The mean C—H distance for the benzene rings is 0·97 ($\pm$0·02) Å.

The oxadiazole ring is planar and shows a certain amount of shortening of bond lengths, consistent with some aromatic nature. For example, it is interesting to note that the C(7)—N(1) bond |1·380 (3) Å| is shorter than the average value of 1·475 Å reported for the C—N bond, when N is sp^2 hybridized (Sutton, 1965). On the other hand, the above-mentioned bond is comparable with the length of 1·369 Å in other aromatic compounds, like some substituted purines (Glusker, van der Helm, Lowe, Minkin & Patterson, 1968), or 1·383 (4) Å in pyrrole (Sutton, 1965).

The same trend is also evident in the C(8)—O bond which is consistent with those of similar compounds, e.g. 1·353 (7) Å in 3-hydroxy-5-phenylisoxazole (Biagini, Cannas & Marongiu, 1969).

If the lengths of C(7)—C(4) and C(8)—C(9) [ave. 1·464 (3) Å] are compared with those of sp^2-hybridized C atoms [e.g. 1·497 (2) Å in biphenyl; Robertson, 1961], a certain degree of conjugation between rings may be assumed by analogy with other phenyl-substituted isoxazoles where distances in the range 1·44–1·47 Å have been found (Biagini et al., 1969).

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The Structure of Dimethylammonium Dimethyldithiocarbamate

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Abstract. $[(CH_3)_2NH_2]^+[(CH_3)_2NCS_2]^-$, monoclinic, $P2_1/c$, $a = 9.979$ (3), $b = 7.701$ (1), $c = 12.189$ (3) Å, $\beta = 105.81$ (2)°, $Z = 4$, $V = 901.2$ Å³, $D_m = 1.225$ (flotation), $D_x = 1.226$ g cm⁻³. The structure has been solved by direct methods and refined by full-matrix least squares. $R(F^2) = 0.053$ for 1964 reflexions and 138 varied parameters. The ordered structure is built up of one-dimensional zig-zag chains along b. Each molecular ion is involved in four intermolecular H—S interactions between the ammonium protons and the dithiocarbamate S atoms, at H—S distances of 2·46 (3), 2·61 (3), 2·66 (3) and 2·99 (3) Å.

Introduction. This investigation forms part of the studies on structural principles in crystal structures in progress at this Institute. The present cations and anions show 'biting' and 'hindering' properties.

The compound was synthesized in benzene from dimethylamine and carbon disulfide according to

$$2(CH_3)_2NH + CS_2 \rightarrow [(CH_3)_2NH_2]^+ + [(CH_3)_2NCS_2]^-$$
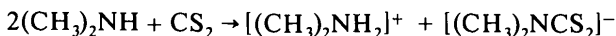
and was recrystallized from carbon disulfide. The transparent colorless crystals were generally elongated along $|010|$. They slowly turned 'floury' on free contact with air and sublimed or decomposed on heating. The space-group assumption was made from systematically absent reflexions among $h0l$ for odd l and $0k0$ for odd k. The unit-cell parameters were based on Cr $K\alpha_1$ radiation, the internal standard cobalt triphosphide ($a = 7.70778$ Å) and 27 Guinier–Hägg powder diffraction lines.

A compact crystal, with an approximate diameter ranging from 0.11 to 0.16 mm, was sealed in a thin-walled glass capillary tube. A unique set of intensities was collected in one quadrant of reciprocal space on a Stoe–Philips four-circle PDP 8/I computer-controlled diffractometer equipped with graphite mono-chromatized Mo $K\alpha$ radiation. Automatic selection of filters and scan speed allowed the count rate to be reduced by the factor 2^n, $0 \le n \le 5$. The longest scan time, in the ω–2θ mode, was 211 s. The background was measured on each side of the reflexion for 30 s. Three standard reflexions showed stability of the crystal and the instrument. Corrections were applied for background and for Lorentz and polarization effects, but not for absorption $[\mu(\text{Mo } K\alpha) = 4.98$ cm$^{-1}]$. A total of 2447 intensities were reduced to 1964 structure factors, $F_m^2 \ge -2.75\sigma(F^2)$ and $0.060 < (\sin\theta)/\lambda < 0.639$ Å^{-1}.

