N4	0.1547 (4)	0.69254 (9)	0.4750 (1)	0.0343 (4)
N3	0.1909 (5)	0.8494 (1)	0.5206 (1)	0.0426 (5)
C2	−0.1726 (4)	0.7984 (1)	0.3263 (2)	0.0277 (4)
C3	0.0569 (4)	0.7819 (1)	0.4413 (1)	0.0285 (4)
C5	0.0320 (5)	0.6240 (1)	0.3979 (2)	0.0360 (5)
C6	−0.1923 (5)	0.6401 (1)	0.2860 (2)	0.0353 (5)
C7	−0.2862 (5)	0.8949 (1)	0.2865 (2)	0.0334 (5)
H1	−0.549 (7)	0.850 (2)	0.136 (2)	0.085 (9)
H2	0.319 (6)	0.830 (1)	0.589 (2)	0.054 (6)
H3	0.128 (7)	0.910 (2)	0.500 (2)	0.062 (7)

Table 2. *Selected geometric parameters (Å, °)*

O1—C7	1.328 (2)	N4—C5	1.319 (2)
O2—C7	1.216 (2)	N3—C3	1.336 (2)
N1—C2	1.333 (2)	C2—C3	1.423 (2)
N1—C6	1.331 (2)	C2—C7	1.480 (2)
N4—C3	1.354 (2)	C5—C6	1.388 (2)
C2—N1—C6	118.3 (1)	N3—C3—C2	124.5 (2)
C3—N4—C5	117.7 (1)	N4—C5—C6	122.7 (1)
N1—C2—C3	121.1 (1)	N1—C6—C5	120.6 (1)
N1—C2—C7	117.8 (1)	O1—C7—O2	119.0 (2)
C3—C2—C7	121.1 (1)	O1—C7—C2	118.2 (1)
N4—C3—N3	116.0 (1)	O2—C7—C2	122.8 (1)
N4—C3—C2	119.6 (1)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.92 (2)	2.27 (2)	2.730 (2)	111 (2)
N3—H3···O2	0.91 (2)	2.04 (2)	2.718 (2)	131 (2)
O1—H1···N4^{i}	0.92 (2)	2.02 (2)	2.760 (2)	137 (2)
N3—H2···N1^{ii}	0.86 (2)	2.26 (2)	3.116 (2)	173 (2)
N3—H3···O2^{iii}	0.91 (2)	2.41 (2)	2.998 (2)	123 (2)

Symmetry codes: (i) $x-1, \frac{3}{2}-y, z-\frac{1}{2}$; (ii) $1+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iii) $-x, 2-y, 1-z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: FG1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1514–1516

Flurtamone

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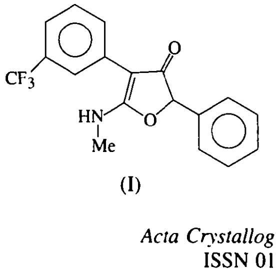
Abstract

Flurtamone, 5-methylamino-2-phenyl-4-(3-trifluoromethylphenyl)furan-3(2*H*)-one, $C_{18}H_{14}F_3NO_2$, is a weed killer. The phenyl substituents are rotated by about 30 and 72° from the mean plane of the central five-membered ring, while the methylamino substituent lies almost within this plane.

Comment

Flurtamone, (I), is an active material used as a weed killer. It is a carotenoid synthesis inhibitor and is applied before planting (pre-emergence or post-emergence) in order to control many grass and broad-leaved weeds. It is selective for cotton, peanuts, sorghum and sunflowers, but is not yet a commercial product. Its identification code is CAS RN [96525-23-4].



(I)

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The solubility of (I) at 293 K in ethanol is 9.92 g per 100 g of solvent. As traces of some growth inhibitors were present in solution, it was necessary to achieve rather large supersaturations, about 50 to 100% with respect to solubility, in order to induce nucleation without stirring the solution. The resulting crystals exhibit a well developed prismatic habit.

The crystal structure of flurtamone, like those of other weed killers linuron (Cadiergue, Pèpe, Astier, Boistelle & Fiard, 1993) and diflufenican (Pèpe, Pfefer, Boistelle & Marchal, 1995), was determined as part of a program of research aimed at predicting from solvent and additive-effect modelling the crystal morphology obtained from different media. The objective was to obtain crystals shaped as isometrically as possible in order to facilitate further handling and treatment.

