(1967), rather than that made by Bocian, Pickett, Rounds & Strauss (1975) that the boat is the most stable conformation.

The butoxy group lies approximately in the plane of the phenyl ring [torsion angle C(15)—C(16)—O(3)—C(21) = 176.2 (3)°; torsion angles along the butyl chain are close to 180°]. The mean plane of the carbamate moiety [atoms N(2), C(14), O(1) and O(2)] makes a dihedral angle of 10.4 (4)° with the mean plane of the phenyl ring and there is intramolecular hydrogen-bond formation between the amide N(2)—H group and O(3) [N(2)···O(3) = 2.531 (3), H···O(3) = 2.12 Å, N(2)—H···O(3) = 98° (H calculated)]. The N(2)—C(14) bond distance of 1.349 (4) Å is somewhat longer than the range of values 1.32–1.33 Å typically found for the N—C distances in the amide group, and the C(15)—N(2) bond length [1.408 (4) Å] is slightly shorter than the value of 1.425 (3) Å found for a pure $C(sp^2)$—N single bond (Adler, Goode, King, Mellor & Miller, 1976). These results suggest some degree of delocalization of the lone pair of electrons on the amide N atom through the phenyl ring, obviously owing to the coplanarity of the carbamate and phenyl groups and to the polarizing effect of the N(2)—H···O(3) hydrogen bond.

From the pharmacological point of view, the most important structural feature of the phenylcarbamate type local anaesthetics is the three-dimensional disposition of the common functional groups, *i.e.* the protonated amino N atom, the N^+—H vector and the phenylcarbamate function (namely, the carbonyl O atom and the π-excessive phenyl ring), which are assumed to define the interaction with the anaesthetic receptor (Remko & Scheiner, 1988). The spatial relationship between these primary anaesthesiophoric groups is defined by torsion angles along the N(1)—C(6)—C(7) chain as follows: τ_1[C(1)—N(1)—C(6)—C(7)] = 69.3 (3), τ_2[N(1)—C(6)—C(7)—C(8)] = −111.8 (3) and τ_3[C(6)—C(7)—C(8)—O(1)] is fixed at 81.9 (3)° by the *cis* position of C(6) and O(1) and by the twist-chair conformation of the cycloheptane ring. As a result, the mean planes of the piperidine and cycloheptane rings make a dihedral angle of 110.3 (4)° with one another.

The principal interaction between the cations and the chloride anions is an $N(1)^+$—H···$Cl^-(\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z)$ hydrogen bond [N(1)···Cl = 3.067 (3), H···Cl = 2.04 Å, N(1)—H···Cl = 176° (H calculated)]. Other packing forces are of coulombic and van der Waals types acting at distances > 3.2 Å.

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Structures of Three Electron Donor–Acceptor Complexes of Dibenz[*a*,*h*]anthracene (DBA)

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Abstract. (I) Dibenz[*a*,*h*]anthracene–1,3,5-trinitrobenzene (1/2) (DBA–TNB), $C_{22}H_{14}.2C_6H_3N_3O_6$, M_r = 704.57, monoclinic, $P2_1/a$, $a = 25.744$ (3), $b = 7.889$ (1), $c = 7.442$ (1) Å, $\beta = 91.61$ (1)°, $V = 1510.8$ (4) Å^3, $Z = 2$, $D_x = 1.549$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.978$ mm^{-1}, $F(000) = 724$, $T = 293$ K, $R = 0.043$ for 2619 data; (II) dibenz[*a*,*h*]anthracene–pyromellitic dianhydride (1/1) (DBA–

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PMDA), $C_{22}H_{14}.C_{10}H_2O_6$, $M_r = 496.47$, monoclinic, $P2_1/n$, $a = 20.784$ (2), $b = 7.357$ (1), $c = 7.524$ (1) Å, $\beta = 97.71$ (1)°, $V = 1140.0$ (3) $Å^3$, $Z = 2$, $D_x = 1.45$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.783$ mm^{-1}, $F(000) = 512$, $T = 293$ K, $R = 0.042$ for 1514 data; (III) dibenz[*a*,*h*]anthracene–1,8:4,5-naphthalenetetracarboxylic dianhydride (1/1) (DBA–NTDA), $C_{22}H_{14}.C_{14}H_4O_6$, $M_r = 546.54$, monoclinic, $P2_1/a$, $a = 19.991$ (4), $b = 8.324$ (2), $c = 7.821$ (2) Å, $\beta = 105.06$ (2)°, $V = 1256.8$ (5) $Å^3$, $Z = 2$, $D_x = 1.444$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.765$ mm^{-1}, $F(000) = 564$, $T = 293$ K, $R = 0.050$ for 1982 data. The donor DBA molecules, located on symmetry centers, stack alternately with the acceptor molecules. The stacks pack in a herringbone motif along the cell axis.

