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Three trifluoromethyl-substituted protoporphyrinogen IX oxidase inhibitors

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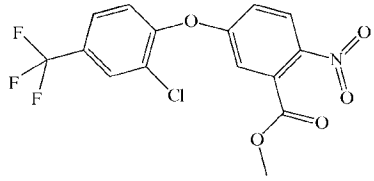
The structures of methyl 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate, $C_{15}H_9ClF_3N_3O_5$, (I), methyl 2-chloro-5-[3-methyl-2,6-dioxo-4-(trifluoromethyl)-1,2,3,6-tetrahydropyrimidin-1-yl]benzoate, $C_{14}H_{10}ClF_3N_2O_4$, (II), and 2-[4-chloro-2-fluoro-5-(prop-2-ynyloxy)phenyl]-4-(trifluoromethyl)piperidine-2,6-dione, $C_{15}H_{10}ClF_4NO_3$, (III), are similar in their dihedral angles and in the distances between the farthest two atoms. There are two independent molecules in the structure of (I). The dihedral angles between the two aromatic rings in each molecule in (I), between the benzene and tetrahydropyrimidine rings in (II), and between the benzene ring and the five-atom planar portion of the piperidine-2,6-dione ring in (III) are 80.78 (11)/89.75 (11), 89.13 (9) and 87.52 (13)°, respectively. The distances between the farthest two atoms, *viz*. O···F in the two molecules of (I), and Cl···F in (II) and (III), are 11.763 (7)/11.953 (6), 10.734 (10) and 10.889 (9) Å, respectively. In all three crystal structures, the molecules are linked to generate sheets of molecules *via* C—H···O interactions.

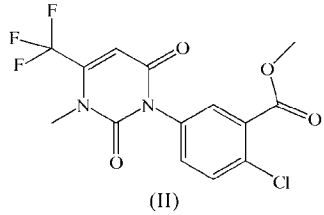
Comment

The protoporphyrinogen IX oxidase inhibitors are structurally very diverse, ranging from diphenyl ethers to 1-heterocycle-2,4,5-trisubstituted benzenes (Tomlin, 2003). They inhibit the activity of protoporphyrinogen IX oxidase (PPO) by binding competitively to the same active site as the substrate protoporphyrinogen IX (Matringe & Scalla, 1988; Duke *et al.*, 1989).

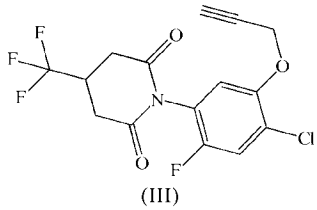
In order to discover new herbicides and to study the interaction between the enzyme and its inhibitors, a number of groups have carried out studies of the structure–activity relationship (SAR) on herbicidal diphenyl ethers and 1-heterocycle-2,4,5-trisubstituted benzenes (Boger & Wakabayashi, 1999). The crystal structures of some PPO inhibitors have played an important role in SAR and QSAR (quantitative structure–activity relationship) studies, especially in 3D-QSAR (three-dimensional quantitative structure–activity relationship) studies (Dayan & Allen, 2000; Kohno *et al.*, 1993; Nandihalli *et al.*, 1992).



(I)



(II)



(III)

At present, only six crystal structures of PPO inhibitors are available in the literature, namely 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoic acid (Acifluorfen; Kennard *et al.*, 1987), 2-(4-chlorophenyl)-1,3,4,5,6,7-hexahydroisoindole-1,3-dione (Chlorophthalim), 3-(4-chloro-5-cyclopentyloxy-2-fluorophenyl)-5-(isopropylidene)oxazolidine-2,4-dione and 2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl)-benzene (Kohno *et al.*, 1993), and 2-[4-chloro-2-fluoro-5-(prop-2-ynyloxy)phenyl]-1,3,4,5,6,7-hexahydroisoindole-1,3-dione and 2-[4-chloro-2-fluoro-5-(prop-2-ynyloxy)phenyl]-*cis*-1,3,3a,4,7,7a-hexahydroisoindole-1,3-dione (Li *et al.*, 2005). More crystal structures are needed for a programme of 3D-QSAR studies on PPO inhibitors carried out by our group, and we report here the crystal structures of methyl 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate, (I), methyl 2-chloro-5-[3-methyl-2,6-dioxo-4-(trifluoromethyl)-1,2,3,6-tetrahydropyrimidin-1-yl]benzoate, (II), and 2-[4-chloro-2-fluoro-5-(prop-2-ynyloxy)phenyl]-4-(trifluoromethyl)piperidine-2,6-dione, (III), all of which contain a

