Crystal structure, PIXEL calculations of intermolecular interaction energies and solid-state characterization of the herbicide isoxaflutole

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In the isoxaflutole molecule [systematic name: (5-cyclopropyl-1,2-oxazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl]methanone; C15H12F3NO4S], the 1,2-oxazole and methanone fragments form an almost coplanar unit, whereas the methanone and phenyl mean planes are inclined by an angle of more than 60°. This conformation differs fundamentally from all other known examples of the 1,2-oxazol-4-yl(phenyl)methanone fragment and is ascribed to the presence of the bulky methylsulfonyl para substituent at the phenyl ring. PIXEL calculations reveal that the largest contributions to the stabilization of the crystal persist within a columnar arrangement of molecules along the twofold screw axis and in interactions between adjacent columns related by an inversion operation. Both these intra-column and inter-column motifs are dominated by the dispersion energy term but also display additional significant stabilization effects as a result of three short intermolecular C—H···O contacts involving the methanesulfonyl-O atoms.

1. Chemical context

The title compound, (I), belongs to the family of isoxazoles and was originally developed by Rhône-Poulenc Agriculture (Cain et al., 1992). Isoxaflutole is a preemergence herbicide that is used against grasses and broadleaf weeds (Luscombe et al., 1995). This compound metabolizes briskly in soils and plants by opening the ring of the isoxazole group. A diketonic derivative is formed in this process, which acts as an inhibitor of 4-hydroxyphenylpyruvate dioxygenase (HPPD) (Pallett et al., 1997; Roberts et al., 1999). Isoxaflutole is marketed in the form of suspension concentrate formulations, water-dispersible granules and wettable powders where it is either the sole active ingredient or combined with other herbicides such as flufenacet.

We have studied the solid-state properties of isoxaflutole as part of a wider investigation of herbicides and present the results in the present communication.
2. Structural commentary

The asymmetric unit of (I) contains one molecule (Fig. 1). The cyclopropyl substituent (C8, C9, C10) of the oxadazol ring is oriented such that its C8—C9 bond lies approximately parallel to the C5—O1 bond of the ring [torsion angle O1—C5—C8—C9 = −15.0 (3)°]. The methanone fragment (O7, C4, C14, C15) and the oxadazol ring (O1, N2, C3, C4, C5) form an almost planar unit. The angle between their respective mean planes is 4.4 (1)°, and the orientation of the methanone group relative to the cyclopropyl substituent of the ring is cis. By contrast, the methanone mean plane forms an angle of 64.28 (5)° with the phenyl ring (C11—C16). The orientation of the methylsulfonyl substituent at the phenyl ring is such that its S17—C22 bond is almost perpendicular to the ring mean plane, which is illustrated by the value of the pseudo-torsion angle C15···C12—S17—C22 of −83.8°.

3. Database survey

The Cambridge Structural Database (version 5.43, June 2022; Groom et al., 2016) contains 15 entries of structures displaying the 1,2-oxazol-4-yl(phenyl)methanone structure fragment (see Table S1 of the supporting information). The conformation of this structure fragment can be rationalized in terms of the relative orientation of three planar units (see Fig. 2, inset), i.e. the methanone (P1), 1,2-oxazole (P2) and phenyl (P3) fragments. In each of the previous examples, the plane of the methanone fragment tends to approach coplanarity with the phenyl ring. The corresponding interplanar angle (P1, P3) ranges between 1.6° and 28.7°. In turn, the methanone and 1,2-oxazole mean planes (P1, P2) form angles in the range from 42.3° to 86.9°. The diagram in Fig. 2 illustrates that for a given molecule, a smaller (P1, P2) angle is generally correlated with a wider (P1, P3) angle and vice versa. Apart from (I), ortho substituents at the phenyl ring are present only in DUHKOI (Cl and F; 28.7°/46.6°) and KOOGOM (—OH; 79.8°/2.4°), which displays an intramolecular O—H···O(methanone) bond. The molecules in the sample group have bulky substituents at both the 3- and 5-positions of the 1,2-oxazol ring, except for (I), YELQAK and YELQE0, which have just one such substituent (supporting information, Table S1). The plot of (P1, P2) against (P1, P3) angles in Fig. 2 illustrates the uniqueness of the conformation of (I) with almost coplanar methanone and 1,2-oxazole units (see previous section), whilst the methanone and phenyl rings planes form an angle (P1, P2) of 64.28 (5)°. This unusual conformation is probably due to the bulky methanesulfonyl group as an ortho substituent of the phenyl ring of (I).