Introduction. The polycyclic aromatic hydrocarbon (PAH) dibenz[*a*,*h*]anthracene (DBA) has been shown to be a carcinogen (Cook, Heiger, Kennaway & Mayneord, 1932; Lecoq, Perin, Strapelias & Duquesne, 1989). Many studies of PAH electron donors in π–π^* complexes with various electron acceptors have been reported (Herbstein, 1971; Mayoh & Prout, 1972; Prout & Kamenar, 1973). The study of the structures described here, DBA complexes (I) with 1,3,5-trinitrobenzene (TNB) (1/2), (II) with pyromellitic dianhydride (PMDA) (1/1), and (III) with 1,8:4,5-naphthalenetetracarboxylic dianhydride (NTDA) (1/1), was undertaken to determine the modes of stacking of the constituent pairs in the complexes and the molecular packing in the respective crystals. Preliminary information on this work was presented in brief (Zacharias, 1979) and is part of a continuing study of carcinogenic PAH compounds and their metabolites.

Experimental. Crystal and experimental data are given in Table 1. Crystals of (I) were obtained from the slow cooling of a hot anhydrous ethanol solution of DBA to which TNB in ethanol had been added. Crystals of (II) were obtained by adding PMDA to a warmed solution of DBA in anhydrous 2-butanone followed by slow evaporation. Crystals of (III) resulted from slow cooling of a solution of DBA and NTDA in ethanol which had been heated to boiling. All data were collected at the ambient temperature using variable θ–2θ scan rates, depending on the reflection intensity, to $(\sin\theta/\lambda)_{max} = 0.61$ $Å^{-1}$. The space groups were established from observed systematic absences and the lattice parameters determined from 14 centered reflections in each case. Values of $\sigma(F)$ were obtained from the relation $\sigma(F) = (F/2)[\sigma^2(I)/I^2 + \delta^2]^{1/2}$ where δ is an instrumental uncertainty obtained from monitoring standard reflections during data collection (four every 92 collected). Empirical absorption corrections as a function of φ derived from 72-point ψ-scans were applied during data reduction. Intensity variations in the standard reflections were less than 1%. Data were collected on a Syntex $P2_1$ diffractometer.

Table 1. *Crystal and experimental data*

	(I)	(II)	(III)
Crystal size (mm)	0.28 × 0.25 × 0.12	0.21 × 0.25 × 0.65	0.10 × 0.12 × 0.43
Crystal habit	Prism	Prism	Prism
Crystal color	Dark orange	Dark red	Dark brown
θ range for cell-parameter determination (°)	18–27	24.5–28	10.5–24.1
Maximum $\sin\theta/\lambda$ for data collection ($Å^{-1}$)	0.606	0.606	0.606
Range h	0→31	0→24	0→24
k	0→9	0→8	−10→0
l	−9→9	−9→9	−9→9
Absorption correction, T_{max}	1.000	1.000	1.000
T_{min}	0.915	0.942	0.842
Total No. of data	3103	2363	2695
No. of unique data	2878	2131	2348
R_{int}	0.011	0.019	0.017
No. of observed data	2619	1514	1982
σ cutoff	$2.00\sigma(I)$	$3.00\sigma(I)$	$3.00\sigma(I)$
δ	0.015	0.021	0.023
R_{obs}	0.043	0.042	0.050
R_{all}	0.045	0.049	0.060
wR_{obs}	0.064	0.055	0.054
wR_{all}	0.071	0.057	0.055
No. of parameters refined	235	204	226
S	0.86	1.64	1.53
$(\Delta/\sigma)_{max}$	0.08	0.05	0.35
$(\Delta\rho)_{max}$ (e $Å^{-3}$)	0.18	0.15	0.17
$(\Delta\rho)_{min}$ (e $Å^{-3}$)	−0.21	−0.17	−0.16

The structures were solved using the direct methods program *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977). Full-matrix least-squares refinements on F were performed with anisotropic vibration parameters for non-H atoms and isotropic for H atoms (located from difference Fourier maps). The quantity minimized was $\sum w||F_o| - |F_c||^2$, where $w = [\sigma^2(F)]^{-1}$. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). In-house computer programs were used in further calculations (Carrell, Shieh & Takusagawa, 1981).

