

Discussion. The atomic coordinates are listed in Table 1,* bond distances and bond angles are displayed in Figs. 1 and 2 for PMDA and Tcn. The bonding geometry of the PMDA molecule agrees well with that found for the acceptor in quite a number of 1:1 *D:A* complexes with donors such as anthracene (Robertson & Stezowski, 1978), phenazine (Bulgarovskaya, Vozzhennikov, Krasavin & Kotov, 1982), carbazole (Stezowski, Binder & Karl, 1982). Tcn bond distances and bond angles may be compared with the average values found in the free Tcn (Robertson, Sinclair & Trotter, 1961). Each of the component molecules is planar. The maximum deviations from the mean planes of the PMDA and Tcn molecules (neglecting H atoms) are 0.021 and 0.012 Å respectively.

The structure of the complex consists of columns of alternately stacked molecules of Tcn and PMDA. The *a* axis of the crystal is the stack axis. The molecular planes are parallel to each other and perpendicular to *a* (the angle between the plane normals is 1.7°, those between the normals to the Tcn and PMDA planes and the *a* axis are 0.7 and 1.5°, respectively), the interplanar separation being 3.36 Å. Because the stacks are not shifted with respect to one another along the *a* axis, the structure can also be described in terms of alternating sheets of Tcn and PMDA molecules. Adjacent Tcn and PMDA molecules are orientated in such a manner that the bonds of the Tcn molecule common to two rings lie over (or under) the centres of the rings of the neighbouring PMDA molecule, Fig. 3.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43514 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

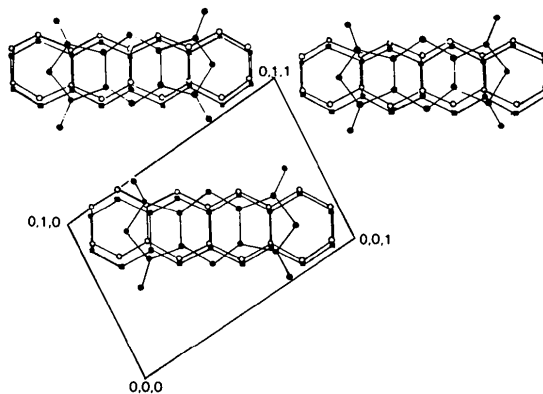


Fig. 3. Molecular packing in Tcn.PMDA viewed normal to the PMDA molecular plane.

References

- BULGAROVSKAYA, I. V., VOZZHENNIKOV, V. M., KRASAVIN, V. P. & KOTOV, B. V. (1982). *Cryst. Struct. Commun.* **11**, 501–504.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- ROBERTSON, B. E. & STEZOWSKI, J. J. (1978). *Acta Cryst.* **B34**, 3005–3011.
- ROBERTSON, J. M., SINCLAIRE, V. C. & TROTTER, J. (1961). *Acta Cryst.* **14**, 697–703.
- SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.
- SILINSH, E. A. (1980). *Organic Molecular Crystals.* Berlin: Springer Verlag.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- STEZOWSKI, J. J., BINDER, W. E. & KARL, N. (1982). *Acta Cryst.* **B38**, 2912–2914.

Acta Cryst. (1987). **C43**, 766–768

Structure of the 1:1 π -Molecular Complex of Chrysene with 1,2:4,5-Pyromellitic Dianhydride*

BY I. V. BULGAROVSKAYA, V. E. ZAVODNIK AND V. M. VOZZHENNIKOV

L. Ya. Karpov Physico-Chemical Institute, ul. Obukha, 10, Moscow, 107120, USSR

(Received 14 July 1986; accepted 10 November 1986)

Abstract. $C_{18}H_{12} \cdot C_{10}H_2O_6$, $M_r = 446.4$, monoclinic, $P2_1/n$, $a = 8.093$ (3), $b = 16.479$ (5), $c = 7.746$ (2) Å, $\gamma = 97.62$ (3)°, $V = 1023.9$ (6) Å³, $Z = 2$, $D_x =$

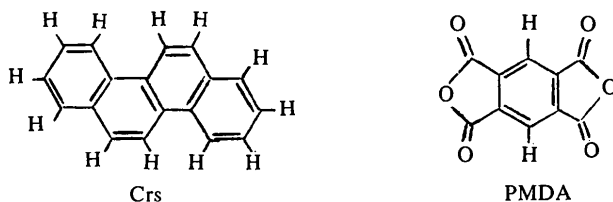
* 1,2:4,5-Pyromellitic dianhydride = 1*H*,3*H*-benzo[1,2-*c*:4,5-*c'*]-difuran-1,3,5,7-tetrone.

1.45 g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo}) = 0.96$ cm⁻¹, $F(000) = 460$, $T = 295$ K, $R = 0.041$ for 845 observed reflections [$I > 3\sigma(I)$]. Alternate planar centrosymmetric molecules form stacks along *c*. The mean separation of the molecular planes is 3.32 Å, the interplanar angle is 2.0°.

