Crystal structure of 1,3-bis[3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione-9-yl]benzene, $C_{40}H_{48}N_2O_4$

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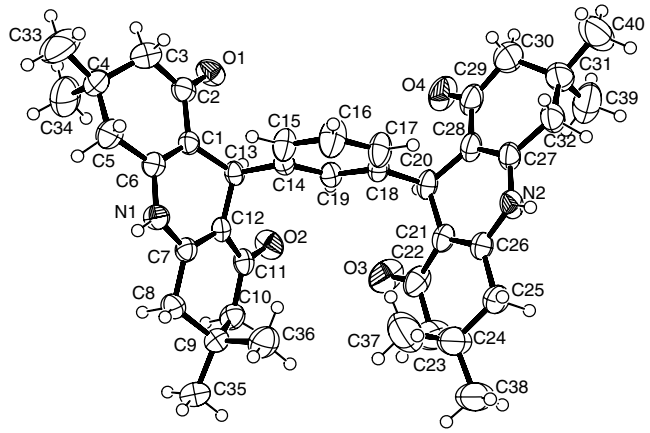

Abstract

$C_{40}H_{48}N_2O_4$, monoclinic, $P12_1/n1$ (No. 14), a = 11.056(2) Å, b = 13.796(2) Å, c = 23.984(2) Å, β = 103.43(1)°, V = 3558.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.063, $wR_{ref}(F^2)$ = 0.225, T = 299 K.

Source of material

A mixture of dimedone (4 mmol) and isophthalaldehyde (1 mmol) was stirred at 313 K – 323 K for 10 min. in aqueous methanol until the solution became cloudy, and the reaction mixture was then allowed to stand at room temperature overnight. The solid obtained was collected by filtration and crystallized from ethanol as pale yellow crystals (36 %), mp 511 K – 513 K. To a solution of the latter (5 mmol) in acetic acid (10 ml) was added ammonium acetate (11 mmol). The reaction mixture was heated under reflux for 2h, cooled and poured over crashed ice. The solid obtained was then collected and crystallized from ethanol to give 1,3-bis[3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridine dione- 9-yl]benzene as pale yellow crystals (yield 48 %).

Discussion

Acridine-1,8-dione dyes have been shown to have very high lasing efficiency [1,2] and are also interesting in view of the electrochemical behaviour of heterocyclic compounds [3]. They have been used as electron donors and electron acceptors [4-6] and in the photoinitiated polymerization of acrylates and methacrylates [7]. In addition to this, these compounds have long been known to be potent farmeshift mutagerns in viruses and bacteria [8].

We report here the crystal structure of the novel 1,3-bis(acridinedione-9-yl)benzene. Bond lengths and angles of the acridine skeleton are in agreement with those of related acridine derivatives [9-12]. The bond lengths in the pyridine ring range from 1.352(3) Å to 1.516(3) Å and show the respective greater or lower degree of single- or double-bond character predicted from comparison with related structures [10,11,13]. The ketone bond lengths, C2=O1, C11=O2, C29=O4 and C22=O3 of 1.236(3) Å, 1.229(3) Å, 1.223(3) Å and 1.222(3) Å, respectively are in agreement with values observed for related structures [14]. The outer six-membered rings of the acridine moieties adopt almost envelope conformations, while the central pyridine rings adopt a distorted boat conformation. The phenyl ring is nearly perpendicular to both acridine moieties, with the dihedral angles between their respective least-squares planes being –95.0(2)° and –97.7(3)°. The structure is stabilized by three N–H···O intermolecular hydrogen bonds N2···O1, N1···O2 and N1···O3 with N···O distances and N–H···O angles of 2.869 Å, 2.874 Å, 3.298 Å and of 167.4(1)°, 157.4(1)°, 113.6(1)°, respectively.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.28 × 0.45 × 0.80 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.71 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	51.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7748, 6938
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4750
$N(param)_{refined}$:	429
Programs:	SHELXS-97 [12], SHELXL-97 [13], ORTEP-3 [14]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*e*	0.8314	1.0453	0.0237	0.088
H(3B)	4*e*	0.9537	1.0933	0.0146	0.088
H(5A)	4*e*	1.0358	1.2480	0.0861	0.072
H(5B)	4*e*	0.9621	1.2599	0.1341	0.072
H(8A)	4*e*	1.1991	1.1718	0.3125	0.056
H(8B)	4*e*	1.2990	1.2255	0.2870	0.056
H(10A)	4*e*	1.3877	0.9588	0.3526	0.057
H(10B)	4*e*	1.2560	0.9987	0.3540	0.057
H(1)	4*e*	1.142(2)	0.914(2)	0.1709(9)	0.035(6)
H(15)	4*e*	1.2567	1.0866	0.0875	0.061
H(16)	4*e*	1.4248	1.0644	0.0476	0.081
H(17)	4*e*	1.5545	0.9328	0.0723	0.067
H(19)	4*e*	1.3453	0.8429	0.1775	0.043
H(2)	4*e*	1.511(3)	0.742(2)	0.170(1)	0.059(8)

