

4,7-Dibromobenzo[*c*]furanan 1-oxide–
pyrene (1/1)

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Key indicators

Single-crystal X-ray study

 $T = 173\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.011\text{ \AA}$ R factor = 0.051 wR factor = 0.127

Data-to-parameter ratio = 16.3

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

The 1:1 complex of 4,7-dibromobenzo[*c*]furanan 1-oxide and pyrene, $\text{C}_6\text{H}_2\text{N}_2\text{O}_2\text{Br}_2 \cdot \text{C}_{16}\text{H}_{10}$, contains π stacks of alternating molecules. The molecules are tilted by $2.5(1)^\circ$ with respect to each other and by $22.6(1)$ and $25.0(1)^\circ$, respectively, away from being normal to the direction of the stack. The average intermolecular distances in the stack alternate between $3.45(2)$ and $3.49(2)\text{ \AA}$. There are no unusual contacts between adjacent stacks.

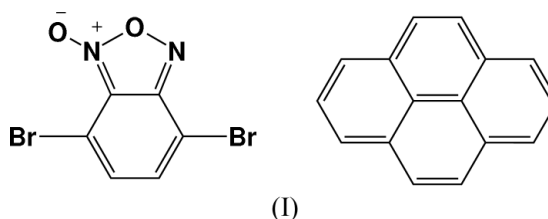
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Comment

In 4,7-dibromobenzo[*c*]furanan 1-oxide (47BF), the molecules are arranged in planar layers with no disorder in the N_2O_2 fragment (Britton *et al.*, 2002). In the 56BF isomer, the molecules are also arranged in planar layers but with complete disorder in the N_2O_2 fragment about a crystallographic twofold axis (Britton *et al.*, 2003). In the 1/1 complex of 56BF and pyrene, the two molecules alternate in π stacks, but the 56BF molecules also associate in chains and are completely ordered. The preparation and structure determination of the 47BF/pyrene complex, (I), were undertaken to see the effects of the expected π complexation on the 47BF–47BF interactions.



There is one molecule of each compound in the asymmetric unit. The anisotropic displacement ellipsoids and atom label-

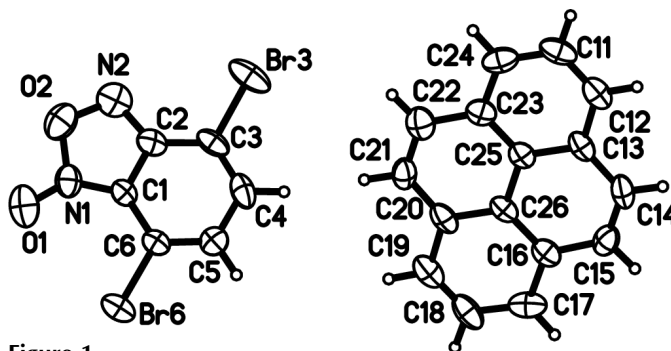


Figure 1

The $\text{C}_6\text{H}_2\text{N}_2\text{O}_2\text{Br}_2$ and $\text{C}_{16}\text{H}_{10}$ molecules in (I). The molecules are in the same orientation as the top layer in Fig. 2. Displacement ellipsoids are shown at the 50% probability level. H atoms are shown with arbitrary radii.

ling are shown in Fig. 1. Bond lengths and angles are normal, and chemically equivalent bonds in the pyrene agree within the rather large experimental error. The volume of the asymmetric unit at 173 K [457.0 (2) Å³] is slightly smaller than the sum of the molecular volumes of the components [459.4 (5) Å³] obtained from 47BF [200.1 (1) Å³; Britton *et al.*, 2002] and pyrene [259.3 (4) Å³; estimated from the values at 113 and 298 K (Kai *et al.*, 1978)].

The molecules stack in columns parallel to the *b* axis. Fig. 2 shows a view along the stacks. The 47BF molecules are tilted 22.6 (1)° and the pyrene molecules 25.0 (1)° away from being normal to the stack direction. Since the two rings are tilted 2.5 (1)° with respect to each other the distances between the C₆ rings and the C₁₆ of the pyrene are not well defined; they are alternately 3.45 (2) and 3.49 (2) Å. There are no significantly short intermolecular contacts.

Given the absence of short contacts involving the N and O atoms, it is surprising that there is no disorder in the N₂O₂ fragment. To confirm this, the structure was also determined at 297 K, where the disorder, if any, would be expected to be larger. Again there was no indication of disorder; the two determinations agreed well except for the larger anisotropic displacement ellipsoids at the higher temperature.

Experimental

The sample of 47BF was provided by Professor Frank B. Mallory. The complex was prepared by dissolving approximately equimolar amounts of the two components in acetone and allowing the solvent to evaporate.

Crystal data

C ₆ H ₂ Br ₂ N ₂ O ₂ ·C ₁₆ H ₁₀	$D_x = 1.803 \text{ Mg m}^{-3}$
$M_r = 496.16$	Mo $K\alpha$ radiation
Monoclinic, Pc	Cell parameters from 2896 reflections
$a = 7.851 (2) \text{ \AA}$	$\theta = 2.6\text{--}24.4^\circ$
$b = 7.657 (2) \text{ \AA}$	$\mu = 4.46 \text{ mm}^{-1}$
$c = 16.296 (4) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\beta = 111.09 (1)^\circ$	Irregular plate, yellow
$V = 914.0 (4) \text{ \AA}^3$	$0.45 \times 0.30 \times 0.15 \text{ mm}$
$Z = 2$	

Data collection

Siemens SMART area-detector	4132 independent reflections
ω scans	2968 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{\text{int}} = 0.058$
(SADABS; Sheldrick, 1996;	$\theta_{\text{max}} = 27.5^\circ$
Blessing, 1995)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.20$, $T_{\text{max}} = 0.51$	$k = -9 \rightarrow 9$
10 268 measured reflections	$l = -20 \rightarrow 21$

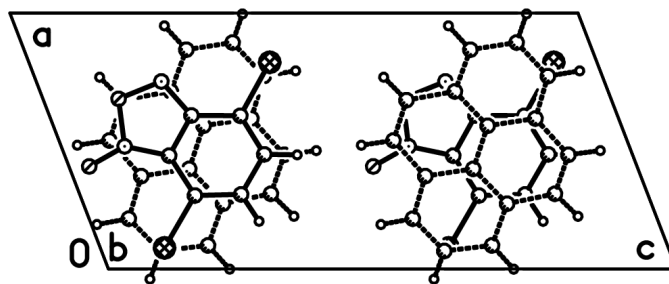


Figure 2

View along *b*. The 47BF molecules are shown with solid bonds. The pyrene is shown with dashed bonds.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$
$wR(F^2) = 0.127$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} = 0.001$
4132 reflections	$\Delta\rho_{\text{max}} = 0.92 \text{ e \AA}^{-3}$
254 parameters	$\Delta\rho_{\text{min}} = -0.54 \text{ e \AA}^{-3}$

A complete sphere of data was collected. All possible Friedel pairs were measured, of which 2031 (97%) of the 2101 pairs contained information about the polarity of the crystal. Inversion twinning was present and the final refinement converged with a Flack (1983) parameter of 0.54 (2).

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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