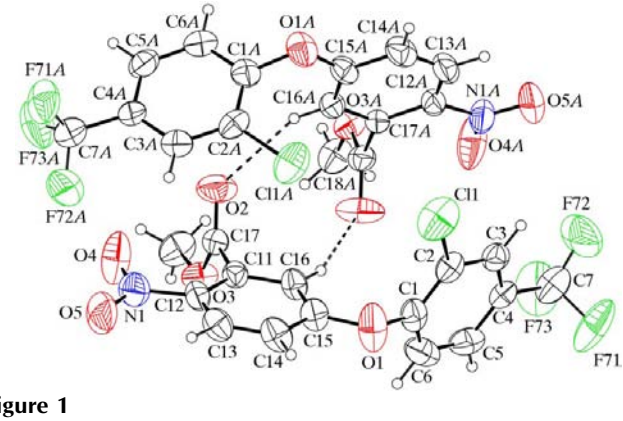


Figure 1
A view of the two independent molecules of (I), with the atom-numbering schemes. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The F atoms of the CF_3 groups are unequally disordered over two orientations; for clarity, only the major orientation is shown in each case.

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trifluoromethyl group, which is thought to be of biological importance, and all of which show high PPO-inhibiting and herbicidal activities (Li, 2005).

The asymmetric unit of (I) contains two independent molecules, as shown in Fig. 1. These are related by a pseudo-twofold axis at ($\frac{1}{4}$, y, $\frac{1}{2}$). The conformations of the two molecules are similar, with the dihedral angles between the aromatic rings being 80.78 (11)° in one molecule and 89.75 (11)° in the other. Relevant torsion angles are C2—C1—O1—C15 = 103.8 (5)° and C2*A*—C1*A*—O1*A*—C15*A* = 82.1 (6)°. The distances between the farthest two atoms are thought to be significant in terms of biological activity and are O5···F71 = 11.763 (7) Å and O5*A*···F71*A* = 11.953 (6) Å. The planes of the carboxylate groups are rotated out of the associated aromatic ring by 83.44 (15) and 88.78 (15)° for carboxylate groups C11/C17/O1/O2 and C11*A*/C17*A*/O1*A*/O2*A*, respectively; the corresponding out-of-plane rotation angles for the 2-nitro groups are 8.5 (4) and 6.5 (3)°. Compound (I) is the methyl ester of Acifluorfen (Kennard *et al.*, 1987); the torsion angle corresponding to C2—C1—O1—C15 is 89.2° in Acifluorfen, and the corresponding longest O···F distance between a nitro O atom and the farthest F atom is 11.83 Å.

The packing of (I) is controlled by C—H···O hydrogen bonds. Checks using *PLATON* (Spek, 2003) show that there are no C—H···π or π–π interactions. The two molecules of the asymmetric unit are weakly linked by C—H···O interactions along C16—H16···O2*A* and C16*A*—H16*A*···O2 (Table 1), generating $R_2^2(10)$ rings (Bernstein *et al.*, 1995). Molecules related by inversion centres at (0, $\frac{1}{2}$, $\frac{1}{2}$), ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$), (1, $\frac{1}{2}$, $\frac{1}{2}$), *etc.*, are then linked by further C—H···O interactions, involving C6—H6 and C6*A*—H6*A* and adjacent nitro O atoms (Table 1), generating $R_2^2(20)$ rings (Fig. 2), resulting in chains of rings along [100]. Parallel chains are propagated by the *c*-glide operation, and these chains are linked by C—H···O interactions involving C5—H5 and C5*A*—H5*A* with adjacent carboxyl O atoms (Table 1), generating corrugated sheets in the (010) plane.

The molecular structure of (II) is shown in Fig. 3. The tetrahydropyrimidine ring adopts a planar conformation and the mean deviation from the plane is 0.0081 Å. The dihedral angle between the tetrahydropyrimidine and benzoate ring planes is 89.13 (9)°. The carboxylate group is rotated 38.84 (12)° about the C11—C17 bond out of the benzoate ring plane. The distance between the farthest two atoms, Cl1···F72, is 10.734 (10) Å.