4. Supramolecular features

The isoxaflutole molecule does not contain any classical hydrogen-bond donor groups. However, two significant short intermolecular C—H···O contacts are found between molecules related by a twofold screw operation (Table 1). The first of these, C16—H16···O21i involves a CH group of the phenyl ring and a methanesulfonyl-O atom (H16···O21i = 2.33 Å). A somewhat longer C10—H10A···O18 contact is formed between the other methanesulfonyl-O atom and the cyclo-
propyl group (H10A···O18i = 2.63 Å). A column-like structure of molecules linked by these contacts propagates parallel to the b axis (Fig. 3). Moreover, columnar structures related by a glide mirror operation of this kind form a layer motif along the c axis with short (1,2-oxazol) C3—H3/C1/C1/C1O21ii(methane-ethanesulfonyl) contacts (H3/C1/C1/C1O21ii = 2.71 Å; Table 1).

Parallel stacking of the these supramolecular bc layers in the a-axis direction results in multiple F/C1/C1/C1Fa and F/C1/C1/C1H interlayer contacts.

5. Quantitative analysis of intermolecular interactions

Intermolecular interaction energies were calculated with the semi-classical density sums (SCDS-PXEL) method using the program OPiX (Gavezzotti, 2007, 2011). C—H distances were recalculated to standard lengths and an electron-density map was calculated at the MP2/6-31G(d,p) level using Gaussian09 (Frisch et al., 2009). The obtained lattice energy of −140 kJ mol$^{-1}$ can be partitioned into contributions from Coulombic ($E_{\text{Col}} = -56.6$ kJ mol$^{-1}$), polarization ($E_{\text{pol}} = -20.7$ kJ mol$^{-1}$), dispersion ($E_{\text{dis}} = -151.2$ kJ mol$^{-1}$) and repulsion ($E_{\text{rep}} = 88.2$ kJ mol$^{-1}$) terms. Their relative values indicate that dispersion energy and electrostatic (Coulombic + polarization) energy contribute with 66% and 34%, respectively, to the stabilization of the crystal structure.

### Table 2

| Index | Symmetry operations | Symmetry element | d (Å) | $E_{\text{Col}}$ | $E_{\text{pol}}$ | $E_{\text{energy-dispersive}}$ | $E_{\text{rep}}$ | Motif | Interactions |
|-------|---------------------|------------------|-------|-----------------|-----------------|-------------------------------|-----------------|-------|--------------|
| 1a,b  | $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$, $2i$ | c | 5.367 | -28.5 | -10.1 | -58.7 | 40.0 | -57.2 | column | C16—H16···O21ii; C10—H10A···O18i |
| 3a,b  | $x, \frac{1}{2} - y, \frac{1}{2} + z$; $x, \frac{1}{2} - y, \frac{1}{2} + z$ | c | 7.002 | -15.9 | -7.1 | -33.4 | 18.7 | -37.6 | layer | C3—H3···O21ii |
| 5     | $1 - x, 1 - y, -z$ | T | 7.261 | -9.8 | -6.3 | -26.0 | 17.0 | -25.2 | layer | |
| 6     | $2 - x, 1 - y, 1 - z$ | T | 8.141 | -6.3 | -2.5 | -21.5 | 10.3 | -19.9 | layer | |
| 7     | $x - 1, x - 1, y, 1 - z$ | T | 9.686 | -5.8 | -1.2 | -14.5 | 8.1 | -13.4 | stack | |
| 8a,b  | $x, \frac{1}{2} - y, \frac{1}{2} + z$; $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$ | c | 12.086 | -6.6 | -1.0 | -9.7 | 3.2 | -8.1 | stack | |
| 10a,b | $x, y, z$; $x + 1, y, z$ | 1 | 13.569 | -2.2 | -0.8 | -6.4 | 5.0 | -4.3 | stack | |
| 12    | $-x, 1 - y, -z$ | T | 14.807 | -1.8 | -0.8 | -7.4 | 5.9 | -4.1 | stack | |

Figure 3

Molecules related by a twofold screw operation form two short C—H···O contacts, resulting in a columnar arrangement along the b axis (motif 1a,b).

