

- LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
 NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., p. 246. Ithaca: Cornell Univ. Press.
 PROUT, K., CAMERON, T. S., FORDER, R. A., CRITCHLEY, S. R., DENTON, B. & REES, G. V. (1974). *Acta Cryst.* **B30**, 2290–2304.
 PROUT, K. & REES, G. V. (1974). *Acta Cryst.* **B30**, 2251–2252.
 ROLLETT, J. S. (1969). *Crystallographic Computing*, edited by F. R. AHMED, pp. 169–172. Copenhagen: Munksgaard.
 ROLLETT, J. S., MCKINLAY, T. G. & NAIGH, N. P. H. (1976). *Crystallographic Computing Techniques*, edited by F. R. AHMED, pp. 413–419. Copenhagen: Munksgaard.
 SCHULTZ, A. J., STEARLEY, K. L., WILLIAMS, J. M., MINK, R. & STUCKY, G. D. (1977). *Inorg. Chem.* **16** (12), 3303–3306.
 WASER, J. (1963). *Acta Cryst.* **16**, 1091–1094.

Acta Cryst. (1982). **B38**, 462–465

Pentameric Methylzinc *tert*-Butylsulphide*

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Abstract

$C_{25}H_{60}S_5Zn_5$, $M_r = 847.93$, is monoclinic, $P2_1/c$, with $a = 9.59$ (2), $b = 39.04$ (6), $c = 12.13$ (2) Å, $\beta = 117.13$ (17)°, $U = 4041.7$ Å³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $Z = 4$ [20 units of $\text{CH}_3\text{ZnSC}(\text{CH}_3)_3$], $D_m = 1.39$ (2), $D_c = 1.38$ Mg m⁻³, $\mu(\text{Cu } K\alpha) = 5.804$ mm⁻¹, $F(000) = 1760$. The structure was determined from visually estimated intensity data by application of Patterson superposition methods, and non-hydrogen atoms were refined by least-squares methods with anisotropic temperature factors. $R = 0.14$ for 2230 reflections classified as observed. The compound exists in the crystal, as in solution, as discrete pentamers. The Zn atoms lie near the corners of a square-based pyramid with the apical atom, Zn(5), closer to Zn(3) and Zn(4). S(2), S(3) and S(4) lie above the centres of three of the triangular faces while S(1), which is only three-coordinate, is above the face defined by Zn(1), Zn(2) and Zn(5) but much closer to Zn(1) and Zn(2). S(5) lies below the basal plane, is closer to Zn(3) and Zn(4) and is five-coordinate, whereas all the Zn atoms and S(2), S(3), S(4) are four-coordinate. There appears to be no appreciable Zn–Zn or S–S bonding in the cluster.

* μ -*tert*-Butylthio-tris(μ_3 -*tert*-butylthio)- μ_4 -*tert*-butylthio-pentakis(methylzinc).

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Introduction

Reaction between dialkylzinc and alkanethiols yields compounds of the empirical formula $RZnSR'$, which show differing degrees of association. Methylzinc methylsulphide is insoluble in benzene and is believed to be polymeric in the solid state. Methylzinc isopropylsulphide is hexameric in benzene solution and has subsequently been found to be octameric in the solid state (Adamson & Shearer, 1969), whereas methylzinc *tert*-butylsulphide and ethylzinc *tert*-butylsulphide are pentameric in benzene solution (Coates & Ridley, 1965). The structure of the simpler pentameric compound was therefore investigated in the solid state.

Experimental

Methylzinc *tert*-butylsulphide was recrystallized from hexane as transparent needles, elongated along **a** and with well developed (010), (001) and (011) faces. The crystal used for data collection had cross-section 0.35 × 0.16 mm and was sealed inside a thin-walled capillary tube in an atmosphere of dry nitrogen. The unit-cell dimensions were determined from precession photographs of the $h0l$ and $hk0$ reciprocal-lattice nets taken with Zr-filtered Mo radiation.

The layers $0kl$ – $6kl$ were recorded photographically using the equi-inclination Weissenberg technique and Ni-filtered Cu radiation. At the end of the data collection, the crystal had acquired a milky appearance, presumably due to the formation of elemental sulphur, but without any noticeable effect on the quality of the data.

Table 1. *Fractional positional parameters (Zn, S $\times 10^5$, C $\times 10^4$) and B_{eq} values with e.s.d.'s in parentheses*

Equivalent isotropic temperature factors were calculated by the method of Willis & Pryor (1975).

