Crystal structure of *N*-(3,5-benzenedicarboxy)-2,5-phenyl-3,4-(9,10-dihydroanthracen-diyl)-pyrrole — acetone — water (1:1:1), $C_{38}H_{25}NO_4 \cdot C_3H_6O \cdot H_2O$

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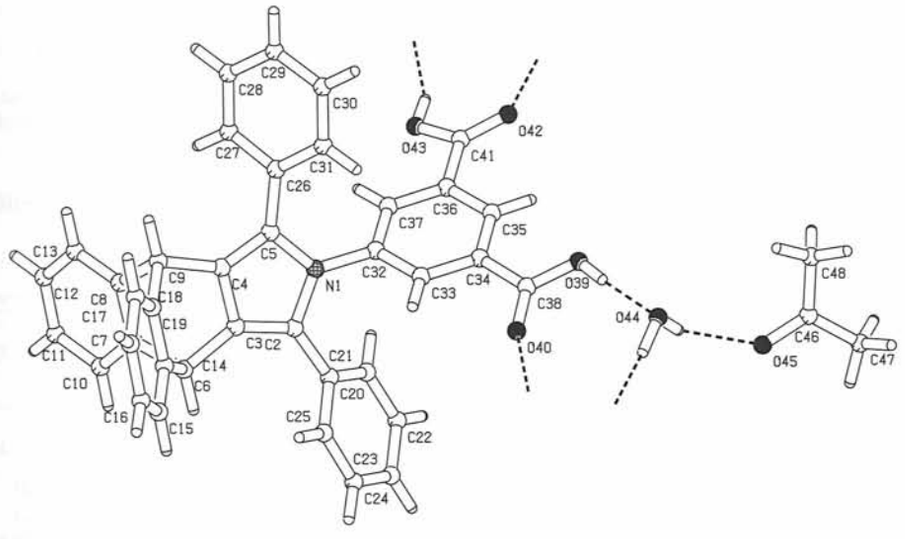

Abstract

$C_{41}H_{33}NO_6$, triclinic, $P\bar{1}$ (No. 2), $a = 9.610(3)$ Å, $b = 12.877(3)$ Å, $c = 15.138(4)$ Å, $\alpha = 109.29(2)°$, $\beta = 105.16(2)°$, $\gamma = 94.24(2)°$, $V = 1680.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.059$, $wR_{ref}(F^2) = 0.161$, $T = 289$ K.

Source of material

The isophtalic acid was obtained by saponification of the dimethyl ester which was prepared by condensation of dimethyl 5-aminoisophtalate and the Diels-Alder adduct of anthracene and dibenzoylethylene in an analogous way as previously published [1]. The total yield for these two steps was 61%. The compound was crystallised from ethanol and the light yellow crystals were washed with acetone. The ^{1}H NMR and ^{13}C NMR data are included in the deposited CIF.

Experimental details

Hydrogen atoms for O44 were found from difference Fourier maps and refined, other hydrogen atoms were placed at calculated positions and refined in the riding mode with a fixed isotropic temperature factor 1.2 times U_{eq} of the parent atom.

Discussion

Highly bulky Diels-Alder adducts of anthracene and their derivatives have been shown to have interesting complexation behavior [2-7]. The combination with the potent structure forming properties of the isophtalic acid group would likely result in modified inclusion properties. The pyrrole ring in the title compound makes an angle with its three phenyl substituents of respectively 41.7(2)° (ring C20-C25), 43.6(2)° (ring C26-C31) and 60.5(2)° (ring C32-C37). The two carboxylic acid groups make an angle of 10.6(2)° and 12.0(2)° with the aromatic rings to which they are attached. By H-bond formation linear chains are formed in the [100] direction. Hydroxyl group O43-H43 forms a H-bond with oxygen atom O40# generated by (1+x, y, z) [d(O43···O40#) = 2.640(4) Å, d(O43—H43) = 0.820 Å, d(H43···O40#) = 1.870 Å, ∠O43–H43···O40# = 156°]. Hydroxyl group O39-H39 forms a water-mediated (O44) H-bond with oxygen atom O42# generated by (x–1, y, z) [d(O39···O44) = 2.612(5) Å, d(O39—H39) = 0.820 Å, d(H39···O44) = 1.790 Å, ∠O39–H39···O44 = 175° and d(O44···O42#) = 2.868(5) Å, d(O44—H44A) = 1.00(5) Å, d(H44A···O42#) = 1.88(6) Å, ∠O44–H44A···O42# = 171(4)°]. Water molecule O44 forms a further H-bond with an acetone molecule [d(O44···O45) = 2.895(7) Å, d(O44—H44B) = 0.72(7) Å, d(H44B···O45) = 2.22(7) Å, ∠O44–H44B···O45 = 158(6)°). In the crystal packing the dihydroanthracene rings are faced towards each other. Ring C7-C13 stacks with a symmetry equivalent one at (–x, 1–y, 1–z) (distance between ring centroids: 3.830(4) Å). No voids larger than 30 Å^3 are present in the unit cell.

