

The authors thank the Natural Sciences and Engineering Research Council of Canada for support.

### References

- AOKI, K., NAGAMO, K. & IITAKA, Y. (1971). *Acta Cryst.* **B27**, 11–23.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
- CANNON, J. R., WHITE, A. H. & WILLIS, A. C. (1976). *J. Chem. Soc. Perkin Trans. 2*, pp. 271–272.
- COTTON, F. A., DAY, V. W., HAZEN, E. E. JR & LARSEN, S. (1973). *J. Am. Chem. Soc.* **95**, 4834–4840.
- COTTON, F. A., HAZEN, E. E. & LEGG, M. J. (1979). *Proc. Natl Acad. Sci. USA*, **76**, 2551–2555.
- CREMER, D. & POPL, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DICKERSON, R. E., DREW, H. R., CONNER, B. N., WING, K. M., FRATINI, A. V. & KOPKA, M. L. (1982). *Science*, **216**, 475–485.
- EZRA, F. S. & COLLIN, R. L. (1973). *Acta Cryst.* **B29**, 1398–1403.
- FURBERG, S. & SOLBAKK, J. (1972). *Acta Chem. Scand.* **26**, 3699–3707.
- GERMAIN, G. & WOOLFSON, M. M. (1968). *Acta Cryst.* **B24**, 91–96.
- GIARDA, L., GARBASSI, F. & CALCATERRA, M. (1973). *Acta Cryst.* **B29**, 1826–1829.
- HAAS, D. J., HARRIS, D. R. & MILLS, H. H. (1965). *Acta Cryst.* **19**, 676–679.
- HAZEL, J. D. & COLLIN, R. L. (1972). *Acta Cryst.* **B28**, 2951–2957.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- KIM, S. H., BERMAN, H. M., SEEMAN, N. C. & NEWTON, M. D. (1973). *Acta Cryst.* **B29**, 703–710.
- KYOGOKU, Y. & IITAKA, Y. (1966). *Acta Cryst.* **21**, 49–56.
- LEWIN, S. (1967). *J. Theor. Biol.* **17**, 181–212.
- LEWIN, S. (1969). *J. Theor. Biol.* **23**, 279–284.
- NEWTON, M. D. (1973). *J. Am. Chem. Soc.* **95**, 256–258.
- PINKERTON, A. A. & SCHWARZENBACH, D. (1978). *J. Chem. Soc. Dalton Trans.* pp. 989–996.
- SAENGER, W. & WAGNER, K. G. (1972). *Acta Cryst.* **B28**, 2237–2244.
- SPRINGS, B. & HAAKE, P. (1977). *Tetrahedron Lett.* **37**, 3223–3226.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- STOUT, G. H. & JENSEN, L. H. (1968). In *X-ray Structure Determination*. London: Macmillan.
- SUCK, D., MANOR, P. C. & SAENGER, W. (1976). *Acta Cryst.* **B32**, 1727–1737.
- SUSSMAN, J. L., SEEMAN, N. C., KIM, S. H. & BERMAN, H. M. (1972). *J. Mol. Biol.* **66**, 403–421.
- SUWALSKY, N. (1968). Thesis, The Weizmann Institute.

*Acta Cryst.* (1982). **B38**, 2955–2958

## Structure of 3-Benzyl-1,4,7,10,13-pentaoxacyclopentadecane-2,5,9-trione

BY T. I. MALINOWSKY, YU. G. GANIN AND YU. A. SIMONOV

*Institute of Applied Physics of the Academy of Sciences MSSR, Akademicheskaya 5, Kishinev 277028, USSR*

AND YU. A. POPKOV, N. G. LUKYANENKO AND A. V. BOGATSKY

*Physico-Chemical Institute of the Academy of Sciences of Ukr.SSR, Chernomorskaya doroga 86, 270080 USSR*

(Received 14 January 1982; accepted 7 June 1982)

