

Crystal structure of 4,5-dichloro-anthracen-9(10H)-one, C₁₄H₈Cl₂O

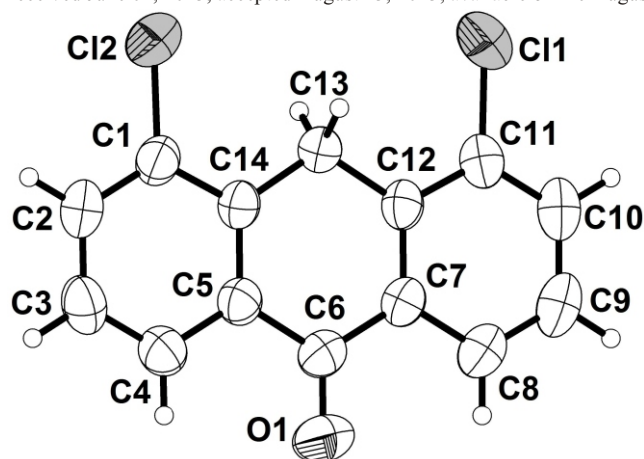
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Abstract

C₁₄H₈Cl₂O, monoclinic, *P*2₁/*c* (no. 14), *a* = 7.4510(4) Å, *b* = 15.0430(7) Å, *c* = 10.5973(4) Å, β = 104.829(3)°, *V* = 1148.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0470, *wR*_{ref}(*F*²) = 0.1494, *T* = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow needles, size 0.09 0.18 0.88 mm
Wavelength:	Cu <i>K</i> radiation (1.54178 Å)
μ:	48.94 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
2θ _{max} :	139.68°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	8175, 2119
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1540
<i>N</i> (<i>param</i>) _{refined} :	155
Programs:	SHELX [10]

Source of material

The synthesis of 4,5-dichloro-9(10*H*)-anthracenone was carried out according to the modified procedure [1]. To a suspension of the 1,8-dichloroanthracene-9,10(8*aH*,10*aH*)-dione (5 g, 18 mmol) in DMF (125 mL) and water (125 mL) was added Na₂S₂O₄ (35 g, 201.15 mmol), and the solution was slowly heated to 90 °C within 2h under N₂. The mixture was cooled to room temperature and then poured into water (1 L) and extracted with CH₂Cl₂ (4 50 mL). The combined organic phase was washed with water (4 100 mL), dried over Na₂SO₄, evaporated, and purified by column chromatography using CH₂Cl₂ as eluent to provide the 4,5-dichloro-9(10*H*)-anthracenone. The crystals of this compound were grown by slow evaporation of CH₂Cl₂.

Experimental details

Only a dataset with a low resolution has been collected which causes a low data-to-parameter ratio.

Discussion

Anthraquinones are group of functionally diverse aromatic chemicals, structurally related to anthracene [2]. Many anthraquinones exhibit biological activities such as antifungal, antioxidant, antibacterial, antiparasitic, insecticidal, antiviral and anti-tumor [3, 4]. Anthraquinone derivatives have also been widely used in analytical chemistry, mainly as strong chelating agents and chromophores [5]. In the textile industry, anthraquinone derivatives are used as dyes for natural and synthetic fibers [6]. In microelectronics, they were found to have application as semiconductors [7]. There is one complete molecule in the asymmetric unit. The title molecule is planar within the experimental standard uncertainties, which is in accord to related structures [8, 9]. The molecules are stacked parallel with the centroids of the outer rings to be only 3.73 Å apart.

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2A)	4e	0.5776	0.2083	0.7997	0.079
H(3A)	4e	0.6223	0.0729	0.9081	0.083
H(4A)	4e	0.4890	0.0539	0.8023	0.076
H(8A)	4e	0.1106	0.2056	0.4143	0.081
H(9A)	4e	0.0713	0.2031	0.2038	0.091
H(10A)	4e	0.1290	0.0696	0.0913	0.085
H(13A)	4e	0.2936	0.1176	0.3729	0.064
H(13B)	4e	0.1176	0.1342	0.4244	0.064

