The molecular packing of 8OH-DPAT showed that there are few close contacts between molecules. The molecules lie in an infinite stack with pairs of molecules related by inversion centres. The propyl arms are badly disordered; this hindered the search for close contacts between the molecules. It is possible that there is a similar salt-bridge effect to that found in 8MeO-PAT although the distances are a little long. The C(11), C(12) and C(13) propyl chain is closer to the nearest Cl ion than the C(14)—C(16) propyl chain; the H atoms on C(11) come within 3 Å of the Cl ion. The other closest atoms to the Cl ion are the H atoms bound to C(6) and C(7).

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Mesoionic Compounds. 2. Structure of 2-Morpholino-5-propyl-1,3-dithiolium-4-thiolate

By Kung-Kai Cheung*

Department of Chemistry, University of Hong Kong, Bonham Road, Hong Kong

Sérgio Galembeck

Universidade de São Paulo, FFCL-RP, Campus Universitário, 14049 *Ribeirão Preto, SP, Brazil*

Joseph Miller

Universidade de São Paulo, FFCL-RP, Campus Universitário, 14049 *Ribeirão Preto, SP, Brazil,*
Universidade de São Paulo, IQ, Cidade Universitária, Cx. Postal 20780, 01498 *São Paulo, SP, Brazil,*
and Universidade Federal da Paraíba, LTF, Campus Universitário I, *Cx. Postal* 5009, 58059 *João Pessoa, PB, Brazil*

Magali B. de Oliveira

Universidade de São Paulo, FFCL-RP, Campus Universitário, 14049 *Ribeirão Preto, SP, Brazil*

Arnaldo B. Pereira

Universidade de São Paulo, FCF-RP, Campus Universitário, 14049 *Ribeirão Preto, SP, Brazil*

and Alfredo M. Simas

Universidade Federal de Pernambuco, DQF, Cidade Universitária, 50739 *Recife, PE, Brazil*

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Abstract. $C_{10}H_{15}NOS_3$, $M_r = 261.43$, monoclinic, $P2_1/c$, $a = 7.511$ (1), $b = 10.088$ (1), $c = 17.383$ (1) Å, $\beta = 112.41$ (1)°, $V = 1217.7$ (4) Å^3, $Z = 4$, $D_x = 1.426$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 5.6$ cm^{-1}, $F(000) = 552$, $T = 297$ K, $R = 0.030$ for 1339 reflections with $|F_o| > 3\sigma(|F_o|)$. In the pentatomic ring

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* Author to whom correspondence should be addressed.

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system of this mesoionic compound there are two short C—S bonds (1.715 and 1.710 Å) which are comparable to the C—S$^-$ (thiolate) bond of 1.719 Å, and two longer C—S bonds (1.764 and 1.753 Å). The structure also features a substituted morpholine molecule.

Introduction. The title compound is a mesoionic compound related to 5-methyl-2-piperidino-1,3-thiolium-4-thiolate (Cheung, Galembeck, Miller, de Oliveira, Pereira & Simas, 1991). The structure analysis was carried out to obtain further structural information and to throw light on the extent of the delocalization of the positive charge within and outside the pentatomic heterocycle, and the degree of aromaticity or level of aromatic stabilization energy.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
S(1)	0.2447 (1)	0.53308 (8)	0.42704 (4)	2.86 (2)
S(2)	0.2387 (1)	0.36454 (8)	0.55918 (4)	3.04 (2)
S(3)	0.3040 (1)	0.78397 (8)	0.51903 (5)	3.91 (2)
O	0.0447 (3)	0.0619 (2)	0.2921 (1)	4.24 (5)
N	0.1724 (3)	0.2726 (2)	0.4069 (1)	2.92 (6)
C(1)	0.2145 (4)	0.3758 (3)	0.4575 (2)	2.51 (6)
C(2)	0.2774 (4)	0.6150 (3)	0.5212 (2)	2.70 (6)
C(3)	0.2754 (4)	0.5339 (3)	0.5824 (2)	2.69 (6)
C(4)	0.3028 (4)	0.5754 (3)	0.6690 (2)	3.24 (7)
C(5)	0.4636 (4)	0.5044 (3)	0.7388 (2)	3.40 (7)
C(6)	0.5022 (5)	0.5700 (3)	0.8224 (2)	4.70 (9)
C(7)	0.1665 (4)	0.1369 (3)	0.4364 (2)	3.54 (7)
C(8)	0.0159 (5)	0.0575 (3)	0.3689 (2)	4.13 (8)
C(9)	0.0299 (5)	0.1945 (3)	0.2632 (2)	4.01 (8)
C(10)	0.1820 (4)	0.2821 (3)	0.3241 (2)	3.51 (7)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

