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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*

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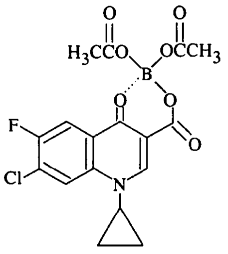
Abstract

In the title compound, bis(acetato-*O*)(7-chloro-1-cyclopropyl-6-fluoro-4-oxo-1,4-dihydroquinoline-3-carboxylato-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 (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)

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,

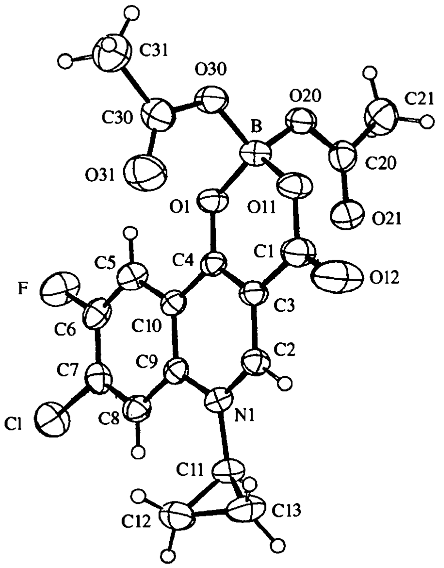


Fig. 1. *ORTEP*II (Johnson, 1971) view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Henning & Gelbin, 1991) are 1.467 and 1.468 Å. The coordination sphere is completed by two O atoms of the acetate groups. The C4—O1 distance [1.295 (4) Å] agrees well with the corresponding distance in the difluoroboric–quinolone complex [1.298 Å], but differs markedly from that in the copper–quinolone complex [1.268 (3) Å; Turel, Leban & Bukovec, 1994]. Similarly, the C1—O11 distance in both the title compound and the difluoroboric–quinolone complex (1.318 Å) differs from the corresponding distance in the copper complex [1.271 (3) Å]. These discrepancies could be explained by differences in the size of the B and Cu atoms, and by their different electronic configurations.

The distances and angles between the atoms of the remaining part of the quinolone molecule are similar to those of the quinolone molecules reported earlier (Turel, Leban & Bukovec, 1994; Turel, Gruber, Leban & Bukovec, 1996; Turel, Leban, Zupančič, Bukovec & Gruber, 1996).

Experimental

Colourless crystals suitable for single-crystal diffraction work were prepared by recrystallization of the microcrystalline title compound from aqueous solution. A synthetic route reported elsewhere was used for the isolation of the title compound (Dolenc, Šket, Barbo & Zupet, 1995).

Crystal data

$C_{17}H_{14}BClFNO_7$
$M_r = 409.55$
Orthorhombic
Pbca
$a = 8.754 (2)$ Å
$b = 15.055 (3)$ Å
$c = 26.742 (5)$ Å
$V = 3524.4 (12)$ Å^3
$Z = 8$
$D_x = 1.544$ Mg m^{-3}
$D_m = 1.53 (5)$ Mg m^{-3}
D_m measured by flotation in chlorobenzene/CCl_4

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–12°$
$\mu = 0.270$ mm^{-1}
$T = 293 (2)$ K
Plate
0.62 × 0.41 × 0.19 mm
Colourless

Data collection

Modified Stoe diffractometer
Fixed 1.2° ω scans
Absorption correction: none
3501 measured reflections
3107 independent reflections
1978 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.0492$

$\theta_{\max} = 27.49°$
$h = 0 \rightarrow 10$
$k = -1 \rightarrow 19$
$l = -1 \rightarrow 34$
3 standard reflections every 400 reflections intensity decay: 2.2%

Refinement

Refinement on F^2
$R(F) = 0.0534$
$wR(F^2) = 0.1395$
$S = 1.076$
3101 reflections
253 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 2.7713P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.059$
$\Delta\rho_{\max} = 0.269$ e Å^{-3}
$\Delta\rho_{\min} = -0.259$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C4—O1	1.295 (4)	O1—B	1.501 (4)
O11—C1	1.302 (4)	B—O20	1.451 (5)
O11—B	1.458 (5)	B—O30	1.458 (5)
O20—B—O11	110.2 (3)	O20—B—O1	109.3 (3)
O20—B—O30	105.6 (3)	O11—B—O1	113.1 (3)
O11—B—O30	110.5 (3)	O30—B—O1	107.9 (3)

All H atoms were found in the difference electron-density map and were included in the refinement at calculated positions. For all H atoms, the values of the isotropic displacement parameters were taken to be 1.3 times those of the attached heavy atoms.

Data collection: local program (Kratky, 1993). Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989), *ORTEP*II (Johnson, 1971) and *PLUTON* (Spek, 1991). Software used to prepare material for publication: *SHELXL*93.

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