Discussion. The final fractional coordinates and U_{eq} values are given in Table 2.* Interatomic bond lengths and bond angles are listed in Table 3. Fig. 1 shows the thermal ellipsoid representations of the donor–acceptor pairs and atomic numbering (Johnson, 1965; Davies, 1983). Fig. 2 shows stereoscopic views of the molecular packing. In (I) and (III), the centrosymmetric donor DBA occupies the crystallographic symmetry centers 0,0,0 and $\frac{1}{2},\frac{1}{2}$,0 in the unit cell; in (II), the corresponding positions are 0,0,0 and $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. In (II) and (III), the centrosymmetric acceptor molecules are arrayed midway between the parallel

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* Lists of refined positions of H atoms, anisotropic temperature factors, least-squares-planes data and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55866 (64 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1016]

Table 2. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

U_{eq} = (1/3) (trace orthogonalized U_{ij} matrix). C atoms of acceptor molecules TNB, PMDA and NTDA have *T*, *P* and *N*, respectively, in their labeling.

	x	y	z	U_{eq}
(I)				
O(1)	0.37654 (6)	0.6648 (2)	0.3184 (3)	0.0831 (8)
O(2)	0.30002 (5)	0.5688 (2)	0.2578 (2)	0.0728 (6)
O(3)	0.26100 (5)	−0.0177 (2)	0.3948 (2)	0.0711 (6)
O(4)	0.30994 (5)	−0.1452 (2)	0.5925 (2)	0.0702 (6)
O(5)	0.48632 (5)	0.0567 (2)	0.6853 (2)	0.0662 (6)
O(6)	0.50952 (5)	0.2889 (2)	0.5606 (3)	0.089 (1)
N(1)	0.34414 (5)	0.5528 (2)	0.3174 (2)	0.0539 (6)
N(2)	0.30050 (5)	−0.0286 (2)	0.4892 (2)	0.0526 (5)
N(3)	0.47731 (5)	0.1845 (2)	0.5974 (2)	0.0534 (6)
C(1*T*)	0.35977 (5)	0.3874 (2)	0.3934 (2)	0.0437 (5)
C(2*T*)	0.32299 (5)	0.2609 (2)	0.4002 (2)	0.0446 (5)
C(3*T*)	0.33877 (5)	0.1085 (2)	0.4776 (2)	0.0434 (5)
C(4*T*)	0.38852 (5)	0.0802 (2)	0.5440 (2)	0.0451 (5)
C(5*T*)	0.42346 (5)	0.2124 (2)	0.5301 (2)	0.0428 (5)
C(6*T*)	0.41076 (5)	0.3679 (2)	0.4555 (2)	0.0443 (6)
C(1)	0.31622 (8)	0.0950 (2)	−0.0295 (2)	0.0623 (8)
C(2)	0.36263 (8)	0.0243 (2)	0.0388 (2)	0.0602 (8)
C(3)	0.40765 (6)	0.1181 (2)	0.0457 (2)	0.0544 (7)
C(4)	0.40850 (6)	0.2881 (2)	−0.0148 (2)	0.0499 (6)
C(5)	0.45518 (6)	0.3920 (2)	−0.0058 (2)	0.0486 (6)
C(6)	0.50289 (6)	0.3362 (2)	0.0658 (2)	0.0511 (6)
C(7)	0.54697 (6)	0.4378 (2)	0.0740 (2)	0.0503 (6)
C(8)	0.59573 (7)	0.3753 (2)	0.1473 (2)	0.0577 (7)
C(9)	0.63891 (6)	0.4706 (2)	0.1515 (2)	0.0571 (7)
C(10)	0.63853 (6)	0.6427 (2)	0.0854 (2)	0.0529 (7)
C(11)	0.68408 (7)	0.7409 (2)	0.0913 (2)	0.0602 (8)
(II)				
O(1)	0.32662 (6)	0.8372 (2)	0.4507 (2)	0.087 (1)
O(2)	0.38730 (6)	0.8061 (2)	0.7212 (2)	0.0711 (7)
O(3)	0.46958 (8)	0.8093 (2)	0.9468 (2)	0.087 (1)
C(1*P*)	0.45406 (8)	1.0087 (2)	0.3388 (2)	0.0489 (8)
C(2*P*)	0.43870 (7)	0.9375 (2)	0.4981 (2)	0.046 (5)
C(3*P*)	0.48305 (8)	0.9288 (2)	0.6526 (2)	0.0465 (8)
C(4*P*)	0.4497 (1)	0.8440 (3)	0.7942 (3)	0.061 (1)
C(5*P*)	0.37683 (9)	0.8586 (3)	0.5411 (3)	0.062 (1)
C(1)	0.26204 (9)	0.2056 (3)	0.6663 (4)	0.085 (1)
C(2)	0.3148 (1)	0.2133 (3)	0.7990 (3)	0.081 (1)
C(3)	0.37314 (9)	0.2856 (3)	0.7647 (3)	0.064 (1)
C(4)	0.38031 (8)	0.3528 (2)	0.5925 (2)	0.0512 (8)
C(5)	0.44119 (7)	0.4271 (2)	0.5491 (2)	0.0436 (8)
C(6)	0.49698 (8)	0.4433 (2)	0.6738 (2)	0.0458 (8)
C(7)	0.55503 (8)	0.5131 (2)	0.6296 (2)	0.0449 (8)
C(8)	0.61149 (9)	0.5273 (3)	0.7622 (2)	0.057 (1)
C(9)	0.66743 (9)	0.5945 (3)	0.7210 (3)	0.064 (1)
C(10)	0.67374 (8)	0.6566 (3)	0.5428 (3)	0.059 (1)
C(11)	0.7326 (1)	0.7301 (3)	0.5019 (4)	0.077 (1)
(III)				
C(1)	0.74434 (9)	0.8632 (2)	0.0509 (3)	0.083 (1)
C(2)	0.69679 (9)	0.8501 (2)	−0.1124 (3)	0.078 (1)
C(3)	0.63821 (8)	0.7590 (2)	−0.1300 (3)	0.0676 (9)
C(4)	0.62499 (7)	0.6786 (2)	0.0156 (2)	0.0574 (8)
C(5)	0.56169 (7)	0.5865 (2)	0.0037 (2)	0.0552 (8)
C(6)	0.50917 (8)	0.5726 (2)	−0.1513 (2)	0.0571 (8)
C(7)	0.44817 (8)	0.4893 (2)	−0.1591 (2)	0.0556 (8)
C(8)	0.39464 (9)	0.4784 (2)	−0.3215 (2)	0.066 (1)
C(9)	0.33604 (9)	0.3925 (2)	−0.3318 (3)	0.069 (1)
C(10)	0.32550 (8)	0.3107 (2)	−0.1809 (3)	0.0637 (9)
C(11)	0.26507 (9)	0.2159 (2)	−0.1948 (3)	0.075 (1)
O(1)	0.56971 (7)	−0.0898 (2)	0.4471 (2)	0.0785 (8)
O(2)	0.66161 (7)	0.0575 (2)	0.4580 (2)	0.101 (1)
O(3)	0.47915 (8)	−0.2393 (2)	0.4528 (2)	0.1035 (9)
C(1*N*)	0.6081 (1)	0.0094 (3)	0.3675 (3)	0.072 (1)
C(2*N*)	0.57911 (8)	0.0456 (2)	0.1781 (2)	0.0557 (8)
C(3*N*)	0.61545 (8)	0.1402 (2)	0.0894 (3)	0.067 (1)
C(4*N*)	0.51398 (7)	−0.0183 (2)	0.0903 (2)	0.0483 (7)
C(5*N*)	0.47650 (8)	−0.1167 (2)	0.1773 (2)	0.0566 (8)
C(6*N*)	0.41284 (9)	−0.1753 (2)	0.0893 (3)	0.069 (1)
C(7*N*)	0.5066 (1)	−0.1556 (2)	0.3659 (3)	0.074 (1)