S(1)—C(1)	1.715 (4)	N—C(7)	1.468 (4)
S(1)—C(2)	1.764 (3)	N—C(10)	1.472 (4)
S(2)—C(1)	1.710 (3)	C(2)—C(3)	1.346 (5)
S(2)—C(3)	1.753 (3)	C(3)—C(4)	1.500 (4)
S(3)—C(2)	1.719 (4)	C(4)—C(5)	1.525 (4)
O—C(8)	1.432 (4)	C(5)—C(6)	1.521 (4)
O—C(9)	1.419 (4)	C(7)—C(8)	1.512 (4)
N—C(1)	1.321 (4)	C(9)—C(10)	1.511 (4)

C(1)—S(1)—C(2)	97.7 (1)	S(3)—C(2)—C(3)	131.0 (2)
C(1)—S(2)—C(3)	96.9 (1)	S(2)—C(3)—C(2)	117.2 (2)
C(8)—O—C(9)	109.8 (2)	S(2)—C(3)—C(4)	116.9 (2)
C(1)—N—C(7)	122.3 (2)	C(2)—C(3)—C(4)	125.9 (3)
C(1)—N—C(10)	120.7 (2)	C(3)—C(4)—C(5)	115.6 (3)
C(7)—N—C(10)	114.9 (2)	C(4)—C(5)—C(6)	111.1 (3)
S(1)—C(1)—S(2)	114.0 (2)	N—C(7)—C(8)	109.7 (3)
S(1)—C(1)—N	123.3 (2)	O—C(8)—C(7)	111.9 (3)
S(2)—C(1)—N	122.7 (2)	O—C(9)—C(10)	111.6 (2)
S(1)—C(2)—S(3)	114.9 (2)	N—C(10)—C(9)	109.9 (3)
S(1)—C(2)—C(3)	114.2 (2)		

C(7)—N—C(1)—S(1)	173.7 (2)	N—C(7)—C(8)—O	53.9 (3)
C(7)—N—C(1)—S(2)	−7.8 (4)	C(8)—O—C(9)—C(10)	61.5 (3)
C(10)—N—C(1)—S(1)	10.9 (4)	C(9)—O—C(8)—C(7)	−61.5 (3)
C(10)—N—C(1)—S(2)	−170.6 (2)	C(10)—N—C(7)—C(8)	−48.6 (4)
		O—C(9)—C(10)—N	−54.6 (4)

Experimental. Orange crystal of dimensions 0.07 × 0.15 × 0.25 mm. Enraf–Nonius CAD-4 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation and scintillation counter. Cell parameters by least squares from setting angles of 25 reflections ($10 < 2\theta < 15°$), measured on the diffractometer. The intensities of 4527 reflections ($2\theta_{max} = 50°$), in the range $-8 \le h \le 8$, $0 \le k \le 12$, $-20 \le l \le 20$ were measured using ω–2θ scans, ω-scan angle $(1.00 + 0.344\tan\theta)°$ at 1.10–5.49° min^{-1}, extended 25% on each side for background measurements. Three standard reflections measured every 2 h showed no decay. Systematic absences indicated $P2_1/c$ and this was confirmed by the structure solution. The intensity data were corrected for Lorentz and polarization effects. After equivalent reflections were averaged, the 4527 measured data gave 2268 independent reflections, of which 1339 with $I > 1.5\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2)$ (S = scan, B_1 and B_2 = background counts), were considered observed. R_{int} = 0.021 for observed reflections and 0.034 for all reflections. The structure was solved with the ring-fragment $NC(CS)_2S$ as input using *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Smith, 1984), which gave all non-H atoms, and was refined by full-matrix least squares with atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAX II computer. The H atoms in calculated positions were not refined. Convergence for 136 variables by least squares with $w = 4F_o^2/[\sigma^2(F_o^2) + (0.03F_o^2)^2]$ was reached at $R = 0.030$, $wR = 0.032$ and $S = 1.010$ for 1339 reflections with $|F_o| > 3\sigma(|F_o|)$. $(\Delta/\sigma)_{max}$ = 0.05. A final difference Fourier map was featureless, with maximum positive and negative peaks of 0.19 and 0.20 e Å^{-3} respectively. There were no intermolecular distances less than 3.5 Å.

