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The Structure of Octamethyltetraoxaquaterene*

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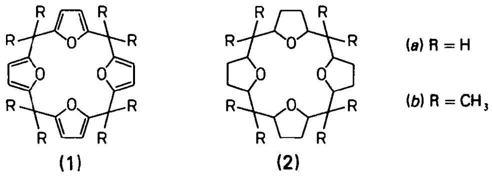
Abstract. $C_{28}H_{32}O_4$, $M_r = 432{\cdot}6$, tetragonal, $P4_1$, $a = b = 10{\cdot}055\ (2)$, $c = 24{\cdot}311\ (7)$ Å, $Z = 4$, $V = 2458\ (1)$ Å^3, $D_x = 1{\cdot}169$ Mg m^{-3}, $F(000) = 1728$, Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 0{\cdot}72$ cm^{-1}, $T = 295$ K. $R(F) = 0{\cdot}046$ for 1315 reflexions [$I > 3\sigma(I)$] and 386 variables. The molecule has approximate symmetry $\bar{4}$ with the oxygens almost coplanar, the furan groups make angles of 76–80° with the plane through the C and O atoms. The diameter of the central cavity is 2·02 Å. The molecules pack in layers perpendicular to *c*, diffuse scattering suggests stacking faults and energy calculations show how these faults may occur.

Introduction. Although the crystal structures of a large number of crown ethers have been determined, little attention has been paid until recently to the tetraoxaquaterenes (1) which are 16-crown-4 ethers. The structures of two modifications of the fully hydrogenated compound (2)(*b*) and lithium perchlorate complex of one of them were determined by van Beylen, Roland, King & Aerts (1985). Recently the structures of two forms of the parent compound (1)(*a*) (Haas, Knipp, Sicken, Lex & Vogel, 1988) and its dication (Vogel, Haas, Knipp, Lex & Schmickler, 1988) have been reported. The structure of the octamethyl derivative (1)(*b*) has been determined and is described here.



(1) (2)

(*a*) R = H

(*b*) R = CH_3

Experimental. (1)(*b*) was synthesized by the condensation of acetone and furan (Chastrette, Chastrette & Sabadie, 1977; De Sousa Healy & Rest, 1981) and recrystallized from ethanol. A crystal, 0·26 × 0·31 × 0·35 mm, was mounted on a Huber diffractometer. Cell dimensions were determined from the setting angles of 16 reflexions with $16 < 2\theta < 22°$ measured with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å) at 2θ, ω, χ, φ; -2θ, $-\omega$, χ, φ; 2θ, ω, $180 + \chi$, φ; and -2θ, $-\omega$, $180 + \chi$, φ. Intensities were measured out to $(\sin\theta/\lambda)_{\max} = 0{\cdot}59$ Å^{-1} using an ω–2θ scan and Nb filtered Mo $K\alpha$ radiation, 2θ-scan width $(1{\cdot}6 + 0{\cdot}692\tan\theta)°$, divided into 50 steps, 1 s/step. Reflexions with $-11 \le h \le 11$, $0 \le k \le 11$, $0 \le l \le 28$ were measured giving 2210 independent reflexions of which 1315 had $I > 3\sigma_{cs}(I)$, $R_{\rm int} = 0{\cdot}059$. Reflexions 200 and 0,0,12 were monitored every 50 reflexions and showed no systematic variation in intensity. No absorption correction was required. As 004 was too intense to be corrected for dead-time it was remeasured at 25 kV and 10 mA.

The structure was determined using *MULTAN*80 (Main *et al.*, 1980), H-atom positions were determined from a difference map. Least-squares refinement (on *F*), all non-H atoms refined anisotropically, H atoms isotropically with a common $U_{\rm iso}$, isotropic extinction factor $g = 0{\cdot}010\ (2) \times 10^{-4}$, for $\bar{t} = 1$, corresponding to a minimum value of $I/I_{\rm corrected}$ of 0·725. The weighting scheme was $w = 1/\sigma(F)$ where $\sigma(F) = [\sigma_{cs}(F^2) + 1{\cdot}03F^2]^{1/2} - |F|$ and $\sigma_{cs}(F^2)$ is the standard deviation of F^2 from counting statistics. Final $R = 0{\cdot}046$,† $wR = 0{\cdot}046$, $S = 1{\cdot}32$, $(\Delta/\sigma)_{\max} = 0{\cdot}35$, $\Delta\rho = 0{\cdot}23\ (5)$ to $-0{\cdot}25\ (5)$ e Å^{-3}. Fractional coordinates are listed in Table 1, bond distances, angles and torsion angles in Table 2.

