

The variation in B—O distances in (3) reflects the relative contributions of B—O π bonding in the bonds. Atoms B4 and B6 compete for π -electron density most effectively when interacting with O3 and O1 respectively. When both B4 and B6 compete for electron density from the same O atom, O5, the π component is reduced. The same situation was observed in [Ph₄B₃O₃]⁻ and both the amine adducts of (PhBO)₃ previously reported.

Only the primary amine group of DMED interacts with (PhBO)₃ in (3). The B—N distance of 1.636 (6) Å is shorter than that in 2(PhBO)₃.3(*p*-NH₂C₆H₄NH₂) [1.663 (8) Å], and much shorter than that reported for 2(PhBO)₃.N(CH₂CH₂)₃N [1.714 (10) Å] (Yalpani & Boese, 1983). In both of the latter adducts an N atom was bonded to B atoms in separate (PhBO)₃ rings. In (3) there is an intramolecular hydrogen-bonding interaction N1—H...N2 [N1...N2 2.737 (5) Å, N—H...N 116°]; the large deviation of the N—H...N angle from the 180° value often found is a consequence of the intramolecular nature of this hydrogen bond. Another point of difference between (3) and (4) or (5) was that space-filling molecules [two *p*-NH₂C₆H₄NH₂ in (4) and three benzene molecules in (5)] were present in the crystal lattices of the latter two adducts; compound (3) does not have this feature.

The B—C(Ph) distances [B(*sp*³)—C 1.601 (6) and mean values of B(*sp*²)—C 1.570 (6) Å in (3)] are comparable with mean values of B(*sp*²)—C [1.541 (8) Å] in (PhBO)₃ and B(*sp*³)—C [1.626 (10) Å]

and B(*sp*²)—C [1.566 (9) Å] in [Ph₄B₃O₃]⁻. The C—C and C—N distances in (3) are all normal. There are no untoward intermolecular contacts; surprisingly, while one amino H atom is involved in intramolecular hydrogen bonding, the other is involved in neither intra- nor intermolecular hydrogen bonding.

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Phenyl(*p*-tolylsulfonylamino)(*p*-tolylsulfonylimino)- λ^4 -sulfane, a Reaction Product of Diphenyl Disulfide and Chloramine-T

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Abstract. *N*¹-Tosyl-*N*²-tosylbenzenesulfinamidine, C₂₀H₂₀N₂O₄S₃, *M*_r = 448.59, triclinic, *P* $\bar{1}$, *a* = 10.140 (1), *b* = 12.465 (2), *c* = 9.665 (2) Å, α = 101.88 (1), β = 114.57 (1), γ = 81.18 (1)°, *V* =

1084.0 (6) Å³, *Z* = 2, *D*_x = 1.37 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 3.5 cm⁻¹, *F*(000) = 468, *T* = 294 K, *R* = 0.035 for 3376 observed reflections. The analysis establishes that the reaction product is PhS(=NSO₂C₆H₄CH₃)(NHSO₂C₆H₄CH₃). The S^{VI}—N and S^{VI}—N(H) distances are 1.611 (2) and 1.630 (2) Å respectively, the S^{IV}—N and S^{IV}—N(H)

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distances are 1.593 (2) and 1.678 (2) Å respectively, and there are two short intramolecular S...O contacts of 2.866 (1) and 2.932 (2) Å, indicative of a highly polar, ylidic structure. The molecules are linked into centrosymmetric dimers by linear N—(H)...O—(S) hydrogen bonds, with N...O 2.800 (3) Å.

Introduction. The reactions of organic sulfur compounds with chloramine-T [sodium *N*-chlorotoluene-*p*-sulfonamide, Na⁺.CH₃C₆H₄SO₂NCl⁻, (1)] yield a wide diversity of products (Campbell & Johnson, 1978). Thus, whereas sulfides (*R*₂S) yield both sulfilimines (*R*₂S=NSO₂C₆H₄CH₃) and sulfoxides (*R*₂SO), examples of which we have recently characterized structurally (Ferguson, Lough, Glidewell & Low, 1989), thiols (*R*SH) and disulfides (*R*₂S₂) give a common product (Clarke, Kenyon & Philips, 1930; Alexander & McCombie, 1932) in which two tolylsulfonylimino groups are present for each *RS* unit. Here we report the structure of the product from Ph₂S₂, PhS(=NSO₂C₆H₄CH₃)(NHSO₂C₆H₄CH₃), phenyl(*p*-tolylsulfonylamino)(*p*-tolylsulfonylimino)-λ⁴-sulfane.

