C4	0.3123 (7)	0.1556 (3)	0.6684 (3)	0.063 (3)
C5	0.5211 (7)	0.1921 (3)	0.5922 (3)	0.060 (3)
C6	0.6123 (6)	0.2969 (3)	0.5993 (3)	0.052 (3)
C7	0.5979 (6)	0.4830 (3)	0.6917 (3)	0.049 (2)
C11	0.5603 (6)	0.6597 (3)	0.8006 (3)	0.052 (2)
C12	0.7621 (6)	0.7168 (3)	0.7310 (3)	0.061 (3)
C13	0.8193 (7)	0.8174 (4)	0.7645 (4)	0.065 (3)
C14	0.6774 (7)	0.8613 (3)	0.8636 (3)	0.067 (3)
C15	0.4746 (7)	0.8090 (4)	0.9323 (3)	0.068 (3)
C16	0.4168 (7)	0.7074 (3)	0.9004 (3)	0.061 (3)
O6	0.8170 (4)	0.3240 (2)	0.5177 (2)	0.065 (2)
O7	0.7885 (4)	0.5078 (2)	0.6190 (2)	0.065 (2)
N7	0.4866 (6)	0.5548 (3)	0.7752 (3)	0.061 (3)
F2	0.1467 (3)	0.3965 (2)	0.8510 (2)	0.072 (2)
F3	−0.0141 (4)	0.1927 (2)	0.8318 (2)	0.089 (2)
F4	0.2253 (4)	0.0552 (2)	0.6602 (3)	0.089 (2)
F5	0.6380 (4)	0.1236 (2)	0.5064 (2)	0.082 (2)
F14	0.7374 (5)	0.9589 (2)	0.8962 (2)	0.097 (2)

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

C1—C2	1.389 (4)	C7—N7	1.341 (4)
C1—C6	1.413 (4)	C11—C12	1.392 (4)
C1—C7	1.486 (4)	C11—C16	1.383 (4)
C6—O6	1.342 (4)	C11—N7	1.416 (4)
C7—O7	1.236 (4)		
C2—C1—C6	115.8 (3)	O7—C7—N7	121.2 (3)
C2—C1—C7	126.3 (3)	C12—C11—C16	119.6 (3)
C6—C1—C7	117.9 (3)	C12—C11—N7	124.2 (3)
C1—C7—O7	119.6 (3)	C16—C11—N7	116.3 (3)
C1—C7—N7	119.2 (3)	C7—N7—C11	129.2 (3)

Data collection: *CAD-4 Diffractometer Control Software* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Diffractometer Control Software*. Data reduction: *PROCESS TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *LS TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *FINISH TEXSAN*. Literature survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1141). 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. (1995). C**51**, 1429–1431

1-(2-Benzothiazolyl)-3-methoxycarbonylmethyl-2-thioxo-4-imidazolidinone, $C_{13}H_{11}N_3O_3S_2$

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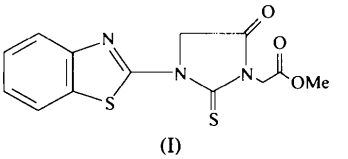
(*Received* 27 *June* 1994; *accepted* 3 *January* 1995)

Abstract

The reaction of methyl isothiocyanatoacetate with 2-aminothiophenol gave as the unexpected major product the title compound, methyl 1-(2-benzothiazolyl)-4-oxo-2-thioxo-3-imidazolidinylacetate, which was identified by X-ray crystal structure analysis.

Comment

In a continuation of our studies on the synthesis of heterocyclic compounds obtained from isothiocyanato carboxylates, we have reported the synthesis of 2-amino substituted derivatives of benzimidazoles (Floch, Uher & Leško, 1989). It was expected that the synthetic procedures developed there should be similarly applicable to the synthesis of 2-amino-substituted derivatives of benzothiazoles. Nevertheless, the reaction of methyl isothiocyanatoacetate with 2-aminothiophenol furnished, in high yield, an unexpected compound (I) having the



(I)

molecular formula $C_{13}H_{11}N_3O_3S_2$ and a molecular ion (M^+) having m/z = 321 in the mass spectrum. Spectral data (IR, UV, ^1H and ^{13}C NMR) were not sufficient to elucidate the structure, so an X-ray diffraction study was undertaken. This showed the molecule to consist of a 2-thiohydantoin ring substituted in the 1-position by a 2-benzothiazolyl ring and in the 3-position by a methoxycarbonylmethyl group.

The distances in the benzene ring are normal [1.367 (6)–1.396 (5) Å]. There is a high degree of π-electron delocalization in the $O1_{sp}$—$N3_{sp^2}$—$C10_{sp^2}$—$N2_{sp^2}$—$C7_{sp^2}$—$N1_{sp}$ unit.

