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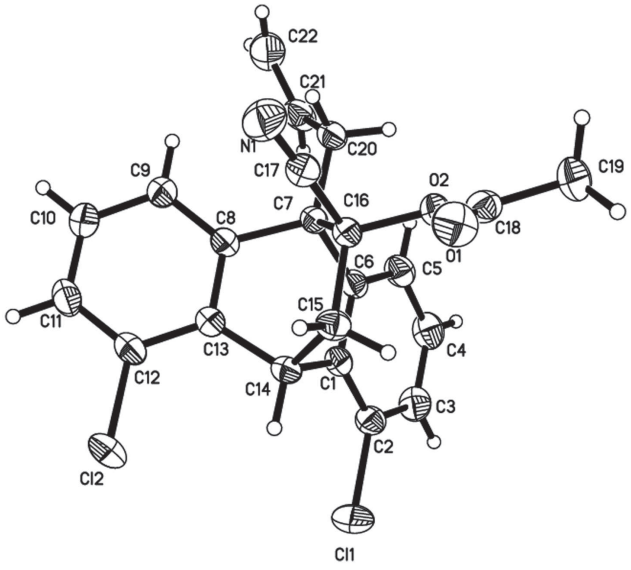
Mujeeb A. Sultan*, Usama Karama, Abdulrahman I. Almansour, Adel Al-saeedi and Hazem A. Ghabbour

Crystal structure of 9-allyl-4,5-dichloro-12-cyano-9,10-dihydro-9,10-ethanoanthracen-12-yl acetate, $C_{22}H_{17}Cl_2NO_2$



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Abstract

$C_{22}H_{17}Cl_2NO_2$, monoclinic, $P2_1/c$ (no. 14), $a = 8.9960(2)$ Å, $b = 15.9202(4)$ Å, $c = 13.2049(3)$ Å, $\beta = 94.369(1)°$, $V = 1885.69(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0420$, $wR_{ref}(F^2) = 0.1237$, $T = 100$ K.

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***Corresponding author: Mujeeb A. Sultan,** Chemistry Department, College of Science, King Saud University, P. O. Box 2455, Riyadh 11451, Kingdom of Saudi Arabia, e-mail: Alhosami1983@yahoo.co.uk
Usama Karama, Abdulrahman I. Almansour and Adel Al-saeedi: Chemistry Department, College of Science, King Saud University, P. O. Box 2455, Riyadh 11451, Kingdom of Saudi Arabia
Hazem A. Ghabbour: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P.O.Box 2457, Riaydh 11451, Saudi Arabia; and Department of Medicinal Chemistry, Faculty of Pharmacy, University of Mansoura, Mansoura 35516, Egypt

Table 1: Data collection and handling.

Crystal:	Colourless, plate, size 0.12×0.36×0.51 mm
Wavelength:	CuK_α radiation (1.54178 Å)
μ:	32.35 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	133.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11660, 3155
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2672
$N(param)_{refined}$:	245
Programs:	SHELX [14], Bruker programs [15]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(3A)	4e	0.6459	0.6626	0.0840	0.054
H(4A)	4e	0.4008	0.6605	0.0175	0.056
H(5A)	4e	0.2378	0.5583	0.0657	0.048
H(9A)	4e	0.2282	0.2675	0.1143	0.044
H(10A)	4e	0.3836	0.1520	0.1055	0.050
H(11A)	4e	0.6255	0.1563	0.1778	0.052
H(14A)	4e	0.6755	0.4315	0.3061	0.038
H(15A)	4e	0.4821	0.4008	0.4175	0.042
H(15B)	4e	0.4811	0.4980	0.3964	0.042
H(19A)	4e	0.1363	0.6464	0.4924	0.086
H(19B)	4e	−0.0021	0.5972	0.4435	0.086
H(19C)	4e	0.1063	0.6428	0.3739	0.086
H(20A)	4e	0.0764	0.3834	0.1959	0.044
H(20B)	4e	0.0869	0.4786	0.1689	0.044
H(21A)	4e	0.1615	0.4282	0.0016	0.054
H(22C)	4e	−0.0658	0.3265	0.0536	0.072
H(22A)	4e	−0.0150	0.3425	−0.0592	0.072

The figure shows the asymmetric unit of the title structure. Tables 1–3 contain details of the methods used and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

1-Cyanoviyle acetate (2.5 mL, 23.4 mmol) was added to a solution of 10-allyl-1,8-dichloroanthracene (2.4 g, 8.3 mmol)