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (\AA^2)
Zn(1)	10706 (53)	30608 (10)	27402 (42)	7.32 (27)
Zn(2)	23773 (49)	34877 (10)	54613 (35)	6.49 (17)
Zn(3)	-701 (47)	41712 (10)	42125 (38)	6.51 (20)
Zn(4)	-15716 (45)	37329 (12)	14044 (36)	7.71 (19)
Zn(5)	20423 (52)	41563 (10)	24886 (41)	6.99 (18)
S(1)	33983 (86)	31400 (18)	44525 (75)	6.15 (32)
S(2)	25904 (79)	40457 (18)	46164 (61)	5.43 (28)
S(3)	-6528 (85)	43117 (19)	20711 (71)	6.28 (32)
S(4)	9898 (83)	35942 (18)	16381 (66)	5.59 (29)
S(5)	-5932 (78)	35500 (17)	36211 (61)	5.05 (26)
C(1)	-228 (46)	2675 (8)	1932 (32)	8.5 (17)
C(2)	2440 (35)	3441 (9)	7183 (26)	7.8 (15)
C(3)	-803 (38)	4433 (8)	5250 (32)	8.0 (16)
C(4)	-3654 (34)	3588 (11)	-32 (31)	10.6 (18)
C(5)	3340 (49)	4454 (10)	1943 (40)	10.4 (22)
C(6)	4098 (41)	2722 (7)	5384 (36)	8.7 (18)
C(7)	5466 (43)	2842 (10)	6631 (36)	4.6 (19)
C(8)	2702 (48)	2614 (8)	5732 (38)	9.4 (21)
C(9)	4327 (38)	2465 (7)	4621 (37)	8.6 (17)
C(10)	3950 (34)	4382 (9)	5724 (34)	8.9 (17)
C(11)	3218 (54)	4714 (8)	5147 (43)	11.3 (19)
C(12)	3935 (40)	4310 (10)	6985 (32)	9.1 (17)
C(13)	5632 (47)	4293 (15)	5927 (42)	13.7 (29)
C(14)	-1713 (50)	4689 (8)	1309 (35)	9.4 (18)
C(15)	-611 (80)	4981 (11)	1792 (55)	17.4 (31)
C(16)	-2030 (88)	4655 (9)	-115 (46)	18.5 (31)
C(17)	-3482 (84)	4684 (20)	1451 (71)	21.4 (50)
C(18)	887 (39)	3555 (8)	52 (22)	7.0 (13)
C(19)	274 (50)	3866 (11)	-643 (36)	10.3 (21)
C(20)	2622 (43)	3463 (10)	209 (37)	9.0 (18)
C(21)	-193 (42)	3212 (10)	-616 (30)	9.4 (18)
C(22)	-2223 (34)	3404 (10)	3896 (35)	8.8 (18)
C(23)	-2971 (68)	3065 (15)	3146 (59)	16.5 (36)
C(24)	-3522 (41)	3681 (10)	3443 (33)	9.4 (19)
C(25)	-1695 (42)	3406 (9)	5292 (30)	10.1 (16)

The intensities were estimated visually* by comparison with a calibrated scale and were placed on a common scale using photographs on which were recorded parts of a number of nets. The intensities were corrected for Lorentz and polarization factors, for absorption (Busing & Levy, 1957), and for spot length (Phillips, 1956) in the case of the upper-level reflections.

Structure solution and refinement

The positions of the five Zn atoms in the asymmetric unit were determined from the Patterson function and those of the S and C atoms from successive Fourier syntheses. Based on the Zn and S atoms alone, the residual *R* was 0.36.

* The analysis was completed in 1966 before a diffractometer became available at Durham. This paper is part of an effort by Dr Shearer's co-workers to complete the publication of the work he supervised.

Refinement was carried out by least squares, using the block-diagonal approximation. With anisotropic thermal parameters for the Zn and S atoms and isotropic ones for C, the residual became 0.17 and further refinement, with anisotropic parameters for C also, reduced this to 0.14 for the 2230 observed reflections. Refinement of layer scale factors produced no further improvement. A final $F_o - F_c$ synthesis showed only one pronounced feature near any of the atomic sites namely a peak of height *ca* 0.5 e \AA^{-3} in the vicinity of C(17). In the final least-squares cycle, the greatest parameter shift was 0.6 of its e.s.d. The weighting scheme was $w = 1.0/(300.0 + |F_o| + 0.1 \times |F_o|^2)$, the unobserved reflections being given zero weight. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The real part of the dispersion correction was applied in the case of Zn and S. Final positional parameters and equivalent isotropic temperature factors are given in Table 1.* Computer programs were supplied by Professor D. W. J. Cruickshank and Dr J. Sime of Glasgow University.