0108-2701/87/040766-03\$01.50

© 1987 International Union of Crystallography

Introduction. The title compound is a charge-transfer (CT) complex formed by pyromellitic dianhydride (PMDA) as an acceptor with chrysene (Crs). The crystals of the compound studied exhibit moderately high photoconductivity in the regions corresponding to the CT-absorption band and to the singlet transitions of the component molecules. We report here the results of the X-ray analysis undertaken as part of our investigations with the object of establishing the role of the crystal structure in photoconductivity.



Experimental. Prismatic red crystals $0.35 \times 0.25 \times 0.15$ mm grown by high-vacuum plate sublimation method. Syntex P1 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice parameters from 12 reflections, $\theta/2\theta$ scan mode, $2\theta < 48^\circ$, three standard reflections with variation within counter statistics. 991 unique reflections collected, 845 non-zero [$I > 3\sigma(I)$], range of hkl : $h \pm 9$, $k 0 \rightarrow 18$, $l 0 \rightarrow 8$; Lp correction, absorption ignored. Direct methods, refinement by full-matrix least squares on F . H positions calculated

Table 1. Fractional atomic coordinates (and *e.s.d.*'s) of non-hydrogen atoms ($\times 10^4$) and hydrogen atoms ($\times 10^3$), and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i a_j a_j$$

	x	y	z	U_{eq} Or U_{iso}
O(1)	6321 (3)	5604 (2)	1044 (4)	69 (1)
O(2)	7474 (4)	6744 (2)	-371 (4)	88 (1)
O(3)	5906 (3)	4358 (2)	2279 (4)	78 (1)
C(1)	9501 (4)	4225 (2)	853 (5)	49 (1)
C(2)	8498 (4)	4839 (2)	761 (5)	46 (1)
C(3)	8974 (4)	5578 (2)	-54 (5)	46 (1)
C(4)	6791 (5)	4847 (2)	1486 (6)	58 (2)
C(5)	7564 (5)	6072 (2)	133 (6)	62 (2)
C(6)	5794 (4)	78 (2)	349 (5)	50 (1)
C(7)	6313 (5)	877 (2)	1191 (5)	48 (1)
C(8)	7870 (5)	1077 (2)	1940 (5)	58 (1)
C(9)	8256 (6)	1795 (3)	2786 (6)	63 (2)
C(10)	7129 (6)	2358 (2)	2942 (5)	60 (2)
C(11)	5606 (6)	2200 (2)	2170 (6)	57 (2)
C(12)	5155 (4)	1446 (2)	1276 (5)	52 (1)
C(13)	3575 (5)	1258 (2)	484 (5)	57 (2)
C(14)	3089 (5)	520 (2)	-314 (5)	49 (1)
H(1)	910 (4)	374 (2)	142 (5)	47 (13)*
H(8)	855 (4)	56 (2)	172 (5)	78 (12)*
H(9)	928 (5)	192 (2)	330 (5)	75 (15)*
H(10)	737 (5)	286 (2)	364 (5)	67 (14)*
H(11)	487 (4)	254 (2)	227 (5)	59 (12)*
H(13)	271 (5)	169 (2)	56 (5)	98 (15)*
H(14)	200 (4)	30 (2)	-86 (5)	76 (13)*

* U_{iso} .

using geometric considerations: final $R = 0.041$, $wR = 0.047$, $w = 1/[\sigma^2(F) + 0.0005F^2]$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965). $(\Delta/\sigma)_{max} = 0.06$, $(\Delta\rho)_{max} = 0.10 \text{ e \AA}^{-3}$. Calculations carried out with *SHELXTL* (Sheldrick, 1978) on the Nova 3 computer incorporated in the Nicolet R3 crystallographic system.

Discussion. The atomic coordinates are listed in Table 1,* interatomic distances and bond angles are presented in Figs. 1 and 2. The dimensions of the PMDA molecule agree well with those in the complexes with anthracene (Robertson & Stezowski, 1978),

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43513 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

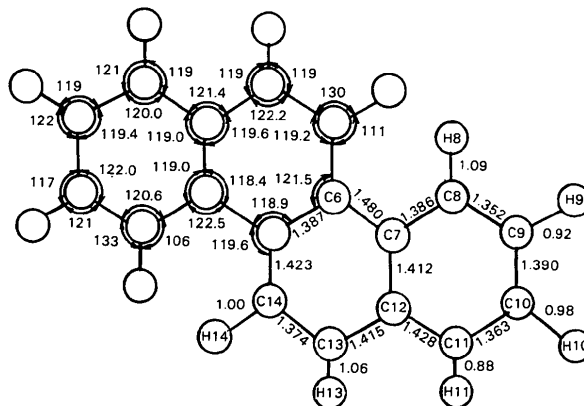


Fig. 1. Bond distances (\AA) and bond angles ($^\circ$) for Crs. *E.s.d.*'s in bond distances are 0.005–0.007 \AA , in bond angles 0.3–0.4 $^\circ$ (0.03–0.04 \AA for C–H bonds and 2–3 $^\circ$ for C–C–H angles).

