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Acta Cryst. (1995). C**51**, 2671–2672

Diflufenican, *N*-(2,4-Difluorophenyl)-2-[3-(trifluoromethyl)phenoxy]-3-pyridinecarboxamide

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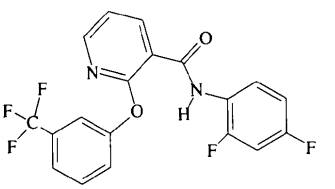
(*Received* 17 *January* 1995; *accepted* 15 *June* 1995)

Abstract

Diflufenican, $C_{19}H_{11}F_5N_2O_2$, has an approximately planar molecular skeleton, except for the 3-trifluoromethylphenyl group which lies almost perpendicular to the rest of the molecule. The amide N—H group and ether O atom are linked by an intramolecular hydrogen bond.

Comment

Diflufenican, (I), is an active material used as a weed killer. More precisely, it acts as a carotenoid synthesis and photosynthetic electron-flow inhibitor. Its identification code is CAS RN [83164-33-4]. This product is conditioned, in association with another weed killer (isoproturon), as a flowable and concentrated suspension (under the trademarks Quartz GT, Javelin or Fenican). It is applied on autumn-sown wheat and barley (before emergence or in early emergence) in order to control grass and broad-leaved weeds. In order to obtain good quality crystals for X-ray analysis, (I) was recrystallized from *p*-xylene and acetone solutions, using non-industrial solvents. The solubilities at 293 K in these solvents were 3.36 and 8.25 g per 100 g of solvent, respectively. Supersaturation was achieved by dissolving an excess of 10 to 20% of material, with respect to solubility, in hot solutions, which were then allowed to cool to 293 K without stirring. In both solvents, thick plate-like crystals appeared within one or two days. The crystal structure of diflufenican was determined as part of an investigation of the effect of different media on the crystal morphology. The ultimate objective was to obtain crystals shaped as isometrically as possible in order to facilitate the conditioning.



(I)

A strong intramolecular N9···O18 hydrogen bond [2.65 (1) Å] is observed. The π-system deformation can be characterized by the torsion angles C12—C17—C10—O11 3.8 (3), C24—C19—O18—C16 −77.4 (5), C5—C4—N9—C10 −6.3 (5) and C17—C10—N9—C4 −174.8 (5)°. The large values of the displacement parameters of the F atoms of the CF_3 group indicate slight disorder of these atom positions. The crystal is stabilized by π–π and van der Waals interactions.

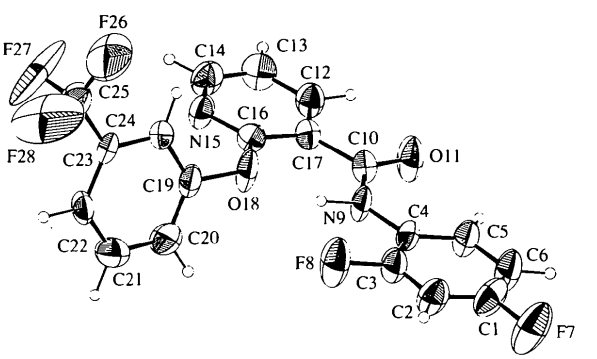


Fig. 1. *ORTEP*II drawing (Jonhson, 1976) of the molecule of diflufenican with displacement ellipsoids of 50% probability.

Experimental

The title compound was recrystallized from *p*-xylene and acetone solutions (see *Comment*). The crystal density D_m was measured by flotation.

Crystal data

$C_{19}H_{11}F_5N_2O_2$ — Cu $K\alpha$ radiation
M_r = 394.3 — λ = 1.5418 Å

Monoclinic
$P2_1/c$
a = 12.523 (3) Å
b = 8.583 (2) Å
c = 15.930 (3) Å
β = 90.52 (3)°
V = 1712.2 (4) Å^3
Z = 4
D_x = 1.53 Mg m^{-3}
D_m = 1.52 (2) Mg m^{-3}

Cell parameters from 25 reflections
θ = 15–45°
μ = 1.17 mm^{-1}
T = 293 K
Thick plate
0.3 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ scans
Absorption correction: none
3407 measured reflections
3407 independent reflections
2545 observed reflections [$I > 3\sigma(I)$]

θ_{max} = 45°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
R = 0.0570
wR = 0.057
S = 0.84
2545 reflections
286 parameters
H-atom positions refined freely with U = 0.05 Å^2
Unit weights applied

$(\Delta/\sigma)_{max}$ = 0.17
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
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)

