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## Structure of Bis[ $\mu$ -4-amino-3,5-bis(aminomethyl)-1,2,4-triazole- $N'$ , $N^1$ , $N^2$ , $N''$ ]-bis[diaquacopper(II)] Bis(sulfate) Tetrahydrate

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**Abstract.**  $[\text{Cu}(\text{C}_4\text{H}_{10}\text{N}_6)(\text{H}_2\text{O})_2]_2(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$ ,  $M_r = 747.6$ , monoclinic,  $C2/c$ ,  $a = 19.009$  (8),  $b = 11.273$  (4),  $c = 13.144$  (7) Å,  $\beta = 103.50$  (4)°,  $Z = 4$  (dinuclear units),  $D_x = 1.81$  g cm<sup>-3</sup>,  $\lambda = 0.71073$  Å,  $\mu = 17.88$  cm<sup>-1</sup>,  $F(000) = 1544$ ,  $T = 293$  K, final  $R = 0.0583$ ,  $wR = 0.0619$  for 1019 reflections [ $I > 2\sigma(I)$ ]. The title compound represents the first doubly  $N1, N2$  triazole-bridged copper(II) compound in which the anion is not involved in coordination. The symmetry-dependent copper(II) ions are in a distorted octahedral environment, with four N-donor atoms of the triazole ligand in the equatorial positions of which the Cu—N(triazole) distances [1.954 (9) and 1.96 (1) Å] are shorter than the Cu—N(amino) distances [2.04 (1) and 2.052 (9) Å]. Two water molecules are coordinated axially at 2.60 (1) and 2.63 (1) Å. The sulfate anions are not coordinated and are fixed in the lattice by an extended hydrogen-bonding network with the amino group of the triazole and the water molecules.

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**Introduction.** One of the strategies in obtaining polynuclear transition-metal(II) coordination compounds is the use of polyfunctional ligands that contain  $N_2$  diazine moieties such as 1,2,4-triazole. The most extensively studied are the dinuclear metal(II) compounds where the metal ions are linked by an  $N1, N2$  triazole bridge (Prins, Birker, Haasnoot, Verschoor & Reedijk, 1985; Koomen-van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989; Keij, de Graaff, Haasnoot & Reedijk, 1984). For this group of compounds the structures have been studied in relation to their magnetic properties and magneto-structural correlations have been made. Recently, a class of compounds of the general formula  $[\text{Cu}(\text{aamt})A(\text{H}_2\text{O})_2A_2(\text{H}_2\text{O})_x(\text{CH}_3\text{OH})_y]$  [aamt = 4-amino-3,5-bis(aminomethyl)-1,2,4-triazole ( $\text{C}_4\text{H}_{10}\text{N}_6$ );  $A = \text{Br}^-$ ,  $\text{ClO}_4^-$ ,  $\text{CF}_3\text{SO}_3^-$ ,  $\text{BF}_4^-$ ,  $\text{NO}_3^-$ ] has been reported (Koomen-van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989). Here the equatorial positions of copper(II) are occupied by four ligand N-donor atoms. The axial positions are occupied by a bromine anion and a water molecule. In analogy

Table 1. *Experimental data*

Quadrants	<i>h, k, l</i>
$\theta$ range (°)	2–30
Standard reflections	$\bar{1}400, \bar{8}00, \bar{3}00$
Variation in standard intensities (%)	7.16
No. of reflections measured	4347
No. of independent reflections	4184
$R_{\text{int}}$	0.072
Significant reflections [ $I > 2\sigma(I)$ ]	1019
<i>h</i>	–26 to 26
<i>k</i>	0 to 15
<i>l</i>	0 to 18
Crystal size (mm)	$0.3 \times 0.2 \times 0.15$
Transmission factor range	0.4546–0.6031
Max. shift/e.s.d. in final cycle	0
$\rho_{\text{max}}$ (e Å <sup>–3</sup> )	0.79
$\rho_{\text{min}}$ (e Å <sup>–3</sup> )	–0.47
<i>R</i>	0.0583
<i>wR</i>	0.0619
Weighting scheme	$1/\sigma^2(F)$
$\sigma^2(F)$	$\sigma_a^2(F) + 0.014F^2$

