angles are listed in Table 2. Fig. 1 shows the *ORTEP* drawing (Johnson, 1965) of the molecule with atomic labeling. Fig. 2 presents the crystal structure.

The tetrazinone ring is almost planar, with maximum displacement out of the least-squares plane of −0·010 (5) Å for N(1) and N(3). This planarity and the endocyclic bond distances indicate an extensive delocalization of electron density which also extends over N(8) and O(7). The N(2)—N(3), N(5)—C(6) and C(4)—O(7) distances, 1·285 (4), 1·317 (4) and 1·223 (4) Å, respectively, indicate the contribution of canonical form (I) shown in the *Introduction*. The N(2)—N(1)—C(6) angle of 112·8 (2)° inclinable to sp^3 hybridization also indicates the localization of an electron (negative charge) on the N(1) position. However, the delocalizability of the electron density in this ring is higher than that found in 4-dimethylamino-1-phenyl-1,2,3,5-tetrazin-6-one (Lindley, Walton, Baydar & Boyd, 1985) because of its poor planarity [maximum displacement out of the least-squares plane of −0·070 (6) Å] and contribution of the canonical form [1·278 (4), 1·312 (6), 1·213 (6) Å, respectively, for corresponding double-bond lengths in the tetrazinone ring].

Though the C(9)—N(2) distance of 1·470 (4) Å indicates a single-bond linkage between sp^2-hybridized C and N atoms, the dihedral angle between the heterocyclic and phenyl-ring planes is small [4·2 (2)°]. This indicates the attractive interactions at H(10)···N(1) and H(14)···N(3) [H(10) and H(14) are connected to C(10) and C(14), respectively], whose short contacts are 2·34 (2) and 2·35 (3) Å, respectively (Taylor & Kennard, 1982).

In the crystal structure of this compound the hydrogen bonds N(8)···N(5) [3·041 (4) Å] and N(8)···O(7) [2·883 (4) Å] are observed.

References

Baydar, A. E., Boyd, G. V., Lindley, P. F. & Walton, A. R. (1985). *J. Chem. Soc. Parkin Trans.* 1, pp. 415–418.

Butler, R. N., Cunningham, D., McArdle, P. & O'Halloran, G. A. (1988). *J. Chem. Soc. Chem. Commun.* pp. 232–234.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Lindley, P. F., Walton, A. R., Baydar, A. E. & Boyd, G. V. (1985). *Acta Cryst.* **C41**, 1669–1672.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Neunhoeffer, H. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 3, edited by A. J. Boulton & A. McKillop, pp. 531–572. Oxford: Pergamon Press.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Thiele, J. & Schleussner, K. (1897). *Justus Liebigs Ann. Chem.* **295**, 129.

Yao, J.-X., Zheng, C.-D., Qian, J.-Z., Han, F.-S., Gu, Y.-X. & Fan, H.-F. (1985). *SAPI85. A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Institute of Physics, Academia Sinica, Beijing, People's Republic of China.

Acta Cryst. (1990). C46, 263–267

Structure of Tetramethyl (2*R*,3*R*,11*R*,12*R*)-*α*-Benzyl-1,4,7,10,13,16-hexaoxacyclooctadecane-2,3,11,12-tetra[(2*S*)-carboxamidoacetate] (an 18-Crown-6 Derivative)

By P. Singh, M. L. Miles and S. Davis

Department of Chemistry, North Carolina State University, Raleigh, North Carolina 27695-8204, *USA*

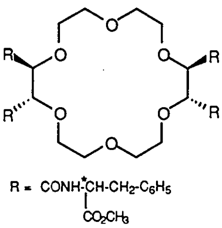
(*Received* 12 *December* 1988; *accepted* 9 *May* 1989)

Abstract. $C_{56}H_{68}N_4O_{18}$, M_r = 1085·18, monoclinic, $P2_1$, a = 9·750 (3), b = 21·389 (6), c = 13·999 (5) Å, β = 93·22 (3)°, V = 2915 (2) Å^3, Z = 2, D_x = 1·24 g cm^{-3}, λ(Mo $K\alpha$) = 0·71096 Å, μ = 0·9 cm^{-1}, F(000) = 1152, T = 295 K, R = 0·050 for 3903 unique reflections. The 18-crown-6 moiety of the molecule has approximate C_2 symmetry, the pseudo twofold axis passing through the midpoints of the C—C bonds containing the side chains. The six O—C—C—O segments have the conformations g^+g^-a, ag^+a, ag^-a, ag^+a, ag^-g^+ and ag^-a. The two *gga* conformations have caused unusual bending in the crown ring resulting in intramolecular inter side-chain hydrogen bonding. One of these hydrogen bonds covers the top face of the crown and the other covers the bottom face. Molecules along the a axis form a polymeric chain *via* a pair of N—H···O hydrogen bonds.