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

	x	y	z	U_{eq}
C1	0.2635 (3)	0.3530 (5)	0.0236 (3)	0.07 (2)
C2	0.3593 (3)	0.4136 (5)	0.0492 (3)	0.06 (2)
C3	0.4304 (3)	0.3032 (4)	0.0761 (2)	0.05 (2)
C4	0.4133 (2)	0.1442 (4)	0.0798 (2)	0.04 (1)
C5	0.3163 (3)	0.0906 (5)	0.0515 (3)	0.05 (2)
C6	0.2408 (3)	0.1986 (5)	0.0229 (3)	0.07 (2)
F7	0.1896 (2)	0.4567 (4)	−0.0038 (2)	0.11 (2)
F8	0.5280 (2)	0.3538 (3)	0.1039 (2)	0.08 (1)
N9	0.4948 (2)	0.0525 (3)	0.1120 (2)	0.04 (1)
C10	0.4938 (2)	−0.1008 (4)	0.1290 (2)	0.04 (1)
O11	0.4196 (2)	−0.1860 (3)	0.1119 (2)	0.07 (2)
C12	0.5934 (3)	−0.3260 (4)	0.1832 (2)	0.05 (2)
C13	0.6824 (3)	−0.3984 (4)	0.2166 (3)	0.06 (2)
C14	0.7663 (3)	−0.3074 (5)	0.2379 (3)	0.05 (1)
N15	0.7688 (2)	−0.1520 (3)	0.2280 (2)	0.04 (1)
C16	0.6835 (3)	−0.0869 (4)	0.1960 (2)	0.04 (1)
C17	0.5919 (2)	−0.1670 (4)	0.1711 (2)	0.06 (1)
O18	0.6841 (2)	0.0715 (3)	0.1869 (2)	0.04 (1)
C19	0.7785 (2)	0.1544 (4)	0.2022 (2)	0.06 (2)
C20	0.8315 (3)	0.2169 (5)	0.1354 (2)	0.07 (3)
C21	0.9209 (3)	0.3078 (6)	0.1470 (3)	0.05 (2)
C22	0.9571 (3)	0.3361 (4)	0.2274 (2)	0.05 (2)
C23	0.9013 (2)	0.2723 (4)	0.2939 (2)	0.04 (1)
C24	0.8110 (2)	0.1833 (4)	0.2828 (2)	0.04 (1)
C25	0.9397 (3)	0.3037 (6)	0.3806 (3)	0.07 (2)
F26	0.8662 (3)	0.2952 (6)	0.4371 (2)	0.15 (3)
F27	1.0097 (3)	0.2059 (6)	0.4047 (2)	0.17 (3)
F28	0.9791 (5)	0.4392 (5)	0.3922 (2)	0.19 (4)

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

C1—C2	1.366 (7)	C14—N15	1.343 (6)
C1—C6	1.355 (7)	C16—N15	1.305 (5)
C1—F7	1.353 (6)	C16—C17	1.392 (6)
C2—C3	1.366 (7)	C16—O18	1.367 (5)
C3—C4	1.382 (6)	C19—O18	1.399 (5)
C3—F8	1.367 (5)	C19—C20	1.368 (6)
C4—C5	1.371 (6)	C19—C24	1.366 (5)
C4—N9	1.384 (5)	C20—C21	1.375 (7)
C5—C6	1.397 (7)	C21—C22	1.376 (7)
C10—N9	1.343 (5)	C22—C23	1.387 (6)
C10—O11	1.211 (5)	C23—C24	1.374 (5)
C10—C17	1.505 (5)	C23—C25	1.483 (7)
C12—C13	1.378 (6)	C25—F26	1.295 (7)
C12—C17	1.378 (6)	C25—F27	1.270 (7)
C13—C14	1.350 (7)	C25—F28	1.276 (8)
C2—C1—F7	116.2 (11)	N15—C16—O18	117.5 (9)
C6—C1—F7	119.8 (12)	C10—C17—C12	116.5 (8)
C1—C2—C3	113.4 (11)	C10—C17—C16	127.4 (9)
C2—C3—C4	126.6 (10)	C12—C17—C16	116.0 (9)
C2—C3—F8	117.4 (10)	C16—O18—C19	119.5 (8)
C4—C3—F8	116.0 (9)	C20—C19—O18	118.6 (9)
C3—C4—C5	117.0 (9)	C20—C19—C24	121.1 (9)
C3—C4—N9	117.6 (8)	C24—C19—O18	120.0 (8)
C5—C4—N9	125.4 (9)	C19—C20—C21	121.2 (11)
C4—C5—C6	118.7 (11)	C20—C21—C22	119.1 (12)
C1—C6—C5	120.3 (12)	C21—C22—C23	118.5 (10)
C10—N9—C4	128.6 (8)	C22—C23—C24	122.7 (9)
C17—C10—N9	116.8 (8)	C22—C23—C25	118.6 (9)
C17—C10—O11	119.6 (9)	C24—C23—C25	118.7 (9)
N9—C10—O11	123.7 (9)	C19—C24—C23	117.4 (8)
C13—C12—C17	120.7 (10)	C23—C25—F26	114.2 (11)
C12—C13—C14	117.5 (11)	C23—C25—F27	112.2 (12)
C13—C14—N15	124.3 (11)	C23—C25—F28	115.0 (12)
C14—N15—C16	116.8 (9)	F26—C25—F27	104.2 (12)
C17—C16—N15	124.7 (9)	F26—C25—F28	103.1 (13)
C17—C16—O18	117.8 (9)	F27—C25—F28	107.1 (13)

Data collection: CAD-4 diffractometer software (Enraf–Nonius, 1977). Cell refinement: 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: CF1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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