Computations were carried out on a VAX 11/780 computer with the following programs: *INTEG* – based on the Nelmes (1975) algorithm for integration of intensities; *DATAP* and *DSORT* (State University of New York) – data processing; modified *ORFLS* (Busing, Martin & Levy, 1962) – least-squares refinement; *ORFFE* (Busing, Martin & Levy, 1964) – geometry; *ORTEP* (Johnson, 1965) – drawings; *ENERGY* – energy calculations. Scattering curves those of Cromer & Mann (1968) for O and C, Stewart, Davidson & Simpson (1965) for H. Parameters for the van der Waals repulsion terms in the energy calculations were from *MMP*2 (Burkert & Allinger, 1982).

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* 2,2,7,7,12,12,17,17-Octamethyl-21,22,23,24-tetraoxapentacyclo-$[16.2.1.1^{3,6}.1^{8,11}.1^{13,16}]$tetracosa-3,5,8,10,13,15,18,20-octaene.

† Lists of structure factors, anisotropic thermal parameters and some bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51380 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional coordinates* ($\times 10^4$ *for* C *and* O, $\times 10^3$ *for* H) *and* U_{eq}

	x	y	z	U_{eq}*(Å^2 × 10^3)
O(11)	6801 (4)	1934 (4)	−13	24 (3)
C(12)	6983 (7)	872 (7)	331 (3)	31 (4)
C(13)	6684 (8)	1229 (8)	853 (4)	49 (5)
C(14)	6292 (7)	2602 (7)	844 (4)	42 (5)
C(15)	6384 (7)	2957 (6)	311 (4)	32 (4)
C(16)	6158 (6)	4273 (7)	17 (4)	34 (4)
C(17)	5069 (8)	4080 (8)	−428 (4)	44 (5)
C(18)	5653 (9)	5302 (8)	442 (4)	47 (5)
O(21)	8498 (4)	4865 (5)	75 (2)	35 (3)
C(22)	7383 (7)	4785 (7)	−255 (4)	37 (5)
C(23)	7701 (8)	5176 (7)	−760 (4)	45 (5)
C(24)	9057 (8)	5553 (8)	−747 (4)	49 (5)
C(25)	9525 (7)	5348 (6)	−238 (3)	38 (5)
C(26)	10832 (7)	5572 (6)	43 (4)	33 (4)
C(27)	10681 (9)	6573 (9)	515 (4)	50 (6)
C(28)	11811 (8)	6123 (9)	−379 (4)	48 (5)
O(31)	11411 (4)	3207 (4)	−85 (2)	31 (3)
C(32)	11329 (7)	4261 (7)	271 (3)	30 (4)
C(33)	11801 (8)	3898 (8)	767 (4)	49 (5)
C(34)	12183 (9)	2535 (9)	727 (4)	52 (6)
C(35)	11921 (7)	2151 (7)	213 (3)	30 (4)
C(36)	12083 (8)	843 (7)	−90 (4)	39 (5)
C(37)	13084 (9)	1005 (9)	−560 (4)	46 (5)
C(38)	12620 (9)	−209 (10)	322 (5)	52 (6)
O(41)	9760 (4)	236 (5)	48 (2)	33 (3)
C(42)	10801 (7)	385 (7)	−322 (4)	34 (4)
C(43)	10377 (8)	43 (9)	−822 (4)	50 (5)
C(44)	9011 (8)	−331 (8)	−767 (4)	46 (5)
C(45)	8637 (8)	−213 (8)	−243 (4)	38 (5)
C(46)	7406 (6)	−424 (8)	81 (4)	34 (4)
C(47)	7600 (10)	−1451 (9)	533 (4)	54 (6)
C(48)	6337 (8)	−915 (9)	−317 (5)	51 (6)
				U_{iso}(Å^2 × 10^2)
H(13)	672 (7)	58 (7)	110 (3)	51 (3)
H(14)	609 (6)	320 (6)	119 (3)	
H(171)	504 (7)	494 (7)	−62 (3)	
H(172)	428 (7)	376 (6)	−28 (3)	
H(173)	548 (6)	335 (6)	−75 (3)	
H(181)	540 (7)	612 (7)	24 (3)	
H(182)	637 (6)	556 (6)	74 (3)	
H(183)	481 (7)	493 (7)	68 (3)	
H(23)	723 (7)	526 (7)	−106 (3)	
H(24)	959 (6)	595 (6)	−103 (3)	
H(271)	1166 (7)	669 (6)	75 (3)	
H(272)	1017 (7)	620 (7)	79 (3)	
H(273)	1054 (6)	761 (7)	35 (2)	
H(281)	1198 (7)	562 (7)	−66 (3)	
H(282)	1157 (7)	707 (7)	−49 (3)	
H(283)	1268 (8)	607 (6)	−23 (3)	
H(33)	1183 (7)	445 (7)	107 (3)	
H(34)	1239 (7)	204 (7)	96 (3)	
H(371)	1319 (6)	13 (7)	−75 (3)	
H(372)	1394 (7)	134 (6)	−47 (3)	
H(373)	1280 (7)	166 (7)	−84 (3)	
H(381)	1312 (6)	−83 (7)	9 (3)	
H(382)	1198 (6)	−34 (7)	67 (3)	
H(383)	1341 (7)	17 (7)	43 (3)	
H(43)	1092 (6)	9 (6)	−119 (3)	
H(44)	851 (7)	−65 (7)	−104 (3)	
H(471)	834 (7)	−117 (7)	77 (3)	
H(472)	783 (6)	−238 (8)	35 (3)	
H(473)	680 (7)	−152 (6)	75 (3)	
H(481)	534 (7)	−99 (6)	−15 (3)	
H(482)	626 (7)	−27 (6)	−63 (3)	
H(482)	656 (6)	−179 (7)	−50 (3)	