Figure 4

Cluster consisting of a central molecule (orange) and neighbouring molecules representing the twelve most important molecule/molecule interactions (see Table 3). The interactions 1a,b (blue molecules) constitute a column along the twofold screw axis, whilst 3a,b, 5 and 6 are interactions between adjacent columns related by a c glide mirror operation.
Considering the individual interaction energies computed for pairs of molecules, the largest absolute total contribution by far \( E_{\text{tot}} = -57.2 \, \text{kJ mol}^{-1} \) is obtained for two symmetry-equivalent interactions between a central and two neighbouring molecules related to each other by twofold screw operations (denoted as 1a,b in Table 2 and Figs. 3, 4). The sum of total energies of all molecule/molecule interactions in the crystal \( E_{\text{tot,S}} = -144.8 \, \text{kJ mol}^{-1} \), which means that this columnar motif parallel to the \( b \) axis (see above) alone accounts for approximately 40% of the stabilization of the structure. This arrangement is associated with a large contact area of van der Waals surfaces (Fig. 4) and also with significant Coulombic and polarization terms \( E_{\text{Col}} = -28.5 \, \text{kJ mol}^{-1} \) and \( E_{\text{pol}} = -10.1 \, \text{kJ mol}^{-1} \), which may confirm the attractive nature of the short intermolecular \( \text{C1}--\text{H1} \) contacts discussed in the previous section (Table 1, Fig. 3).

Another set of two symmetry-equivalent interactions (denoted as 3a,b; \( E_{\text{tot}} = -36.7 \, \text{kJ mol}^{-1} \)) are associated with glide mirror interactions, i.e. the assembly of neighbouring column motifs into a layer structure along the \( c \) axis. Significant Coulombic \( E_{\text{Col}} = -15.9 \, \text{kJ mol}^{-1} \) and polarization \( E_{\text{pol}} = -7.3 \, \text{kJ mol}^{-1} \) terms, coinciding with the short \( \text{C}--\text{H} \cdots \text{O}1^{\text{ii}} \) contact mentioned in the previous section (Table 1), are observed in addition to the dominant dispersion energy contributions \( E_{\text{disp}} = -33.4 \, \text{kJ mol}^{-1} \). The diagram in Fig. 4 shows a central molecule and its twelve most important molecular neighbours, which together account for approximately 96% of the sum of pairwise PIXEL energies (see section 2 of the supporting information). Altogether, intra-column (along the \( b \) axis) interactions and interaction between neighbouring columns (along the \( c \) axis) contribute approximately with 42% and 43%, respectively, to the stabilization of the crystal structure. The rest (15%) originates from the stacking of molecular \( bc \) layers in the \( a \)-axis direction.

### 6. Synthesis and crystallization

Isoxaliflute (technical quality) was recrystallized from a hot saturated acetonitrile (p.a.) solution yielding a colourless crystalline product used for further characterization. The reported form was also the only crystalline phase encountered in the reported form was also the only crystalline phase encountered and included as rigid groups allowed to rotate but not tip (C—H = 0.98 Å). H atoms bonded to aromatic CH (C—H = 0.95 Å), secondary CH2 and tertiary CH carbon atoms (C—H = 0.99 Å) were positioned geometrically. The \( U_{\text{iso}} \) parameters of all H atoms were refined freely. Two outlier reflections (102, 202) were omitted from the final refinement.

### 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were identified in difference-Fourier maps. Methyl H atoms were idealized and included as rigid groups allowed to rotate but not tip (C—H = 0.98 Å). H atoms bonded to aromatic CH (C—H = 0.95 Å), secondary CH2 and tertiary CH carbon atoms (C—H = 0.99 Å) were positioned geometrically. The \( U_{\text{iso}} \) parameters of all H atoms were refined freely. Two outlier reflections (102, 202) were omitted from the final refinement.

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### Table 3

**Experimental details.**

| Property | Value |
|----------|-------|
| Crystal data | C15H12F3NO4S |
| Chemical formula | C15H12F3NO4S |
| Molar mass | 359.32 |
| Crystal system, space group | Monoclinic, \( P2_1/c \) |
| Temperature (K) | 193 |
| \( \beta \) (\( \text{Å} \)) | 118.530 (15) |
| \( Z \) | 4 |
| Radiation type | Mo Ka |
| \( \mu \) (mm\(^{-1}\)) | 0.27 |
| Crystal size (mm) | 0.25 × 0.10 × 0.08 |
| Data collection | Xcalibur, Ruby, Gemini ultra |
| Absorption correction | Multi-scan (CrysAlis PRO; Rigaku OD, 2020) |
| No. of reflections | 9962, 3275, 2667 |
| \( R \) (\( I > 2\sigma(I) \)) reflections | 0.036 |
| \( R_{\text{ref}} \) | 0.641 |
| Refinement | |
| \( R_{\text{F}} \) | 0.038, 0.097, 1.02 |
| No. of reflections | 3275 |
| No. of parameters | 231 |
| H-atom treatment | Only H-atom displacement parameters refined |
| \( \Delta \rho_{\text{max}}, \Delta \rho_{\text{min}} \) (e \( \text{Å}^{-3} \)) | 0.31, −0.35 |

