

Data collection

Enraf–Nonius CAD-4
diffractometer $\theta_{\max} = 24.98^\circ$
 $h = -38 \rightarrow 38$
 $2\theta/\omega$ scans $k = 0 \rightarrow 6$
 Absorption correction: none $l = 0 \rightarrow 26$
 3603 measured reflections 3 standard reflections
 3603 independent reflections every 100 reflections
 3285 reflections with frequency: 120 min
 $I > 2\sigma(I)$ intensity decay: 4%

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} = 0.053$
 $R[F^2 > 2\sigma(F^2)] = 0.054$ $\Delta\rho_{\max} = 0.311 \text{ e } \text{Å}^{-3}$
 $wR(F^2) = 0.121$ $\Delta\rho_{\min} = -0.291 \text{ e } \text{Å}^{-3}$
 $S = 1.128$ Extinction correction: none
 3285 reflections Scattering factors from
 259 parameters *International Tables for*
 H atoms riding *Crystallography* (Vol. C)

$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Table 1. Selected geometric parameters (Å, °)

S1—C1	1.738 (2)	O2—C5	1.203 (2)
S1—C3	1.736 (2)	O3—C5	1.293 (2)
S2—C1	1.617 (2)	C2—C3	1.482 (2)
N—C1	1.385 (2)	C3—C6	1.352 (2)
N—C2	1.397 (2)	C4—C5	1.513 (2)
N—C4	1.431 (2)	C6—C7	1.443 (2)
O1—C2	1.197 (2)		
C1—S1—C3	92.96 (8)	N—C2—C3	109.46 (12)
C1—N—C2	116.78 (13)	C6—C3—C2	119.36 (13)
C1—N—C4	123.57 (13)	C6—C3—S1	130.21 (13)
C2—N—C4	118.61 (12)	C2—C3—S1	110.42 (11)
N—C1—S2	126.24 (13)	N—C4—C5	110.93 (14)
N—C1—S1	110.34 (11)	O2—C5—O3	125.0 (2)
S2—C1—S1	123.42 (10)	O2—C5—C4	123.35 (15)
O1—C2—N	122.4 (2)	O3—C5—C4	111.69 (14)
O1—C2—C3	128.12 (15)	C3—C6—C7	131.89 (14)

Most H atoms were geometrically placed, except for H3 which was unambiguously located by difference Fourier methods, and were constrained with a riding model.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *Xtal.GX* (Hall & du Boulay, 1995) and *PLUTON92* (Spek, 1992).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1236). Services for accessing these data are described at the back of the journal.

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2,2'-Biphenol

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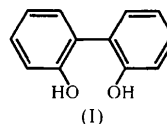
(Received 15 October 1997; accepted 9 February 1998)

Abstract

The structure of anhydrous 2,2'-biphenol, C₁₂H₁₀O₂, is described, together with the hydrogen-bonding scheme. The first hydrogen bond is an intramolecular one, while the second one connects the biphenol molecules to build infinite chains running in the **b** direction. A comparison with the structure of 2,2'-biphenol monohydrate is made.

Comment

In relation to the synthesis of Lewis acid derivatives of biphenol (Schaverien *et al.*, 1992), we are interested in the organization of pure phenols in the solid state. Recently, biphenol has been used as an activator of coordination catalysts (Matsukawa & Mikami, 1996; Mikami & Matsukawa, 1997), but little is known about the structures of these complexes. The structure of 2,2'-biphenol monohydrate, (II), was recently described (Chen *et al.*, 1996), and a comparison can be made of the structures of the monohydrate, (II), and the anhydrous form, (I).



All bond lengths and angles found in (I) (Table 1) agree with those in the literature (Allen *et al.*, 1987). The C6—C7 single bond is significantly longer than the C—C bonds in the two aromatic rings. The dihedral angles between the aromatic rings, namely 48.4(1)° in (I) and 67.6(1)° in (II), are significantly different.

It is also worth noting that the two hydroxyl groups of (I) are *cis* with respect to one another, while in the monohydrate, this is not true. This *cis* arrangement allows the formation of an intramolecular hydrogen bond, in which the O...O distance is 2.606(1) Å (Table 2). There is also a second hydrogen bond, linking the biphenyl molecules into a supramolecular chain running along the **b** direction (Fig. 1). This contrasts with the case of the monohydrate, where the *trans* arrangement of the O atoms inhibits an intramolecular bond, and hydrogen bonding *via* the lattice water molecules connects the biphenyl molecules into a two-dimensional network.

