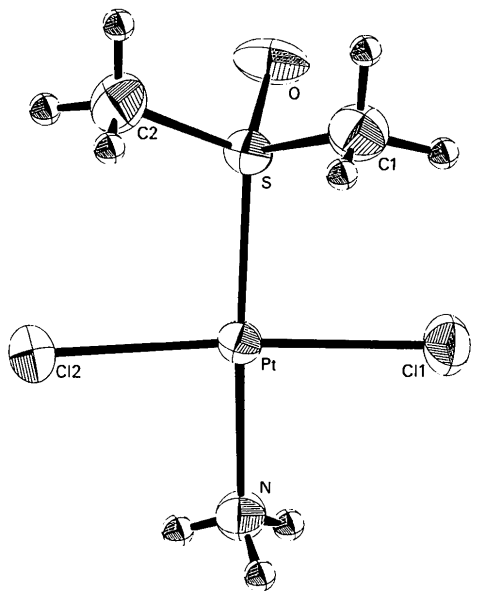

Fig. 1. Dessin de la molécule vue en perspective et noms utilisés pour désigner ses atomes.

liaisons dans le Tableau 2. La Fig. 1 représente la molécule et la Fig. 2 une vue stéréoscopique de la structure. Les numéros attribués aux atomes sont indiqués sur la Fig. 1.

Littérature associée. Structure du diméthylsulfoxyde (Thomas, Shoemaker & Ericks, 1966). Structure du *cis*-amminedichloro(diméthylsulfoxyde)platine(II) (Melanson & Rochon, 1978*a*). Structure du *trans*-dichloro(diméthylsulfoxyde)(picoline-2)platine(II) (Melanson & Rochon, 1978*b*). Structure du chlorure de *trans*-diamminechloro(diméthylsulfoxyde)platine-(II) (Delafontaine, Khodadad, Toffoli & Rodier, 1985). Structure du bis[trichloro(diméthylsulfoxyde)-platinate(II)] de tétraammineplatine(II) (Khodadad & Rodier, 1987).

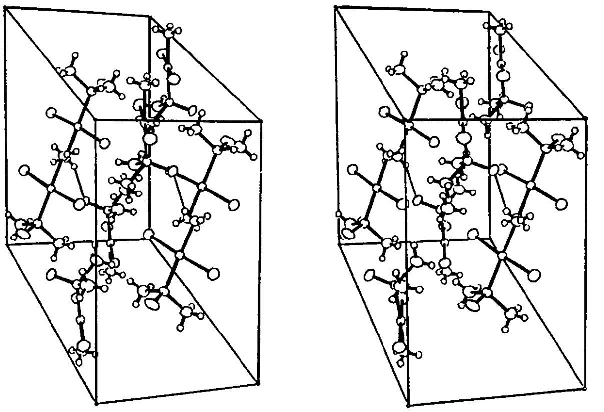

Fig. 2. Vue stéréoscopique de la structure. *x* est dirigé de l'arrière vers l'avant, *y* est horizontal et dirigé de gauche à droite et *z* est vertical ascendant.

Références

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, EU, et Enraf–Nonius, Delft, Pays-Bas.

Delafontaine, J.-M., Khodadad, P., Toffoli, P. & Rodier, N. (1985). *Acta Cryst.* **C41**, 702–703.

Johnson, C. K. (1976). *ORTEP*II. Rapport ORNL-5138. Oak Ridge National Laboratory, Tennessee, EU.

Khodadad, P. & Rodier, N. (1987). *Acta Cryst.* **C43**, 1690–1692.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. de York, Angleterre, et de Louvain, Belgique.

Melanson, R. & Rochon, F. D. (1978*a*). *Acta Cryst.* **B34**, 941–948.

Melanson, R. & Rochon, F. D. (1978*b*). *Acta Cryst.* **B34**, 1125–1127.

Thomas, R., Shoemaker, C. B. & Ericks, K. (1966). *Acta Cryst.* **21**, 12–20.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 159–166.

Acta Cryst. (1991). **C47**, 1317–1319

Structure of 11,12-Dibenzoyldibenzobarrelene*

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Abstract. $C_{30}H_{20}O_2$, $M_r = 412{\cdot}49$, monoclinic, Cc, $a = 15{\cdot}764\,(5)$, $b = 9{\cdot}202\,(2)$, $c = 15{\cdot}562\,(3)$ Å, $\beta = 106{\cdot}26\,(2)°$, $V = 2167\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}264$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70930$ Å, $\mu = 0{\cdot}7$ cm^{-1}, $F(000) = 864$, $T = 295$ K, $R = 0{\cdot}030$ for 777 reflections. The two carbonyl groups are rotated

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* 11,12-Dibenzoyl-9,10-dihydro-9,10-ethenoanthracene.