Molecules of (II) are linked to form centrosymmetric $R_2^2(8)$ dimers *via* C—H···O interactions involving C5—H5 and an adjacent pyrimidine carbonyl O atom (Table 2). Further C—H···O interactions involving C16—H16 and an adjacent (screw-axis related) carboxyl atom, *viz*. O3 (Table 2), generate sheets of molecules in the (101) plane (Fig. 4).

In compound (III) (Fig. 5), the piperidine-2,6-dione ring adopts an envelope conformation, with atom C4 disordered

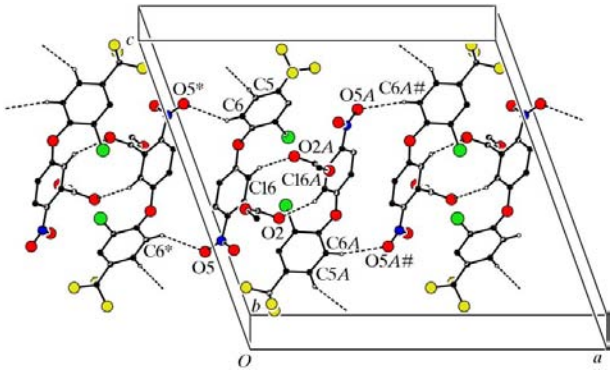

Figure 2
A view of a chain of molecules of (I) linked by weak C—H···O interactions along [100]. Atoms labelled with an asterisk (*) or a hash (#) are at the symmetry positions ($-x, 1-y, 1-z$) and ($1-x, 1-y, 1-z$), respectively.

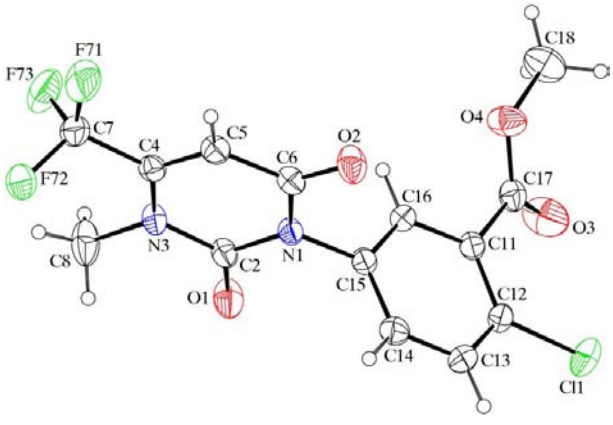

Figure 3
A view of (II), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

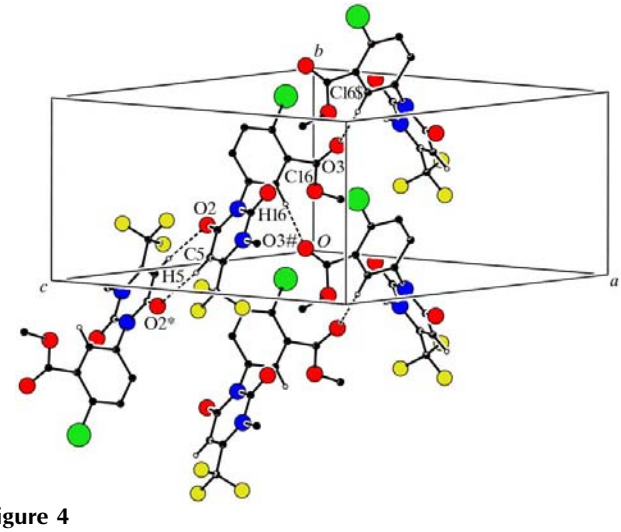

Figure 4
A view showing part of a sheet of molecules of (II) linked by weak C—H···O interactions in the (101) plane. Atoms labelled with an asterisk (*), hash (#) or dollar ($) are at the symmetry positions ($-x, -y, 1-z$), ($\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$) and ($\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$), respectively.

equally over two orientations (labelled C4 and C4*A*), with these atoms being 0.439 (1) and −0.333 (13) Å, respectively, from the plane defined by atoms N1/C2/C3/C5/C6. The dihedral angle between the plane of these five atoms and the benzene ring (C11–C16) is 87.52 (13)°. This conformation has atom F1 essentially equidistant from carbonyl atoms O1 and O2 [3.457 (5) and 3.513 (3) Å, respectively]. The orientation of the propargyl group relative to the aromatic ring is defined by the torsion angle C15—O3—C17—C18 of 84.3 (4)°. The distance between the farthest two atoms, Cl1···F71, is 10.889 (9) Å.

