

LITTLE, R. G., PAUTLER, D. & COPPENS, P. (1971). *Acta Cryst.* B27, 1493–1499.  
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

SHELDRIK, G. M. (1976). *SHELX 76*. Program for crystal structure determination. Univ. of Cambridge, England.  
 SHIMANOCHI, H., ASHIDA, T., SASADA, Y. & KAKUDO, M. (1967). *Tetrahedron Lett.* 1, 61–67.  
 SHIONO, R. (1968). Private communication.  
 SHMUELI, U., SHANAN-ATIDI, H., HORWITZ, H. & SHVO, Y. (1973). *J. Chem. Soc. Perkin Trans. 2*, pp. 657–662.

*Acta Cryst.* (1982). B38, 859–863

## The Structure of the 1:1 Molecular Complex of Pyrene and Dicyanomethylenecroconate\*

BY R. M. DOHERTY† AND J. M. STEWART

*Chemistry Department, University of Maryland, College Park, Maryland 20742, USA*

AND A. D. MIGHELL, C. R. HUBBARD AND A. J. FATIADI

*National Measurement Laboratory, National Bureau of Standards, Washington, DC 20234, USA*

(Received 5 March 1981; accepted 23 September 1981)

### Abstract

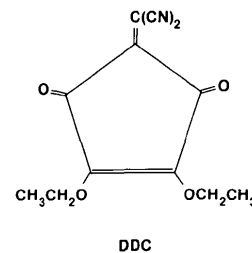
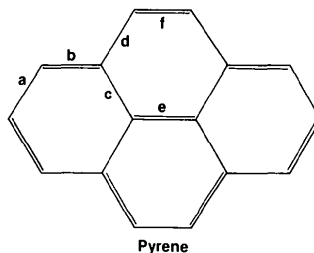
$C_{16}H_{10} \cdot C_{12}H_{10}N_2O_4$  is triclinic,  $P\bar{1}$ , with  $a = 10.085$  (3),  $b = 10.646$  (3),  $c = 11.037$  (3) Å,  $\alpha = 98.73$  (2),  $\beta = 92.61$  (2),  $\gamma = 107.36$  (2)°,  $Z = 2$ ,  $D_x = 1.339$ ,  $D_m = 1.35$  g cm<sup>-3</sup>,  $\mu$ (Cu  $K\alpha$ ) = 6.96 cm<sup>-1</sup>. Final  $R = 0.048$  for 2767 observed reflections. The compound, which is a 1:1 molecular complex of pyrene and dicyanomethylenecroconate, is formed upon evaporation of a solution containing equimolar amounts of the two substances. The structure of this charge-transfer complex consists of stacks of alternating donor and acceptor molecules.

### Introduction

Treatment of diethylcroconate in *N,N*-dimethylformamide with malononitrile produces golden plates of 2-(3,4-diethoxy-2,5-dioxo-3-cyclopenten-1-ylidene)-malononitrile (DDC) in 85% yield (Fatiadi, 1978*a*). DDC is an electron acceptor; for example, with polycyclic aromatic hydrocarbons such as pyrene, benz[*a*]pyrene, or anthracene it forms deeply colored charge-transfer complexes. This paper describes the crystal and molecular structure of a 1:1 molecular complex (hereafter referred to as P-DDC) formed from pyrene and DDC.

\* Dicyanomethylenecroconate is 2-(3,4-diethoxy-2,5-dioxo-3-cyclopenten-1-ylidene)malononitrile.

† From a dissertation submitted to the Graduate School, University of Maryland, in partial fulfillment of the requirements for a PhD degree in chemistry.



