Crystal structure of 1¹⁰-methoxy-6-thia-1(1,8)-anthracena-4,8(1,3)-dibenzacyclodeca-2,9-diynaphane, C₃₃H₂₂OS

G. T. Crisp, P. D. Turner and E. R. T. Tiekink*

The University of Adelaide, Department of Chemistry, Australia 5005

Received June 4, 1999, CCDC-No. 1267/203

Abstract

C₃₃H₂₂OS, triclinic, $P\bar{1}$ (No. 2), $a = 11.504(7)$ Å, $b = 11.621(8)$ Å, $c = 10.337(8)$ Å, $\alpha = 95.46(6)^\circ$, $\beta = 115.96(5)^\circ$, $\gamma = 98.48(5)^\circ$, $V = 1209.0$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.075$, $wR(F) = 0.088$, $T = 293$ K.

Discussion

Little delocalisation of π -electron density throughout the molecule is noted as evidenced by the magnitude of the acetylenic C2—C3 and C17—C18 bond distances of 1.197(7) Å and 1.193(7) Å, respectively. Further, the molecule is not planar as seen in the dihedral angles formed between the anthracene moiety

and the C1, C27–C31 and C19–C23, C32 six-membered rings of 12.0° and 38.9°, respectively. In the lattice, centrosymmetrically related molecules associate via π – π interactions (closest contact of 3.433(7) Å occurs between the C27 and C33) atoms and in addition via close O33...H26b' contacts of 2.77 Å such that $d(\text{O33} \cdots \text{C26}') = 3.491(7)$ Å.

Table 1. Data collection and handling.

Crystal:	yellow, multi-faceted, size 0.24 × 0.32 × 0.40 mm
Wavelength:	Mo K_{α} radiation (0.7107 Å)
μ :	1.58 cm ^{−1}
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
2 θ_{max} :	55.2°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5866, 5590
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 3 \sigma(I_{\text{obs}})$, 2469
$N(\text{param})_{\text{refined}}$:	316
Programs:	SIR92 [2], teXsan [3], DIFABS [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(5)	2i	−0.2221	0.5116	−0.2657	0.075
H(6)	2i	−0.1495	0.6589	−0.0625	0.073
H(7)	2i	0.0728	0.7464	0.07	0.067
H(10)	2i	0.1561	0.4621	−0.3006	0.057
H(13)	2i	0.532	0.7945	0.1319	0.068
H(14)	2i	0.679	0.7323	0.0668	0.077
H(15)	2i	0.6088	0.5856	−0.1429	0.081
H(20)	2i	0.1827	0.1741	−0.4617	0.078
H(22)	2i	0.2262	0.0022	−0.787	0.083
H(23)	2i	0.3721	0.1676	−0.7794	0.081
H(24a)	2i	0.0458	−0.009	−0.5797	0.105
H(24b)	2i	0.1208	−0.0971	−0.6159	0.105
H(26a)	2i	−0.0331	0.1203	−0.8321	0.082
H(26b)	2i	−0.1654	0.0516	−0.962	0.082
H(28)	2i	−0.3501	−0.0333	−0.8938	0.076
H(29)	2i	−0.4695	0.0328	−0.7892	0.087
H(30)	2i	−0.3757	0.1913	−0.5921	0.076
H(31)	2i	−0.0408	0.2351	−0.6395	0.064
H(32)	2i	0.4321	0.3342	−0.6019	0.075
H(34a)	2i	0.2889	0.7171	0.2736	0.099
H(34b)	2i	0.4328	0.7401	0.2944	0.099
H(34c)	2i	0.3787	0.8424	0.3412	0.099