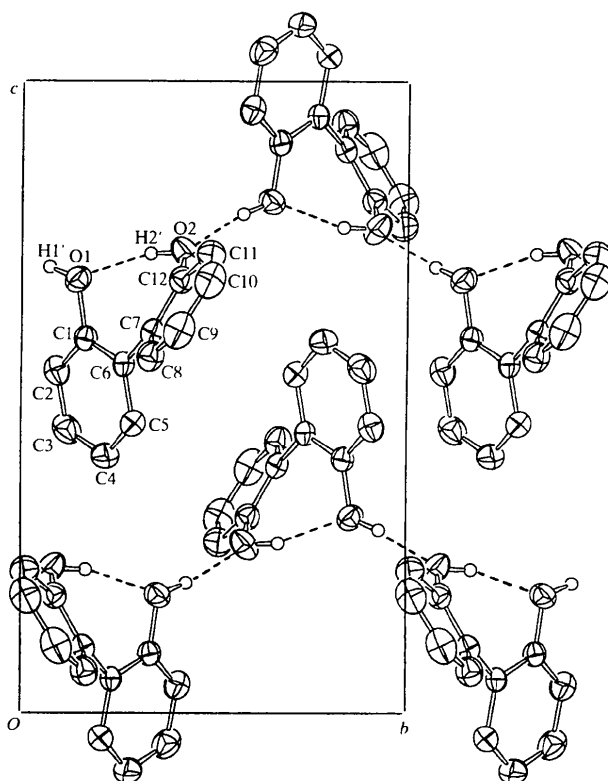


Fig. 1. An ORTEP (Johnson, 1976) projection of the crystal structure of (I) along the **a** direction, shown with 35% probability displacement ellipsoids. The hydrogen bonds are represented by dashed lines.

Experimental

Crystals of the title compound were obtained by slow evaporation of a solution of amorphous 2,2'-biphenol (1 mmol) in dry toluene (10 ml).

Crystal data

C₁₂H₁₀O₂
M_r = 186.21

Mo K α radiation
 λ = 0.7107 Å

Monoclinic
*P*2₁/*c*
a = 7.392 (2) Å
b = 8.965 (6) Å
c = 14.925 (4) Å
 β = 102.90 (2)°
V = 964.1 (6) Å³
Z = 4
*D*_x = 1.283 Mg m⁻³
*D*_m not measured

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω scans
Absorption correction: none
3079 measured reflections
2982 independent reflections
1695 reflections with *I* > 1.8 σ (*I*)

Refinement

Refinement on *F*
R = 0.042
wR = 0.053
S = 1.768
1695 reflections
168 parameters
All H atoms refined
w = 1/[$\sigma^2(F_o)$ + 0.00042|*F_o*|²]
(Δ/σ)_{max} = 0.050

Cell parameters from 24 reflections
 θ = 9.9–11.5°
 μ = 0.087 mm⁻¹
T = 293 K
Stout monoclinic prism
0.50 × 0.48 × 0.42 mm
Translucent pale yellow

*R*_{int} = 0.019
 θ _{max} = 29.96°
h = -10 → 10
k = 0 → 12
l = 0 → 20
2 standard reflections
frequency: 120 min
intensity decay: none

$\Delta\rho$ _{max} = 0.16 e Å⁻³
 $\Delta\rho$ _{min} = -0.24 e Å⁻³
Extinction correction: Zachariasen (1967, 1968)
Extinction coefficient: 3.20 (4) × 10⁻⁵
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

