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Crystal structure of bis(9-aminoacridin-10-ium) tetrachloridocuprate(II) monohydrate, $C_{26}H_{24}Cl_4CuN_4O$

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Abstract

$C_{26}H_{24}Cl_4CuN_4O$, triclinic, $P\bar{1}$ (no. 2), $a = 9.967(1)$ Å, $b = 11.853(1)$ Å, $c = 13.370(2)$ Å, $\alpha = 101.981(8)^\circ$, $\beta = 106.586(8)^\circ$, $\gamma = 113.856(9)^\circ$, $V = 1287.0(3)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0300$, $wR_{ref}(F^2) = 0.0771$, $T = 295(2)$ K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The 9-aminoacridine hydrochloride monohydrate (49 mg/0.20 mmol) and $CuCl_2$ (27 mg/0.20 mmol) were dissolved in 15 mL water + ethanol (v/v 1:1), acidified with 2 mL of concentrated hydrochloric acid. The mixture was heated and stirred for 30 minutes. The clear solution was allowed to evaporate for a few days to give dark orange crystals of title compound (m.p. = 516.7 K).

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Table 1: Data collection and handling.

Crystal:	Plate, dark orange
Size:	$0.52 \times 0.31 \times 0.05$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.29 mm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Ruby CCD, ω -scans
$\theta_{\max}$, completeness:	26° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18871, 5061, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4392
$N(\text{param})_{\text{refined}}$:	349
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], ORTEPII [4]

Experimental details

All H atoms were positioned geometrically and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Comment

This study is part of our continuing interest in synthesis and structural characterisation of 9-aminoacridinium salts [5, 6].

