N3—C11—S5	115.7 (2)	N3a—C11a—S5a	116.0 (2)
S6—C11—S5	121.0 (2)	S6a—C11a—S5a	120.9 (2)
C17—C16—C21	119.3 (3)	C17a—C16a—C21a	119.0 (3)
C17—C16—Te1	120.2 (2)	C17a—C16a—Te1a	120.8 (2)
C21—C16—Te1	120.6 (2)	C21a—C16a—Te1a	120.1 (2)

S2—Te1—S1—C1	4.88 (10)
S4—Te1—S3—C6	0.16 (9)
S6—Te1—S5—C11	−3.02 (10)
C4—N1—C1—S2	−9.1 (5)
C2—N1—C1—S1	−7.0 (4)
C1—N1—C2—C3	90.7 (4)
C1—N1—C4—C5	95.9 (4)
C7—N2—C6—S3	−0.8 (4)
C9—N2—C6—S4	3.1 (4)
C6—N2—C7—C8	−87.6 (4)
C6—N2—C9—C10	84.6 (5)
C12—N3—C11—S6	0.9 (5)
C14—N3—C11—S5	−15.4 (5)
C14′—N3—C11—S5	34.9 (6)
C11—N3—C12—C13	−93.3 (5)
C11—N3—C14—C15	97.0 (5)
C11—N3—C14′—C15′	−114.2 (8)
S5—Te1—C16—C17	152.9 (3)
S1—Te1—C16—C17	−127.7 (3)
S4—Te1—C16—C17	74.5 (3)
S2—Te1—C16—C17	−62.4 (3)
S3—Te1—C16—C17	10.8 (3)
S2a—Te1a—S1a—C1a	4.44 (10)
S4a—Te1a—S3a—C6a	0.41 (11)
S6a—Te1a—S5a—C11a	9.95 (9)
C2a—N1a—C1a—S2a	6.1 (4)
C4a—N1a—C1a—S1a	3.5 (4)
C1a—N1a—C2a—C3a	−91.3 (4)
C1a—N1a—C4a—C5a	−88.6 (4)
C7a—N2a—C6a—S3a	3.4 (5)
C9a—N2a—C6a—S4a	1.7 (4)
C6a—N2a—C7a—C8a	84.1 (4)
C6a—N2a—C9a—C10a	−91.3 (5)
C12a—N3a—C11a—S6a	1.4 (4)
C14″—N3a—C11a—S5a	−17.6 (5)
C14a—N3a—C11a—S5a	6.6 (4)
C11a—N3a—C12a—C13a	−107.0 (4)
C11a—N3a—C14a—C15a	−93.8 (5)
C11a—N3a—C14″—C15″	−77.4 (8)
S5a—Te1a—C16a—C17a	−42.1 (2)
S4a—Te1a—C16a—C17a	−120.2 (2)
S1a—Te1a—C16a—C17a	37.5 (2)
S2a—Te1a—C16a—C17a	102.5 (2)
S3a—Te1a—C16a—C17a	174.9 (2)

The C14–C15 ethyl group in (2) is disordered in both independent molecules. Two positions were refined isotropically for each molecule with occupancy factor ratios of 0.67/0.33 and 0.6/0.4, respectively. Refinement was carried out on F^2 for all reflections except for 66 [structure (1)] and 24 [structure (2)] with very negative F^2 or flagged for potential systematic errors (e.g. extinction).

For both compounds, program(s) used to solve structures: SHELXS86 (Sheldrick, 1985); program(s) used to refine structures: SHELXL93 (Sheldrick, 1993).

References

Bondi, A. (1964). J. Phys. Chem. 68, 441–451.
Dakternieks, D., Di Giacomo, R., Gable, R. W. & Hoskins, B. F. (1988). J. Am. Chem. Soc. 110, 6762–6768.