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Computer programs: CrysAlis PRO (Rigaku OD, 2020), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), XP (Bruker, 1998), Mercury (Macrae et al., 2020), PLATON (Spek, 2020) and publICIF Westrip (2010).
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Crystal structure, PIXEL calculations of intermolecular interaction energies and solid-state characterization of the herbicide isoxaflutole

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Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2020); cell refinement: CrysAlis PRO (Rigaku OD, 2020); data reduction: CrysAlis PRO (Rigaku OD, 2020); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: XP (Bruker, 1998) and Mercury (Macrae et al., 2020); software used to prepare material for publication: PLATON (Spek, 2020) and publCIF Westrip (2010).

(5-Cyclopropyl-1,2-oxazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl]methanone

Crystal data

\[ \text{C}_15\text{H}_{12}\text{F}_3\text{NO}_4\text{S} \]

Mr = 359.32

Monoclinic, \( P2_1/c \)

\( a = 13.5689 \) (16) Å

\( b = 9.2906 \) (8) Å

\( c = 13.4358 \) (15) Å

\( \beta = 118.530 \) (15)°

\( V = 1488.1 \) (3) Å³

\( Z = 4 \)

\( F(000) = 736 \)

\( D_\text{x} = 1.604 \text{ Mg m}^{-3} \)

Mo \( K\alpha \) radiation, \( \lambda = 0.71073 \) Å

Cell parameters from 2902 reflections

\( \theta = 5.0–29.8° \)

\( \mu = 0.27 \text{ mm}^{-1} \)

\( T = 193 \) K

Prism, colourless

0.25 × 0.10 × 0.08 mm

Data collection

Xcalibur, Ruby, Gemini ultra diffractometer

Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 10.3575 pixels mm⁻¹

\( \omega \) scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2020)

\( T_{\text{min}} = 0.925, T_{\text{max}} = 1.000 \)

9962 measured reflections

3275 independent reflections

2667 reflections with \( I > 2\sigma(I) \)

\( R_{\text{int}} = 0.036 \)

\( \theta_{\text{max}} = 27.1°, \theta_{\text{min}} = 2.8° \)

\( h = -17\rightarrow16 \)

\( k = -11\rightarrow9 \)

\( l = -17\rightarrow17 \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.038 \)

\( wR(F^2) = 0.097 \)

\( S = 1.02 \)

3275 reflections

231 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

Only H-atom displacement parameters refined

\( w = 1/[(\sigma^2(F^2) + (0.0428P)^2 + 0.6729P)] \)

where \( P = (F^2 + 2F^2)/3 \)
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.31 e Å^{-3}
Δρ_{min} = −0.35 e Å^{-3}

