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# Crystal structure of the first characterized polymeric copper and sodium complex diaqua-(tris-acetato-κ*O*,*O*′)(μ²-acetato-κ*O*′′)dinatrium copper(II) monohydrate, $C_8H_{18}CuNa_2O_{11}$

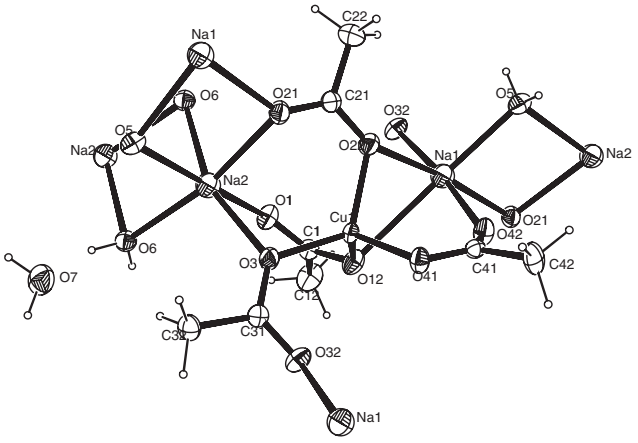


**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.71 × 0.55 × 0.23 mm |
| Wavelength: | Mo *Kα* radiation (0.71073 Å) |
| $\mu$: | 15.0 $cm^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$ |
| $2\theta_{max}$, completeness: | 56.6°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 22366, 3912, 0.017 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3628 |
| $N(param)_{refined}$: | 227 |
| Programs: | Bruker programs [1], SHELX [2, 3], ShelXle [4], ORTEP-3 [5], PLATON [6], Mercury CSD [7] |

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## Abstract

$C_8H_{18}CuNa_2O_{11}$, monoclinic, $P2_1/n$ (no. 14), $a = 9.9625(5)$ Å, $b = 14.0145(7)$ Å, $c = 11.3376(6)$ Å, $\beta = 97.602(2)°$, $V = 1569.04(14)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0178$, $wR_{ref}(F^2) = 0.0482$, $T = 200$ K.

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The crystal structure is shown in the figure. 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 material

31% HBr/acetic acid (1 mol equiv) was added to a solution of D-glucose (1 mol equiv) in acetic anhydride (6 mol equiv) under vigorous stirring, at 0 °C for 30 min. The temperature of the reaction mixture was maintained at room temperature and additional 31% HBr/acetic acid (5 mol equiv) was added. The resulting mixture was stirred for 5 h followed by the addition of anhydrous sodium acetate (10 g), $CuSO_4 \cdot 5H_2O$ (1 g) and zinc (5 g) in a solution of water (10 mL) and acetic acid (15 mL). Stirring was continued for further 3 h, solid was removed and the fitrate was washed with ethylacetate (200 mL) and then with water (100 mL). The organic layer of the filtrate was washed with saturated aqueous $NaHC0_3$ (100 mL), then with brine (50 mL) and dried ($Na_2SO_4$). The crude product obtained was recrystallized in water-ethanol mixture and afforded crystals suitable for crystal structure analysis.

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cu1 | 0.62979(2) | 0.30982(2) | 0.58404(2) | 0.01601(5) |
| Na1 | 0.74790(4) | 0.15630(3) | 0.78110(4) | 0.02386(10) |
| Na2 | 0.84320(4) | 0.47470(3) | 0.52589(4) | 0.01975(9) |
| O5 | 0.69319(9) | 0.14333(6) | 0.97858(8) | 0.02546(17) |
| O6 | 0.94525(8) | 0.49504(6) | 0.35076(7) | 0.02241(16) |
| O7 | 0.91756(12) | 0.64184(8) | 0.17966(10) | 0.0406(2) |
| O11 | 0.91637(9) | 0.30969(6) | 0.53285(8) | 0.02725(19) |
| O12 | 0.74826(9) | 0.20532(6) | 0.54116(8) | 0.02674(18) |
| O21 | 0.75407(8) | 0.48535(6) | 0.71032(7) | 0.02392(17) |
| O22 | 0.71298(8) | 0.33388(6) | 0.74973(7) | 0.02405(17) |
| O31 | 0.63107(8) | 0.40883(6) | 0.46384(7) | 0.02380(17) |
| O32 | 0.48033(8) | 0.33742(6) | 0.32996(8) | 0.02654(18) |
| O41 | 0.43975(8) | 0.27975(6) | 0.60135(7) | 0.02322(17) |
| O42 | 0.51953(8) | 0.15852(6) | 0.71535(8) | 0.02670(18) |
| C11 | 0.86739(11) | 0.22910(8) | 0.51831(10) | 0.0213(2) |
| C12 | 0.94723(14) | 0.15036(10) | 0.47001(14) | 0.0354(3) |
| H12A | 0.8856 | 0.1085 | 0.4191 | 0.053* |
| H12B | 0.9944 | 0.1133 | 0.5362 | 0.053* |
| H12C | 1.0136 | 0.1780 | 0.4234 | 0.053* |
| C21 | 0.75449(10) | 0.41799(8) | 0.78056(10) | 0.0196(2) |
| C22 | 0.80233(14) | 0.43330(11) | 0.91107(11) | 0.0338(3) |
| H22A | 0.8699 | 0.4845 | 0.9204 | 0.051* |
| H22B | 0.8429 | 0.3743 | 0.9457 | 0.051* |
| H22C | 0.7252 | 0.4511 | 0.9519 | 0.051* |
| C31 | 0.55643(10) | 0.40478(8) | 0.36274(10) | 0.0188(2) |
| C32 | 0.56903(12) | 0.48937(9) | 0.28313(11) | 0.0257(2) |
| H32A | 0.5128 | 0.4792 | 0.2065 | 0.038* |
| H32B | 0.6638 | 0.4970 | 0.2703 | 0.038* |
| H32C | 0.5387 | 0.5470 | 0.3208 | 0.038* |
| C41 | 0.42670(11) | 0.21166(8) | 0.67322(9) | 0.0182(2) |
| C42 | 0.28600(12) | 0.19667(10) | 0.70671(13) | 0.0324(3) |
| H42A | 0.2400 | 0.2584 | 0.7085 | 0.049* |
| H42B | 0.2927 | 0.1669 | 0.7855 | 0.049* |
| H42C | 0.2342 | 0.1551 | 0.6479 | 0.049* |
| H5A | 0.7588(13) | 0.1666(13) | 1.0241(15) | 0.052(5)* |
| H5B | 0.6222(12) | 0.1645(13) | 1.0018(16) | 0.054(6)* |
| H6A | 0.9360(17) | 0.5415(9) | 0.3036(13) | 0.045(5)* |
| H6B | 0.9677(18) | 0.4484(9) | 0.3118(13) | 0.045(5)* |
| H7A | 0.961(2) | 0.6932(10) | 0.186(2) | 0.075(8)* |
| H7B | 0.8723(19) | 0.6448(15) | 0.1121(11) | 0.066(6)* |

