The structure was solved by direct methods. The H atoms were placed at calculated positions and refined as riding using the *SHELXL*93 defaults: O—H = 0.82, C—H = 0.93 Å, U(H) = $1.5U_{iso}$(parent atom). Examination of the crystal structure with *PLATON* (Spek, 1995) showed that there is one small (14 Å^3) solvent-accessible void in the asymmetric unit cell located at (0.425, 0.175, 0.735). However, the very small residual density at the void position excludes the possibility of occupation by a water molecule. All calculations were performed on a Pentium 150 MHz PC running LINUX.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: Enraf–Nonius *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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A Cyclic Monomer of Tetraethyleneglycol Succinate

Leroy Cronin, Madeleine H. Moore, J. Anthony Semlyen and Barry R. Wood

Department of Chemistry, University of York, Heslington, York YO1 5DD, England. E-mail: jas3@york.ac.uk

Abstract

The title compound, 1,4,7,10,13-pentaoxacycloheptadecane-14,17-dione, $C_{12}H_{20}O_7$, was prepared in a mixture of large ether–ester rings and isolated by preparative gel-permeation chromatography. The maximum internal diameter of its cavity was found to be 7.5 Å.

Comment

As part of a general study into large-ring molecules and cyclic polymers (Semlyen, 1986; Clarson & Semlyen, 1993; Semlyen, 1996), we have been developing a method of forming large cyclic esters and ether–esters (Wood, Semlyen & Hodge, 1997; Hamilton & Semlyen, 1997). The synthetic route involves producing a linear polymer from suitable monomers in a polyesterification reaction, then equilibrating the polymer in dilute solution to yield a range of cyclic compounds. These are then separated into a series of either individual compounds or narrow molar mass fractions by preparative gel-permeation chromatography (GPC). A monomeric ether–ester cyclic compound, (I), was recovered from a mixture of $[O(CH_2CH_2O)_4CO(CH_2)_2CO]_x$, where $x = 1$.

(I)
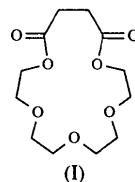
The structure (Fig. 1) indeed demonstrates that the polymer is cyclic with a maximum internal diameter (C3$\cdots$C8) of 7.55 (1) Å. The ester unit is planar with O4—C7 and O5—C7 bond lengths of 1.350 (8) and 1.199 (9) Å, respectively. The ester O4—C7—O5 angle is 121.4 (7)°. The ether linkages O2—C2 and O2—C3 are 1.431 (10) and 1.390 (9) Å, respectively, with a C2—O2—C3 bond angle of 114.6 (6)°.

The ring contains two planar ester units and two ether units, which are not crystallographically equivalent. The two ether units connected *via* O2 are buckled in the same manner, with O2—C2—C1—O1 and O2—

C3—C4—O3 torsion angles of 69.7 (7) and −70.9 (8)°, respectively. However, the two ether–ester units are quite different: O4 and O3 are mutually *anti* while O7 and O1 are *syn*, as seen in the values of the O4—C6—C5—O3 and O7—C11—C12—O1 torsion angles of 177.7 (7) and −76.4 (8)°, respectively. The two ester units are connected at the top of the ring by C8 and C9, and the whole unit, C7—C8—C9—C10, has a torsion angle of −59.9 (9)°, completing the ring.
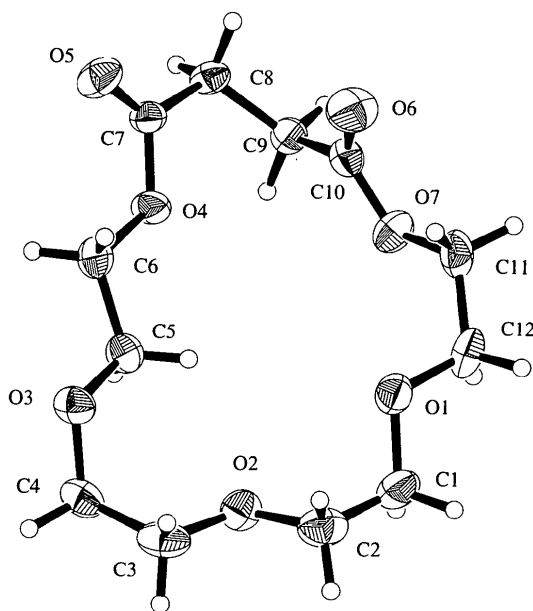

Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids and H atoms as spheres of arbitrary radii.

Experimental

The preparation of the cyclic tetraethyleneglycol succinate was carried out in two parts (Wood, Semlyen & Hodge, 1997). The first part involved the preparation of a high molar mass chain polymer by the condensation reaction between dimethylsuccinate and tetraethylene glycol using tetraisopropylorthotitanate as the catalyst. The reaction was carried out under melt conditions at 393 K for 48 h under a dry nitrogen atmosphere. The second part involved the generation of cyclic oligomers using a dilute solution ring-chain transesterification reaction. The high molar mass chain polymer was refluxed in chlorobenzene at a 1:50 dilution (weight of polymer to weight of solvent) at 403 K for 96 h, using dibutyltin bis(2-ethylhexanoate) as the transesterification catalyst. The cyclic oligomers were then separated into a series of sharp molar mass fractions by preparative GPC. The monomeric ring component was collected as a pure single compound, whereas higher oligomers were collected as mixtures consisting of different ring sizes. The monomeric ring compound was found to crystallize from the melt. Subsequent recrystallization was from *n*-pentane.