All three rings are roughly coplanar and the angle between the plane of the benzene ring and the plane of the imidazolidinone ring is 2.4 (1)°. The imidazolidinone ring plane makes an angle of 86.0 (2)° with the mean plane of the methoxycarbonylmethyl group. The molecular structure with the atom-numbering scheme is shown in Fig. 1 (*ORTEP*II; Johnson, 1971).

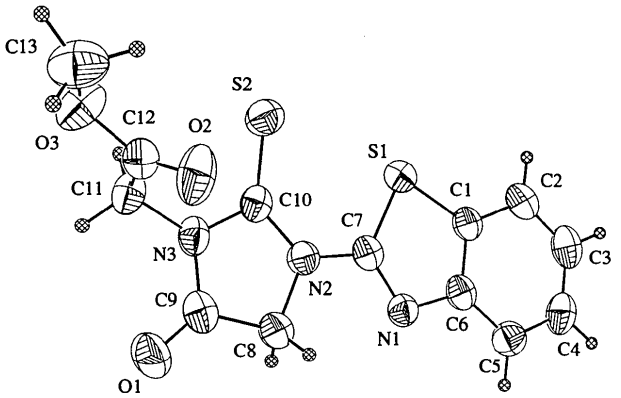


Fig. 1. View of $C_{13}H_{11}N_3O_3S_2$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

Experimental

Thin transparent colourless crystals of the title compound were obtained from acetone solution.

Crystal data

$C_{13}H_{11}N_3O_3S_2$
M_r = 321.37
Monoclinic
$P2_1/c$
a = 14.339 (1) Å
b = 4.804 (1) Å
c = 20.812 (2) Å
β = 101.48 (1)°
V = 1404.8 (3) Å^3
Z = 4
D_x = 1.520 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 16–25°
μ = 0.336 mm^{-1}
T = 293 (2) K
Rod
0.75 × 0.03 × 0.03 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2553 measured reflections
2474 independent reflections
1240 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0415
θ_{max} = 25.01°
$h = -16 \rightarrow 17$
$k = 0 \rightarrow 5$
$l = -24 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: not significant

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0417
$wR(F^2)$ = 0.0905
S = 1.075
2473 reflections
200 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.3112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0002 (9)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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}
S1	0.39160 (7)	0.2147 (2)	1.01990 (5)	0.0432 (3)
S2	0.34586 (8)	−0.1533 (3)	0.89750 (5)	0.0521 (4)
O1	0.0599 (2)	−0.6364 (7)	0.9406 (2)	0.069 (1)
O2	0.0943 (2)	−0.1909 (7)	0.7972 (2)	0.069 (1)
N1	0.2556 (2)	0.1236 (7)	1.0840 (2)	0.040 (1)
N2	0.2374 (2)	−0.1405 (7)	0.98952 (15)	0.040 (1)
N3	0.1893 (2)	−0.4384 (7)	0.9097 (2)	0.043 (1)
C1	0.3934 (3)	0.3963 (8)	1.0921 (2)	0.038 (1)
C2	0.4581 (3)	0.5981 (9)	1.1214 (2)	0.048 (1)
C3	0.4447 (3)	0.7141 (10)	1.1788 (2)	0.055 (1)
C4	0.3689 (3)	0.6380 (9)	1.2068 (2)	0.053 (1)
C5	0.3045 (3)	0.4407 (9)	1.1782 (2)	0.049 (1)
C6	0.3167 (3)	0.3215 (9)	1.1198 (2)	0.039 (1)
C7	0.2878 (3)	0.0565 (8)	1.0322 (2)	0.037 (1)
C8	0.1517 (3)	−0.2686 (9)	1.0051 (2)	0.045 (1)
C9	0.1245 (3)	−0.4708 (10)	0.9495 (2)	0.049 (1)
C10	0.2578 (3)	−0.2379 (8)	0.9329 (2)	0.038 (1)
C11	0.1830 (3)	−0.5794 (9)	0.8477 (2)	0.047 (1)
C12	0.1342 (3)	−0.4023 (10)	0.7913 (2)	0.043 (1)
O3	0.1416 (2)	−0.5169 (7)	0.73558 (14)	0.059 (1)
C13	0.0918 (3)	−0.3817 (12)	0.6760 (2)	0.070 (2)