stacks of DBA and, in (I), the acceptor TNB molecules lie over and under the outer rings of DBA and form an approximately linear array parallel to the **a** direction of the cell.

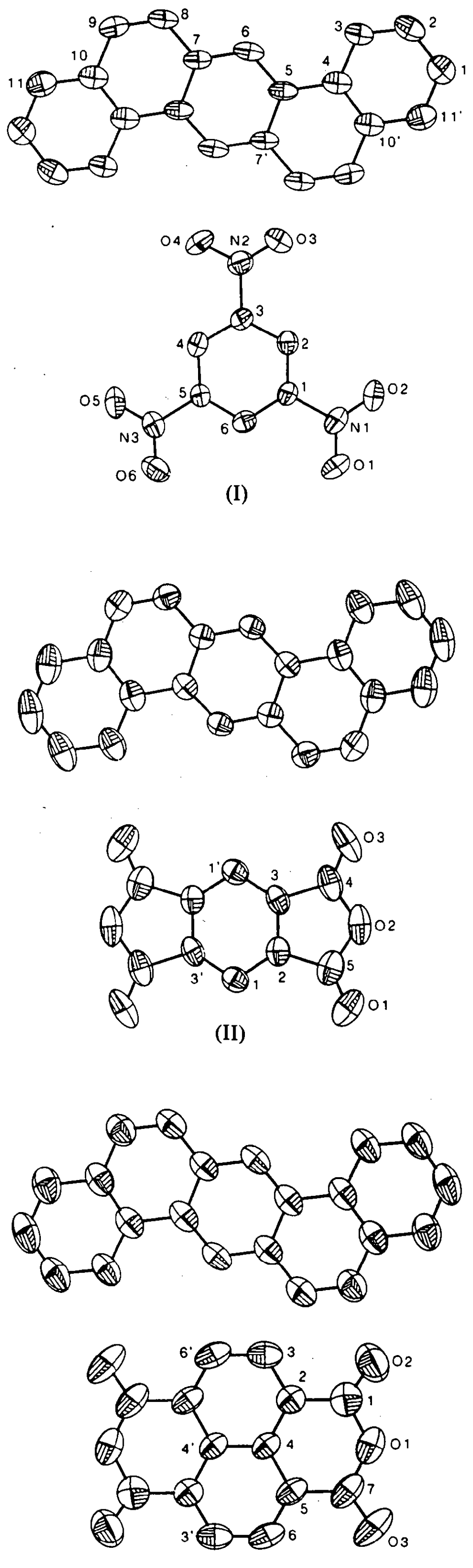


Fig. 1. Thermal ellipsoid (50%) representations (*ORTEP*; Johnson, 1965).