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(23A)	4*e*	1.7317	0.9153	0.3226	0.105
H(23B)	4*e*	1.8103	0.8243	0.3147	0.105
H(25A)	4*e*	1.9110	0.9471	0.1980	0.065
H(25B)	4*e*	1.9444	0.8487	0.2306	0.065
H(30A)	4*e*	1.5090	0.5953	–0.0129	0.098
H(30B)	4*e*	1.5156	0.7067	–0.0244	0.098
H(32A)	4*e*	1.8395	0.7242	0.0469	0.071
H(32B)	4*e*	1.7378	0.7892	0.0079	0.071
H(33A)	4*e*	0.7898	1.2983	0.0434	0.141
H(33B)	4*e*	0.7346	1.2117	0.0032	0.141
H(33C)	4*e*	0.8631	1.2567	0.0001	0.141
H(34A)	4*e*	0.7577	1.2009	0.1239	0.120
H(34B)	4*e*	0.8176	1.0977	0.1355	0.120
H(34C)	4*e*	0.7077	1.1152	0.0820	0.120
H(35A)	4*e*	1.4574	1.1089	0.4176	0.075
H(35B)	4*e*	1.3182	1.1420	0.4064	0.075
H(35C)	4*e*	1.4185	1.2119	0.3919	0.075

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(36A)	4*e*	1.5506	1.0672	0.3349	0.084
H(36B)	4*e*	1.5153	1.1700	0.3082	0.084
H(36C)	4*e*	1.4704	1.0766	0.2720	0.084
H(37A)	4*e*	1.8230	1.0810	0.2366	0.123
H(37B)	4*e*	1.7515	1.0666	0.2853	0.123
H(37C)	4*e*	1.6978	1.0229	0.2242	0.123
H(38A)	4*e*	2.0134	1.0096	0.2970	0.118
H(38B)	4*e*	2.0085	0.9125	0.3304	0.118
H(38C)	4*e*	1.9395	1.0048	0.3453	0.118
H(39A)	4*e*	1.8254	0.5526	0.0489	0.116
H(39B)	4*e*	1.6970	0.5001	0.0285	0.116
H(39C)	4*e*	1.7218	0.5662	0.0832	0.116
H(40A)	4*e*	1.8044	0.6235	–0.0443	0.146
H(40B)	4*e*	1.6844	0.6798	–0.0753	0.146
H(40C)	4*e*	1.6781	0.5680	–0.0647	0.146
H(1N)	4*e*	1.157(3)	1.238(1)	0.201(1)	0.056
H(2N)	4*e*	1.852(2)	0.851(2)	0.114(1)	0.054

