oxygen atom in CIN, C1—O11—C8, is 114·8 (1)°, very close to the mean of the values found in the Sn complexes (Barnes, Sampson & Weakley, 1980). A librational calculation on the CIN molecule alone confirms its rigidity: $RG = 0{\cdot}046$ compared with 0·28 for the whole complex.

All three methyl groups are well defined, with no suggestion of disorder. The quinol molecule is identical with those found in quinol itself (Wallwork & Powell, 1980) and in quinol complexes. The angle C23—C21—O27 has the typical value of 123·1 (3)° to minimize the C23···H271 interaction. O27 is 0·03 (2) Å above the ring plane while H271 is 0·24 (2) Å below the plane, with C21—O27—H271 = 109·9 (12)°.

The hydrogen-bond length O11···O27 is 2·724 (2) Å, with O11···H271 1·700 (26) Å and O27—H271···O11 173·9 (10)°. In the Sn complexes the average hydrogen-bond distances are 2·71 Å to hydroxyl and 2·59 Å to water.

C4, C8, O11 and C1 are constrained to be approximately coplanar by the ring system. Thus the limiting possibilities are that the hydrogen bond to H(271) uses only one lone pair of electrons from O11, in which case the torsion angle C10—C8—O11—H271 would be close to 0 (or 120)° or that the hydrogen bond uses the combined lone pairs contributing equally, with the torsion angle about 60°. The experimental value is 32·3 (12)°, implying that both lone pairs contribute, but unequally.

References

BAEYER, A. & VILLIGER, V. (1902). *Chem. Ber.* **35**, 1201–1212.

BARNES, J. C. & NICOLL, G. Y. R. (1985). *Inorg. Chim. Acta*, **110**, 47–50.

BARNES, J. C., SAMPSON, H. A. & WEAKLEY, T. J. R. (1980). *J. Chem. Soc. Dalton Trans.* pp. 949–953.

BARNES, J. C. & WEAKLEY, T. J. R. (1976). *J. Chem. Soc. Dalton Trans.* pp. 1786–1790.

CHAN, T.-L. & MAK, T. C. W. (1983). *J. Chem. Soc. Perkin Trans.* 2, pp. 777–782.

HASSEL, O. & HVOSLEV, J. (1954). *Acta Chem. Scand.* **8**, 1953–1955.

LEE, J. D. & WALLWORK, S. C. (1959). *Acta Cryst.* **12**, 210–216.

MAHMOUD, M. M. & WALLWORK, S. C. (1975). *Acta Cryst.* **B31**, 338–342.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

ROBERTS, P. & SHELDRICK, G. M. (1975). *XANADU*. Program for molecular geometry calculations. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SUMAROVA, T. & NEVSKAYA, Y. (1956). *J. Gen. Chem. USSR*, **27**, 3375–3377.

WALLWORK, S. C. & POWELL, H. M. (1980). *J. Chem. Soc. Perkin Trans.* 2, pp. 641–646.

Acta Cryst. (1988). C**44**, 120–122

Structure of 4-(*N*-Benzyl-*N*-methylamino)-1,7,8,9-tetrachloro-10,10-dimethoxy-3-oxa-4-azatricyclo[5.2.1.$0^{2,6}$]deca-4,8-diene

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Abstract. $C_{18}H_{18}Cl_4N_2O_3$, $M_r = 452{\cdot}16$, monoclinic, $P2_1/n$, $a = 9{\cdot}692\,(5)$, $b = 24{\cdot}204\,(4)$, $c = 8{\cdot}664\,(5)$ Å, $\beta = 94{\cdot}45\,(5)°$, $V = 2026\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}48$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 56{\cdot}168$ cm^{-1}, $F(000) = 928$, room temperature, $R = 0{\cdot}070$ for 3192 symmetry-independent observed reflections [$I > 3\sigma(I)$]. The five-membered ring fused to the norbornene moiety is in an *endo* configuration. The dihedral angle between the least-squares planes through the benzene and five-membered heterocyclic rings is 80·7 (2)°.