Deuten, K. von, Schnabel, W. & Klar, G. (1980). Phosphorus Sulfur, 9, 93–98.
Esperås, S. & Husebye, S. (1972). Acta Chem. Scand. 26, 3293–3304.
Foss, O. (1953). Acta Chem. Scand. 7, 227–228.
Husebye, S. (1979). Acta Chem. Scand. Ser. A, 33, 485–490.
Husebye, S., Maartmann-Moe, K. & Steffensen, W. (1990). Acta Chem. Scand. 44, 579–583.
Husebye, S. & Thowsen, A. G. (1981). Acta Chem. Scand. Ser. A, 35, 443–449.
Rao, G. V. N. A., Seshasayee, M., Aravamudan, G. & Radha, K. (1983). Inorg. Chem. 22, 2590–2593.
Sheldrick, G. M. (1985). SHELXS86. Program for the Solution of Crystal Structures. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. Univ. of Göttingen, Germany.
Wieber, M. & Lang, S. (1994). Z. Anorg. Allg. Chem. 620, 1397–1402.

Benz[4,5]isoquino[1,2-b]quinazoline-7,9-dione and a Rearrangement Product of its Hydrolysis, 2-(1,8-Naphthalenedicarboximido)benzamide

Sergey V. Lindeman, Igor I. Ponomarev and Alexandr L. Rusanov

Institute of Organoelement Compounds, 28 Vavilov Street, Moscow 117813, Russia

Abstract

Benz[4,5]isoquino[1,2-b]quinazoline-7,9-dione, (1), $C_{19}H_{10}N_2O_2$, was isolated as a product of the reaction between naphthalic anhydride and anthranylamide after crystallization from dimethylformamide. Recrystallization from concentrated (95%) formic acid resulted in 2-(1,8-naphthalenedicarboximido)benzamide, (2), $C_{19}H_{12}N_2O_3$, as a rearrangement product of hydrolysis [alternative systematic name: 2-(1,3-dioxo-2,3-dihydro-1H-benz[de]isoquinol-2-yl)benzamide]. The two crystallographically independent molecules of (1) [(1a) and (1b)] are substantially non-planar as a result of strong intramolecular steric repulsion between their cis-arranged carbonyl groups [the O···O distances are 2.584 (3) and 2.664 (3) Å, and the dihedral angles between the naphthalene and benzene nuclei are 8.75 (7) and 14.46 (7)°, respectively]. Molecule (2) is also sterically hindered with an approximately orthogonal orientation of the naphthalenedicarboximide and o-phenylene groups [dihedral angle 87.05 (4)°] and absence of π-conjugation between the o-phenylene and amide groups [dihedral angle 40.02 (6)°].

Comment

As part of our investigations into the synthesis of regular polynaphthoylenequinazolones from bis-naphthalic anhydrides and bis-anthranylamides, the model reaction between naphthalic anhydride and anthranylamide has been carried out (see scheme below) (Ponomarev, Skuratova, Lindeman, Sinichkin, Vinogradova & Rusanov, 1994).
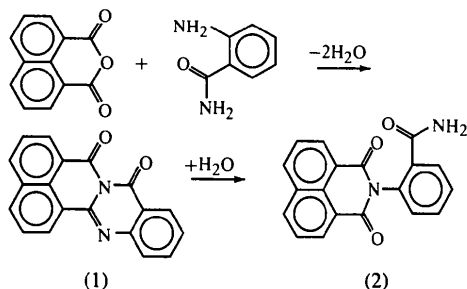
Only one of the two possible isomeric products, benz-[4,5]isoquino[1,2-b]quinazoline-7,9-dione, (1), was isolated from the reaction mixture by crystallization from dimethylformamide. Recrystallization from concentrated (95%) formic acid resulted in the formation of 2-(1,8-naphthalenedicarboximido)benzamide, (2), which may be considered as a rearrangement product of the hydrolysis of (1).

The two crystallographically independent molecules of (1) [(1a) and (1b) in Fig. 1] are substantially distorted relative to the ideal π-conjugated planar structure. The dihedral angles between the planes of the naphthalene and benzene nuclei are 8.75 (7) for (1a) and 14.46 (7)° for (1b). This results from bending of the non-aromatic rings: the diazoline ring of the quinazoline moiety adopts a sofa conformation [atoms N1 and N1a are shifted from the planes of the related benzene rings by 0.106 and 0.208 (4) Å, respectively, whereas the deviation of the other atoms does not exceed 0.044 (3) Å]. The tetrahydropyridine ring of the isoquinoline moiety has a boat-like conformation with atoms N1 (N1a) and C12 (C12a) shifted by 0.194 (3) Å [0.258 (3) Å] and 0.103 (3) Å [0.104 (3) Å] from the mean plane of the naphthalene moiety; atom C11 (C11a) deviates by 0.085 (3) Å [0.034 (3) Å] in the opposite direction.

