The structure was solved by direct methods. The H atoms were placed at calculated positions and refined as riding using the SHELXL93 defaults: O—H = 0.82, C—H = 0.93 Å, U(H) = 1.5Uiso(parent atom). Examination of the crystal structure with PLATON (Spek, 1995) showed that there is one small (14 Å³) 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.


We are indebted to the Cultural Service of the German Federal Republic Embassy, the Deutscher Akademischer Austauschdienst (DAAD) and the German Agency for Technical Cooperation (GTZ) for the offer of a CAD-4 automatic diffractometer which enabled the experimental work to be carried out. This work was supported by JNICT and the CIÊNCIA program.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1084). Services for accessing these data are described at the back of the journal.

References
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.

**Crystal data**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>C12H20O7</td>
<td>Mr = 276.28</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>Space group</td>
<td>P212121</td>
</tr>
<tr>
<td>a</td>
<td>8.97 (2) Å</td>
</tr>
<tr>
<td>b</td>
<td>18.89 (3) Å</td>
</tr>
<tr>
<td>c</td>
<td>8.355 (8) Å</td>
</tr>
<tr>
<td>V</td>
<td>1416 (4) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Dm</td>
<td>not measured</td>
</tr>
</tbody>
</table>

**Data collection**

- Rigaku AFC-6S diffractometer
- Mo Kα radiation
- λ = 0.71069 Å
- μ = 0.107 mm⁻¹
- T = 293 (2) K
- Plate
- 0.80 × 0.60 × 0.05 mm
- Colourless

**Refinement**

- Refinement on F²
- R[F² > 2σ(F²)] = 0.0527
- wR(F²) = 0.3111
- S = 1.053
- 1449 reflections
- 172 parameters
- H atoms riding
- w = 1/[σ²(F²) + (0.1P)²]
- where P = (F² + 2F²)³
- (Δσ/σ)max < 0.001

**H atoms**

H atoms were introduced at geometrically calculated positions and thereafter allowed to ride on their parent C atoms with Uiso(H) = 1.2Ueq(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.

LC and BRW would like to thank the EPSRC for funding.

**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 tetrakis-propylorthotitanate 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.

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

<table>
<thead>
<tr>
<th>Bond</th>
<th>Value (Å)</th>
<th>Bond</th>
<th>Value (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O2—C3</td>
<td>1.390 (9)</td>
<td>O5—C7</td>
<td>1.199 (9)</td>
</tr>
<tr>
<td>O3—C4</td>
<td>1.418 (8)</td>
<td>O3—C5</td>
<td>1.495 (9)</td>
</tr>
<tr>
<td>O4—C7</td>
<td>1.350 (8)</td>
<td>C5—C6</td>
<td>1.486 (10)</td>
</tr>
<tr>
<td>O4—C6</td>
<td>1.442 (7)</td>
<td>C8—C9</td>
<td>1.510 (10)</td>
</tr>
<tr>
<td>C3—O2—C2</td>
<td>114.6 (6)</td>
<td>C4—O4—C5</td>
<td>104.8 (6)</td>
</tr>
<tr>
<td>C4—O3—C5</td>
<td>111.6 (6)</td>
<td>C5—O7—C4</td>
<td>121.4 (7)</td>
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<tr>
<td>C7—O4—C6</td>
<td>119.1 (6)</td>
<td>C6—O2—C3</td>
<td>127.1 (7)</td>
</tr>
<tr>
<td>O2—C3—C4</td>
<td>103.3 (6)</td>
<td>O4—C7—C8</td>
<td>115.5 (6)</td>
</tr>
<tr>
<td>O3—C4—C3</td>
<td>112.6 (6)</td>
<td>C7—C8—C9</td>
<td>114.9 (6)</td>
</tr>
<tr>
<td>O3—C5—C6</td>
<td>107.4 (6)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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.

References


Boron Complex of a Member of the Quinolone Family
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Abstract
In the title compound, bis(acetato-O)(7-chloro-1-cyclopropyl-6-fluoro-4-oxo-1,4-dihydroquinoline-3-carboxylato-O3,Oa)boron, C17H16BC1F5NO7, 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-napthyridine-3-carboxylic acid) was synthesized in the early 1960s and its crystal structure has been determined (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.

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, Henning & Gelbin, 1991).

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