
C8	0.6252 (12)	0.8788 (11)	0.3162 (11)	0.084 (3)
H8A	0.723922	0.876368	0.370487	0.126*
H8B	0.573436	0.950570	0.371279	0.126*
H8C	0.655593	0.900916	0.245180	0.126*
C9	0.3799 (11)	0.3064 (9)	0.1113 (8)	0.055 (2)
C10	0.3599 (15)	0.2162 (11)	0.1705 (9)	0.080 (3)
H10	0.267985	0.210169	0.209962	0.096*
C11	0.4686 (15)	0.1368 (12)	0.1732 (10)	0.083 (3)
H11	0.447999	0.068052	0.205282	0.099*
C12	0.5986 (13)	0.1563 (11)	0.1318 (11)	0.070 (3)
C13	0.6360 (12)	0.2401 (12)	0.0699 (11)	0.090 (4)
H13	0.733464	0.245200	0.036741	0.108*
C14	0.5113 (13)	0.3240 (11)	0.0579 (10)	0.077 (3)
H14	0.524458	0.384409	0.015910	0.093*
C15	0.8561 (13)	0.1021 (16)	0.1053 (14)	0.137 (7)
H15A	0.925082	0.038937	0.112025	0.206*
H15B	0.835017	0.092238	0.014517	0.206*

H15C	0.912545	0.198818	0.165580	0.206*
C16	0.1083 (10)	0.8821 (9)	0.3002 (8)	0.056 (2)
C17	0.0083 (9)	0.7542 (9)	0.1774 (8)	0.049 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I4	0.0529 (4)	0.0449 (3)	0.0430 (3)	0.0166 (2)	-0.0062 (2)	0.0102 (2)
F16	0.085 (5)	0.135 (7)	0.130 (7)	-0.041 (5)	0.028 (5)	-0.023 (5)
F17	0.099 (5)	0.068 (4)	0.098 (5)	0.038 (4)	-0.031 (4)	-0.021 (3)
F18	0.212 (9)	0.098 (5)	0.057 (4)	0.025 (5)	-0.042 (5)	0.017 (4)
O1	0.067 (4)	0.062 (4)	0.040 (3)	0.014 (3)	-0.004 (3)	0.007 (3)
O3	0.069 (4)	0.068 (4)	0.077 (5)	0.009 (3)	0.009 (4)	0.035 (4)
O6	0.075 (5)	0.071 (5)	0.073 (4)	-0.005 (4)	0.003 (4)	0.029 (4)
O7	0.080 (5)	0.101 (5)	0.063 (4)	0.023 (4)	0.009 (4)	0.037 (4)
O12	0.105 (6)	0.095 (6)	0.093 (5)	0.038 (5)	0.019 (5)	0.047 (5)
O17	0.089 (5)	0.054 (4)	0.060 (4)	0.020 (3)	-0.024 (3)	0.011 (3)
O18	0.096 (5)	0.057 (4)	0.083 (5)	0.032 (4)	-0.019 (4)	0.013 (4)
N2	0.070 (5)	0.067 (5)	0.068 (5)	0.006 (4)	-0.002 (4)	0.019 (5)
C3	0.052 (5)	0.046 (5)	0.054 (5)	0.012 (4)	-0.015 (4)	-0.002 (4)
C4	0.057 (5)	0.060 (6)	0.045 (5)	0.027 (5)	0.000 (4)	0.011 (4)
C5	0.056 (5)	0.065 (6)	0.055 (5)	0.019 (5)	0.002 (4)	0.013 (5)
C6	0.058 (6)	0.069 (7)	0.057 (6)	0.027 (5)	-0.002 (5)	0.021 (5)
C7	0.078 (8)	0.118 (10)	0.118 (10)	0.029 (7)	0.036 (7)	0.073 (9)
C8	0.065 (6)	0.067 (7)	0.091 (8)	-0.002 (5)	-0.010 (5)	0.020 (6)
C9	0.059 (5)	0.051 (5)	0.043 (4)	0.023 (4)	-0.006 (4)	0.010 (4)
C10	0.115 (9)	0.073 (7)	0.051 (5)	0.054 (6)	0.009 (5)	0.016 (5)
C11	0.099 (9)	0.090 (8)	0.062 (6)	0.044 (7)	0.016 (6)	0.028 (6)
C12	0.057 (6)	0.083 (7)	0.079 (7)	0.012 (5)	0.002 (5)	0.051 (6)
C13	0.055 (6)	0.083 (8)	0.078 (7)	0.000 (6)	0.025 (5)	-0.015 (6)
C14	0.074 (7)	0.060 (6)	0.079 (7)	0.014 (5)	0.025 (6)	0.010 (5)
C15	0.042 (6)	0.154 (13)	0.122 (11)	-0.036 (7)	0.039 (7)	-0.018 (9)
C16	0.043 (5)	0.057 (6)	0.051 (5)	0.008 (4)	-0.003 (4)	0.013 (4)
C17	0.043 (4)	0.048 (5)	0.047 (5)	0.018 (4)	0.005 (4)	0.012 (4)

Geometric parameters (Å, °)

I4—C9	2.087 (8)	C7—H7A	0.9800
I4—C4	2.092 (8)	C7—H7B	0.9800
F16—C16	1.268 (11)	C7—H7C	0.9800
F17—C16	1.290 (10)	C8—H8A	0.9800
F18—C16	1.307 (11)	C8—H8B	0.9800
O1—C5	1.347 (10)	C8—H8C	0.9800
O1—N2	1.397 (10)	C9—C14	1.353 (13)
O3—C3	1.297 (12)	C9—C10	1.388 (14)
O3—C8	1.520 (11)	C10—C11	1.353 (14)
O6—C6	1.154 (11)	C10—H10	0.9500
O7—C6	1.344 (11)	C11—C12	1.268 (15)

