C′13—C′14—O′3	107.6 (6)	C′3A—C′4A—C′5A	121.4 (5)
O′2—C′14—O′3	125.5 (7)	C′4A—C′5A—C′6A	120.7 (5)
C′14—O′3—C′15	117.7 (6)	C′4A—C′5A—C18A	120.8 (5)
O′3—C′15—C′16	107.6 (8)	C′6A—C′5A—C18A	118.4 (5)
C′1—C′17—O′4	112.4 (5)	C′1A—C′6A—C′5A	120.5 (5)
C′17—O′4—C′18	111.9 (4)	C′1A—C′6A—O′1A	121.8 (5)
O′4—C′18—C″5	109.9 (4)	C′5A—C′6A—O′1A	117.4 (5)
C″2—C″1—C″6	117.7 (5)	C′3A—C′7A—C′8A	120.4 (5)
C″2—C″1—C″17	119.4 (4)	C′3A—C′7A—C′12A	122.1 (5)
C″6—C″1—C″17	122.9 (4)	C′8A—C′7A—C′12A	117.4 (5)
C″1—C″2—C″3	122.0 (5)	C′7A—C′8A—C′9A	120.9 (6)
C″2—C″3—C″4	118.4 (5)	C′8A—C′9A—C′10A	120.7 (6)
C″2—C″3—C″7	120.0 (5)	C′9A—C′10A—C′11A	119.1 (6)
C″4—C″3—C″7	121.6 (5)	C′10A—C′11A—C′12A	120.9 (6)
C″3—C″4—C″5	121.2 (4)	C′7A—C′12A—C′11A	121.0 (5)
C″4—C″5—C″6	121.5 (4)	C′6A—O′1A—C′13A	118.4 (4)
C″4—C″5—C′18	120.0 (4)	O′1A—C′13A—C′14A	109.1 (5)
C″6—C″5—C′18	118.5 (4)	C′13A—C′14A—O′2A	127.3 (7)
C″1—C″6—C″5	121.8 (5)	C′13A—C′14A—O′3A	108.2 (6)
C″1—C″6—O″1	118.3 (4)	O′2A—C′14A—O′3A	124.5 (8)
C″5—C″6—O″1	119.7 (4)	C′14A—O′3A—C′15A	117.1 (7)
C″3—C″7—C″8	122.6 (6)	O′3A—C′15A—C′16A	113.0 (10)
C″3—C″7—C″12	122.0 (6)	C′1A—C′17A—O′4A	112.1 (4)
C″8—C″7—C″12	115.4 (6)	C′17A—O′4A—C′18A	114.0 (4)
C″7—C″8—C″9	122.5 (8)	O′4A—C′18A—C″5A	108.4 (4)
C″8—C″9—C″10	122.6 (9)	C″2A—C″1A—C″6A	117.3 (5)
C″9—C″10—C″11	116.7 (9)	C″2A—C″1A—C″17A	119.7 (5)
C″10—C″11—C″12	121.5 (9)	C″6A—C″1A—C″17A	122.9 (5)
C″7—C″12—C″11	121.0 (8)	C″1A—C″2A—C″3A	122.6 (5)
C″2A—C″3A—C″4A	118.1 (5)	C″8A—C″9A—C″10A	121.8 (10)
C″2A—C″3A—C″7A	120.7 (5)	C″9A—C″10A—C″11A	119.2 (10)
C″4A—C″3A—C″7A	121.2 (5)	C″10A—C″11A—C″12A	119.0 (9)
C″3A—C″4A—C″5A	120.6 (5)	C″7A—C″12A—C″11A	120.3 (7)
C″4A—C″5A—C″6A	121.3 (5)	C″6A—O″1A—C″13A	114.4 (4)
C″4A—C″5A—C′18A	120.2 (5)	O″1A—C″13A—C″14A	110.5 (5)
C″6A—C″5A—C′18A	118.5 (5)	C″13A—C″14A—O″2A	126.3 (5)
C″1A—C″6A—C″5A	122.4 (5)	C″13A—C″14A—O″3A	112.0 (5)
C″1A—C″6A—O″1A	121.2 (5)	O″2A—C″14A—O″3A	121.6 (5)
C″5A—C″6A—O″1A	116.4 (5)	C″14A—O″3A—C″15A	122.8 (5)
C″3A—C″7A—C″8A	120.5 (5)	O″3A—C″15A—C″16A	110.5 (7)
C″3A—C″7A—C″12A	121.3 (5)	C″1A—C″17A—O″4A	113.3 (5)
C″8A—C″7A—C″12A	118.2 (6)	C″17A—O″4A—C″18A	113.4 (4)
C″7A—C″8A—C″9A	121.2 (8)	O″4A—C″18A—C5A	112.0 (4)