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Table 1. *Positional (fractional* × 10^4) *and equivalent isotropic* (U × 10^3 Å^2) *thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}$(trace of diagonalized U tensor).

	x	y	z	U_{eq}
C1	−978 (7)	384 (9)	−713 (7)	50
C2	−1582 (8)	182 (10)	−219 (8)	66
C3	−1707 (8)	1284 (11)	335 (8)	63
C4	−1233 (7)	2566 (9)	426 (8)	56
C4a	−624 (6)	2712 (9)	−56 (7)	41
C5	1601 (7)	3744 (10)	1046 (7)	59
C6	2402 (8)	2998 (13)	1226 (8)	72
C7	2510 (8)	1907 (12)	650 (10)	75
C8	1845 (8)	1529 (10)	−107 (8)	61
C8a	1052 (7)	2266 (8)	−270 (7)	40
C9	234 (7)	1985 (8)	−1058 (7)	41
C9a	−488 (7)	1639 (9)	−623 (7)	39
C10	0	3999 (8)	0	46
C10a	931 (7)	3364 (9)	292 (7)	43
C11	−2 (7)	3463 (9)	−1516 (7)	42
C12	−123 (7)	4501 (8)	−963 (6)	39
C13	−94 (7)	3681 (9)	−2484 (7)	43
C14	543 (8)	2966 (8)	−2891 (7)	43
C15	272 (7)	2457 (9)	−3761 (8)	55
C16	878 (10)	1840 (10)	−4136 (7)	70
C17	1755 (9)	1721 (10)	−3657 (9)	70
C18	2020 (8)	2210 (11)	−2781 (9)	69
C19	1413 (8)	2836 (10)	−2396 (7)	59
C20	−368 (7)	6034 (9)	−1233 (7)	48
C21	216 (8)	6914 (8)	−1630 (7)	47
C22	1112 (9)	6701 (9)	−1355 (8)	61
C23	1670 (8)	7562 (12)	−1686 (8)	77
C24	1319 (12)	8584 (13)	−2322 (10)	92
C25	436 (11)	8771 (11)	−2619 (9)	85
C26	−131 (8)	7954 (10)	−2267 (7)	65
O1	−700 (6)	4437 (6)	−2939 (6)	61
O2	−1031 (6)	6565 (7)	−1093 (6)	71

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C1—C2	1·394 (11)	C11—C13	1·486 (10)
C1—C9a	1·375 (9)	C12—C20	1·492 (10)
C2—C3	1·380 (12)	C13—C14	1·483 (11)
C3—C4	1·383 (11)	C13—O1	1·232 (8)
C4—C4a	1·380 (10)	C14—C15	1·382 (10)
C4a—C9a	1·379 (9)	C14—C19	1·376 (10)
C4a—C10	1·526 (9)	C15—C16	1·373 (11)
C5—C6	1·396 (12)	C16—C17	1·378 (13)
C5—C10a	1·385 (10)	C17—C18	1·385 (13)
C6—C7	1·387 (13)	C18—C19	1·388 (12)
C7—C8	1·384 (13)	C20—C21	1·484 (11)
C8—C8a	1·382 (11)	C20—O2	1·227 (8)
C8a—C9	1·533 (10)	C21—C22	1·371 (11)
C8a—C10a	1·384 (9)	C21—C26	1·376 (10)
C9—C9a	1·512 (9)	C22—C23	1·387 (12)
C9—C11	1·533 (9)	C23—C24	1·365 (14)
C10—C10a	1·526 (10)	C24—C25	1·349 (15)
C10—C12	1·527 (9)	C25—C26	1·392 (14)
C11—C12	1·335 (9)		

