

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

O(1)—P	1.572 (4)	O(2)—P	1.562 (4)
O(3)—P	1.456 (3)	N(1)—P	1.684 (4)
C(1)—S	1.690 (4)	C(4)—O(1)	1.487 (6)
C(6)—O(2)	1.493 (5)	C(1)—N(1)	1.412 (5)
C(3)—N(1)	1.486 (6)	C(1)—N(2)	1.320 (6)
C(2)—N(2)	1.455 (6)	C(11)—C(2)	1.519 (7)
C(21)—C(3)	1.522 (6)	C(5)—C(4)	1.506 (8)
C(6)—C(5)	1.499 (9)	C(7)—C(6)	1.511 (8)
C(12)—C(11)	1.365 (7)	C(16)—C(11)	1.402 (9)
C(13)—C(12)	1.385 (7)	C(14)—C(13)	1.395 (9)
C(15)—C(14)	1.375 (7)	C(16)—C(15)	1.391 (7)
C(22)—C(21)	1.403 (8)	C(26)—C(21)	1.363 (7)
C(23)—C(22)	1.386 (6)	C(24)—C(23)	1.385 (7)
C(25)—C(24)	1.401 (9)	C(26)—C(25)	1.384 (7)
O(2)—P—O(1)	105.3 (2)	O(3)—P—O(1)	115.2 (2)
O(3)—P—O(2)	115.0 (2)	N(1)—P—O(1)	102.7 (2)
N(1)—P—O(2)	104.2 (2)	N(1)—P—O(3)	113.1 (2)
C(4)—O(1)—P	118.1 (4)	C(6)—O(2)—P	120.7 (3)
C(1)—N(1)—P	124.3 (3)	C(3)—N(1)—P	116.7 (3)
C(3)—N(1)—C(1)	118.6 (4)	C(2)—N(2)—C(1)	123.6 (4)
N(1)—C(1)—S	121.0 (3)	N(2)—C(1)—S	122.1 (3)
N(2)—C(1)—N(1)	116.9 (4)	C(11)—C(2)—N(2)	116.9 (5)
C(21)—C(3)—N(1)	116.6 (5)	C(5)—C(4)—O(1)	108.0 (4)
C(6)—C(5)—C(4)	113.4 (4)	C(5)—C(6)—O(2)	109.7 (5)
C(7)—C(6)—O(2)	105.4 (4)	C(7)—C(6)—C(5)	115.8 (4)
C(12)—C(11)—C(2)	118.6 (5)	C(16)—C(11)—C(2)	122.7 (4)
C(16)—C(11)—C(12)	118.7 (4)	C(13)—C(12)—C(11)	122.1 (6)
C(14)—C(13)—C(12)	119.4 (5)	C(15)—C(14)—C(13)	118.9 (5)
C(16)—C(15)—C(14)	121.4 (6)	C(15)—C(16)—C(11)	119.5 (4)
C(22)—C(21)—C(3)	121.4 (4)	C(26)—C(21)—C(3)	120.3 (5)
C(26)—C(21)—C(22)	118.3 (4)	C(23)—C(22)—C(21)	120.5 (4)
C(24)—C(23)—C(22)	120.3 (6)	C(25)—C(24)—C(23)	119.4 (5)
C(26)—C(25)—C(24)	119.0 (5)	C(25)—C(26)—C(21)	122.5 (6)

Table 3. Torsion angles (°) in the 1,3,2-dioxaphosphorinane ring

E.s.d.'s are in the range 0.5–1.0°.			
P—O(2)—C(6)—C(5)	47.8	C(5)—C(4)—O(1)—P	–56.7
O(2)—C(6)—C(5)—C(4)	–55.0	C(4)—O(1)—P—O(2)	45.7
C(6)—C(5)—C(4)—O(1)	59.8	O(1)—P—O(2)—C(6)	–41.2

As in other *N,N'*-dibenzyl trisubstituted thioureas (Karolak-Wojciechowska *et al.*, 1979, 1983), the P and H(3) atoms are *trans* oriented with respect to sulphur.