* Correspondence author

(e-mail: edward.tiekink@adelaide.edu.au)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(25)	2i	-0.0550(2)	-0.0778(1)	-0.8272(2)	0.068(1)	0.047(1)	0.131(2)	0.0075(8)	0.039(1)	-0.010(1)
O(33)	2i	0.3129(3)	0.8019(3)	0.1327(4)	0.047(2)	0.049(2)	0.052(2)	0.007(2)	0.009(2)	0.003(2)
C(1)	2i	-0.1970(5)	0.2315(4)	-0.5984(5)	0.035(3)	0.041(3)	0.052(3)	0.008(2)	0.005(3)	0.009(3)
C(2)	2i	-0.1376(5)	0.3310(5)	-0.4784(6)	0.033(3)	0.050(4)	0.061(4)	0.008(3)	0.011(3)	0.017(3)
C(3)	2i	-0.0939(5)	0.4110(4)	-0.3768(6)	0.033(3)	0.039(3)	0.057(3)	0.002(2)	0.005(3)	0.004(3)
C(4)	2i	-0.0459(5)	0.5038(4)	-0.2565(6)	0.032(3)	0.044(3)	0.056(3)	0.005(2)	0.012(3)	0.012(3)
C(5)	2i	-0.1303(5)	0.5449(5)	-0.2118(7)	0.032(3)	0.051(4)	0.084(4)	0.002(3)	0.012(3)	0.008(3)
C(6)	2i	-0.0873(5)	0.6345(5)	-0.0906(6)	0.040(3)	0.055(4)	0.076(4)	0.010(3)	0.019(3)	0.000(3)
C(7)	2i	0.0437(5)	0.6862(4)	-0.0130(6)	0.046(3)	0.043(3)	0.063(4)	0.013(3)	0.012(3)	0.001(3)
C(8)	2i	0.1373(5)	0.6513(4)	-0.0544(5)	0.038(3)	0.037(3)	0.046(3)	0.007(2)	0.009(3)	0.007(2)
C(9)	2i	0.0953(5)	0.5590(4)	-0.1769(5)	0.034(3)	0.038(3)	0.052(3)	0.008(2)	0.010(2)	0.013(3)
C(10)	2i	0.1856(5)	0.5248(4)	-0.2199(5)	0.039(3)	0.037(3)	0.050(3)	0.003(2)	0.007(3)	0.003(2)
C(11)	2i	0.3200(5)	0.5815(4)	-0.1456(5)	0.036(3)	0.042(3)	0.052(3)	0.009(2)	0.011(3)	0.012(3)
C(12)	2i	0.3643(5)	0.6742(4)	-0.0215(5)	0.035(3)	0.043(3)	0.050(3)	0.004(2)	0.010(3)	0.010(3)
C(13)	2i	0.5018(5)	0.7307(5)	0.0522(6)	0.040(3)	0.046(3)	0.063(4)	0.000(3)	0.010(3)	0.007(3)
C(14)	2i	0.5879(5)	0.6965(5)	0.0119(7)	0.036(3)	0.060(4)	0.080(4)	-0.004(3)	0.017(3)	0.007(3)
C(15)	2i	0.5460(5)	0.6059(5)	-0.1132(7)	0.039(3)	0.064(4)	0.091(5)	0.011(3)	0.023(3)	0.019(4)
C(16)	2i	0.4147(5)	0.5485(5)	-0.1906(6)	0.037(3)	0.051(3)	0.062(4)	0.010(3)	0.012(3)	0.013(3)
C(17)	2i	0.3755(5)	0.4560(5)	-0.3101(7)	0.038(3)	0.062(4)	0.064(4)	0.014(3)	0.020(3)	0.019(3)
C(18)	2i	0.3470(5)	0.3760(6)	-0.4072(7)	0.042(4)	0.072(4)	0.067(4)	0.020(3)	0.011(3)	0.008(4)
C(19)	2i	0.3118(5)	0.2746(5)	-0.5192(6)	0.039(3)	0.072(4)	0.053(4)	0.023(3)	0.012(3)	0.013(3)
C(20)	2i	0.2202(6)	0.1738(6)	-0.5276(6)	0.051(4)	0.084(5)	0.063(4)	0.033(3)	0.021(3)	0.025(4)
C(21)	2i	0.1866(6)	0.0735(5)	-0.6338(6)	0.057(4)	0.065(4)	0.056(4)	0.021(3)	0.023(3)	0.019(3)
C(22)	2i	0.2458(6)	0.0724(5)	-0.7198(7)	0.060(4)	0.065(4)	0.070(4)	0.016(3)	0.019(3)	0.005(3)
C(23)	2i	0.3348(6)	0.1691(6)	-0.7134(6)	0.048(4)	0.088(5)	0.062(4)	0.017(3)	0.021(3)	0.006(4)
C(24)	2i	0.0802(7)	-0.0321(6)	-0.6446(8)	0.116(6)	0.072(5)	0.094(5)	0.040(4)	0.053(5)	0.040(4)
C(26)	2i	-0.1094(6)	0.0598(5)	-0.8603(6)	0.060(4)	0.063(4)	0.068(4)	0.020(3)	0.014(3)	0.009(3)
C(27)	2i	-0.1823(5)	0.0978(4)	-0.7816(6)	0.045(3)	0.043(3)	0.052(3)	0.009(3)	0.011(3)	0.008(3)
C(28)	2i	-0.3117(6)	0.0361(5)	-0.8206(6)	0.056(4)	0.048(4)	0.064(4)	0.001(3)	0.014(3)	-0.006(3)
C(29)	2i	-0.3807(5)	0.0729(5)	-0.7556(7)	0.037(4)	0.060(4)	0.090(5)	-0.004(3)	0.012(3)	-0.008(4)
C(30)	2i	-0.3267(5)	0.1690(5)	-0.6408(6)	0.038(3)	0.064(4)	0.070(4)	0.007(3)	0.012(3)	0.004(3)
C(31)	2i	-0.1285(5)	0.1936(4)	-0.6696(6)	0.031(3)	0.039(3)	0.066(4)	0.003(2)	0.002(3)	0.013(3)
C(32)	2i	0.3679(5)	0.2687(5)	-0.6101(6)	0.038(3)	0.077(4)	0.061(4)	0.011(3)	0.015(3)	0.009(3)
C(33)	2i	0.2723(5)	0.7068(4)	0.0193(5)	0.042(3)	0.035(3)	0.050(3)	0.006(2)	0.008(3)	0.002(3)
C(34)	2i	0.3567(6)	0.7729(6)	0.2716(7)	0.076(5)	0.074(5)	0.064(4)	0.014(4)	0.006(4)	0.001(4)

Acknowledgment. The Australian Research Council is thanked for the support.

References

1. Bodwell, G. J.; Houghton, T. J.; Koury, H. E.; Yarlagaadda, B.: Sodium sulfide adsorbed on alumina as a reagent for the facile synthesis of 2,11-dithia[3.3]metacyclopheanes. *Synlett* (1995) 751-752.
2. Altomare, A.; Cascarano, M.; Giacovazzo, C.; Guagliardi, A.: SIR92 - a program for automatic solution of crystal structures by direct methods. *J. Appl. Crystallogr.* **26** (1993) 343.
3. Molecular Structure Corporation. (1997-1998). *teXsan* for Windows version 1.03. Single Crystal Structure Analysis Software. Version 1.04. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
4. Walker, N.; Stuart, D.: An empirical method for correcting diffractometer data for absorption effects. *Acta Crystallogr. A* **39** (1983) 159-166.