Redetermination of 9,10-dimethyl-anthracene

Bernard Marciniak

Institute of Chemistry and Environment Protection, Jan Dlugosz University, al. Armii Krajowej 13/15, 42-200 Czestochowa, Poland
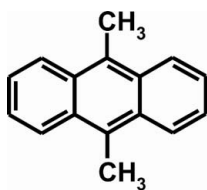Correspondence e-mail: crystal@cz.onet.pl

The crystal structure of the title compound, $C_{16}H_{14}$, was refined by Iball & Low [*Acta Cryst.* (1974). B**30**, 2203–2205] to an R value of 0.080. The present redetermination confirms the previous study but with improved precision and with all H atoms located and refined. The atoms of the methyl group are disordered over two sites with occupancies 0.55 (3) and 0.45 (3). The molecular symmetry is C_i. The crystal structure is stabilized by van der Waals interactions.

Related literature

For the previous structure determination, see: Iball & Low (1974) [refcode DMANTR, Cambridge Structural Database, Version 5.28; Allen, 2002)].

Experimental

Crystal data

$C_{16}H_{14}$	V = 557.98 (7) Å^3
M_r = 206.27	Z = 2
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 7.8229 (6) Å	μ = 0.07 mm^{-1}
b = 5.3093 (4) Å	T = 290 (2) K
c = 13.4649 (10) Å	$0.50 \times 0.16 \times 0.04$ mm
β = 93.861 (7)°	

Data collection

Oxford Diffraction Xcalibur3 CCD diffractometer	3013 measured reflections
	1003 independent reflections
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2007)	692 reflections with $I > 2\sigma(I)$
	R_{int} = 0.020
T_{min} = 0.947, T_{max} = 0.994	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.137	
S = 1.11	
1003 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
93 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Brandenburg, K. (2006). *DIAMOND*. Release 3.1.e. Crystal Impact GbR, Bonn, Germany.
Iball, J. & Low, J. M. (1974). *Acta Cryst.* B**30**, 2203–2205.
Oxford Diffraction (2007). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.31. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supplementary materials

Redetermination of 9,10-dimethylanthracene

B. Marciniak

Comment

In the literature, there is a single paper related to the crystal structure of 9,10-dimethylanthracene (I) [Iball & Low, 1974; CSD refcode DMANTR (Cambridge Structural Database, Version 5.28; Allen, 2002)]. In that determination, a reliability factor was R=0.080 for 712 data, and hydrogen atom coordinates were included in calculated positions without any refinement. However, the redetermination of (I) from recollected intensity data is presented here. The disorder of methyl group was detected over two sites (Fig. 1) with site occupancy factors 0.55 (3) and 0.45 (3). Apart from this disorder and the higher precision of the geometric parameters [σ(C—C) = 0.002–0.003 Å in the present work, compared with 0.009–0.012 Å in the earlier work (Iball & Low, 1974)], the results obtained agree well with the already published data. In the crystal structure of (I) the asymmetric unit consists of a half-molecule whereas the other half is generated by a centre of inversion. The molecule is planar with the largest r.m.s. deviation from the best least-square plane being 0.005 (1) Å for C5. The crystal packing is stabilized by van der Waals interactions (Fig. 2).

Experimental

The crystals of (I) were obtained from the vapour in a specially constructed glass apparatus consisting of the two main parts, namely a cold copper plate used as a substrate and an electrically heated molybdenic boat, both equipped with the Fe/constantan thermocouples which allowed an independent control of the T_1 and T_2 temperatures of these parts, respectively. Under a low background pressure of 10^{-3} mbar, the crystals were nucleated at 311 K/330 K (T_1/T_2) and grown at 318 K /330 K.