Description and discussion of structure

Bond lengths and angles, with their e.s.d.'s, are shown in Tables 2 and 3 respectively and some non-bonding intramolecular distances in Table 4.

As in solution, the molecules are pentamers. The Zn atoms lie near the corners of a square-based pyramid, as shown in Fig. 1. The atoms Zn(1), Zn(2), Zn(3) and

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36379 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

 Table 2. *Bond distances* (\AA)

Zn(1)-S(1)	2.272 (9)	S(1)-C(6)	1.92 (3)
Zn(1)-S(4)	2.457 (8)	S(2)-C(10)	1.91 (4)
Zn(1)-S(5)	2.982 (8)	S(3)-C(14)	1.79 (4)
Zn(2)-S(1)	2.322 (9)	S(4)-C(18)	1.89 (2)
Zn(2)-S(2)	2.455 (8)	S(5)-C(22)	1.83 (3)
Zn(2)-S(5)	2.712 (8)		
Zn(3)-S(2)	2.419 (8)	C(6)-C(7)	1.56 (5)
Zn(3)-S(3)	2.457 (9)	C(6)-C(8)	1.63 (5)
Zn(3)-S(5)	2.514 (8)	C(6)-C(9)	1.45 (5)
Zn(4)-S(3)	2.426 (9)	C(10)-C(11)	1.49 (5)
Zn(4)-S(4)	2.403 (8)	C(10)-C(12)	1.56 (5)
Zn(4)-S(5)	2.514 (8)	C(10)-C(13)	1.56 (5)
Zn(5)-S(2)	2.426 (8)	C(14)-C(15)	1.48 (7)
Zn(5)-S(3)	2.472 (9)	C(14)-C(16)	1.62 (8)
Zn(5)-S(4)	2.439 (8)	C(14)-C(17)	1.78 (8)
		C(18)-C(19)	1.44 (5)
Zn(1)-C(1)	1.92 (4)	C(18)-C(20)	1.63 (5)
Zn(2)-C(2)	2.07 (3)	C(18)-C(21)	1.66 (5)
Zn(3)-C(3)	1.98 (3)	C(22)-C(23)	1.58 (7)
Zn(4)-C(4)	2.04 (3)	C(22)-C(24)	1.55 (5)
Zn(5)-C(5)	2.02 (4)	C(22)-C(25)	1.53 (5)

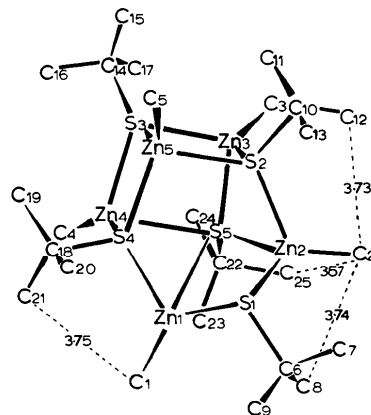
Table 3. Bond angles ($^{\circ}$)

