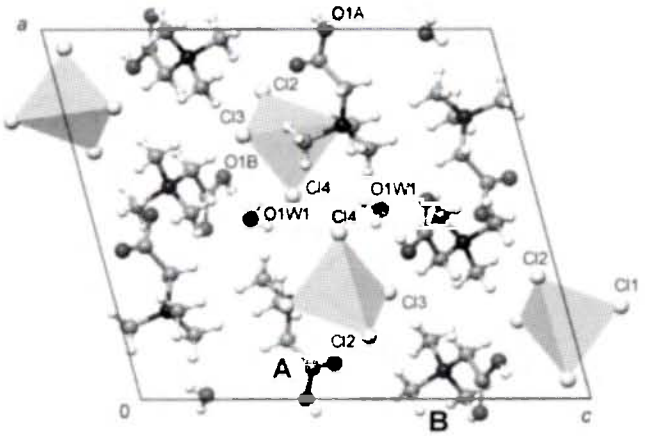
Z. Kristallogr. NCS 221 (2006) 525-526 / DOI 10.1524/ncrs.2006.0183

Crystal structure of bis(hydrogenbetaine) tetrachlorocuprate(II) monohydrate, $[(CH_3)_3NCH_2COOH]_2[CuCl_4] \cdot H_2O$

J. Schreuer, L. Wiehl*, A. Wagner and P. Hofmann

Universität Frankfurt, Institut für Geowissenschaften, Senckenberganlage 30, 60054 Frankfurt am Main, Germany

Received October 17, 2006, accepted and available on-line December 18, 2006; CCDC no. 1267/1912



Abstract

$C_{10}H_{26}Cl_4CuN_2O_5$, monoclinic, $P12_1/n1$ (no. 14), a = 14.053(2) Å, b = 9.1089(7) Å, c = 16.409(2) Å, β = 105.013(9)°, V = 2028.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.085, T = 293 K.

Source of material

Stoichiometric amounts of betaine monohydrate ($C_5H_{11}NO_2 \cdot H_2O$) and copper dichloride dihydrate ($CuCl_2 \cdot 2H_2O$) were dissolved in distilled water with an addition of hydrochloric acid (HCl) under stirring at about 320 K. By evaporation of the solvent at about 293 K crystals of two different phases emerged. Green needles could be identified as $CuCl_2 \cdot 2H_2O$. Bulky masses of amber yellow crystals proved to be a new phase, which is described here. All starting materials were commercial products.

Discussion

The structure consists of distorted $[CuCl_4]^{2-}$ anions, betaine cations, $[(CH_3)_3NCH_2COOH]^+$, and water molecules. The protonation of both inequivalent betaine molecules is clearly evident from the two different C—O bond lengths of their carboxylic groups, namely d(C5A—O1A) = 1.316 Å and d(C5A—O2A) = 1.188 Å for betaine A and, respectively, d(C5B—O1B) = 1.310 Å and d(C5B—O2B) = 1.193 A) for betaine B. Pairs of severely flattened tetrachlorocuprate tetrahedra, which are related by an inversion center, are linked by two water molecules via hydrogen bonds characterized by d(O1W1···Cl3) = 3.377 Å and d(O1W1···Cl4) = 3.370 Å. Further, the betaine molecule A is linked to Cl2 by a hydrogen bond (d(O1A···Cl2) = 3.096 Å), resulting in a linear arrangement of the type betaine –[$CuCl_4$]–$(H_2O)_2$–[$CuCl_4$]–betaine. An additional strong hydrogen bond connects the betaine molecule B with the water molecule (d(O1B···O1W1) = 2.634 Å).

Table 1. Data collection and handling.

Crystal:	amber yellow block, size 0.15 × 0.17 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	16.22 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Xcalibur3 & Sapphire3 CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28624, 3944
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3186
$N(param)_{refined}$:	303
Programs:	SHELXS-97 [1], SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(12A)	4e	0.467(3)	0.366(4)	0.881(2)	0.06(1)
H(11A)	4e	0.640(2)	0.374(3)	0.807(2)	0.041(8)
H(10A)	4e	0.670(2)	0.482(3)	0.871(2)	0.043(8)
H(9A)	4e	0.800(3)	0.380(4)	0.786(2)	0.07(1)
H(8A)	4e	0.888(2)	0.356(3)	0.860(2)	0.043(8)
H(7A)	4e	0.825(2)	0.500(4)	0.861(2)	0.07(1)
H(6A)	4e	0.766(3)	0.287(4)	1.011(2)	0.07(1)
H(5A)	4e	0.799(3)	0.437(5)	0.993(3)	0.09(1)
H(4A)	4e	0.868(3)	0.312(4)	0.994(2)	0.07(1)
H(3A)	4e	0.727(2)	0.116(4)	0.894(2)	0.05(1)
H(2A)	4e	0.830(3)	0.130(4)	0.883(2)	0.061(9)
H(1A)	4e	0.737(3)	0.156(4)	0.810(3)	0.09(1)
H(12B)	4e	0.567(3)	–0.216(5)	0.327(3)	0.09(2)
H(11B)	4e	0.652(3)	0.093(4)	0.299(2)	0.07(1)
H(10B)	4e	0.667(3)	0.004(4)	0.227(2)	0.06(1)
H(9B)	4e	0.624(3)	0.343(5)	0.135(3)	0.09(1)
H(8B)	4e	0.700(2)	0.206(3)	0.157(2)	0.055(9)
H(7B)	4e	0.688(3)	0.298(4)	0.228(3)	0.08(1)
H(6B)	4e	0.488(3)	0.336(6)	0.186(3)	0.11(2)
H(5B)	4e	0.538(4)	0.294(6)	0.275(3)	0.12(2)
H(4B)	4e	0.452(3)	0.199(4)	0.227(2)	0.07(1)
H(3B)	4e	0.509(3)	0.174(4)	0.071(2)	0.07(1)
H(2B)	4e	0.469(3)	0.043(4)	0.113(2)	0.07(1)
H(1B)	4e	0.573(3)	0.039(4)	0.089(2)	0.07(1)
H(1W1)	4e	–0.024(4)	0.067(6)	0.128(4)	0.13(3)
H(2W1)	4e	0.032(4)	0.146(5)	0.115(3)	0.09(2)