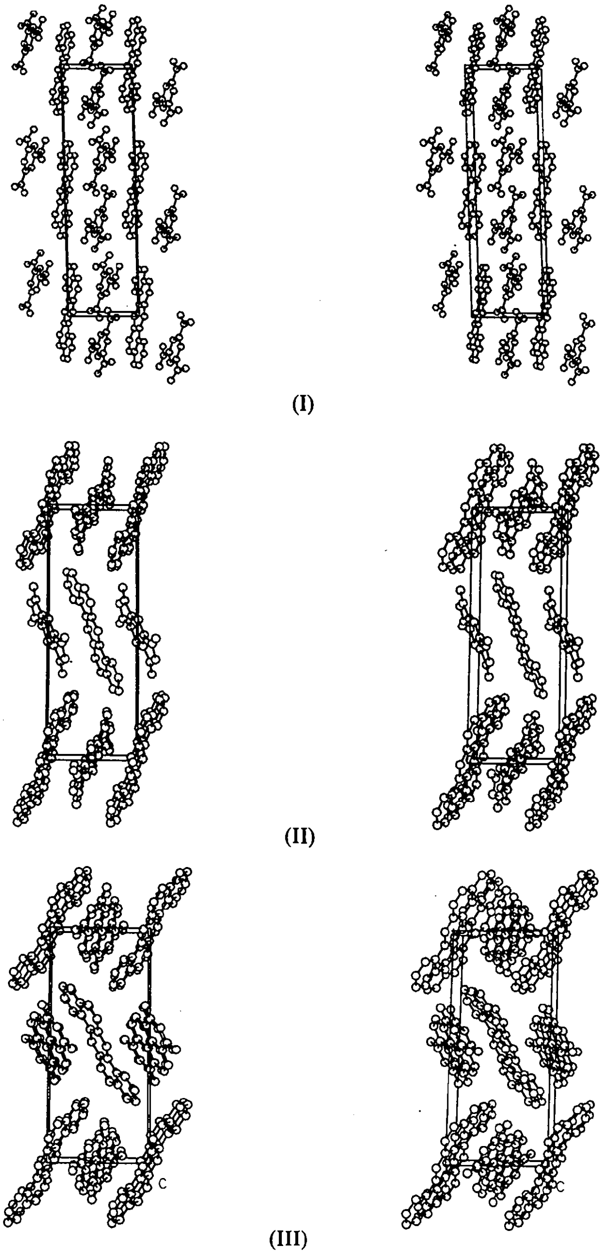
Table 3. *Bond lengths* (Å) *and bond angles* (°)

Values for the isolated molecule of DBA (IV) are from Iball, Morgan & Zacharias (1975). Symmetry relations for primed atoms: DBA: (I) 1 − *x*, 1 − *y*, − *z*; (II) 1 − *x*, 1 − *y*, 1 − *z*; (III) 1 − *x*, 1 − *y*, − *z*; (IV) − *x*, − *y*, − *z*; PMDA: 1 − *x*, 2 − *y*, 1 − *z*; NTDA: 1 − *x*, − *y*, − *z*.