Experimental. The title compound was prepared by reaction of Ph₂S₂ with chloramine-T in *tert*-butanol at 313 K (Dell'erba, Poluzzi Corallo, Novi & Leandri, 1981). NMR (CD₂Cl₂): δ_H 2.40 (*s*, 6H, CH₃), 4.5 (*s*, br, 1H, NH), 7.22 and 7.64 (*AB*, *J* 8 Hz, 8H, —C₆H₄—), 7.4–7.7 (*m*, 5H, C₆H₅); δ_C 21.8 (*q*, CH₃) 127.2 (*d*), 127.3 (*d*), 130.1 (*d*), 130.2 (*d*), 133.0 (*d*), 137.0 (*s*), 138.7 (*s*), 144.6 (*s*) (aromatic). Colourless needles were grown from ethanol. Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range 12 ≤ θ ≤ 20°. Crystal dimensions 0.36 × 0.13 × 0.50 mm; intensities of reflections with indices *h* –12 to 12, *k* –15 to 15, *l* 0 to 12; ω–2θ scans, ω-scan width (0.60 + 0.35tanθ)°, graphite-monochromated Mo *K*α radiation, 2θ_{max} = 54°. Intensities of three reflections measured every 2 h showed a small loss of intensity (2.2% over the course of the data collection), anisotropic decay correction, correction factors on *I* ranged from 0.951 to 1.109 with an average value of 1.042. 4713 unique reflections were measured, 3376 with *I* ≥ 3σ(*I*) were labelled observed and used in structure solution and refinement. Data were corrected for Lorentz and polarization effects, no absorption correction. Space group *P*1 or $\bar{P}1$, $\bar{P}1$ was chosen and confirmed by the successful refinement. Structure solution was by direct methods (Sheldrick, 1986) followed by difference Fourier syntheses. Refinement (Frenz, 1983) was by full-matrix least-squares calculations, initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the

refinement, difference maps showed maxima in positions consistent with the expected locations of the H atoms. H atoms bound to carbon were included in the refinement as riding atoms with *d*(C—H) of 0.95 Å. The methyl hydrogens appeared as a torus of electron density; they were allowed for by including six half-H atoms at 60° intervals round the torus. The hydrogen bound to nitrogen N(1) was clearly located (Fig. 1) and its position was refined with an individual isotropic thermal parameter. A secondary-extinction coefficient (Zachariasen, 1963) refined to a value of 3 × 10⁻⁷. The final cycle of refinement on *F* included 267 variable parameters, *R* = 0.035, *wR* = 0.046, *w* = 1/[σ²(*F*_o) + 0.04(*F*_o)²]. Max. shift/e.s.d. in the last cycle was <0.005; density in final difference map ±0.24 (5) e Å⁻³; no chemically significant features. Scattering factors and anomalous-dispersion corrections were from *International Tables for X-ray Crystallography* (1974, Vol. IV). The calculations were performed on a PC-XT (SHELXS86) and an enhanced PDP-11/73 computer (SDP-Plus). Atomic coordinates and details of molecular geometry are given in Tables 1 and 2.* Fig. 2 is a view of the molecule with the atom-numbering scheme, prepared using ORTEPII (Johnson, 1976); Fig. 3 shows the intermolecular hydrogen bonding

Discussion. The crystalline product obtained from the reaction of Ph₂S₂ with chloramine-T had an elemental analysis consistent with its formulation as (2) but its NMR spectra in solution suggested that

* Tables of calculated hydrogen coordinates, thermal parameters, mean-planes data and torsion angles, and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53143 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

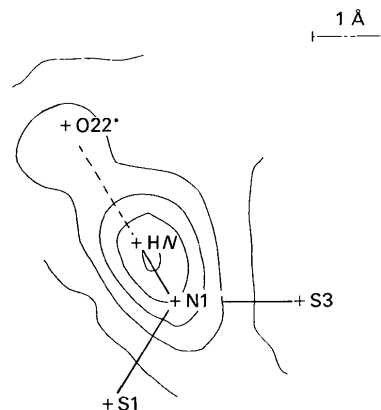


Fig. 1. Section of the difference Fourier map in the S(1)N(1)S(3) plane, showing the location of the H atom bound to nitrogen: successive contour levels represent electron-density increments of 0.05 e Å⁻³.