O1—C1	1.374 (1)	C6—C7	1.486 (2)
O2—C12	1.381 (2)	C7—C8	1.396 (2)
C1—C2	1.383 (2)	C7—C12	1.390 (2)
C1—C6	1.389 (2)	C8—C9	1.375 (2)
C2—C3	1.372 (2)	C9—C10	1.367 (2)
C3—C4	1.370 (2)	C10—C11	1.376 (2)
C4—C5	1.377 (2)	C11—C12	1.378 (2)
C5—C6	1.395 (2)		
O1—C1—C2	121.3 (1)	C6—C7—C8	119.1 (1)
O1—C1—C6	117.07 (9)	C6—C7—C12	124.2 (1)
C2—C1—C6	121.6 (1)	C8—C7—C12	116.7 (1)
C1—C2—C3	119.6 (1)	C7—C8—C9	121.8 (1)
C2—C3—C4	120.3 (1)	C8—C9—C10	119.9 (1)
C3—C4—C5	119.8 (1)	C9—C10—C11	119.9 (1)
C4—C5—C6	121.6 (1)	C10—C11—C12	120.0 (1)
C1—C6—C5	116.9 (1)	O2—C12—C7	121.2 (1)
C1—C6—C7	123.23 (9)	O2—C12—C11	117.2 (1)
C5—C6—C7	119.8 (1)	C7—C12—C11	121.5 (1)

Table 2. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1'...O2'	0.86 (2)	1.79 (2)	2.645 (1)	168 (1)
O2—H2'...O1	0.86 (2)	1.80 (2)	2.606 (1)	156 (1)

Symmetry code: (i) -*x*, *y* - ½, ¾ - *z*.

The H atoms were found in a final difference Fourier map and were refined isotropically.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR88* (Burla *et al.*, 1989). Program(s) used to

refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

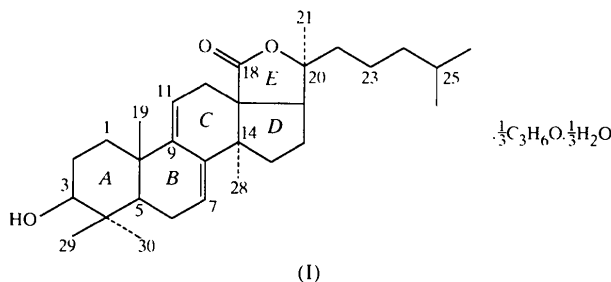
Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1005). Services for accessing these data are described at the back of the journal.

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Comment

Two types of transformation of the pentacyclic holostane skeleton in triterpene glycosides of holothurians resulting from the action of protonic acids are well known (Elyakov & Stonik, 1986). One type is related to a double-bond migration in aglycones with a labile 9 β -H-7(8)-ene fragment. The stereochemical details of this migration have been studied carefully (Ilyin *et al.*, 1991). The second transformation is typical for aglycones with a labile 12 α -hydroxy-9(11)-ene fragment and forms holosta-7,9-dienes under appropriate conditions (Chanley & Rossi, 1969). We report herein the structure of a product (Fig. 1) of this type of transformation, namely the artificial genin of the holothurian *Bohadschia argus* glycosides (Antonov & Stonik, 1986), as its $\frac{1}{3}$ -acetone solvate $\frac{1}{3}$ -hydrate, (I). This holothurinogenin, seychellogenin, has been isolated from the sea cucumber *Bohadschia koellikeri* (Roller *et al.*, 1969).



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(20S)-3 β -Hydroxyholosta-7,9-diene

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Abstract

The crystal and molecular structures of the title triterpenoid have been determined. There are three molecules of this triterpenoid (C₃₀H₄₆O₃), one molecule of acetone (C₃H₆O) and a molecule of water (H₂O) in the asymmetric unit, *i.e.* formula C₃₀H₄₆O₃ · $\frac{1}{3}$ C₃H₆O · $\frac{1}{3}$ H₂O. The molecules of the triterpenoid have different conformations of both the side chains and the γ -lactone rings.

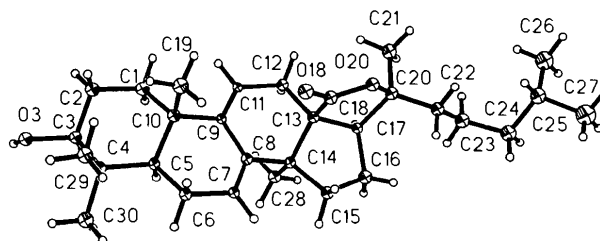


Fig. 1. The molecular structure of molecule *A* of the title compound with 30% probability non-H-atom displacement spheres (H atoms as spheres of arbitrary radii).