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Table 1. Data collection and handling.

Crystal:	light yellow blocks, size 0.15 × 0.20 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	Siemens P4-PC, ω
$2\theta_{max}$:	44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4911, 4026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2659
$N(param)_{refined}$:	441
Programs:	SHELXS-97 [8], SHELXL-97 [9], PLATON [10]

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

Atom	Site	x	y	z	U_{iso}
H(6)	2*i*	–0.2812	0.2359	0.5612	0.052
H(9)	2*i*	0.0651	0.5467	0.7599	0.050
H(10)	2*i*	–0.1624	0.2103	0.4211	0.076
H(11)	2*i*	0.0280	0.2670	0.3683	0.092
H(12)	2*i*	0.2042	0.4187	0.4705	0.091
H(13)	2*i*	0.1950	0.5191	0.6258	0.068
H(15)	2*i*	–0.4867	0.3589	0.5647	0.068
H(16)	2*i*	–0.5531	0.5328	0.6228	0.083
H(17)	2*i*	–0.3792	0.6847	0.7283	0.077
H(18)	2*i*	–0.1355	0.6685	0.7735	0.063
H(21)	2*i*	–0.0437	0.0244	0.7542	0.063
H(22)	2*i*	–0.1869	–0.1488	0.6963	0.082
H(23)	2*i*	–0.4337	–0.1743	0.6182	0.096
H(24)	2*i*	–0.5372	–0.0252	0.5977	0.098
H(25)	2*i*	–0.3949	0.1512	0.6609	0.076
H(27)	2*i*	0.2895	0.4953	0.8456	0.067
H(28)	2*i*	0.4587	0.6395	0.9711	0.082
H(29)	2*i*	0.4577	0.6821	1.1304	0.087
H(30)	2*i*	0.2862	0.5805	1.1657	0.080
H(31)	2*i*	0.1123	0.4378	1.0403	0.060
H(33)	2*i*	–0.1631	0.2146	0.9337	0.046
H(35)	2*i*	0.1343	0.1129	1.1130	0.044
H(37)	2*i*	0.2450	0.2160	0.9143	0.049
H(39)	2*i*	–0.1713	0.1059	1.1740	0.076
H(43)	2*i*	0.5248	0.1327	1.0178	0.083
H(44A)	2*i*	–0.413(6)	0.084(4)	1.185(3)	0.085
H(44B)	2*i*	–0.322(7)	0.018(5)	1.215(4)	0.085
H(47A)	2*i*	–0.3456	–0.245	1.3859	0.169
H(47B)	2*i*	–0.2147	–0.2883	1.3497	0.169
H(47C)	2*i*	–0.3679	–0.3039	1.2733	0.169
H(48A)	2*i*	–0.0596	–0.0845	1.4129	0.232
H(48B)	2*i*	–0.1781	–0.0605	1.4677	0.232
H(48C)	2*i*	–0.1581	0.0042	1.3993	0.232

