atom, was also observed in the ambient-pressure and high-pressure structures of CHD (Katrusiak, 1990).

We are grateful to Dr R. O. Gould of the Department of Chemistry, University of Edinburgh, for his help in solving this structure. This study was partly supported by the British Council and the Polish Academy of Sciences Project CPBP 01.12.

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Acta Cryst. (1990). C**46**, 1293–1295

N-Methyl-*N*′-sulfamidoisothiourea

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(*Received* 9 *May* 1989; *accepted* 27 *September* 1989)

Abstract. (1,2-Dimethyl-3-isothiourea)sulfonamide, $C_3H_9N_3O_2S_2$, M_r = 183·3, triclinic, $P\bar{1}$, a = 7·216 (2), b = 7·932 (3), c = 8·201 (2) Å, α = 66·37 (2), β = 84·78 (2), γ = 64·10 (2)°, V = 385 Å^3, Z = 2, D_x = 1·58 Mg m^{-3}, Cu $K\alpha$ radiation, λ = 1·54178 Å, μ = 5·82 mm^{-1}, $F(000)$ = 192, room temperature, R = 0·041 for 1026 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The C—S bond to the methylated sulfur is longer than the analogous bond in urea. The C—N and N—S bonds show considerable delocalization. An intramolecular hydrogen bond exists between the methylated amine hydrogen and an oxygen of the sulfamide group.

Introduction. We are interested in cyclization reactions at metal centres and have prepared several unusual metal–sulfur–nitrogen complexes (Kelly & Woollins, 1986; Jones, Kelly, Williams & Woollins, 1988; Jones, Kelly, Warrens, Williams & Woollins, 1986). Recently, there has been a report on sulfamido–isothiourea derivatives (Michael, Ross & Rees, 1985) which have been used in *organic* cyclization reactions. The potential for these compounds in *coordination* chemistry has not been investigated and structural information is not available. We are involved in a study of the coordinating properties of this type of compound with a view to forming new materials with interesting physical properties. Prior to a study of their complexing ability we have determined the structure of one example, *N*-methyl-*N*′-sulfamidoisothiourea (1).

Experimental. Colourless single crystals of (1) suitable for X-ray crystallography were grown at room temperature by slow diffusion of *n*-hexane into a dichloromethane solution of the compound. Crystal size 0·16 × 0·14 × 0·20 mm. Refined unit-cell parameters obtained from setting angles of 16 reflections with $11{\cdot}8 \le \theta \le 28{\cdot}1°$. Nicolet *R*3*m* diffractometer. 1039 independent reflections ($2\theta \le 116°$), Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1026 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −7/7, k −7/8, l 0/8; two check reflections measured every 50 reflections, no significant change in net counts; Lorentz and polarization corrections, numerical absorption correction (face-indexed crystal), maximum and minimum transmission factors 0·460 and 0·230. Structure solved by direct methods. The non-H atoms were refined anisotropically. The leading protons on the methyl groups were located from a ΔF map. The protons on N(1) and N(3) were located from a ΔF map and refined isotropically. The positions of the remaining H atoms were idealized C—H 0·96 Å, assigned isotropic thermal parameters,

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Table 1. *Atomic coordinates* ($\times 10^4$) *and temperature factors* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S(1)	1638 (1)	1306 (1)	8786 (1)	51 (1)
C(1)	2397 (4)	3114 (4)	8813 (3)	38 (1)
N(1)	2730 (4)	2994 (3)	10431 (3)	46 (1)
N(2)	2593 (4)	4404 (3)	7272 (3)	42 (1)
S(2)	3652 (1)	5857 (1)	7148 (1)	37 (1)
O(1)	3241 (3)	7281 (3)	5317 (2)	49 (1)
O(2)	3066 (3)	6705 (3)	8482 (2)	48 (1)
N(3)	6153 (4)	4502 (4)	7528 (3)	48 (1)
C(2)	1544 (6)	1830 (5)	6455 (4)	60 (2)
C(3)	2590 (6)	1473 (5)	12110 (4)	64 (2)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

S(1)—C(1)	1·752 (4)	S(1)—C(2)	1·788 (4)
C(1)—N(1)	1·329 (4)	C(1)—N(2)	1·312 (3)
N(1)—C(3)	1·453 (4)	N(2)—S(2)	1·607 (3)
S(2)—O(1)	1·431 (2)	S(2)—O(2)	1·444 (2)
S(2)—N(3)	1·628 (2)		