Introduction. In order to establish the stereochemistry of the product in the (4+2)-cycloaddition reaction of 2-(*N*-benzyl-*N*-methylamino)oxazole with 1,1-dimethoxytetrachlorocyclopentadiene (Dondoni, Fogagnolo, Mastellari, Pedrini & Ugozzoli, 1986) we have determined the crystal and molecular structure of the title compound.

Experimental. Colourless prismatic crystal, 0·29 × 0·30 × 0·60 mm approximately, Siemens AED single-

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crystal diffractometer, Ni-filtered Cu $K\alpha$ radiation, cell parameters from 29 $(\theta,\chi,\varphi)_{hkl}$ reflections accurately measured with maximum experimental angular error of 0·01°, room temperature, one check reflection measured every 50 collected without significant variation, profile analysis with Lehmann & Larsen (1974) procedure modified by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979), $3 \leq \theta \leq 70°$, Lp correction, absorption correction with Walker & Stuart (1983) procedure using *ABSORB* (Ugozzoli, 1986), maximum and minimum values of the absorption coefficients in the polar angles φ and μ are 1·28 and 0·75 respectively, $-11 < h < 11$, $0 < k < 29$, $0 < l < 10$, 4226 reflections collected, 3878 symmetry-independent ($R_{\rm int} = 0{\cdot}063$), 3192 observed at $3\sigma(I)$ level, direct methods with *SHELX*76 (Sheldrick, 1976), full-matrix least-squares refinement on F, heavy atoms anisotropic, H in calculated positions with geometrical constraint C—H = 1·08 Å, 12 reflections omitted in last cycles, 268 parameters refined, final $R = 0{\cdot}070$, unit weights, $(\Delta/\sigma)_{\rm max} = 0{\cdot}3$, $(\Delta\rho)_{\rm max} = 0{\cdot}47$ e Å^{-3}, scattering factors of *SHELX*76, all calculations performed on a Gould SEL 32/77 computer.

Discussion. The final atomic parameters are given in Table 1;* bond lengths, bond angles and selected torsion angles are reported in Table 2. A perspective view of the molecule is shown in Fig. 1.

The C(sp^2)—Cl and C(sp^3)—Cl bond lengths in the ranges 1·689 (4)–1·701 (5) and 1·752 (5)–1·759 (4) Å, respectively, do not differ significantly from the values determined in the crystal structures of similar compounds containing these bonds (Smith, Kennard & Shields, 1977; Smith & Kennard, 1977; Van Hemelrijk, Lenstra & Geise, 1981; Galešić, Matijašić & Bruvo, 1985).

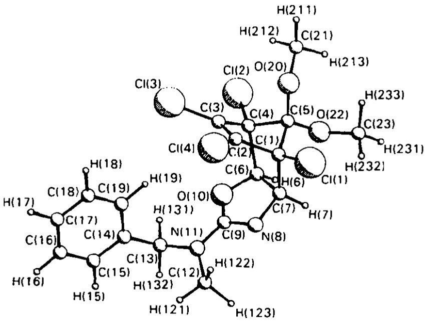

Fig. 1. Perspective view of the molecule with atom numbering.

Table 1. *Final atomic coordinates and equivalent isotropic temperature factors of the non-hydrogen atoms with e.s.d.'s in parentheses*