Ruby-red crystals of P-DDC (m.p. 441–443 K) were prepared by slow evaporation to dryness of a dichloromethane solution of an equimolar amount of pyrene and DDC (Fatiadi, 1978*a*). An elemental analysis of the product gave the following results. Calculated (found) for  $C_{28}H_{20}N_2O_4$ : C 74.99 (75.15), H 4.49 (4.68), N 6.25 (6.38)%. The mass spectrum showed only two peaks, one at  $m/e$  202 (pyrene) and one at  $m/e$  246 (DDC). The ultraviolet spectrum, recorded in dichloromethane, showed bands at  $\lambda_{max}$  of 439*sh* ( $\epsilon = 10\ 000$ ), 413 ( $\epsilon = 12\ 700$ ), 338.7 ( $\epsilon = 54\ 000$ ), 322.4 ( $\epsilon = 32\ 000$ ), 307 ( $\epsilon = 16\ 300$ ), and 295 nm ( $\epsilon = 8200$ ). The infrared spectrum (KBr) showed the following maxima: 2220*w* (C≡N), 1660*s* (C=O, C=C), 1640*w*, 1600*w*, 1552*s*, 1470*m*, 1429*m*, 1390*s*, 1360*m*, 1330*s*, 1235*m*, 1176*m*, 1136*s*, 1108*w*, 1075*w*, 1010*s*, 901*m*, 862*m*, 847*s*, 826*w*, 785*m*, 763*m*, 754*s*, 714*s* cm<sup>-1</sup>.

Data were collected on a crystal of dimensions 0.20 × 0.20 × 0.05 mm using an automated four-circle diffractometer with graphite-monochromated Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å. Cell dimensions were determined by a least-squares refinement of the setting angles of 15 reflections with  $2\theta$  values ranging between

8 and 33°. Using reduction techniques, the crystal system was established as triclinic. The centrosymmetric space group,  $P\bar{1}$ , was assigned on the basis of a successful refinement. Integrated diffraction intensities were measured in the range  $8.0 \leq 2\theta \leq 154.0^\circ$ . Standard reflections, which were measured periodically, showed no apparent decrease in intensity during data collection. The estimated standard deviation in intensity,  $\sigma(I)$ , was calculated from  $\sigma^2(I) = TC + 0.000108TC^2$  where TC is the total observed counts and the constant was derived from a statistical analysis of the intensity distributions of the four standard reflections. The data were corrected for Lorentz and polarization effects, but not for absorption. Of the 4014 unique reflections measured, 2767 had  $I \geq 3\sigma(I)$  and were subsequently used for the structure determination and refinement.

### Solution and refinement

The structure was solved by direct methods using *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Several attempts were required to solve the structure. In the first attempt, an *E* map was calculated that revealed only a fragment of one pyrene molecule. In the second attempt, a useful *E* map did not result even though an idealized pyrene molecule in a random orientation was assumed. In a third and successful attempt, an *E* map was calculated that revealed 31 of the 34 nonhydrogen atoms. To obtain this solution, an idealized pyrene (in the orientation deduced from the first *E* map) and an idealized fragment of DDC (in a random orientation) were assumed.

After refinement of the partial model with block-diagonal least-squares calculations, an  $F_o$  Fourier map was calculated from which the remaining three nonhydrogen atoms were located. In the final model, H atoms were assigned ideal calculated positions with C–H distances of 1.0 Å and fixed thermal parameters with  $U = 0.080 \text{ \AA}^2$ . For the H atoms in the methyl groups of DDC, the positions were assigned so that they are staggered (*i.e.* dihedral angles of approximately  $60^\circ$ ) with respect to the methylene H atoms. Anisotropic refinement of the nonhydrogen atoms resulted in a final  $R$  ( $= \sum |F_o| - |F_c| / \sum |F_o|$ ) of 0.048. In this refinement, the atomic parameters were blocked into three large groups: DDC and the two independent half-pyrene molecules. The function minimized was  $\sum w(|F_o| - |F_c|)^2$  where  $w = [\sigma(F_o)]^{-2}$ . The average and maximum shift divided by error were 0.084 and 0.369, respectively. Analysis of the final difference map revealed no peak greater than  $0.25 \text{ e \AA}^{-3}$ . The scattering factors used were those of Cromer & Mann (1968) for C, N, and O, and that of Stewart, Davidson & Simpson (1965) for H. All calculations (except

Table 1. *Positional and isotropic thermal parameters* ( $\text{\AA}^2$ ) *with estimated standard deviations in parentheses*

Equivalent isotropic temperature factors for the nonhydrogen atoms were calculated from  $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ .

