

Table 3. Bond lengths (Å) and bond angles (°) with *e.s.d.*'s in parentheses

C1—C2	1.552 (7)	C5—C6	1.548 (8)
C1—C3	1.551 (8)	C5—Br2	1.963 (5)
C1—Br1	1.968 (5)	C6—Br3	1.965 (5)
C1—Br4	1.958 (5)	C7—C8	1.398 (7)
C2—C5	1.564 (8)	C7—C9	1.393 (8)
C2—C9	1.505 (7)	C8—C11	1.376 (9)
C3—C4	1.550 (7)	C9—C10	1.388 (8)
C3—Br5	1.947 (5)	C10—C12	1.390 (8)
C4—C6	1.549 (7)	C11—C12	1.37 (1)
C4—C7	1.498 (7)		
C3—C1—C2	109.4 (4)	Br1—C1—C2	107.7 (4)
Br1—C1—C3	108.3 (4)	Br4—C1—C2	109.3 (4)
Br4—C1—C3	116.2 (4)	Br4—C1—Br1	105.4 (2)
C5—C2—C1	106.7 (4)	C9—C2—C1	108.8 (4)
C9—C2—C5	107.1 (4)	C4—C3—C1	109.4 (4)
Br5—C3—C1	116.3 (3)	Br5—C3—C4	110.4 (4)
C6—C4—C3	108.2 (4)	C7—C4—C3	105.4 (4)
C7—C4—C6	109.4 (4)	C6—C5—C2	108.7 (4)
Br2—C5—C2	107.7 (4)	Br2—C5—C6	116.6 (3)
C5—C6—C4	110.5 (4)	Br3—C6—C4	108.9 (3)
Br3—C6—C5	116.3 (3)	C8—C7—C4	126.3 (5)
C9—C7—C4	113.3 (4)	C9—C7—C8	120.2 (5)
C11—C8—C7	119.0 (6)	C7—C9—C2	114.1 (5)
C10—C9—C2	125.9 (5)	C10—C9—C7	120.0 (5)
C12—C10—C9	119.2 (6)	C12—C11—C8	121.0 (5)
C11—C12—C10	120.6 (6)		

density is high, probably due to the repulsions between Br atoms in the structure.

The X-ray structure analysis of the title compound has revealed that bromine addition has occurred in (3) contrary to our expectation in a *syn* fashion. Studies concerning the mechanism of *syn* addition show that the *syn* adduct can arise either from direct *syn* collapse of the ion pair or from rotation followed by anti-collapse (Heasley, Bower, Dougharty & Easdon, 1980). Because of the rigid skeleton in (2) a bond rotation is out of the question. Another

possible mechanism for *syn* addition is the effect of the neighbouring Br atom at C3 which can attack the preliminary formed cyclic bromonium ion from the underside. The Br ion attacks the cyclic bromonium ion from the *syn* face of the molecule to give the *syn* adduct (3).

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References

- BALCI, M., ÇAKMAK, O. & HARMANDAR, M. (1985). *Tetrahedron Lett.* **26**, 5469–5472.
- ÇAKMAK, O. & BALCI, M. (1989). *J. Org. Chem.* **54**, 181–187.
- ÇAKMAK, O., HÖKELEK, T. & BALCI, M. (1990). In preparation.
- DAVIES, E. K. (1982). *SNOOPI*. Program for drawing crystal and molecular diagrams. Univ. of Oxford, England.
- HEASLEY, G. E., BOWER, T. R., DOUGHARTY, K. W. & EASDON, J. C. (1980). *J. Org. Chem.* **45**, 5150–5155.
- HIXON, S. S., MARIANO, P. S. & ZIMMERMAN, H. E. (1973). *Chem. Rev.* **73**, 531–555.
- HÖKELEK, T., ÇAKMAK, O. & BALCI, M. (1990). In preparation.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.
- WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, D. W. (1985). *CRYSTALS*. Program for crystal structure solution. Univ. of Oxford, England.