The asymmetric unit of the title compound contains two 9-aminoacridin-10-ium cations, denoted A and B, one tetrachloridocuprate(II) anion and one water molecule (cf. left part of the figure). The geometric parameters (bond lengths and angles) characterising the 9-aminoacridin-10-ium and tetrachloridocuprate(II) ions are typical for these units.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cu1	0.59434(3)	0.67495(3)	0.29105(2)	0.03662(9)
Cl1	0.43066(8)	0.59752(7)	0.10760(5)	0.05417(16)
Cl2	0.61729(8)	0.78136(7)	0.45790(5)	0.05411(17)
Cl3	0.80395(7)	0.83689(6)	0.28255(6)	0.05367(16)
Cl4	0.52329(7)	0.47415(5)	0.30815(5)	0.04293(14)
O1W	0.2275(2)	0.2793(2)	−0.01533(19)	0.0623(5)
H1W	0.231(4)	0.268(4)	−0.077(3)	0.094 [*]
H2W	0.268(4)	0.360(4)	0.013(3)	0.094 [*]
C1A	0.9788(3)	0.2218(2)	0.46737(18)	0.0414(5)
H1A	1.061635	0.201984	0.475884	0.050 [*]
C2A	0.8668(3)	0.1644(2)	0.5068(2)	0.0490(6)
H2A	0.872481	0.104294	0.540517	0.059 [*]
C3A	0.7436(3)	0.1953(2)	0.49701(18)	0.0445(5)
H3A	0.669701	0.157341	0.526131	0.053 [*]
C4A	0.7302(2)	0.2800(2)	0.44563(16)	0.0372(5)
H4A	0.647865	0.300035	0.439792	0.045 [*]
C5A	0.8890(3)	0.5469(2)	0.23422(19)	0.0419(5)
H5A	0.800738	0.559192	0.227158	0.050 [*]
C6A	0.9869(3)	0.6019(2)	0.1850(2)	0.0506(6)
H6A	0.965947	0.652591	0.144811	0.061 [*]
C7A	1.1186(3)	0.5831(2)	0.1939(2)	0.0497(6)
H7A	1.184063	0.620631	0.159070	0.060 [*]
C8A	1.1521(3)	0.5104(2)	0.25320(18)	0.0393(5)
H8A	1.240347	0.498734	0.258562	0.047 [*]
C9A	1.0851(2)	0.37554(19)	0.37118(15)	0.0303(4)
N10A	0.8232(2)	0.41661(17)	0.34504(14)	0.0312(4)
H10A	0.754(3)	0.434(2)	0.3412(19)	0.037 [*]
C11A	0.9704(2)	0.31121(19)	0.41364(15)	0.0302(4)
C12A	0.8414(2)	0.33717(19)	0.40124(15)	0.0288(4)
C13A	1.0545(2)	0.45227(19)	0.30673(16)	0.0299(4)
C14A	0.9214(2)	0.47142(19)	0.29593(16)	0.0302(4)
N15A	1.2153(2)	0.3637(2)	0.39003(18)	0.0453(5)
H15A	1.236(3)	0.329(3)	0.430(2)	0.054 [*]
H15B	1.283(3)	0.403(3)	0.368(2)	0.054 [*]
C1B	0.3425(3)	0.1156(2)	0.18767(18)	0.0388(5)
H1B	0.310137	0.171866	0.162874	0.047 [*]
C2B	0.2683(3)	0.0464(2)	0.24289(19)	0.0466(6)
H2B	0.186318	0.056078	0.256408	0.056 [*]
C3B	0.3152(3)	−0.0395(2)	0.27951(19)	0.0474(6)
H3B	0.262380	−0.087889	0.316138	0.057 [*]
C4B	0.4362(3)	−0.0536(2)	0.26257(18)	0.0410(5)
H4B	0.465913	−0.111071	0.287536	0.049 [*]
C5B	0.8548(3)	0.0624(2)	0.13128(19)	0.0428(5)
H5B	0.884585	0.007701	0.161328	0.051 [*]
C6B	0.9365(3)	0.1293(2)	0.0781(2)	0.0467(6)
H6B	1.022322	0.119829	0.071937	0.056 [*]
C7B	0.8943(3)	0.2120(2)	0.03256(18)	0.0432(5)
H7B	0.951917	0.257176	−0.003488	0.052 [*]
C8B	0.7691(3)	0.2267(2)	0.04079(17)	0.0369(5)
H8B	0.741536	0.281845	0.009995	0.044 [*]
C9B	0.5488(2)	0.17315(19)	0.10772(15)	0.0296(4)
N10B	0.6407(2)	0.00808(17)	0.19260(15)	0.0352(4)
H10B	0.666(3)	−0.037(2)	0.217(2)	0.042 [*]
C11B	0.4685(2)	0.10336(18)	0.16717(15)	0.0296(4)
C12B	0.5165(2)	0.01881(19)	0.20725(15)	0.0310(4)
C13B	0.6804(2)	0.15944(19)	0.09542(15)	0.0293(4)
C14B	0.7246(2)	0.07629(19)	0.14069(16)	0.0319(4)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N15B	0.5039(3)	0.2486(2)	0.06516(17)	0.0420(5)
H15C	0.429(3)	0.252(3)	0.069(2)	0.050 [*]
H15D	0.548(3)	0.285(3)	0.028(2)	0.050 [*]

Analysis of the intermolecular interactions in the structure of title compound shows that adjacent 9-aminoacridin-10-ium cations interact via π – π interactions (with centroid···centroid distances in the 3.606(1)–4.058(1) Å range and separations from 3.418(1) to 3.491(1) Å), forming infinite π –stacked columns in the ABBA arrangement. The neighbouring columns are linked through N_(amino)–H···Cl (N15B···Cl1 = 3.276(2) Å), N_(amino)–H···O_(water) (N15B···O1W = 2.857(4) Å) and O_(water)–H···Cl (O1W···Cl1 = 3.221(2) Å) hydrogen bonds producing a cyclic [$\cdots$ N–H···O–H···Cl $\cdots$]₂ synthon [7] (*cf.* right part of the figure, highlighted in dark grey) to form blocks. In these blocks, the N_(acridine)–H···Cl (N10A···Cl4 = 3.254(2) Å and N10B···Cl3 = 3.283(2) Å) and O_(water)–H···Cl (O1W···Cl3 = 3.425(2) Å) hydrogen bonds are also observed. In the crystal packing, adjacent blocks are connected via N_(amino)–H···Cl (N15A···Cl2 = 3.422(3) Å and N15A···Cl4 = 3.406(3) Å) hydrogen bonds to form a cyclic [$\cdots$ N–H···Cl–Cu–Cl $\cdots$]₂ synthon [7], (*cf.* right part of the figure, highlighted in light grey) producing a three-dimensional structure.

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