	(I)	(II)	(III)	(IV)
DBA				
C(1)—C(2)	1.401 (3)	1.381 (3)	1.384 (3)	1.403 (2)
C(1)—C(11′)	1.374 (3)	1.369 (4)	1.359 (3)	1.358 (2)
C(2)—C(3)	1.375 (3)	1.380 (3)	1.372 (2)	1.372 (2)
C(3)—C(4)	1.415 (2)	1.413 (3)	1.404 (2)	1.407 (2)
C(4)—C(5)	1.455 (2)	1.455 (2)	1.462 (2)	1.454 (2)
C(4)—C(10′)	1.416 (2)	1.413 (2)	1.412 (2)	1.412 (2)
C(5)—C(6)	1.396 (2)	1.395 (2)	1.387 (2)	1.398 (2)
C(5)—C(7′)	1.436 (2)	1.428 (2)	1.428 (2)	1.424 (2)
C(6)—C(7)	1.389 (2)	1.392 (2)	1.390 (2)	1.392 (2)
C(7)—C(8)	1.442 (2)	1.438 (2)	1.435 (2)	1.438 (2)
C(8)—C(9)	1.341 (2)	1.338 (2)	1.357 (2)	1.338 (2)
C(9)—C(10)	1.444 (2)	1.439 (3)	1.425 (3)	1.436 (2)
C(10)—C(11)	1.405 (2)	1.408 (2)	1.423 (2)	1.413 (2)
C(2)—C(1)—C(11′)	119.6 (2)	119.9 (2)	121.1 (2)	119.9 (2)
C(1)—C(2)—C(3)	120.6 (2)	120.8 (2)	120.0 (2)	119.9 (2)
C(2)—C(3)—C(4)	121.2 (2)	120.6 (2)	121.2 (2)	121.6 (2)
C(3)—C(4)—C(5)	122.7 (1)	122.6 (1)	122.7 (2)	122.6 (2)
C(3)—C(4)—C(10′)	117.6 (1)	118.2 (1)	118.5 (1)	118.0 (2)
C(5)—C(4)—C(10′)	119.7 (1)	119.2 (2)	118.7 (1)	119.1 (2)
C(4)—C(5)—C(6)	123.8 (1)	123.4 (1)	123.1 (1)	123.3 (2)
C(4)—C(5)—C(7′)	119.2 (1)	119.1 (1)	119.1 (1)	119.5 (2)
C(6)—C(5)—C(7′)	117.0 (1)	117.5 (1)	117.8 (1)	117.3 (2)
C(5)—C(6)—C(7)	122.9 (1)	122.7 (1)	122.4 (1)	122.5 (2)
C(6)—C(7)—C(5′)	120.0 (1)	119.8 (1)	119.8 (1)	120.0 (2)
C(6)—C(7)—C(8)	121.4 (1)	120.9 (1)	121.0 (1)	120.9 (2)
C(8)—C(7)—C(5′)	118.6 (1)	119.2 (1)	119.3 (1)	118.9 (2)
C(7)—C(8)—C(9)	122.0 (1)	121.3 (2)	121.3 (2)	121.4 (2)
C(8)—C(9)—C(10)	121.4 (2)	121.6 (2)	120.9 (2)	121.6 (2)
C(9)—C(10)—C(11)	120.7 (2)	121.2 (2)	120.4 (2)	121.6 (2)
C(9)—C(10)—C(4′)	119.1 (1)	119.5 (2)	120.6 (1)	119.5 (2)
C(11)—C(10)—C(4′)	120.2 (1)	119.4 (2)	119.0 (2)	119.3 (2)
C(10)—C(11)—C(1′)	120.8 (2)	121.1 (2)	120.2 (2)	121.3 (2)

