

Crystal structure of 4,6-dibenzoyloxy-1H-indole-2,3-dione methanolate (4/1), $C_{22}H_{17}NO_4 \cdot 0.25 (CH_3OH)$

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Received June 3, 1996, CSD-No. 402521

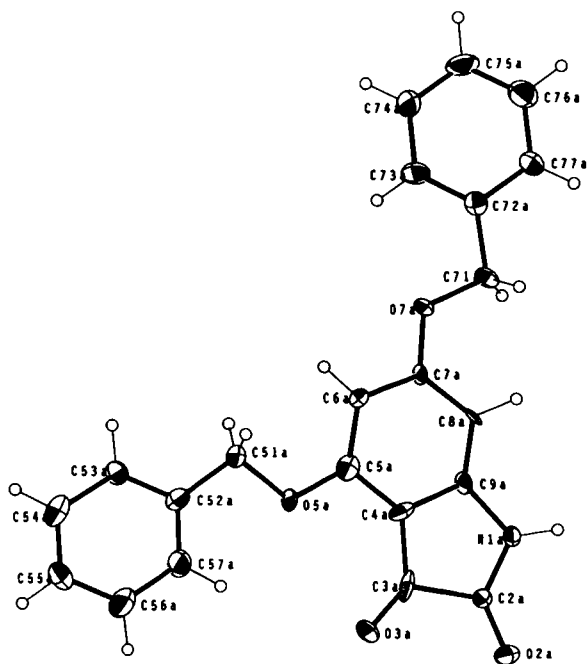


Fig. 1. Molecule A.

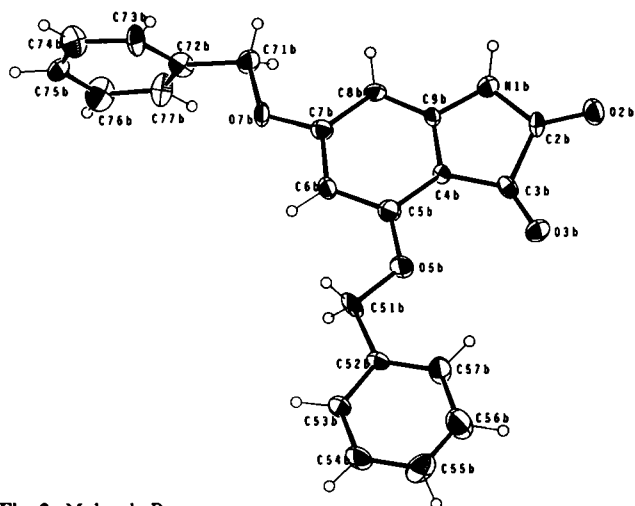


Fig. 2. Molecule B.

Table 1. Parameters used for the X-ray data collection

Crystal:	yellow needle, size 0.100 x 0.150 x 0.300 mm
Wavelength:	Mo $K\alpha$ radiation (0.7107 Å)
μ :	0.82 cm^{-1}
Diffractometer:	AFC7R
Scan mode:	$\omega/2\theta$
$T_{measurement}$:	293 K
$2\theta_{max}$:	45°
$N(hkl)_{unique}$:	1419
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(param)_{refined}$:	492
Program:	teXsan

Source of material: Oxalyl chloride (2 ml) was added to the hydrochloride salt of 4,6-dibenzoyloxylaniline (1.1 g) and heated at 438 K - 443 K for 1.5 hrs. The mixture was allowed to cool, methanol added (10 mL), the solution reheated, filtered while hot, and cooled slowly to give yellow crystals. The compound was recrystallised from methanol.

The methanol molecule was disordered over the inversion centre (0,1/2,0) with a site occupation of 0.5 for O(1s) and C(1s). The two independent molecules (A and B) differ only in that the phenyl ring (C72-C77) lies out of the molecular plane defined by the aromatic fragment in molecule B only. Molecule A is H-bonded to molecule B by one H-bond (H1a-O2b). Molecule B is H-bonded to molecule A through 2 H-bonds (H1b-O2a, O3a).