	x	y	z	$U_{\rm eq}$(Å^2)
Cl(1)	7269 (1)	4394 (0)	−1519 (2)	532 (4)
Cl(2)	4749 (2)	2318 (1)	−1512 (2)	623 (5)
Cl(3)	2377 (1)	3293 (1)	−1478 (2)	583 (4)
Cl(4)	3928 (1)	4552 (1)	−1346 (2)	578 (5)
C(1)	6254 (5)	3834 (2)	−979 (5)	361 (14)
C(2)	4700 (5)	3927 (2)	−1271 (5)	375 (14)
C(3)	4101 (5)	3434 (2)	−1314 (6)	386 (15)
C(4)	5197 (5)	3001 (2)	−1015 (6)	382 (15)
C(5)	6428 (5)	3272 (2)	−1841 (6)	370 (16)
C(6)	5770 (5)	3069 (2)	718 (6)	390 (17)
C(7)	6539 (5)	3630 (2)	717 (6)	407 (15)
N(8)	5894 (4)	3963 (1)	1826 (4)	332 (12)
C(9)	4879 (6)	3651 (2)	2333 (6)	474 (18)
O(10)	4714 (4)	3145 (2)	1792 (4)	579 (14)
N(11)	4103 (5)	3876 (2)	3378 (5)	532 (16)
C(12)	4497 (7)	4396 (2)	4128 (8)	656 (23)
C(13)	2905 (6)	3586 (2)	3902 (7)	602 (22)
C(14)	1597 (6)	3917 (2)	3576 (7)	550 (19)
C(15)	699 (7)	4004 (3)	4720 (9)	759 (27)
C(16)	−509 (9)	4294 (4)	4420 (13)	1076 (41)
C(17)	−841 (10)	4497 (4)	2950 (15)	1132 (43)
C(18)	46 (11)	4428 (4)	1819 (13)	1141 (46)
C(19)	1261 (9)	4127 (3)	2115 (9)	878 (32)
O(20)	6223 (4)	3367 (1)	−3425 (4)	494 (12)
O(21)	6318 (8)	2897 (3)	−4432 (8)	714 (24)
O(22)	7602 (3)	2967 (1)	−1414 (4)	462 (12)
C(23)	8888 (5)	3182 (3)	−1929 (9)	635 (24)

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

Table 2. *Bond distances* (Å), *bond angles* (°), *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

Cl(1)—C(1)	1·759 (5)	C(7)—N(8)	1·434 (6)
Cl(2)—C(4)	1·754 (5)	N(8)—C(9)	1·341 (7)
Cl(3)—C(3)	1·700 (5)	C(9)—O(10)	1·317 (7)
Cl(4)—C(2)	1·687 (5)	C(9)—N(11)	1·337 (7)
C(1)—C(2)	1·524 (7)	N(11)—C(12)	1·454 (7)
C(1)—C(5)	1·567 (7)	N(11)—C(13)	1·459 (8)
C(1)—C(7)	1·554 (7)	C(13)—C(14)	1·508 (8)
C(2)—C(3)	1·326 (7)	C(14)—C(15)	1·385 (10)
C(3)—C(4)	1·500 (7)	C(14)—C(19)	1·379 (10)
C(4)—C(5)	1·581 (7)	C(15)—C(16)	1·373 (11)
C(4)—C(6)	1·569 (7)	C(16)—C(17)	1·380 (17)
C(5)—O(20)	1·391 (6)	C(17)—C(18)	1·363 (17)
C(5)—O(22)	1·382 (6)	C(16)—C(19)	1·392 (13)
C(6)—C(7)	1·549 (7)	O(20)—C(21)	1·441 (8)
C(6)—O(10)	1·447 (7)	O(22)—C(23)	1·453 (7)