These distortions are caused principally by forced short intramolecular contacts between cis-disposed carbonyl groups: the O···O distances are 2.584 and 2.664 (3) Å in molecules (1a) and (1b), respectively (at such a mutual orientation of the polar carbonyl groups, an additional electrostatic repulsion of their dipoles evidently takes place). In addition, the short intramolecular contacts O1···H—C7 [2.57 (3) and 2.55 (3) Å in molecules (1a) and (1b), respectively], O1'···H—C6' [2.56 (3) and 2.60 (3) Å] and N2···H—C2 [2.40 (3) and 2.46 (3) Å], are also seemingly of the repulsive type. As a result, atoms O1 and N2 deviate in opposite directions from the mean plane of the naphthalene moiety

by 0.404 (3) and −0.117 (3) Å in (1a) and by 0.293 (3) and −0.088 (3) Å in (1b), and atom O1' deviates from the mean plane of the phenylene moiety by 0.186 (4) in (1a) and 0.085 (4) Å in (1b).

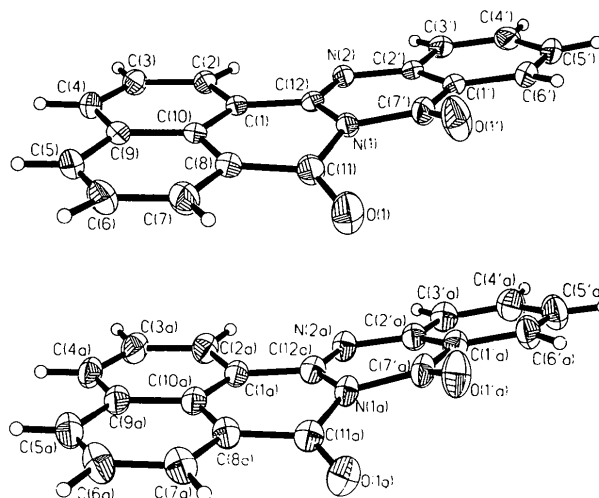Fig. 1. Perspective view of the two crystallographically independent molecules of (1) [top (1a), bottom (1b)] in comparable orientations [molecule (1b) is shown inverted relative to the table of coordinates] showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

The noticeable difference in the degree of nonplanarity for the two crystallographically independent molecules (1a) and (1b) seems to result from their different crystal environments. In (1), the molecules are packed in stacks [···(1b')···(1b)···(1a')···(1a)···]ₙ along [10̅1] (Fig. 2). In the stacks, (1a) is overlapped fully and closely (interplanar distances are ca 3.42 Å)
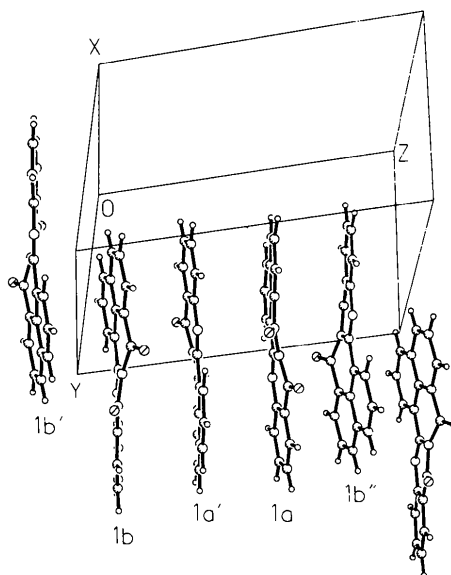

Fig. 2. Perspective view of a stack of molecules of (1) in the crystal.

by two antiparallel neighbouring molecules ($1b''$) and ($1a'$), but only the naphthalenedicarboximide moiety is overlapped by the packing of the antiparallel ($1b$) and ($1b'$) molecules, where the interplanar distance is ca 3.53 Å.