with the copper(II) trifluoromethanesulfonate compound with 3,5-bis(2-pyridinyl)-1,2,4-triazole (Prins, Birker, Haasnoot, Verschoor & Reedijk, 1985), where the trifluoromethanesulfonate has been found to be involved in coordination, it was supposed that in these aamt compounds the anions were in coordinative positions. In order to elucidate further the nature of the anions present in the aamt compounds, the X-ray crystal structure determination of the sulfate compound was undertaken. The anion was selected in view of the versatility of its coordination modes, including the possibility that it acts as a free anion (Nakamoto, 1978). In this respect it is notable that in a copper(II) sulfate compound with the related ligand 3,5-bis(2-pyridinyl)-4-amino-1,2,4-triazole, the sulfate was found to act as a bidentate dianion linking two copper ions as well as a monodentate dianion (van Koningsbruggen, 1991).

**Experimental.** The aamt ligand has been prepared according to the literature (Krysin, Levchenko, Andronova & Nabenina, 1975; Koomen-van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989). Crystals suitable for X-ray analysis were synthesized according to the following method: 10 mmol of aamt dissolved in 10 ml of hot water was added to 10 mmol of Cu(SO<sub>4</sub>)<sub>2</sub>·5H<sub>2</sub>O in 20 ml of hot water. Dark blue crystals were obtained on slow evaporation at room temperature. Experimental data are shown in Table 1. Enraf-Nonius CAD-4 four-circle diffractometer with graphite-monochromatized Mo *K* $\alpha$  radiation. Cell constants were determined from setting angles of 24 reflections ( $2 \leq \theta \leq 30^\circ$ ). The intensities were corrected for Lorentz and polarization effects. Absorption correction was applied by the use of Monte-Carlo methods (de Graaff, 1973). Scattering factors and corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The copper ion was located from a Patterson map. The structure was solved by automatic Fourier techniques, using the computer program *AUTOFOUR* (Kinneging & de Graaff, 1984). Least-squares refinement using *F*'s of non-H-atom positional and anisotropic thermal parameters. Positions of H atoms were calculated, except for those on the amino N atoms, with C–H = 0.90 Å for H atoms belonging to the methylene groups. H atoms belonging to the water molecules could not be located owing to positional disorder. Isotropic temperature factors for the methylene H atoms were 5 Å<sup>2</sup>, for the amino H atoms 3 Å<sup>2</sup>. Leiden University Computer (IBM 3083); programs written or modified by Mrs E. W. Rutten-Keulemans and Mr R.A.G. de Graaff.

**Discussion.** Positional parameters and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 2.\* Bond distances and angles are given in Table 3. An *ORTEP* drawing (Johnson, 1965) of [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub><sup>4+</sup>, with the atomic labelling used in Tables 2, 3 and 4, is given in Fig. 1. The structure consists of dinuclear [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub><sup>4+</sup> cations, non-coordinating sulfate anions and non-coordinating water molecules. The [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub><sup>4+</sup> cation is very similar to the previously reported related compound [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)Br(H<sub>2</sub>O)]<sub>2</sub>Br<sub>2</sub>·2H<sub>2</sub>O·CH<sub>3</sub>OH (Koomen-van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989). The aamt ligand acts as a doubly bidentate ligand, linking the metal ions which are related by the crystallographic centre of symmetry at the middle of the copper–copper vector. The Cu–Cu' distance within the dinuclear cation is 4.088 (3) Å, which is comparable to the 4.0694 (7) Å found for the bromide compound. The equatorial coordination around each copper(II) ion can be described as distorted square planar. The metal ion is displaced by 0.07 (9) Å out of the least-squares plane through the equatorial N donor atoms in a direction towards O1. The innermost angles N1–Cu–N2, which are 91.9 (4) and 92.1 (1)° for the copper sulfate and the copper bromide compound, respectively, are comparable. The outermost angles N31–Cu–N51 differ slightly and are 104.1 (4)° for the sulfate compound and 105.7 (1)° for the bromide derivative. The bite angles in the copper sulfate compound are slightly larger [N1–Cu–N51 = 81.5 (4) and N2–Cu–N31 = 82.3 (4)°] than in the bromide compound

