

Structure of 2,6-Dicyclohexylnaphthalene

By P. MOREAU, J. SOLOFO, P. GENESTE AND A. FINIELS

URA 418 – CNRS Laboratoire de Chimie Organique Physique et Cinétique Chimique Appliquées,
ENSCM 8, Rue de l'Ecole Normale, 34053 Montpellier CEDEX 1, France.

J. RAMBAUD

Chimie Physique Générale, Faculté de Pharmacie, 34060 Montpellier CEDEX, France

AND J.-P. DECLERCQ

Laboratoire de Chimie Physique et de Cristallographie, Université Catholique de Louvain, 1,
Place Louis Pasteur, 1348, Louvain-La-Neuve, Belgium

(Received 12 March 1991; accepted 2 August 1991)

Abstract. C₂₂H₂₈, *M_r* = 292.4, monoclinic, *C*2/*c*, *a* = 25.691 (4), *b* = 5.663 (1), *c* = 13.279 (2) Å, β = 113.83 (1)°, *V* = 1767.3 (4) Å³, *Z* = 4, *D_x* = 1.06 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.63 mm⁻¹, *F*(000) = 640, *T* = 293 K, *R* = 0.048 for 1137 observed reflections. The 2,6-dicyclohexylnaphthalene contains a crystallographic symmetry centre. The presence of two bulky substituents involves only slight deviation from flatness for the naphthalene ring. The cyclohexyl substituents adopt a chair conformation.

Experimental. The title compound was prepared by the cyclohexylation reaction of naphthalene over zeolites (Solofo, Moreau, Geneste & Finiels, 1990). Single crystals were obtained by recrystallization from ethanol at room temperature, colorless thin plates 0.6 × 0.3 × 0.2 mm, lattice parameters refined using 30 reflections in the range 7 ≤ 2θ ≤ 25°. *D_m* not measured. Huber 424 + 511 diffractometer, graphite-monochromatized Mo *K*α radiation. The asymmetric unit is comprised of 1725 independent reflections to (sinθ)/λ < 0.617 Å⁻¹. 1137 were observed with *I* ≥ 2.5σ(*I*). Index range: *h* - 30 to 28, *k* 0 to 7, *l* 0 to 16, standard reflection (400) checked every 50 reflections: no significant deviation. No absorption correction. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The asymmetric unit is comprised of half a molecule because of the presence of a crystallographic symmetry centre. H atoms from difference Fourier synthesis. Anisotropic least-squares refinement on *F* with *SHELX76* (Sheldrick, 1976). H isotropic. *R* = 0.048, *wR* = 0.061, *S* = 0.85 for 1137 observed reflections, *w* = 1/[σ²(*F*) + 0.007701*F*²]. Final (Δ/σ)_{max} = 0.051. Maximum and minimum heights in final difference Fourier synthesis

Table 1. Fractional atomic coordinates (× 10⁵) with *e.s.d.*'s in parentheses and equivalent isotropic temperature factors (Å²)

$$B_{eq} = 8/3\pi^2 \sum_i U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
C(1')	89024 (8)	4074 (37)	14790 (16)	4.31
C(2')	89249 (11)	25787 (43)	21716 (20)	5.33
C(3')	85391 (11)	23409 (51)	27836 (21)	5.96
C(4')	79358 (11)	17567 (55)	20160 (25)	6.54
C(5')	79105 (14)	-4118 (64)	13313 (31)	7.70
C(6')	82929 (10)	-1573 (55)	7105 (22)	6.20
C(1)	96763 (7)	-10978 (31)	9470 (14)	3.91
C(2)	92831 (7)	5994 (32)	8544 (14)	3.84
C(3)	92250 (9)	25254 (34)	1304 (16)	4.50
C(4)	95555 (9)	27007 (34)	-4508 (16)	4.43
C(8a)	100299 (7)	-9702 (30)	3577 (13)	3.64

0.14 and -0.16 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Atomic coordinates are listed in Table 1.* Bond lengths and bond angles are listed in Table 2. Fig. 1 shows that the cyclohexyl substituent has a chair conformation. The presence of the cyclohexyl substituents on positions 2 and 6 has only a very small effect upon the flatness of the naphthalenic ring, as shown by the study of the deviations of all the atoms from plane C(4)-C(4a)-C(8a) (Table 3) (*XANADU*; Roberts & Sheldrick, 1975).