Molecule (2), to our knowledge, is the first aryl-substitued 1,8-naphthalenedicarboximide to be investigated structurally. As a result of steric restrictions enforced by the two carbonyl groups, the benzamide moiety is almost perpendicular to the planar naphthalenedicarboximide moiety [dihedral angle 87.05 (4)°; Fig. 3]. The N1—C2' bond length of 1.449 (1) Å is longer than the standard value of 1.371 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The amide group is also twisted around the C1'—C7' bond [by 40.02 (6)°] such that it is not π-conjugated with the benzene ring. Nevertheless, the length of this bond [1.502 (2) Å] is normal (Allen *et al.*, 1987).
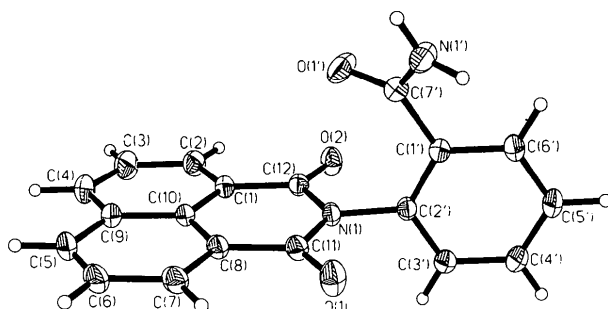

Fig. 3. Perspective view of molecule (2) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

The bond-length distribution in the naphthalenedicarboximide moieties of (1) and (2) is very similar to that found earlier in *N*-ethyl-1,8-naphthalimide (Easton, Gulbis, Hoskins, Scharfbillig & Tiekink, 1992), but formation of the quinazoline ring leads to a substantial loosening of the N1—C11 bond [1.449 (3) in ($1a$) and 1.439 (3) Å in ($1b$) in comparison with 1.394 (1) Å in (2)], whereas its equivalent, the N1—C12 bond, remains constant [1.401 (3) and 1.399 (3) Å versus 1.399 (1) Å]. This may be of importance to the mechanism of hydrolysis of (1).

Experimental

Compounds (1) and (2) were obtained as described earlier (Ponomarev, Skuratova, Lindeman, Sinichkin, Vinogradova & Rusanov, 1994). (1) was crystallized from dimethylformamide; (2) from formic acid.

Compound (1)

Crystal data

$C_{19}H_{10}N_2O_2$	Mo $K\alpha$ radiation
$M_r = 298.29$	$\lambda = 0.71069$ Å

Triclinic
$P\bar{1}$
$a = 9.065$ (2) Å
$b = 10.858$ (2) Å
$c = 14.118$ (3) Å
$\alpha = 89.59$ (3)°
$\beta = 71.94$ (3)°
$\gamma = 83.63$ (3)°
$V = 1312.4$ (5) Å^3
$Z = 4$
$D_x = 1.510$ Mg m^{-3}

Cell parameters from 24
  reflections
$\theta = 10$–11°
$\mu = 0.100$ mm^{-1}
$T = 173$ (2) K
Prism
0.5 × 0.3 × 0.25 mm
Yellow

Data collection

Siemens $P3$ diffractometer
$\omega/2\theta$ scans
Absorption correction:
  none
5811 measured reflections
5810 independent reflections
3772 observed reflections
  $[I > 2\sigma(I)]$
$R_{int} = 0.191$