	x	y	z	U
C(11)	0.80107 (23)	0.81496 (25)	0.05607 (24)	0.061 (2)
C(12)	0.76185 (22)	0.92368 (26)	0.08083 (23)	0.063 (2)
C(13)	0.84883 (22)	1.05122 (23)	0.06048 (28)	0.053 (1)
C(14)	0.81012 (27)	1.16748 (28)	0.08465 (24)	0.074 (2)
C(15)	0.89850 (30)	1.28720 (27)	0.06614 (26)	0.081 (2)
C(16)	1.02564 (28)	1.29682 (24)	0.02096 (25)	0.067 (2)
C(17)	1.06837 (22)	1.18222 (22)	-0.00749 (21)	0.051 (1)
C(18)	0.97955 (20)	1.05855 (20)	0.01293 (20)	0.044 (1)
C(21)	0.26857 (23)	-0.07983 (27)	0.58886 (24)	0.067 (2)
C(22)	0.69088 (23)	0.18753 (25)	0.43704 (25)	0.064 (2)
C(23)	0.56168 (23)	0.18208 (22)	0.49028 (22)	0.053 (1)
C(24)	0.51471 (28)	0.29309 (25)	0.51867 (26)	0.069 (2)
C(25)	0.38990 (29)	0.28145 (28)	0.56907 (27)	0.079 (2)
C(26)	0.30758 (26)	0.16124 (28)	0.59238 (25)	0.072 (2)
C(27)	0.34954 (22)	0.04765 (24)	0.56627 (22)	0.054 (1)
C(28)	0.47831 (20)	0.05754 (21)	0.51402 (21)	0.046 (1)
C(31)	0.24383 (20)	1.15536 (19)	0.27446 (20)	0.041 (1)
C(32)	0.36474 (20)	1.14604 (20)	0.22524 (21)	0.042 (1)
C(33)	0.36745 (20)	1.00756 (20)	0.20761 (20)	0.045 (1)
C(34)	0.23482 (19)	0.92815 (19)	0.24819 (20)	0.041 (1)
C(35)	0.15589 (19)	1.02175 (19)	0.29001 (20)	0.042 (1)
C(312)	0.08730 (22)	1.27255 (21)	0.35391 (23)	0.053 (1)
C(313)	0.09431 (27)	1.41687 (24)	0.37808 (29)	0.081 (2)
C(322)	0.46871 (24)	1.37322 (22)	0.19823 (25)	0.060 (2)
C(323)	0.59933 (29)	1.44868 (26)	0.15162 (30)	0.088 (2)
C(341)	0.19143 (21)	0.79625 (20)	0.24705 (21)	0.046 (1)
C(3411)	0.05890 (23)	0.72771 (20)	0.28676 (22)	0.053 (1)
C(3413)	0.27280 (23)	0.70960 (21)	0.20437 (22)	0.055 (1)
N(3412)	-0.04437 (21)	0.66550 (19)	0.31601 (22)	0.073 (1)
N(3414)	0.32822 (24)	0.63451 (21)	0.17209 (22)	0.086 (2)
O(311)	0.21652 (14)	1.26939 (13)	0.30243 (15)	0.056 (1)
O(321)	0.47215 (14)	1.23646 (14)	0.19304 (15)	0.054 (1)
O(331)	0.45949 (15)	0.96629 (15)	0.16764 (16)	0.064 (1)
O(351)	0.04212 (14)	0.99105 (14)	0.32864 (16)	0.059 (1)

*MULTAN*) were performed with the XRAY system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Table 1\* lists the final atomic parameters.

### Discussion

The unit cell contains two crystallographically independent pyrene molecules, each of which lies on a center of symmetry, and two DDC molecules. Together they form a 1:1 charge-transfer complex.

#### (a) *The donor: pyrene*

Bond distances and angles for pyrene are given in Fig. 1. The bond lengths found in the two pyrene molecules agree with those reported in the literature (Table 2). Least-squares planes were calculated through the pyrene molecules. For pyrene I, the maximum deviation from the plane is  $0.017$  (4) Å for

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36432 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(14) and the average deviation is 0.007 Å. For pyrene 2, the maximum deviation from the plane is 0.005 (4) Å for C(26) and the average deviation is 0.002 Å. To within the limits of error, the two pyrene molecules may be considered equally planar.