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55263 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L10125]

Table 2. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters [ $\times 10^2$  for Cu and S(1);  $\times 10$  for the other atoms]

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>iso</sub> (Å <sup>2</sup> )
Cu	-911 (1)	953 (2)	24 (2)	265 (5)
N1	-698 (5)	-743 (8)	31 (8)	21 (3)
N2	-89 (6)	-1368 (9)	-6 (9)	21 (3)
C3	-242 (7)	-2484 (12)	0 (11)	19 (4)
N4	-945 (6)	-2586 (9)	69 (10)	23 (3)
N41	-1368 (5)	-3598 (10)	98 (9)	33 (4)
C5	-1197 (7)	-1457 (12)	52 (11)	20 (4)
C31	332 (7)	-3371 (12)	-65 (11)	29 (4)
N31	1034 (5)	-2761 (8)	18 (9)	23 (3)
C51	-1938 (6)	-976 (12)	74 (11)	36 (5)
N51	-1925 (5)	323 (10)	-18 (9)	26 (3)
O1	-728 (4)	1008 (9)	2045 (7)	35 (3)
O2	-1250 (5)	968 (9)	-2036 (8)	45 (3)
O3	-2638 (5)	1503 (8)	1514 (8)	38 (3)
O4	-4280 (6)	1931 (11)	2567 (9)	73 (5)
S(1)	-3437 (2)	954 (4)	7571 (3)	306 (11)
O(1)	-3890 (5)	235 (9)	6740 (8)	46 (3)
O(2)	-2702 (5)	600 (10)	7655 (11)	78 (5)
O(3)	-3617 (7)	736 (10)	8558 (8)	74 (5)
O(4)	-3520 (6)	2173 (9)	7291 (9)	66 (5)

 Table 3. Bond lengths (Å) and angles (°) within the [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub><sup>4+</sup> ion; estimated standard deviations include allowance for cell-parameter errors

Primed atoms are generated by the symmetry operation  $-x, -y, -z$ .