$C_{22}H_{17}NO_4$, triclinic, $P\bar{1}$ (No. 2), $a = 16.584(7)$ Å, $b = 23.560(8)$ Å, $c = 5.156(1)$ Å, $\alpha = 95.86(2)^\circ$, $\beta = 93.77(3)^\circ$, $\gamma = 70.26(3)^\circ$, $V = 1885.2$ Å³, $Z = 4$, $R(F) = 0.082$, $R_w(F) = 0.086$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(2a)	2i	0.0980(9)	-0.0558(7)	1.030(3)	0.06(1)	0.06(1)	0.07(1)	-0.012(9)	0.013(9)	0.03(1)
O(2b)	2i	0.103(1)	-0.1240(7)	1.471(3)	0.07(1)	0.05(1)	0.10(2)	0.00(1)	0.03(1)	0.02(1)
O(3a)	2i	0.2701(9)	-0.0935(6)	0.819(3)	0.04(1)	0.05(1)	0.08(1)	0.012(9)	0.006(9)	0.03(1)
O(3b)	2i	0.173(1)	-0.2397(7)	1.716(3)	0.07(1)	0.07(1)	0.07(1)	-0.024(9)	0.02(1)	0.02(1)
O(5a)	2i	0.3410(8)	-0.0401(6)	0.410(3)	0.02(1)	0.06(1)	0.10(1)	0.009(8)	0.024(9)	0.04(1)
O(5b)	2i	0.3294(9)	-0.3497(7)	1.590(3)	0.07(1)	0.06(1)	0.10(1)	-0.01(1)	0.02(1)	0.04(1)
O(7a)	2i	0.1269(8)	0.1304(6)	0.071(3)	0.033(9)	0.04(1)	0.06(1)	0.004(8)	0.002(8)	0.027(9)
O(7b)	2i	0.5037(9)	-0.3137(6)	0.974(3)	0.05(1)	0.04(1)	0.09(1)	0.005(8)	0.041(9)	0.013(9)
N(1a)	2i	0.066(1)	0.0191(8)	0.734(3)	0.03(1)	0.07(1)	0.04(1)	-0.01(1)	0.00(1)	0.02(1)
N(1b)	2i	0.230(1)	-0.1655(9)	1.242(3)	0.06(1)	0.06(2)	0.06(2)	-0.01(1)	0.02(1)	0.02(1)
C(2a)	2i	0.117(1)	-0.029(1)	0.864(4)	0.03(2)	0.07(2)	0.05(2)	0.00(1)	0.01(1)	0.03(2)
C(2b)	2i	0.171(1)	-0.166(1)	1.417(5)	0.04(2)	0.06(2)	0.06(2)	0.01(1)	0.02(1)	0.00(2)
C(3a)	2i	0.211(1)	-0.050(1)	0.751(5)	0.03(2)	0.09(3)	0.06(2)	-0.03(2)	0.02(1)	-0.01(2)
C(3b)	2i	0.207(1)	-0.226(1)	1.549(5)	0.04(2)	0.03(2)	0.06(2)	-0.01(1)	-0.01(1)	0.00(1)
C(4a)	2i	0.201(2)	-0.007(1)	0.553(4)	0.07(2)	0.06(2)	0.04(2)	-0.03(2)	0.01(2)	0.02(1)
C(4b)	2i	0.285(1)	-0.2569(9)	1.406(4)	0.04(2)	0.02(1)	0.03(2)	-0.01(1)	0.00(1)	-0.01(1)
C(5a)	2i	0.259(1)	0.002(1)	0.389(4)	0.05(2)	0.08(2)	0.03(2)	-0.03(2)	0.00(1)	-0.01(1)
C(5b)	2i	0.347(1)	-0.314(1)	1.425(5)	0.05(2)	0.07(2)	0.07(2)	-0.02(2)	0.00(1)	0.02(2)
C(6a)	2i	0.230(1)	0.049(1)	0.225(4)	0.04(2)	0.08(2)	0.06(2)	-0.02(1)	0.00(1)	0.04(2)
C(6b)	2i	0.419(1)	-0.3349(9)	1.262(4)	0.04(2)	0.04(2)	0.07(2)	0.00(1)	0.01(1)	0.01(1)
C(7a)	2i	0.147(1)	0.0877(9)	0.239(4)	0.02(1)	0.02(1)	0.07(2)	0.01(1)	0.02(1)	0.01(1)
C(7b)	2i	0.429(1)	-0.293(1)	1.102(4)	0.05(2)	0.05(2)	0.06(2)	-0.02(1)	0.00(1)	0.01(1)
C(8a)	2i	0.089(1)	0.0799(8)	0.395(4)	0.02(1)	0.03(2)	0.04(2)	0.00(1)	-0.02(1)	0.02(1)
C(8b)	2i	0.367(2)	-0.233(1)	1.087(4)	0.08(2)	0.04(2)	0.05(2)	-0.01(1)	0.02(2)	0.02(1)
C(9a)	2i	0.116(1)	0.036(1)	0.557(4)	0.02(1)	0.06(2)	0.04(2)	0.01(1)	0.01(1)	0.01(1)
C(9b)	2i	0.299(1)	-0.218(1)	1.243(4)	0.04(2)	0.04(2)	0.03(2)	0.01(1)	0.00(1)	0.00(1)