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Structure of 5,19-Diamino-9,12,15-trioxatricyclo[14.4.0.0^{3,8}]jcosa-1(16),3,5,7,17,19-hexaene

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Abstract. C₁₇H₂₀N₂O₃, *M_r* = 300.356, monoclinic, *P*2₁/*c*, *a* = 14.443 (2), *b* = 13.883 (2), *c* = 7.455 (3) Å,

β = 94.08 (2)°, *V* = 1491.0 (2) Å³, *Z* = 4, *D_m* = 1.3, *D_x* = 1.338 g cm⁻³, λ(Cu *Kα*) = 1.54180 Å, μ = 7.119 cm⁻¹, *F*(000) = 640, *T* = 293 K, *R* = 0.038 for 2384 observed reflections. The title compound adopts

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a highly non-symmetric, irregular conformation. In particular, the $-\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2-$ sequence of atoms in the macrocyclic ring is more flexible and irregular than the rest of the molecule. The individual phenyl rings are planar but twisted with respect to each other with a torsion angle of 65.6° .

Introduction. The new 12-crown-3 ligand, [12]K(O3-4,4'-diamino-2,2'-diphenylmethano-2₄-coronand-3), containing a benzylic $-\text{CH}_2-$ group has been prepared by reduction of the corresponding dinitro polyether (coronand). It is the smallest member of the 2,3,5,6-bis[5'(5')-aminobenzo]-(3*n* + 3)-crown-*n* (*n* = number of oxygens) type of crown ethers.

The macrocyclic polyethers are known to be complexation agents for the alkali and alkaline-earth cations (Pedersen, 1967; Lockhart, McDonnell & Clegg, 1984), but the title compound does not form a complex with the Li^+ cation, because a CH_2 group (in place of an O atom) and the steric hindrance of the bulky $\text{C}_6\text{H}_4-\text{CH}_2-\text{C}_6\text{H}_4$ group might prevent any wrapping of cations in the hole of the macrocycle. In contrast, the flexible 12-crown-4-polyether forms a complex with the Li^+ cation (Dale & Kristiansen, 1972; Krane, Dale & Daasvatn, 1980).

On the other hand, the title compound is a suitable complexation agent for the transition-metal cations, having two NH_2 groups on the benzene rings. The corresponding dinitro derivative containing a benzylic CH_2 group is very important, because the CH_2 functional group may give the opportunity to introduce substituents to this centre and to produce new chiral ligands.

A structure determination of the title compound was undertaken to permit a comparison of its structure with those of analogous macrocyclic polyethers (coronands).

Experimental. The starting compound 5,19-dinitro-9,12,15-trioxatricyclo[14.4.0.0^{3,8}]jicosa-1(16),3,5,7,17,19-hexaene (I), was synthesized from ethyleneglycol-ditosylate and 2,2'-methylenebis(4-nitrophenol), then purified according to the methods described previously (Kılıç & Gündüz, 1986).

The coronand, 5,19-diamino-9,12,15-trioxatricyclo[14.4.0.0^{3,8}]jicosa-1(16),3,5,7,17,19-hexaene (II), was prepared by reduction of the dinitro derivative (I) with a Pd-C/hydrazine system as follows: 4 mmol of (I) was dissolved in 200 cm³ of ethanol and 0.40 g of Pd-C/hydrazine was added as a catalyst and boiled under reflux for 15 min. Then a solution of 10 cm³ of hydrazine hydrate in 50 cm³ of ethanol was added dropwise to the mixture over a period of 30 min. After refluxing for 2 h, the hot mixture was filtered and solvent was removed *in vacuo*. The resulting solid residue was crystallized from isopro-