* $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond lengths* (Å), *angles, and torsion angles* (°): *the atoms are labelled so that e.g.* C(2) *of side* 3 *is* C(32) *of Fig.* 1, *the side number refers to the first atom e.g.* C(6)–C(2)–O(1) *of side* 4 *is* C(46)–C(12)–O(11); *the standard deviations of the mean values are the sample standard deviations*

	side 1	side 2	side 3	side 4	mean
O(1)–C(2)	1·368 (8)	1·381 (8)	1·372 (8)	1·388 (8)	1·379 (5)
O(1)–C(5)	1·362 (8)	1·371 (7)	1·384 (7)	1·406 (8)	
C(2)–C(3)	1·353 (10)	1·328 (10)	1·344 (11)	1·332 (11)	1·339 (3)
C(4)–C(5)	1·347 (10)	1·339 (10)	1·335 (11)	1·336 (11)	
C(3)–C(4)	1·436 (12)	1·415 (11)	1·427 (12)	1·430 (11)	1·427 (4)
C(5)–C(6)	1·520 (9)	1·498 (9)	1·515 (10)	1·482 (10)	1·500 (5)
C(6)–C(2)	1·491 (9)	1·515 (9)	1·481 (10)	1·499 (10)	
C(6)–C(7)	1·552 (10)	1·535 (11)	1·533 (12)	1·520 (12)	1·537 (4)
C(6)–C(8)	1·548 (10)	1·526 (10)	1·552 (11)	1·529 (10)	