* Correspondence author (e-mail: l.wiehl@kristall.uni-frankfurt.de)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	4e	0.78645(2)	0.79764(4)	0.01757(2)	0.0326(2)	0.0488(2)	0.0423(2)	0.0050(2)	0.0118(1)	0.0003(2)
Cl(1)	4e	0.75109(6)	0.77572(9)	−0.12202(4)	0.0718(6)	0.0594(5)	0.0414(4)	−0.0024(4)	0.0071(3)	−0.0011(3)
Cl(2)	4e	0.66880(7)	0.6476(1)	0.04454(6)	0.0579(5)	0.0689(5)	0.0966(7)	−0.0059(4)	0.0433(5)	0.0061(5)
Cl(3)	4e	0.78805(8)	0.9673(1)	0.11464(6)	0.0865(7)	0.0678(5)	0.0728(6)	0.0083(5)	0.0283(5)	−0.0213(4)
Cl(4)	4e	0.94929(7)	0.7832(2)	0.03997(6)	0.0342(5)	0.191(1)	0.0717(6)	0.0232(6)	0.0120(4)	−0.0096(7)
O(1A)	4e	0.5004(2)	0.3930(3)	0.8684(2)	0.036(1)	0.080(2)	0.084(2)	0.009(1)	0.025(1)	0.029(1)
O(2A)	4e	0.5963(2)	0.2306(3)	0.9520(2)	0.053(1)	0.072(2)	0.079(2)	0.007(1)	0.030(1)	0.034(1)
N(1A)	4e	0.7612(2)	0.3208(2)	0.8890(1)	0.034(1)	0.039(1)	0.037(1)	0.0036(9)	0.0111(9)	0.0009(9)
C(5A)	4e	0.5842(2)	0.3245(3)	0.9001(2)	0.036(2)	0.048(2)	0.049(2)	0.001(1)	0.015(1)	0.003(1)
C(4A)	4e	0.6605(2)	0.3859(3)	0.8602(2)	0.033(2)	0.045(2)	0.043(2)	0.003(1)	0.009(1)	0.008(1)
C(3A)	4e	0.8251(3)	0.4013(4)	0.8435(2)	0.039(2)	0.062(2)	0.057(2)	0.003(2)	0.023(2)	0.007(2)
C(2A)	4e	0.8046(3)	0.3415(5)	0.9819(2)	0.044(2)	0.082(3)	0.038(2)	0.011(2)	0.006(1)	−0.003(2)
C(1A)	4e	0.7614(3)	0.1622(4)	0.8668(3)	0.057(3)	0.045(2)	0.087(3)	0.004(2)	0.021(2)	−0.010(2)
N(1B)	4e	0.5753(2)	0.1659(3)	0.1899(1)	0.038(1)	0.050(1)	0.046(1)	0.001(1)	0.016(1)	0.005(1)
O(1B)	4e	0.6012(2)	−0.1671(3)	0.3170(2)	0.049(2)	0.075(2)	0.073(2)	0.003(1)	0.004(1)	0.030(1)
O(2B)	4e	0.4625(2)	−0.0620(3)	0.2453(2)	0.037(1)	0.077(2)	0.076(2)	0.000(1)	0.014(1)	0.023(1)
C(5B)	4e	0.5502(2)	−0.0639(3)	0.2695(2)	0.040(2)	0.059(2)	0.036(1)	0.003(1)	0.010(1)	0.006(1)
C(4B)	4e	0.6199(2)	0.0492(4)	0.2504(2)	0.035(2)	0.063(2)	0.050(2)	0.001(2)	0.009(1)	0.007(2)
C(3B)	4e	0.6567(3)	0.2639(4)	0.1782(3)	0.058(2)	0.058(2)	0.083(3)	−0.008(2)	0.036(2)	−0.002(2)
C(2B)	4e	0.5056(4)	0.2590(5)	0.2233(3)	0.061(3)	0.066(2)	0.098(3)	0.013(2)	0.043(2)	0.011(2)
C(1B)	4e	0.5241(3)	0.1009(5)	0.1063(2)	0.070(3)	0.088(3)	0.040(2)	−0.017(2)	0.007(2)	0.013(2)
O(1W1)	4e	0.0123(3)	0.1251(4)	0.1480(2)	0.078(2)	0.072(2)	0.064(2)	−0.009(2)	0.031(2)	−0.016(1)

Acknowledgment. The authors gratefully acknowledge the financial support of the DFG (Forschergruppe 412 'Spin- und Ladungskorrelationen in niedrigdimensionalen Festkörpern').

References

1. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
2. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.