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}
N(1)	2*i*	–0.0014(3)	0.2683(3)	0.8363(2)	0.036(2)	0.051(2)	0.042(2)	0.005(2)	0.008(2)	0.028(2)
C(2)	2*i*	–0.1165(4)	0.2193(3)	0.7472(3)	0.037(2)	0.045(3)	0.040(2)	0.006(2)	0.010(2)	0.020(2)
C(3)	2*i*	–0.1208(4)	0.2943(3)	0.7000(3)	0.046(3)	0.046(3)	0.038(2)	0.005(2)	0.010(2)	0.019(2)
C(4)	2*i*	–0.0113(4)	0.3896(3)	0.7605(3)	0.037(2)	0.046(3)	0.041(2)	0.004(2)	0.013(2)	0.021(2)
C(5)	2*i*	0.0624(4)	0.3739(3)	0.8441(3)	0.038(2)	0.049(3)	0.043(2)	0.001(2)	0.009(2)	0.025(2)
C(6)	2*i*	–0.2072(4)	0.3024(3)	0.6039(3)	0.045(2)	0.044(3)	0.034(2)	–0.002(2)	0.001(2)	0.016(2)
C(7)	2*i*	–0.0926(5)	0.3291(4)	0.5566(3)	0.061(3)	0.051(3)	0.039(3)	0.019(2)	0.015(2)	0.027(2)
C(8)	2*i*	0.0124(5)	0.4238(4)	0.6181(3)	0.052(3)	0.057(3)	0.044(3)	0.015(2)	0.017(2)	0.032(2)
C(9)	2*i*	–0.0091(4)	0.4802(3)	0.7175(3)	0.041(2)	0.044(2)	0.043(2)	0.000(2)	0.008(2)	0.023(2)
C(10)	2*i*	–0.0899(6)	0.2714(4)	0.4628(3)	0.092(4)	0.060(3)	0.042(3)	0.027(3)	0.019(3)	0.021(2)
C(11)	2*i*	0.0240(7)	0.3062(5)	0.4311(4)	0.103(5)	0.105(5)	0.057(3)	0.053(4)	0.050(4)	0.044(3)
C(12)	2*i*	0.1286(6)	0.3968(5)	0.4919(4)	0.076(4)	0.118(5)	0.073(4)	0.043(4)	0.041(3)	0.065(4)
C(13)	2*i*	0.1237(5)	0.4567(4)	0.5851(3)	0.052(3)	0.079(3)	0.065(3)	0.023(2)	0.027(2)	0.049(3)
C(14)	2*i*	–0.2712(4)	0.4097(3)	0.6339(3)	0.043(3)	0.047(3)	0.035(2)	0.004(2)	0.010(2)	0.021(2)
C(15)	2*i*	–0.4156(5)	0.4208(4)	0.6066(3)	0.043(3)	0.065(3)	0.059(3)	–0.002(2)	0.006(2)	0.030(3)
C(16)	2*i*	–0.4554(5)	0.5251(5)	0.6419(4)	0.049(3)	0.081(4)	0.089(4)	0.019(3)	0.024(3)	0.042(3)
C(17)	2*i*	–0.3517(6)	0.6157(4)	0.7040(4)	0.074(4)	0.058(3)	0.071(3)	0.023(3)	0.034(3)	0.025(3)
C(18)	2*i*	–0.2058(5)	0.6061(4)	0.7314(3)	0.057(3)	0.051(3)	0.047(3)	–0.002(2)	0.017(2)	0.019(2)
C(19)	2*i*	–0.1645(4)	0.5042(3)	0.6963(3)	0.046(3)	0.040(3)	0.037(2)	0.006(2)	0.012(2)	0.019(2)
C(20)	2*i*	–0.2028(4)	0.1072(3)	0.7143(3)	0.039(2)	0.045(3)	0.043(2)	0.005(2)	0.011(2)	0.020(2)
C(21)	2*i*	–0.1435(5)	0.0157(4)	0.7236(3)	0.050(3)	0.050(3)	0.056(3)	0.010(2)	0.011(2)	0.022(2)
C(22)	2*i*	–0.2291(6)	–0.0884(4)	0.6884(4)	0.090(4)	0.043(3)	0.069(3)	0.012(3)	0.015(3)	0.023(3)
C(23)	2*i*	–0.3762(7)	–0.1039(4)	0.6418(4)	0.092(4)	0.052(3)	0.085(4)	–0.012(3)	0.008(3)	0.030(3)
C(24)	2*i*	–0.4378(6)	–0.0151(4)	0.6303(4)	0.054(3)	0.069(4)	0.107(4)	–0.014(3)	0.000(3)	0.035(3)