As was found in 2-[4-chloro-2-fluoro-5-(prop-2-ynyloxy)-phenyl]-*cis*-1,3,3a,4,7,7a-hexahydroisoindole-1,3-dione (Li *et al.*, 2005), there is also a well defined acetylenic C—H···O bond in (III) [C19—H19···O2^{i}; symmetry code: (i) $x, y, z-1$], which generates chains along [100] (Fig. 6). Adjacent chains are linked by C—H···O hydrogen bonds, involving the propargyl moiety C17—H17 with an adjacent piperidine carbonyl O atom, and also the piperidine C4—H4 group with the other piperidine carbonyl O atom (Table 3). In this way, a sheet structure with $R_3^2(15)$ and $R_3^3(21)$ rings is propagated in the (010) plane (Fig. 6).

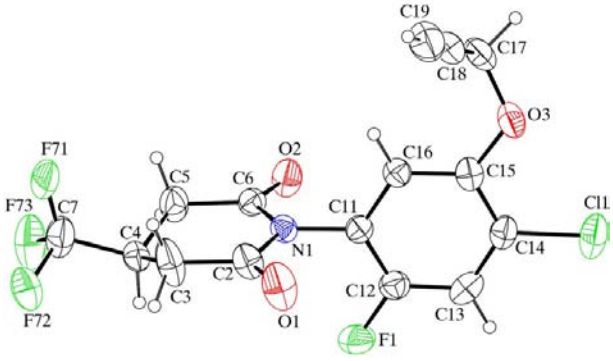

Figure 5
A view of (III), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The C4—C7F$_3$ moiety is disordered equally over two sites; for clarity, only one of these is shown.

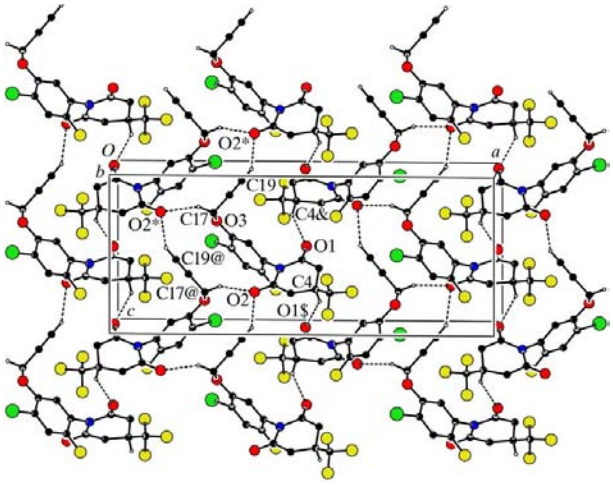

Figure 6
A view showing part of a sheet of molecules of (III) linked by C—H···O interactions in the (010) plane. Atoms labelled with an asterisk (*), hash (#), dollar ($), ampersand (&) or 'at' symbol (@) are at the symmetry positions $(x, y, z-1)$, $(\frac{1}{2}-x, y, z-\frac{1}{2})$, $(1-x, 1-y, \frac{1}{2}+z)$, $(1-x, 1-y, z-\frac{1}{2})$ and $(\frac{1}{2}-x, y, \frac{1}{2}+z)$, respectively.