Table 1. Positional and thermal parameters and their e.s.d.'s

	x	y	z	B (Å ²)*
S(1)	0.04979 (6)	0.29081 (4)	0.25251 (6)	3.76 (1)
S(2)	-0.20402 (5)	0.63239 (4)	0.10893 (5)	3.45 (1)
S(3)	0.03686 (5)	0.52913 (4)	0.30251 (5)	3.01 (1)
O(11)	0.1771 (2)	0.2179 (1)	0.2743 (2)	5.78 (5)
O(12)	-0.0045 (2)	0.3212 (1)	0.3708 (2)	4.74 (4)
O(21)	-0.2722 (1)	0.5766 (1)	0.1747 (2)	4.53 (4)
O(22)	-0.2605 (2)	0.6183 (2)	-0.0569 (2)	4.80 (4)
N(1)	0.0834 (2)	0.4052 (1)	0.2210 (2)	3.72 (4)
N(2)	-0.0310 (2)	0.6030 (1)	0.1691 (2)	3.60 (4)
C(11)	-0.0894 (2)	0.2354 (2)	0.0819 (2)	3.75 (5)
C(12)	-0.2315 (2)	0.2761 (2)	0.0487 (3)	5.11 (6)
C(13)	-0.3396 (3)	0.2253 (3)	-0.0797 (3)	6.22 (7)
C(14)	-0.3101 (3)	0.1360 (2)	-0.1741 (3)	6.37 (7)
C(15)	-0.1663 (4)	0.0994 (2)	-0.1400 (3)	7.10 (9)
C(16)	-0.0563 (3)	0.1481 (2)	-0.0135 (3)	5.68 (7)
C(17)	-0.4278 (4)	0.0767 (3)	-0.3113 (4)	10.3 (1)
C(21)	-0.2197 (2)	0.7746 (2)	0.1741 (2)	3.81 (5)
C(22)	-0.2811 (3)	0.8481 (2)	0.0734 (3)	5.64 (6)
C(23)	-0.2904 (3)	0.9585 (2)	0.1292 (3)	7.42 (8)
C(24)	-0.2379 (3)	0.9982 (2)	0.2842 (4)	7.32 (8)
C(25)	-0.1780 (4)	0.9234 (3)	0.3828 (4)	7.8 (1)
C(26)	-0.1683 (3)	0.8123 (2)	0.3295 (3)	6.45 (8)
C(27)	-0.2507 (4)	1.1196 (3)	0.3441 (5)	11.2 (1)
C(31)	0.2124 (2)	0.5775 (2)	0.4093 (2)	3.20 (4)
C(32)	0.2785 (2)	0.6301 (2)	0.3463 (3)	4.44 (6)
C(33)	0.4180 (2)	0.6619 (2)	0.4387 (3)	5.42 (7)
C(34)	0.4858 (2)	0.6425 (2)	0.5882 (3)	5.27 (7)
C(35)	0.4185 (2)	0.5899 (2)	0.6482 (3)	5.19 (6)
C(36)	0.2808 (2)	0.5561 (2)	0.5584 (2)	4.24 (5)
HN	0.139 (2)	0.398 (2)	0.170 (2)	3.4 (4)†

* Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $\frac{1}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + abc\cos\gamma B_{12} + accos\beta B_{13} + bc\cos\alpha B_{23})$.

† HN was refined with an isotropic thermal parameter.

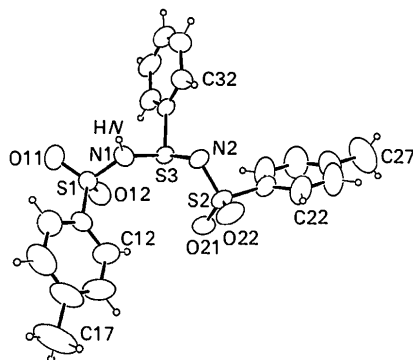


Fig. 2. Perspective view of the molecule, showing the atom-numbering scheme.

the two toluenesulfonamido fragments are equivalent. The X-ray analysis of this product established its formulation as (2), and reveals not only weak hydrogen bonds between pairs of molecules, but short intramolecular S^{IV}...O bonds, indicative of a highly polar, ylidic structure.