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}
C(1)	4*e*	1.0739(2)	1.0452(2)	0.13297(9)	0.032(1)	0.039(1)	0.039(1)	0.0014(9)	0.0106(9)	–0.0034(9)
C(2)	4*e*	0.9833(2)	1.0066(2)	0.0847(1)	0.035(1)	0.057(2)	0.046(1)	–0.004(1)	0.014(1)	–0.008(1)
C(3)	4*e*	0.9079(3)	1.0769(3)	0.0433(1)	0.068(2)	0.089(2)	0.054(2)	0.014(2)	–0.006(2)	–0.012(2)
C(4)	4*e*	0.8752(3)	1.1698(2)	0.0700(1)	0.052(2)	0.075(2)	0.045(1)	0.021(1)	0.003(1)	0.002(1)
C(5)	4*e*	0.9888(3)	1.2132(2)	0.1092(1)	0.066(2)	0.052(2)	0.058(2)	0.018(1)	0.004(1)	0.008(1)
C(6)	4*e*	1.0728(2)	1.1410(2)	0.1456(1)	0.042(1)	0.038(1)	0.042(1)	0.006(1)	0.007(1)	0.0006(9)
C(7)	4*e*	1.2147(2)	1.1160(2)	0.2364(1)	0.039(1)	0.032(1)	0.042(1)	0.0042(9)	0.0077(9)	0.0003(9)
C(8)	4*e*	1.2662(2)	1.1622(2)	0.2932(1)	0.054(2)	0.034(1)	0.048(1)	0.005(1)	0.005(1)	–0.006(1)
C(9)	4*e*	1.3687(2)	1.1033(2)	0.3321(1)	0.038(1)	0.048(1)	0.045(1)	0.001(1)	0.006(1)	–0.003(1)
C(10)	4*e*	1.3201(2)	0.9996(2)	0.3325(1)	0.050(1)	0.046(1)	0.045(1)	0.007(1)	0.008(1)	0.008(1)
C(11)	4*e*	1.2678(2)	0.9561(2)	0.2746(1)	0.044(1)	0.033(1)	0.047(1)	0.0070(9)	0.017(1)	0.0049(9)
C(12)	4*e*	1.2200(2)	1.0198(2)	0.22665(9)	0.032(1)	0.029(1)	0.043(1)	0.0036(8)	0.0131(9)	–0.0006(9)
C(13)	4*e*	1.1741(2)	0.9795(2)	0.16666(9)	0.033(1)	0.025(1)	0.044(1)	–0.0006(8)	0.0136(9)	–0.0021(9)
C(14)	4*e*	1.2828(2)	0.9669(2)	0.13717(9)	0.031(1)	0.033(1)	0.039(1)	–0.0028(8)	0.0101(9)	–0.0058(9)
C(15)	4*e*	1.3080(2)	1.0330(2)	0.0979(1)	0.047(1)	0.050(1)	0.062(2)	0.013(1)	0.022(1)	0.016(1)
C(16)	4*e*	1.4090(3)	1.0196(2)	0.0740(1)	0.061(2)	0.074(2)	0.078(2)	0.019(2)	0.038(2)	0.037(2)
C(17)	4*e*	1.4867(2)	0.9409(2)	0.0887(1)	0.045(1)	0.065(2)	0.068(2)	0.012(1)	0.031(1)	0.018(1)
C(18)	4*e*	1.4636(2)	0.8740(2)	0.1279(1)	0.030(1)	0.042(1)	0.050(1)	–0.0001(9)	0.013(1)	0.000(1)
C(19)	4*e*	1.3615(2)	0.8880(2)	0.1513(1)	0.033(1)	0.033(1)	0.045(1)	–0.0043(9)	0.0147(9)	–0.0004(9)
C(20)	4*e*	1.5527(2)	0.7893(2)	0.1478(1)	0.029(1)	0.042(1)	0.063(2)	0.0008(9)	0.018(1)	0.004(1)
C(21)	4*e*	1.6632(2)	0.8241(2)	0.1931(1)	0.033(1)	0.048(1)	0.052(1)	0.003(1)	0.014(1)	0.009(1)
C(22)	4*e*	1.6516(2)	0.8365(2)	0.2517(1)	0.042(1)	0.087(2)	0.055(2)	0.003(1)	0.018(1)	0.021(2)
C(23)	4*e*	1.7626(3)	0.8779(3)	0.2946(1)	0.065(2)	0.152(4)	0.048(2)	–0.022(2)	0.021(2)	0.001(2)