(b) *The acceptor: DDC*

Bond distances and angles for DDC are given in Fig. 2. The C(31)–C(32) bond distance in DDC is 1.381 (3) Å which is significantly longer than the equivalent distances in other molecules containing the O=C–C=C–C=O conjugated system (Table 3). The lengthening of this bond can be attributed to the resonance structures shown in Fig. 3. These resonance structures would be expected to cause a contraction of the C–C single bond and a lengthening of the carbonyl bond. The data in Table 3 show that the C–C single bond is the shortest while the C=O bond is of intermediate length.

Bond distances in the dicyanomethylene group in DDC can be compared to corresponding distances in this group in TCNQ [see Table 6 in Himes, Mighell, Hubbard & Fatiadi (1980) for a summary of data on

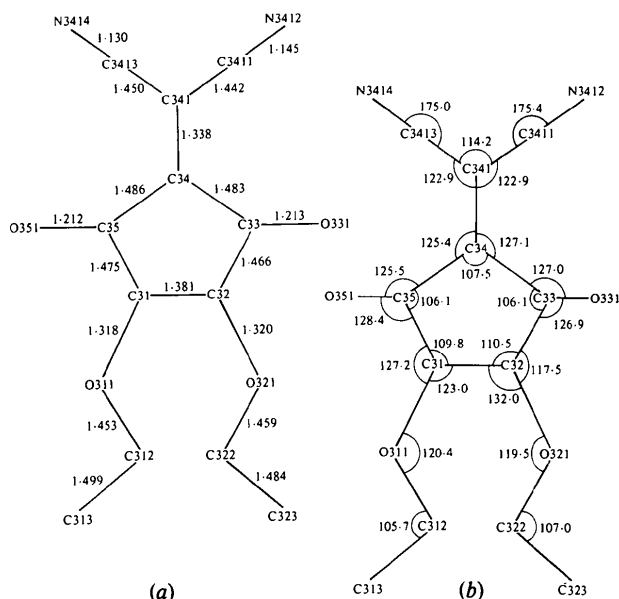


Fig. 2. Bond distances (Å) and angles (°) in DDC. The e.s.d.'s in the distances range between 0.003 and 0.004 Å and those in the angles between 0.2 and 0.3°.

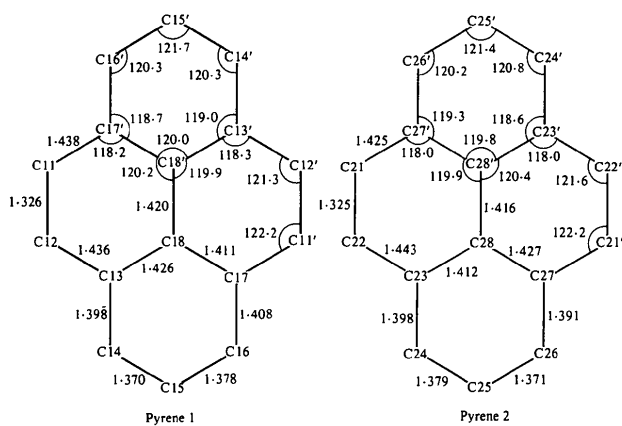


Fig. 1. Bond distances (Å) and angles (°) in pyrene. The e.s.d.'s in the distances range between 0.003 and 0.004 Å and those in the angles between 0.2 and 0.3°.

Table 2. Average distances for chemically equivalent bonds in pyrene–DDC, in pyrene and in pyrene–PMDA

Bond*	Pyrene–DDC	Pyrene	Pyrene–PMDA
a	1.374 (3)†	1.386 (1)‡	1.403 (6)§
b	1.399 (4)	1.402 (2)	1.394 (5)
c	1.419 (5)	1.422 (2)	1.430 (2)
d	1.436 (5)	1.436 (1)	1.445 (3)
e	1.418 (3)	1.427 (2)	1.426 (1)
f	1.326 (1)	1.353 (2)	1.352 (2)

\* See figure in *Introduction* for identification of bonds.  
 † Average of equivalent bonds in pyrene 1 and pyrene 2.  
 ‡ Kai, Hama, Yasuoka & Kasai (1978).  
 § Herbstein & Snyman (1969).