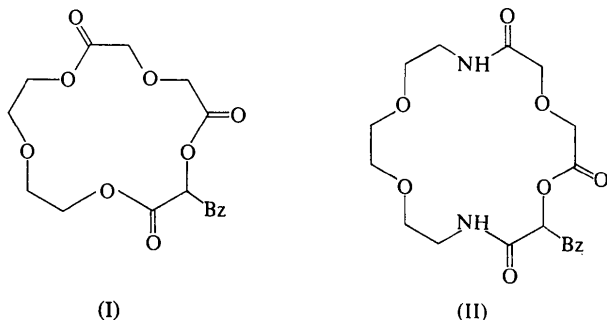
**Abstract.**  $C_{17}H_{20}O_8$ , monoclinic,  $P2_1/b$ ,  $Z = 4$ ,  $a = 10.798$  (11),  $b = 9.057$  (10),  $c = 17.585$  (31) Å,  $\gamma = 93.02$  (2)°,  $V = 1717.4$  Å<sup>3</sup>,  $D_x = 1.36$  Mg m<sup>-3</sup>,  $\mu = 0.106$  mm<sup>-1</sup>. The structure was solved by direct methods and refined by least-squares procedures to a final  $R$  of 0.048 for 2254 independent reflections. The interatomic distances and bond angles are in good agreement with previously published values. The 15-membered ring forms a distorted square. The three ester groups are in the *trans* form: two groups are almost perpendicular to the mean plane of the macrocycle and the third is displaced from it. The overall conformation is stabilized by both intra- and

intermolecular  $C=O \cdots C=O$  and  $C-H \cdots O$  (ester or ether) interactions.

**Introduction.** Publications on structures of macrocyclic molecules are devoted mainly to the cyclic polyethers (crown ethers) and their complexes (Dalley, 1978; Ovchinnikov, Ivanov & Shkrob, 1974). However, little information is presently available about crown-ether derivatives containing ester and amide groups.

We have previously reported (Ganin, Lukyanenko, Dvorkin, Popkov, Simonov & Bogatsky, 1981; Simonov *et al.*, 1981) the results of X-ray structure

determinations of 15- and 18-membered crown ethers containing ester and amide groups. This paper is an extension of our studies on this new synthetic ligand and on the conformational changes in a polyether cycle in the presence of carbonyl groups, other substituents and hetero-atoms. Further, we describe the crystal and molecular structures of the title compound (I).



**Experimental.** The synthesis of (I) was performed according to the method described by Bogatsky, Lukyanenko, Popkov, Zakharov & Varava (1981). The crystals obtained were colourless, rhombic bipyramids  $0.7 \times 0.5 \times 0.7$  mm.

Intensity measurements were made on a DAR-UMB three-circle automatic diffractometer, using graphite-monochromated Mo  $K\alpha$  radiation and the  $\omega$ - $\theta/2\theta$  scan mode. 3380 symmetry-independent reflections, 2254 with  $I(hkl) \geq 3\sigma[I(hkl)]$ , were processed to obtain  $F$  uncorrected for absorption.

The structure was solved by direct methods using the program *YANX*, adapted for the EC series of computers.  $E$  maps and chemical assumptions allowed the construction of a model with identification of 25 non-H atoms. The atomic parameters thus obtained were refined by a full-matrix least-squares method. The function minimized was  $\sum (|F_o| - |F_c|)^2$ . Unit weights were used for all reflections. Several cycles of refinement with isotropic and then anisotropic thermal parameters for non-H atoms led to  $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.079$ . At this stage of the refinement all H atoms were located from an  $F$  synthesis. Additional cycles were then computed including H atoms with isotropic thermal parameters. The final  $R$  was 0.048.\* None of the positional parameters of the non-H atoms shifted by more than  $0.4\sigma$  in the last cycle. A final difference map revealed no peaks greater than  $0.11 \text{ e } \text{\AA}^{-3}$ . Positional parameters are given in Table 1. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