	side 1	side 2	side 3	side 4	mean
C(2)–O(1)–C(5)	106·1 (6)	108·0 (5)	106·5 (5)	108·3 (6)	107·2 (5)
O(1)–C(2)–C(3)	109·6 (7)	108·9 (6)	109·6 (6)	108·8 (6)	109·1 (6)
O(1)–C(5)–C(4)	112·2 (6)	107·6 (7)	110·0 (7)	106·4 (7)	
C(2)–C(3)–C(4)	107·6 (7)	106·9 (7)	107·1 (7)	106·9 (7)	107·3 (3)
C(3)–C(4)–C(5)	104·5 (6)	108·6 (7)	106·8 (7)	109·6 (7)	
O(1)–C(5)–C(6)	115·6 (7)	117·4 (6)	116·9 (6)	116·7 (7)	116·8 (3)
C(6)–C(2)–O(1)	115·7 (6)	117·4 (6)	116·3 (6)	118·0 (6)	
C(4)–C(5)–C(6)	132·2 (7)	135·0 (7)	133·2 (7)	136·9 (7)	134·1 (6)
C(6)–C(2)–C(3)	135·4 (7)	132·9 (7)	134·9 (7)	132·3 (7)	
C(5)–C(6)–C(7)	108·9 (6)	110·7 (6)	109·9 (7)	112·0 (7)	
C(5)–C(6)–C(8)	108·5 (6)	108·3 (6)	108·4 (7)	107·3 (7)	
C(5)–C(6)–C(2)	112·7 (6)	109·0 (6)	111·1 (6)	109·1 (7)	109·5 (2)
C(7)–C(6)–C(8)	108·6 (6)	109·2 (7)	109·0 (7)	109·2 (8)	
C(7)–C(6)–C(2)	108·4 (6)	109·2 (7)	108·7 (7)	109·5 (7)	
C(8)–C(6)–C(2)	109·7 (6)	110·5 (6)	109·7 (7)	109·7 (6)	

	side 1	side 2	side 3	side 4
C(2)–O(1)–C(5)–C(6)	178·1 (6)	177·6 (6)	178·8 (6)	−179·5 (7)
O(1)–C(5)–C(6)–C(7)	60·4 (8)	−59·7 (9)	62·8 (9)	−59·8 (10)
O(1)–C(5)–C(6)–C(8)	178·4 (6)	−179·3 (6)	−178·1 (6)	−179·6 (6)
O(1)–C(5)–C(6)–C(2)	−59·9 (8)	60·5 (8)	−57·5 (9)	61·6 (8)
C(5)–C(6)–C(2)–O(1)	−52·1 (8)	53·3 (8)	−56·5 (8)	55·0 (8)
C(7)–C(6)–C(2)–O(1)	−172·7 (6)	174·4 (6)	−177·6 (6)	177·9 (6)
C(8)–C(6)–C(2)–O(1)	68·9 (9)	−65·5 (8)	63·4 (9)	−62·3 (9)
C(6)–C(2)–O(1)–C(5)	179·9 (14)	178·2 (6)	−179·5 (6)	178·1 (6)

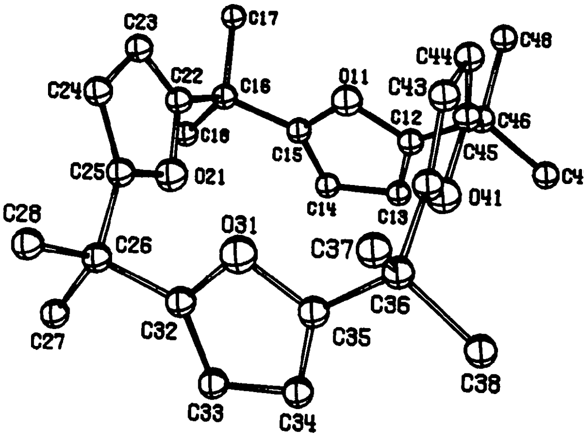
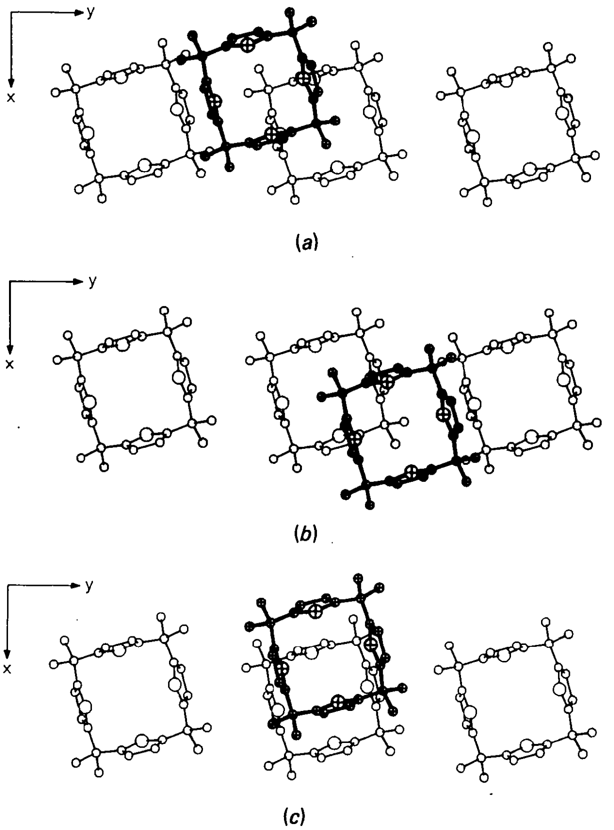


Fig. 1. A perspective view of one molecule showing the labelling of the atoms. H atoms, not shown, have the same numbers as the C atoms to which they are attached.