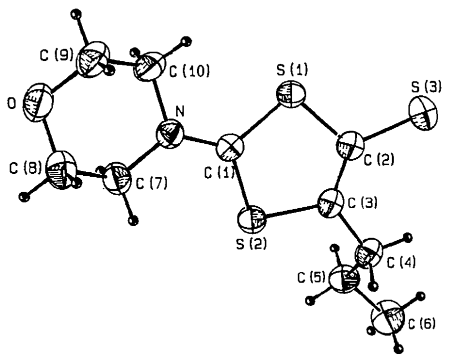

Fig. 1. *ORTEP*II drawing of the molecule with the numbering of atoms.

Discussion. An *ORTEP*II drawing (Johnson, 1976) of the molecule with atomic numbering scheme is shown in Fig. 1. The atomic parameters of C, N, O and S atoms are listed in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. The C(2)—C(3) bond of 1.346 (5) Å is comparable to the C=C bond of 1.362 (25) Å in thiophenes (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The S(3)—C(2) bond of 1.719 (4) Å is longer than the C=S bond of 1.683 (1) Å in the substituted thione 4-phenylthiazole-2(3*H*)-thione (Nalini & Desiraju, 1989) and is comparable to the S(1)—C(1) and S(2)—C(1) bonds of 1.715 (4) and 1.710 (3) Å respectively, but is significantly shorter than the S(1)—C(2) and S(2)—C(3) bonds of 1.764 (3) and 1.753 (3) Å respectively. The short S(3)—C(2) bond can be viewed as a single bond with the S atom carrying a negative charge so that C(2) is positive relative to S(3). The comparable and short S(1)—C(1) and S(2)—C(1) bonds can be viewed as a positive charge in the pentatomic ring close to C(1) so that C(1) is positive relative to the S(1) and S(2) atoms. The pentatomic ring system is nearly planar and the torsion angles involving the five ring atoms are close to 0 and 180°. All the features of the pentatomic heterocyclic system in this compound agree very well with those in 5-methyl-2-piperidino-1,3-dithiolium-4-thiolate (Cheung *et al.*, 1991). The C(1)—N bridging distance between the two ring systems is 1.321 (4) Å. The morpholinyl ring has a chair conformation with N and O deviating −0.560 (2) and +0.670 (2) Å respectively from the plane through C(7), C(8), C(9) and C(10). The C_{sp^3}—N_{sp^3} bonds are 1.468 (4) and 1.472 (4) Å, the C_{sp^3}—C_{sp^3} bonds are 1.511 (4) and 1.512 (4) Å, while the C_{sp^3}—O bonds are 1.432 (4) and 1.419 (4) Å, and all six bond angles are close to 109.5°.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and a complete list of bonding interactions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54586 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of an Intermediate Methylated Product in the Synthesis of Drimanes

By Kalyan Das and U. C. Sinha*

Department of Physics, Indian Institute of Technology, Bombay-400 076, *India*

S. R. Desai and S. V. Bhat

Department of Chemistry, Indian Institute of Technology, Bombay-400 076, *India*

and S. S. Tavale and V. G. Puranaik

Physical and Structural Chemistry Division, National Chemical Laboratory, Pune-411 008, *India*

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Abstract. 5-Methoxymethyl-1,1,4a-trimethyl-6-[(phenylthio)methyl]-1,5,8,8a-tetrahydro-4(4a*H*)-naphthalenone, $C_{22}H_{28}O_2S$, m.p. = 382 K, M_r = 356.53, monoclinic, $P2_1/c$, a = 11.810 (1), b = 8.929 (2), c = 18.716 (1) Å, β = 92.773 (8)°, V = 1971.32 Å^3, Z = 4, D_x = 1.201 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ(Mo $K\alpha$) = 0.137 mm^{-1}, $F(000)$ = 768, T = 291 K, final R = 0.055, wR = 0.074 using 2674 reflections. The bicyclic skeleton of the molecule is *cis* fused. An intermolecular C—H···O interaction [C(20)—H(20)···O(1′) 3.306 (3) Å] was observed.

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* To whom correspondence should be addressed.

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