Experimental

Compound (I) was synthesized by refluxing benzoic acid in methanol, as described by Johnson (1977). The oily product was cooled in a refrigerator and the solid which formed was grown from a solution in acetone to afford colourless single crystals suitable for X-ray diffraction [m.p. 331–332 K (semi-solid; Johnson, 1977)]. ^{1}H NMR ($CDCl_3$): δ 3.93 (*s*, 3H, CH_3O), 7.08–7.13 (*m*, 2H, Ph), 7.27 (*dd*, 1H, J = 9 and 0.9 Hz, NO_2-Ph), 7.63 (*dd*, 1H, J = 7.8 and 1.5 Hz, CF_3-Ph), 7.82 (*d*, 1H, J = 1.8 Hz, CF_3-Ph), 8.05 (*d*, 1H, J = 9 Hz, NO_2-Ph). Compound (II) was sythesized according to the procedure of Li *et al.* (2001), by stirring methyl 2-chloro-5-[2,6-dioxo-4-(trifluoromethyl)-1,2,3,6-tetrahydropyrimidin-1-yl]benzoate, Na_2CO_3 and iodomethane in dimethylformamide for 4 h at room temperature. After pouring the reaction mixture into water, the precipitate which formed was filtered off and recrystallized from chloroform, which gave colourless single crystals suitable for X-ray diffraction (m.p. 441–442 K). Analysis, $C_{14}H_{10}ClF_3N_2O_4$ requires: C 46.36, H 2.78, N 7.72%; found: C 46.39, H 2.61, N 7.70%; ^{1}H NMR ($CDCl_3$): δ 3.55 (*s*, 3H, CH_3N), 3.91 (*s*, 3H, CH_3O), 6.37 (*s*, 1H, py), 7.29 (*d*, 1H, J = 8.9 Hz, Ph), 7.59 (*d*, 1H, J = 8.9 Hz, Ph), 7.79 (*d*, 1H, J = 2.4 Hz, Ph). Compound (III) was synthesized by refluxing 4-chloro-2-fluoro-5-(prop-2-ynyloxy)phenylamine with the corresponding anhydride in acetic acid for 1 h, as described by Lange *et al.* (1991). The crude products were purified by silica-gel column chromatography and then grown from a solution in acetone to afford colourless single crystals suitable for X-ray diffraction [m.p. 379–380 K; literature value 361–394 K (Lange *et al.*, 1991)]. ^{1}H NMR ($CDCl_3$): δ 2.57 (*t*, 1H, J = 2.4 Hz, C*CH), 2.89–3.15 (*m*, 5H), 4.72 (*d*, 2H, J = 2.4 Hz, CH_2C*C), 6.84 (*d*, 1H, J = 6.6 Hz, Ph), 7.29 (*d*, 1H, J = 9 Hz, Ph).

Compound (I)

Crystal data

$C_{15}H_9ClF_3NO_5$
M_r = 375.68
Monoclinic, $P2_1/c$
a = 19.280 (5) Å
b = 9.715 (3) Å
c = 18.050 (5) Å
β = 109.509 (5)°
V = 3186.8 (15) Å^3
Z = 8
D_x = 1.566 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 997 reflections
θ = 2.3–19.5°
μ = 0.30 mm^{-1}
T = 293 (2) K
Prism, colourless
0.44 × 0.36 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.853, T_{max} = 0.925
16 078 measured reflections
5616 independent reflections
2057 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 25.0°
$h = -22 \rightarrow 14$
$k = -11 \rightarrow 11$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.163
S = 0.87
5616 reflections
475 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0715P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O2A	0.93	2.47	3.359 (5)	159
C16A—H16A···O2	0.93	2.51	3.373 (5)	155
C6—H6···O5^{i}	0.93	2.70	3.323 (6)	125
C6A—H6A···O5A^{ii}	0.93	2.56	3.389 (6)	149
C5—H5···O2^{iii}	0.93	2.60	3.511 (6)	167
C5A—H5A···O2A^{iv}	0.93	2.74	3.578 (6)	151

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, \frac{1}{2}-y, z+\frac{1}{2}$; (iv) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

Compound (II)

Crystal data

$C_{14}H_{10}ClF_3N_2O_4$
M_r = 362.69
Monoclinic, $P2_1/n$
a = 14.326 (13) Å
b = 7.802 (7) Å
c = 14.337 (13) Å
β = 110.599 (15)°
V = 1500 (2) Å^3
Z = 4
D_x = 1.606 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 823 reflections
θ = 2.5–25.4°
μ = 0.31 mm^{-1}
T = 293 (2) K
Prism, colourless
0.26 × 0.22 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.806, T_{max} = 1.000
7542 measured reflections
2646 independent reflections
2007 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -12 \rightarrow 17$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.10
2646 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 1.2049P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0072 (12)