S(1)—Zn(1)—S(4)	99.0 (3)	Zn(1)—S(4)—Zn(5)	128.9 (3)
S(1)—Zn(1)—S(5)	92.8 (3)	Zn(4)—S(4)—Zn(5)	92.9 (3)
S(4)—Zn(1)—S(5)	76.3 (3)	Zn(1)—S(4)—C(18)	117.4 (10)
S(1)—Zn(1)—C(1)	135.5 (11)	Zn(4)—S(4)—C(18)	108.8 (11)
S(4)—Zn(1)—C(1)	122.4 (10)	Zn(5)—S(4)—C(18)	108.9 (10)
S(5)—Zn(1)—C(1)	111.0 (11)	Zn(1)—S(5)—Zn(2)	72.8 (2)
S(1)—Zn(2)—S(2)	99.4 (3)	Zn(1)—S(5)—Zn(3)	130.6 (3)
S(1)—Zn(2)—S(5)	99.0 (3)	Zn(1)—S(5)—Zn(4)	78.4 (2)
S(2)—Zn(2)—S(5)	80.4 (2)	Zn(2)—S(5)—Zn(3)	81.6 (2)
S(1)—Zn(2)—C(2)	130.9 (10)	Zn(2)—S(5)—Zn(4)	130.2 (3)
S(2)—Zn(2)—C(2)	122.0 (10)	Zn(3)—S(5)—Zn(4)	87.7 (3)
S(5)—Zn(2)—C(2)	112.0 (8)	Zn(1)—S(5)—C(22)	120.4 (12)
S(2)—Zn(3)—S(3)	88.3 (3)	Zn(2)—S(5)—C(22)	119.0 (11)
S(2)—Zn(3)—S(5)	85.2 (3)	Zn(3)—S(5)—C(22)	108.9 (12)
S(3)—Zn(3)—S(5)	89.7 (3)	Zn(4)—S(5)—C(22)	110.5 (11)
S(2)—Zn(3)—C(3)	128.5 (10)	S(1)—C(6)—C(7)	103 (2)
S(3)—Zn(3)—C(3)	125.5 (10)	S(1)—C(6)—C(8)	105 (2)
S(5)—Zn(3)—C(3)	126.6 (10)	S(1)—C(6)—C(9)	109 (2)
S(3)—Zn(4)—S(4)	88.5 (3)	C(7)—C(6)—C(8)	105 (3)
S(3)—Zn(4)—S(5)	90.4 (3)	C(7)—C(6)—C(9)	122 (3)
S(4)—Zn(4)—S(5)	87.0 (3)	C(8)—C(6)—C(9)	112 (3)
S(3)—Zn(4)—C(4)	127.4 (12)	S(2)—C(10)—C(11)	104 (2)
S(4)—Zn(4)—C(4)	126.0 (11)	S(2)—C(10)—C(12)	105 (2)
S(5)—Zn(4)—C(4)	125.2 (11)	S(2)—C(10)—C(13)	107 (3)
S(2)—Zn(5)—S(3)	87.7 (3)	C(11)—C(10)—C(12)	114 (3)
S(2)—Zn(5)—S(4)	97.9 (3)	C(11)—C(10)—C(13)	120 (4)
S(3)—Zn(5)—S(4)	86.7 (3)	C(12)—C(10)—C(13)	106 (3)
S(2)—Zn(5)—C(5)	125.7 (12)	S(3)—C(14)—C(15)	107 (3)
S(3)—Zn(5)—C(5)	122.3 (12)	S(3)—C(14)—C(16)	106 (3)
S(4)—Zn(5)—C(5)	125.2 (12)	S(3)—C(14)—C(17)	106 (3)
Zn(1)—S(1)—Zn(2)	95.0 (3)	C(15)—C(14)—C(16)	104 (4)
Zn(1)—S(1)—C(6)	110.8 (11)	C(15)—C(14)—C(17)	121 (4)
Zn(2)—S(1)—C(6)	107.7 (11)	C(16)—C(14)—C(17)	112 (4)
Zn(2)—S(2)—Zn(5)	125.7 (3)	S(4)—C(18)—C(19)	110 (2)
Zn(2)—S(2)—Zn(3)	89.0 (3)	S(4)—C(18)—C(20)	109 (2)
Zn(3)—S(2)—Zn(5)	93.0 (3)	S(4)—C(18)—C(21)	107 (2)
Zn(2)—S(2)—C(10)	118.5 (12)	C(19)—C(18)—C(20)	112 (3)
Zn(3)—S(2)—C(10)	108.8 (10)	C(19)—C(18)—C(21)	113 (3)
Zn(5)—S(2)—C(10)	112.0 (12)	C(20)—C(18)—C(21)	105 (2)
Zn(3)—S(3)—Zn(4)	91.0 (3)	S(5)—C(22)—C(23)	111 (3)
Zn(3)—S(3)—Zn(5)	90.9 (3)	S(5)—C(22)—C(24)	110 (3)
Zn(4)—S(3)—Zn(5)	91.5 (3)	S(5)—C(22)—C(25)	109 (2)
Zn(3)—S(3)—C(14)	122.1 (13)	C(23)—C(22)—C(24)	108 (3)
Zn(4)—S(3)—C(14)	124.1 (13)	C(23)—C(22)—C(25)	118 (3)
Zn(5)—S(3)—C(14)	127.1 (14)	C(24)—C(22)—C(25)	101 (3)
Zn(1)—S(4)—Zn(4)	91.8 (3)		

