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A 1:1 complex of 2,4,5,6-tetrachloro-1,3-dicyano-benzene with pyrene

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In the crystal structure of the title compound, 2,4,5,6-tetrachloro-1,3-dicyanobenzene–pyrene (1/1), $C_8Cl_4N_2$·$C_{16}H_{10}$, the dicyanobenzene molecules located on twofold rotation axes of symmetry and the pyrene molecules on inversion centers. The primary intermolecular interaction is in an alternate π-stacked arrangement. There are no unusual interactions between the stacks.

Comment

In an earlier paper (Britton, 2002), the formation of two-dimensional arrays of the isomers of tetrachlorodicyano-benzene, TCDB, by formation of complexes with hexa-methylbenzene was described. At the same time, the possibility of forming similar arrangements, with other π bases replacing the hexamethylbenzene, was explored. While a number of complexes were formed, none of them had the desired layers of TCDB. The structure of the complex, *m*-TCDB/pyrene 1/1, (I), is described here.
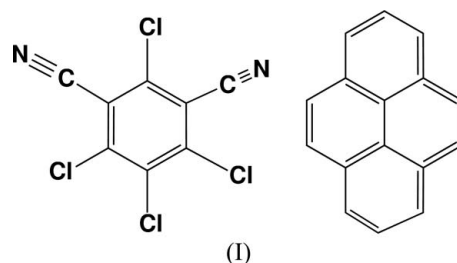

(I)

Fig. 1 shows the labelling and the structures of the two molecules. The TCDB molecule lies on a twofold axis; the pyrene molecule lies on a center of symmetry. Bond lengths and angles are normal.

The primary intermolecular interaction in this complex between alternating molecules in a π stack parallel to the [101]
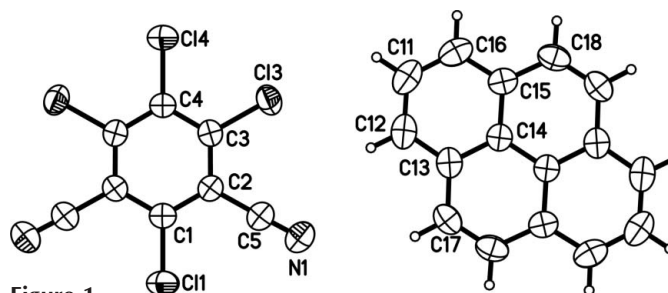

Figure 1
Both molecules in TCDB/pyrene. Displacement ellipsoids are drawn at the 50% probability level. Unlabeled atoms in TCDB are generated by the symmetry operation $(1 - x, y, \frac{1}{2} - z)$, and those in pyrene by $(\frac{1}{2} - x, \frac{1}{2} - y, 2 - z)$.

direction. The molecules are approximately parallel to (201), being tilted 2.1 (1)° away from parallel with each other, and are 3.47 (7) Å apart in the stack; the large uncertainty is a consequence of the deviation from a parallel arrangement. There are no unusual interactions between the stacks.

Experimental

Crystals of the complex were obtained by dissolving equimolar amounts of the two components in acetone and allowing the solution to evaporate.

Crystal data

$C_8Cl_4N_2 \cdot C_{16}H_{10}$	$D_x = 1.576$ Mg m^{-3}
$M_r = 468.14$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25
$a = 12.032$ (3) Å	reflections
$b = 15.808$ (4) Å	$\theta = 10.4–21.0°$
$c = 10.673$ (3) Å	$\mu = 0.62$ mm^{-1}
$\beta = 103.66$ (3)°	$T = 296$ (2) K
$V = 1972.6$ (9) Å^3	Needle, yellow
$Z = 4$	$0.50 \times 0.15 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 27.0°$
	$h = -15 \rightarrow 15$
$\omega–2\theta$ scans	$k = 0 \rightarrow 20$
Absorption correction: none	$l = -13 \rightarrow 13$
5654 measured reflections	3 standard reflections
2154 independent reflections	frequency: 75 min
1695 reflections with $I > 2\sigma(I)$	intensity decay: <1%
$R_{int} = 0.051$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$+ 0.061P]$
$wR(F^2) = 0.115$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2154 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
138 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	

H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1983); cell refinement: *CAD-4 Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); 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*.

References

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