Introduction. Macrocyclic polyethers, also known as 'crown' ethers owing to the appearance of their molecular models, have the remarkable property of

0108-2701/90/020263-05$03.00

complexing with small molecules and ions. The mode of complexation involves attractive interactions between positively charged ions or polar groups on the guest molecule and the ether oxygens surrounding the cavity of the macrocyclic host. The most stable ionic complexes are formed by cations having a diameter similar to that of the cavity (2·67 to 2·86 Å for the 18-crown-6 ether) (Hilgenfeld & Saenger, 1982, Table 9) while the most stable complexes involving organic ligands are formed by molecules containing acidic protons which can hydrogen bond with the ether oxygens, such as primary ammonium ions, R'—NH_3^+ (Pedersen, 1967; Cram & Cram, 1978; Lehn, 1979). Lehn and co-workers (Behr & Lehn, 1980) have shown that one can carry out a chemical reaction between the R'-group of the guest molecule and another functional group, R, by attaching the latter to the crown ether as a side chain. R and R' are designed by the use of —CH_2— spacers to bring their reaction centers in close proximity in space. As a preliminary to such studies we have synthesized, following the procedure of Behr & Lehn (1980), an 18-crown-6 derivative containing L-phenylalanine in the side chain (I). Since our initial binding studies were not successful, we decided to carry out a crystal structure investigation of (I) in order to gain more insight into the conformational requirements for complex formation by the modified 18-crown-16 derivative. The results are compared with those obtained by Lehn and co-workers on ionic complexes of related 18-crown-6 polyethers (Behr, Lehn, Moras & Thiery, 1981; Daly, Schonholzer, Behr & Lehn, 1981).



(1)

Experimental. The title compound, a phenylalanine derivative of 18-crown-6, was synthesized following the procedure of Behr & Lehn (1980) for the tryptophan derivative, and crystallized as thick plates from a methanol solution by slow evaporation at room temperature. A crystal of dimensions 0·85 × 0·60 × 0·15 mm was mounted on a glass fiber and was secured to an X–Y goniometer head. All X-ray measurements were made on a Nicolet $R3m/\mu$ diffractometer equipped with a graphite monochromator. Unit-cell dimensions were derived from a least-squares refinement of the setting angles of 25 reflections with $20 \leq 2\theta \leq 30°$. The intensity data were collected up to a maximum 2θ of 52° by the $\theta/2\theta$ scan technique at a variable scan rate between 4 and 29·3° $\min^{-1}$; range in Miller indices, hkl, $-11 \leq h \leq 12$, $0 \leq k \leq 26$, $0 \leq l \leq 17$. Stationary backgrounds were measured for half of the scan time on each side of a peak; two standards (040 and 211) were measured after every 70 reflections; their intensity variation was 2%. Most reflections at higher 2θ values were weak. Out of a total of 5899 reflections measured, 3903 with $I \geq 1{\cdot}5(I)$ were used in the structure refinement. Intensities were corrected for background and Lorentz and polarization effects. The structure was solved by the random tangent refinement technique using the crystallographic program package *SHELXTL* (Sheldrick, 1985). All 78 non-hydrogen atoms were located from the first E map. The hydrogen atoms attached to the carbon atoms were placed in calculated positions 0·96 Å away from the carbon atom to which they were attached and were assigned isotropic thermal vibration parameters 1·2 times that of the attached carbon. They were refined in the riding mode varying their thermal parameters only. The four N—H hydrogens were located from a difference Fourier map and refined with isotropic thermal vibration parameters. The non-hydrogen atoms were refined with anisotropic thermal vibration parameters.