Extinction correction: SHELXL,

Fc^2 = kFc[1+0.001xFc^2/\sin(2θ)]^{1/4}
Extinction coefficient: 0.0203 (15)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| x       | y       | z       | U_{eq} | U_{eq}^* |
|---------|---------|---------|--------|---------|
| O1      | 0.24820 (10) | 0.73141 (14) | 0.02968 (10) | 0.0294 (3) |
| N2      | 0.32693 (13) | 0.75772 (18) | −0.01032 (12) | 0.0306 (4) |
| C3      | 0.42144 (15) | 0.7107 (2) | 0.06926 (14) | 0.0258 (4) |
| H3      | 0.4898 | 0.7158 | 0.0662 | 0.033 (5)* |
| C4      | 0.41253 (14) | 0.65039 (18) | 0.16220 (13) | 0.0217 (4) |
| C5      | 0.30138 (14) | 0.66771 (19) | 0.13150 (13) | 0.0225 (4) |
| C6      | 0.49824 (14) | 0.58226 (19) | 0.26500 (14) | 0.0242 (4) |
| O7      | 0.47773 (11) | 0.52442 (17) | 0.33418 (11) | 0.0398 (4) |
| C8      | 0.23389 (15) | 0.6311 (2) | 0.18562 (15) | 0.0283 (4) |
| H8      | 0.2763 | 0.6121 | 0.2689 | 0.051 (6)* |
| C9      | 0.11929 (16) | 0.6956 (2) | 0.14281 (18) | 0.0364 (5) |
| H9A     | 0.0941 | 0.7183 | 0.1991 | 0.047 (6)* |
| H9B     | 0.0930 | 0.7637 | 0.0785 | 0.061 (8)* |
| C10     | 0.12928 (17) | 0.5426 (2) | 0.11990 (19) | 0.0381 (5) |
| H10A    | 0.1094 | 0.5152 | 0.0413 | 0.050 (7)* |
| H10B    | 0.1105 | 0.4698 | 0.1620 | 0.059 (7)* |
| C11     | 0.61644 (14) | 0.58208 (18) | 0.28108 (13) | 0.0216 (4) |
| C12     | 0.70654 (13) | 0.65149 (18) | 0.37110 (13) | 0.0193 (3) |
| C13     | 0.81286 (13) | 0.64965 (18) | 0.38042 (13) | 0.0203 (3) |
| H13     | 0.8729 | 0.6992 | 0.4408 | 0.024 (5)* |
| C14     | 0.83109 (14) | 0.57524 (18) | 0.30129 (13) | 0.0206 (3) |
| C15     | 0.74423 (14) | 0.50231 (18) | 0.21388 (14) | 0.0234 (4) |
| H15     | 0.7574 | 0.4491 | 0.1610 | 0.035 (5)* |
| C16     | 0.63762 (14) | 0.50723 (19) | 0.20371 (14) | 0.0255 (4) |
| H16     | 0.5778 | 0.4584 | 0.1426 | 0.031 (5)* |
| S17     | 0.69237 (4) | 0.74778 (5) | 0.47805 (3) | 0.02312 (14) |
| O18     | 0.78878 (12) | 0.83833 (16) | 0.53321 (11) | 0.0387 (4) |
| C19     | 0.94598 (15) | 0.5802 (2) | 0.31081 (14) | 0.0258 (4) |
| F20     | 1.02531 (9) | 0.53642 (16) | 0.41237 (10) | 0.0490 (4) |
| O21     | 0.58332 (12) | 0.81258 (15) | 0.42868 (11) | 0.0375 (4) |
| C22     | 0.69982 (18) | 0.6145 (2) | 0.57370 (15) | 0.0336 (4) |
| H22A    | 0.7714 | 0.5632 | 0.6026 | 0.048 (7)* |
| H22B    | 0.6378 | 0.5465 | 0.5351 | 0.057 (7)* |
| H22C    | 0.6945 | 0.6594 | 0.6370 | 0.046 (6)* |
| F23     | 0.95559 (9) | 0.49764 (13) | 0.23536 (10) | 0.0394 (3) |
| F24     | 0.97390 (10) | 0.71318 (13) | 0.29720 (12) | 0.0467 (3) |
### Atomic displacement parameters (Å²)