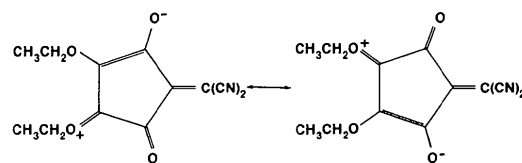


Fig. 3. Possible resonance contributions to DDC.

Table 3. Bond distances (Å) in some conjugated systems

Compound	C=C	C–C	C=O
DDC	1.381 (3) Å	1.470 (2) Å	1.212 (2) Å
4-Cyclopentene-1,3-dione <sup>a</sup>	1.341 (5)	1.493 (5)	1.208 (2)
Fumaldehyde <sup>b</sup>	1.337 (5)	1.480 (3)	1.207 (3)
<i>p</i> -Benzoquinone <sup>c</sup>	1.344 (3)	1.481 (2)	1.225 (2)

References: (a) Hagen & Hedberg (1978a). (b) Paulen & Trætberg (1975). (c) Hagen & Hedberg (1978b).

this group in a series of TCNQ complexes]. Inspection of the table shows that in TCNQ the double bond in the dicyanomethylene moiety is longer and the single bond shorter than for this group in DDC.

The acceptor, DDC, may also be viewed as a neutral pseudo-oxocarbon (Fatiadi, 1978b, 1980a,b) formed by substituting a dicyanomethylene group for a carbonyl O atom. As such, it may be compared with the five-membered ring in 1,2,3-indantrione (triketo-indane) TKI (Bolton, 1965) which is shown in Fig. 4. Bolton postulates a contribution of about 7% from the resonance forms *A* and *B*. In TKI the C(1)–C(2) bond distance is 1.392 Å, and the C(2)–C(3) and C(1)–

C(5) bond distances are each 1.478 Å. In DDC, the equivalent bond distances are 1.381 (3), 1.466 (3) and 1.475 (3) Å, respectively. This similarity in bond distances supports the hypothesis of contributions from the resonance forms for DDC shown in Fig. 3.

(c) *The complex*

The structure of P-DDC consists of stacks of alternating donor and acceptor molecules (Fig. 5). Fig. 6 shows the projection of DDC onto the planes of the two pyrene molecules adjacent to it in the stack. The planes of the projections are defined by atoms C(13), C(17), and C(18) for pyrene 1 and by atoms C(23), C(27) and C(28) for pyrene 2.

The least-squares planes of the molecules are not quite parallel to one another. The angles formed by the intersection of the planes are 1.9° for pyrene 1 and DDC, 3.5° for pyrene 2 and DDC, and 2.6° for pyrene 1 and pyrene 2. Such nonparallelism of the molecules in other donor-acceptor complexes has been cited as evidence of specific localized interaction between the donor and acceptor molecules. For

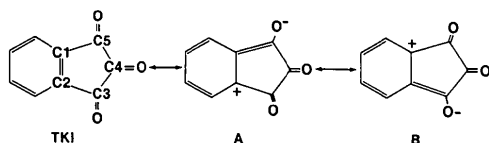


Fig. 4. Resonance contributions to 1,2,3-indantrione.

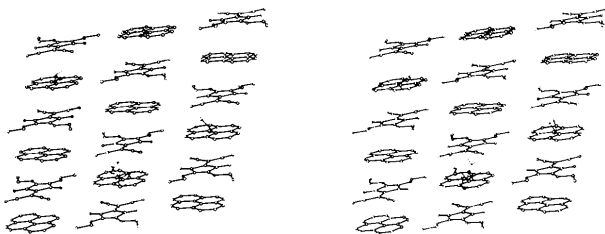


Fig. 5. Stereoview of the packing in P-DDC.