C(1)—S(1)—C(2)	101·4 (2)	S(1)—C(1)—N(1)	114·9 (2)
S(1)—C(1)—N(2)	117·8 (3)	N(1)—C(1)—N(2)	127·3 (3)
C(1)—N(1)—C(3)	125·4 (3)	C(1)—N(2)—S(2)	121·9 (2)
N(2)—S(2)—O(1)	105·5 (1)	N(2)—S(2)—O(2)	113·1 (1)
O(1)—S(2)—O(2)	116·9 (1)	N(2)—S(2)—N(3)	109·0 (1)
O(1)—S(2)—N(3)	106·3 (1)	O(2)—S(2)—N(3)	105·7 (1)

C(2)—S(1)—C(1)—N(1)	−175·8 (2)	C(2)—S(1)—C(1)—N(2)	3·5 (2)
S(1)—C(1)—N(1)—C(3)	2·5 (3)	N(2)—C(1)—N(1)—C(3)	−176·8 (3)
S(1)—C(1)—N(2)—S(2)	−169·2 (1)	N(1)—C(1)—N(2)—S(2)	10·1 (3)
C(1)—N(2)—S(2)—O(1)	−169·6 (2)	C(1)—N(2)—S(2)—O(2)	−40·6 (2)
C(1)—N(2)—S(2)—N(3)	76·7 (2)		

[$U(H) = 1{\cdot}2U_{eq}(C)$] and allowed to ride on their parent C atoms. The methyl groups were refined as rigid bodies. An empirical extinction correction was applied [$g = 0{\cdot}0385\ (82)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}041$; $wR = 0{\cdot}054$ [$w^{-1} = \sigma^2(F) + 0{\cdot}00057F^2$]. $S = 2{\cdot}73$; 110 refined parameters; $(\Delta/\sigma)_{max} = 0{\cdot}078$; residual electron density in difference map within 0·36 and $-0{\cdot}36$ e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computations carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1985).

Discussion. The structure of (1) with the atomic numbering scheme is shown in Fig. 1. Table 1* lists the fractional atomic coordinates of the non-H atoms and Table 2 the bond lengths, angles and torsion angles. Methylation of S(1) results in the expected lengthening of the C(1)—S(1) bond relative to that in thioureas and increased delocalization over N(1)—C(1)—N(2)—S(2). This is most pronounced in the two C—N bonds though the N(2)—S(2) distance [1·607 (3) Å] is also appreciably shorter than in a formal single bond, *ca* 1·7 Å (Jones, Williams & Woollins, 1985). The S(2)—N(3) distance [1·628 (2) Å] is also somewhat shorter than that in sulfamic acid (1·73 Å) but comparable to that in sulfamide, 1·600 (9) Å (Trueblood & Mayer, 1956). This suggests that neither N(3) nor N(1) will have readily available lone pairs suitable for coordination of the neutral molecule. N(1) is trigonal and N(3) is pyramidal. A consequence of the pattern of deloca-

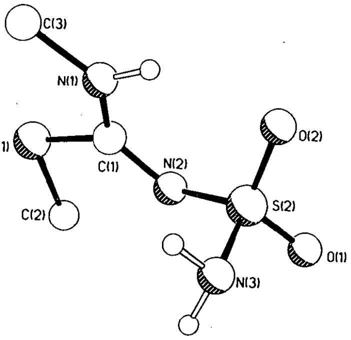

Fig. 1. Ball-and-stick representation of the structure of (1) showing the atomic numbering scheme.

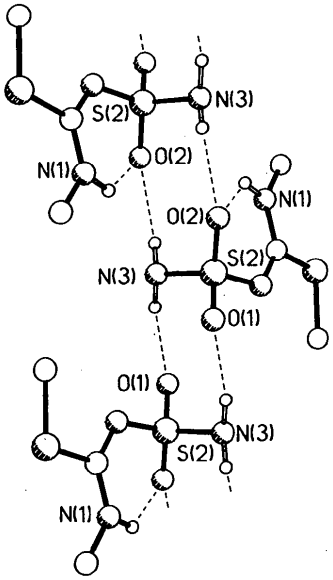

Fig. 2. Part of the continuous chain of hydrogen-bonded molecules of (1) in the crystal. N(3)—H···O(2) 3·05, H···O(2) 2·19 Å, N—H···O(2) angle 146°; N(3)—H···O(1) 3·09, H···O(1) 2·14 Å, N—H···O(1) angle 166°.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52689 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

lization is an essentially planar backbone with maximum departures from *syn* and *anti* geometry of *ca* 10°.