\* Lists of structure factors, anisotropic thermal parameters and distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36973 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional parameters and isotropic thermal factors, with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}/U_{\text{iso}} (\text{\AA}^2)^\dagger$
O(1)	0.1044 (2)	0.1681 (3)	0.8703 (1)	0.036 (1)
C(2)	0.1761 (3)	0.3079 (4)	0.8651 (2)	0.036 (1)
C(3)	0.3105 (3)	0.2816 (4)	0.8465 (2)	0.037 (1)
O(4)	0.3225 (2)	0.1540 (3)	0.8101 (1)	0.041 (1)
C(5)	0.4456 (3)	0.1258 (4)	0.7820 (2)	0.045 (2)
C(6)	0.5254 (2)	0.0620 (4)	0.8418 (2)	0.046 (2)
O(7)	0.4783 (2)	-0.0835 (3)	0.8596 (2)	0.048 (1)
C(8)	0.5558 (2)	-0.1531 (3)	0.9134 (2)	0.049 (2)
C(9)	0.4898 (4)	-0.2902 (4)	0.9434 (2)	0.049 (2)
O(10)	0.3874 (2)	-0.2467 (3)	0.9907 (2)	0.049 (1)
C(11)	0.2735 (4)	-0.2607 (4)	0.9626 (3)	0.046 (2)
C(12)	0.1855 (4)	-0.1933 (4)	1.0172 (2)	0.044 (2)
O(13)	0.0705 (2)	-0.1606 (2)	0.9845 (2)	0.042 (1)
C(14)	0.0734 (3)	-0.0693 (4)	0.9189 (2)	0.039 (2)
C(15)	0.1346 (3)	0.0822 (4)	0.9300 (2)	0.034 (1)
O(16)	0.3938 (2)	0.3696 (3)	0.8610 (2)	0.051 (1)
O(17)	0.2468 (3)	-0.3166 (4)	0.9022 (2)	0.073 (2)
O(18)	0.2038 (2)	0.1228 (3)	0.9794 (2)	0.046 (1)
C(19)	0.1181 (3)	0.3932 (4)	0.8008 (2)	0.042 (2)
C(20)	-0.0150 (4)	0.4250 (4)	0.8164 (2)	0.038 (1)
C(21)	-0.0452 (4)	0.5300 (4)	0.8690 (2)	0.046 (2)
C(22)	-0.1683 (4)	0.5583 (4)	0.8829 (3)	0.057 (2)
C(23)	-0.2613 (4)	0.4830 (5)	0.8441 (3)	0.059 (2)
C(24)	-0.2327 (4)	0.3793 (5)	0.7915 (3)	0.059 (2)
C(25)	-0.1097 (4)	0.3494 (4)	0.7771 (2)	0.049 (2)
H(1)	0.169 (3)	0.376 (4)	0.917 (2)	0.042 (10)
H(2)	0.483 (4)	0.233 (4)	0.761 (2)	0.063 (11)
H(3)	0.430 (4)	0.047 (4)	0.741 (2)	0.070 (12)
H(4)	0.614 (3)	0.058 (4)	0.822 (2)	0.053 (11)
H(5)	0.533 (3)	0.131 (4)	0.894 (2)	0.057 (11)
H(6)	0.642 (3)	-0.192 (4)	0.885 (2)	0.067 (12)
H(7)	0.580 (4)	-0.095 (5)	0.960 (3)	0.101 (15)
H(8)	0.458 (3)	-0.357 (4)	0.903 (2)	0.038 (10)
H(9)	0.577 (3)	-0.341 (4)	0.977 (2)	0.057 (11)
H(10)	0.163 (3)	-0.265 (4)	1.062 (2)	0.056 (11)
H(11)	0.228 (3)	-0.115 (4)	1.039 (20)	0.043 (10)
H(12)	0.120 (3)	-0.109 (3)	0.881 (2)	0.035 (9)
H(13)	-0.008 (3)	-0.064 (3)	0.902 (2)	0.029 (8)
H(14)	0.168 (3)	0.482 (4)	0.802 (2)	0.068 (12)
H(15)	0.124 (3)	0.327 (4)	0.754 (2)	0.053 (11)
H(16)	0.026 (3)	0.575 (4)	0.895 (2)	0.063 (12)
H(17)	-0.199 (4)	0.647 (5)	0.917 (3)	0.102 (15)
H(18)	-0.349 (4)	0.506 (4)	0.852 (2)	0.074 (3)
H(19)	-0.302 (4)	0.340 (4)	0.762 (2)	0.079 (13)
H(20)	-0.081 (4)	0.267 (4)	0.741 (2)	0.081 (13)

$^\dagger U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ ;  $U_{\text{iso}}$  is for H atoms.

**Discussion.** In contrast to the 18-membered macrocycle (II) (Ganin *et al.*, 1981), in which the benzyl group occupies a pseudoaxial position and in which the benzyl ring forms a sandwich structure, macrocycle (I) has the substituent in a pseudoequatorial position with the benzyl-ring plane perpendicular to the mean plane of the macrocycle.