Table 1. *Experimental data and structure-refinement parameters*

Method of measuring D_n	Flotation in aqueous KI
Crystal shape and size (mm)	Rod-shaped, $0.2 \times 0.3 \times 0.5$
Diffractometer used and data-collection technique	Four-circle diffractometer (Enraf-Nonius CAD-4), ω - 2θ scan
Number and θ range of reflections used for measuring lattice parameters	25 reflections with $12 \leq 2\theta \leq 40^\circ$
Maximum value of $(\sin\theta/\lambda)$ reached in intensity measurements (\AA^{-1})	0.617
Range of <i>h</i> , <i>k</i> and <i>l</i>	$-1 \leq h \leq 17$, $-1 \leq k \leq 17$, $-9 \leq l \leq 9$
Standard reflections and their intensity variations throughout experiment	031, 060, 020, variation $-1, 0, 0\%$ respectively
Number of reflections measured	3829
Number of unique reflections	2928
Number of unobserved reflections	544
Criterion for recognizing unobserved reflections	$I \leq 3\sigma(I)$
Method used to solve structure	Direct methods
Use of F or F^2 magnitudes in least-squares refinement	F
Parameters refined	278
Values of R , wR	0.038, 0.046, S not calculated
Method used to calculate w	Chebyshev
Final residual electron densities ($e \text{\AA}^{-3}$) for max. and min. peaks	+0.69 and -0.69
Max. ($\Delta\sigma$)	+0.01
Source of atomic scattering factors and f' , f'' values	<i>International Tables for X-ray Crystallography</i> (1974).
Computer programs used	SHELXS86 (Sheldrick, 1986), CRYSTALS (Watkin, Carruthers & Betteridge, 1985), SNOOPI (Davies, 1983)

pyl alcohol. In about three days, yellow rod-shaped crystals were obtained in a benzene/dichloromethane/hexane mixture.

Experimental data, the method used to solve the structure and other related parameters and procedures are given in Table 1. All of the H atoms were located on difference Fourier maps and refined isotropically while the non-H atoms were included with anisotropic thermal parameters.

Discussion. The final coordinates with isotropic or equivalent isotropic thermal parameters are given in Table 2.* The molecular structure of the title compound is shown in Fig. 1. The bond lengths, angles and torsion angles are given in Table 3.

The C6—C7—C8 angle [$116.1(1)^\circ$] is larger than the expected value for sp^3 hybridization ($\sim 109^\circ$). Also, each phenyl ring in the structure is planar and twisted with respect to the other as in the related crown ethers and their complexes (Lockhart, McDonnell & Clegg, 1984). The torsion angle between the phenylene planes is 65.6° . There is a short contact between the benzylic H72 atom and neighbouring O1 atom, O1—H72 (2.41 Å). The

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53035 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Atomic coordinates ($\times 10^4$) and isotropic or equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) with *e.s.d.*'s in parentheses

$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$U/U_{\text{eq}}(\text{\AA}^2)$
C1	6541-1 (9)	2145 (1)	2509 (2)	345 (6)
C2	5842 (1)	2254 (1)	3669 (2)	417 (7)
C3	5279 (1)	3060 (1)	3587 (2)	426 (8)
C4	5405-2 (9)	3775 (1)	2320 (2)	384 (7)
C5	6095-3 (9)	3644 (1)	1116 (2)	346 (7)
C6	6667-1 (9)	2838-1 (9)	1182 (2)	312 (6)
C7	7399 (1)	2663 (1)	-144 (2)	372 (7)
C8	7720-8 (9)	3536-7 (9)	-1126 (2)	316 (6)
C9	7183-5 (9)	3916 (1)	-2582 (2)	344 (6)
C10	7481-7 (9)	4689-8 (9)	-3585 (2)	335 (6)
C11	8363-9 (9)	5067 (1)	-3135 (2)	361 (7)
C12	8901-7 (9)	4707 (1)	-1677 (2)	372 (7)
C13	8581-5 (9)	3961 (1)	-653 (2)	323 (6)
C14	8947 (1)	3805 (1)	2532 (2)	421 (8)
C15	9424 (1)	3058 (1)	3745 (2)	499 (9)
C16	8269 (1)	2045 (1)	4798 (2)	439 (9)
C17	7660 (1)	1207 (1)	4267 (2)	461 (8)
O1	7115-3 (7)	1339-7 (7)	2581 (1)	414 (5)
O2	8976-7 (7)	2144-6 (8)	3581 (1)	481 (6)
O3	9168-9 (6)	3586-8 (8)	731 (1)	401 (5)
N1	4847 (1)	4601 (1)	2201 (2)	567 (9)
N2	6891 (1)	5093 (1)	-4953 (2)	472 (8)
HN11	5104 (16)	5170 (17)	1797 (33)	793 (71)
HN12	4434 (17)	4661 (17)	3025 (32)	762 (66)
HN21	7196 (14)	5520 (15)	-5699 (27)	600 (53)
HN22	6518 (17)	4688 (18)	-5457 (33)	795 (72)
H21	5742 (12)	1755 (14)	4553 (24)	495 (46)
H31	4784 (14)	3121 (15)	4438 (27)	619 (52)
H51	6162 (10)	4150 (12)	228 (21)	381 (39)
H71	7108 (13)	2188 (14)	-1068 (26)	577 (51)
H72	7932 (13)	2336 (13)	451 (25)	507 (47)
H91	6547 (12)	3631 (12)	-2941 (22)	438 (42)
H111	8608 (11)	5599 (13)	-3830 (23)	446 (42)
H121	9523 (13)	4989 (13)	-1332 (24)	480 (44)
H141	8249 (12)	3809 (12)	2639 (21)	419 (40)
H142	9177 (12)	4501 (14)	2866 (23)	514 (47)
H151	10065 (15)	2978 (15)	3380 (28)	650 (55)
H152	9445 (14)	3309 (16)	5053 (31)	733 (62)
H161	7877 (12)	2649 (13)	4797 (23)	450 (43)
H162	8575 (13)	1949 (15)	6070 (28)	632 (54)
H171	8034 (14)	597 (16)	4054 (27)	644 (55)
H172	7218 (14)	1081 (14)	5219 (26)	600 (51)