$\theta_{max} = 28.97°$
$h = 0 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 18$
2 standard reflections
  monitored every 98
  reflections
  intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0696$
$wR(F^2) = 0.1679$
$S = 1.134$
5805 reflections
495 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.1255P)^2 + 0.1195P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.572$ e Å^{-3}
$\Delta\rho_{min} = -0.392$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
  from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
O1	0.3067 (2)	0.09524 (15)	−0.4100 (2)	0.0472 (5)
N1	0.0458 (2)	0.06307 (15)	−0.36653 (13)	0.0231 (4)
N2	−0.2044 (2)	0.02515 (15)	−0.37377 (13)	0.0237 (4)
C1	−0.0115 (2)	−0.1467 (2)	−0.39405 (14)	0.0222 (4)
C2	−0.1150 (3)	−0.2259 (2)	−0.4030 (2)	0.0260 (5)
C3	−0.0694 (3)	−0.3540 (2)	−0.4128 (2)	0.0298 (5)
C4	0.0771 (3)	−0.4026 (2)	−0.4142 (2)	0.0312 (5)
C5	0.3435 (3)	−0.3682 (2)	−0.4131 (2)	0.0331 (5)
C6	0.4483 (3)	−0.2886 (2)	−0.4094 (2)	0.0353 (6)
C7	0.4034 (3)	−0.1606 (2)	−0.4010 (2)	0.0322 (5)
C8	0.2531 (2)	−0.1140 (2)	−0.3949 (2)	0.0261 (5)
C9	0.1880 (3)	−0.3239 (2)	−0.4080 (2)	0.0276 (5)
C10	0.1421 (2)	−0.1945 (2)	−0.39816 (14)	0.0236 (4)
C11	0.2109 (3)	0.0207 (2)	−0.3912 (2)	0.0298 (5)
C12	−0.0619 (2)	−0.0139 (2)	−0.37832 (14)	0.0211 (4)
O1'	0.0752 (2)	0.2488 (2)	−0.3064 (2)	0.0472 (5)
C1'	−0.1636 (2)	0.2315 (2)	−0.33544 (15)	0.0247 (5)
C2'	−0.2568 (2)	0.1494 (2)	−0.35534 (14)	0.0231 (4)
C3'	−0.4093 (3)	0.1912 (2)	−0.3543 (2)	0.0272 (5)
C4'	−0.4657 (3)	0.3135 (2)	−0.3333 (2)	0.0319 (5)
C5'	−0.3736 (3)	0.3957 (2)	−0.3131 (2)	0.0334 (5)
C6'	−0.2240 (3)	0.3551 (2)	−0.3139 (2)	0.0299 (5)
C7'	−0.0058 (3)	0.1888 (2)	−0.3345 (2)	0.0283 (5)
O1a	0.4834 (2)	0.20825 (15)	0.21748 (13)	0.0377 (4)
N1a	0.2854 (2)	0.19098 (15)	0.15327 (13)	0.0248 (4)

$C_{19}H_{10}N_2O_2$ AND $C_{19}H_{12}N_2O_3$

N2a	0.0439 (2)	0.1451 (2)	0.13639 (13)	0.0276 (4)
C1a	0.2465 (3)	−0.0197 (2)	0.11641 (15)	0.0255 (5)
C2a	0.1590 (3)	−0.0994 (2)	0.0904 (2)	0.0308 (5)
C3a	0.2205 (3)	−0.2220 (2)	0.0652 (2)	0.0355 (6)
C4a	0.3686 (3)	−0.2649 (2)	0.0651 (2)	0.0347 (6)
C5a	0.6157 (3)	−0.2253 (2)	0.0927 (2)	0.0353 (6)
C6a	0.7014 (3)	−0.1473 (2)	0.1203 (2)	0.0381 (6)
C7a	0.6380 (3)	−0.0258 (2)	0.1485 (2)	0.0334 (5)
C8a	0.4902 (2)	0.0166 (2)	0.1458 (2)	0.0271 (5)
C9a	0.4620 (3)	−0.1862 (2)	0.0907 (2)	0.0303 (5)
C10a	0.3998 (3)	−0.0619 (2)	0.11701 (14)	0.0266 (5)
C11a	0.4253 (3)	0.1451 (2)	0.1760 (2)	0.0272 (5)
C12a	0.1846 (2)	0.1106 (2)	0.13731 (15)	0.0248 (5)
O1′a	0.3434 (2)	0.39510 (14)	0.14197 (14)	0.0421 (5)
C1′a	0.0861 (3)	0.3599 (2)	0.1510 (2)	0.0288 (5)
C2′a	−0.0110 (3)	0.2701 (2)	0.1477 (2)	0.0267 (5)
C3′a	−0.1666 (3)	0.3069 (2)	0.1524 (2)	0.0326 (5)
C4′a	−0.2218 (3)	0.4304 (2)	0.1602 (2)	0.0384 (6)
C5′a	−0.1252 (3)	0.5200 (2)	0.1640 (2)	0.0414 (6)
C6′a	0.0271 (3)	0.4850 (2)	0.1587 (2)	0.0362 (6)
C7′a	0.2471 (3)	0.3233 (2)	0.1487 (2)	0.0295 (5)