Cl(1)—C(1)—C(7)	115·7 (3)	C(4)—C(6)—C(7)	103·1 (4)
Cl(1)—C(1)—C(5)	117·3 (3)	C(7)—C(6)—O(10)	104·7 (4)
Cl(1)—C(1)—C(2)	114·1 (3)	C(1)—C(7)—C(6)	103·2 (4)
C(5)—C(1)—C(7)	99·0 (4)	C(6)—C(7)—N(8)	104·9 (4)
C(2)—C(1)—C(7)	107·7 (4)	C(1)—C(7)—N(8)	113·6 (4)
C(2)—C(1)—C(5)	101·0 (4)	C(7)—N(8)—C(9)	105·8 (3)
Cl(4)—C(2)—C(1)	124·6 (4)	N(8)—C(9)—N(11)	117·3 (4)
C(1)—C(2)—C(3)	107·2 (4)	N(8)—C(9)—O(10)	118·6 (5)
Cl(4)—C(2)—C(3)	127·9 (4)	O(10)—C(9)—N(11)	124·1 (5)
Cl(3)—C(3)—C(2)	127·4 (4)	C(6)—O(10)—C(9)	105·9 (4)
C(2)—C(3)—C(4)	108·7 (4)	C(9)—N(11)—C(13)	121·2 (4)
Cl(3)—C(3)—C(4)	123·5 (4)	C(9)—N(11)—C(12)	120·9 (5)
Cl(2)—C(4)—C(3)	117·2 (4)	C(12)—N(11)—C(13)	117·7 (5)
C(3)—C(4)—C(6)	106·6 (4)	N(11)—C(13)—C(14)	111·3 (4)
C(3)—C(4)—C(5)	100·4 (4)	C(13)—C(14)—C(19)	120·2 (5)
Cl(2)—C(4)—C(6)	113·4 (3)	C(13)—C(14)—C(15)	120·8 (5)
Cl(2)—C(4)—C(5)	117·4 (3)	C(15)—C(14)—C(19)	118·9 (6)
C(5)—C(4)—C(6)	99·6 (4)	C(14)—C(15)—C(16)	121·1 (7)
C(1)—C(5)—C(4)	91·8 (3)	C(15)—C(16)—C(17)	119·4 (8)
C(4)—C(5)—O(22)	107·0 (4)	C(16)—C(17)—C(18)	120·3 (9)
C(4)—C(5)—O(20)	117·4 (4)	C(17)—C(18)—C(19)	120·2 (9)

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* Lists of anisotropic thermal parameters, H coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44307 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2 (*cont.*)

C(1)—C(5)—O(22)	116·9 (4)	C(14)—C(19)—C(18)	119·9 (7)
C(1)—C(5)—O(20)	108·3 (4)	C(5)—O(20)—C(21)	117·1 (4)
O(20)—C(5)—O(22)	113·8 (4)	C(5)—O(22)—C(23)	115·8 (4)
C(4)—C(6)—O(10)	114·4 (4)		