The projection of the molecular structure on the least-square plane through atoms O(1), O(4), O(7) and O(13) (atoms are coplanar within  $0.071 \text{ \AA}$ ) is given in Fig. 1. Interatomic distances and bond angles are given in Table 2. The mean values of C—O [ $1.435 (4) \text{ \AA}$ ] and

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

1-2-3	Distances 1-2	Angles 1-2-3	1-2-3	Distances 1-2	Angles 1-2-3
O(1)-C(2)-C(3)	1.452 (4)	110.3 (3)	C(19)-C(2)-C(3)		110.0 (3)
C(2)-C(3)-O(4)	1.519 (5)	112.1 (3)	C(2)-C(19)-C(20)		112.6 (3)
C(3)-O(4)-C(5)	1.333 (4)	116.6 (3)	C(19)-C(20)-C(21)	1.506 (5)	121.2 (3)
O(4)-C(5)-C(6)	1.453 (3)	112.3 (3)	C(19)-C(20)-C(25)		120.1 (3)
C(5)-C(6)-O(7)	1.495 (6)	109.1 (3)	C(20)-C(21)-C(22)	1.378 (5)	120.6 (4)
C(6)-O(7)-C(8)	1.422 (4)	111.4 (3)	C(21)-C(22)-C(23)	1.388 (6)	120.4 (4)
O(7)-C(8)-C(9)	1.431 (5)	109.6 (3)	C(22)-C(23)-C(24)	1.366 (6)	119.7 (4)
C(8)-C(9)-O(10)	1.495 (6)	108.2 (3)	O(16)-C(3)-C(2)	1.197 (4)	122.7 (3)
C(9)-O(10)-C(11)	1.454 (5)	118.4 (3)	O(16)-C(3)-O(4)		125.1 (3)
O(10)-C(11)-C(12)	1.325 (5)	109.0 (3)	O(17)-C(11)-O(10)	1.205 (5)	124.7 (4)
C(11)-C(12)-O(13)	1.503 (6)	114.0 (3)	O(17)-C(11)-C(12)		126.3 (4)
C(12)-O(13)-C(14)	1.414 (5)	117.4 (3)	O(18)-C(15)-O(1)	1.191 (4)	123.6 (3)
O(13)-C(14)-C(15)	1.419 (4)	114.9 (3)	O(18)-C(15)-C(14)		127.9 (3)
C(14)-C(15)-O(1)	1.504 (5)	108.4 (3)	C(23)-C(24)-C(25)	1.365 (7)	120.6 (4)
C(15)-O(1)-C(2)	1.357 (4)	114.6 (3)	C(24)-C(25)-C(20)	1.393 (6)	120.0 (4)
C(19)-C(2)-O(1)	1.523 (5)	105.9 (3)	C(25)-C(20)-C(21)	1.385 (5)	118.7 (3)

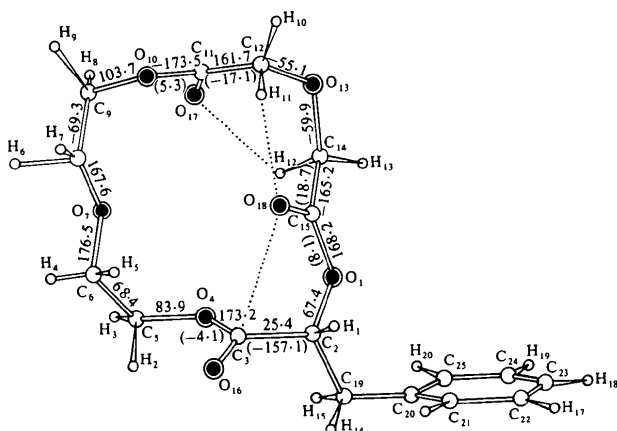


Fig. 1. Projection of the molecular structure on the plane defined by the atoms O(1), O(4), O(7) and O(13) with non-valency interactions indicated. (Torsion angles in deg.)

C-C [1.506 (5) Å] are characteristic of crown ethers (Dalley, 1978). The structure of the three ester groups and benzene corresponds to a  $\pi$ -delocalized system: C=O 1.197 (5), C...O 1.339 (4) and C...C 1.379 (6) Å.

Valence angles at tetrahedral C atoms range from 109.1 (3) to 114.9 (3)°, while those at trigonal C atoms of the benzene ring range from 118.8 (3) to 120.6 (4)°. The average angles are: O...C-C 109.9 (3), O=C...O 124.4 (3), O=C-C 125.6 (3), C-O-C 114.4 (3), C...O-C 116.1 (3)°. The following parameters are characteristic of the fragments containing H atoms: C-H 1.02 (4) Å, H-C-O 109 (2), H-C-C 108 (2), H-C-H 109 (3) and H-C...C 120 (2)°.

