Crystal structure of dimethyl 5-(acridin-9-yloxy)isophthalate — methanol (1:1), $C_{23}H_{17}NO_5 \cdot CH_3OH$

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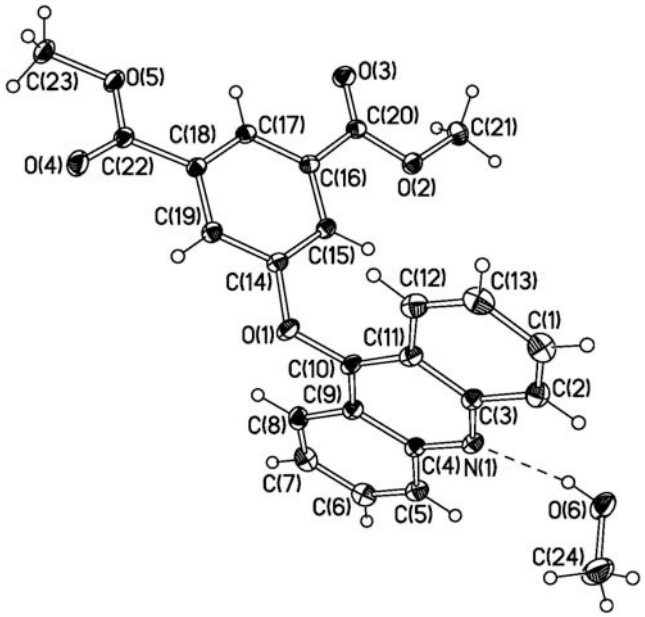

Abstract

$C_{24}H_{21}NO_6$, triclinic, $P\bar{1}$ (no. 2), a = 8.681(2) Å, b = 9.769(2) Å, c = 13.214(3) Å, α = 71.34(3)°, β = 78.45(3)°, γ = 77.18(3)°, V = 1025.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.146, T = 293 K.

Source of material

The title compound has been synthesized from 9-chloroacridine and the commercially available dimethyl 5-hydroxyisophthalate by a Williamson reaction. To a solution of NaOH (375 mg, 9.4 mmol) in *t*-BuOH (40 mL), dimethyl 5-hydroxy-isophthalate (1.5 g, 7 mmol) in DMSO (30 mL) was added and stirred under argon atmosphere for 30 minutes. 9-Chloroacridine (1.25 g, 5.8 mmol) in DMSO (25 mL) was then added and the mixture was heated under reflux for 1.5 h. The solid obtained by precipitation after ice addition was solved in $CHCl_3$ and worked-up as usual to yield the title compound (1.5 g, 83 %), which was recrystallized from ethyl acetylacetate/methanol (1:1, *v*/*v*).

Experimental details

The hydrogen atoms were positioned geometrically, with C—H distances constrained to 0.93 Å (aromatic) and 0.96 Å (methyl), and refined in riding mode with $U_{iso}(H) = x\,U_{eq}(C)$, where x = 1.5 for methyl H atoms and x = 1.2 for all other H atoms, respectively. The hydroxyl hydrogen atom was refined with a distance restraint of 0.82 Å, starting from the difference Fourier map coordinates and with $U_{iso}(H) = 1.5\,U_{eq}(O)$.

Discussion

The title molecule, dimethyl 5-(acridin-9-yloxy)isophthalate, is a fluorescent molecule with a structure based on a dimethyl benzene-1,3-bis-carboxylate (dimethyl isophthalate) framework and a phenolic function at C5 position derivatized as an acridine ether group. The diamine derivative of title compound has been used on supramolecular chemistry as a fluorescente spacer in pyrrole receptors for the recognition and sensing of bis-carboxylate anions [1].

The crystal contains an unique molecule and a molecule of the solvent in the asymmetric unit. All the bond lengths and angles are within the normal ranges. The title molecule consists of a benzene ring with two methyl esters and one acridine ring as susbtituents. The planar conformation of the ester groups is established from the torsion angles O5–C22–O4–C18 (178.8(2)°) and O2–C20–O3–C16 (179.0(2)º). In the molecule, the acridine group is almost perpendicular to the aromatic ring with the torsion angle C14–O1–C10–C9 of 89.3(6)º. A strong intermolecular interaction between the hydroxyl of the solvent molecule and nitrogen atom of the acridine group is seen. The details of the hydrogen bond O6–H6O···N1 are: d(O6—H6O) = 0.82 Å, d(H6O···N1) = 2.05 Å, d(O6···N1) = 2.857(1) Å, ∠O6–H6O···N1 = 167 º.