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

S1—C1	1.734 (4)	N3—C11	1.444 (5)
S1—C7	1.734 (4)	C1—C6	1.386 (5)
S2—C10	1.635 (4)	C1—C2	1.396 (5)
O1—C9	1.207 (5)	C2—C3	1.367 (6)
O2—C12	1.184 (5)	C3—C4	1.379 (6)
N1—C7	1.296 (5)	C4—C5	1.373 (6)
N1—C6	1.403 (5)	C5—C6	1.385 (6)
N2—C10	1.354 (5)	C8—C9	1.502 (6)
N2—C7	1.397 (5)	C11—C12	1.505 (5)
N2—C8	1.467 (5)	C12—O3	1.306 (5)
N3—C9	1.370 (5)	O3—C13	1.455 (5)
N3—C10	1.391 (5)		
C1—S1—C7	87.6 (2)	C1—C6—N1	114.7 (3)
C7—N1—C6	108.8 (3)	N1—C7—N2	118.2 (4)
C10—N2—C7	127.8 (4)	N1—C7—S1	118.2 (3)
C10—N2—C8	112.5 (3)	N2—C7—S1	123.6 (3)

C7—N2—C8	119.6 (3)	N2—C8—C9	101.6 (3)
C9—N3—C10	112.7 (3)	O1—C9—N3	125.1 (4)
C9—N3—C11	123.8 (4)	O1—C9—C8	128.2 (4)
C10—N3—C11	123.2 (4)	N3—C9—C8	106.8 (4)
C6—C1—C2	120.8 (4)	N2—C10—N3	106.3 (3)
C6—C1—S1	110.7 (3)	N2—C10—S2	128.8 (3)
C2—C1—S1	128.4 (3)	N3—C10—S2	124.9 (3)
C3—C2—C1	117.8 (4)	N3—C11—C12	111.7 (3)
C2—C3—C4	121.4 (5)	O2—C12—O3	125.5 (4)
C5—C4—C3	121.2 (5)	O2—C12—C11	124.2 (4)
C4—C5—C6	118.3 (4)	O3—C12—C11	110.3 (4)
C5—C6—C1	120.4 (4)	C12—O3—C13	117.1 (4)
C5—C6—N1	124.8 (4)		

The structure was solved by Patterson methods using *SHELX*86 (Sheldrick, 1990) and refined by least squares with *SHELXL*93 (Sheldrick, 1993). Calculations were carried out using *PARST* (Nardelli, 1983), which was also used to prepare material for publication. The figure was produced with *ORTEP*II (Johnson, 1971).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: HR1040). 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. (1995). C**51**, 1431–1434

A 1,5-Diarylpyrrole Derivative

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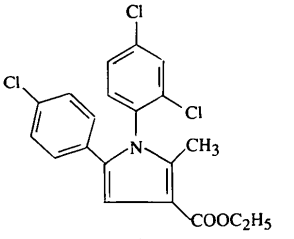
(*Received* 27 *May* 1994; *accepted* 13 *January* 1995)

Abstract

The structure of the 1,5-diarylpyrrole derivative, ethyl 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-2-methyl-3-pyrrolecarboxylate, $C_{20}H_{16}Cl_3NO_2$, has been determined. The orientation assumed by the phenyl rings corresponds to the deepest of the two minima obtained from *ab initio* calculations. All bond distances and angles are in the expected ranges.

Comment

A large number of diarylpyrrole derivatives have been synthesized in order to develop new fungicides against *Candida* strains (Scalzo, Biava, Porretta & Cerreto, 1991; Cerreto, Villa, Retico & Scalzo, 1992). In the course of this study, the compound 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-3-(dimethylamino)-2-methylpyrrole, (1), along with some of its analogs, was found to be remarkably active. Studies of the structure–activity relationships of this class of compound (Scalzo *et al.*, 1991), together with structural analogies to some azole antifungal agents, suggested that their biological activity could be related to interactions with the heme group of the fungicide target enzyme (cytochrome P-450). In order to optimize the antifungal activity of these compounds, a computer graphic study was undertaken using (1) as a model structure. Several attempts were made to obtain crystals suitable for X-ray analysis. Finally, a crystal structure determination was undertaken on the title 1,5-diarylpyrrole derivative, ethyl 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-2-methyl-3-pyrrolecarboxylate, (2). Though inactive, compound (2) could give valuable information on the orientation of the phenyl rings, which is critical for the mode of interaction in the active site of the enzyme.



(2)

A view of the title compound projected onto the plane of the pyrrole ring is shown in Fig. 1. One phenyl ring, Ph1 (C6–C11), is almost perpendicular to the plane of the pyrrole ring (76°) and the other phenyl ring, Ph2 (C17–C22), is a little twisted out of this plane (28°). The dihedral angles in three pyrrole derivatives (De Kimpe, Sulmon, De Buyck, Verhe, Schamp, Declercq & Van Meerssche, 1984; Toupet, Mazari, Texier & Carrie, 1991), which describe the relative positions of the planes of adjacent phenyl rings, are given in Table 3. In these compounds, one phenyl ring is oriented similar to Ph1 in (2), while the other assumes a slightly different orientation with an increased inclination on the pyrrole plane. This feature is also present