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1,4-Diphenylbutane

Michel Fleck[a]* and Martin Walter[b]

[a]Universität Wien – Geozentrum, Institut für Mineralogie und Kristallographie, Althanstrasse 14, A-1090 Wien, Austria, and [b]Universität Wien – Pharmaziezentrum, Department für Medizinische-Pharmazeutische Chemie, Althanstrasse 14, A-1090 Wien, Austria

Correspondence e-mail: michel.fleck@univie.ac.at

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.048
wR factor = 0.143
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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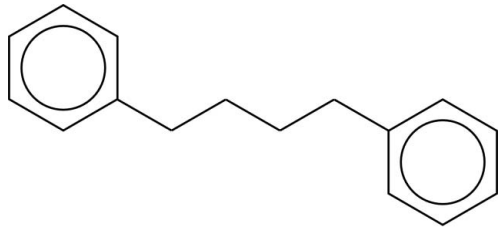
All molecule of the title compound, $C_{16}H_{18}$, centrosymmetric.

Comment

Although a simple molecule, the crystal structure of 1,4-diphenylbutane has not hitherto been described in the literature. Bonsma & Nowacki (1959) reported the crystal structure of 2,3-diphenylbutane. In that paper, they also presented the cell parameters of 1,4-diphenylbutane, but no structural analysis had been conducted.



(I)

1,4-Diphenylbutane, (I), was found as an impurity in the distilled raw product resulting from a reaction using lithium diisopropylamide (LDA) as reagent. As confirmed by the supplier, 1,4-diphenylbutane is an intrinsic side product of commercial LDA, arising from the production procedure which can be traced back to the experiments of Ziegler *et al.* (1934). In fact, lithiation of styrene and subsequent hydrolysis with diisopropylamine leads to LDA, ethylbenzene and, as dimerization product, 1,4-diphenylbutane.

Comparing the literature boiling point of 1,4-diphenylbutane [417–419 K at 4–5 Torr (Suga *et al.*, 1972)] and of fractions containing the desired product (428–430 K at 11 Torr), it is not surprising that separation by distillation is not successful. 1,4-Diphenylbutane was isolated from the distilled raw product by vacuum-flash chromatography over silica gel with light petroleum–ethyl acetate (20:1 *v*/*v*) as the eluting solvent. *n*-Hexane was used as solvent for the crystallization of suitable crystals.

The molecular geometry of 1,4-diphenylbutane does not exhibit any unusual chemical features. Aromatic C—C bond distances range from 1.365 (2) to 1.3814 (19) Å, while the aliphatic C—C bond distances are 1.5211 (17) and 1.520 (2) Å. C—H distances range from 0.946 (19) to 0.992 (17) Å (no constraints were applied in the refinement). The molecule is centrosymmetric; the mid-point of the central C—C bond is located on a centre of inversion. The butane chain is therefore in a perfect *trans* conformation [torsion angle C7—C8—C8^{i}—C7^{i} = 180°; symmetry code: (i) $2-x, -y, 1-z$].

Experimental

After aqueous work-up of an LDA reaction and concentration of the organic phase, the remainder was distilled at 428–430 K and 11 Torr. 1,4-Diphenylbutane was isolated from this crude product by vacuum-flash chromatography over silica gel with light petroleum–ethyl acetate (20:1 *v*/*v*) as the eluting solvent. Crystals were obtained by dissolving diphenylbutane in *n*-hexane and allowing the solvent to evaporate slowly at normal pressure. The synthesis yielded small colourless crystals up to a size of 1 mm.

Crystal data

$C_{16}H_{18}$
M_r = 210.30
Monoclinic, $P2_1/c$
a = 5.787 (1) Å
b = 8.776 (2) Å
c = 12.617 (3) Å
β = 91.25 (3)°
V = 640.6 (2) Å^3
Z = 2
D_x = 1.090 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1288 reflections
θ = 26.6–1288°
μ = 0.06 mm^{-1}
T = 293 (2) K
Prism, colourless
0.10 × 0.06 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO*; Otwinowski & Minor, 1997)
T_{min} = 0.994, T_{max} = 0.997
2835 measured reflections
1464 independent reflections
1139 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 27.5°
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.143
S = 1.07
1464 reflections
110 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.0552P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.09 (3)

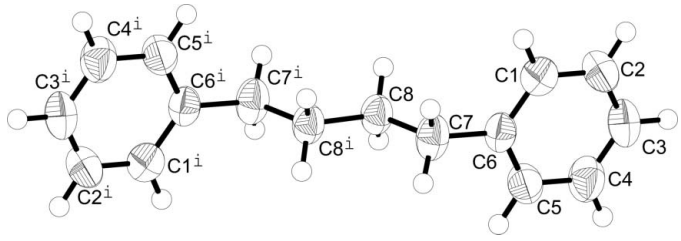


Figure 1
The molecular structure of (I), shown with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $2 - x, -y, 1 - z$.]

Data collection: *COLLECT* (Nonius, 2003); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Version 2.1; Bergerhoff *et al.*, 1997); software used to prepare material for publication: *SHELXL97*.

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