All calculations, except those for hydrogen bonds given in Table 2, were performed on a Data General Microeclipse computer with the program package *SHELXTL* (Sheldrick, 1985) which has a block-cascade least-squares algorithm for structure refinement (Sparks, 1961). The calculations for hydrogen bonds were made with Professor Baur's program *SADIAN* (Baur, 1968) for an IBM 360. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F) + gF^2]$; g is an adjustable parameter whose final value was 0·0013. Final $R = 0{\cdot}050$, $wR = 0{\cdot}056$. The e.s.d.'s, $\sigma(F_o)$, were derived from counting statistics. Max. $\Delta/\sigma = 0{\cdot}60$. The goodness-of-fit parameter, S, was 1·1 and the isotropic secondary-extinction coefficient was $2{\cdot}0\,(3) \times 10^{-6}$. The final difference Fourier was featureless, the largest positive and negative peaks being $+0{\cdot}25$ and $-0{\cdot}17$ e Å^{-3}, respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Atomic fractional coordinates for non-hydrogen atoms are given in Table 1 and selected bond distances and bond angles, and hydrogen-bonding parameters are given in Table 2.*

---

* Lists of structure factor amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52222 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	*U**
C1	939 (5)	118	−1107 (3)	75 (2)
C2	1654 (5)	−439 (3)	−939 (4)	98 (2)
C3	1494 (6)	−761 (3)	−114 (4)	101 (2)
C4	627 (6)	−538 (3)	556 (4)	99 (2)
C5	−58 (5)	22 (3)	383 (4)	81 (2)
C6	66 (4)	361 (2)	−442 (3)	61 (2)
C7	−683 (5)	954 (2)	−611 (3)	62 (2)
C8	110 (4)	1535 (2)	−230 (3)	59 (2)
C9	−648 (6)	2125 (3)	−526 (4)	81 (2)
O1	−1058 (6)	2513 (2)	−8 (3)	141 (2)
O2	−889 (6)	2174 (3)	−1442 (3)	139 (2)
C10	−1576 (9)	2716 (4)	−1824 (5)	162 (4)
N1	366 (3)	1521 (2)	795 (2)	52 (1)
C11	1568 (4)	1697 (2)	1215 (3)	46 (1)
O3	2567 (3)	1835 (2)	765 (2)	64 (1)
C12	1660 (4)	1732 (2)	2291 (2)	45 (1)
O4	548 (3)	1436 (1)	2731 (2)	52 (1)
C13	755 (5)	799 (2)	2942 (3)	69 (2)
C14	1835 (5)	672 (3)	3733 (4)	82 (2)
O5	3169 (3)	700 (2)	3343 (2)	66 (1)
C15	4247 (5)	840 (2)	4015 (3)	68 (2)
C16	5509 (5)	977 (2)	3489 (3)	63 (2)
O6	5363 (3)	1571 (1)	3022 (2)	50 (1)
C17	6538 (4)	1733 (2)	2518 (3)	50 (1)
C18	6540 (4)	1408 (2)	1552 (3)	51 (1)
O7	7625 (3)	1200 (2)	1267 (2)	78 (1)
N2	5340 (3)	1346 (2)	1070 (2)	54 (1)
C19	5166 (4)	1038 (2)	151 (3)	58 (2)
C20	4591 (6)	1469 (3)	−623 (4)	78 (2)
O8	3779 (5)	1292 (2)	−1255 (3)	113 (2)
O9	5046 (4)	2046 (2)	−543 (2)	91 (2)
C21	4495 (9)	2486 (3)	−1247 (4)	138 (4)
C22	4314 (5)	446 (3)	197 (4)	74 (2)
C23	4872 (5)	−35 (2)	898 (4)	66 (2)
C24	6155 (5)	−295 (2)	820 (4)	84 (2)
C25	6640 (7)	−752 (3)	1455 (5)	105 (3)
C26	5852 (7)	−953 (3)	2168 (4)	107 (3)
C27	4583 (9)	−704 (4)	2247 (4)	110 (3)
C28	4116 (7)	−251 (3)	1629 (4)	96 (3)
C29	6181 (7)	4504 (3)	4195 (5)	113 (3)
C30	6426 (11)	4957 (4)	3556 (8)	151 (4)
C31	5493 (9)	5126 (3)	2883 (5)	144 (4)
C32	4221 (14)	4839 (5)	2820 (7)	164 (5)
C33	3961 (8)	4354 (4)	3478 (5)	120 (3)
C34	4945 (5)	4183 (2)	4148 (4)	71 (2)
C35	4704 (5)	3669 (2)	4848 (3)	64 (2)
C36	5705 (4)	3132 (2)	4799 (3)	62 (2)
C37	5551 (6)	2680 (3)	5596 (4)	86 (2)
O10	5134 (9)	2810 (3)	6347 (3)	184 (3)
O11	5936 (4)	2106 (2)	5413 (3)	97 (2)
C38	5849 (10)	1645 (3)	6154 (4)	146 (4)
N3	5664 (3)	2842 (2)	3871 (2)	54 (1)
C39	6778 (4)	2764 (2)	3363 (3)	52 (1)
O12	7927 (3)	2946 (2)	3643 (2)	77 (1)
C40	6557 (4)	2440 (2)	2400 (3)	49 (1)
O13	5301 (3)	2622 (1)	1915 (2)	51 (1)
C41	5367 (5)	3221 (2)	1476 (3)	68 (2)
C42	4019 (5)	3356 (3)	954 (3)	75 (2)
O14	3036 (3)	3473 (2)	1634 (2)	68 (1)
C43	1660 (5)	3496 (3)	1228 (4)	80 (2)
C44	694 (5)	3363 (2)	2007 (3)	69 (2)
O15	522 (2)	2721 (1)	2199 (2)	53 (1)
C45	1717 (4)	2414 (2)	2601 (3)	46 (1)
C46	1876 (4)	2467 (2)	3677 (3)	44 (1)
O16	2979 (3)	2327 (1)	4099 (2)	59 (1)
N4	805 (3)	2676 (2)	4138 (2)	48 (1)
C47	891 (4)	2720 (2)	5170 (3)	51 (1)
C48	612 (5)	2095 (3)	5622 (3)	70 (2)
O17	−116 (6)	1699 (2)	5288 (3)	128 (2)
O18	1299 (4)	2035 (2)	6455 (3)	107 (2)
C49	1155 (9)	1454 (4)	6969 (5)	139 (3)
C50	−77 (4)	3219 (2)	5524 (3)	62 (2)
C51	360 (4)	3870 (2)	5291 (3)	61 (2)
C52	−93 (6)	4164 (3)	4446 (4)	85 (2)
C53	363 (7)	4763 (3)	4261 (5)	112 (3)
C54	1245 (8)	5066 (4)	4897 (7)	129 (3)
C55	1679 (7)	4776 (3)	5714 (7)	121 (3)
C56	1251 (5)	4186 (3)	5909 (4)	88 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond distances* (Å) *and bond angles* (°) *in the crown ether moiety, and distances* (Å) *and angles* (°) *associated with possible hydrogen bonds*