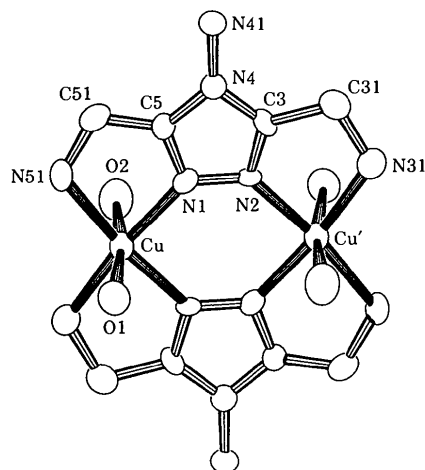
Cu—Cu'	4.088 (3)	N4—C5	1.36 (1)
Cu—N1	1.954 (9)	C5—N1	1.25 (1)
Cu—N51	2.04 (1)	C3—C31	1.50 (2)
Cu—O1	2.60 (1)	C31—N31	1.48 (1)
Cu—O2	2.63 (1)	C5—C51	1.52 (1)
Cu—N2'	1.96 (1)	C51—N51	1.47 (2)
Cu—N31'	2.052 (9)	S(1)—O(1)	1.47 (1)
N1—N2'	1.37 (1)	S(1)—O(2)	1.43 (1)
N2—C3	1.29 (1)	S(1)—O(3)	1.44 (1)
C3—N4	1.37 (1)	S(1)—O(4)	1.42 (1)
N4—N41	1.40 (1)		
N1—Cu—N51	81.5 (4)	Cu'—N2—N1	135.1 (8)
N1—Cu—O1	92.3 (4)	Cu'—N2—C3	116.8 (9)
N1—Cu—O2	90.7 (4)	Cu'—N31—C31	111.4 (7)
N1—Cu—N2'	91.9 (4)	Cu—N51—C51	112.2 (7)
N1—Cu—N31'	174.2 (4)	N2—N1—C5	109 (1)
N51—Cu—O1	86.3 (4)	N1—N2—C3	108 (1)
N51—Cu—O2	88.4 (4)	N2—C3—N4	108 (1)
N51—Cu—N2'	173.1 (4)	N2—C3—C31	119 (1)
N51—Cu—N31'	104.1 (4)	N4—C3—C31	133 (1)
O1—Cu—O2	173.4 (3)	C3—N4—C5	105 (1)
O1—Cu—N2'	96.2 (4)	N41—N4—C3	124 (1)
O1—Cu—N31'	89.5 (4)	N41—N4—C3	130 (1)
O2—Cu—N2'	89.5 (4)	N1—C5—N4	110 (1)
O2—Cu—N31'	88.1 (4)	N1—C5—C51	119 (1)
N2'—Cu—N31'	82.3 (4)	N4—C5—C51	131 (1)
Cu—N1—N2	132.9 (8)	N31—C31—C3	110 (1)
Cu—N1—C5	118.3 (9)	N51—C51—C5	109 (1)

[N1—Cu—N51 = 80.8 (1) and N2—Cu—N31 = 81.0 (1)°]. This is also reflected in the geometry of the aamt ligand which shows larger C3—C31—N31 and C5—C51—N51 angles [110 (1) and 109 (1)° in the sulfate compound compared to 107.1 (3) and 107.1 (3)° in the bromide compound] as well as longer C3—C31 and C5—C51 distances [1.50 (2) and

Table 4. Distances (Å) and angles (°) for the hydrogen bonding in the title compound; estimated standard deviations include allowance for cell-parameter errors

Primed atoms are generated by the symmetry operation  $-0.5-x, -0.5-y, 1-z$ ; doubly primed atoms are generated by  $-0.5-x, -0.5+y, 0.5-z$

<i>A</i> —H... <i>B</i>	<i>A</i> ... <i>B</i>	<i>A</i> —H	H... <i>B</i>	<i>A</i> —H... <i>B</i>
N41—H(N411)...O(3)'	2.99 (2)	0.7312	2.39 (2)	140.3 (4)
N41—H(N412)...O(1)''	2.89 (2)	1.0798	2.12 (2)	126.3 (4)


 Fig. 1. Projection of [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub><sup>4+</sup> with the atomic labelling system. H atoms have been omitted for clarity.

1.52 (1) Å in the sulfate compound, 1.487 (4) and 1.492 (4) Å in the bromide compound]. In the present compound the axial ligands are coordinated at somewhat shorter distances than the water molecule [Cu(1)—O(1) = 2.647 (3) Å] and the Br atom [Cu(1)—Br(1) = 2.8971 (6) Å] in the bromide compound.

As has been observed in related compounds (Prins, Birker, Haasnoot, Verschoor & Reedijk, 1985; Koomen—van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989; Keij, de Graaff, Haasnoot & Reedijk, 1984) the axial ligands are bent away from the centre of symmetry towards the *exo* side of the molecule. The O1—Cu—O2 angle of 173.4 (3)° is comparable to the O1—Cu—Br angle of 174.22 (7)° found in the bromide compound. The sulfate anion is involved in extensive hydrogen bonding with the amino group at the N4 of the aamt ligand, as is summarized in Table 4.