The bond lengths involving carbon are all entirely typical of their types (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) as are the S—O bond lengths involving S(1); however, the bonds

Table 2. Molecular dimensions

(a) Bond lengths (Å)			
S(1)—O(11)	1.425 (2)	C(14)—C(17)	1.510 (4)
S(1)—O(12)	1.426 (2)	C(15)—C(16)	1.369 (3)
S(1)—N(1)	1.630 (2)	C(21)—C(22)	1.364 (3)
S(1)—C(11)	1.756 (2)	C(21)—C(26)	1.372 (3)
S(2)—O(21)	1.436 (2)	C(22)—C(23)	1.375 (4)
S(2)—O(22)	1.442 (1)	C(23)—C(24)	1.373 (4)
S(2)—N(2)	1.611 (2)	C(24)—C(25)	1.363 (5)
S(2)—C(21)	1.761 (2)	C(24)—C(27)	1.511 (4)
S(3)—N(1)	1.678 (2)	C(25)—C(26)	1.378 (4)
S(3)—N(2)	1.593 (2)	C(31)—C(32)	1.373 (4)
S(3)—C(31)	1.773 (2)	C(31)—C(36)	1.379 (3)
C(11)—C(12)	1.377 (3)	C(32)—C(33)	1.390 (3)
C(11)—C(16)	1.370 (3)	C(33)—C(34)	1.373 (4)
C(12)—C(13)	1.379 (3)	C(34)—C(35)	1.362 (4)
C(13)—C(14)	1.367 (4)	C(35)—C(36)	1.379 (3)
C(14)—C(15)	1.379 (4)		
(b) Bond angles (°)			
O(11)—S(1)—O(12)	120.5 (1)	C(13)—C(14)—C(15)	117.7 (2)
O(11)—S(1)—N(1)	106.8 (1)	C(13)—C(14)—C(17)	122.7 (3)
O(11)—S(1)—C(11)	107.49 (9)	C(15)—C(14)—C(17)	119.6 (3)
O(12)—S(1)—N(1)	106.0 (1)	C(14)—C(15)—C(16)	121.6 (3)
O(12)—S(1)—C(11)	108.9 (1)	C(11)—C(16)—C(15)	119.4 (3)
N(1)—S(1)—C(11)	106.5 (1)	S(2)—C(21)—C(22)	121.5 (2)
O(21)—S(2)—O(22)	116.88 (9)	S(2)—C(21)—C(26)	119.2 (2)
O(21)—S(2)—N(2)	112.96 (9)	C(22)—C(21)—C(26)	119.3 (2)
O(21)—S(2)—C(21)	108.8 (1)	C(21)—C(22)—C(23)	119.6 (2)
O(22)—S(2)—N(2)	107.0 (1)	C(22)—C(23)—C(24)	122.1 (3)
O(22)—S(2)—C(21)	106.7 (1)	C(23)—C(24)—C(25)	117.4 (3)
N(2)—S(2)—C(21)	103.51 (9)	C(23)—C(24)—C(27)	121.6 (3)
N(1)—S(3)—N(2)	105.94 (9)	C(25)—C(24)—C(27)	121.0 (3)
N(1)—S(3)—C(31)	99.22 (8)	C(24)—C(25)—C(26)	121.5 (3)
N(2)—S(3)—C(31)	102.2 (1)	C(21)—C(26)—C(25)	120.1 (3)
S(1)—N(1)—S(3)	122.6 (1)	S(3)—C(31)—C(32)	122.3 (1)
S(1)—N(1)—HN	116 (1)	S(3)—C(31)—C(36)	115.8 (2)
S(3)—N(1)—HN	121 (1)	C(32)—C(31)—C(36)	121.9 (2)
S(2)—N(2)—S(3)	115.6 (1)	C(31)—C(32)—C(33)	117.6 (2)
S(1)—N(1)—C(12)	119.9 (2)	C(32)—C(33)—C(34)	120.8 (3)
S(1)—C(11)—C(16)	119.6 (2)	C(33)—C(34)—C(35)	120.5 (2)
C(12)—C(11)—C(16)	120.5 (2)	C(34)—C(35)—C(36)	119.9 (2)
C(11)—C(12)—C(13)	118.6 (2)	C(31)—C(36)—C(35)	119.2 (3)
C(12)—C(13)—C(14)	122.2 (2)		
(c) Hydrogen-bond dimensions (Å and °) and short intramolecular contacts (Å)			
N(1) O(22)	2.800 (3)	O(22) HN	179 (2)
HN O(22)	1.926 (22)		
HN N(1)	0.874 (23)		
S(3) O(12)	2.932 (2)		
S(3) O(21)	2.866 (1)		

Symmetry code: (i) $-x, 1-y, -z$.