Table 2. Selected geometric parameters (Å, °) for (1)

O1—C11	1.221 (3)	O1a—C11a	1.169 (3)
N1—C12	1.401 (3)	N1a—C12a	1.399 (3)
N1—C7′	1.422 (2)	N1a—C11a	1.439 (3)
N1—C11	1.449 (3)	N1a—C7′a	1.446 (3)
N2—C12	1.296 (3)	N2a—C12a	1.293 (3)
N2—C2′	1.378 (2)	N2a—C2′a	1.385 (3)
C1—C12	1.460 (3)	C1a—C12a	1.460 (3)
C8—C11	1.467 (3)	C8a—C11a	1.464 (3)
O1′—C7′	1.188 (3)	O1′a—C7′a	1.216 (3)
C1′—C7′	1.458 (3)	C1′a—C7′a	1.459 (3)
C12—N1—C7′	119.5 (2)	C12a—N1a—C11a	121.4 (2)
C12—N1—C11	122.0 (2)	C12a—N1a—C7′a	119.4 (2)
C7′—N1—C11	118.4 (2)	C11a—N1a—C7′a	119.1 (2)
C12—N2—C2′	119.3 (2)	C12a—N2a—C2′a	119.0 (2)
C2—C1—C10	119.8 (2)	C2a—C1a—C10a	119.9 (2)
C2—C1—C12	119.7 (2)	C2a—C1a—C12a	119.3 (2)
C10—C1—C12	120.5 (2)	C10a—C1a—C12a	120.6 (2)
C7—C8—C10	120.6 (2)	C7a—C8a—C10a	120.9 (2)
C7—C8—C11	119.4 (2)	C7a—C8a—C11a	119.9 (2)
C10—C8—C11	119.9 (2)	C10a—C8a—C11a	119.2 (2)
C1—C10—C8	120.7 (2)	C8a—C10a—C1a	121.2 (2)
O1—C11—N1	120.3 (2)	O1a—C11a—N1a	120.9 (2)
O1—C11—C8	123.5 (2)	O1a—C11a—C8a	121.9 (2)
N1—C11—C8	116.3 (2)	N1a—C11a—C8a	117.3 (2)
N2—C12—N1	124.0 (2)	N2a—C12a—N1a	124.3 (2)
N2—C12—C1	118.1 (2)	N2a—C12a—C1a	118.7 (2)
N1—C12—C1	117.9 (2)	N1a—C12a—C1a	117.0 (2)
C2′—C1′—C7′	120.6 (2)	C2′a—C1′a—C7′a	120.3 (2)
C6′—C1′—C7′	120.5 (2)	C6′a—C1′a—C7′a	120.2 (2)
N2—C2′—C1′	121.2 (2)	N2a—C2′a—C1′a	121.5 (2)
N2—C2′—C3′	118.8 (2)	N2a—C2′a—C3′a	119.0 (2)
O1′—C7′—N1	120.3 (2)	O1′a—C7′a—N1a	121.7 (2)
O1′—C7′—C1′	125.2 (2)	O1′a—C7′a—C1′a	124.5 (2)
N1—C7′—C1′	114.4 (2)	N1a—C7′a—C1′a	113.8 (2)

Compound (2)

Crystal data

$C_{19}H_{12}N_2O_3$
$M_r = 316.31$
Monoclinic
$P2_1/c$
$a = 9.354$ (2) Å
$b = 14.130$ (3) Å
$c = 11.923$ (2) Å
$\beta = 111.28$ (3)°
$V = 1468.4$ (5) Å^3
$Z = 4$
$D_x = 1.431$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 24 reflections
$\theta = 10\text{–}11°$
$\mu = 0.099$ mm^{-1}
$T = 173$ (2) K
Prism
$0.5 \times 0.4 \times 0.35$ mm
Yellow

Data collection

Siemens P3 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5391 measured reflections
5132 independent reflections
3967 observed reflections
$[I > 2\sigma(I)]$
$R_{int} = 0.012$