Cl(1)—C(1)—C(2)—Cl(4)	−25·6 (6)	Cl(2)—C(4)—C(5)—O(22)	−62·7 (5)
Cl(1)—C(1)—C(7)—N(8)	79·8 (5)	C(6)—C(4)—C(5)—C(1)	−58·9 (4)
Cl(1)—C(1)—C(7)—C(6)	−167·2 (4)	C(5)—C(4)—C(6)—C(7)	35·5 (5)
Cl(1)—C(1)—C(5)—O(20)	−54·1 (5)	C(5)—C(4)—C(6)—O(10)	148·5 (4)
Cl(1)—C(1)—C(5)—O(22)	76·1 (5)	C(6)—C(4)—C(5)—O(22)	60·0 (5)
Cl(1)—C(1)—C(5)—C(4)	−173·9 (4)	C(6)—C(4)—C(5)—O(20)	−170·6 (4)
Cl(1)—C(1)—C(2)—C(3)	159·6 (4)	C(4)—C(5)—O(22)—C(23)	−174·1 (5)
C(5)—C(1)—C(2)—Cl(4)	−152·4 (4)	C(1)—C(5)—O(22)—C(23)	−73·0 (6)
C(7)—C(1)—C(2)—Cl(4)	104·3 (5)	C(4)—C(5)—O(20)—C(21)	−77·0 (6)
C(5)—C(1)—C(7)—N(8)	−153·9 (4)	C(1)—C(5)—O(20)—C(21)	−179·1 (5)
C(2)—C(1)—C(7)—N(8)	−49·2 (6)	O(20)—C(5)—O(22)—C(23)	54·6 (6)
C(5)—C(1)—C(7)—C(6)	−40·9 (5)	O(22)—C(5)—O(20)—C(21)	49·0 (7)
C(2)—C(1)—C(7)—C(6)	63·8 (5)	C(4)—C(6)—C(7)—C(1)	3·0 (5)
C(2)—C(1)—C(5)—O(20)	70·4 (5)	C(4)—C(6)—O(10)—C(9)	−113·5 (5)
C(2)—C(1)—C(5)—O(22)	−159·3 (4)	C(4)—C(6)—C(7)—N(8)	122·2 (4)
C(7)—C(1)—C(5)—C(4)	60·9 (4)	O(10)—C(6)—C(7)—C(1)	−116·9 (4)
C(2)—C(1)—C(5)—C(4)	−49·3 (4)	C(7)—C(6)—O(10)—C(9)	−1·4 (6)
C(5)—C(1)—C(2)—C(3)	32·8 (5)	O(10)—C(6)—C(7)—N(8)	2·3 (5)
C(7)—C(1)—C(2)—C(3)	−70·5 (5)	C(6)—C(7)—N(8)—C(9)	−2·2 (5)
C(7)—C(1)—C(5)—O(22)	−49·2 (5)	C(1)—C(7)—N(8)—C(9)	109·8 (5)
C(7)—C(1)—C(5)—O(20)	−179·4 (4)	C(7)—N(8)—C(9)—O(10)	1·5 (7)
Cl(4)—C(2)—C(3)—Cl(3)	0·8 (8)	C(7)—N(8)—C(9)—N(11)	−179·2 (5)
C(1)—O(2)—C(3)—Cl(3)	175·3 (4)	N(8)—C(9)—O(10)—C(6)	0·1 (7)
C(1)—C(2)—C(3)—C(4)	1·6 (6)	N(8)—C(9)—N(11)—C(12)	−10·5 (8)
Cl(4)—C(2)—C(3)—C(4)	−173·0 (4)	N(8)—C(9)—N(11)—C(13)	174·9 (5)
Cl(3)—C(3)—C(4)—Cl(2)	22·5 (6)	N(11)—C(9)—O(10)—C(6)	−179·2 (5)
C(2)—C(3)—C(4)—Cl(2)	−163·4 (4)	O(10)—C(9)—N(11)—C(12)	168·8 (6)
C(2)—C(3)—C(4)—C(5)	−35·0 (6)	O(10)—C(9)—N(11)—C(13)	−5·8 (9)
Cl(3)—C(3)—C(4)—C(5)	150·9 (4)	C(9)—N(11)—C(13)—C(14)	−121·0 (6)
C(2)—C(3)—C(4)—C(6)	68·4 (6)	C(12)—N(11)—C(13)—C(14)	64·3 (7)
Cl(3)—C(3)—C(4)—C(6)	−105·7 (5)	N(11)—C(13)—C(14)—C(15)	−130·8 (6)
Cl(2)—C(4)—C(5)—C(1)	178·4 (4)	N(11)—C(13)—C(14)—C(19)	50·8 (8)
C(3)—C(4)—C(5)—C(1)	50·1 (4)	C(13)—C(14)—C(19)—C(18)	179·4 (7)
C(3)—C(4)—C(6)—C(7)	−68·5 (5)	C(13)—C(14)—C(15)—C(16)	−178·5 (7)
Cl(2)—C(4)—C(6)—C(7)	161·1 (4)	C(15)—C(14)—C(19)—C(18)	1·0 (11)
C(3)—C(4)—C(6)—O(10)	44·5 (6)	C(19)—C(14)—C(15)—C(16)	−0·0 (11)
Cl(2)—C(4)—C(6)—O(10)	−85·9 (5)	C(14)—C(15)—C(16)—C(17)	0·7 (14)
C(3)—C(4)—C(5)—O(20)	−61·7 (6)	C(15)—C(16)—C(17)—C(18)	−2·4 (16)
Cl(2)—C(4)—C(5)—O(20)	66·6 (6)	C(16)—C(17)—C(18)—C(19)	3·4 (16)
C(3)—C(4)—C(5)—O(22)	169·0 (4)	C(17)—C(18)—C(19)—C(14)	−2·7 (14)

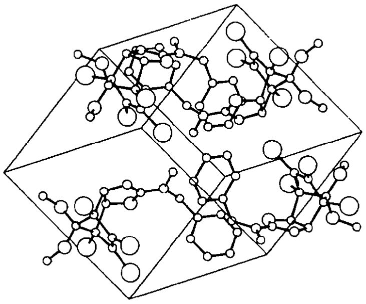

Fig. 2. Minimum-overlap view of the molecular packing without H atoms.