There is an intramolecular hydrogen bond (2·84 Å) between N(1) and O(2) [H(1)···O(2) 2·11 Å, N—H···O angle 130°]. Symmetry-related molecules are linked *via* N—H···O hydrogen bonds (Fig. 2) between the amine N and sulfone O atoms to form continuous chains in the crystal.

Despite certain unfavourable characteristics, the overall geometry of (1), in particular the spatial relationship of N(1) and O(2), indicates a potential for chelate coordination to one or two metal atoms with retention of planarity within the ligand.

We wish to thank Dr J. Michael, Hoechst UK, for supplying the original sample of (1).

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Acta Cryst. (1990). C**46**, 1295–1297

Structure of 2-Amino-1-(*p*-methoxyphenyl)-4-imidazolecarbaldehyde

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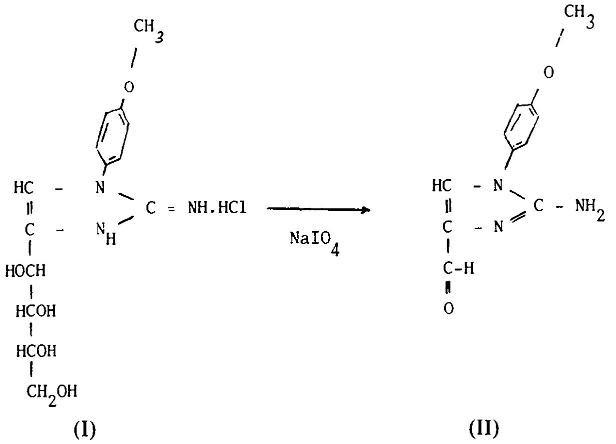
(*Received* 28 *March* 1989; *accepted* 15 *August* 1989)

Abstract. $C_{11}H_{11}N_3O_2$, $M_r = 217·2$, triclinic, $P\bar{1}$, $a = 8·020\,(2)$, $b = 8·490\,(4)$, $c = 9·031\,(4)$ Å, $\alpha = 117·19\,(6)$, $\beta = 102·66\,(4)$, $\lambda = 72·22\,(4)°$, $V = 518·4\,(5)$ Å^3, $Z = 2$, $D_x = 1·39$, $D_m = 1·40$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·9$ cm^{-1}, $F(000) = 228$, room temperature, final $R = 0·06$ for 1329 observed reflections. The dihedral angle between the imidazole plane and the aromatic ring is 55·3 (1)°. The double bonds in the imidazole ring are delocalized. The molecules are linked by van der Waals forces, although there is one short contact between the N(amino) and the O(carbonyl) atoms.

Introduction. This is one of a series of new imidazole derivatives that we are investigating. We have previously determined the crystal structures of 2-amino-3-methyl-5-(D-*arabino*-1,2,3,4-tetrahydroxybutyl)imidazolium chloride (Vega, López-Castro & Márquez, 1988) and 2-amino-1-methyl-4-imidazolecarbaldehyde (Pérez-Garrido, Moreno, Criado & López-Castro, 1988). The title compound (II) has been synthesized at the Organic Chemistry Department of Seville University by Professor Fdez-Bolaños and co-workers by oxidation with $NaIO_4$ of 2-amino-3-(*p*-methoxyphenyl)-5-(D-*arabino*-1,2,3,4-tetrahydroxybutyl)imidazolium chloride (I).



(I) (II)

The compound, as with other analogues, has been studied by UV, IR and NMR (Alaiz-Barragán, 1987), and the crystal-structure determination of the title compound was undertaken in order to establish possible correlations with the spectroscopic data. The resolution of the structure confirms the molecular conformation and the canonical form that contributes most to the structure, as shown by the spectroscopic data.

0108-2701/90/071295-03$03.00