$\theta_{max} = 35.06°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 22$
$l = -19 \rightarrow 17$
2 standard reflections
monitored every 98 reflections
intensity decay: 2.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0475$
$wR(F^2) = 0.1194$
$S = 1.049$
5126 reflections
265 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.4423P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.498$ e Å^{-3}
$\Delta\rho_{min} = -0.201$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (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}
O1	0.76656 (11)	−0.03540 (8)	0.29891 (9)	0.0333 (2)
O2	0.97954 (10)	0.13342 (7)	0.07635 (8)	0.0274 (2)
N1	0.87226 (10)	0.05097 (7)	0.18882 (8)	0.0187 (2)
C1	0.71021 (13)	0.15024 (8)	0.02347 (10)	0.0201 (2)
C2	0.69214 (15)	0.20842 (9)	−0.07381 (11)	0.0269 (2)
C3	0.5455 (2)	0.24371 (11)	−0.14309 (13)	0.0349 (3)
C4	0.4204 (2)	0.22262 (10)	−0.11415 (13)	0.0336 (3)
C5	0.30892 (14)	0.13750 (10)	0.01793 (13)	0.0312 (3)
C6	0.32633 (14)	0.07640 (10)	0.11088 (14)	0.0312 (3)
C7	0.47099 (14)	0.03727 (9)	0.17626 (12)	0.0261 (2)
C8	0.59680 (12)	0.06213 (8)	0.14816 (10)	0.0202 (2)
C9	0.43501 (13)	0.16293 (9)	−0.01500 (11)	0.0259 (2)
C10	0.58193 (12)	0.12545 (8)	0.05319 (10)	0.0200 (2)
C11	0.74773 (13)	0.02145 (8)	0.21783 (10)	0.0207 (2)
C12	0.86378 (12)	0.11319 (8)	0.09533 (10)	0.0191 (2)
O1′	0.97394 (13)	0.21800 (7)	0.32657 (9)	0.0356 (2)
N1′	1.06258 (14)	0.17028 (9)	0.52013 (10)	0.0295 (2)
C1′	1.10306 (12)	0.06917 (8)	0.37163 (10)	0.0196 (2)
C2′	1.02245 (12)	0.01878 (7)	0.26692 (9)	0.0186 (2)
C3′	1.07862 (14)	−0.06540 (9)	0.23987 (11)	0.0242 (2)
C4′	1.21895 (15)	−0.10010 (9)	0.31768 (12)	0.0276 (2)
C5′	1.30281 (14)	−0.04983 (9)	0.41986 (12)	0.0267 (2)
C6′	1.24509 (13)	0.03433 (9)	0.44686 (11)	0.0241 (2)
C7′	1.04014 (14)	0.15870 (8)	0.40362 (11)	0.0233 (2)

Table 4. Selected geometric parameters (Å, °) for (2)

O1—C11	1.2195 (14)	C1—C12	1.476 (2)
O2—C12	1.2179 (14)	C8—C11	1.470 (2)
N1—C11	1.3940 (14)	O1′—C7′	1.2315 (15)
N1—C12	1.3991 (14)	N1′—C7′	1.338 (2)
N1—C2′	1.4485 (14)	C1′—C7′	1.502 (2)
C11—N1—C12	125.09 (9)	N1—C11—C8	117.41 (10)
C11—N1—C2′	116.70 (9)	O2—C12—N1	119.89 (10)
C12—N1—C2′	118.09 (9)	O2—C12—C1	123.54 (10)
C2—C1—C10	120.12 (10)	N1—C12—C1	116.57 (10)
C2—C1—C12	119.70 (11)	C6′—C1′—C7′	120.22 (10)
C10—C1—C12	120.18 (10)	C2′—C1′—C7′	121.48 (10)
C7—C8—C10	120.80 (11)	C3′—C2′—N1	118.78 (10)
C7—C8—C11	119.44 (11)	C1′—C2′—N1	119.84 (10)
C10—C8—C11	119.76 (10)	O1′—C7′—N1′	122.58 (11)

C8—C10—C1	120.90 (10)	O1'—C7'—C1'	121.40 (11)
O1—C11—N1	120.03 (10)	N1'—C7'—C1'	116.02 (10)
O1—C11—C8	122.56 (11)		

Refinement was performed on F^2 for all reflections except for five for structure (1) and six for structure (2) with very negative F^2 or flagged for potential systematic errors (e.g. extinction).