C(24)	4*e*	1.8473(3)	0.9399(3)	0.2713(1)	0.055(2)	0.128(3)	0.059(2)	–0.024(2)	0.022(2)	–0.022(2)
C(25)	4*e*	1.8789(2)	0.8963(2)	0.2185(1)	0.036(1)	0.071(2)	0.057(2)	–0.006(1)	0.015(1)	–0.004(1)
C(26)	4*e*	1.7711(2)	0.8486(2)	0.1784(1)	0.034(1)	0.045(1)	0.050(1)	0.001(1)	0.014(1)	0.003(1)
C(27)	4*e*	1.7067(2)	0.7689(2)	0.0874(1)	0.034(1)	0.045(1)	0.061(2)	–0.000(1)	0.016(1)	–0.009(1)
C(28)	4*e*	1.5958(2)	0.7443(2)	0.0982(1)	0.031(1)	0.045(1)	0.068(2)	0.002(1)	0.015(1)	–0.005(1)
C(29)	4*e*	1.5148(2)	0.6769(2)	0.0599(1)	0.033(1)	0.059(2)	0.088(2)	0.000(1)	0.011(1)	–0.013(2)
C(30)	4*e*	1.5485(3)	0.6554(3)	0.0025(2)	0.047(2)	0.102(3)	0.091(2)	–0.007(2)	0.008(2)	–0.034(2)
C(31)	4*e*	1.6856(3)	0.6473(2)	0.0075(1)	0.050(2)	0.076(2)	0.072(2)	–0.011(1)	0.014(1)	–0.029(2)
C(32)	4*e*	1.7508(2)	0.7369(2)	0.0356(1)	0.042(1)	0.071(2)	0.068(2)	–0.003(1)	0.021(1)	–0.021(1)
C(33)	4*e*	0.8097(5)	1.2406(3)	0.0251(2)	0.124(4)	0.108(3)	0.090(3)	0.040(3)	–0.035(3)	0.017(2)
C(34)	4*e*	0.7805(4)	1.1433(3)	0.1063(2)	0.064(2)	0.127(3)	0.115(3)	0.022(2)	0.033(2)	–0.002(3)
C(35)	4*e*	1.3930(3)	1.1454(2)	0.3927(1)	0.055(2)	0.073(2)	0.052(2)	–0.001(1)	–0.002(1)	–0.010(1)
C(36)	4*e*	1.4873(3)	1.1044(3)	0.3097(1)	0.046(2)	0.090(2)	0.075(2)	–0.009(2)	0.017(2)	–0.006(2)
C(37)	4*e*	1.7725(4)	1.0372(3)	0.2525(2)	0.098(3)	0.112(3)	0.109(3)	–0.005(3)	0.046(3)	–0.032(3)
C(38)	4*e*	1.9626(3)	0.9694(4)	0.3150(2)	0.067(2)	0.165(4)	0.064(2)	–0.027(2)	0.016(2)	–0.034(2)
C(39)	4*e*	1.7374(3)	0.5579(3)	0.0457(2)	0.057(2)	0.072(2)	0.157(4)	0.002(2)	0.017(2)	–0.032(2)
C(40)	4*e*	1.7159(4)	0.6279(4)	–0.0493(2)	0.078(3)	0.182(5)	0.111(3)	–0.032(3)	0.034(2)	–0.092(3)
N(1)	4*e*	1.1529(2)	1.1764(1)	0.19339(9)	0.060(1)	0.0246(9)	0.049(1)	0.0072(9)	–0.003(1)	–0.0024(8)
N(2)	4*e*	1.7875(2)	0.8285(2)	0.12493(9)	0.034(1)	0.051(1)	0.058(1)	–0.0103(9)	0.0228(9)	–0.0107(9)
O(1)	4*e*	0.9734(2)	0.9183(1)	0.07629(8)	0.050(1)	0.057(1)	0.059(1)	–0.0177(8)	0.0180(9)	–0.0185(9)
O(2)	4*e*	1.2613(2)	0.8675(1)	0.26924(8)	0.088(1)	0.0297(9)	0.058(1)	0.0083(8)	0.018(1)	0.0078(8)
O(3)	4*e*	1.5586(2)	0.8120(2)	0.26733(9)	0.051(1)	0.147(2)	0.062(1)	–0.012(1)	0.027(1)	0.025(1)
O(4)	4*e*	1.4209(2)	0.6441(2)	0.0715(1)	0.037(1)	0.077(1)	0.110(2)	–0.015(1)	0.015(1)	–0.012(1)

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