The bond distances and bond angles are listed in pairs to bring out their approximate equivalence due to the pseudo twofold axis passing through the midpoints of bonds C12—C45 and C17—C40.

C12—C45	1·522 (4)	C12—O4—C13	114·7 (3)
C17—C40	1·520 (6)	C45—O15—C44	114·9 (3)
C13—C14	1·510 (6)	C14—O5—C15	114·4 (4)
C43—C44	1·508 (7)	C43—O14—C42	113·3 (3)
C15—C16	1·498 (6)	C16—O6—C17	112·4 (3)
C41—C42	1·495 (7)	C41—O13—C40	113·3 (3)
C12—O4	1·425 (4)	O4—C12—C45	108·6 (3)
C45—O15	1·425 (4)	O15—C45—C12	108·3 (3)
C13—O4	1·406 (5)	O4—C13—C14	114·4 (4)
C44—O15	1·410 (5)	O15—C44—C43	114·1 (4)
C14—O5	1·440 (6)	O5—C14—C13	108·8 (4)
C43—O14	1·428 (6)	O14—C43—C44	108·4 (4)
C15—O5	1·403 (5)	O5—C15—C16	108·5 (4)
C42—O14	1·411 (6)	O14—C42—C41	108·4 (4)
C16—O6	1·431 (5)	O6—C16—C15	109·6 (4)
C41—O13	1·423 (5)	O13—C41—C42	109·0 (4)
C17—O6	1·422 (5)	O6—C17—C40	108·2 (3)
C40—O13	1·421 (4)	O13—C40—C17	108·0 (3)