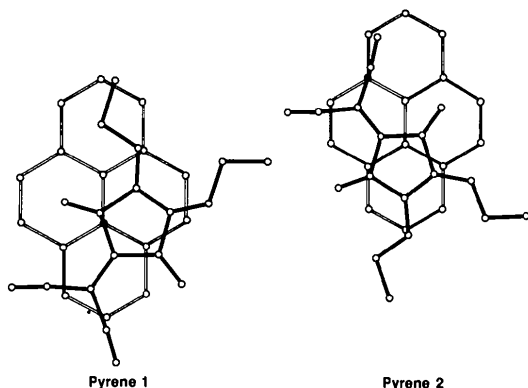


Fig. 6. Projections of DDC onto the two pyrene molecules.

example, in perylene-fluoranil, the individual molecules are planar, but they are inclined toward one another at an angle of 1.8° (Hanson, 1963). In perylene-PMDA (PMDA is pyromellitic dianhydride), it is the anhydride parts of the acceptor molecule that are bent out of the plane of the benzene ring in PMDA and, hence, are inclined toward the perylene molecules (Boeyens & Herbstein, 1965). Similarly, in the pyrene-PMDA molecular complex, the angles of inclination between the two independent pyrene molecules and the PMDA molecules are 1.3° and 0.0° (Herbstein & Snyman, 1969). Again the anhydride portions of the PMDA molecules are distorted from the plane of the central benzene ring.

The closest interplanar contact between C atoms in pyrene found by Camerman & Trotter (1965) was 3.55 Å. If this is taken as an indication of the normal van der Waals separation, the interplanar contacts closer than 3.55 Å may reflect some charge-transfer interaction. For P-DDC, all interplanar contacts less than 3.5 Å are given in Table 4. These data suggest two modes of charge-transfer interaction between pyrene and DDC: (1) a highly localized interaction involving the O atom in one of the ethoxy groups and (2) interaction of the double bond of the acceptor with the aromatic ring in the donor. The former (consistent with the resonance structures in Fig. 3) is supported by the proximity [3.319 (4) Å] of C(25) and O(311). The latter is supported by the close approach of pyrene 1 to the dicyanomethylene end of DDC [C(15)⋯C(3413) = 3.391 (4) Å]. The orientation of the dicyanomethylene group with respect to pyrene 1 is equivalent to that reported (Kuroda, Ikemoto & Akamatu, 1966) for a pyrene-TCNE complex. However, the donor-acceptor interatomic distances are slightly smaller in the TCNE complex and the average distance of the two atoms in the double bond of the dicyanomethylene group [C(34), C(3411)] from the plane of the pyrene molecule is less in pyrene-TCNE (3.32 vs 3.36 Å). Thus, although this second mode of interaction is probably the same in both complexes, the strength would be slightly greater in pyrene-TCNE.

Table 4. Shortest intermolecular contacts between pyrene 1 and DDC, and pyrene 2 and DDC (Å)

Pyrene 1 DDC		Pyrene 2 DDC	
C(15)⋯C(3413 <sup>i</sup> )	3.391 (4)	C(25)⋯O(311 <sup>ii</sup> )	3.319 (4)
C(16)⋯C(3411 <sup>i</sup> )	3.415 (4)	C(23)⋯C(32 <sup>ii</sup> )	3.365 (3)
C(16)⋯C(341 <sup>i</sup> )	3.428 (4)	C(25)⋯C(31 <sup>ii</sup> )	3.406 (4)
C(18)⋯C(35 <sup>i</sup> )	3.434 (3)	C(28)⋯C(33 <sup>ii</sup> )	3.424 (3)
C(12)⋯C(32 <sup>i</sup> )	3.446 (3)	C(24)⋯C(32 <sup>ii</sup> )	3.439 (4)
C(15)⋯C(341 <sup>i</sup> )	3.444 (4)	C(27)⋯C(35 <sup>ii</sup> )	3.474 (3)
C(13)⋯C(33 <sup>i</sup> )	3.456 (3)	C(27)⋯O(331 <sup>iii</sup> )	3.485 (3)

Symmetry code: (i) 1 - x, 2 - y, -z; (ii) x, y - 1, z; (iii) 1 - x, 1 - y, 1 - z.