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## Polarized Ethylenes: Structures of [Dimethylamino(methylthio)methylene]malononitrile (I), C<sub>7</sub>H<sub>9</sub>N<sub>3</sub>S, and [Bis(dimethylamino)methylene]malononitrile (II), C<sub>8</sub>H<sub>12</sub>N<sub>4</sub>

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**Abstract.** (I):  $M_r = 167$ , orthorhombic,  $Pna2_1$ ,  $Z = 4$ ,  $\lambda = 0.7107 \text{ \AA} = 0.299 \text{ mm}^{-1}$ ,  $F(000) = 352$ ,  $T = 293 \text{ K}$ . (II):  $M_r = 164$ , orthorhombic,  $Pcab$ ,  $Z = 8$ ,  $a = 7.899 (3)$ ,  $b = 8.670 (2)$ ,  $c = 12.956 (6) \text{ \AA}$ ,  $V = 887.2 \text{ \AA}^3$ ,  $D_m = 1.25$ ,  $D_x = 1.250 \text{ Mg m}^{-3}$ ,  $\mu(\text{Mo K}\alpha)$ ,  $a = 7.876 (2)$ ,  $b = 14.430 (4)$ ,  $c = 16.312 (5) \text{ \AA}$ ,  $V =$

0108-2701/83/050589-04\$01.50

The molecular configuration in the present structure is sterically favoured and is stabilized by an intramolecular hydrogen bond between N(1) and O(3) of 2.623 (6) Å.

The C=S bond length of 1.690 (4) Å has a similar value to that found for thiourea [1.70 (1) Å, Truter, 1967].

In the molecule there are two C(*sp*<sup>2</sup>)-N bonds. The C(1)-N(1) bond of 1.412 (5) Å is longer by 0.09 Å than C(1)-N(2) = 1.320 (6) Å. This lengthening is a consequence of the presence of the electronegative phosphoryl group bonded to N(1). For the same reason the P-N(1) bond of 1.684 (4) Å is shorter than the generally accepted single P-N bond (1.77 Å).

The configuration at the P atom in *N*-phosphorylthiourea (2a) is identical with that in the parent thioacid [*cis*-(1)] (the methyl group in the 4-position of the dioxaphosphorinane ring is in both cases *trans* with respect to the phosphoryl oxygen). This result proves that the S→N migration of the phosphoryl group in *S*-phosphorylisothiourea (3) proceeds with retention of configuration at the P atom.

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1853.9 Å<sup>3</sup>,  $D_m = 1.18$ ,  $D_x = 1.175$  Mg m<sup>-3</sup>,  $\mu(\text{Mo } K\alpha, \lambda = 0.7107 \text{ \AA}) = 0.083$  mm<sup>-1</sup>,  $F(000) = 704$ ,  $T = 293$  K. Both structures were solved by direct methods and refined to  $R = 4.1\%$  (542 significant reflections) for (I) and  $R = 4.9\%$  (709 significant reflections) for (II). The C=C bond lengths are 1.397 (8) Å in (I) and 1.407 (4) Å in (II). The molecules are twisted about the C=C bond by 21.7° in (I) and 29.3° in (II).

**Introduction.** As part of a programme of X-ray studies on the structure and conformation of crystalline 'push-pull' ethylenes, we reported earlier the results obtained for 2-benzoyl-3-dimethylamino-3-methylthio-2-propenenitrile, (1,3-dimethyl-2-imidazolidinylidene)malononitrile and (1,3-dimethyl-2-perhydropyrimidinylidene)malononitrile (Adhikesavalu & Venkatesan, 1981, 1982).

In this paper our findings on the molecular geometry of (I) and (II) are discussed. Samples of (I) and (II) were provided by Professor Jan Sandström, University of Lund, Sweden.