A—H···*B*	*A*—H	H···*B*	*A*···*B*	*A*—H···*B*
N1—H···O4	0·83	2·28	2·712	113
N1—H···O7*	0·83	2·16	2·872	145
N2—H···O3	0·85	2·21	2·907	140
N2—H···O6	0·85	2·51	2·774	99
N2—H···O13	0·85	2·55	2·976	112
N3—H···O16	0·86	2·10	2·875	150
N3—H···O13	0·86	2·58	2·781	94
N3—H···O6	0·86	2·58	2·976	109
N4—H···O15	0·83	2·35	2·715	107
N4—H···O12*	0·83	2·14	2·910	154

* Intermolecular hydrogen bonds. Acceptor atoms O7 and O12 belong to the same molecule at symmetry position $-1+x, y, z$. All other hydrogen bonds are intramolecular. Note that all the acidic protons are involved in bifurcated or trifurcated hydrogen bonds owing to a preponderance of acceptor oxygen atoms in the molecule.

Discussion. The conformation of the molecule, looking approximately edgewise at the polyether ring, is shown in Fig. 1, along with the atom-numbering system. The two side-chain *R* groups attached to adjacent chiral carbon atoms, the latter denoted as C*, have a diaxial orientation, the dihedral angles C11—C*12—C*45—C46 and C18—C*17—C*40—C39 being −177·4 and −171·2°, respectively. This diaxial orientation of side groups is analoguous to that observed in two alkali metal complexes of an 18-crown-6 macrocycle containing similarly substituted side groups COO^- and CONHAr (Behr, Lehn, Moras & Thiery, 1981). The amide nitrogen has a nearly eclipsed or *syn* orientation with the nearest ring ether oxygen, the NH—CO—C*H—O torsion angles being 13·6, 38·0, 38·4 and 12·6° for the four moieties containing atoms N1, N2, N3 and N4, respectively. This configuration was also observed by Behr *et al.* (1981) who suggested that it was stabilized by five-membered intramoiety bent hydrogen bonds, *viz* N1—H···O4, N2—H···O6, N3—H···O13 and N4—H···O15 (Table 2 and Fig. 1).

The two side-chain *R* groups protruding out of the same face of the macrocyclic ring are hydrogen bonded to each other, through N2—H···O3 over the top face and N3—H···O16 over the bottom face; Fig. 1. It is obvious that these hydrogen bonds would have to be broken before any binding can take place between a substrate and the ether oxygens, which might explain why the macrocycle is relatively unreactive.

The crystal packing is governed by two intermolecular hydrogen bonds, N1—H···O7 and N4—H···O12, which join molecules in a polymeric chain along the *a* axis (Fig. 1 and Table 2).

The 18-membered macrocyclic ether ring has a pseudo twofold axis passing through the middle of the C*—C* bonds, *viz* C*12—C*45 and C*17—C*40. This can be seen from the torsion angles and the deviations of the ring atoms from their mean plane (Fig. 2). The coordinates of the midpoints of C*12—C*45 and C*17—C*40, (0·6547, 0·2087, 0·2459) and (0·1688, 0·2073, 0·2446), respectively, show that the pseudo twofold axis is nearly parallel to the crystallographic *a* axis. With a shift of origin along the *b* axis, the latter being polar, its approximate location can be described as $(x,0,\frac{1}{4})$. A projection of the molecule down the *a* axis (Fig. 3) shows that the entire molecule conforms to this pseudosymmetry, albeit to a lesser extent than the macrocyclic ether ring alone.

The six O—CH₂—CH₂—O segments, starting from O4 in Fig. 2 (bottom left) in a counterclockwise direction, have the conformation g^+g^-a, ag^+a, ag^-a, ag^+a, ag^-g^+ and ag^-a, which is different from the most commonly observed conformation, *i.e.* $ag^{\pm}a$, observed for all segments in complexed 18-crown-6 macrocycles. Therefore, two segments, *i.e.* those with conformations g^+g^-a and