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4*e*	0.81378(6)	0.56287(4)	0.22405(4)	0.0311(3)	0.0660(4)	0.0635(3)	−0.0095(2)	0.0059(2)	0.0028(3)
Cl(2)	4*e*	0.80010(6)	0.27827(4)	0.28241(5)	0.0335(3)	0.0576(4)	0.0740(4)	0.0139(2)	−0.0063(2)	0.0038(3)
O(1)	4*e*	0.2165(1)	0.51350(8)	0.33572(9)	0.0350(7)	0.0350(8)	0.0359(7)	0.0021(5)	0.0063(5)	−0.0045(5)
O(2)	4*e*	0.2401(2)	0.4932(1)	0.5050(1)	0.057(1)	0.069(1)	0.0357(8)	0.0004(7)	0.0070(6)	−0.0045(7)
N(1)	4*e*	0.1626(2)	0.3093(1)	0.3951(2)	0.062(1)	0.051(1)	0.066(1)	−0.0095(9)	0.0145(9)	0.008(1)
C(1)	4*e*	0.5344(2)	0.4981(1)	0.2030(1)	0.031(1)	0.033(1)	0.0305(9)	0.0042(6)	0.0043(6)	−0.0014(7)
C(2)	4*e*	0.6291(2)	0.5600(1)	0.1733(1)	0.031(1)	0.041(1)	0.038(1)	−0.0007(7)	0.0086(7)	−0.0027(8)
C(3)	4*e*	0.5809(2)	0.6213(1)	0.1037(2)	0.049(1)	0.040(1)	0.048(1)	−0.0017(8)	0.0139(8)	0.0045(9)
C(4)	4*e*	0.4344(2)	0.6197(1)	0.0642(2)	0.055(1)	0.040(1)	0.044(1)	0.0080(8)	0.0054(8)	0.0116(9)
C(5)	4*e*	0.3364(2)	0.5584(1)	0.0928(2)	0.039(1)	0.043(1)	0.038(1)	0.0075(7)	−0.0014(7)	0.0022(8)
C(6)	4*e*	0.3862(2)	0.4973(1)	0.1618(1)	0.030(1)	0.033(1)	0.0313(9)	0.0040(6)	0.0027(6)	−0.0036(7)
C(7)	4*e*	0.2932(2)	0.4250(1)	0.2003(1)	0.026(1)	0.032(1)	0.0318(9)	0.0025(6)	0.0008(6)	−0.0040(7)
C(8)	4*e*	0.3815(2)	0.3435(1)	0.1876(1)	0.028(1)	0.033(1)	0.0321(9)	0.0039(6)	0.0018(6)	−0.0004(7)
C(9)	4*e*	0.3264(2)	0.2701(1)	0.1418(1)	0.035(1)	0.035(1)	0.041(1)	0.0014(7)	−0.0001(7)	−0.0021(8)
C(10)	4*e*	0.4194(2)	0.2008(1)	0.1375(2)	0.045(1)	0.034(1)	0.047(1)	0.0012(7)	0.0044(8)	−0.0046(9)
C(11)	4*e*	0.5644(2)	0.2033(1)	0.1801(2)	0.044(1)	0.036(1)	0.052(1)	0.0111(8)	0.0089(8)	0.0027(9)
C(12)	4*e*	0.6179(2)	0.2768(1)	0.2263(1)	0.031(1)	0.040(1)	0.040(1)	0.0081(7)	0.0042(7)	0.0063(8)
C(13)	4*e*	0.5287(2)	0.3474(1)	0.2302(1)	0.030(1)	0.033(1)	0.0316(9)	0.0026(6)	0.0033(6)	0.0015(7)
C(14)	4*e*	0.5706(2)	0.4301(1)	0.2800(1)	0.025(1)	0.035(1)	0.0342(9)	0.0018(6)	−0.0004(6)	0.0010(7)
C(15)	4*e*	0.4636(2)	0.4433(1)	0.3654(1)	0.031(1)	0.043(1)	0.0301(9)	−0.0009(7)	0.0009(6)	−0.0009(7)
C(16)	4*e*	0.3003(2)	0.4372(1)	0.3198(1)	0.029(1)	0.032(1)	0.0327(9)	0.0014(6)	0.0044(6)	−0.0017(7)
C(17)	4*e*	0.2215(2)	0.3659(1)	0.3641(1)	0.035(1)	0.039(1)	0.039(1)	0.0000(7)	0.0047(7)	−0.0003(8)
C(18)	4*e*	0.1922(2)	0.5341(1)	0.4334(2)	0.036(1)	0.046(1)	0.038(1)	−0.0074(8)	0.0093(7)	−0.0091(9)
C(19)	4*e*	0.0999(3)	0.6121(2)	0.4360(2)	0.057(2)	0.055(2)	0.062(1)	0.007(1)	0.018(1)	−0.018(1)
C(20)	4*e*	0.1296(2)	0.4238(1)	0.1573(1)	0.028(1)	0.040(1)	0.042(1)	0.0035(7)	0.0001(7)	−0.0064(8)
C(21)	4*e*	0.1009(2)	0.4032(1)	0.0470(2)	0.033(1)	0.056(1)	0.044(1)	0.0098(8)	−0.0061(7)	−0.0105(9)
C(22)	4*e*	−0.0032(3)	0.3526(2)	0.0103(2)	0.053(2)	0.057(2)	0.066(2)	0.008(1)	−0.024(1)	−0.015(1)

in xylene (8 mL). The mixture was stirred and refluxed at 150°C for 24 h, cooled to room temperature and the solvent removed in vacuo. The residue was purified *via* flash column chromatography on silica gel using ethyl acetate/ hexane (1:10–1:5) to afford the 9-allyl-4,5-dichloro-12-cyano-9,10-dihydro-9,10-ethanoanthracen-12-yl acetate (2.8 g, 84.3%). The crystals of this compound were grown by slow evaporation of the solvent.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 13, 23, 43 or 137 option) [14].

Discussion

Ethano-bridged anthracene derivatives represent an important class of bioactive molecules that showed antidepressant activity [1]. The recent discovery that some antidepressants show a wide range of pharmaceutical activities such as antidepressants [2], antiproliferative agent against BL cell lines [3] and anti- multi-drug resistance (MDR) activity on cancer cells and the malaria strain *P. Falciparum* [4–6] led many groups work on the synthesis of novel 9,10-dihydro-9,10-ethanoanthracene derivatives [2, 7–13]. The asymmetric unit of the title structure contains one independent molecule. All bond lengths and angles of the title molecule are in the expected ranges. The packing of the title structure shows at least one non-classical hydrogen bond between C17A—H17B$\cdots$O1A with symmetry code: $-x+2$, $-y+1$, $-z+1$. In the absence of any classical hydrogen bond donor group there are no classical hydrogen bonds.

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