Table 1. *Fractional coordinates* (S × 10^5; C, N × 10^4; H × 10^3)

	x	y	z
S(1)	23944 (7)	49750 (9)	23269 (5)
S(2)	14286 (7)	42605 (10)	43721 (5)
C(3)	2686 (2)	4109 (3)	3668 (2)
N(4)	3892 (2)	3301 (2)	4152 (2)
C(5)	4994 (4)	3093 (6)	3584 (4)
C(6)	4189 (4)	2516 (5)	5282 (3)
N(7)	−1033 (2)	3584 (3)	1929 (2)
C(8)	−1175 (4)	3925 (5)	0715 (3)
C(9)	−1995 (4)	4625 (5)	2399 (3)
H(51)	583 (4)	353 (4)	403 (3)
H(52)	506 (4)	202 (6)	343 (3)
H(53)	482 (3)	384 (4)	289 (3)
H(61)	494 (4)	295 (5)	564 (3)
H(62)	437 (4)	129 (5)	521 (3)
H(63)	347 (4)	258 (4)	562 (3)
H(71)	−012 (3)	382 (4)	229 (2)
H(72)	−120 (3)	245 (4)	207 (2)
H(81)	−105 (3)	514 (5)	061 (2)
H(82)	−211 (3)	347 (4)	030 (2)
H(83)	−047 (3)	323 (4)	050 (2)
H(91)	−180 (3)	584 (5)	229 (3)
H(92)	−293 (3)	438 (4)	199 (3)
H(93)	−180 (3)	436 (4)	321 (3)

The structure was solved by direct methods (Long, 1965). The H atom positions were obtained from a difference synthesis. The full-matrix least-squares refinements allowed anisotropic thermal parameters for the non-hydrogen atoms and isotropic for H. In the last cycle 138 independent parameters were varied and all the structure factors were included. The resulting fractional coordinates are given in Table 1.* The largest (parameter $\Delta)/\sigma$ were 0.6 $[y$-C(5), y-H(52), z-H(61)$]$, 0.4 $[x$-C(6),x-H(51)$]$ and 0.3 $[y$-H(63)$]$. The expression minimized was $\sum w\delta^2$, where $\delta = F_m^2 - F_c^2$ and $w^{-1} = \sigma^2_{\text{count}}(F^2) + (0.02F_m^2)^2 = \sigma^2(F^2)$. The refinements converged at $R(F^2) = \sum |\delta|/\sum F_m^2 = 0.053$ and $R_w(F^2) = (\sum w\delta^2/\sum wF_m^4)^{1/2} = 0.074$. The quantity $|\delta|/\sigma(F^2)$ was >2.0 for 149 reflexions; the largest value was 8.0 for the reflexion $10\bar{2}$. A normal ΔR probability plot (Abrahams & Keve, 1971) had a slope of 0.90 and an intercept of -0.05. A difference synthesis showed some spurious peaks of density less than 0.14 e Å^{-3}. Atomic scattering factors for S, N, C and H, and the f' and f'' anomalous dispersion corrections for S, N and C were taken from *International Tables for X-ray Crystallography* (1974). Lundgren (1976) has described the computer programs. The calculations were performed on the IBM 370/155 and IBM 1800 computers in Uppsala.

Discussion. The chain: the molecular ions are linked by hydrogen bonds into one-dimensional zig-zag chains, extending along b. There is one independent chain in the structure. Part of a chain is shown in Fig. 1. The chains are packed at approximately the van der Waals distances (Bondi, 1964) in the structure.

The molecular ions in the chain are described by a 2_1 screw axis. One of the ammonium protons forms inter-molecular bonds with both S atoms in one anion. The H—S distances (corresponding N—S distances are given in brackets) are H(71)$\cdots$S(1) $= 2.66$ (3) Å $[3.490$ (2) Å$]$ and H(71)$\cdots$S(2) $= 2.61$ (3) Å $[3.343$ (2) Å$]$. The other proton interacts with both S atoms in a 2_1-related anion as H(72)$\cdots$S(1^i) $= 2.46$ (3) Å $[3.326$ (3) Å$]$ and H(72)$\cdots$S(2^i) $= 2.99$ (3) Å $[3.662$ (3) Å$]$. H—S distances of similar or somewhat shorter values have been observed in hydrazinium($+1$) hydrogen sulfide (Lazarini & Vardjan-Jarec, 1975). The long distance H(72)$\cdots$S(2^i) is close to the van der Waals distance of 3.00 Å (Bondi, 1964). Each 'bite' atom (ammonium proton or dithiocarbamate S atom) participates in two intermolecular bonds. Thus, the coordination number per molecular ion is 4.