## Experimental details

The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bonds to best fit the experimental electron density (HFIX 137 in the SHELX program suite [2]), with $U_{iso}$(H) set to 1.5$U_{eq}$(C). The H atoms of the water molecules were located on a difference Fourier map and refined with the O—H bond lengths and the H···H 1,3 distance (to restrain the H—O—H bond angle) restrained to 0.84 Å and 1.34 Å, respectively.

## Discussion

The asymmetric unit contains one copper cation, two sodium cations, four acetato anions and three water molecules, which pack to give a three-dimensional polymeric structure. Four oxygen atoms (O12, O22, O31, O41) from each of the four acetate anions are coordinated to the copper centre, which adopts a distorted tetrahedral geometry. The shortest and longest Cu—O bond distances are 1.9461(9) and 1.9814(9) Å and involve O31 and O12, respectively. The smallest and largest O—Cu—O bond angles are 99.82(4) and 120.16(4)°, respectively. The Cu atom is bridged to each of the sodium cations via three acetate anions. The copper to sodium distances are 3.2109(5) and 3.2658(5) Å (to Na1 and Na2, respectively). Na1 has a square pyramid geometry with five oxygen atoms, four (O21 [3/2−*x*, −1/2+*y*, 3/2−*z*], O22, O32 [1/2+*x*, 1/2−*y*, 1/2+*z*] and O42) from acetate anions and one (O5) from a water molecule. The shortest and longest Na1—O bond distances are 2.2987(9) and 2.5315(9) Å and involve O42 and O22, respectively. The tau-descriptor for 5-coordination is 0.06 [8]. The least-square plane defined by the four acetate oxygen anions bonded to Na1 has a r.m.s. deviation of 0.0116 with Na1 0.0842(6) Å above the plane. Atom O12 has a distance of 2.8062(10) Å to Na1 and would give Na1 an octahedral geometry if O12 were coordinating. Na2 has an octahedral geometry with six oxygen atoms, three (O5 [3/2−*x*, 1/2+*y*, 3/2−*z*], O6 and O6 [2−*x*, 1−*y*, 1−*z*]) from water molecules and three (O11, O21 and O31) from acetate anions. The shortest and longest Na2—O bond distances are 2.3281(9) and 2.4229(10) Å (to O31 and O11, respectively). The two sodium cations Na1 and Na2 [3/2−*x*, −1/2+*y*, 3/2−*z*] are bridged by two oxygen atoms, one from water O5 and one from acetate anion O21 [3/2−*x*, −1/2+*y*, 3/2−*z*]. The Na1 to Na2 distance is 3.5518(6) Å. Two Na2 atoms are also bridged by two water O atoms, O6 and O6 [2−*x*, 1−*y*, 1−*z*]. The Na2 to Na2 [2−*x*, 1−*y*, 1−*z*] distance is 3.3307(6) Å. There are a number of hydrogen bonds in the structure. The shortest interaction is O6—H6B···O42 [1/2+*x*, 1/2−*y*, −1/2+*z*] with a distance of 1.964(14) Å. The uncoordinated water molecule, O7, receives one hydrogen bond and donates two.

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