Crystal data

$C_{12}H_{20}O_7$	Mo $K\alpha$ radiation
$M_r = 276.28$	$\lambda = 0.71069$ Å
Orthorhombic	Cell parameters from 20
$P2_12_12_1$	reflections
$a = 8.97$ (2) Å	$\theta = 5.25$–$7.55°$
$b = 18.89$ (3) Å	$\mu = 0.107$ mm^{-1}
$c = 8.355$ (8) Å	$T = 293$ (2) K
$V = 1416$ (4) Å^3	Plate
$Z = 4$	$0.80 \times 0.60 \times 0.05$ mm
$D_x = 1.296$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Rigaku AFC-6S diffractometer	$\theta_{max} = 25°$
ω–2θ scans	$h = 0 \rightarrow 10$
Absorption correction: none	$k = 0 \rightarrow 22$
1457 measured reflections	$l = 0 \rightarrow 9$
1457 independent reflections	3 standard reflections
758 reflections with	every 150 reflections
$I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.0527$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
$wR(F^2) = 0.3111$	Extinction correction: none
$S = 1.053$	Scattering factors from
1449 reflections	*International Tables for*
172 parameters	*Crystallography* (Vol. C)
H atoms riding	Absolute configuration:
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$	Flack (1983)
where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter not
$(\Delta/\sigma)_{max} < 0.001$	reliably determined

Table 1. *Selected geometric parameters (Å, °)*

O2—C3	1.390 (9)	O5—C7	1.199 (9)
O3—C4	1.418 (8)	C3—C4	1.502 (11)
O3—C5	1.419 (7)	C5—C6	1.495 (9)
O4—C7	1.350 (8)	C7—C8	1.486 (10)
O4—C6	1.442 (7)	C8—C9	1.510 (10)
C3—O2—C2	114.6 (6)	O4—C6—C5	104.8 (6)
C4—O3—C5	111.6 (6)	O5—C7—O4	121.4 (7)
C7—O4—C6	119.1 (6)	O5—C7—C8	127.1 (7)
O2—C3—C4	110.3 (6)	O4—C7—C8	111.5 (6)
O3—C4—C3	112.6 (6)	C7—C8—C9	114.9 (6)
O3—C5—C6	107.4 (6)		

H atoms were introduced at geometrically calculated positions and thereafter allowed to ride on their parent C atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The structure is not of high precision and this is mainly due to the crystal size which was extremely small in one dimension (0.05 mm), giving a weak room-temperature data set.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1992). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1137). Services for accessing these data are described at the back of the journal.

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(Achari & Neidle, 1976). Numerous related compounds were prepared in the last 30 years and they are often called quinolones. Newer quinolones are well known chemotherapeutics with broad antibacterial activity. The title boron complex, (I), is an intermediate in the synthesis of some quinolone molecules (Dolenc, Šket, Barbo & Zupet, 1995) and the structure of a difluoroboric–quinolone complex has already been published (Jordis, Sauter, Burkart, Henning & Gelbin, 1991). The present investigation was aimed at determining the crystal structure of a new boron–quinolone complex and comparing the results with data from known quinolone complexes.

(I)

Boron Complex of a Member of the Quinolone Family

Iztok Turel,[a] Ivan Leban,[a] Peter Bukovec[a] and Martin Barbo[b]

[a]*Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, PO Box 537, 1001 Ljubljana, Slovenia, and* [b]*Krka Pharmaceutical Factory, Šmarješka c. 6, 8000 Novo mesto, Slovenia. E-mail: iztok.turel@uni-lj.si*

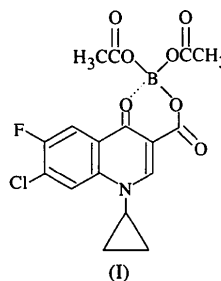## Abstract

In the title compound, bis(acetato-*O*)(7-chloro-1-cyclopropyl-6-fluoro-4-oxo-1,4-dihydroquinoline-3-carboxylato-O^3,O^4)boron, $C_{17}H_{14}BClFNO_7$, the B atom is coordinated by four O atoms (two of the quinolone moiety and one each of the two acetate groups) and adopts a slightly distorted tetrahedral geometry. The B—O distances are 1.458 (5) and 1.501 (4) Å for quinolone, and 1.451 (5) and 1.458 (5) Å for acetate.

Comment

Nalidixic acid (1-ethyl-1,4-dihydro-7-methyl-4-oxo-1,8-naphtyridine-3-carboxylic acid) was synthesized in the early 1960s and its crystal structure has been determined

The B atom is coordinated by four O atoms and adopts a slightly distorted tetrahedral geometry, with O—B—O angles between 105.6 (3) and 113.1 (3)° (Fig. 1). The B atom is bonded to both the carboxylic and carbonyl O atoms of the quinolone molecule, the B—O11 distance being somewhat shorter than B—O1 (Table 1). The corresponding distances in the difluoroboric–quinolone complex (Jordis, Sauter, Burkart,
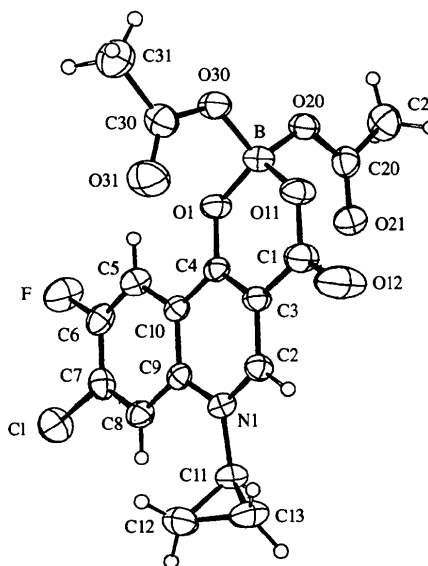

Fig. 1. *ORTEP*II (Johnson, 1971) view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.