C2—C1—C9a	120·4 (8)	C9—C11—C13	122·2 (7)
C1—C2—C3	119·0 (8)	C12—C11—C13	124·5 (7)
C2—C3—C4	121·5 (8)	C10—C12—C11	114·5 (7)
C3—C4—C4a	117·8 (8)	C10—C12—C20	121·0 (7)
C4—C4a—C9a	122·3 (7)	C11—C12—C20	124·5 (7)
C4—C4a—C10	125·4 (8)	C11—C13—C14	119·2 (7)
C9a—C4a—C10	112·3 (7)	C11—C13—O1	119·8 (8)
C6—C5—C10a	118·6 (9)	C14—C13—O1	121·0 (7)
C5—C6—C7	119·6 (9)	C13—C14—C15	120·5 (8)
C6—C7—C8	122·1 (9)	C13—C14—C19	119·2 (8)
C7—C8—C8a	117·6 (8)	C15—C14—C19	120·3 (8)
C8—C8a—C9	125·4 (8)	C14—C15—C16	119·7 (8)
C8—C8a—C10a	121·4 (8)	C15—C16—C17	121·0 (9)
C9—C8a—C10a	113·3 (7)	C16—C17—C18	119·0 (9)
C8a—C9—C9a	104·3 (6)	C17—C18—C19	120·4 (9)
C8a—C9—C11	105·2 (6)	C14—C19—C18	119·6 (8)
C9a—C9—C11	106·3 (6)	C12—C20—C21	119·4 (8)
C1—C9a—C4a	118·9 (7)	C12—C20—O2	119·5 (8)
C1—C9a—C9	127·0 (8)	C21—C20—O2	121·0 (8)
C4a—C9a—C9	114·0 (7)	C20—C21—C22	119·8 (8)
C4a—C10—C10a	105·7 (6)	C20—C21—C26	120·7 (9)
C4a—C10—C12	106·0 (6)	C22—C21—C26	119·4 (9)
C10a—C10—C12	104·8 (6)	C21—C22—C23	120·5 (9)
C5—C10a—C8a	120·8 (8)	C22—C23—C24	119·5 (10)
C5—C10a—C10	126·7 (8)	C23—C24—C25	120·4 (11)
C8a—C10a—C10	112·5 (7)	C24—C25—C26	120·7 (10)
C9—C11—C12	113·2 (7)	C21—C26—C25	119·3 (9)

out of the C11=C12 plane; torsion angles C=C—C=O are −41 (1)° at C11 [synclinal, cos^2(angle) = 0·58] and 124 (1)° at C12 [anticlinal, cos^2(angle) = 0·32]. The phenyl rings are rotated out of the carbonyl planes by 35 (1)°.

Experimental. Crystal dimensions 0·3 × 0·3 × 0·4 mm. Nonius CAD-4F diffractometer, monochromatized Mo $K\alpha$ radiation, lattice parameters from 25 reflections with θ = 7–12°. Intensities for $\theta \le 27{\cdot}5°$, *hkl*: −20 to 20, 0 to 11, −20 to 0 ($\bar{h}k0$ not measured), ω–2θ scan, ω-scan width $(0{\cdot}50 + 0{\cdot}35\tan\theta)°$ at 0·9–10° min^{-1}, extended 25% on each side for background measurement; three standard reflections show no significant variation. Lp but no absorption corrections. 2472 unique reflections, 777 (only 31·4%, weak data at higher angles) with $I \ge 3\sigma(I)$. Structure solved by direct methods, refined by full-matrix least-squares procedures, x and z of C10 fixed to define the origin, H atoms placed in calculated positions. Refinement on F, with $w = 1/\sigma^2(F)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102). Locally written or locally modified versions of standard computer programs were used.* Final R = 0·030, wR = 0·028 for 288 parameters, 777 reflections with $I \ge 3\sigma(I)$ (data/parameter ratio only 2·7; isotropic refinement, ratio 6·1, had R = 0·12 without H-atom contribution), R = 0·20 for all 2472 reflections (high value is a result of the large number of weak high-angle reflections), extinction correction, g = 2·7 (5) × 10^4, S = 1·1, Δ/σ = 0·003 (mean), 0·014 (maximum), $\Delta\rho$ = −0·19 to +0·16 e Å^{-3}. Positional parameters are

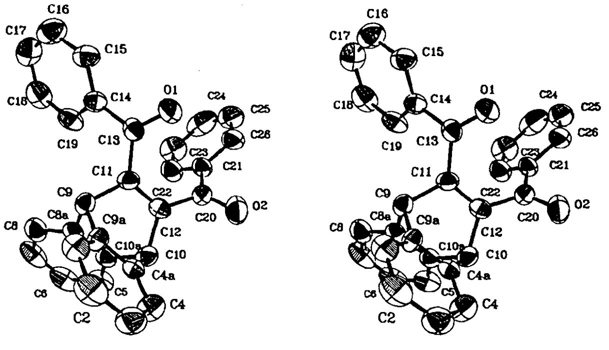

Fig. 1. Stereoview of the molecule.

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* *MULTAN*, *ORFLS*, *ORFFE* and *ORTEP*II; references given in Garcia-Garibay, Scheffer, Trotter & Wireko (1990).

given in Table 1, bond lengths and angles in Table 2, and a view of the molecule is shown in Fig. 1.*

Related literature. This work is part of a photochemical study of dibenzobarrelene diesters (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990).