Torsion angles describing the conformation of macrocycle (I) are given in Fig. 1. The least-squares deviations of the angles, shown in Fig. 1 and Table 3, are 0.3°.

The angles shown in Fig. 1 in parentheses refer to the carbonyl groups.

The 15-membered macrocycle forms a distorted square ring with three C atoms [C(2), C(5), C(9)] and

Table 3. *Some torsion angles (°)*

C(15)-O(1)-C(2)-C(19)	-173.6	C(23)-C(24)-C(25)-C(20)	0.0
O(1)-C(2)-C(19)-C(20)	61.7	C(24)-C(25)-C(20)-C(21)	0.0
C(2)-C(19)-C(20)-C(21)	73.2	C(24)-C(25)-C(20)-C(19)	180.0
C(19)-C(20)-C(21)-C(22)	180.0	C(25)-C(20)-C(21)-C(22)	0.0
C(20)-C(21)-C(22)-C(23)	0.0	O(4)-C(3)-C(2)-C(19)	-91.1
C(21)-C(22)-C(23)-C(24)	0.0	C(3)-C(2)-C(19)-C(20)	-179.1
C(22)-C(23)-C(24)-C(25)	0.0	O(16)-C(3)-C(2)-C(19)	86.4

Table 4. *Least-squares planes and deviations (Å) of individual atoms from them*

E.s.d.'s of deviations are given in parentheses.

$$\text{Plane (1): } 3.4887x + 4.6838y + 13.7288z = 13.0391$$

O(1)	0.061 (4)	O(7)	0.040 (5)
O(4)	-0.071 (4)	O(13)	-0.029 (4)

$$\text{Plane (2): } 2.9768x - 3.7700y + 15.3523z = 12.8671$$

O(1)	0.171 (4)	C(3)	-0.009 (6)	C(5)	-0.009 (6)
C(2)	-0.222 (6)	O(4)	-0.051 (4)	O(16)	0.130 (5)

$$\text{Plane (3): } -5.2576x + 4.3451y + 13.1142z = 8.4656$$

C(5)	-0.006 (6)	O(7)	-0.070 (7)	C(9)	0.070 (7)
C(6)	0.081 (6)	C(8)	-0.074 (6)		

$$\text{Plane (4): } 1.5034x + 7.8428y - 8.1713z = 9.4045$$

C(9)	0.156 (7)	C(11)	-0.043 (7)	O(13)	0.206 (4)
O(10)	-0.043 (5)	C(12)	-0.144 (7)	O(17)	-0.080 (6)

$$\text{Plane (5): } -8.5892x + 3.8133y + 8.3064z = 6.8757$$

O(1)	0.098 (4)	O(13)	0.084 (4)	C(15)	0.007 (7)
C(2)	-0.028 (6)	C(14)	-0.138 (6)	O(18)	-0.023 (5)

$$\text{Plane (6): } 0.0797x + 6.3504y - 12.5143z = 7.5158$$

C(20)	-0.003 (6)	C(22)	-0.001 (7)	C(24)	0.001 (8)
C(21)	0.003 (7)	C(23)	-0.001 (8)	C(25)	0.001 (7)

Dihedral angles between planes (°): 1-2 56.3; 1-3 48.1; 1-4 81.4; 1-5 71.6; 1-6 100.2; 2-3 70.5; 3-4 91.0; 4-5 89.9; 2-5 87.7.

one O atom [O(13)] at the corners. The dihedral angles between planes of each side of the quadrangle are given in Table 4 (planes 2–5).

The three ester groups exist in the energetically favoured *trans* form (Miyazava, 1959). The torsion angles around the  $C_{sp^2}-C_{sp^3}$  bonds for C(11)–C(12) and C(14)–C(15) are near  $180^\circ$ , and around C(2)–C(3) are near  $0^\circ$ . The atoms in the groups of type O–C–C–O are in the energetically favourable *gauche* conformation (Flory, 1969). On the other hand, the C–O–C–C groups show a *trans* preference (Flory, 1969), but only two [C(6)–O(7) and

O(7)–C(8)] of the seven C–O bonds of the title compound have this conformation.

The shortest intramolecular distances, reflecting 1,4-interactions, are: C(11)=O(17)···H(12)–C(14) (O···H 2.41; O···C 3.01 Å; C–H–O  $125^\circ$ ), C(15)=O(18)···H(11)–C(12) (O···H 2.42; O···C 2.94 Å; C–H–O  $118^\circ$ ) and C(15)=O(18)···C(3)=O(16) (C···O 2.95 Å). The latter dipole–dipole interaction leads to pyramidalization of the C(2), C(3), O(16), O(4) ester group: C(3) deviates from the plane defined by C(2), O(4) and O(16) towards the nucleophile O(18) by 0.018 Å (e.s.d. 0.006 Å). This feature for C=O···C=O interactions was noted by Bürgi, Dunitz & Shefter (1974).