Table 4. Some non-bonding intramolecular distances (\AA)

Zn(1)···Zn(2)	3.387 (6)	S(1)···S(2)	3.645 (11)
Zn(1)···Zn(3)	4.996 (6)	S(1)···S(3)	5.842 (11)
Zn(1)···Zn(4)	3.491 (6)	S(1)···S(4)	3.598 (11)
Zn(1)···Zn(5)	4.417 (7)	S(1)···S(5)	3.835 (11)
Zn(2)···Zn(3)	3.418 (6)	S(2)···S(3)	3.395 (11)
Zn(2)···Zn(4)	4.741 (6)	S(2)···S(4)	3.668 (11)
Zn(2)···Zn(5)	4.343 (6)	S(2)···S(5)	3.341 (10)
Zn(3)···Zn(4)	3.483 (6)	S(3)···S(4)	3.370 (11)
Zn(3)···Zn(5)	3.513 (6)	S(3)···S(5)	3.505 (11)
Zn(4)···Zn(5)	3.509 (6)	S(4)···S(5)	3.384 (10)
Zn(5)···S(1)	4.504 (9)		

Zn(4) form the base of the pyramid and are nearly coplanar, the distances of the atoms from their mean plane being 0.03 \AA . The apical atom Zn(5) is much closer to Zn(3) and Zn(4) than to Zn(1) and Zn(2). S(2), S(3) and S(4) lie above three of the triangular faces of the pyramid and each forms three bonds to

Fig. 1. Structure of the pentamer (distances are in \AA).

zinc. S(1) is edge-bridging as the distance between S(1) and Zn(5) of 4.503 \AA is so large as to rule out the possibility of bonding between these atoms. S(5) lies below the basal plane but is closer to Zn(3) and Zn(4) than to Zn(1) and Zn(2).

A methyl C atom is attached to each Zn atom and a tertiary C atom of a *tert*-butyl group to each sulphur; all the Zn and S atoms are four-coordinate except S(1), which is three-coordinate, and S(5), which is five-coordinate.

This arrangement results in the atoms Zn(3), Zn(4), Zn(5), S(2), S(3), S(4) and S(5) lying near seven of the corners of a cube. In addition, the molecule possesses an approximate mirror plane which passes near S(1), S(3), S(5) and Zn(5). Of the seven pairs of Zn—S bonds related in this way, the greatest difference in distance within a pair is 0.05 \AA except in the case of Zn(1)—S(5) and Zn(2)—S(5) where the difference is 0.27 \AA .

The nine Zn—S bonds involving S(2), S(3) and S(4), which are all four-coordinate, have lengths between 2.403 and 2.472 \AA similar to those found (2.346–2.489 \AA) for $(\text{MeZnSPR})_8$ (Adamson & Shearer, 1969). The sum of the tetrahedral covalent radii (Pauling, 1960) is 2.35 \AA , this being equal to the Zn—S distance in zinc sulphide (Pauling, 1960), the bridging distance in polymeric $(\mu\text{-SPh})_6\text{Zn}_4\text{CH}_3(\text{OH})\text{SPh}$ (Dance, 1980) and in $(\text{NH}_2)_2\text{CS}_2\text{ZnCl}_2$ (Kunchur & Truter, 1958). In the present case the S—Zn—S angles range from 76 to 99 $^{\circ}$ and the Zn—S—Zn angles at S(2), S(3) and S(4) are roughly 90 $^{\circ}$ except for Zn(5)—S(2)—Zn(2) and Zn(5)—S(4)—Zn(1), which are 126 and 129 $^{\circ}$ respectively. An increase in the lengths of these bonds compared with those in ZnS might be expected in view of the smaller bond angles and hence, perhaps, increased *p* character in the orbitals used by Zn and S.

The sulphur atom S(1) is three-coordinate. The Zn(1)—S(1) and Zn(2)—S(1) lengths, 2.272 and 2.322 \AA , are less than those of the other Zn—S bonds in this compound and also less than the sum of the

tetrahedral covalent radii. The relative shortness of the Zn—S(1) bonds may be due to S(1) bridging only two Zn atoms, the others bridging three or four. Both Zn(1) and Zn(2) interact only weakly with S(5) so that each has three near neighbours with the fourth somewhat further away. The use of tetrahedral radii in these circumstances is not wholly satisfactory and some shortening in bond length might be expected.