Table 1. Data collection and handling.

Crystal:	colorless prismatic, size 0.15 × 0.20 × 0.30 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	8.13 cm^{-1}
Diffractometer, scan mode:	SEIFERT XRD 3003 SC, $2\theta/\omega$
$2\theta_{max}$:	120°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3031, 3031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2290
$N(param)_{refined}$:	285
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL^{TM} [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.7177	1.0500	–0.1749	0.083
H(2)	2*i*	0.4827	0.9802	–0.1660	0.070
H(5)	2*i*	0.1097	0.6585	–0.0041	0.065
H(6)	2*i*	0.0292	0.4599	0.1254	0.077
H(7)	2*i*	0.1868	0.3190	0.2570	0.078

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(8)	2*i*	0.4295	0.3716	0.2570	0.067
H(12)	2*i*	0.8228	0.7244	0.0892	0.073
H(13)	2*i*	0.8850	0.9266	–0.0452	0.086
H(15)	2*i*	0.4965	0.7175	0.2766	0.051
H(17)	2*i*	0.6923	0.5872	0.5511	0.050
H(19)	2*i*	0.8448	0.3664	0.3308	0.052
H(21A)	2*i*	0.1960	1.0450	0.3672	0.100
H(21B)	2*i*	0.3228	1.0583	0.4314	0.100

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(21C)	2*i*	0.1973	0.9545	0.4888	0.100
H(23A)	2*i*	0.9570	0.2838	0.7615	0.093
H(23B)	2*i*	1.0885	0.2570	0.6663	0.093
H(23C)	2*i*	0.9502	0.1646	0.7071	0.093
H(24A)	2*i*	0.0965	0.8873	–0.2771	0.221
H(24B)	2*i*	0.2583	0.8038	–0.2352	0.221
H(24C)	2*i*	0.2505	0.9590	–0.3201	0.221
H(6O)	2*i*	0.2120	0.9146	–0.1292	0.123