**Experimental.** Single crystals of (I) were obtained from a mixture of toluene and ligroin and those of (II) from toluene by slow evaporation. Crystals of (I) and (II) with approximate dimensions 0.30 × 0.30 × 0.1 mm and 0.25 × 0.30 × 0.20 mm, respectively, used for data collection; preliminary Weissenberg photographs indicated that crystals of (I) and (II) are orthorhombic with four and eight molecules, respectively, in the unit cell; intensity data collected on a Nonius CAD-4 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation,  $\omega/2\theta$  mode, scan speed 1° min<sup>-1</sup>,  $\theta < 23^\circ$ ; 650 reflections for (I) [1266 for (II)] were collected of which 542 [709] were judged significant ( $|F_o| \geq 3\sigma|F_o|$ ); intensities not corrected for absorption. The normalized structure factor distribution was clearly acentric for (I) and hence the space group  $Pna2_1$  was chosen for the structure solution and refinement. Both structures solved by direct methods using the program *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolson, 1980); full-matrix refinement of a scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, located from a difference map) converged to an  $R$  factor of 4.1% ( $R_w = 3.7\%$ ) for (I) and 4.9% ( $R_w = 4.5\%$ ) for (II);\*  $\sum w(|F_o| - |F_c|)^2$  minimized, where  $w = 1.1263/\sigma^2[|F_o| + 0.0004 \times (|F_o|)^2]$  and  $w = 1.5238/\sigma^2[|F_o| + 0.0004(|F_o|)^2]$  were used for (I) and (II) respectively; *SHELX76* (Sheldrick, 1976) was used for the refinement.

\* Lists of structure factors and anisotropic thermal parameters for (I) and (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38299 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** Positional coordinates are given in Tables 1 and 2. Bond lengths and bond angles involving non-hydrogen atoms are provided in Table 3, and Table 4 gives some of the important torsion angles. Figs. 1 and 2 give the numbering of atoms for (I) and (II) respectively. The main objective in selecting these two compounds was to investigate the bond lengths of the ethylene bond and the rotation about this bond in these two molecules; (I) carries SMe and NMe<sub>2</sub> groups as donors whereas (II) has two NMe<sub>2</sub> groups. The barrier to rotation about the C=C bond as measured from NMR studies is 55.2 kJ mol<sup>-1</sup> for (I) (Sandström & Wennerbeck, 1978). For (II), it could not be measured for reasons of symmetry (Kessler, 1968). It is known that the barrier is less for an ethylene with one SMe and one NMe<sub>2</sub> group than for one with two NMe<sub>2</sub> groups as donors, suggesting that the combination one SMe and one NMe<sub>2</sub> group is a better donor compared to two NMe<sub>2</sub> groups (Sandström & Wennerbeck, 1978). However, this is not borne out by X-ray results. The C=C bond lengths in (I) and (II) are 1.397 (8) and 1.407 (4) Å respectively; these differ significantly from the normal C=C bond found in ethylene [1.336 (2) Å; Bartell, Roth, Hollowell, Kuchitsu & Young (1965)], but there is no significant difference between these two bond lengths.

Owing to steric effects both (I) and (II) are twisted about the C=C bond. The twist angle, defined as the angle between the plane through C(1), C(2) and C(4) and the plane through C(3), N(3) and S, is 21.7 (5)° in (I) and 29.3 (5)° in (II). The smaller twist in (I) is due to the orientation of the methyl group on S away from

Table 1. Final positional parameters ( $\times 10^4$ , for H  $\times 10^3$ ) and isotropic thermal parameters for (I)