Fig. 2 shows the molecular packing of the title compound. We particularly note the C(15)···O(13) intermolecular distance (2.799 Å) [O(13) at  $(-x, -y, 2-z)$ ]. This interaction is reflected in an observed deviation of C(15) from the plane of the atoms C(14), O(18) and O(1) ( $\Delta = 0.026$  Å). Other intermolecular interactions with indications of their types are given in Table 5.

Table 5. Intermolecular interactions

(I) C–H···O

C(6)–H(5)···O(10) [C···O 3.492, H···O 2.42 Å, C–H–O  $161^\circ$ ; O(10) at  $(1-x, -y, 2-z)$ ]

(II) C–H···O=C

C(5)–H(3)···O(16) [C···O 3.448, H···O 2.43 Å, C–H–O  $176^\circ$ ; O(16) at  $(x, y-0.5, 1.5-z)$ ]

C(8)–H(7)···O(18) [C···O 3.209, H···O 2.59 Å, C–H–O  $130^\circ$ ; O(18) at  $(-x, -y, 2-z)$ ]

(III) O=C···O

O(18)–C(15)···O(13) [C···O 2.799 Å; O(13) at  $(-x, -y, 2-z)$ ]

(IV) C–H···H–C

C(24)–H(19)···H(1)–C(2) [H···H 2.47 Å; H(1) at  $(x-1, y, z)$ ]

C(22)–H(17)···H(6)–C(8) [H···H 2.38 Å; H(6) at  $(x-1, 1+y, z)$ ]

C(19)–H(15)···H(12)–C(14) [H···H 2.45 Å; H(12) at  $(x, 0.5+y, 1.5-z)$ ]

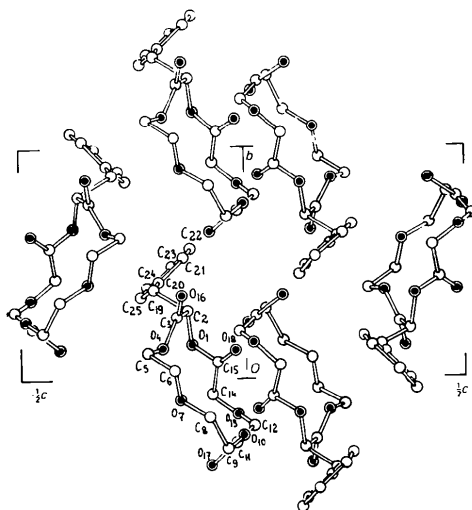


Fig. 2. Projection of the crystal structure along the *a* axis.

### References

- BOGATSKY, A. V., LUKYANENKO, N. G., POPKOV, YU. A., ZAKHAROV, K. S. & VARAVA, V. M. (1981). *Zh. Org. Khim.* **17**, 1062–1068.
- BÜRGI, H. B., DUNITZ, J. D. & SHEFTER, E. (1974). *Acta Cryst.* **B30**, 1517–1527.
- DALLEY, N. K. (1978). *Synthetic Multidentate Macrocyclic Compounds*, edited by R. M. IZATT & J. J. CHRISTENSEN, pp. 207–243. New York: Academic Press.
- FLORY, J. (1969). *Statistical Mechanics of Chain Molecules*. New York: Interscience.
- GANIN, YU. G., LUKYANENKO, N. G., DVORKIN, A. A., POPKOV, YU. A., SIMONOV, YU. A. & BOGATSKY, A. V. (1981). *Dokl. Akad. Nauk SSSR*, **258**, 646–649 (in Russian).
- International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- MIYAZAVA, T. (1959). *J. Chem. Phys.* **30**, 1076–1086.
- OVCHINNIKOV, YU. A., IVANOV, V. T. & SHKROB, A. M. (1974). *Membranoaktivnye kompleksy*. Moscow: Nauka.
- SIMONOV, YU. A., MALINOWSKY, T., GANIN, YU. G., DVORKIN, A., BOGATSKY, A., LUKYANENKO, N. & POPKOV, YU. A. (1981). *Acta Cryst.* **A37**, C213.