Intermolecular distances between ammonium protons, shorter than 4.0 Å, are intra-chain

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33698 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH 1 2HU, England.
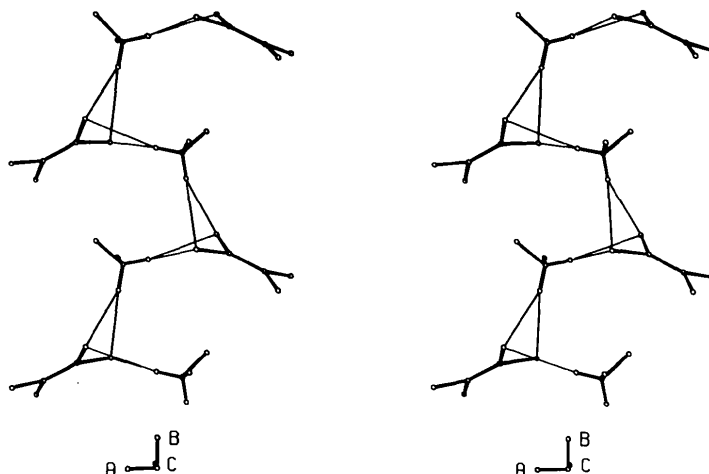
Fig. 1. A part of the one-dimensional chain. Only the ammonium protons have been drawn, other H atoms are omitted for clarity.

H(71)···H(72^i) of 3·10 (4) Å and H(72)···H(72^i) of 3·88 (7) Å. Intermolecular intra-chain S—S distances are greater than 5·00 Å. The inter-chain distance S(2)···S(2^{ii}) of 3·762 (2) Å is notably short. This type of distance generally appears to be greater than 3·9 Å (Jennische, 1975). The van der Waals distance between 'uncharged' S atoms is 3·66 Å (Bondi, 1964).

The dimethylammonium cation: distances and angles are given in Fig. 2. The cation has pseudo *mm* symmetry and is very similar to that in the X-ray structure of dimethylammonium hydrogen oxalate (Thomas & Pramatus, 1975). The two methyl protons close to the non-hydrogen atom plane have the configuration of maximal separation; the atoms H(83) and H(93) are located at 0·05 (3) and 0·04 (3) Å, respectively, from the plane through atoms N(7), C(8) and C(9).

The dimethyldithiocarbamate anion: distances and angles are given in Fig. 3. The non-hydrogen atom geometry is very similar to that in the *mm*-symmetric anion in cesium dimethyldithiocarbamate (Wahlberg,

1976). The present methyl groups have the same configurations as those in the cation. The configurations in cesium dimethyldithiocarbamate are a little different since the methyl protons in the dithiocarbamate plane are minimally separated.

The distance S(2)—C(3) is *ca* 4σ shorter than S(1)—C(3). This discrepancy may be the result of S(1) forming two normal intermolecular hydrogen bonds, while S(2) participates in one normal bond and one quite long intermolecular interaction. The mean C—S distance is 1·711 Å. The S₂CNC₂ part of the anion is generally observed as 'planar within experimental errors'. This is ascribed to sp^2 hybridization and a delocalized π-bond. In the present anion the following distances to the least-squares plane through the non-hydrogen atoms are observed: S(1) 0·000 (1), S(2) 0·001 (1), C(3) −0·007 (2), N(4) −0·005 (2), C(5) 0·020 (5), C(6) 0·010 (4), H(53) −0·16 (3) and H(63) 0·05 (3) Å. The 'planar part' is a little bent: both C(3) and N(4) lie significantly below the other non-hydrogen atoms.
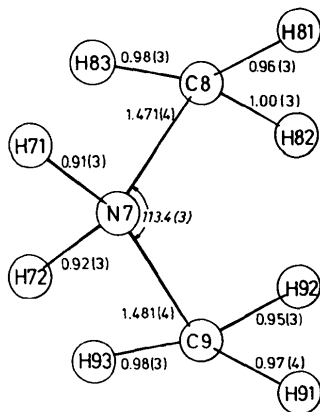