		$B_{\text{eq}}(\text{\AA}^2 \times 10^2)$			
S		2494 (2)	1934 (1)	2939	361 (3)
N(1)		1121 (10)	4121 (7)	6468 (5)	630 (21)
N(2)		507 (9)	-209 (7)	4977 (5)	558 (18)
N(3)		1719 (7)	4791 (5)	3606 (4)	403 (12)
C(1)		1144 (9)	3489 (7)	5704 (6)	407 (17)
C(2)		1183 (7)	2659 (6)	4764 (4)	311 (13)
C(3)		1742 (8)	3292 (6)	3833 (5)	297 (14)
C(4)		817 (8)	1049 (7)	4847 (5)	373 (17)
C(5)		619 (11)	5863 (7)	4155 (7)	631 (25)
C(6)		2857 (10)	5487 (7)	2868 (9)	639 (24)
C(7)		1699 (9)	2594 (8)	1695 (5)	420 (18)
Bonded to		$x$	$y$	$z$	$B(\text{\AA}^2)$
H(1)	C(7)	79 (9)	314 (7)	172 (6)	2.6 (1.6)
H(2)	C(7)	281 (8)	311 (6)	127 (5)	3.0 (1.4)
H(3)	C(7)	133 (11)	154 (10)	133 (7)	3.5 (2.1)
H(4)	C(6)	351 (12)	464 (12)	259 (9)	9.0 (3.4)
H(5)	C(6)	224 (15)	577 (14)	226 (11)	9.6 (3.7)
H(6)	C(6)	328 (17)	636 (14)	311 (13)	11.6 (3.9)
H(7)	C(5)	125 (13)	636 (12)	469 (10)	7.1 (3.0)
H(8)	C(5)	52 (14)	679 (11)	368 (8)	7.9 (2.5)
H(9)	C(5)	-15 (17)	544 (14)	430 (10)	8.8 (3.5)

For non-H atoms  $B_{\text{eq}} = \frac{1}{3} \sum_i \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$ .

For H atoms the temperature factor is of the form  $T = \exp(-B \sin^2 \theta / \lambda^2)$ .

Table 2. Final positional parameters ( $\times 10^4$ , for H  $\times 10^3$ ) and isotropic thermal parameters for (I)For non-H atoms  $B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$ .For H atoms the temperature factor is of the form  $T = \exp(-B \sin^2 \theta / \lambda^2)$ .

	x	y	z	$B_{eq}(\text{\AA}^2 \times 10^2)$
N(1)	2410 (6)	1166 (3)	3874 (2)	702 (13)
N(2)	4564 (6)	2907 (3)	1938 (2)	715 (14)
N(3)	2863 (4)	3530 (2)	4653 (2)	345 (8)
N(4)	2528 (4)	4403 (2)	3475 (2)	368 (8)
C(1)	2774 (6)	1889 (3)	3634 (2)	453 (12)
C(2)	3182 (5)	2786 (2)	3350 (2)	336 (10)
C(3)	2862 (4)	3579 (2)	3827 (2)	298 (9)
C(4)	3931 (6)	2853 (3)	2567 (2)	473 (12)
C(5)	3895 (7)	2868 (4)	5098 (3)	477 (14)
C(6)	1700 (8)	4095 (3)	5135 (3)	487 (15)
C(7)	1676 (8)	4479 (4)	2686 (3)	522 (15)
C(8)	3103 (7)	5276 (3)	3840 (3)	558 (14)

## Bonded

to	x	y	z	$B(\text{\AA}^2)$
H(1) C(5)	496 (6)	264 (3)	477 (3)	7.7 (1.3)
H(2) C(5)	430 (5)	315 (3)	545 (2)	4.1 (1.1)
H(3) C(5)	323 (7)	232 (4)	530 (3)	10.4 (1.7)
H(4) C(6)	72 (6)	434 (3)	483 (2)	6.2 (1.3)
H(5) C(6)	131 (5)	372 (3)	551 (2)	5.3 (1.1)
H(6) C(6)	228 (5)	461 (3)	536 (2)	4.8 (0.9)
H(7) C(7)	112 (5)	386 (3)	250 (2)	5.8 (0.9)
H(8) C(7)	73 (7)	498 (4)	269 (3)	8.1 (1.3)
H(9) C(7)	248 (6)	461 (3)	230 (2)	5.7 (1.2)
H(10) C(8)	401 (8)	509 (4)	449 (3)	12.8 (1.7)
H(11) C(8)	353 (5)	563 (3)	340 (3)	6.9 (1.2)
H(12) C(8)	221 (7)	560 (4)	417 (3)	10.6 (1.7)