Refinement

All C-bound H atoms, except those on C8 atom were included in the refinement at the geometrically calculated positions and refined using a riding model with C—H distances of 0.93 Å and with U_{iso}(H)=1.2U_{eq}(C). The H atoms of methyl group were located in a difference Fourier map and refined with U_{iso}(H)=1.5U_{eq}(C). These atoms were found to be disordered over two sites with refined occupancies of 0.55 (3): 0.45 (3).
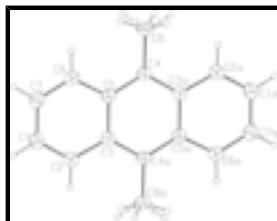
Figures

Fig. 1. View of molecule of (I) showing the atom-numbering scheme and disordered H atoms of methyl group. Displacement ellipsoids are drawn at the 20% probability level and H atoms are shown as small spheres of arbitrary radii. Symmetry code (*a*): $1 - x$, $-y$, $1 - z$.
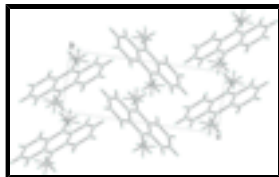
Fig. 2. The crystal packing, viewed along the *a* axis.

9,10-dimethylanthracene

Crystal data

$C_{16}H_{14}$	$F_{000} = 220$
$M_r = 206.27$	$D_x = 1.228$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3013 reflections
$a = 7.8229$ (6) Å	$\theta = 2.6–25.4°$
$b = 5.3093$ (4) Å	$\mu = 0.07$ mm^{-1}
$c = 13.4649$ (10) Å	$T = 290$ (2) K
$\beta = 93.861$ (7)°	Columnar, yellow
$V = 557.98$ (7) Å^3	$0.50 \times 0.16 \times 0.04$ mm
$Z = 2$	

Data collection

Oxford Diffraction Xcalibur3 CCD diffractometer	1003 independent reflections
Radiation source: fine-focus sealed tube	692 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
$T = 290(2)$ K	$\theta_{max} = 25.4°$
ω scans	$\theta_{min} = 2.6°$
Absorption correction: analytical (CrysAlis RED; Oxford Diffraction, 2007)	$h = -9\rightarrow9$
$T_{min} = 0.947$, $T_{max} = 0.994$	$k = -3\rightarrow6$
3013 measured reflections	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 0.0148P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.11$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
1003 reflections	$\Delta\rho_{min} = -0.17$ e Å^{-3}

93 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.13 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.1584 (2)	0.1546 (3)	0.30410 (13)	0.0641 (5)	
H1	0.0550	0.1503	0.2657	0.077*	
C2	0.1862 (2)	−0.0076 (3)	0.38108 (13)	0.0571 (5)	
H2	0.1008	−0.1222	0.3944	0.069*	
C3	0.34228 (18)	−0.0089 (2)	0.44254 (10)	0.0464 (5)	
C4	0.62951 (19)	0.1785 (2)	0.47711 (11)	0.0468 (5)	
C5	0.47336 (19)	0.1693 (2)	0.41972 (10)	0.0464 (5)	
C6	0.4366 (2)	0.3365 (3)	0.33742 (12)	0.0580 (5)	
H6	0.5191	0.4533	0.3216	0.070*	
C7	0.2856 (2)	0.3297 (3)	0.28208 (13)	0.0649 (6)	
H7	0.2658	0.4412	0.2293	0.078*	
C8	0.7676 (3)	0.3651 (4)	0.45414 (18)	0.0626 (6)	
H8A	0.798 (6)	0.474 (9)	0.514 (4)	0.094*	0.55 (3)
H8B	0.732 (7)	0.460 (10)	0.402 (4)	0.094*	0.55 (3)
H8C	0.871 (7)	0.268 (9)	0.440 (4)	0.094*	0.55 (3)
H8D	0.801 (8)	0.352 (10)	0.379 (5)	0.094*	0.45 (3)
H8E	0.876 (9)	0.355 (11)	0.495 (4)	0.094*	0.45 (3)
H8F	0.732 (8)	0.540 (11)	0.449 (5)	0.094*	0.45 (3)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0578 (11)	0.0703 (11)	0.0630 (11)	0.0084 (9)	−0.0039 (9)	−0.0041 (9)
C2	0.0506 (9)	0.0576 (10)	0.0629 (10)	−0.0015 (7)	0.0020 (8)	−0.0076 (8)
C3	0.0471 (9)	0.0439 (8)	0.0487 (9)	0.0016 (6)	0.0077 (7)	−0.0091 (6)
C4	0.0459 (9)	0.0446 (8)	0.0511 (9)	−0.0023 (6)	0.0116 (7)	−0.0077 (6)
C5	0.0507 (9)	0.0422 (8)	0.0476 (8)	0.0021 (6)	0.0126 (7)	−0.0057 (6)
C6	0.0615 (11)	0.0539 (10)	0.0596 (10)	0.0013 (7)	0.0109 (8)	0.0031 (7)