Counting only the electrons in the atomic *s* and *p* orbitals, six valence electrons are available to bind S(5) to Zn(1), Zn(2), Zn(3) and Zn(4) if there are two electrons per bond in all other cases and an additional two electrons occupy a non-bonding orbital on S(1). Similar situations arise in respect of the compounds B₅H₉ (Dulmage & Lipscomb, 1952) and B₅H₁₁ (Lavine & Lipscomb, 1954). However, the position of S(5) is not symmetrical. Of the four Zn—S bonds formed by S(5), the bonds to Zn(3) and Zn(4) have lengths of 2.514 Å, which is not much outside the range of the lengths of the Zn—S bonds formed by S(2), S(3) and S(4). However, the Zn(1)—S(5) and Zn(2)—S(5) bond distances of 2.982 and 2.712 Å are much greater and are comparable with the value of 2.815 Å found in zinc diethyldithiocarbamate (Bonamico, Mazzone, Vaciano & Zambonelli, 1965) for one Zn—S interaction.

The Zn—C bond lengths range from 1.92 Å for Zn(1)—C(1) to 2.07 Å for Zn(2)—C(2). The sum of the tetrahedral covalent radii (Pauling, 1960) is 2.08 Å whereas a mean value of 1.94 Å was found in tetrameric methylzinc methoxide (Shearer & Spencer, 1980) and the same value was reported for dimethylzinc (Rundle, Olson, Stucky & Engebretson, 1963) where the Zn atoms are two-coordinate. Some variation in length would be expected in view of the differing environments of the Zn atoms but in the present structure the bond lengths are not accurate enough to show any such effects.

The S—C bonds lie in the range 1.79 Å for S(3)—C(14) to 1.92 Å for S(1)—C(6), and are the same within experimental error. In methanethiol (Kojima & Nishikawa, 1957), the S—C bond length is 1.819 Å. The mean C—C bond length is 1.57 Å and all are the same within experimental error as the value of 1.5445 Å in diamond. The C—C—C angles in the *tert*-butyl groups show no significant deviations from expected values.

No intra- or intermolecular non-bonding distances involving C atoms are shorter than the van der Waals distances.

The thermal parameters of the Zn atoms are greater than those of S suggesting that the Zn atoms carry some positive charge and the S atoms a negative one, since the form factors used were those of neutral atoms. In general, the thermal parameters, particularly those of the C atoms, are large. This may arise from the roughly spherical shape of the molecules whose outer region is composed largely of alkyl groups but may reflect some positional disorder related to the decomposition of the crystal.

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References

- ADAMSON, G. W. & SHEARER, H. M. M. (1969). *Chem. Commun.* pp. 897–898.
- BONAMICO, M., MAZZONE, G., VACIAGO, A. & ZAMBONELLI, L. (1965). *Acta Cryst.* **19**, 898–909.
- BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 180–182.
- COATES, G. E. & RIDLEY, D. R. (1965). *J. Chem. Soc.* pp. 1870–1877.
- DANCE, I. G. (1980). *J. Am. Chem. Soc.* **102**, 3445–3451.
- DULMAGE, W. I. & LIPSCOMB, W. N. (1952). *Acta Cryst.* **5**, 260–264.
- International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- KOJIMA, J. & NISHIKAWA, T. (1957). *Phys. Soc. Jpn*, **12**, 680–686.
- KUNCHUR, N. R. & TRUTER, M. R. (1958). *J. Chem. Soc.* pp. 3478–3484.
- LAVINE, L. R. & LIPSCOMB, W. N. (1954). *J. Chem. Phys.* **22**, 614–620.
- PAULING, L. (1960). *The Nature of the Chemical Bond*. Ithaca: Cornell Univ. Press.
- PHILLIPS, D. C. (1956). *Acta Cryst.* **9**, 819–821.
- RUNDLE, R. E., OLSON, D. H., STUCKY, G. D. & ENGBRETSON, G. R. (1963). *Acta Cryst.* **16**, A71.
- SHEARER, H. M. M. & SPENCER, C. B. (1980). *Acta Cryst.* **B36**, 2046–2050.
- WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*. Cambridge Univ. Press.