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*	0.6702(2)	0.5100(2)	0.1928(1)	0.058(1)	0.070(1)	0.0472(9)	0.0209(8)	–0.0238(8)	–0.0298(8)
O(2)	2*i*	0.3706(2)	0.8735(2)	0.3903(1)	0.056(1)	0.053(1)	0.055(1)	0.0086(8)	–0.0151(8)	–0.0225(8)
O(3)	2*i*	0.4751(2)	0.8098(2)	0.5441(1)	0.070(1)	0.068(1)	0.057(1)	0.0052(9)	–0.0164(8)	–0.0318(9)
O(4)	2*i*	0.9924(2)	0.2538(2)	0.4930(1)	0.063(1)	0.065(1)	0.064(1)	0.0142(9)	–0.0213(9)	–0.0216(9)
O(5)	2*i*	0.8825(2)	0.3678(2)	0.6184(1)	0.062(1)	0.061(1)	0.0465(9)	0.0028(8)	–0.0232(8)	–0.0141(8)
N(1)	2*i*	0.3644(2)	0.7639(2)	–0.0306(1)	0.043(1)	0.058(1)	0.039(1)	–0.0006(9)	–0.0080(8)	–0.0158(9)
C(1)	2*i*	0.6886(3)	0.9695(3)	–0.1194(2)	0.061(2)	0.069(2)	0.072(2)	–0.018(1)	0.008(1)	–0.019(1)
C(2)	2*i*	0.5498(3)	0.9273(3)	–0.1148(2)	0.057(2)	0.062(2)	0.050(1)	–0.008(1)	–0.002(1)	–0.012(1)
C(3)	2*i*	0.5045(3)	0.8022(2)	–0.0321(2)	0.043(1)	0.054(1)	0.040(1)	–0.001(1)	–0.0028(9)	–0.019(1)
C(4)	2*i*	0.3228(2)	0.6444(2)	0.0468(2)	0.039(1)	0.052(1)	0.037(1)	–0.000(1)	–0.0025(9)	–0.019(1)
C(5)	2*i*	0.1743(3)	0.6034(3)	0.0482(2)	0.043(1)	0.068(2)	0.054(1)	–0.001(1)	–0.008(1)	–0.025(1)
C(6)	2*i*	0.1267(3)	0.4848(3)	0.1252(2)	0.053(2)	0.070(2)	0.074(2)	–0.013(1)	0.000(1)	–0.029(2)
C(7)	2*i*	0.2224(3)	0.3991(3)	0.2044(2)	0.066(2)	0.058(2)	0.066(2)	–0.013(1)	0.007(1)	–0.018(1)
C(8)	2*i*	0.3660(3)	0.4312(3)	0.2052(2)	0.061(2)	0.051(1)	0.047(1)	0.005(1)	–0.006(1)	–0.013(1)
C(9)	2*i*	0.4199(3)	0.5560(2)	0.1271(2)	0.043(1)	0.053(1)	0.037(1)	0.004(1)	–0.0040(9)	–0.020(1)
C(10)	2*i*	0.5639(3)	0.5986(3)	0.1232(2)	0.043(1)	0.058(1)	0.038(1)	0.011(1)	–0.0123(9)	–0.023(1)
C(11)	2*i*	0.6100(2)	0.7233(3)	0.0450(2)	0.037(1)	0.060(1)	0.047(1)	0.003(1)	–0.0051(9)	–0.027(1)
C(12)	2*i*	0.7539(3)	0.7737(3)	0.0385(2)	0.043(1)	0.077(2)	0.069(2)	0.001(1)	–0.011(1)	–0.033(2)
C(13)	2*i*	0.7908(3)	0.8938(3)	–0.0416(3)	0.047(2)	0.080(2)	0.095(2)	–0.017(1)	0.000(1)	–0.040(2)
C(14)	2*i*	0.6697(2)	0.5387(2)	0.2890(2)	0.039(1)	0.054(1)	0.038(1)	–0.004(1)	–0.0071(9)	–0.017(1)
C(15)	2*i*	0.5691(2)	0.6535(2)	0.3205(2)	0.035(1)	0.049(1)	0.041(1)	–0.0031(9)	–0.0071(9)	–0.0123(9)
C(16)	2*i*	0.5790(2)	0.6714(2)	0.4194(2)	0.035(1)	0.046(1)	0.042(1)	–0.0100(9)	–0.0039(9)	–0.0137(9)
C(17)	2*i*	0.6865(2)	0.5748(2)	0.4852(2)	0.038(1)	0.049(1)	0.040(1)	–0.013(1)	–0.0061(9)	–0.0119(9)
C(18)	2*i*	0.7852(2)	0.4597(2)	0.4523(2)	0.034(1)	0.045(1)	0.041(1)	–0.0105(9)	–0.0071(9)	–0.0093(9)
C(19)	2*i*	0.7775(2)	0.4425(2)	0.3536(2)	0.035(1)	0.050(1)	0.046(1)	–0.0024(9)	–0.0068(9)	–0.016(1)
C(20)	2*i*	0.4721(3)	0.7908(2)	0.4591(2)	0.041(1)	0.047(1)	0.044(1)	–0.011(1)	–0.0051(9)	–0.014(1)
C(21)	2*i*	0.2626(3)	0.9928(3)	0.4221(2)	0.066(2)	0.052(2)	0.081(2)	0.014(1)	–0.017(1)	–0.030(1)
C(22)	2*i*	0.8987(3)	0.3495(2)	0.5213(2)	0.039(1)	0.048(1)	0.048(1)	–0.007(1)	–0.011(1)	–0.010(1)
C(23)	2*i*	0.9775(3)	0.2592(3)	0.6947(2)	0.066(2)	0.065(2)	0.051(1)	–0.007(1)	–0.027(1)	–0.003(1)
C(24)	2*i*	0.1934(6)	0.8984(5)	–0.2583(3)	0.209(5)	0.132(4)	0.107(3)	0.076(3)	–0.088(3)	–0.075(3)
O(6)	2*i*	0.1577(2)	0.9621(2)	–0.1771(2)	0.081(1)	0.092(2)	0.065(1)	0.018(1)	–0.028(1)	–0.023(1)

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