TNB					
O(1)—N(1)	1.215 (2)	O(2)—N(1)	1.214 (2)	O(3)—N(2)	1.223 (2)
O(4)—N(2)	1.219 (2)	O(5)—N(3)	1.221 (2)	O(6)—N(3)	1.206 (2)
N(1)—C(1*T*)	1.474 (2)	N(2)—C(3*T*)	1.467 (2)	N(3)—C(5*T*)	1.477 (2)
C(1*T*)—C(2*T*)	1.377 (2)	C(1*T*)—C(6*T*)	1.388 (2)	C(2*T*)—C(3*T*)	1.389 (2)
C(3*T*)—C(4*T*)	1.378 (2)	C(4*T*)—C(5*T*)	1.383 (2)	C(5*T*)—C(6*T*)	1.382 (2)
O(1)—N(1)—O(2)	124.2 (1)	O(3)—N(2)—O(4)	124.3 (1)	O(5)—N(3)—O(6)	124.4 (2)
O(1)—N(1)—C(1*T*)	117.5 (1)	O(2)—N(1)—C(1*T*)	118.3 (1)	O(3)—N(2)—C(3*T*)	117.6 (1)
O(4)—N(2)—C(3*T*)	118.1 (1)	O(5)—N(3)—C(5*T*)	117.9 (1)	O(6)—N(3)—C(5*T*)	117.8 (1)
N(1)—C(1*T*)—C(2*T*)	118.3 (1)	N(1)—C(1*T*)—C(6*T*)	118.1 (1)	C(1*T*)—C(2*T*)—C(3*T*)	116.7 (1)
N(2)—C(3*T*)—C(2*T*)	118.3 (1)	N(2)—C(3*T*)—C(4*T*)	118.6 (1)	C(2*T*)—C(3*T*)—C(4*T*)	123.2 (1)
N(3)—C(5*T*)—C(4*T*)	117.9 (1)	N(3)—C(5*T*)—C(6*T*)	118.5 (1)	C(3*T*)—C(4*T*)—C(5*T*)	116.8 (1)
C(4*T*)—C(5*T*)—C(6*T*)	123.5 (1)	C(5*T*)—C(6*T*)—C(1*T*)	116.3 (1)	C(6*T*)—C(1*T*)—C(2*T*)	123.6 (1)
PMDA					
O(1)—C(5*P*)	1.177 (2)	O(2)—C(5*P*)	1.398 (2)	O(2)—C(4*P*)	1.368 (2)
O(3)—C(4*P*)	1.194 (2)	C(1*P*)—C(2*P*)	1.384 (2)	C(1*P*)—C(3*P*′)	1.379 (2)
C(2*P*)—C(3*P*)	1.385 (2)	C(2*P*)—C(5*P*)	1.485 (2)	C(3*P*)—C(4*P*)	1.484 (2)
C(4*P*)—O(2)—C(5*P*)	110.4 (1)	C(2*P*)—C(1*P*)—C(3*P*′)	114.4 (1)	C(1*P*)—C(2*P*)—C(3*P*)	122.7 (1)
C(1*P*)—C(2*P*)—C(5*P*)	130.0 (2)	C(3*P*)—C(2*P*)—C(5*P*)	107.3 (1)	C(2*P*)—C(3*P*)—C(1*P*′)	123.0 (1)
C(4*P*)—C(3*P*)—C(1*P*′)	129.6 (1)	C(2*P*)—C(3*P*)—C(4*P*)	107.5 (1)	O(2)—C(4*P*)—O(3)	121.9 (1)
O(2)—C(4*P*)—C(3*P*)	107.8 (1)	O(3)—C(4*P*)—C(3*P*)	130.2 (2)	O(1)—C(5*P*)—O(2)	122.3 (2)
O(1)—C(5*P*)—C(2*P*)	130.7 (2)	O(2)—C(5*P*)—C(2*P*)	107.0 (1)		
NTDA					
O(1)—C(1*N*)	1.381 (2)	O(1)—C(7*N*)	1.371 (2)	O(2)—C(1*N*)	1.188 (2)
O(3)—C(7*N*)	1.200 (2)	C(2*N*)—C(3*N*)	1.375 (2)	C(2*N*)—C(4*N*)	1.409 (2)
C(1*N*)—C(2*N*)	1.475 (3)	C(4*N*)—C(5*N*)	1.400 (2)	C(5*N*)—C(6*N*)	1.370 (2)
C(4*N*)—C(4*N*′)	1.411 (2)	C(3*N*)—C(6*N*′)	1.396 (3)	C(5*N*)—C(7*N*)	1.477 (3)
C(1*N*)—O(1)—C(7*N*)	125.7 (1)	O(1)—C(1*N*)—O(2)	116.9 (2)	O(1)—C(1*N*)—C(2*N*)	117.1 (1)
O(2)—C(1*N*)—C(2*N*)	126.0 (2)	C(1*N*)—C(2*N*)—C(3*N*)	120.1 (1)	C(1*N*)—C(2*N*)—C(4*N*)	119.0 (1)
C(3*N*)—C(2*N*)—C(4*N*)	120.9 (2)	C(2*N*)—C(3*N*)—C(6*N*′)	119.7 (1)	C(2*N*)—C(4*N*)—C(4*N*′)	118.8 (1)
C(2*N*)—C(4*N*)—C(5*N*)	121.9 (1)	C(5*N*)—C(4*N*)—C(4*N*′)	119.3 (1)	C(4*N*)—C(5*N*)—C(6*N*)	120.6 (2)
C(4*N*)—C(5*N*)—C(7*N*)	118.9 (1)	C(6*N*)—C(5*N*)—C(7*N*)	120.5 (1)	C(5*N*)—C(6*N*)—C(3*N*′)	120.7 (2)
O(1)—C(7*N*)—O(3)	118.1 (2)	O(1)—C(7*N*)—C(5*N*)	117.5 (1)	O(3)—C(7*N*)—C(5*N*)	124.4 (2)

In the donor–acceptor pairs, the distances between pairs of parallel planes fitted through DBA C atoms and the acceptor atoms are: (I) 3.370, (II) 3.316, (III) 3.345 Å. The angles between the normals to the least-squares best planes through DBA C atoms and the C atoms of the acceptor molecules are 1.18, 1.06 and 0.48°, respectively. The angles between normals to the best planes through DBA molecules related by the screw-glide operation are 38.5, 48.4 and 71.4°, respectively. Fig. 3 shows the molecular overlap of each acceptor pair projected on the plane of DBA. Short intermolecular distances indicating attractive interactions are: (I) H(6*T*)···O(6) 2.42, H(2*T*)···O(4) 2.61 Å between trinitrobenzene molecules, and five others in the range 2.6–2.67 Å between DBA H atoms and TNB O atoms; (II) H(6)···O(3) 2.53 Å; (III) H(6)···O(3) 2.46 Å. Table 4 summarizes the geometry of these interactions.