H51—H91 distance (2.53 Å) can lead to steric hindrance between the two phenyl rings. This steric hindrance can reduce the flexibility of the macrocyclic ring by increasing the C6—C7—C8 angle [116.1(1)°] and twisting the phenyl rings. The replacement of the CH₂ group with the O atom probably reduces the coordination ability of the macrocyclic ring. However, it seems to serve as a functional group to introduce substituents into the above-mentioned ring (von Itter, Vögtle, Weber & Sheldrick, 1983). The structure of the molecule reveals a non-symmetric overall geometry. This is apparent mainly from the conformations at C17—C16—O2—C15 and C16—O2—C15—C14 which are *gauche* (164.8 and 89.7°, respectively), while C7—C6—C1—O1 and O3—C13—C8—C7 (2.0 and 0.3°, respectively) have eclipsed conformations. There seems to be no report on 12-ring macrocyclics in the literature with which to compare the torsion angles obtained here, and it is not worthwhile to compare them with the torsion angles of 15-crown-5, 18-crown-6 and other analogous crown ethers. In the macrocyclic ring the C1—O1—C17 [115.0(1)°] and C13—O3—C14 [116.8(1)°] angles are larger than C15—O2—C16 [112.0(1)°] in the irregular part. The value of C15—O2—C16 [112.0(1)°] is in agreement with the corresponding one in 18-crown-6 (Dobler, Dunitz & Seiler, 1974; Seiler, Dobler & Dunitz, 1974) and other analogous crown ethers (Weber, Newkome, Fronczek & Franken, 1988). In the macrocyclic ring, the C16—O2—C15 and O2—C15—C14 angles have equivalent values [112.0(1)°], and C15—C14—O3 [107.0(1)°] is smaller than O1—C17—C16 [113.9(1)°]. The angles O1—C1—C6 [118.2(1)°] and C1—C6—C7 [118.8(1)°] are smaller than O3—C13—C8 [121.0(1)°] and C13—C8—C7 [121.1(1)°], due to the short contact O1—H72