| Atom | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|------|-----------|-----------|-----------|-----------|-----------|-----------|
| O1   | 0.0211 (7)| 0.0400 (8)| 0.0249 (6)| 0.0034 (5)| 0.0093 (5)| 0.0067 (5)|
| N2   | 0.0295 (9)| 0.0387 (10)| 0.0257 (7)| −0.0010 (7)| 0.0150 (7)| 0.0041 (6)|
| C3   | 0.0232 (9)| 0.0302 (10)| 0.0261 (8)| −0.0031 (7)| 0.0133 (7)| 0.0004 (7)|
| C4   | 0.0185 (8)| 0.0246 (9)| 0.0229 (8)| −0.0039 (7)| 0.0106 (7)| −0.0007 (7)|
| C5   | 0.0198 (8)| 0.0243 (9)| 0.0214 (8)| −0.0009 (7)| 0.0081 (7)| 0.0002 (7)|
| C6   | 0.0188 (8)| 0.0295 (10)| 0.0249 (8)| −0.0023 (7)| 0.0110 (7)| 0.0016 (7)|
| O7   | 0.0245 (7)| 0.0600 (10)| 0.0369 (7)| 0.0013 (7)| 0.0162 (6)| 0.0207 (7)|
| C8   | 0.0191 (9)| 0.0374 (11)| 0.0299 (9)| −0.0001 (8)| 0.0129 (7)| 0.0032 (8)|
| C9   | 0.0270 (10)| 0.0378 (11)| 0.0528 (12)| 0.0047 (9)| 0.0259 (9)| 0.0006 (9)|
| C10  | 0.0296 (11)| 0.0376 (11)| 0.0547 (12)| −0.0076 (9)| 0.0263 (10)| −0.0072 (9)|
| C11  | 0.0176 (8)| 0.0243 (9)| 0.0230 (8)| 0.0003 (7)| 0.0099 (7)| 0.0054 (6)|
| C12  | 0.0181 (8)| 0.0202 (8)| 0.0207 (7)| 0.0016 (6)| 0.0100 (6)| 0.0020 (6)|
| C13  | 0.0171 (8)| 0.0213 (8)| 0.0207 (8)| −0.0003 (6)| 0.0076 (6)| 0.0004 (6)|
| C14  | 0.0193 (8)| 0.0210 (8)| 0.0235 (8)| 0.0024 (6)| 0.0118 (7)| 0.0042 (6)|
| C15  | 0.0263 (9)| 0.0231 (9)| 0.0235 (8)| 0.0000 (7)| 0.0140 (7)| −0.0008 (7)|
| C16  | 0.0227 (9)| 0.0281 (10)| 0.0240 (8)| −0.0061 (7)| 0.0097 (7)| −0.0044 (7)|
| S17  | 0.0255 (3)| 0.0244 (2)| 0.0244 (2)| 0.00307 (17)| 0.01593 (19)| 0.00053 (16)|
| O18  | 0.0450 (9)| 0.0421 (8)| 0.0388 (7)| −0.0153 (7)| 0.0280 (7)| −0.0174 (6)|
| C19  | 0.0238 (9)| 0.0284 (10)| 0.0296 (9)| 0.0024 (7)| 0.0164 (7)| 0.0029 (7)|
| F20  | 0.0193 (6)| 0.0889 (10)| 0.0363 (6)| 0.0101 (6)| 0.0112 (5)| 0.0143 (6)|
| O21  | 0.0378 (8)| 0.0411 (8)| 0.0414 (8)| 0.0201 (6)| 0.0252 (7)| 0.0109 (6)|
| C22  | 0.0411 (12)| 0.0370 (11)| 0.0263 (9)| 0.0069 (9)| 0.0191 (9)| 0.0082 (8)|
| F23  | 0.0358 (7)| 0.0455 (7)| 0.0497 (7)| 0.0046 (5)| 0.0306 (6)| −0.0083 (5)|
| F24  | 0.0436 (7)| 0.0327 (7)| 0.0844 (9)| −0.0068 (6)| 0.0471 (7)| 0.0000 (6)|

### Geometric parameters (Å, °)

| Bond | Distance | Angle |
|------|----------|-------|
| O1—C5 | 1.341 (2) | C11—C12 | 1.400 (2) |
| O1—N2 | 1.4276 (19) | C12—C13 | 1.387 (2) |
| N2—C3 | 1.292 (2) | C12—S17 | 1.7794 (16) |
| C3—C4 | 1.426 (2) | C13—C14 | 1.386 (2) |
| C3—H3 | 0.9500 | C13—H13 | 0.9500 |
| C4—C5 | 1.370 (2) | C14—C15 | 1.380 (2) |
| C4—C6 | 1.458 (2) | C14—C19 | 1.503 (2) |
| C5—C8 | 1.456 (2) | C15—C16 | 1.387 (2) |
| C6—O7 | 1.215 (2) | C15—H15 | 0.9500 |
| C6—C11 | 1.513 (2) | C16—H16 | 0.9500 |
| C8—C9 | 1.501 (3) | S17—O18 | 1.4291 (14) |
| C8—C10 | 1.508 (3) | S17—O21 | 1.4333 (14) |
| C8—H8 | 1.0000 | S17—C22 | 1.7521 (18) |
| C9—C10 | 1.474 (3) | C19—F23 | 1.326 (2) |
| C9—H9A | 0.9900 | C19—F24 | 1.330 (2) |
| C9—H9B | 0.9900 | C19—F20 | 1.335 (2) |
| C10—H10A | 0.9900 | C22—H22A | 0.9800 |
| C10—H10B | 0.9900 | C22—H22B | 0.9800 |
### Hydrogen-bond geometry (Å, °)

| D—H···A          | D—H | H···A | D···A   | D—H···A |
|------------------|------|-------|---------|---------|
| C16—H16···O21\(i\) | 0.95 | 2.35  | 3.214 (2) | 151     |
| C10—H104···O18\(i\) | 0.99 | 2.63  | 3.355 (2) | 130     |
| C3—H3···O21\(ii\)   | 0.95 | 2.71  | 3.522 (2) | 144     |

Symmetry codes: (i) \(-x+1, y-1/2, -z+1/2\); (ii) \(x, -y+3/2, z-1/2\).