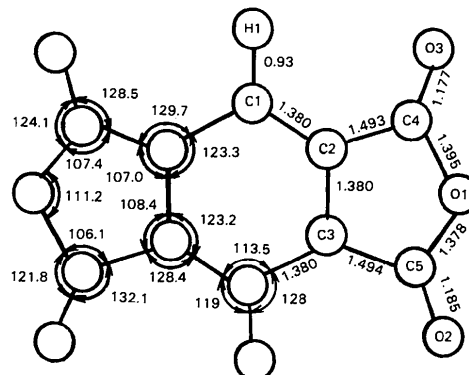


Fig. 2. Bond distances (\AA) and bond angles ($^\circ$) for PMDA. *E.s.d.*'s in bond distances are 0.005–0.007 \AA and in bond angles 0.3–0.4 $^\circ$ (0.03–0.04 \AA for C–H bonds and 2–3 $^\circ$ for C–C–H angles).

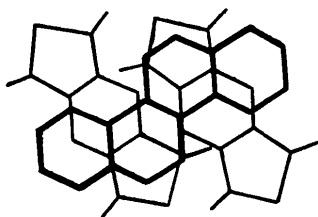


Fig. 3. Molecular overlapping diagram.

phenanthrene (Evans & Robinson, 1977), phenazine (Bulgarovskaya, Vozzhennikov, Krasavin & Kotov, 1982). The dimensions of the Crs molecule compare well with those of the free Crs (Burns & Iball, 1960) and of Crs in the complexes with 7,7,8-tetracyanoquinodimethane (Munnoch & Wright, 1974) and fluoranil (Munnoch & Wright, 1975). The PMDA molecule is planar, the maximum deviation from the least-squares best plane through the molecule being 0.008 Å. The Crs molecule is approximately planar. The individual rings are each planar; the maximum deviation from the best planes is 0.019 Å.

In the crystal the component molecules are arranged in mixed stacks extending along the *c* axis. The molecular planes are parallel to each other (the dihedral angle between the planes of the PMDA and Crs molecules is 2.0°) and make angles of 31.2 and 30.4° respectively with the *c* axis. The mean separation

between the PMDA and Crs planes is 3.32 Å. The overlap diagram is shown in Fig. 3. A particular PMDA molecule is located above one portion of the neighbouring Crs molecule; the next PMDA molecule in the stack takes up an analogous position and orientation with respect to the other centrosymmetric portion of the Crs molecule sandwiched between them, and so on along the stack.

References

- BULGAROVSKAYA, I. V., VOZZHENNIKOV, V. M., KRASAVIN, V. P. & KOTOV, B. V. (1982). *Cryst. Struct. Commun.* **11**, 501–504.
- BURNS, D. M. & IBALL, J. (1960). *Proc. R. Soc. London*, **257**, 491–514.
- EVANS, D. L. & ROBINSON, W. T. (1977). *Acta Cryst.* **B34**, 2891–2893.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- MUNNOCH, P. J. & WRIGHT, J. D. (1974). *J. Chem. Soc. Perkin Trans. 2*, pp. 1397–1400.
- MUNNOCH, P. J. & WRIGHT, J. D. (1975). *J. Chem. Soc. Perkin Trans. 2*, pp. 1071–1074.
- ROBERTSON, B. E. & STEZOWSKI, J. J. (1978). *Acta Cryst.* **B34**, 3005–3011.
- SHELDRIK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1987). **C43**, 768–770

Structure of the 1:1 π -Molecular Complex of 9,10-Dibromoanthracene with 1,2:4,5-Pyromellitic Dianhydride*

BY I. V. BULGAROVSKAYA, V. K. BELSKY AND V. M. VOZZHENNIKOV

L. Ya. Karpov Physico-Chemical Institute, ul. Obukha, 10, Moscow, 107120, USSR

(Received 14 July 1986; accepted 10 November 1986)

Abstract. $C_{14}H_8Br_2 \cdot C_{10}H_2O_6$, $M_r = 554.15$, monoclinic, $A2/m$, $a = 7.134$ (2), $b = 9.740$ (3), $c = 14.310$ (4) Å, $\gamma = 90.53$ (2)°, $V = 994.3$ (3) Å³, $Z = 2$, $D_x = 1.85$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 40.85$ cm⁻¹, $F(000) = 544$, $T = 295$ K, $R = 0.026$ for 755 observed reflections [$I > 3\sigma(I)$]. The crystal structure consists of stacks of alternating 9,10-dibromoanthracene and pyromellitic dianhydride molecules 3.36 Å apart. Both molecules are situated in special positions of the $2/m$ symmetry.

* 1,2:4,5-Pyromellitic dianhydride = 1*H*,3*H*-benzo[1,2-*c*:4,5-*c'*]-difuran-1,3,5,7-tetrone.

Introduction. In recent years a number of crystal structures of donor–acceptor (*D:A*) complexes based on pyromellitic dianhydride (PMDA) as an acceptor have been reported. The 1:1 complex between PMDA and anthracene was a first choice among a number of possible combinations with donor molecules. This complex possesses interesting physical properties extensively studied and characterized. Substituted anthracene-like materials such as 9,10-dibromoanthracene (DBA) are known to possess photoconductive and luminescence characteristics different from those of the parent hydrocarbon, anthracene (Smith & Bock, 1962). As physical properties of the complex