The five-membered ring is essentially planar [maximum deviation 0.020 (4) Å]. The degree of delocalization of the π system can be characterized by the torsion angles C1—C6—C11—C12 −29.6 (4), C15—C14—C19—C20 72.4 (5) and O13—C12—N17—C18 3.4 (3)°. The high values of the displacement parameters of the F atoms of the CF_3 group indicate, as in diflufenican (Pèpe, Pfefer, Boistelle & Marchal, 1995), possible disorder for these atoms. The crystal structure is stabilized by an intermolecular hydrogen bond [O16···N17^{i} 2.76 (1) Å; symmetry code: (i) $x, \frac{1}{2}-y, -\frac{1}{2}+z$] and by van der Waals interactions.

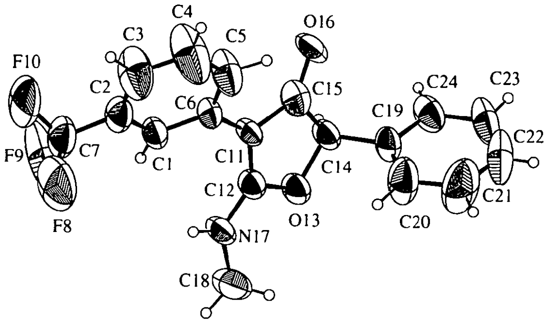

Fig. 1. The molecular structure of flurtamone with displacement ellipsoids shown at the 50% probability level.

Experimental

The crystals of flurtamone used for X-ray analysis were grown from ethanol solution.

Crystal data

$C_{18}H_{14}F_3NO_2$
$M_r = 333.3$
Monoclinic
$P2_1/c$
$a = 10.359\ (2)$ Å
$b = 12.218\ (2)$ Å
$c = 13.530\ (3)$ Å
$\beta = 110.64\ (3)°$
$V = 1602.5\ (4)$ Å^3
$Z = 4$
$D_x = 1.38$ Mg m^{-3}
$D_m = 1.37\ (2)$ Mg m^{-3}
D_m measured by flotation in $CH_2Cl_2/CHCl_3$

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 15–45°$
$\mu = 0.87$ mm^{-1}
$T = 293$ K
Hexagonal prism
0.4 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ scans
Absorption correction: none
3254 measured reflections
3254 independent reflections
2214 observed reflections [$I > 3\sigma(I)$]

$\theta_{max} = 45°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.056$
$wR = 0.056$
$S = 0.78$
2214 reflections
259 parameters
H atoms riding with fixed U_{iso}
Unit weights applied

$(\Delta/\sigma)_{max} = 0.16$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.1264 (4)	0.3428 (3)	0.7366 (3)	0.05 (1)
C2	0.0540 (4)	0.4307 (3)	0.7557 (3)	0.06 (2)
C3	0.0446 (6)	0.5264 (4)	0.7023 (5)	0.11 (4)
C4	0.1154 (8)	0.5366 (4)	0.6361 (6)	0.13 (5)
C5	0.1837 (5)	0.4507 (4)	0.6118 (4)	0.09 (3)
C6	0.1948 (3)	0.3512 (3)	0.6651 (2)	0.05 (1)
C7	−0.0167 (5)	0.4218 (5)	0.8320 (4)	0.08 (3)
F8	−0.0489 (5)	0.3208 (3)	0.8482 (3)	0.17 (3)
F9	0.0625 (4)	0.4460 (4)	0.9283 (3)	0.17 (3)
F10	−0.1263 (4)	0.4743 (4)	0.8120 (3)	0.19 (3)
C11	0.2717 (3)	0.2599 (3)	0.6410 (2)	0.04 (1)
C12	0.3389 (3)	0.1752 (3)	0.7053 (2)	0.05 (1)
O13	0.4051 (2)	0.1063 (2)	0.6604 (2)	0.05 (1)
C14	0.3786 (4)	0.1441 (3)	0.5533 (2)	0.05 (2)
C15	0.2942 (3)	0.2462 (3)	0.5436 (3)	0.07 (2)
O16	0.2526 (3)	0.3016 (2)	0.4606 (2)	0.06 (1)
N17	0.3534 (3)	0.1492 (2)	0.8023 (2)	0.05 (1)
C18	0.4375 (6)	0.0599 (4)	0.8616 (3)	0.08 (3)
C19	0.5110 (4)	0.1645 (3)	0.5355 (3)	0.06 (2)
C20	0.6155 (5)	0.2172 (5)	0.6075 (4)	0.10 (3)
C21	0.7350 (5)	0.2416 (6)	0.5838 (5)	0.12 (5)
C22	0.7488 (7)	0.2083 (5)	0.4953 (6)	0.11 (4)
C23	0.6426 (6)	0.1564 (4)	0.4222 (4)	0.08 (3)
C24	0.5257 (5)	0.1334 (3)	0.4416 (3)	0.07 (2)