Related literature. Bond lengths for the naphthalene ring in 2,6-dicyclohexylnaphthalene are in agreement

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54482 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses

C(2)—C(1')	1.523 (3)	C(2)—C(1)	1.364 (3)
C(6')—C(1')	1.518 (3)	C(8a)—C(1)	1.420 (2)
C(2)—C(1')	1.520 (2)	C(3)—C(2)	1.421 (3)
C(3')—C(2')	1.520 (2)	C(4)—C(3)	1.362 (3)
C(4')—C(3')	1.509 (4)	C(8a)—C(4a)	1.420 (3)
C(5')—C(4')	1.514 (4)	C(4a)—C(4)	1.416 (3)
C(6')—C(5')	1.523 (3)		
C(6')—C(1')—C(2')	110.7 (2)	C(8a)—C(1)—C(2)	122.0 (2)
C(2)—C(1')—C(2')	113.3 (2)	C(1)—C(2)—C(1')	121.1 (2)
C(2)—C(1')—C(6')	111.4 (2)	C(3)—C(2)—C(1')	120.6 (2)
C(3')—C(2')—C(1')	112.2 (2)	C(3)—C(2)—C(1)	118.3 (2)
C(4')—C(3')—C(2')	111.8 (2)	C(4)—C(3)—C(2)	121.3 (2)
C(5')—C(4')—C(3')	111.5 (2)	C(3)—C(4)—C(4a)	121.2 (2)
C(6')—C(5')—C(4')	111.6 (2)	C(1)—C(8a)—C(4a)	119.2 (2)
C(5')—C(6')—C(1')	111.8 (2)	C(8a)—C(4a)—C(4)	117.9 (2)

Table 3. Deviation from plane C(4)—C(4a)—C(8a)

Equation of plane: (0.6797)x _D + (0.5117)y _D + (0.5256)z _D = 10.5273			
Deviation from plane		Deviation from plane	
C(1')	-0.0052	C(4)*	-0.0000
C(2)	1.1311	C(8a)*	-0.0000
C(3')	1.0831	C(4a)*	-0.0000
C(4')	-0.2570	C(8)	-0.0000
	-1.3898	C(5)	-0.0021
C(6')	-1.3468	C(7)	0.0069
C(1)	0.0021	H(8)	-0.0221
C(2)	0.0153	C(6)	-0.0153
C(3)	0.0069	H(5)	-0.0254
		H(7)	-0.0187
		C(1'')	0.0052

hexane molecule by Davis & Hassel (1963), except the C(4')—C(3') and C(5')—C(4') bonds and the C(6')—C(1')—C(2') angle which are slightly modified.

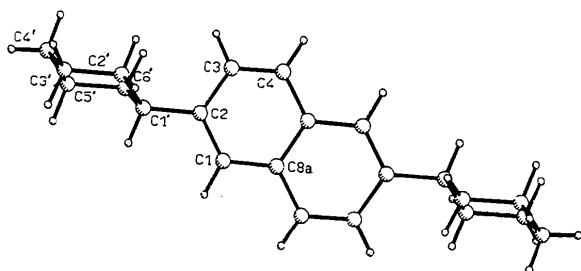


Fig. 1. Projection on the naphthalenic plane showing the numbering of atoms (PLUTO; Motherwell & Clegg, 1978).

with those found by Cruickshank (1957) for the naphthalene molecule, whereas some small differences appear in the angle values. In the same way, all the bond lengths and angles of the cyclohexyl ring fall within the range of values obtained in the cyclo-

References

- CRUICKSHANK, D. W. J. (1957). *Acta Cryst.* **10**, 504–508.
 DAVIS, M. & HASSEL, O. (1963). *Acta Chem. Scand.* **17**, 1181.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System for Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 ROBERT, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SOLOFO, J., MOREAU, P., GENESTE, P. & FINIELS, A. (1990). Demande Internationale: PCT/CH90/00178.

Acta Cryst. (1992). **C48**, 398–400

Absolute Configuration of (S)_p-O-Menthoxy-N-anilinophenylthiophosphonate*

BY ANNA E. KOZIOŁ

Department of Chemistry, Maria Curie-Skłodowska University, pl. Marii Curie-Skłodowskiej 3,
20-031 Lublin, Poland

AND KINGA SUWIŃSKA

Institute of Physical Chemistry, Polish Academy of Sciences, ul. Kasprzaka 44/52, 01-224 Warszawa, Poland

(Received 29 May 1991; accepted 20 August 1991)

Abstract. C₂₂H₃₀NOPS, *M_r* = 387.53, orthorhombic, *P*2₁2₁2₁, *a* = 6.0211 (9), *b* = 8.6175 (8), *c* =

43.095 (5) Å, *V* = 2236.0 (5) Å³, *Z* = 4, *D_x* = 1.15, *D_m* = 1.146 g cm⁻³ (floatation in chlorobenzene/dichloroethane mixture), λ(Cu Kα) = 1.54178 Å, μ = 19.2 cm⁻¹, *F*(000) = 832, *T* = 293 K, *R* = 0.069 and *wR* = 0.064 for 3860 reflections collected on a

* Alternative nomenclature: (S)_p-O-2-menthyl (anilino)-(phenyl)thiophosphinate.