(I)

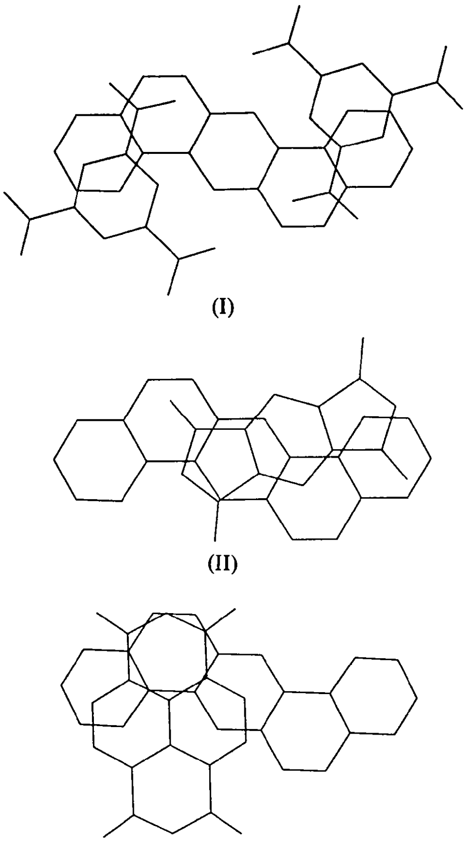
(II)

(III)

Fig. 2. Stereoscopic views of the molecular packing of (I) DBA–TNB (along *b*), (II) DBA–PMDA (along *c*) and (III) DBA–NTDA (along *c*).

The molecular geometry of DBA comports with that previously found in the isolated molecule (Iball, Morgan & Zacharias, 1975). Of particular note are the C(8)—C(9) and C(4)—C(5) bond lengths. The former shorter bond is the so-called K-region which is presumed to invite electrophilic attack by degradative enzymes of biological systems. The longer bond, C(4)—C(5), in the 'bay-region' of the phenanthrene portion of the molecule is accompanied by angles involving it that are 2.5–3.8° greater than 120°, indicating some H(3)···H(6) steric repulsive interaction. The distances between these are (I) 2.09, (II) 2.13 and (III) 1.97 Å. In the DBA structure determination (Iball, Morgan & Zacharias, 1975) this distance is 2.04 Å. The in-plane splaying of the angles is the major means of strain relief, as the greatest deviation of the torsion angles about the C(4)—C(5) bond from 0 or 180° is 2.2° [in (I)].

The molecular geometry of the TNB molecule is consistent with that previously found (Choi & Abel, 1972; Zacharias, Prout, Myers & Glusker, 1991). The PMDA and NTDA molecules both have geometry consonant with those reported (Robertson &



(I)

(II)

(III)

Fig. 3. Overlap of acceptor molecules projected on the plane of the DBA donor: (I) DBA–TNB, (II) DBA–PMDA, (III) DBA–NTDA.

Table 4. *Geometry of* (C)—H···O *interactions* (Å, °)

	C—H	H···O	C···O	C—H···O
(I)				
C(2*T*)—H(2*T*)···O(4^{i})	0.99 (2)	2.61 (2)	3.503 (2)	151 (2)
C(6*T*)—H(6*T*)···O(6^{ii})	0.99 (2)	2.42 (2)	3.401 (2)	174 (2)
(II)				
C(6)—H(6)···O(3^{iii})	0.94 (2)	2.53 (2)	3.399 (2)	153 (2)
(III)				
C(6)—H(6)···O(3^{iv})	1.00 (2)	2.46 (2)	3.382 (2)	153 (1)

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $1-x, 1-y, 2-z$; (iv) $x, y+1, z-1$.

Stezowski, 1978; Aravamudhan, Haeberlen, Irngartinger & Krieger, 1979; Born & Heywang, 1990). An interesting feature of the anhydride $O\overset{1}{=}C\overset{2}{-}O\overset{3}{-}C\overset{4}{=}O$ grouping is seen here: if bond 1 is longer than bond 4, then bond 2 is shorter than bond 3. This condition has been attributed to differences in the crystal-packing environments of the two carbonyl groups as seen in a low-temperature study of the PMDA–anthracene (1/1) complex (Robertson & Stezowski, 1978), and as is seen also in the low-temperature structures of PMDA and NTDA.

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The Monoclinic Phase of the 2/1 Inclusion Compound Between Deoxycholic Acid and *o*-Xylene

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Abstract. (3α,5β,12α)-3,12-Dihydroxycholan-24-oic acid–*o*-xylene (2/1), $2C_{24}H_{40}O_4.C_8H_{10}$, $M_r = 891.328$, monoclinic, $P2_1$, $a = 7.238$ (7), $b = 26.171$ (12), $c = 13.510$ (9) Å, $\beta = 90.91$ (9)°, $V = 2559$ (3) Å^3, $Z = 2$, $D_m = 1.16$, $D_x = 1.157$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 5.15$ cm^{-1}, $F(000) = 980$, $T = 293$ K, $R = 0.123$ ($wR = 0.153$) for 2662 observed reflections and 518 refined parameters. The crystals of the title compound belong to the monoclinic system. However, the presence of pseudo-twofold screw axes among the host molecules makes the structure of the bilayer, as well as the spatial relationships between them, similar to that of all so far known orthorhombic crystals. The guest molecules fill the space in

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