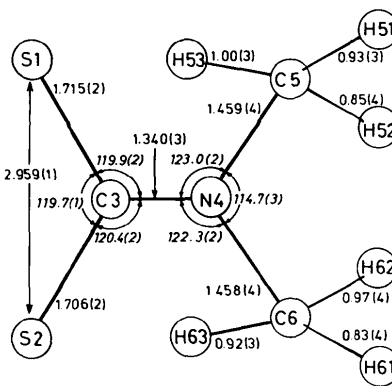Fig. 2. Distances (Å) and angles (°) in the dimethylammonium cation.

Fig. 3. Distances (Å) and angles (°) in the dimethyldithio-carbamate anion.

The C(methyl)—N bonds are shorter in the anion than in the cation. The differences are between 3σ and 6σ.

I wish to express my gratitude to Professor Ivar Olovsson for the facilities placed at my disposal. I am indebted to Dr Rolf Hesse and Dr Per Jennische for stimulating discussions. The English text was checked by Mr Rajesh Kumar.

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3-Methyl-α,α-diphenyl-1-isoquinolineethanol

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Abstract. $C_{24}H_{21}NO$, monoclinic, $P2_1/c$, $a = 13.990$ (1), $b = 9.703$ (1), $c = 15.053$ (1) Å, $\beta = 117.32$ (1)°, $Z = 4$, $\rho_m = 1.241$, $\rho_c = 1.242$ (1) g cm^{-3}, $\mu = 5.94$ cm^{-1}, final $R(F^2) = 0.061$. The condensation of benzophenone with the monolithiated derivative of 1,3-dimethylisoquinoline is shown to produce the title compound. The geometrical parameters of the aromatic rings are normal. The alcohol group is involved in an intramolecular hydrogen bond with the isoquinoline nitrogen [O⋯N, 2.702 (2) Å]. The average aromatic C—C distance (1.386 Å) and C—N distance (1.340 Å) are not unusual. The isoquinoline distances are compared with those found in another 1,3 derivative.

Introduction. The compound was prepared by condensation of benzophenone with the monolithiated derivative of 1,3-dimethylisoquinoline (McClure & Kaiser, 1977).
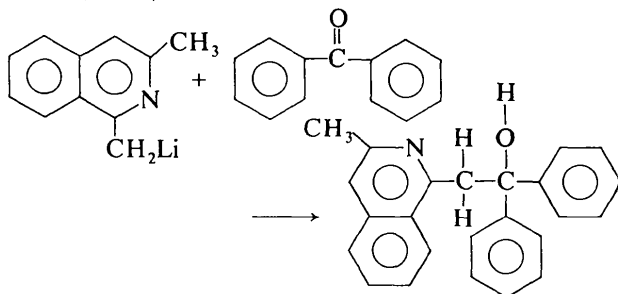
The prismatic crystals were obtained by slow evapora-

tion of an ether solution. Precession photographs gave the systematic absences $h0l$, $l = 2n + 1$ and $0k0$, $k = 2n + 1$, establishing the space group as $P2_1/c$. A crystal (approximate dimensions $0.25 \times 0.3 \times 0.5$ mm) was mounted on a programmed Picker four-circle diffractometer. Accurate cell dimensions and an orientation matrix were obtained by a least-squares fit* to the setting angles of 24 manually centered reflections, and a complete intensity data set was collected with Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å) for $2\theta \le 97°$. The θ–2θ scan technique ($1°$ min^{-1}) was used with a variable width to account for α_1–α_2 splitting. Stationary counter–stationary crystal background measurements were made for 20 s at both ends of each scan. Intensities of 2348 reflections were measured, of which 1862 were independent. Three standards were measured after every fifty reflections and indicated no significant crystal decomposition. The agreement factor between equivalent reflections was 1.1%. The 1840