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1,8-Dichloroanthracene

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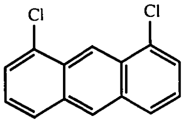
Abstract

The C—Cl distances in the title compound, $C_{14}H_8Cl_2$, average 1.751 (3) Å. The C atoms of the anthracene ring system exhibit a maximum deviation of 0.016 (5) Å from coplanarity and the Cl atoms are slightly displaced on opposite sides of the anthracene plane by −0.045 (1) and 0.035 (1) Å.

Comment

The structure of the title compound, (1), has been determined as part of our continuing work on the activation of 1,8-disubstituted anthracenes towards nucleophilic substitution. The unit-cell dimensions and space group of (1) have been previously reported by Desvergne, Chekpo & Bouas-Laurent (1978) to be a = 15.25, b = 18.90 and c = 4.00 Å, and *Pnma* (with Z = 4). The crystals were described as needles and measurements were made from Weissenberg photographs. As these authors apparently did not measure intensities and do not report coordinates, their space-group determination is ambiguous and space group $Pna2_1$ cannot be ruled out. Although the a and c dimensions match the present determination to approximately 1%, the b axial length is 4.5% longer than the c dimension here. While we cannot rule out the possibility of polymorphism, we note that a likely typographical error (18.09 *versus* 18.90) would account for the difference. We strongly suspect that the two crystal phases are identical.



(1)

The C—Cl distances in compound (1) [1.745 (4) and 1.756 (4) Å] agree well with the value of 1.749 Å reported for 1,8-dichloro-10-methylanthracene (Desvergne, Gaultier & Hauw, 1970) and are marginally longer than that of 1,8-dichloro-9-methylanthracene [1.726 (7) Å; Dellaca, Penfold & Robinson, 1969]. The molecular plane normal is inclined by 28.4 (1)° to the b-axis direction and the interplanar distance is 3.477 (1) Å. This stack of molecules forms a dihedral angle of 56.8 (2)° with that related to it by the a glide.

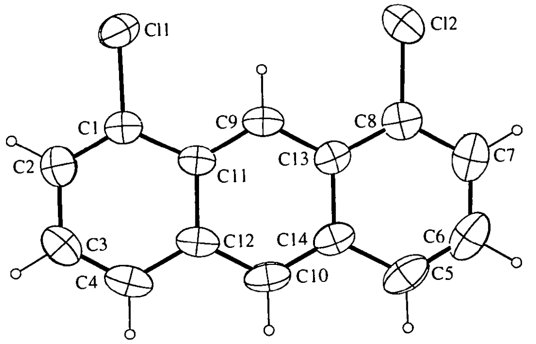


Fig. 1. The molecular structure of (1) showing 40% probability ellipsoids. H atoms are represented by spheres of arbitrary radii.

Experimental

The title compound was prepared according to the procedure of Collman *et al.* (1992) and crystallized from 2-propanol by slow evaporation.

Crystal data

$C_{14}H_8Cl_2$
M_r = 247.13
Orthorhombic
$Pna2_1$
a = 15.410 (3) Å
b = 3.953 (1) Å
c = 18.081 (6) Å
V = 1101.4 (9) Å^3
Z = 4
D_x = 1.490 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.554 mm^{-1}
T = 295 K
Lath
0.60 × 0.40 × 0.12 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min}$ = 0.949, $T_{\max}$ = 0.999
1952 measured reflections
1664 independent reflections

1358 observed reflections [$I > \sigma(I)$]
$\theta_{\max}$ = 30°
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 25$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F
R = 0.057

$\Delta\rho_{\max}$ = 0.49 e Å^{-3}
$\Delta\rho_{\min}$ = −0.35 e Å^{-3}

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$wR = 0.059$
$S = 1.955$
1358 reflections
145 parameters
H atoms riding (C—H 0.95 Å)
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$
$(\Delta/\sigma)_{\max} = 0.003$

Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: 0.18 (1) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl1	0.39882 (6)	0.0936 (3)	0.00000	0.0658 (3)
Cl2	0.44907 (7)	0.6548 (3)	0.24897 (8)	0.0660 (3)
C1	0.5098 (2)	0.008 (1)	−0.0047 (2)	0.0448 (9)
C2	0.5405 (3)	−0.152 (1)	−0.0644 (3)	0.056 (1)
C3	0.6307 (3)	−0.224 (1)	−0.0690 (3)	0.061 (1)
C4	0.6847 (3)	−0.134 (1)	−0.0134 (3)	0.058 (1)
C5	0.7328 (3)	0.388 (1)	0.2314 (3)	0.065 (1)
C6	0.7020 (3)	0.550 (1)	0.2901 (3)	0.073 (1)
C7	0.6134 (4)	0.633 (1)	0.2974 (3)	0.067 (1)
C8	0.5590 (3)	0.545 (1)	0.2401 (3)	0.053 (1)
C9	0.5320 (2)	0.282 (1)	0.1184 (2)	0.0448 (9)
C10	0.7075 (2)	0.126 (1)	0.1091 (3)	0.056 (1)
C11	0.5625 (2)	0.114 (1)	0.0564 (2)	0.0411 (9)
C12	0.6538 (2)	0.033 (1)	0.0506 (2)	0.047 (1)
C13	0.5861 (2)	0.376 (1)	0.1763 (2)	0.044 (1)
C14	0.6778 (2)	0.293 (1)	0.1716 (3)	0.049 (1)

Table 2. *Selected geometric parameters* (Å, °)

Cl1—C1	1.745 (4)	C6—C7	1.411 (8)
Cl2—C8	1.756 (4)	C7—C8	1.376 (7)
C1—C2	1.339 (6)	C8—C13	1.397 (6)
C1—C11	1.434 (6)	C9—C11	1.385 (6)
C2—C3	1.421 (7)	C9—C13	1.389 (6)
C3—C4	1.352 (7)	C10—C12	1.391 (6)
C4—C12	1.415 (6)	C10—C14	1.388 (7)
C5—C6	1.327 (7)	C11—C12	1.447 (5)
C5—C14	1.423 (7)	C13—C14	1.453 (5)
Cl1—C1—C2	118.5 (3)	C12—C10—C14	123.3 (4)
Cl1—C1—C11	117.5 (3)	C1—C11—C9	124.8 (3)
C2—C1—C11	124.0 (4)	C1—C11—C12	115.5 (3)
C1—C2—C3	119.2 (4)	C9—C11—C12	119.7 (3)
C2—C3—C4	120.4 (5)	C4—C12—C10	123.0 (4)
C3—C4—C12	121.6 (4)	C4—C12—C11	119.3 (4)
C6—C5—C14	121.4 (4)	C10—C12—C11	117.7 (4)
C5—C6—C7	122.2 (5)	C8—C13—C9	124.8 (4)
C6—C7—C8	117.4 (4)	C8—C13—C14	116.6 (4)
Cl2—C8—C7	117.2 (4)	C9—C13—C14	118.6 (4)
Cl2—C8—C13	118.8 (3)	C5—C14—C10	123.2 (4)
C7—C8—C13	124.1 (4)	C5—C14—C13	118.4 (4)
C11—C9—C13	122.3 (3)	C10—C14—C13	118.4 (4)

The crystal used for analysis was mounted in a capillary to prevent sublimation. The structure with the opposite sense of the polar axis was refined under identical circumstances, however, no significant coordinate shifts or differences in agreement indices were noted. The reported configuration was arbitrarily chosen.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: direct methods (*MULTAN*80; Main *et al.*, 1980). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN CIF IN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with a unit-cell stereopacking diagram, have been deposited with the IUCr (Reference: FG1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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n-Butyl(carboxymethyl)dimethylammonium Bromide and (Carboxymethyl)ethyldimethylammonium Bromide

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Abstract

The title compounds, $C_6H_{14}NO_2^+.Br^-$ and $C_8H_{18}NO_2^+.Br^-$, each crystallize forming three-dimensional networks of $Br^-\cdots N^+$ contacts and $Br^-\cdots H—O$ hydro-