Fig. 6 shows the orientation of the double bond of the dicyanomethylene group in DDC with respect to pyrene 1 and pyrene 2. In both cases the double bond is roughly centered over an aromatic ring. However, in the case of pyrene 2 there is no interatomic approach involving the double bond in the dicyanomethylene group and pyrene 2 less than 3.5 Å. Therefore one may conclude that the dicyanomethylene-pyrene interaction is much weaker for pyrene 2 than for pyrene 1.

### Conclusion

DDC is capable of behaving as an electron acceptor in complexes in the same way as TCNE. There is, however, another way in which DDC can interact with an electron donor, *viz.* through an O atom in one of the two ethoxy groups. In P-DDC the latter appears to be the stronger interaction.

The computer facilities for this project were provided in part by the Computer Science Center of the University of Maryland. We wish to thank T. Blizzard and R. Wood for their technical assistance.

### References

- BOEYENS, J. C. A. & HERBSTEIN, F. H. (1965). *J. Chem. Phys.* **69**, 2160.  
 BOLTON, W. (1965). *Acta Cryst.* **18**, 5–10.  
 CAMERMAN, A. & TROTTER, J. (1965). *Acta Cryst.* **18**, 636–643.

*Acta Cryst.* (1982). **B38**, 863–867

## Structure du (16βH)-Δ14-Vincanol

PAR NOËL RODIER

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, Rue J.-B. Clément,  
 92290 Chatenay-Malabry, France

ET SALAH BAASSOU, HACHEM MEHRI ET MICHEL PLAT

Laboratoire de Pharmacie chimique, Faculté des Sciences pharmaceutiques et biologiques, Rue J.-B. Clément,  
 92290 Chatenay-Malabry, France

(Reçu le 21 avril 1980, accepté le 25 septembre 1981)

### Abstract

C<sub>19</sub>H<sub>22</sub>N<sub>2</sub>O is orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, with *a* = 10.285 (8), *b* = 16.30 (2), *c* = 18.38 (2) Å, *Z* = 8, *D*<sub>c</sub> = 1.268 Mg m<sup>-3</sup>. The structure was solved by direct methods and refined to a final *R* value of 0.047 for 2497 independent reflections. H atoms were located

0567-7408/82/030863-05\$01.00

- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.  
 FATIADI, A. J. (1978a). *Synthesis*, **165**, 241–282.  
 FATIADI, A. J. (1978b). *J. Am. Chem. Soc.* **100**, 2586–2587.  
 FATIADI, A. J. (1980a). *J. Org. Chem.* **45**, 1338–1339.  
 FATIADI, A. J. (1980b). *Oxocarbons*, edited by R. WEST, ch. 4. New York: Academic Press.  
 HAGEN, K. & HEDBERG, K. (1978a). *J. Mol. Struct.* **44**, 195–202.  
 HAGEN, K. & HEDBERG, K. (1978b). *J. Chem. Phys.* **59**, 158–162.  
 HANSON, A. W. (1963). *Acta Cryst.* **16**, 1147–1151.  
 HERBSTEIN, F. H. & SNYMAN, J. A. (1969). *Philos. Trans. R. Soc. London Ser. A*, **264**, 635–662.  
 HIMES, V. L., MIGHELL, A. D., HUBBARD, C. R. & FATIADI, A. J. (1980). *J. Res. Natl Bur. Stand. US*, **85**, 87–97.  
 KAI, Y., HAMA, F., YASUOKA, N. & KASAI, N. (1978). *Acta Cryst.* **B34**, 1263–1270.  
 KURODA, H., IKEMOTO, I. & AKAMATU, H. (1966). *Bull. Chem. Soc. Jpn*, **39**, 547–551.  
 MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN 77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.  
 PAULEN, G. & TRÆTTEBERG, M. (1975). *Acta Chem. Scand. Ser. A*, **28**, 1155–1161.  
 STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY system – version of March 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

and their positional parameters refined. The structure consists of layers which run parallel with the (001) plane and its cohesion is due to hydrogen bonds and van der Waals interactions. The ring conformations are sofa for *C* and *E*(1), half-chair for *D* and chair for *E*(2). The ring-junction configurations are planar for *A/B*, *B/C* and *B*(1)/*E*(1), *cis* for *C/D* and *D/E*, *trans* for

© 1982 International Union of Crystallography