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Bilayered structure of *N*,*N*′-diphenyl-4,4′-biphthalimide

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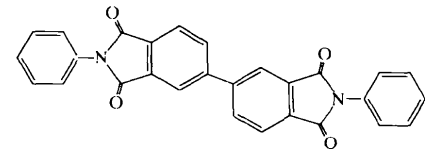
Abstract

The molecular and crystal structure of the title compound ($C_{28}H_{16}N_2O_4$), which corresponds to the monomer unit of the thermally stable polyimide, has been determined. The molecule is composed of a central phthalimide plane with phenyl ring planes at both ends twisted by 61.24 (8)°. The repeating unit along the *c* axis consists of a two-layer structure, in which the molecules tilt in opposite directions in adjacent layers.

Comment

Thermally stable polyimide, obtained by condensation polymerization of 3,3′,4,4′-biphenyltetracarboxylic dianhydride and bis(4-aminophenyl) ether, is one of the commercially available polymers that have several industrial applications. The title compound, (I), was synthesized using aniline instead of bis(4-aminophenyl) ether to investigate the stereochemistry and the physical properties of the chemical repeating unit of this polyimide.



(I)

Compound (I) has an inversion center in its chemical structure which coincides with the crystallographic inversion center. The molecule consists of a phthalimide plane at the center, with phenyl rings at both ends. The phthalimide plane, which is defined by N1, C7, C8, C9, C10, C11, C12, C13 and C14, has good planarity with a maximum deviation of 0.027 (2) Å. The phenyl ring plane is twisted from the phthalimide plane by 61.24 (8)°. The molecules are packed in a bilayered structure in which they tilt in opposite directions in adjacent layers. The carbonyl-O atoms have several short contacts with C atoms in adjacent molecules [O1···C6 3.234 (3) and O2···C10 3.299 (2) Å]. The distances between the O atoms and the H atoms attached to C6 and C10 are shorter than the van der Waals contacts, which suggests C—H···O hydrogen bonding.

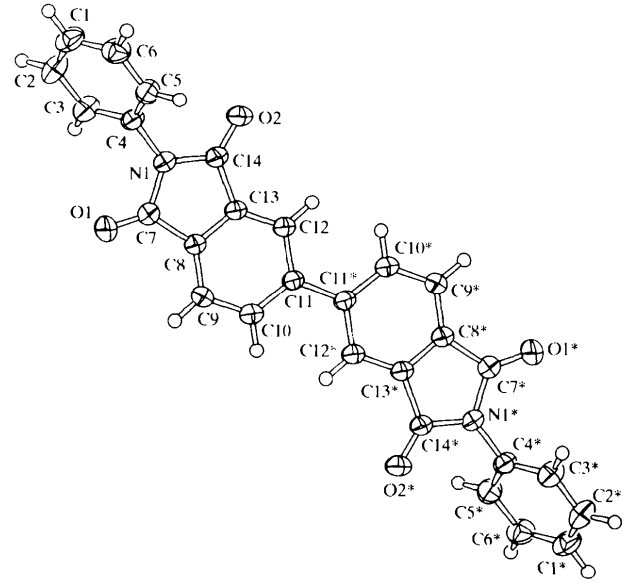


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title molecule with the atomic numbering scheme and non-H atoms represented by 50% probability displacement ellipsoids. Symmetrically generated atoms are denoted by *.

Experimental

The title compound was synthesized from 3,3′,4,4′-biphenyltetracarboxylic dianhydride and aniline. Single crystals used for X-ray diffraction were colorless and prismatic, and were obtained by the slow sublimation of a powdered sample in

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a glass tube oven at about 543 K under reduced pressure at 2 mmHg.

Crystal data

$C_{28}H_{16}N_2O_4$
M_r = 444.45
Monoclinic
$P2_1/c$
a = 8.3987 (15) Å
b = 6.890 (4) Å
c = 17.8585 (15) Å
β = 90.868 (11)°
V = 1033.3 (7) Å^3
Z = 2
D_x = 1.428 Mg m^{-3}
D_m = 1.425 Mg m^{-3}
D_m measured by flotation in aqueous KI
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 21.9–24.8°
μ = 0.097 mm^{-1}
T = 296.2 K
Prismatic
0.60 × 0.40 × 0.30 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: none
3608 measured reflections
3273 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 30.0°
h = 0 → 11
k = 0 → 9
l = −24 → 24
3 standard reflections
every 100 reflections
intensity decay: 0.14%

Refinement

Refinement on F
R = 0.049
wR = 0.067
S = 1.145
1833 reflections
154 parameters
H-atom: see text
$w = 1/[\sigma^2(F_o) + 0.0009|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C7	1.196 (2)	C5—C6	1.385 (3)
O2—C14	1.205 (2)	C7—C8	1.480 (3)
N1—C4	1.434 (2)	C8—C9	1.377 (2)
N1—C7	1.409 (2)	C8—C13	1.384 (3)
N1—C14	1.396 (3)	C9—C10	1.384 (3)
C1—C2	1.369 (3)	C10—C11	1.401 (3)
C1—C6	1.378 (3)	C11—C11^i	1.488 (3)
C2—C3	1.382 (3)	C11—C12	1.405 (2)
C3—C4	1.376 (3)	C12—C13	1.371 (3)
C4—C5	1.383 (3)	C13—C14	1.486 (2)
C4—N1—C7	123.7 (2)	C7—C8—C13	108.7 (2)
C4—N1—C14	124.4 (2)	C9—C8—C13	120.6 (2)
C7—N1—C14	111.8 (1)	C8—C9—C10	117.4 (2)
C2—C1—C6	120.3 (2)	C9—C10—C11	123.0 (2)
C1—C2—C3	120.6 (2)	C10—C11—C11^i	120.9 (2)
C2—C3—C4	119.0 (2)	C10—C11—C12	118.3 (2)
N1—C4—C3	119.8 (2)	C11^i—C11—C12	120.9 (2)
N1—C4—C5	119.1 (2)	C11—C12—C13	118.3 (2)
C3—C4—C5	121.1 (2)	C8—C13—C12	122.5 (2)
C4—C5—C6	119.1 (2)	C8—C13—C14	108.3 (2)
C1—C6—C5	119.9 (2)	C12—C13—C14	129.2 (2)
O1—C7—N1	125.0 (2)	O2—C14—N1	125.5 (2)
O1—C7—C8	129.5 (2)	O2—C14—C13	128.7 (2)
N1—C7—C8	105.5 (2)	N1—C14—C13	105.8 (2)
C7—C8—C9	130.8 (2)		

Symmetry code: (i) 1 − x, −y, 1 − z.

H atoms were fixed geometrically and were not included in the refinement procedure.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1075). Services for accessing these data are described at the back of the journal.

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Equilin

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Abstract

3-Hydroxyestra-1,3,5(10),7-tetraen-17-one, $C_{18}H_{20}O_2$, crystallizes in space group $P2_12_12_1$ from ethyl acetate. The planarity of the *B* ring, and the difference in puckering of the *C* and *D* rings from that of estrone, are due to the presence of the C7=C8 double bond, which may explain its function as an inhibitor of human type 1 17β-hydroxysteroid dehydrogenase, instead of being its substrate.

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