All the C(sp^3)—C(sp^3) bond lengths, ranging from 1·549 (6) to 1·581 (6) Å, are somewhat lengthened. The dihedral angle between the least-squares planes through the C(1), C(2), C(3), C(4) and the C(1), C(4), C(6), C(7) atoms is 118·7 (3)°. The atoms of the five-membered ring [C(6), C(7), C(9), N(8), O(10)] deviate from their mean plane within 0·016 (5) Å. This mean plane shows angles of 127·4 (2) and 119·4 (2)° with the mean planes through C(1), C(2), C(3), C(4) and C(1), C(4), C(6), C(7), respectively, while the angle with the plane through C(1), C(4), C(5) is 1·9 (2)°. The quoted mean-plane angles are comparable with those found in the related but essentially unsubstituted norbornene derivatives with *endo* five-membered rings (Bellabono, Destro, Gramaccioli & Simonetta, 1969; Destro, Filippini, Gramaccioli & Simonetta, 1969). The *endo* configuration of the five-membered hetero ring with regard to the norbornene ring has resulted in the rather close intramolecular distances C(2)—N(8) 2·840 (6), C(3)—C(9) 3·234 (6) and C(3)—O(10) 2·799 (6) Å.

The C—C—C and O—C—O bridge angles of 91·8 (4) and 113·8 (4)°, as well as the O(10)—C(5)—O(22)—C(23) and O(22)—C(5)—O(20)—C(21) torsion angles of 54·6 (6) and 49·0 (7)° respectively, are in accordance with corresponding literature data (Van Hemelrijk, Lenstra & Geise, 1981, 1982; Van Hemelrijk & Lenstra, 1981; Galešić, Matijašić & Bruvo, 1985).

The nitrogen atom from the tertiary amino group, N(11), is 0·038 (5) Å out of the plane of the three adjacent carbon atoms.

The benzene ring, which is planar within ±0·017 (10) Å, makes a dihedral angle of 80·7 (2)° with the plane of the five-membered heterocyclic ring, and of 101·7 (3)° with the plane through the C(1), C(4), C(5) atoms. It is remarkable that the standard deviations of bond lengths in the aromatic ring and also the thermal parameters for these atoms are somewhat greater than for the other part of molecule. This is especially evident for C(16), C(17) and C(18) atoms.

The molecular packing of the compound, which is consistent with usual van der Waals radii, is shown in Fig. 2.

References

Bellabono, I. R., Destro, R., Gramaccioli, C. M. & Simonetta, M. (1969). *J. Chem. Soc. B*, pp. 710–716.

Belletti, D., Ugozzoli, F., Cantoni, A. & Pasquinelli, G. (1979). *Gestione on line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo* 220. Internal Reports 1–3/79, Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.

Destro, R., Filippini, G., Gramaccioli, C. M. & Simonetta, M. (1969). *Acta Cryst.* **B25**, 2465–2472.

Dondoni, A., Fogagnolo, M., Mastellari, A., Pedrini, P. & Ugozzoli, F. (1986). *Tetrahedron Lett.* **27**, 3915–3918.

Galešić, N., Matijašić, I. & Bruvo, M. (1985). *Acta Cryst.* **C41**, 1662–1664.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* **A30**, 580–589.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination, Univ. of Cambridge, England.

Smith, G. & Kennard, C. H. L. (1977). *Aust. J. Chem.* **30**, 1117–1122.

Smith, G., Kennard, C. H. L. & Shields, K. G. (1977). *Aust. J. Chem.* **30**, 911–916.

Ugozzoli, F. (1986). *Comput. Chem.* **11**(2), 109–120.

Van Hemelrijk, D. & Lenstra, A. T. H. (1981). *Cryst. Struct. Commun.* **10**, 603–612.

Van Hemelrijk, D., Lenstra, A. T. H. & Geise, H. J. (1981). *Cryst. Struct. Commun.* **10**, 1269–1276.

Van Hemelrijk, D., Lenstra, A. T. H. & Geise, H. J. (1982). *Acta Cryst.* **B38**, 2760–2763.

Walker, M. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.