C(51a)	2i	0.400(1)	-0.036(1)	0.230(5)	0.04(2)	0.08(2)	0.10(2)	-0.01(1)	0.01(2)	0.04(2)
C(51b)	2i	0.386(1)	-0.408(1)	1.613(5)	0.06(2)	0.03(2)	0.12(2)	0.01(1)	0.01(2)	0.03(2)
C(52a)	2i	0.483(2)	-0.076(1)	0.310(5)	0.06(2)	0.07(2)	0.06(2)	-0.03(2)	0.00(2)	0.02(2)
C(52b)	2i	0.351(1)	-0.437(1)	1.795(4)	0.06(2)	0.05(2)	0.06(2)	-0.01(1)	0.00(1)	0.03(2)
C(53a)	2i	0.555(2)	-0.073(1)	0.219(7)	0.04(2)	0.19(4)	0.32(5)	0.05(2)	0.06(3)	0.21(4)
C(53b)	2i	0.393(1)	-0.496(1)	1.852(5)	0.08(2)	0.05(2)	0.08(2)	0.00(1)	0.02(2)	0.03(2)
C(54a)	2i	0.638(2)	-0.111(2)	0.285(8)	0.07(2)	0.24(5)	0.23(4)	-0.03(3)	0.03(3)	0.14(4)
C(54b)	2i	0.364(2)	-0.528(1)	2.007(5)	0.08(2)	0.05(2)	0.11(3)	-0.01(2)	0.01(2)	0.05(2)
C(55a)	2i	0.648(2)	-0.155(1)	0.433(6)	0.05(2)	0.15(3)	0.10(3)	-0.01(2)	-0.01(2)	0.06(2)
C(55b)	2i	0.290(2)	-0.500(1)	2.130(5)	0.10(3)	0.10(3)	0.10(3)	-0.06(2)	0.03(2)	0.02(2)
C(56a)	2i	0.577(2)	-0.159(2)	0.521(9)	0.07(3)	0.32(6)	0.45(7)	0.02(3)	0.07(4)	0.31(5)
C(56b)	2i	0.249(2)	-0.440(1)	2.082(6)	0.07(2)	0.11(3)	0.19(4)	0.00(2)	0.04(2)	0.08(3)
C(57a)	2i	0.495(2)	-0.116(1)	0.472(7)	0.05(2)	0.08(2)	0.21(4)	0.00(2)	0.03(2)	0.08(2)
C(57b)	2i	0.277(2)	-0.408(1)	1.918(5)	0.08(2)	0.06(2)	0.14(3)	0.00(2)	0.06(2)	0.05(2)
C(71a)	2i	0.040(1)	0.1706(9)	0.057(4)	0.05(2)	0.03(2)	0.04(2)	0.00(1)	-0.01(1)	0.00(1)
C(71b)	2i	0.530(2)	-0.274(1)	0.834(4)	0.07(2)	0.06(2)	0.09(2)	-0.01(1)	0.03(2)	0.02(2)
C(72a)	2i	0.038(1)	0.216(1)	-0.126(4)	0.05(2)	0.03(2)	0.06(2)	-0.01(1)	0.00(1)	-0.01(1)
C(72b)	2i	0.619(2)	-0.307(1)	0.760(6)	0.04(2)	0.07(2)	0.10(3)	-0.04(2)	0.00(2)	0.03(2)
C(73a)	2i	0.108(2)	0.219(1)	-0.238(5)	0.08(2)	0.05(2)	0.10(2)	-0.02(2)	0.00(2)	0.03(2)
C(73b)	2i	0.639(2)	-0.348(1)	0.536(6)	0.03(2)	0.11(3)	0.11(3)	0.01(2)	0.02(2)	0.01(2)
C(74a)	2i	0.101(2)	0.262(1)	-0.411(6)	0.05(2)	0.07(2)	0.12(3)	0.00(2)	0.02(2)	0.03(2)
C(74b)	2i	0.719(2)	-0.379(1)	0.458(6)	0.07(2)	0.09(2)	0.11(3)	-0.03(2)	0.00(2)	0.00(2)
C(75a)	2i	0.024(2)	0.302(1)	-0.469(5)	0.11(3)	0.07(2)	0.10(2)	-0.05(2)	-0.01(2)	0.04(2)
C(75b)	2i	0.781(2)	-0.367(1)	0.620(7)	0.08(2)	0.09(3)	0.09(3)	0.00(2)	0.05(2)	0.04(2)
C(76a)	2i	-0.046(2)	0.299(1)	-0.364(6)	0.08(2)	0.08(3)	0.11(3)	-0.04(2)	-0.03(2)	0.03(2)
C(76b)	2i	0.768(2)	-0.327(2)	0.844(6)	0.06(2)	0.14(3)	0.10(3)	-0.05(2)	-0.01(2)	-0.01(2)
C(77a)	2i	-0.041(1)	0.259(1)	-0.179(5)	0.05(2)	0.07(2)	0.12(3)	0.00(2)	0.00(2)	0.05(2)
C(77b)	2i	0.683(2)	-0.299(1)	0.899(5)	0.07(2)	0.09(2)	0.10(3)	-0.02(2)	0.03(2)	-0.02(2)

Acknowledgment. We gratefully acknowledge support of this work by the Australian Research Council.

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