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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71304 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1048]

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1-S19.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271-383.

Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Ghosh, S. (1993). In preparation.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.

McPhail, A. T., Rimmer, B., Robertson, J. M. & Sim, G. A. (1967). *J. Chem. Soc. B*, pp. 101-106.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.

Schenk, H. & Hall, S. R. (1990). *SIMPEL*. In *Xtal3.0 Reference Manual*, edited by S. R. Hall & J. M. Stewart. Univs. of Western Australia, Australia, and Maryland, USA.

White, D. N. J. & Sim, G. A. (1977*a*). *Cryst. Struct. Commun.* **6**, 681-684.

White, D. N. J. & Sim, G. A. (1977*b*). *Cryst. Struct. Commun.* **6**, 685-688.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558-564.

Acta Cryst. (1993). C**49**, 1975–1976

Structure of *N*,*N'*-Dimethylpiperazine-2,3-dithione: Space Group Correction

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Abstract

The crystal structure of *N*,*N'*-dimethylpiperazine-2,3-dithione, $C_6H_{10}N_2S_2$, has been described and refined in space group *Aa* [Servaas, Stufkens, Oskam, Vernooijs, Baerends, De Ridder & Stam (1989). *Inorg. Chem.* **28**, 4104-4113]. It is described properly in space group *A*2/*a*. Revised coordinates and bond lengths and angles are given.

Comment

The structure of the title compound was reported in space group *Aa* (Servaas *et al.*, 1989). The six-membered ring of the molecule was found to adopt a half-chair conformation with the twofold axis running through the midpoints of the C—C bonds, parallel to the *b* axis. Furthermore, the two N—C(methyl) bond lengths were very dissimilar [1.439 (5) and 1.489 (5) Å]; there is no reason for them to be unequal. This unusual spread of distances undoubtedly resulted from the refinement of a centrosymmetric structure in a non-centrosymmetric space group (Ermer & Dunitz, 1970; Schomaker & Marsh, 1979). Therefore it seemed likely that the structure should properly be described in space group *A*2/*a*.

Starting coordinates were from Table 3 of Servaas *et al.* (1989) and averaged in accordance with *A*2/*a*. Full-matrix minimization of the quantity $\Sigma w(F_o - F_c)^2$ with $w = 1/[\sigma^2(F) + 0.0035F^2]$ was performed. Refinement was anisotropic for the non-H atoms and isotropic for the H atoms. An isotropic extinction coefficient was included in the parameters [Zachariasen, final value $3.4\,(5) \times 10^{-5}$]. Anomalous dispersion was taken into account but no correction for absorption was applied. The H-atom positions were calculated initially on the basis of standard geometry and refined. A final difference Fourier map revealed residual electron density within the range -0.22–0.30 e Å^{-3}.

The values obtained for parameters, bond lengths and angles are close to the averages of pairs of equivalent values obtained in the earlier *Aa* refinement. The two N—C(methyl) bond lengths are now equal by symmetry at 1.473 (3) Å. The general description of the structure remains unchanged.

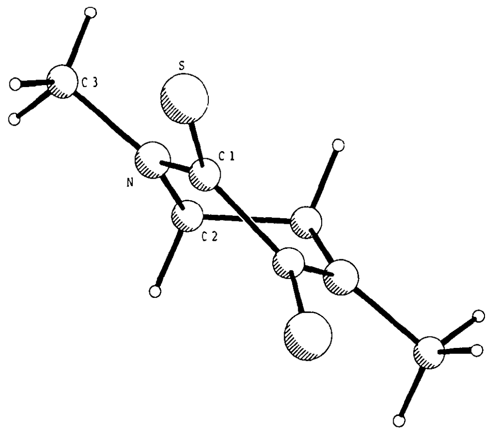

Fig. 1. A *PLUTO* (Motherwell & Clegg, 1978) drawing of $C_6H_{10}N_2S_2$ showing the labelling of the independent non-H atoms. The view is down the twofold axis, with the crystallographic 2_1 axis vertical.