O1—O3 intramolecular distance in the macrocyclic ring is 4.95 Å. In [18]K(O₅-2,2'-diphenylmethano-24-coronand-5) (von Itter, Vögtle, Weber & Sheldrick, 1983), the corresponding values mentioned above are 66.2°, 2.27 and 4.77 Å, respectively, in good agreement with the results obtained. The short contact between O1 and H72 is not conserved in solution since the H¹ NMR spectrum presents a sharp singlet ($\delta = 4.08$ p.p.m.) for the benzylic CH₂ in CDCl₃ solution. So, both of the benzylic CH₂ hydrogens are not distinguishable, and the conformation of the aminodiphenylmethane system in CDCl₃ solution is not fixed at room temperature as observed in [18]K(O₅-2,2'-diphenylmethano-24-coronand-5). Simple crown ethers, *e.g.* 12-crown-4, 15-crown-5 and 18-crown-6 are highly flexible (Dale & Kristiansen, 1972; Krane, Dale & Daasvatn, 1980; Dobler, Dunitz & Seiler, 1974; Seiler, Dobler & Dunitz, 1974). The title compound can be viewed as an ether derivative which is formed by incorporating phenyl rings containing NH₂ substituents and by replacing the oxygen between the two phenyl groups with a CH₂ group in 12-crown-4. The intramolecular

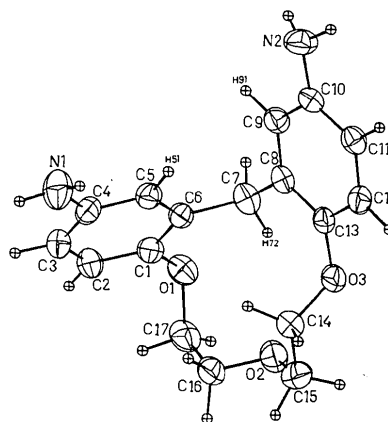


Fig. 1. A SNOOPI (Davies, 1983) drawing of the title molecule with the atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

Table 3. Bond lengths (Å), bond angles (°) and selected torsion angles (°) with e.s.d.'s in parentheses

C1—C2	1.384 (2)	C9—C10	1.394 (2)
C1—C6	1.401 (2)	C10—C11	1.396 (2)
C1—O1	1.391 (2)	C10—N2	1.399 (2)
C2—C3	1.382 (2)	C11—C12	1.384 (2)
C3—C4	1.391 (2)	C12—C13	1.385 (2)
C4—C5	1.400 (2)	C13—O3	1.389 (2)
C4—N1	1.400 (2)	C14—O3	1.435 (2)
C5—C6	1.389 (2)	C14—C15	1.510 (2)
C6—C7	1.518 (2)	C15—O2	1.425 (2)
C7—C8	1.507 (2)	C16—O2	1.421 (2)
C8—C9	1.392 (2)	C16—C17	1.495 (3)
C8—C13	1.398 (2)	C17—O1	1.447 (2)
C2—C1—O1	121.5 (1)	C8—C9—C10	122.2 (1)
C2—C1—C6	120.2 (1)	C9—C10—N2	120.3 (1)
C6—C1—O1	118.2 (1)	C9—C10—C11	118.1 (1)
C1—C2—C3	120.9 (1)	C11—C10—N2	121.6 (1)
C2—C3—C4	120.2 (1)	C10—C11—C12	120.3 (1)
C3—C4—N1	121.6 (1)	C11—C12—C13	120.8 (1)
C3—C4—C5	118.4 (1)	C12—C13—C8	120.1 (1)
C5—C4—N1	119.9 (1)	C12—C13—O3	118.6 (1)
C4—C5—C6	122.1 (1)	C8—C13—O3	121.0 (1)
C5—C6—C1	118.1 (1)	C13—O3—C14	116.8 (1)
C5—C6—C7	123.1 (1)	O3—C14—C15	107.0 (1)
C1—C6—C7	118.8 (1)	C14—C15—O2	112.0 (1)
C6—C7—C8	116.1 (1)	C15—O2—C16	112.0 (1)
C7—C8—C9	120.6 (1)	O2—C16—C17	110.1 (1)
C7—C8—C13	121.1 (1)	C16—C17—O1	113.9 (1)
C9—C8—C13	118.3 (1)	C17—O1—C1	115.0 (1)
C1—O1—C17—C16	69.1	C14—O3—C13—C8	79.7
O1—C17—C16—O2	78.0	O3—C13—C8—C7	0.3
C17—C16—O2—C15	164.8	C13—C8—C7—C6	131.1
C16—O2—C15—C14	89.7	C8—C7—C6—C1	143.4
O2—C15—C14—O3	71.9	C7—C6—C1—O1	2.0
C15—C14—O3—C13	159.2	C6—C1—O1—C17	120.3