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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
Cl(1)	4e	0.0223(1)	0.10442(6)	0.14910(7)	0.1212(7)	0.0886(6)	0.0510(4)	0.0044(4)	0.0021(4)	0.0079(3)
Cl(2)	4e	0.3858(1)	0.26306(5)	0.55209(8)	0.1138(7)	0.0514(4)	0.0800(5)	0.0103(3)	0.0119(4)	0.0054(3)
O(1)	4e	0.3038(4)	0.1490(1)	0.6243(2)	0.136(2)	0.050(1)	0.085(2)	0.002(1)	0.002(1)	0.010(1)
C(1)	4e	0.4169(4)	0.1608(2)	0.6312(2)	0.065(2)	0.052(1)	0.057(1)	0.003(1)	0.018(1)	0.009(1)
C(2)	4e	0.5237(4)	0.1569(2)	0.7577(3)	0.068(2)	0.069(2)	0.060(2)	0.005(1)	0.014(1)	0.020(1)
C(3)	4e	0.5507(4)	0.0761(2)	0.8223(3)	0.065(2)	0.085(2)	0.051(1)	0.003(1)	0.003(1)	0.010(1)
C(4)	4e	0.4710(4)	0.0004(2)	0.7590(3)	0.068(2)	0.064(2)	0.055(1)	0.006(1)	0.011(1)	0.001(1)
C(5)	4e	0.3635(3)	0.0046(2)	0.6303(2)	0.057(1)	0.053(1)	0.051(1)	0.0041(9)	0.015(1)	0.003(1)
C(6)	4e	0.2798(4)	0.0781(2)	0.5663(3)	0.069(2)	0.050(1)	0.064(2)	0.004(1)	0.015(1)	0.002(1)
C(7)	4e	0.1652(3)	0.0738(2)	0.4308(2)	0.059(1)	0.051(1)	0.057(1)	0.001(1)	0.017(1)	0.008(1)
C(8)	4e	0.0881(4)	0.1520(2)	0.3695(3)	0.073(2)	0.056(2)	0.075(2)	0.006(1)	0.019(1)	0.013(1)
C(9)	4e	0.0205(4)	0.1505(2)	0.2438(3)	0.076(2)	0.073(2)	0.079(2)	0.014(1)	0.020(1)	0.031(2)
C(10)	4e	0.0546(4)	0.0710(2)	0.1764(3)	0.069(2)	0.085(2)	0.057(2)	0.009(1)	0.014(1)	0.022(1)
C(11)	4e	0.0226(4)	0.0056(2)	0.2367(2)	0.066(2)	0.070(2)	0.050(1)	0.003(1)	0.014(1)	0.008(1)
C(12)	4e	0.1344(3)	0.0073(2)	0.3639(2)	0.054(1)	0.056(1)	0.049(1)	0.0031(9)	0.0178(9)	0.009(1)
C(13)	4e	0.2176(3)	0.0926(2)	0.4260(2)	0.060(1)	0.050(1)	0.050(1)	0.0012(9)	0.014(1)	0.0026(9)
C(14)	4e	0.3337(3)	0.0854(2)	0.5633(2)	0.052(1)	0.052(1)	0.049(1)	0.0014(9)	0.0149(9)	0.0074(9)

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References

- Prinz, H.; Wiegerebe, W.; Müller, K.: Syntheses of Anthracenones. 1. Sodium Dithionite Reduction of peri-Substituted Anthracenediones. *J. Org. Chem.* **61** (1996) 2853-2856.
- Dave, H.; Ledwani, L.: A review on anthraquinones isolated from Cassia species and their applications. *IJNPR.* **3** (2012) 291-319.
- Kanokmedhakul, S.; Kanokmedhakul, K.; Phonkkered, N.; Soyong, K.; Kongsaree, P.; Suksamrarn, A.: Antimycobacterialanthraquinone-chromanone compound and diketopiperazine alkaloid from the fungus *Chaetomiumglobosum* KMITL-N0802. *Planta Med.* **68** (2002) 834-836.
- Srinivas, G.; Babykutty, S.; Sathiadevan, P.P.; Srinivas, P.: Molecularmechanism of emodin action: transition from laxative ingredient to an antitumor agent. *Med. Res. Rev.* **27** (2007) 591-608.
- Diaz A. N.: Analytical applications of 1,10-Anthraquinones: A review. *Talanta* **38** (1991) 571-588.
- Velmurugan, P.; Lee, Y. H.; Nanthakumar, K.; Kamala-Kannan, S.; Dufossé, L.; Mapari, S.; Oh, B.-T.: Water-soluble red pigments from *Isaria farinosa* and structural characterization of the main colored component. *J. Basic Microbiol.* **50** (2010) 581-590.
- Chaichit, N.; Sihanonth, P.; Petsom, A.; Sangvanich, P.: Austrocortinin: Crystal structure of a natural anthraquinone pigment from fungi. *Dyes Pigments.* **77** (2008) 653-656.
- Ziegler, K.; Gschwind, F.; Jansen, M.: Crystal structure of 9,10-bis-(2,6-difluorophenyl)-anthracene. *Z. Kristallogr. NCS* **228** (2013) 109-110.
- Li, H. M.; Wang, Z.-Q.; Xu, C.: Crystal structure of 10-bromo-9-phenyl-anthracene-anthracene-9,10-dione (2:1). *Z. Kristallogr. NCS* **228** (2013) 183-184.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.