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Powder diffraction study of 1,2:3,4-dibenzanthracene

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Key indicators

Powder X-ray study
T = 295 K
Mean σ(C–C) = 0.092 Å
R factor = 0.036
wR factor = 0.036
Data-to-parameter ratio = 3.1

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

The crystal structure of 1,2:3,4-dibenzanthracene, $C_{22}H_{14}$, was solved by simulated annealing from laboratory X-ray powder diffraction data collected at room temperature to 1.8 Å resolution. Subsequent Rietveld refinement yielded an R_{wp} value of 0.036. The molecules crystallize in space group $P2_1$ with two independent molecules in the asymmetric unit which pack in a stacked arrangement along the b axis.

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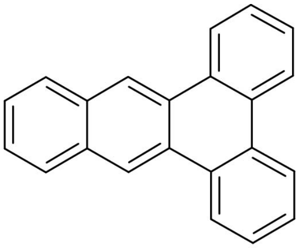
Comment

The title compound, (I), was used as supplied and its crystal structure was solved by simulated annealing using laboratory X-ray powder diffraction data (Fig. 1). The compound crystallizes in space group $P2_1$ with two independent molecules in the asymmetric unit (Fig. 2).



(I)

The crystal packing adopts a γ-type structure, with molecules stacked in the direction of the b axis (Desiraju & Gavezzotti, 1989). The distance between the centres of mass of neighbouring molecules within each stack (R_n) equals the shortest cell axis, 5.062 Å, and the perpendicular distance between the molecular planes within each stack (R_{ip}) is 3.740 Å, with an offset angle α = 43° (Fig. 3).

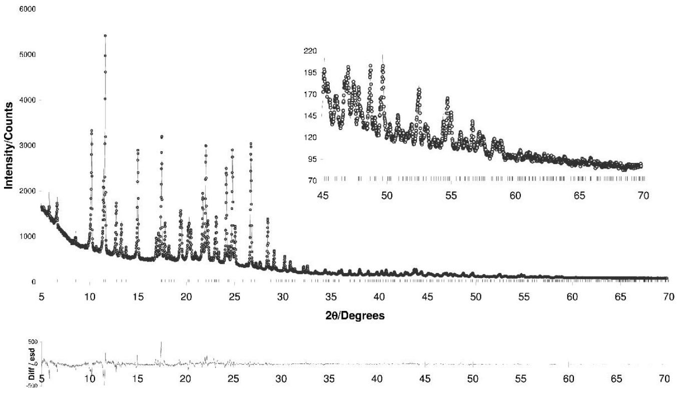


Figure 1
Final observed (points), calculated (line) and difference $[(y_{obs} - y_{calc})/\sigma(y_{obs})]$ profiles for the Rietveld refinement of (I).

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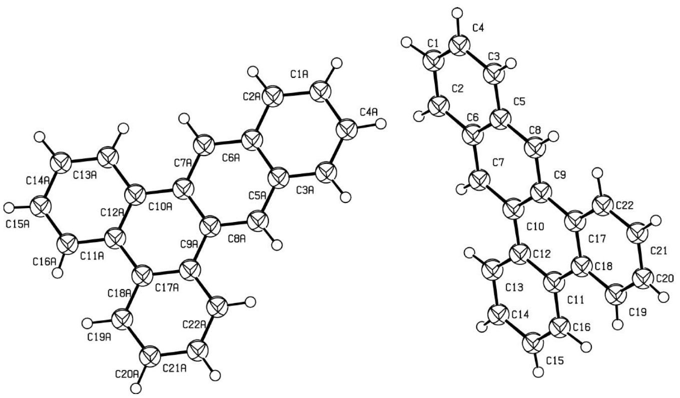

Figure 2
The atomic arrangement in (I), showing the two molecules in the asymmetric unit. The dihedral angle between the least-squares planes through each of the molecules is 47.8 (8)°. Isotropic displacement spheres are shown at the 50% probability level.

Experimental

1,2:3,4-Dibenzanthracene (Sigma–Aldrich) was lightly ground in a mortar, loaded into a 0.7 mm borosilicate glass capillary and mounted on the diffractometer. Data were collected from a sample in a rotating 0.7 mm borosilicate glass capillary using a variable count time scheme (Hill & Madsen, 2002).

Crystal data

$C_{22}H_{14}$
$M_r = 278.33$
Monoclinic, $P2_1$
$a = 18.2966\ (5)$ Å
$b = 5.06225\ (10)$ Å
$c = 15.7245\ (4)$ Å
$\beta = 104.5574\ (15)°$
$V = 1409.68\ (6)$ Å^3
$Z = 4$
$D_x = 1.311$ Mg m^{-3}
Cu $K\alpha_1$ radiation
$\mu = 0.56$ mm^{-1}
$T = 295$ K
Specimen shape: cylinder
12 × 0.7 mm
Specimen prepared at 295 K
Particle morphology: visual estimate, flat plate, pale yellow

Data collection

Bruker D8 Advance diffractometer
Specimen mounting: 0.7 mm borosilicate capillary
Specimen mounted in transmission mode
Scan method: step
Absorption correction: none
$2\theta_{\min} = 4$, $2\theta_{\max} = 69.8°$
Increment in $2\theta = 0.014°$
$h = -13 \rightarrow 13$
$k = -3 \rightarrow 3$
$l = -9 \rightarrow 11$

Refinement

Refinement on F^2
$R_p = 0.036$
$R_{wp} = 0.036$
$R_{exp} = 0.014$
$S = 2.69$
Increment in $2\theta = 0.014°$
Wavelength of incident radiation: 1.54056 Å
Profile function: fundamental parameters with axial divergence correction
739 reflections
241 parameters
Only H-atom coordinates refined
$w = 1/\sigma(y_{obs})^2$
$(\Delta/\sigma)_{\max} = 0.011$
Preferred orientation correction: a spherical harmonics-based preferred orientation correction was applied with *TOPAS* during the Rietveld refinement.