Table 4. Torsion angles ( $^\circ$ ) with e.s.d.'s in parentheses

	(I)	(II)
C(1)–C(2)–C(3)–N(3)	–25.5 (9)	28.8 (5)
C(1)–C(2)–C(3)–N(4)		–150.4 (4)
C(1)–C(2)–C(3)–S	155.0 (5)	
C(4)–C(2)–C(3)–N(3)	162.8 (6)	–151.0 (5)
C(4)–C(2)–C(3)–N(4)		29.8 (5)
C(4)–C(2)–C(3)–S	–16.7 (7)	
C(2)–C(3)–N(3)–C(5)	–20.5 (9)	29.5 (5)
C(2)–C(3)–N(3)–C(6)	154.5 (6)	–145.1 (4)
C(2)–C(3)–N(4)–C(7)		30.8 (5)
C(2)–C(3)–N(4)–C(8)		–145.1 (4)
C(2)–C(3)–S–C(7)	139.8 (5)	
N(3)–C(3)–N(4)–C(7)		–148.4 (4)
N(3)–C(3)–N(4)–C(8)		35.7 (5)
N(3)–C(3)–S–C(7)	–39.7 (6)	
N(4)–C(3)–N(3)–C(5)		–151.2 (4)
N(4)–C(3)–N(3)–C(6)		34.2 (5)
S–C(3)–N(3)–C(5)	159.0 (3)	
S–C(3)–N(3)–C(6)	–26.0 (8)	

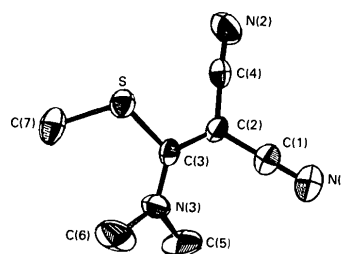


Fig. 1. A perspective view of a molecule of (I).

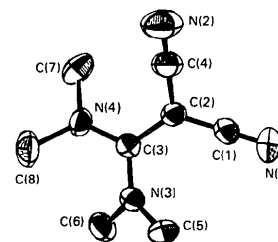


Fig. 2. A perspective view of a molecule of (II).

Table 3. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) involving non-H atoms, with e.s.d.'s in parentheses

	(I)	(II)
C(1)–N(1)	1.132 (10)	1.151 (6)
C(1)–C(2)	1.415 (9)	1.412 (3)
C(2)–C(3)	1.397 (8)	1.407 (4)
C(2)–C(4)	1.430 (8)	1.410 (5)
C(4)–N(2)	1.130 (9)	1.143 (5)
C(3)–N(3)	1.333 (7)	1.349 (5)
C(3)–S	1.755 (6)	
C(3)–N(4)		1.346 (4)
C(5)–N(3)	1.458 (9)	1.449 (6)
C(6)–N(3)	1.444 (10)	1.457 (6)
C(7)–S	1.822 (7)	
C(7)–N(4)		1.456 (6)
C(8)–N(4)		1.465 (5)
N(1)–C(1)–C(2)	178.4 (8)	178.5 (5)
C(1)–C(2)–C(3)	123.4 (5)	121.6 (3)
C(1)–C(2)–C(4)	115.3 (5)	117.1 (3)
C(2)–C(4)–N(2)	175.6 (7)	178.9 (5)
C(4)–C(2)–C(3)	120.8 (5)	121.3 (3)
C(2)–C(3)–N(3)	124.7 (5)	120.6 (3)
C(2)–C(3)–N(4)		121.2 (3)
C(3)–N(3)–C(5)	121.5 (5)	122.3 (3)
C(3)–N(3)–C(6)	123.0 (6)	120.6 (3)
C(5)–N(3)–C(6)	115.3 (6)	116.8 (4)
C(3)–N(4)–C(7)		122.3 (3)
C(3)–N(4)–C(8)		121.7 (3)
C(7)–N(4)–C(8)		115.9 (4)
C(3)–S–C(7)	104.9 (2)	
S–C(3)–N(3)	120.9 (5)	
N(4)–C(3)–N(3)		118.2 (3)
C(2)–C(3)–S	114.4 (4)	

the cyano group. The torsion angle C(2)–C(3)–S–C(7) is 139.8 (5) $^\circ$ .