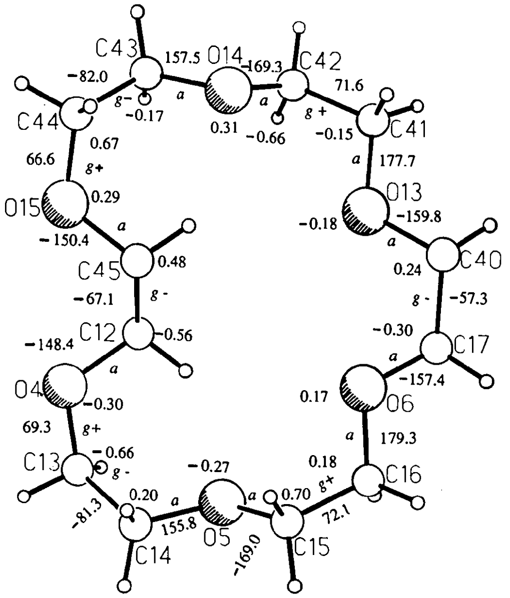


Fig. 2. The 18-crown-6 moiety viewed normal to the mean plane of its carbon and oxygen atoms. Deviations (Å) of atoms from the mean plane are given inside the ring; the letters *g* and *a* refer to the conformation around the C—C or C—O bonds, excluding the hydrogen atoms; the numbers outside the ring are torsion angles (°).

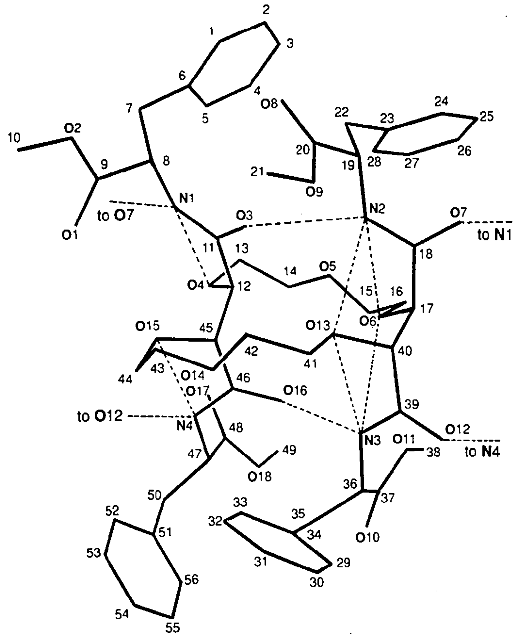


Fig. 1. A view of the molecule looking approximately edgewise at the crown ring showing the numbering system. Hydrogen bonds are shown as dashed lines. Hydrogen atoms are omitted.

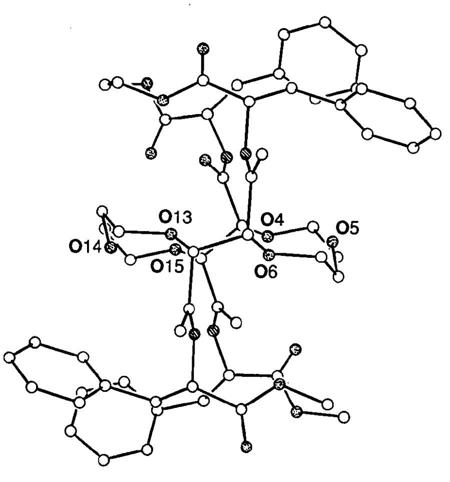


Fig. 3. The molecule projected down the crystallographic *a* axis, the latter being parallel to the pseudo C_2 axis of the molecule.

ag^-g^+, in the above list contain one exception each from the preferred conformation, *i.e.* g^+ instead of *a*. These occur around bonds O4—C13 and O15—C44 and are responsible for a moderate bending (Dale, 1980) in the cyclic structure at O4 and O15. The unusual conformation in this region of the macroring is also responsible for positioning O3 and O16 (Fig. 1) in between the two side arms enabling them to hydrogen bond across the two faces of the ring (see above).