Although the H atoms at the water molecules could not be localized it is clear from the short O...O contacts that there is an important hydrogen-bonding network between the sulfate and both the coordinated and non-coordinated water molecules, as well as between both kinds of water molecules. To

obtain information about the magnitude of the exchange coupling between the copper(II) ions the magnetic susceptibilities have been recorded in the temperature range 300–6.7 K. A least-squares fit on the Bleaney–Bowers equation (Bleaney & Bowers, 1952) based on the exchange Hamiltonian  $\hat{H} = -2J[\hat{S}_1 \cdot \hat{S}_2]$  in which  $J$  = exchange coupling constant and  $\hat{S}$  = spin operator, yielded  $g = 2.02$ ,  $J = -96.5 \text{ cm}^{-1}$  and  $p$  (percentage paramagnetic impurities) = 1.95. These parameters are in the same range as has been found for [Cu(C<sub>4</sub>H<sub>10</sub>N<sub>6</sub>)Br(H<sub>2</sub>O)]<sub>2</sub>·Br<sub>2</sub>·2H<sub>2</sub>O·CH<sub>3</sub>OH (Koomen–van Oudenniel, de Graaff, Haasnoot, Prins & Reedijk, 1989). Apparently, the slightly longer Cu–Cu' distance as well as the differences in the axial coordination sphere do not have much influence on the magnitude of the antiferromagnetic interaction between nearest-neighbour copper(II) ions.

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## Structure of the Hydrogen-Bond-Rich Dinickel(II) Complex of *N,N',N'',N'''*-Tetrakis(2-aminoethyl)-1,1,2,2-ethanetetraamide

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**Abstract.**  $\mu$ -[*N,N',N'',N'''*-Tetrakis(2-aminoethyl)-1,1,2,2-ethanetetraamido-*N,N':N'',N'''*]-dinickel(II) hexahydrate, [Ni<sub>2</sub>(C<sub>14</sub>H<sub>26</sub>N<sub>8</sub>O<sub>4</sub>)]<sub>2</sub>·6H<sub>2</sub>O,  $M_r = 595.93$ , monoclinic,  $P2_1/c$ ,  $a = 7.363$  (3),  $b = 8.886$  (2),  $c = 18.321$  (4) Å,  $\beta = 96.18$  (2)°,  $V = 1191.6$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.65$ ,  $D_x = 1.66 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71069$  Å, graphite monochromator,  $\mu = 16.5 \text{ cm}^{-1}$ ,  $F(000) = 628$ ,  $T = 296 \text{ K}$ ,  $R = 0.037$  and  $wR = 0.037$  for 2362 unique reflections with  $I > 3\sigma(I)$ . The structure is composed of two symmetry-related chelating groups linked through a C–C bond. Each Ni<sup>II</sup> is coordinated by four N atoms (two amino and two amido) and exhibits square-planar geometry. All atoms capable of forming hydrogen bonds do so, resulting in 20 hydrogen bonds in the complex.

**Introduction.** *N,N',N'',N'''*-Tetrakis(2-aminoethyl)-1,1,2,2-ethanetetraamide is a tetradentate double-chelating ligand in which each cavity consists of four N atoms of two aminoethyl-substituted amide groups, and is similar in structure to the double-ring dioxotetraamine ligand (Buttafava, Fabbrizzi, Perotti, Poggi & Seghi, 1984). It is difficult to obtain single crystals of complexes with either of the above ligands owing to the effect of the amide groups. Until now, the crystal structure of the single-ring platinum(II) complex of dioxotetraamine has been the only one reported with this ligand (Kimura, Korenari, Shonoya & Shiro, 1988) and a complex of the double-chelating ligand has been studied only in solution (Hay, Bembi & McLaren, 1981). Some interest has been shown in the physiological functions of dioxotetraamine complexes (Kimura,

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