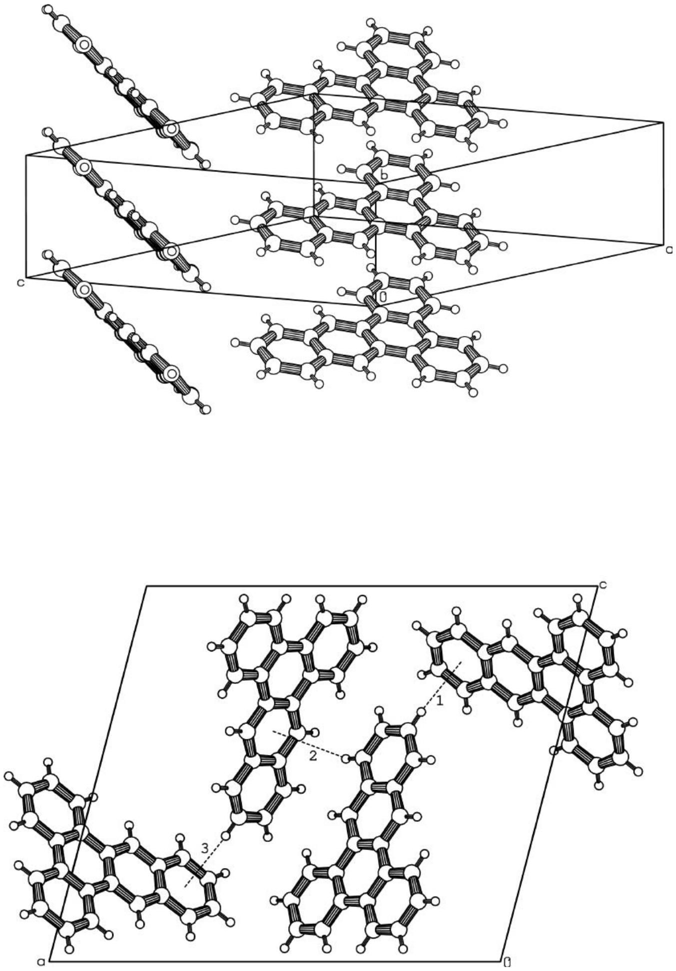

Figure 3
Top: view showing the molecular stacking along the *b* axis in (I) for both unique molecules. Molecules within each stack form offset face-to-face attractive contacts (Hunter *et al.*, 1990). Bottom: view down the *b* axis onto the *ac* plane. The crystal packing arrangement is stabilized by a series of C—H$\cdots\pi$ contacts between adjacent stacks, with H$\cdots$ring-centroid (*Cg*) distances in the range 2.9 (5)–3.3 (5) Å. Dashed lines represent three of these contacts. (1) C4*A*—H26*A*$\cdots$*Cg* of the ring C1–C6 in the molecule at (x, $1 + y$, z) [H26*A*$\cdots$*Cg* 2.9 (5) Å], (2) C2*A*—H24*A*$\cdots$*Cg* of the ring C5*A*–C10*A* in the molecule at ($1 - x, \frac{1}{2} + y, 1 - z$) [H24*A*$\cdots$*Cg* 3.2 (6) Å] and (3) the symmetry equivalent of (1), where H26*A* and *Cg* are in the molecules at ($1 - x, \frac{1}{2} + y, 1 - z$).

The diffraction pattern indexed to a monoclinic cell [$F(25) = 210.1$, $M(25) = 71.8$; *DICVOL-91* (Boultif & Louer, 1991)], and space group $P2_1$ was assigned from volume considerations and a statistical consideration of the systematic absences. The data set was background-subtracted and truncated to $2\theta = 51.9°$ for Pawley fitting (Pawley, 1981; $\chi^2_{Pawley} = 3.96$), and the structure was solved using the simulated annealing (SA) global optimization procedure of David *et al.* (1998), as implemented in the *DASH* computer program (David *et al.*, 2001). The SA structure solution involved the optimization of two independent fragments in the asymmetric unit, totalling 12 degrees of freedom. The best SA solution had a favourable $\chi^2_{SA}/\chi^2_{Pawley}$ ratio of 4.53 and a chemically reasonable packing arrangement, and exhibited no significant misfit to the data. The solved structure was then refined with the full data set (2θ 4–69.8°) using a restrained Rietveld method (Rietveld, 1969), as implemented in *TOPAS* (Coelho, 2003), with the value of R_{wp} falling from 0.146 to 0.036 during the refinement. The *y* coordinate of atom C1 was fixed and all remaining atomic positions (including H atoms) were refined, subject to a series of restraints on bond lengths, bond angles and planarity. Inclusion of a March–Dollase (Dollase, 1986) preferred orientation correction indicated the presence of mild (1.16) preferred orientation along the [010] direction, and a spherical harmonics correction of intensities for preferred orientation was applied in the final refinement. The observed and calculated diffraction patterns for the refined crystal structure are shown in Fig. 1.

Data collection: *DIFFRAC* plus *XRD Commander* (Kienle & Jacob, 2003); cell refinement: *TOPAS* (Coelho, 2003); data reduction: *DASH* (David *et al.*, 2001); structure solution: *DASH*; structure refinement: *TOPAS*; molecular graphics: *PLATON* (Spek, 2003); publication software: *enCIFer* (Allen *et al.*, 2004).

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