The bond distances and bond angles in the 18-crown-6 ring are quite similar to those observed in the unsubstituted 18-crown-6 (Dunitz & Seiler, 1974; Maverick, Seiler, Schweizer & Dunitz, 1980). There are significant differences, however, owing to the side-chain substitution and a change in symmetry from C_i to pseudo C_2. For example, the mean C—C and C—O bond distances, 1·507 and 1·422 Å, respectively, Table 2, happen to be exactly the same as those observed for the unsubstituted crown (Dunitz & Seiler, 1974), but the individual variations, especially in the C—O bond distances, are much more pronounced in the substituted crown than in the unsubstituted. Also, as pointed out in Table 2, the variations in bond distances as well as bond angles are consistent with the presence of the approximate C_2 symmetry. The six C—O—C bond angles in the crown ring vary from 112·9 to 114·8° with a mean of 113·9° (113·5° for the unsubstituted crown; Dunitz & Seiler, 1974) and ten of the twelve O—C—C bond angles vary from 107·9 to 109·7° with a mean of 108·6°. The two exceptions, at C13 and C44, have identical values of 114·7°, and are much larger than other O—C—C angles cited above. The larger O—C—C bond angles at C13 and C44 together with somewhat larger C—O—C bond angles at O4 and O15, Table 2, seem to be related to the anomalous g^+ conformation around O4—C13 and O15—C44 (see above) and may be a result of the *gauche* effect (Bartell, 1959) proposed by Dunitz & Seiler (1974).

The bond distances and bond angles in the four identical side chains are all normal with the following average values: ester moiety, COOCH$_3$: C=O 1·193 (8), C—OCH$_3$ 1·303 (8), O—CH$_3$ 1·449 (9) Å; peptide moiety, CONHCH: C=O 1·233 (5), CO—N 1·331 (5), NH—CH 1·443 (5) Å.

References

Bartell, L. S. (1959). *J. Am. Chem. Soc.* **81**, 3497–3498.

Baur, W. H. (1968). *SADIAN*. Program to calculate distances and angles in crystal structures. Univ. of Illinois at Chicago, USA.

Behr, J. P. & Lehn, J. M. (1980). *Helv. Chim. Acta*, **63**, 2112–2118.

Behr, J. P., Lehn, J. M., Moras, D. & Thiery, J. C. (1981). *J. Am. Chem. Soc.* **103**, 701–703.

Cram, D. J. & Cram, J. M. (1978). *Acc. Chem. Res.* **11**, 8–14.

Dale, J. (1980). *Isr. J. Chem.* **20**, 3–11.

Daly, J. J., Schonholzer, P., Behr, J. P. & Lehn, J. M. (1981). *Helv. Chim. Acta*, **64**, 1444–1451.

Dunitz, J. D. & Seiler, P. (1974). *Acta Cryst.* B**30**, 2739–2741.

Hilgenfeld, R. & Saenger, W. (1982). *Top. Curr. Chem.* **101**, 1–82.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Lehn, J. M. (1979). *Pure Appl. Chem.* **51**, 979–997.

Maverick, E., Seiler, P., Schweizer, W. B. & Dunitz, J. D. (1980). *Acta Cryst.* B**36**, 615–620.

Pedersen, C. J. (1967). *J. Am. Chem. Soc.* **89**, 7017–7036.

Sheldrick, G. M. (1985). *SHELXTL*. Nicolet Instrument Corporation, Madison, WI, USA.

Sparks, R. A. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, edited by R. A. Pepinsky, J. M. Robertson & J. C. Speakman, pp. 170–187. New York: Pergamon Press.

Acta Cryst. (1990). C**46**, 267–271

Structure of Elsinochrome A*

By H. J. Mebius, H. Krabbendam† and A. J. M. Duisenberg

Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit, Padualaan 8, 3584 *CH Utrecht, The Netherlands*

(*Received* 28 *December* 1988; *accepted* 9 *May* 1989)

Abstract. $\mathrm{C_{30}H_{24}O_{10}}$, $M_r = 554{\cdot}49$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}439\,(4)$, $b = 13{\cdot}001\,(7)$, $c = 14{\cdot}957\,(6)$ Å, $V = 2418\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}49$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54184$ Å, $\mu = 8{\cdot}48$ cm^{-1}, $F(000) = 1136$, $T = 293\,(2)$ K, $R = 0{\cdot}054$ for 2037 observed independent reflections with $I > 3\sigma(I)$. The molecule is X-shaped, with the two acetyl groups pointing outwards, perpendicular to the average

---

* *trans*-1,2-Diacetyl-1,2-dihydro-5,10-dihydroxy-3,7,8,12-tetramethoxybenzo[*ghi*]perylene-4,11-dione.

† To whom correspondence should be addressed.

0108-2701/90/020267-05$03.00