Discussion. The molecules, Fig. 1, have the 'square' ring conformation, with furan rings alternately up and down. The approximate symmetry is $\bar{4}$, for carbon and oxygen the r.m.s. deviation from ideal symmetry is 0·03 and the maximum deviation 0·06 Å, the corresponding values for $\bar{4}m$ are 0·07 Å and 0·13 Å. The O atoms are almost coplanar, the furan groups make angles of 76–80° with the best plane through the carbons and oxygens, the furan rings are tilted away from the ring opposite to them so that the dihedral angle between the rings is 24·0 and 24·1°. This conformation is similar to that found in the monoclinic form of the parent compound (2)(*a*) (Haas *et al.*, 1988), isomer 2 of the fully hydrogenated compound (2)(*b*) and its lithium picrate complex (van Beylen *et al.*, 1985), to 1,5,9,13-tetraoxacyclohexadecane (Groth, 1971), and to a nitrogen analogue (Sawitzki & von Schnering, 1976). The conformation is much closer to $\bar{4}m$ than that calculated (Cuthbertson & Glidewell, 1983) by the *MNDO* technique for an isolated molecule of (1)(*a*), *e.g.* the dihedral angles between neighbouring furan rings range from 87·5 to 92·9° whereas the theoretical value is 66·6°.

UV photoelectron spectroscopy of (1)(*b*) (Baker, Armen & Funaro, 1983) shows strong interactions among the 'individual' furan HOMO's. The authors say this can be attributed to through-bond coupling of the furan π systems or that the oxygens point away from the cavity. The structure supports through-bond coupling.

Bond distances and angles (Table 2) have the expected values, except that torsion angles O1—C5—C6—C2′, 59·9 (9)°, are larger than C5—C6—C2′—O1′, 54·2 (1·0)°. The diameter of the central cavity is 2·02 Å, *cf.* 1·8 Å for 15-crown-5 and 2·8 Å for 18-crown-6 (Lamb, Izatt, Christensen & Eatough, 1979).



(*a*)

(*b*)

(*c*)

Fig. 2. Packing and possible modes of disorder. A portion of a layer of molecules at $z = 0$ and one molecule at $z = \frac{1}{4}$ (drawn with thick lines) generated by (*a*) 4_1 symmetry operation, (*b*) 4_1 symmetry operation and a translation of $0{\cdot}35a + 0{\cdot}72c$, (*c*) 4_1 symmetry operation and a translation of $-0{\cdot}045a + 0{\cdot}360c$. (*a*), (*b*), and (*c*) correspond to the minima at (0,0), (0·35, 0·72), and (−0·045, 0·36) of the energy map shown in Fig. 3.

The molecules pack in layers perpendicular to *c* so that a dimethyl group of a molecule in one layer is over the centre of a ring in a neighbouring layer, Fig. 2(*a*). A characteristic feature of precession photographs is the presence of streaks parallel to c^* through the reciprocal-lattice points (though not 00*l*'s), which suggests disorder. Energy calculations in which a molecule was moved in the plane *c*/4 above a layer of molecules shows two almost equally deep minima, Fig. 3, one at (0, 0) corresponds to the position generated by the 4_1 axis, Fig. 2(*a*), the other at (0·35, 0·72) to the molecule being displaced, Fig. 2(*b*), by the vector $0{\cdot}35a + 0{\cdot}72c$, so that the diametrically opposed dimethyl group now lies over the centre of a ring, Fig. 2(*b*). The two ways of packing are remarkably similar and would be identical if the molecules had exact $\bar{4}m$ symmetry. There is a smaller minimum at (−0·045, 0·360), the corresponding packing is shown in Fig. 2(*c*). The stacking faults of the layers account for the streaks parallel to *c*,* since the projection of the disordered structure onto the *c* axis will still have a repeat distance of *c*/4 there are no streaks through the 00*l* reflexions.