There are significant changes in the bond lengths due to conjugation. N(3)–C(3) [1.333 (7)  $\text{\AA}$ ] in (I), and N(3)–C(3) [1.349 (5)  $\text{\AA}$ ] and N(4)–C(3) [1.346 (4)  $\text{\AA}$ ] in (II) are very much shorter than the 1.452 (2)  $\text{\AA}$  reported for a pure  $\text{N}=\text{C}_{sp^2}$  single bond (Ammon, Mazzocchi, Regan & Colicelli, 1979). The  $\text{C}_{sp^2}-\text{C}_{sp}$  bond length in ethylenetetracarbonitrile (Little, Pautler & Coppens, 1971) is 1.437 (2)  $\text{\AA}$ . In comparison, the  $\text{C}_{sp^2}-\text{C}_{sp}$  bond lengths [C(2)–C(4) 1.430 (8), C(2)–C(1) 1.415 (9)  $\text{\AA}$ ] in (I) do not differ significantly whereas those in (II) [C(2)–C(1) 1.412 (3), C(2)–C(4) 1.410 (5)  $\text{\AA}$ ] differ significantly. The S–C(3) bond in (I) is 1.755 (6)  $\text{\AA}$  whereas the S–C $_{sp^2}$  bond in 1,1-bis(methylthio)ethylene as deter-

mined from electron diffraction is slightly longer but not to a highly significant extent [1.767 (5) Å; Jandal, Seip & Torgrimsen (1976)].

There is some pyramidality at the olefinic C and the donor N atoms. C(2) in (I) deviates from the plane of the bonded atoms by 0.056 (4) Å whereas C(3) is planar. There is no significant pyramidality at the olefinic C atoms in the case of (II). N(3) in (I) deviates from the plane of the bonded atoms by 0.034 (5) Å. In (II), N(3) and N(4) deviate by 0.037 (4) and 0.028 (3) Å respectively from the planes of their bonded atoms. In (I), the NMe<sub>2</sub> group is twisted by 23 (1)° with respect to the least-squares plane defined by N(3), S, C(3) and C(2). In (II), the N(3)Me<sub>2</sub> group is twisted by 32 (1)° whereas the N(4)Me<sub>2</sub> group is twisted by 34 (1)° with respect to the least-squares plane through N(3), N(4), C(3) and C(2).

Figs. 3 and 4 show the packing of the molecules in the unit cells of (I) and (II) respectively.

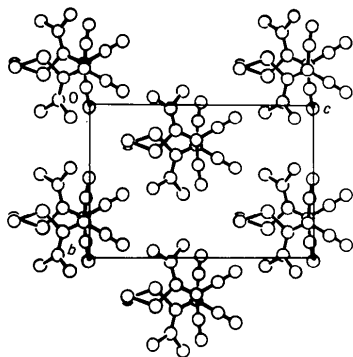


Fig. 3. Packing of the molecules viewed down *a* for (I).

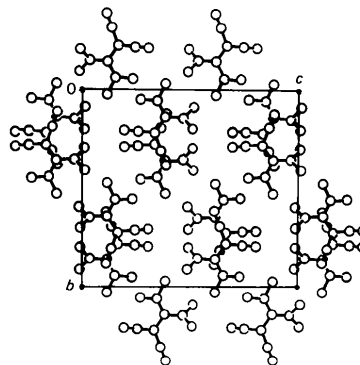


Fig. 4. Packing of the molecules viewed down *a* for (II).

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### Structure of Tetramethyl Bicyclo[2.2.2]oct-5-ene-2-endo,3-exo,7-endo,8-exo-tetracarboxylate,† C<sub>16</sub>H<sub>20</sub>O<sub>8</sub>

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**Abstract.** *M<sub>r</sub>* = 340, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 6.080 (2), *b* = 11.468 (4), *c* = 22.901 (5) Å, β = 97.67 (4)°,

† IUPAC name: Tetramethyl (5*R*\*,6*R*\*,7*S*\*,8*S*\*)-Bicyclo[2.2.2]oct-2-ene-5,6,7,8-tetracarboxylate.

*V* = 1582.5 (9) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.427 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.73 mm<sup>-1</sup>, *F*(000) = 720, *T* = 295 K. The structure was solved by direct methods and refined by full-matrix least squares to a conventional *R* = 0.060 for 1949 observed reflections. The two