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.93	2.51	3.427 (4)	169
C16—H16···O3^{ii}	0.93	2.47	3.260 (4)	143

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

Compound (III)

Crystal data

$C_{15}H_{10}ClF_4NO_3$
M_r = 363.69
Orthorhombic, $Pca2_1$
a = 22.887 (9) Å
b = 7.250 (3) Å
c = 9.497 (4) Å
V = 1575.7 (11) Å^3
Z = 4
D_x = 1.533 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 908 reflections
θ = 2.8–21.2°
μ = 0.30 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.38 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.853, T_{max} = 0.919
7730 measured reflections
2725 independent reflections
2022 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 25.0°
$h = -16 \rightarrow 27$
$k = -8 \rightarrow 8$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.05
2725 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.3451P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), with 1236 Friedel pairs
Flack parameter: −0.05 (12)

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···O2^{i}	0.93	2.49	3.287 (7)	143
C17—H17B···O2^{ii}	0.97	2.52	3.359 (4)	145
C4—H4···O1^{iii}	0.98	2.33	3.175 (10)	144

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+\frac{1}{2}, y, z-\frac{1}{2}$; (iii) $-x+1, -y+1, z+\frac{1}{2}$.

For all three compounds, possible H-atom sites were revealed in difference maps. H atoms were subsequently allowed for in the refinements as riding atoms, with C—H = 0.93, 0.97 and 0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(parent atom). For (I), both trifluoromethyl groups have the F atoms unequally disordered over two main orientations. This was modelled with DFIX restraints [C—F = 1.33 (5) Å and F···F = 2.08 (5) Å] and tied occupancy parameters, which refined to 0.827 (10)/0.173 (10) and 0.858 (9)/0.142 (9). Each triplet of minor-occupancy F atoms was refined with a common U_{iso} value [final values = 0.111 (7) and 0.122 (8) Å^2]. Compound (III) has disorder of the piperidine C4 atom, with concomitant disorder of the F atoms bonded to C7 (which was not disordered). Initial refinement of tied occupancy parameters for the disordered C4 site led to values which did not differ significantly from 0.5 and, in the final refinement cycles, the occupancies of the two components (labelled C4 and C4A) were set at 0.5. The six half-occupancy F atoms (labelled F71–F73 and F71A–F73A) were refined without restraints.

For all three compounds, data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); structure solution: *SHELXS97* (Sheldrick, 1997); structure refinement: *SHELXL97* (Sheldrick, 1997) in *WinGX* (Farrugia, 1999); molecular graphics: *PLATON* (Spek, 2003); publication software: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1802). Services for accessing these data are described at the back of the journal.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boger, P. & Wakabayashi, K. (1999). *Peroxidizing Herbicides*. Berlin: Springer.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dayan, F. E. & Allen, S. N. (2000). *Pest Manage. Sci.* **56**, 717–722.
Duke, S. O., Lydon, J. & Paul, R. N. (1989). *Weed Sci.* **37**, 152–160.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Johnson, W. O. (1977). US Patent No. 4 031 131.
Kennard, C. H. L., Smith, G. & Hari, T. (1987). *Aust. J. Chem.* **40**, 1131–1135.
Kohno, H., Hirai, K., Hori, M., Sato, Y., Boger, P. & Wakabayashi, K. (1993). *Z. Naturforsch. Teil C,* **48**, 334–338.
Lange, B. C., Ashmore, J. W., Wissinger-Cornille, J. & Tice, C. M. (1991). Eur. Patent Appl. No. 415 642.
Li, B. (2005). PhD thesis, University of Nankai, People's Republic of China.
Li, B., Man, Y., Zhang, Z. J., Xu, L. H. & Hsu, C. T. (2001). US Patent No. 6 251 829.
Li, B., Song, H.-B., Hsu, C.-T., Zhang, K.-L., Wu, C. & Yang, H.-Z. (2005). *Acta Cryst.* C**61**, o114–o117.
Matringe, M. & Scalla, R. (1988). *Plant Physiol.* **86**, 619–662.
Nandihalli, U. B., Duke, M. V. & Duke, S. O. (1992). *Pestic. Biochem. Physiol.* **43**, 193–211.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tomlin, C. (2003). *The Pesticide Manual*, 13th ed. Hampshire: BCPC.