| C7 | 0.0689 (12) | 0.0666 (11) | 0.0592 (10) | 0.0119 (9) | 0.0048 (9) | 0.0048 (8) |
| C8 | 0.0575 (11) | 0.0609 (11) | 0.0699 (13) | −0.0118 (9) | 0.0093 (10) | 0.0028 (10) |

Geometric parameters (Å, °)

C1—C2	1.354 (2)	C6—C7	1.355 (2)
C1—C7	1.408 (3)	C6—H6	0.9300
C1—H1	0.9300	C7—H7	0.9300
C2—C3	1.428 (2)	C8—H8A	1.00 (5)
C2—H2	0.9300	C8—H8B	0.90 (5)
C3—C4^i	1.414 (2)	C8—H8C	0.99 (5)
C3—C5	1.443 (2)	C8—H8D	1.07 (6)
C4—C5	1.402 (2)	C8—H8E	0.98 (6)
C4—C8	1.513 (2)	C8—H8F	0.97 (6)
C5—C6	1.434 (2)		
C2—C1—C7	120.1 (2)	C4—C8—H8A	110 (2)
C2—C1—H1	119.9	C4—C8—H8B	110 (3)
C7—C1—H1	119.9	H8A—C8—H8B	110 (4)
C1—C2—C3	122.3 (2)	C4—C8—H8C	108 (3)
C1—C2—H2	118.9	H8A—C8—H8C	108 (4)
C3—C2—H2	118.9	H8B—C8—H8C	111 (4)
C4^i—C3—C2	122.0 (1)	C4—C8—H8D	112 (3)
C4^i—C3—C5	120.3 (1)	H8A—C8—H8D	137 (4)
C2—C3—C5	117.7 (1)	H8B—C8—H8D	51 (3)
C5—C4—C3^i	119.0 (1)	H8C—C8—H8D	62 (3)
C5—C4—C8	121.2 (2)	C4—C8—H8E	117 (3)
C3^i—C4—C8	119.8 (2)	H8A—C8—H8E	55 (3)
C4—C5—C6	121.7 (1)	H8B—C8—H8E	133 (4)
C4—C5—C3	120.7 (1)	H8C—C8—H8E	53 (3)
C6—C5—C3	117.5 (2)	H8D—C8—H8E	106 (4)
C7—C6—C5	122.0 (2)	C4—C8—H8F	116 (3)
C7—C6—H6	119.0	H8A—C8—H8F	63 (3)
C5—C6—H6	119.0	H8B—C8—H8F	48 (4)
C6—C7—C1	120.3 (2)	H8C—C8—H8F	136 (4)
C6—C7—H7	119.8	H8D—C8—H8F	95 (4)
C1—C7—H7	119.8	H8E—C8—H8F	109 (4)
C7—C1—C2—C3	0.1 (2)	C2—C3—C5—C4	179.9 (1)
C1—C2—C3—C4^i	179.9 (1)	C4^i—C3—C5—C6	−179.9 (1)
C1—C2—C3—C5	−0.5 (2)	C2—C3—C5—C6	0.6 (2)
C3^i—C4—C5—C6	179.8 (1)	C4—C5—C6—C7	−179.6 (1)
C8—C4—C5—C6	−0.5 (2)	C3—C5—C6—C7	−0.2 (2)
C3^i—C4—C5—C3	0.5 (2)	C5—C6—C7—C1	−0.2 (3)
C8—C4—C5—C3	−179.8 (2)	C2—C1—C7—C6	0.2 (3)
C4^i—C3—C5—C4	−0.5 (2)		

Symmetry codes: (i) −x+1, −y, −z+1.

Fig. 1

Fig. 2