(2.41 Å). Hence, O1—C1—C2 [121.5 (1)°] and C5—C6—C7 [123.1 (1)°] are increased while O3—C13—C12 [118.6 (1)°] is decreased. Since C3—C4—C5 [118.4 (1)°] and C9—C10—C11 [118.1 (1)°] are decreased, C4—C5—C6 [122.1 (1)°] and C8—C9—C10 [122.2 (1)°] increase, due to the electron-releasing NH₂ groups. In each of the aromatic rings, the average of the C—C bond lengths is 1.391 (2) Å. Since the N and O atoms bonding to the aromatic rings release electrons to the phenyl groups, the N1—C4 [1.400 (2) Å], N2—C10 [1.399 (2) Å] [average 1.400 (2) Å], O1—C1 [1.391 (2) Å] and O3—C13 [1.389 (2) Å] [average 1.390 (2) Å] are shorter than the conventional C—N (1.47 Å) and C—O (1.43 Å) bond lengths. The average length of the aromatic C—O bond [1.390 (2) Å] is shorter than those of the aliphatic O1—C17 [1.447 (2) Å], O3—C14 [1.435 (2) Å], O2—C15 [1.425 (2) Å] and O2—C16 [1.421 (2) Å] [average 1.432 (2) Å] bonds.

The average value [1.512 (2) Å] of C6—C7 [1.518 (2) Å] and C7—C8 [1.507 (2) Å] is a little longer than the average value [1.502 (2) Å] of the aliphatic C14—C15 [1.510 (2) Å] and C16—C17 [1.495 (3) Å] bonds. In 2,6-pyridino (Newkome, Fronczek & Kohli, 1981) and 2,2'-bipyridino (Weber, Newkome, Fronczek & Franken, 1988) macrocyclic polyethers, the average values of the aliphatic C—C bond lengths are 1.471 (10) and 1.495 (4) Å, while the polyetheral C—O bond lengths are 1.421 (9) and 1.495 (4) Å, respectively, to be compared with the results given above.

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References

- DALE, J. A. & KRISTIANSEN, P. O. (1972). *Acta Chem. Scand. Ser. B*, **26**, 1471–1478.
- DAVIES, K. (1983). *SNOOPI*. Program for drawing crystal and molecular diagrams. Univ. of Oxford, England.
- DOBLER, M., DUNITZ, J. D. & SEILER, P. (1974). *Acta Cryst.* **B30**, 2741–2743.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- ITTER, F. A. VON, VÖGTLE, F., WEBER, G. & SHELDRIK, G. M. (1983). *Z. Naturforsch. Teil B*, **38**, 262–264.
- KILIÇ, Z. & GÜNDÜZ, N. (1986). *Tetrahedron*, **42**, 137–141.
- KRANE, J., DALE, J. & DAASVATN, K. (1980). *Acta Chem. Scand. Ser. B*, **34**, 59–62.
- LOCKHART, J. C., MCDONNELL, M. B. & CLEGG, W. (1984). *J. Chem. Soc. Chem. Commun.* pp. 365–367.
- NEWKOME, G. R., FRONCZEK, F. R. & KOHLI, D. K. (1981). *Acta Cryst.* **B37**, 2114–2117.
- PEDERSEN, C. J. (1967). *J. Am. Chem. Soc.* **89**, 7017–7036.
- SEILER, P., DOBLER, M. & DUNITZ, J. D. (1974). *Acta Cryst.* **B30**, 2744–2745.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.
- WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, D. W. (1985). *CRYSTALS*. Program for crystal structure solution. Univ. of Oxford, England.
- WEBER, E., NEWKOME, G. R., FRONCZEK, F. R. & FRANKEN, S. (1988). *J. Incl. Phenom.* **6**, 1–7.