Each H atom attached to the methyl groups was treated as part of a rigid unit with its original orientation taken from the strongest H-atom peak on a difference Fourier synthesis. Other H atoms were located from $\Delta\rho$ syntheses and constrained to be 1.08 Å from the corresponding C atoms, their positions being dictated by the geometry of the molecule; they were included as riding atoms in the structure-factor calculations. No H-atom parameters were refined.

Data collection and cell refinement: Enraf–Nonius CAD-4 software. Program(s) used to solve structure: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1110). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Revised Structure for Diethyl 5,11,17,23-Tetra-*tert*-butyl-26,28-bis(2-methoxyethoxy)calix[4]arene-25,27-bis(oxyacetate)

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Abstract

The crystal structure of diethyl {5,11,17,23-tetra-*tert*-butyl-26,28-bis(2-methoxyethoxy)pentacyclo-$[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]$octacosa-1(25),3,5,7(28),9,11,-13(27),15,17,19(26),21,23-dodecaene-25,27-diyldioxy}diacetate, $C_{58}H_{80}O_{10}$, is re-refined in space group *C*2/*c* rather than *Cc*, leading to improved values of bond lengths and angles. Revised coordinates are given.

Comment

The structure of this macrocyclic compound has been described (Guelzim, Khrifi, Baert, Asfari & Vicens, 1993) in space group *Cc* [monoclinic; $a = 25.767\,(2)$, $b = 13.897\,(2)$, $c = 19.867\,(4)$ Å, $\beta = 126.91\,(1)°$, $Z = 4$]. It is better described in *C*2/*c*. Refinement in *C*2/*c* was based on the 3068 'observed'

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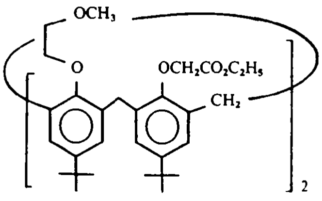
† Contribution No. 8926.

reflections [$I > 3\sigma(I)$] recovered from SUP 71234 and the starting coordinates were taken from Table 1 of Guelzim *et al.* (1993), suitably adjusted for the change in symmetry. A full-matrix refinement proceeded to an R factor of 0.064 for 310 parameters, close to the R factor of 0.062 for 613 parameters reported for the *Cc* refinement. H atoms were placed in calculated positions with C—H = 0.95 Å. Difference maps in the planes of the methyl C atoms of the two independent *tert*-butyl groups, calculated at a late stage in the refinement, indicated disorder between major and minor conformers for both groups; the population parameters were adjusted [the final values being 0.85 (1) and 0.79 (1) for the major conformers] but the atoms of the minor conformers were held fixed in tetrahedral sites (C—C = 1.50 Å). All other atoms were assigned anisotropic U_{ij}'s and an extinction coefficient was adjusted [the final value being 0.090 (3) × 10^{-6}]. The largest final shift was 0.09σ; the largest feature in the final difference Fourier map was a peak of 0.35 e Å^{-3} near a disordered *tert*-butyl group. Final coordinates are given in Table 1.



(I)

In this revised centrosymmetric structure the molecule lies on a twofold axis. Bond lengths and angles are considerably improved. For example, the two chemically equivalent C═O bond lengths in the acetate groups, which were given as 1.303 (15) and 1.147 (13) Å in the earlier work, are now equal by symmetry at 1.191 (8) Å. Because of the disorder and large libration coefficients, the C—C distances in the *tert*-butyl groups are unreliable [range in this work = 1.47 (1)–1.58 (1) Å; range in the previous work = 1.36 (2)–1.54 (1) Å]. The terminal ethyl group C(9)—C(10) shows the largest displacement coefficients of all – up to 0.6 Å r.m.s. – and a much shortened C—C bond, at [1.39 (2) Å; in the earlier study, the two separate values for this bond length were 1.21 (2) and 1.55 (3) Å.