S(2)—O(21) and S(2)—O(22), 1.436 (2) and 1.442 (1) Å respectively, are both longer than the upper quartile for such bonds (Allen *et al.*, 1987). The four independent S—N bonds in the molecule all exhibit different lengths, ranging from 1.593 (2) Å for S(3)—N(2) to 1.678 (2) Å for S(3)—N(1). The S(3)—N(2) bond length is very close to that found for the S—N bond in chloramine-T itself (1), 1.590 (2) Å (Olmstead & Power, 1986), which was interpreted as a double bond. On the other hand, the S(3)—N(1) distance is typical of S—N single bonds (Olsen & Barrick, 1973; Holt, Holt & Watson, 1974). The remaining S—N bond lengths, S(1)—N(1) 1.630 (2) and S(2)—N(2) 1.611 (2) Å are both close to the S—N bond lengths 1.629 (2) and 1.602 (2) Å

found in compound (3) (Ferguson *et al.*, 1989), and 1.639 (2) and 1.605 (2) Å found in compound (4) (Beer, Hordvik, Naylor & Wright, 1981): in each compound the S—N bonds were interpreted as being midway between the single and double bonds. Compounds (1)–(4) are shown in Fig. 4.

There are two intramolecular S...O contacts, S(3)...O(12) 2.932 (2) and S(3)...O(21) 2.866 (1) Å, which are considerably shorter than the sum of the van der Waals radii, 3.3 Å (Bondi, 1964). Such short interactions have been noticed previously in (4) and in a range of other sulfilimines (Ferguson *et al.*, 1989), as well as in arsenic and antimony ylides (Ferguson, Glidewell, Lloyd, Metcalf & Ruhl, 1987; Ferguson, Glidewell, Gosney, Lloyd, Lumbroso & Metcalfe, 1988), and have been interpreted consistently in terms of an attractive interaction between negatively charged oxygen and positively charged sulfur, arsenic or antimony, in a polar ylidic structure, as for example (3a) ↔ (3b). As in such other

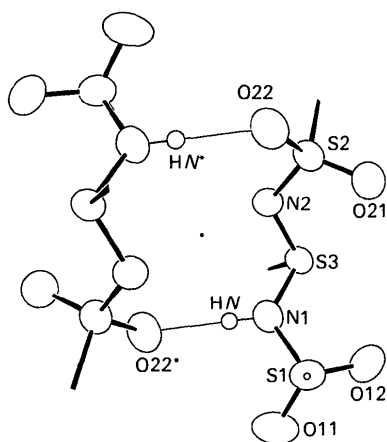


Fig. 3. The intermolecular hydrogen bonding forming centrosymmetric dimers; for clarity the phenyl and *p*-tolyl rings are not shown.

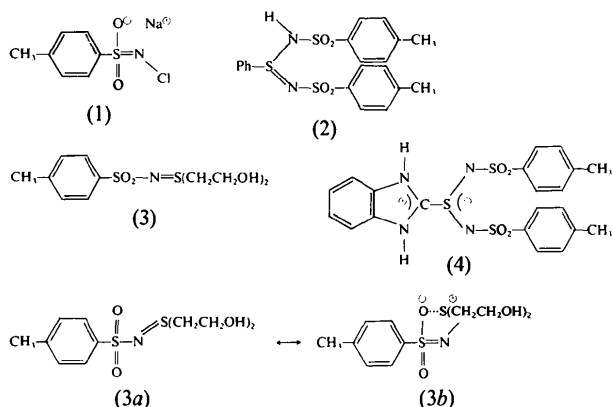


Fig. 4. Compounds (1)–(4).

