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Redetermination of 1,3-Dimethylimidazole-2-thione

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Abstract

The title compound, 1,3-dimethylimidazole-2(3H)-thione, $C_5H_8N_2S$, has molecular symmetry $mm2$ (C_{2v}) in the crystal.

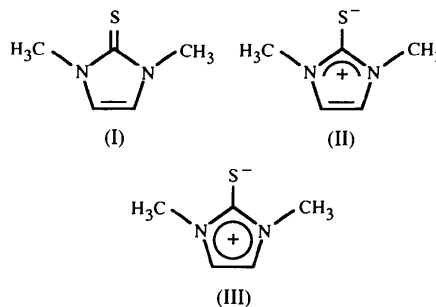
Comment

The present study is part of a program investigating the structure/property relationships among nonlinear optical (NLO) materials. A solution of the title compound, DMIT, exhibits optical nonlinearities, including nonlinear refraction and two-photon absorption. DMIT was initially chosen for our investigations because the structure was known and it represents the basic template for a series of substituted compounds in which we also have interest. In earlier work, DMIT (Ansell, Forkey &

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Moore, 1970) was reported to have crystallized in the orthorhombic space group $Bmmb$. A redetermination of the structure was undertaken since the previous authors reported no atomic coordinates.



The C1—S bond length corresponds to a partial double bond. Based on bond distances within the ring, Ansell, Forkey & Moore (1970) concluded that the electronic structure of DMIT would best be represented by a resonance hybrid of structures (I) and (II). The bond distances found in this study correspond to the more delocalized resonance structure (III).

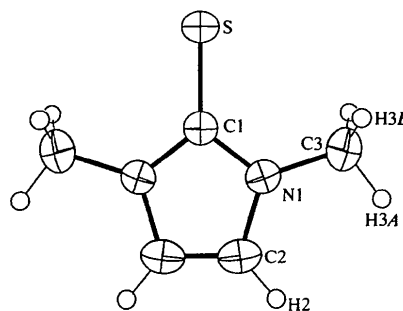


Fig. 1. The molecular structure of DMIT. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was synthesized from *N*-methylimidazole by alkylation with methyl iodide to give the 1,3-dimethylimidazolium halide. Subsequent treatment with sulfur in methanolic carbonate (Ansell, Forkey & Moore, 1970) yields DMIT (m.p. 454 K).

Crystal data

$C_5H_8N_2S$
 $M_r = 128.19$
 Orthorhombic
 $Cmcm$
 $a = 8.4680$ (14) Å
 $b = 11.1997$ (7) Å
 $c = 6.8220$ (9) Å
 $V = 646.99$ (14) Å³
 $Z = 4$
 $D_x = 1.316$ Mg m⁻³
 $D_m = 1.286$ Mg m⁻³
 D_m measured by flotation in CCl_4 /hexane

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 4-13^\circ$
 $\mu = 0.39$ mm⁻¹
 $T = 296$ K
 Parallelepiped
 $0.40 \times 0.15 \times 0.10$ mm
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: Gaussian, calculated
 $T_{\min} = 0.900$, $T_{\max} = 0.963$
 1350 measured reflections
 654 independent reflections
 248 reflections with $I > \sigma(I)$

$R_{\text{int}} = 0.036$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 10$
 $k = -13 \rightarrow 13$
 $l = 0 \rightarrow 8$
 3 standard reflections
 frequency: 240 min
 intensity decay: 3%

Refinement

Refinement on F^2
 $R = 0.049$
 $wR = 0.040$
 $S = 1.169$
 248 reflections
 30 parameters
 H atoms: see below
 Weights: calculated from measured e.s.d.'s

$(\Delta/\sigma)_{\max} = 0.0002$
 $\Delta\rho_{\max} = 0.322 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.428 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1189). Services for accessing these data are described at the back of the journal.

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N-Carboxy-DL-phenylalanine Anhydride

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

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

	x	y	z	U_{eq}
S	1	0.21604 (14)	1/4	0.0509 (9)
C1	1	0.3662 (5)	1/4	0.038 (3)
C2	1.0785 (6)	0.5578 (3)	1/4	0.050 (2)
C3	1.2903 (5)	0.3993 (4)	1/4	0.060 (3)
N1	1.1263 (4)	0.4391 (3)	1/4	0.0404 (16)

Table 2. Selected geometric parameters (\AA , $^\circ$)

S—C1	1.681 (5)	C2—N1	1.390 (5)
C1—N1	1.346 (4)	C3—N1	1.458 (5)
C2—C2 ⁱ	1.329 (7)		
N1 ⁱ —C1—N1	105.3 (4)	C1—N1—C2	110.4 (4)
N1—C1—S	127.4 (2)	C1—N1—C3	124.8 (3)
C2 ⁱ —C2—N1	106.9 (4)	C2—N1—C3	124.7 (4)
S—C1—N1—C2	180.0	H2—C2—N1—C3	0.0
S—C1—N1—C3	0.0	N1—C2—C2 ⁱ —N1 ⁱ	0.0
H2—C2—N1—C1	180.0	H2—C2—C2 ⁱ —N1 ⁱ	180.0

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

All non-H atoms were located from electron density maps and were refined anisotropically by full-matrix least squares. H atoms were located from difference Fourier synthesis and were not refined positionally, while the displacement parameters were refined isotropically.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *Xtal3.4* (Hall, Flack & Stewart, 1995). Program(s) used to solve structure: *Xtal3.4*. Program(s) used to refine structure: *Xtal3.4*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *Xtal3.4*. Software used to prepare material for publication: *Xtal3.4*.

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Abstract

The N1—H1 imino group of the five-membered ring of the title compound, C₁₀H₉NO₃, forms intermolecular hydrogen bonds between O1 along the *a* axis and between O3 along the *b* axis. Thus, a two-dimensional hydrogen-bonding network forms a layer perpendicular to the *c* axis. The layer, which consists of one of two independent molecules, stacks alternately along the *c* axis and produces a sandwich structure.

Comment

The relationship between the crystal structures of *N*-carboxy anhydrides (NCAs) of *L*-amino acids and their polymerizability in the solid state has been studied by Kanazawa (1992a) and Kanazawa & Kawai (1980). The crystal structures of glycine NCA (Kanazawa, Matsura, Tanaka, Kakudo, Komoto & Kawai, 1976a), *L*-alanine NCA (Kanazawa, Matsura, Tanaka, Kakudo, Komoto & Kawai, 1976b), γ -benzyl-*L*-glutamate NCA (Kanazawa, Ohashi, Sasada & Kawai, 1978a), *L*-leucine NCA (Kanazawa, Ohashi, Sasada & Kawai, 1978b), *L*-valine NCA (Kanazawa, Ohashi & Sasada, 1984) and *DL*-valine NCA (Takenaka, Ohashi & Kanazawa, 1994) have been determined so far.