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Experimental

Crystal data

$C_6H_{10}N_2S_2$
M_r = 174.29
Monoclinic
$A2/a$
a = 10.653 (1) Å
b = 7.2936 (6) Å
c = 10.6398 (9) Å
β = 95.26 (1)°
V = 823.2 (1) Å^3
Z = 4

D_x = 1.406 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 23 reflections
θ = 38–44°
μ = 5.20 mm^{-1}
T = 293 K
Needle shaped crystals
0.25 × 0.20 × 0.13 mm
Dark red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1710 measured reflections
770 independent reflections
731 observed reflections [$I > 2.5\sigma(I)$]

$\theta_{\max}$ = 69.71°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 12$
2 standard reflections
frequency: 66.67 min
intensity variation: none

Refinement

Refinement on F
Final R = 0.037
wR = 0.07
S = 1.167
714 reflections
67 parameters refined
$w = 1/[\sigma^2(F) + 0.0035F^2]$
$(\Delta/\sigma)_{\max}$ = 0.783
$\Delta\rho_{\max}$ = 0.294 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}

Extinction correction: Zachariasen (1967)
Extinction coefficient: g = 3.4 (5) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

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

The origin is located at a centre of symmetry on the glide plane a.
$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S	0.11142 (4)	0.18040 (6)	0.05913 (4)	0.0460 (6)
C1	0.1977 (1)	0.3686 (3)	0.0442 (1)	0.0331 (9)
C2	0.2460 (3)	0.6922 (3)	0.0694 (3)	0.057 (1)
C3	0.0912 (2)	0.5405 (4)	0.2013 (2)	0.058 (1)
N	0.1807 (1)	0.5247 (2)	0.1041 (1)	0.0425 (9)

Table 2. *Geometric parameters* (Å, °)

S—C1	1.668 (2)	C2—N	1.470 (3)
C1—N	1.325 (2)	C2—C2^{i}	1.487 (4)
C1—C1^{i}	1.523 (2)	C3—N	1.473 (3)
S—C1—N	124.1 (1)	C1—N—C2	120.3 (2)
S—C1—C1^{i}	120.0 (1)	C1—N—C3	122.0 (2)
N—C1—C1^{i}	115.9 (2)	C2—N—C3	117.6 (2)
N—C2—C2^{i}	108.7 (2)		

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

Crystals were prepared by Dr P. C. Servaas according to the literature procedure described by Isaksson, Liljefors & Sandström (1981). Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* program comparable to *Xtal LATCON* (Hall, Flack & Stewart, 1992). Data reduction: *Xtal ADDREF, SORTRF*. Structure solved by the heavy-atom method. Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *Xtal BONDLA, CIFIO.*

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71346 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1061]

References

Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
Ermer, O. & Dunitz, J. (1970). *Acta Cryst.* **A26**, 163.
Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal3.2 Reference Manual*. Univs. of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
Isaksson, R., Liljefors, T. & Sandström, J. (1981). *J. Chem. Res. (M)*, pp. 664–682.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.
Schomaker, V. & Marsh, R. E. (1979). *Acta Cryst.* **B35**, 1933–1934.
Servaas, P. C., Stufkens, D. J., Oskam, A., Vernooijs, P., Baerends, E. J., De Ridder, D. J. A. & Stam, C. H. (1989). *Inorg. Chem.* **28**, 4104–4113.
Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

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Structure of Dimethyl 2-[*o*-(3,5-Dimethyl-1-pyrazolyl)anilino]-3-methoxymaleate

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Abstract

In the title compound the pyrazole and benzene rings form a dihedral angle of 64.5 (1)°. Neither ring deviates significantly from planarity. There is an intramolecular hydrogen bond between atom N12 and atom O23 [N12···O23 2.682 (4) Å, N12—H12···O23 102.9 (2)°]; all other bond distances and angles are within the expected ranges.

Comment

This work is part of a more complex study on the chemistry of the Ramsden's Class *B* heteropentalenes. The ti-