I am indebted to Kim Nørkjær for synthesizing the compound and growing the crystals used and to the Carlsberg Foundation and to the Danish Science Research Council for the diffractometer.

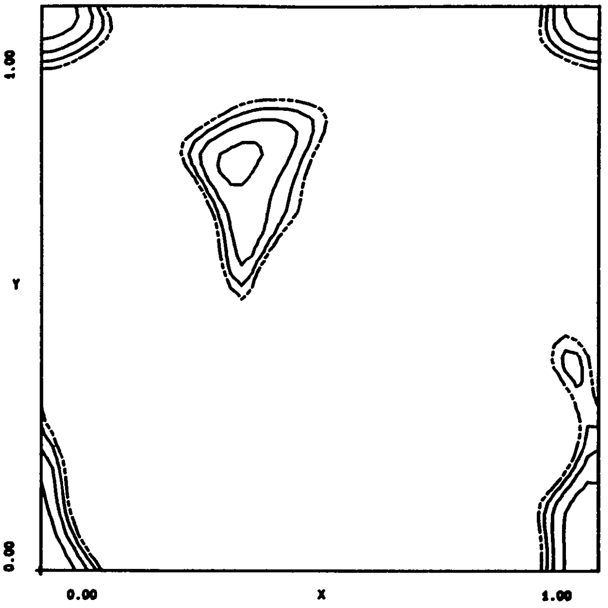


Fig. 3. Energy of interaction, in kJ mol^{-1}, between one molecule at $z = \frac{1}{4}$ and a layer of molecules at $z = 0$. Contours are drawn at intervals of 20 kJ mol^{-1}, the zero contour is shown by stippled lines, positive contours are omitted.

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Structure of *N*-(2-Dimethylaminoethyl)dithiocarbamic Acid

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Abstract. $C_5H_{12}N_2S_2$, M_r = 164·3, monoclinic, $P2_1/n$, a = 11·057 (1), b = 11·866 (2), c = 6·5885 (7) Å, β = 96·19 (1)°, V = 859·39 Å^3, Z = 4, D_m = 1·262, D_x = 1·269 Mg m^{-3}, m.p. = 431 K, Mo $K\alpha$ X-rays, λ = 0·71069 Å, μ = 0·526 mm^{-1}, $F(000)$ = 352, T = 295 K, R = 0·046 for 1207 unique observed reflections. The molecule exists as a zwitterion $(CH_3)_2$-$N^+H(CH_2)_2NHCS_2^-$. In the planar NCS_2 dithiocarbamate group the lengths of the partially double C—S bonds, 1·730 (4) and 1·682 (4) Å, differ significantly. The C—N bond, 1·340 (6) Å, also has strong double-bond character. A bifurcated N—H···S_2 and a normal N—H···S intermolecular hydrogen bond result in infinite interconnected zigzag chains of molecules parallel to the *b* axis.

Introduction. As part of a continuing study of the structure of dithiocarbamates, we recently reported the structures of three zwitterionic alkylaminoalkyldithiocarbamates (see below). These substances were chosen to help delineate the effects of the substituents *R* on the structure of dithiocarbamate ligands, $-S_2CNR_2$ [$R = R'_2NH(CH_2)_n$], without the attendant effects of a complexing metal ion. The availability of one additional compound in this series, *N*-(2-dimethylaminoethyl)dithiocarbamic acid (DMAE hereafter) prompted us to extend this study to provide additional insights into the factors affecting the molecular and crystal structure of these substances.

Experimental. Colourless transparent crystals prepared by one of us (PK), 0·45 × 0·34 × 0·31 mm, D_m by flotation in CCl_4/CH_3COCH_3; computer-controlled Philips PW1100 four-circle single-crystal diffractometer, graphite-monochromated Mo $K\alpha$, ω-scan mode; cell parameters and their e.s.d.'s by least-squares analysis of measured θ angles of the 42 strongest reflections; space group from systematic absences; three standard reflections per 90 min (120, 202, $\bar{1}31$) without significant intensity variation; no absorption correction, 2960 measured reflections, θ = 3–25°, max. *hkl* 13, 14, ±7, 1458 unique reflections, $R_{\rm int}$ = 0·038,

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