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* Lists of anisotropic thermal parameters, hydrogen positions, torsion angles and structure factors, and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53756 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Reference

Garcia-Garibay, M., Scheffer, J. R., Trotter, J. & Wireko, F. C. (1990). *Acta Cryst.* B**46**, 431–440.

Acta Cryst. (1991). C**47**, 1319–1320

Structure of Di-*n*-propyl Dibenzobarrelene-11,12-dicarboxylate*

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Abstract. $C_{24}H_{24}O_4$, $M_r = 376{\cdot}45$, triclinic, $P\overline{1}$, $a = 9{\cdot}6978\,(4)$, $b = 10{\cdot}2945\,(5)$, $c = 10{\cdot}3828\,(3)$ Å, $\alpha = 82{\cdot}414\,(3)$, $\beta = 81{\cdot}671\,(3)$, $\gamma = 85{\cdot}817\,(3)^\circ$, $V = 1015{\cdot}1\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}232$ g cm^{-3}, Cu $K\alpha_1$, $\lambda = 1{\cdot}54056$ Å, $\mu = 6{\cdot}3$ cm^{-1}, $F(000) = 400$, $T = 295$ K, $R = 0{\cdot}067$ for 2597 reflections. The two carboxyl groups have different orientations with respect to the C11=C12 bond; torsion angles C=C—C=O are $-107{\cdot}3\,(5)^\circ$ at C11 [anticlinal, $\cos^2(\text{angle}) = 0{\cdot}09$, not conjugated, C11—CO = 1·487 (5) Å] and $-175{\cdot}1\,(4)^\circ$ at C12 [antiperiplanar, $\cos^2(\text{angle}) = 0{\cdot}99$, fully conjugated, C12—CO = 1·475 (5) Å].

Experimental. Crystal dimensions 0·3 × 0·3 × 0·4 mm. Nonius CAD-4F diffractometer, monochromatized Cu $K\alpha$ radiation, lattice parameters from 25 reflections with $\theta = 35$–44°. Intensities for $\theta < 75^\circ$, hkl: -12 to 12, -12 to 0, -13 to 13 ($\bar{h}0l$ removed from data set), ω–2θ scan, ω-scan width $(0{\cdot}75 + 0{\cdot}14\tan\theta)^\circ$ at 1·3–10° min^{-1}, extended 25% on each side for background measurement; three standard reflections showed no significant variation. Lp but no absorption corrections. 4169 unique reflections, 2597 (62·3%) with $I \geq 3\sigma(I)$. Structure solved by direct methods, refined by full-matrix least-squares procedures, H atoms placed in calculated positions and not refined. Refinements on F, with $w = 1/\sigma^2(F)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102). Locally written or locally modified versions of standard computer programs were used.† Final $R = 0{\cdot}067$, $wR = 0{\cdot}086$ for 254 parameters, 2597 reflections with $I \geq 3\sigma(I)$, $R = 0{\cdot}10$ for all 4169 reflections, extinction correction, $g = 3{\cdot}0\,(4) \times 10^4$, $S = 3{\cdot}4$, $\Delta/\sigma = 0{\cdot}002$ (mean), 0·028 (maximum), $\Delta\rho = -0{\cdot}50$ to $+0{\cdot}20$ e Å^{-3}. Positional parameters are given in Table 1, bond lengths and angles in Table 2 and a view of the molecule is shown in Fig. 1.‡

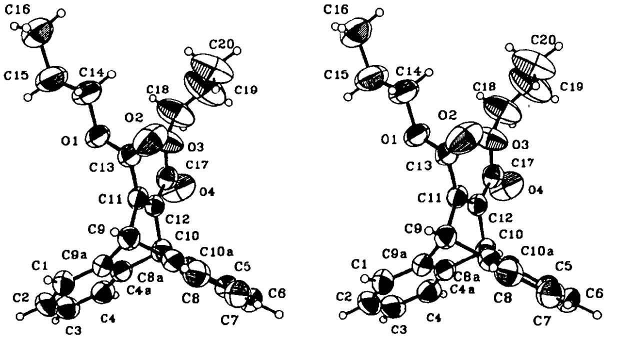

Fig. 1. Stereoview of the molecule.

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* Dipropyl 9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate.

† *MULTAN*, *ORFLS*, *ORFFE* and *ORTEP*II; references given in Garcia-Garibay, Scheffer, Trotter & Wireko (1990).

‡ Lists of anisotropic thermal parameters, hydrogen positions, torsion angles and structure factors, and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53757 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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