O7—C7	1.459 (13)	C11—H11	0.9500
O12—C15	1.355 (12)	C12—C13	1.374 (15)
O12—C12	1.449 (12)	C13—C14	1.515 (16)
O17—C17	1.221 (9)	C13—H13	0.9500
O18—C17	1.214 (10)	C14—H14	0.9500
N2—C3	1.308 (12)	C15—H15A	0.9800
C3—C4	1.445 (13)	C15—H15B	0.9800
C4—C5	1.334 (13)	C15—H15C	0.9800
C5—C6	1.453 (14)	C16—C17	1.524 (11)
C9—I4—C4	90.8 (3)	C10—C9—I4	117.9 (7)
C5—O1—N2	110.3 (7)	C11—C10—C9	121.3 (11)
C3—O3—C8	113.8 (8)	C11—C10—H10	119.3
C6—O7—C7	114.1 (9)	C9—C10—H10	119.3
C15—O12—C12	114.6 (11)	C12—C11—C10	118.4 (12)
C3—N2—O1	104.8 (8)	C12—C11—H11	120.8
O3—C3—N2	125.4 (9)	C10—C11—H11	120.8
O3—C3—C4	123.4 (8)	C11—C12—C13	127.0 (11)
N2—C3—C4	111.1 (10)	C11—C12—O12	116.1 (10)
C5—C4—C3	104.5 (8)	C13—C12—O12	116.5 (10)
C5—C4—I4	129.6 (8)	C12—C13—C14	115.5 (9)
C3—C4—I4	125.8 (7)	C12—C13—H13	122.2
C4—C5—O1	109.2 (9)	C14—C13—H13	122.2
C4—C5—C6	130.6 (9)	C9—C14—C13	115.1 (10)
O1—C5—C6	120.2 (9)	C9—C14—H14	122.5
O6—C6—O7	123.9 (10)	C13—C14—H14	122.5
O6—C6—C5	125.4 (9)	O12—C15—H15A	109.5
O7—C6—C5	110.8 (9)	O12—C15—H15B	109.5
O7—C7—H7A	109.5	H15A—C15—H15B	109.5
O7—C7—H7B	109.5	O12—C15—H15C	109.5
H7A—C7—H7B	109.5	H15A—C15—H15C	109.5
O7—C7—H7C	109.5	H15B—C15—H15C	109.5
H7A—C7—H7C	109.5	F16—C16—F17	107.8 (9)
H7B—C7—H7C	109.5	F16—C16—F18	103.2 (9)
O3—C8—H8A	109.5	F17—C16—F18	105.7 (8)
O3—C8—H8B	109.5	F16—C16—C17	110.8 (8)
H8A—C8—H8B	109.5	F17—C16—C17	115.2 (7)
O3—C8—H8C	109.5	F18—C16—C17	113.2 (8)
H8A—C8—H8C	109.5	O18—C17—O17	128.3 (8)
H8B—C8—H8C	109.5	O18—C17—C16	116.1 (7)
C14—C9—C10	122.3 (9)	O17—C17—C16	115.6 (7)
C14—C9—I4	119.7 (7)		
C5—O1—N2—C3	0.0 (9)	O1—C5—C6—O7	-7.7 (11)
C8—O3—C3—N2	-7.7 (13)	C14—C9—C10—C11	-2.6 (15)
C8—O3—C3—C4	174.8 (8)	I4—C9—C10—C11	-177.5 (8)
O1—N2—C3—O3	-177.8 (8)	C9—C10—C11—C12	6.9 (16)
O1—N2—C3—C4	-0.1 (10)	C10—C11—C12—C13	-7.7 (18)

O3—C3—C4—C5	177.9 (8)	C10—C11—C12—O12	179.4 (9)
N2—C3—C4—C5	0.1 (10)	C15—O12—C12—C11	-173.9 (10)
O3—C3—C4—I4	0.4 (12)	C15—O12—C12—C13	12.4 (14)
N2—C3—C4—I4	-177.4 (6)	C11—C12—C13—C14	3.9 (17)
C3—C4—C5—O1	-0.1 (10)	O12—C12—C13—C14	176.8 (8)
I4—C4—C5—O1	177.3 (5)	C10—C9—C14—C13	-1.1 (13)
C3—C4—C5—C6	179.6 (9)	I4—C9—C14—C13	173.7 (6)
I4—C4—C5—C6	-3.0 (15)	C12—C13—C14—C9	0.7 (13)
N2—O1—C5—C4	0.1 (10)	F16—C16—C17—O18	86.1 (11)
N2—O1—C5—C6	-179.7 (8)	F17—C16—C17—O18	-151.2 (9)
C7—O7—C6—O6	-1.6 (14)	F18—C16—C17—O18	-29.4 (12)
C7—O7—C6—C5	177.5 (8)	F16—C16—C17—O17	-96.1 (11)
C4—C5—C6—O6	-8.3 (16)	F17—C16—C17—O17	26.7 (12)
O1—C5—C6—O6	171.4 (9)	F18—C16—C17—O17	148.5 (9)
C4—C5—C6—O7	172.5 (9)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C8—H8C \cdots O12 ⁱ	0.98	2.58	3.475 (14)	152
C11—H11 \cdots F16 ⁱⁱ	0.95	2.56	3.405 (17)	148
C15—H15C \cdots O6 ⁱⁱⁱ	0.98	2.62	3.503 (14)	151

Symmetry codes: (i) $x, y+1, z$; (ii) $x, y-1, z$; (iii) $x+1, y, z$.