

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1077). Services for accessing these data are described at the back of the journal.

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## 2-tert-Butyl-9,10-dibromoanthracene

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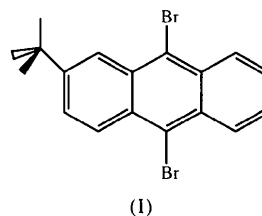
## Abstract

The title compound, C<sub>18</sub>H<sub>16</sub>Br<sub>2</sub>, crystallized on a crystallographic mirror plane. There was disorder of the 'Bu group with a major orientation at 59%. Interplanar spacings are 3.509 (4) Å.

## Comment

The title compound, (I), was structurally characterized in order to measure interplanar distances for studies aimed at assessing the forces of attraction in this molecule, and to make comparisons with other molecules such as anthracene (Lehmann & Pawley, 1972) and 9,10-dibromoanthracene (Kitaigorodski, 1946; Trotter, 1958, 1986; Bulgarovskaya *et al.*, 1987) of a similar nature. We find that the interplanar spacing in (I) of 3.509 (4) Å is similar to that in 9,10-dibromoanthracene of 3.52 (1) Å (Trotter, 1986). It is also interesting to note that the packing arrangement in 9,10-dibromoanthracene, where the molecules are stacked on top of each other, is very different from that observed in (I). Clearly, the 'Bu group is responsible for these differences. Additionally, the closest Br···Br intermolecular distance of 4.004 (2) Å in (I) is smaller than that of 4.068 (1) Å in 9,10-dibromoanthracene (Trotter, 1968). It is likely that the skewed arrangement of the rings in (I) which

reduces Br–Br repulsions, also results in a decrease in the interplanar spacings in (I).



Molecules of (I) in the crystal are arranged in a herring-bone manner parallel to the *bc* plane and perpendicular to the *a* axis. The angle for the intramolecular Br···Br vectors situated between adjacent coplanar rows is 90.9(1)°. Adjacent pairs of molecules are skewed, with 'Bu groups on opposite sides and displaced by one ring so that only the two fused rings furthest from the 'Bu group are aligned on top of each other. The bond distance and angles within the anthracene rings in (I) and 9,10-dibromoanthracene are similar.

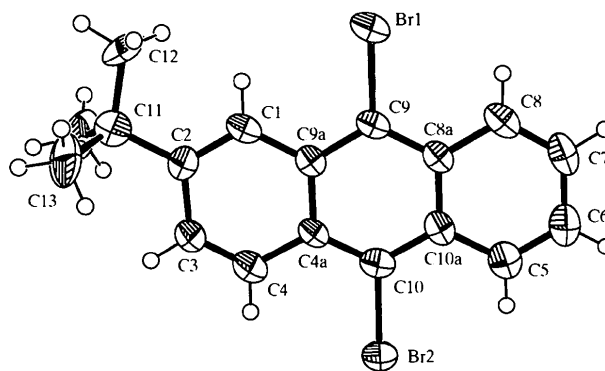


Fig. 1. View of the major contributor to (I) (50% probability displacement ellipsoids). H atoms, represented by circles of arbitrary radii, are not labeled.

## Experimental

Crystals of (I) prepared as detailed previously (Lee & Mendenhall, 1988) were obtained by slow crystallization from an ethanol solution.

### Crystal data

C<sub>18</sub>H<sub>16</sub>Br<sub>2</sub>  
*M<sub>r</sub>* = 392.14  
 Orthorhombic  
*Pnma*  
*a* = 23.433 (5) Å  
*b* = 7.018 (4) Å  
*c* = 9.554 (4) Å  
*V* = 1571.2 (12) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.658 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
*λ* = 0.71069 Å  
 Cell parameters from 25 reflections  
*θ* = 10.4–15.0°  
*μ* = 5.15 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism  
 0.22 × 0.20 × 0.08 mm  
 Yellow

**Data collection**

Enraf–Nonius CAD-4 diffractometer	1183 reflections with $I > 2\sigma(I)$
Non-profiled $\omega$ scans	$\theta_{\max} = 27.47^\circ$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 30$ $k = 0 \rightarrow 9$ $l = 0 \rightarrow 12$
$T_{\min} = 0.304$ , $T_{\max} = 0.674$	3 standard reflections
1930 measured reflections	frequency: 60 min
1930 independent reflections	intensity decay: 2%

**Refinement**

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.002$
$R(F) = 0.040$	$\Delta\rho_{\max} = 0.73 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.112$	$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$
$S = 1.028$	Extinction correction: <i>SHELXL97</i> (Sheldrick, 1997)
1930 reflections	Extinction coefficient: 0.0006 (4)
135 parameters	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
H atoms constrained	
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.26P]$	
where $P = [\text{Max}(F_o^2, 0) + 2F_c^2]/3$	

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Br1—C9	1.905 (5)	C8a—C9	1.412 (7)
Br2—C10	1.903 (5)	C9—C9a	1.401 (7)
C4a—C10	1.392 (7)	C10—C10a	1.409 (7)
C9a—C9—Br1	118.2 (4)	C4a—C10—Br2	118.6 (4)
C8a—C9—Br1	119.0 (4)	C10a—C10—Br2	118.8 (4)

A difference Fourier map clearly indicated two regions of electron density suggestive of disorder for with 'Bu group. C atoms were inserted and the sum of occupancies of the relevant C atoms were constrained to unity and then refinement continued to convergence both on positions and occupancies.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *XCAD4* (Harms & Wocadlo, 1995). Program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997). Software used to prepare material for publication: *WinGX* (Farrugia, 1998).

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## The *tert*-butylammonium perchlorate complex of 2,3-naphtho-18-crown-6 at 115 K

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**Abstract**

In the title complex, *tert*-butylammonium perchlorate–2,5,8,11,14,17-hexaoxatricyclo[16.8.0<sup>20,25</sup>]hexacosal(26),18,20(25),21,23-pentaene–ethyl acetate–dichloromethane (4/4/1/1),  $\text{C}_4\text{H}_{12}\text{N}^+ \cdot \text{C}_{20}\text{H}_{26}\text{O}_6 \cdot \text{ClO}_4^- \cdot 0.25\text{C}_4\text{H}_8\text{O}_2 \cdot 0.25\text{CH}_2\text{Cl}_2$ , the *tert*-butylammonium cation binds to the macrocyclic host (Chemical Abstracts name: 2,3,5,6,8,9,11,12,14,15-decahydro-1,4,7,10,13,16-hexaoxanaphtho[2,3-*b*]cyclooctadecin) in the expected tripod arrangement, while the perchlorate anion links naphthyl groups in the crystal through  $\text{C}—\text{H} \cdots \text{O}—\text{Cl}—\text{O} \cdots \text{H}—\text{C}$  interactions. Thermal motion analysis indicates that the *tert*-butylammonium group and the perchlorate anion each librate with respect to the host, with amplitudes of 6.2 (4) and 11.4 (2) $^\circ$ , respectively.

**Comment**

The host in the title compound, (I), has been used as a standard in solution studies of structures of hosts and their complexing affinities; see, for example, Kyba *et al.* (1977) and Helgeson *et al.* (1979). The *tert*-butyl-

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