For both compounds, program(s) used to solve structures: SHELXS86 (Sheldrick, 1985); program(s) used to refine structures: SHELXL93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: AB1270). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). J. Chem. Soc. Perkin Trans. 2, pp. S1–S19.
Easton, C. J., Gulbis, J. M., Hoskins, B. F., Scharfbillig, I. M. & Tiekink, E. R. T. (1992). Z. Kristallogr. 199, 249–254.
Ponomarev, I. I., Skuratova, N. A., Lindeman, S. V., Sinichkin, M. K., Vinogradova, S. V. & Rusanov, A. L. (1994). Vysokomol. Soedin. Ser. A, 36, 730–738.
Sheldrick, G. M. (1985). SHELXS86. Program for the Solution of Crystal Structures. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. Univ. of Göttingen, Germany.

Biphenyl-2-methanol

RAJNIKANT AND DAVID WATKIN

Chemical Crystallography Laboratory, University of Oxford, 9 Parks Road, Oxford OX1 3PD, England

GEORGE TRANTER

Department of Physical Sciences, The Wellcome Foundation Limited, Langley Court, South Eden Park Road, Kent BR3 3BS, England

Abstract

Biphenyl-2-methanol, $C_{13}H_{12}O$, crystallizes in the non-centrosymmetric monoclinic space group Pc with four molecules in the asymmetric unit. However, the four independent molecules fall into two pairs related by pseudo centres. The dihedral angle between the phenyl rings in each molecule is 53.3 (1), 58.9 (1), 52.6 (1) and 59.6 (1)°; the dihedral angle between the methanol and

phenyl group is 45.2 (1), 42.2 (1), 54.9 (1) and 45.4 (1)°, respectively.

Comment

Biphenyl and its derivatives have been studied extensively in the past because of the differences found in the inter-ring torsion angle φ in the solid state (Charbonneau & Delugeard, 1976, 1977; Brock, Blackburn & Haller, 1984; Brock & Haller, 1984a,b; Samdal, 1985; Brock & Minton, 1989) and in the gas phase (Almenningen & Bastiansen, 1958; Bastiansen & Traetteberg, 1962). These systems have also been investigated for those structures which pose difficulties in refinement because of a low observed-data-to-parameter ratio (a result of the crystals being non-centrosymmetric, having large overall displacement parameters and growing as thin needles or plates) (Brock, Blackburn & Haller, 1984). In a continuation of our on-going research program aimed at investigating the trends in crystallization and crystal growth of some substituted biphenyls from non-aqueous solutions (Rajnikant, Watkin & Tranter, 1995a,b,c), the crystal and molecular structure of the title compound, (I), is presented.

(I)
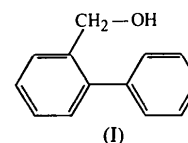
A general view of the molecule indicating the atom-numbering scheme is shown in Fig. 1 and the packing of the molecules viewed along the a axis is depicted in Fig. 2. The four independent molecules in the asymmetric unit are essentially identical except for the torsion angles. The average length of the bond C(n01)—C(n07) [1.454 (6) Å] is slightly less than the standard value for a single bond length between trigonally linked C atoms (1.477 Å; Cruickshank & Sparks, 1960), but is significantly shorter than the value observed
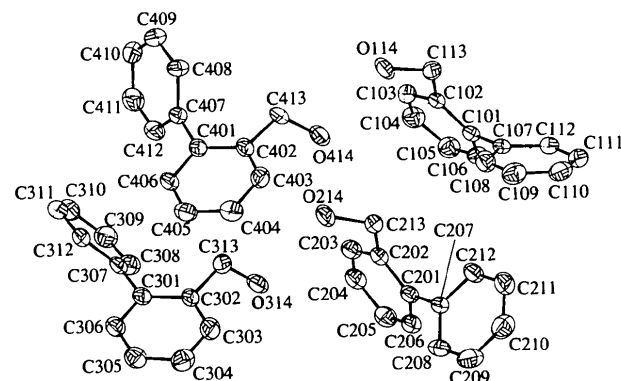

Fig. 1. Minimum overlap view of the four independent molecules in the asymmetric unit of biphenyl-2-methanol. Displacement ellipsoids are drawn at the 50% probability level.