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} or B
C(1)	0.0979 (2)	0.1565 (3)	0.2300 (2)	0.0518 (9)
C(2)	0.1566 (2)	0.1295 (3)	0.3056 (2)	0.0523 (9)
C(3)	0.1931 (2)	0.0590 (3)	0.3009 (2)	0.0621 (11)
C(4)	0.1728 (2)	0.0138 (3)	0.2272 (2)	0.0628 (10)
C(5)	0.1119 (2)	0.0393 (3)	0.1546 (2)	0.0613 (10)
C(6)	0.0743 (2)	0.1101 (3)	0.1553 (2)	0.0534 (9)
O(1)	0.0581 (1)	0.2236 (2)	0.2297 (2)	0.0627 (7)
C(7)	0.0802 (2)	0.3201 (3)	0.2498 (3)	0.0920 (15)
C(8)	0.0621 (2)	0.3815 (4)	0.1781 (4)	0.0920 (16)
O(2)	0.0469 (2)	0.3552 (3)	0.1114 (3)	0.1296 (14)
O(3)	0.0682 (2)	0.4726 (3)	0.1993 (3)	0.1541 (18)
C(9)	0.0546 (4)	0.5407 (5)	0.1322 (6)	0.207 (4)
C(10)	0.0826 (5)	0.6284 (6)	0.1712 (7)	0.258 (5)
C(11)	0.2121 (2)	−0.0660 (4)	0.2242 (3)	0.0826 (13)
C(12)†	0.2742 (3)	−0.0905 (5)	0.3080 (3)	0.110 (2)
C(13)†	0.1708 (3)	−0.1621 (5)	0.1941 (4)	0.133 (3)
C(14)†	0.2230 (3)	−0.0481 (6)	0.1596 (4)	0.124 (2)
C(15)	0.0061 (2)	0.1332 (3)	0.0760 (2)	0.0552 (9)
C(16)	−0.0471 (2)	0.0890 (3)	0.0782 (2)	0.0473 (9)
C(17)	−0.0550 (2)	−0.0101 (3)	0.0733 (2)	0.0542 (10)
C(18)	−0.1026 (2)	−0.0549 (3)	0.0741 (2)	0.0549 (9)
C(19)	−0.1414 (2)	0.0038 (3)	0.0838 (2)	0.0550 (9)
C(20)	−0.1347 (2)	0.1028 (3)	0.0908 (2)	0.0477 (9)
C(21)	−0.0885 (2)	0.1454 (3)	0.0853 (2)	0.0467 (9)
O(4)	−0.0828 (1)	0.2454 (2)	0.0897 (1)	0.0526 (6)
C(22)	−0.1231 (2)	0.2911 (3)	0.0096 (2)	0.0717 (12)
C(23)	−0.1232 (2)	0.3977 (3)	0.0224 (3)	0.0843 (14)
O(5)	−0.1607 (2)	0.4153 (2)	0.0510 (2)	0.0984 (11)
C(24)	−0.1598 (3)	0.5135 (4)	0.0718 (4)	0.149 (3)
C(25)	−0.1125 (2)	−0.1645 (3)	0.0660 (3)	0.0779 (12)
C(26)‡	−0.0649 (5)	−0.2163 (4)	0.0593 (7)	0.163 (4)
C(27)‡	−0.1796 (4)	−0.1866 (5)	−0.0165 (6)	0.174 (4)
C(28)‡	−0.1131 (5)	−0.2033 (5)	0.1345 (6)	0.166 (4)
C(29)	−0.1748 (1)	0.1604 (3)	0.1093 (2)	0.0525 (9)
C(12′)§	0.1755	−0.1057	0.1367	10.0**
C(13′)§	0.2240	−0.1456	0.2831	10.0**
C(14′)§	0.2761	−0.0278	0.2503	10.0**
C(26′)¶	−0.1730	−0.1888	0.0566	10.0**
C(27′)¶	−0.0557	−0.2119	0.1430	10.0**
C(28′)¶	−0.1184	−0.1984	−0.0097	10.0**

† Site population = 0.846 (6).
‡ Site population = 0.790 (8).
§ Site population = 0.154.
¶ Site population = 0.210.
** Isotropic displacement parameter, B.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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