C(25)	2*i*	–0.3518(5)	0.0907(4)	0.6675(3)	0.055(3)	0.050(3)	0.083(3)	0.007(3)	0.008(3)	0.031(3)
C(26)	2*i*	0.1777(4)	0.4533(3)	0.9287(3)	0.036(2)	0.047(3)	0.041(3)	0.002(2)	0.009(2)	0.019(2)
C(27)	2*i*	0.2863(5)	0.5132(4)	0.9097(3)	0.045(3)	0.067(3)	0.054(3)	–0.006(2)	0.009(2)	0.028(3)
C(28)	2*i*	0.3889(5)	0.5989(4)	0.9851(4)	0.049(3)	0.066(3)	0.081(4)	–0.013(3)	0.008(3)	0.030(3)
C(29)	2*i*	0.3885(5)	0.6242(4)	1.0801(4)	0.056(3)	0.065(3)	0.068(4)	–0.005(3)	–0.003(3)	0.010(3)
C(30)	2*i*	0.2857(5)	0.5640(4)	1.1010(3)	0.064(3)	0.076(4)	0.044(3)	0.011(3)	0.009(3)	0.008(3)
C(31)	2*i*	0.1810(5)	0.4786(4)	1.0256(3)	0.047(3)	0.059(3)	0.045(3)	0.007(2)	0.012(2)	0.021(2)
C(32)	2*i*	0.0356(4)	0.2226(3)	0.9120(3)	0.032(2)	0.044(2)	0.037(2)	0.001(2)	0.007(2)	0.021(2)
C(33)	2*i*	–0.0688(4)	0.2008(3)	0.9550(3)	0.026(2)	0.044(2)	0.047(2)	0.007(2)	0.009(2)	0.023(2)
C(34)	2*i*	–0.0326(4)	0.1582(3)	1.0297(3)	0.028(2)	0.042(2)	0.038(2)	0.004(2)	0.013(2)	0.018(2)
C(35)	2*i*	0.1089(4)	0.1395(3)	1.0618(3)	0.041(2)	0.036(2)	0.035(2)	0.004(2)	0.008(2)	0.018(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(36)	2*i*	0.2125(4)	0.1602(3)	1.0183(3)	0.029(2)	0.038(2)	0.039(2)	0.004(2)	0.008(2)	0.018(2)
C(37)	2*i*	0.1754(4)	0.2019(3)	0.9435(3)	0.031(2)	0.049(3)	0.046(2)	0.002(2)	0.015(2)	0.022(2)
C(38)	2*i*	–0.1483(4)	0.1380(3)	1.0745(3)	0.032(3)	0.046(3)	0.049(3)	0.007(2)	0.009(2)	0.026(2)
O(39)	2*i*	–0.1022(3)	0.1160(3)	1.1537(2)	0.046(2)	0.102(3)	0.066(2)	0.013(2)	0.027(2)	0.054(2)
O(40)	2*i*	–0.2738(3)	0.1403(3)	1.0372(2)	0.036(2)	0.134(3)	0.090(2)	0.019(2)	0.025(2)	0.074(2)
C(41)	2*i*	0.3638(4)	0.1394(3)	1.0537(3)	0.032(2)	0.049(3)	0.050(3)	0.005(2)	0.009(2)	0.025(2)
O(42)	2*i*	0.4090(3)	0.1214(3)	1.1283(2)	0.046(2)	0.112(3)	0.074(2)	0.032(2)	0.018(2)	0.057(2)
O(43)	2*i*	0.4427(3)	0.1446(3)	0.9956(2)	0.031(2)	0.112(3)	0.088(2)	0.022(2)	0.027(2)	0.059(2)
O(44)	2*i*	–0.3145(4)	0.0763(3)	1.2226(3)	0.056(2)	0.100(3)	0.076(2)	0.010(2)	0.025(2)	0.053(3)
O(45)	2*i*	–0.3159(6)	–0.1345(4)	1.2525(4)	0.164(5)	0.135(4)	0.127(4)	0.058(3)	0.068(3)	0.083(3)
C(46)	2*i*	–0.2584(6)	–0.1491(6)	1.3288(5)	0.073(4)	0.113(5)	0.097(5)	0.026(4)	0.030(3)	0.074(4)
C(47)	2*i*	–0.3001(8)	–0.2554(6)	1.3349(6)	0.152(7)	0.121(6)	0.162(7)	0.037(5)	0.017(6)	0.090(6)
C(48)	2*i*	–0.156(1)	–0.0660(7)	1.4084(7)	0.147(8)	0.127(7)	0.22(1)	–0.025(6)	–0.014(8)	0.019(7)

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