Table 2. *Selected geometric parameters (Å, °)*

C11—C12	1.373 (4)	C14—O13	1.452 (4)
C11—C15	1.427 (4)	C14—C15	1.502 (4)
C12—O13	1.357 (3)	C18—N17	1.449 (5)
C12—N17	1.307 (4)		
C12—C11—C15	106.6 (2)	O13—C14—C15	104.5 (2)
C11—C12—O13	114.0 (2)	C11—C15—C14	107.2 (3)
C11—C12—N17	132.3 (3)	C11—C15—O16	130.5 (3)
O13—C12—N17	113.7 (2)	C14—C15—O16	122.3 (3)
C12—O13—C14	107.6 (2)	C12—N17—C18	125.0 (3)

Data collection: Enraf–Nonius (1977) CAD-4 diffractometer software. Cell refinement: Enraf–Nonius (1977) CAD-4 diffractometer software. Data reduction: *DATARED* (Pèpe, 1979). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *AME* (Software Systems, 1988).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1058). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Four ω-Phthalimidoaliphatic Peracids

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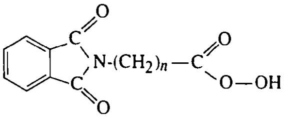
Abstract

Hydrogen bonding between the peracid proton and phthalimido carbonyl O atom connect molecules into chains in the structures of 3-phthalimidoperoxypropanoic acid, $C_{11}H_9NO_5$, and 4-phthalimidoperoxybutanoic acid, $C_{12}H_{11}NO_5$. In contrast, the peracid function acts as both a hydrogen-bond donor and acceptor in the structures of 5-phthalimidoperoxypentanoic acid, $C_{13}H_{13}NO_5$, and 6-phthalimidoperoxyhexanoic acid, $C_{14}H_{15}NO_5$, resulting in a distinctly different chain motif.

Comment

Organic peracids have recently been recognized as potential low-temperature laundry bleaches. To have commercial utility, these materials are required to exhibit high solid-state thermal stability with respect to decomposition to the corresponding carboxylic acid. In order to probe the relationship between peracid crystal structure and this solid-state stability, the structures of the four homologues 3-phthalimidoperoxypropanoic acid, (I), 4-phthalimidoperoxybutanoic acid, (II), 5-phthalimidoperoxypentanoic acid, (III), and 6-phthalimidoperoxyhexanoic acid, (IV), have been determined. In particular, the influence of the alkyl chain length on the type of hydrogen-bonding adopted has been investigated (Feeder, 1992).



(I) $n = 2$
(II) $n = 3$
(III) $n = 4$
(IV) $n = 5$

In all four title structures (Figs. 1–4), the phthalimido groups are close to being planar and have bond lengths and angles similar to those of both a series of ω-phthalimidoaliphatic acids (Feeder & Jones, 1996*a*) and a series of ω-phthalimidoaliphatic acid monohydrates (Feeder & Jones, 1994*a*) determined previously. The alkyl chains each adopt a regular all-*trans* conformation. The position of the peracid proton could not be determined in any of the four structures, although each of the peracid groups defined by the heavy C and O atoms is planar: (I) to 0.001 (4), (II) to 0.001 (1), (III) to 0.004 (4) and (IV) to 0.002 (2) Å. The bond lengths and angles associated with the peracid functions of compounds (II), (III) and (IV) are very similar to those reported previously for peroxypelargonic acid (Belitskus & Jeffrey, 1965), *o*-nitroperoxybenzoic acid (Sax, Beurskens & Chu, 1965), *p*-nitroperoxybenzoic acid (Kim, Chu & Jeffrey, 1970), a series of *p*-amidoperoxybenzoic acids (Feeder & Jones, 1996*b*) and two ω-saccharinperoxyaliphatic acids (Feeder & Jones, 1994*b*). The low O—O bond length in compound (I) (O111—O112) of 1.357 (7) Å is an artefact related to the high atomic displacement parameter (or disorder) of the O112 atom.

Hydrogen bonding in all four structures links molecules into infinite chains. In the structures of (I) and (II), the hydrogen bond is between the peracid proton