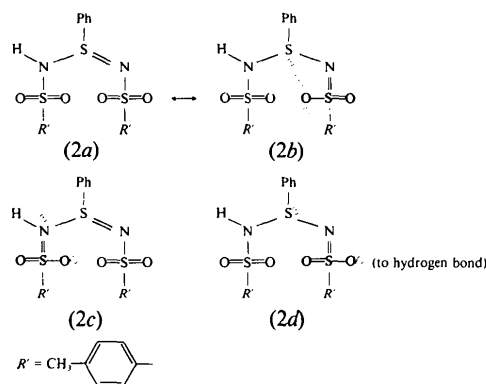


Fig. 5. Four canonical forms of (2).

sulfur, arsenic and antimony compounds, the short intramolecular contacts are associated with almost planar four-atom fragments [*cf.* (3b)]. In the present structure (2) the torsion angles S(3)—N(1)—S(1)—O(12) and S(3)—N(2)—S(2)—O(21) are -7.2 (2) and -6.4 (1)° respectively; in (3) the corresponding S—N—S—O torsion angle is -20.0 (2)° (Ferguson *et al.*, 1989).

The molecules of (2) are joined together by weak hydrogen bonds (Table 2) to form dimer units lying across a centre of inversion (Fig. 3). The S—O bond involved [S(2)—O(22), 1.442 (1) Å] is the longest in the molecule, while the adjacent S—N bond [S(2)—N(2), 1.611 (2) Å] is of length intermediate between single and double bonds. The combination of the short ylidic intramolecular S...O contacts and the intermolecular hydrogen bonding in the solid state thus leads to the requirement of at least four canonical forms (2a)–(2d) (Fig. 5) which together make up the overall electronic structure. In solution the hydrogen bonding is disrupted and the apparent equivalence of the two $\text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{N}$ fragments arises from fast exchange of the unique hydrogen between alternative sites at nitrogen.

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Structure of a Benzanthracene–1,2,4,5-Tetracyanobenzene Complex, C₁₈H₁₂·C₁₀H₂N₄

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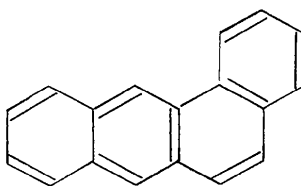
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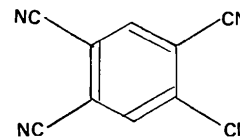
Abstract. Benz[*a*]anthracene–1,2,4,5-benzenetetracarbonitrile, $M_r = 406.45$, monoclinic, $P2_1/n$, $a = 8.216$ (4), $b = 7.965$ (3), $c = 30.709$ (6) Å, $\beta = 91.56$ (2)°, $V = 2009$ (2) Å³, $Z = 4$, $D_x = 1.34$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu(\text{Mo } K\alpha) = 0.8$ cm⁻¹, $F(000) = 840$, $T = 123$ K; refinement with 1444 observed diffractometer data converged to $R = 0.057$. The molecules lie in ribbons in the (206) plane and the benzanthracene component is disordered over two sites. The stacking of molecules in the crystal is similar to that found in a benzanthracene–pyromellitic dianhydride complex, there being two different environments for each donor and acceptor atom.

Introduction. Benz[*a*]anthracene is the parent compound of a large series of carcinogenic compounds but is not regarded as being an active compound itself. No accurate structure determination of benz[*a*]anthracene has been reported to date although the unit cell and space group were first published in 1938 (Iball, 1938) and a two-dimensional structure in 1956 (Friedlander & Sayre, 1956). In an effort to obtain good molecular data for the benz[*a*]anthracene (BA)

nucleus and to study complexes of BA (an electron donor) with various electron acceptors, 1:1 complexes of BA with pyromellitic dianhydride (PMDA) (Foster, Iball, Scrimgeour & Williams, 1976) and with 1,2,4,5-tetracyanobenzene (TCNB) were prepared. We now report the structure of the BA–TCNB complex.



BA



TCNB

Experimental. The crystals were obtained as bright orange rectangular plates by mixing saturated solutions of both components in ethyl acetate in a 1:1 molar ratio. The crystals were recrystallized twice from ethyl acetate. All measurements were made with a crystal 0.10 × 0.50 × 0.50 mm cooled to 123 K. Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $11 < \theta < 19^\circ$. Intensities of reflections with h 0 to 9, k 0 to 8 and l –33 to 33

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