*Note from the authors*: After this manuscript was ready for publication we realized that the crude structure of this compound was actually solved by Sundaralingam & Carrabine (1971), from photographic data. The H-atom locations in this referenced work were unknown. Therefore we think it is useful to publish this new and more precisely determined structure in which the H-atom positions are determined.

The authors wish to thank Dr Bagieu in our Laboratory for her help in tracing the bibliographic data related to the interaction of metal ions with nucleic acids and their constituents.

### References

EICHHORN, G. L. (1962). *Nature (London)*, **194**, 474–475.

EICHHORN, G. L. & SHIN, Y. A. (1968). *J. Am. Chem. Soc.* **90**, 7323–7328.

FRENZ, B. A. (1983). *Enraf–Nonius, Structure Determination Package; SDP Users Guide.* Version 1.1. Enraf–Nonius, Delft, The Netherlands.

FUJINAMI, F., OGAWA, K., ARAKAWA, Y., SHIROTAKE, S., FUJII, S. & TOMIKA, K. I. (1979). *Acta Cryst.* **B35**, 968–970.

HAMILTON, W. C. & IBERS, J. A. (1968). *The Hydrogen Bond in Solids.* New York: Benjamin.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DELCLERCQ, J. -P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

OGAWA, K., KUMIHASHI, M., TOMITA, K.-I. & SHIROTAKE, S. (1980). *Acta Cryst.* **B36**, 1793–1797.

OGAWA, K., NISHITANI, K., FUJIWARA, T., SHIROTAKE, S. & TOMITA, K.-I. (1979). *Acta Cryst.* **B35**, 965–967.

SUNDARALINGAM, M. & CARRABINE J. A. (1971). *J. Mol. Biol.* **61**, 287–309.

WEBER, H. P., CRAVEN, B. M. & MCMULLAN, R. K. (1980). *Acta Cryst.* **B36**, 645–649.

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*Acta Cryst.* (1990). C**46**, 1223–1226

# Structure of the Silver Salt of *N*-(*p*-Aminophenylsulfonyl)acetamide (Silver Sulfacetamide)

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(*Received* 13 *April* 1989; *accepted* 20 *September* 1989)

**Abstract.** $Ag^+.C_8H_9N_2O_3S^-$, $M_r = 321{\cdot}1$, monoclinic, $P2_1/n$, $a = 5{\cdot}849\,(2)$, $b = 7{\cdot}720\,(5)$, $c = 22{\cdot}573\,(5)$ Å, $\beta = 83{\cdot}16\,(1)°$, $V = 1012\,(1)$ Å$^3$, $Z = 4$, $D_x = 2{\cdot}107$ g cm$^{-1}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 21{\cdot}57$ cm$^{-1}$, $T = 295$ K, $F(000) = 632$, final $R = 0{\cdot}0413$ for 1522 observed reflections. Each Ag atom is coordinated to two O atoms and one N atom of two sulfacetamide molecules with Ag—O distances 2·408 (4) and 2·336 (5) Å and Ag—N distance 2·218 (5) Å. Thus a silver sulfacetamide polymer is formed along **a** showing Ag–Ag interaction [3·102 (1) Å] about alternate centres of inversion. The molecules are linked by N—H···O-type hydrogen bonds.

**Introduction.** Sulfacetamide, a highly soluble sulfonamide which does not cause crystalluria, and its sodium salt are extensively used in opthalmic infections, giving solutions which are non-irritating to the delicate tissues of the eye. Crystal structures of sulfacetamide (Basak, Mazumdar & Chaudhuri, 1982) and sulfacetamide sodium monohydrate (Ghosh, Basak & Mazumdar, 1987) have already been reported.

Sulfonamides are selective inhibitors of animal and bacterial carbonic anhydrase and this is partially due to the ionization of this molecule by deprotonation at the amido nitrogen, upon coordination to the metalloenzyme. The present structural analysis was undertaken in order to study the complex as a model sulfonamide–metal ion complex and also to see the structural changes in the ligand as a result of coordination to the metal.

**Experimental.** Reddish square-shaped plate-like crystals obtained by slow evaporation from a 10% ammonia solution at room temperature in the dark. Crystal size 0·15 × 0·25 × 0·40 mm, $\omega/2\theta$ scan, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice parameters from least-squares fit with 25 reflections up to $2\theta = 50°$, three standard reflections ($\bar{2}\bar{2}4$, $22\bar{2}$, $2,\bar{1},10$) moni-

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* $U_{\mathrm{eq}}$ (Å$^2$) *with e.s.d.'s in parentheses*

|  | *x* | *y* | *z* | $U_{\mathrm{eq}}$ |
|---|---|---|---|---|
| Ag | 0·1390 (1) | 0·0575 (1) | 0·0510 (0) | 0·052 (0) |
| S | −0·3607 (2) | −0·0916 (2) | 0·0952 (1) | 0·024 (0) |
| N(1) | −0·2416 (8) | 0·0811 (7) | 0·0633 (2) | 0·028 (1) |
| N(2) | −0·5887 (14) | −0·0064 (10) | 0·3546 (3) | 0·048 (2) |
| O(1) | −0·5635 (7) | −0·1444 (6) | 0·0692 (2) | 0·032 (1) |
| O(2) | −0·1781 (8) | −0·2188 (6) | 0·0898 (2) | 0·038 (1) |
| O(3) | −0·5770 (7) | 0·2411 (6) | 0·0800 (2) | 0·038 (2) |
| C(1) | −0·4410 (10) | −0·0484 (8) | 0·1713 (3) | 0·025 (2) |
| C(2) | −0·6390 (11) | −0·1223 (9) | 0·2001 (3) | 0·033 (2) |
| C(3) | −0·6878 (12) | −0·1083 (10) | 0·2610 (3) | 0·037 (3) |
| C(4) | −0·5418 (11) | −0·0183 (8) | 0·2947 (3) | 0·030 (2) |
| C(5) | −0·3445 (12) | 0·0577 (10) | 0·2642 (3) | 0·036 (2) |
| C(6) | −0·2928 (11) | 0·0416 (9) | 0·2039 (3) | 0·033 (2) |
| C(7) | −0·3694 (10) | 0·2284 (8) | 0·0598 (3) | 0·029 (2) |
| C(8) | −0·2410 (12) | 0·3784 (9) | 0·0304 (3) | 0·040 (2) |

tored, no variation in intensity observed, 1773 unique reflections recorded in the range $-6 \le h \le 6$, $-1 \le k \le 9$, $-1 \le l \le 26$, $0 \le \theta \le 22°$. Data were corrected for Lorentz and polarization effects but not for absorption. Structure solution *via* Patterson function, $\Delta F$ synthesis and full-matrix least-squares refinement on *F* using *SHELX*76 (Sheldrick, 1976), with anisotropic temperature factors for all non-H atoms and isotropic temperature factors for H atoms, refinement with 1522 observed reflections [$I \ge 3\sigma(I)$] and 160 refined parameters, unit weights. Methyl hydrogens were included in calculated positions but not refined. $R = 0{\cdot}0413$, $wR = 0{\cdot}0413$, $(\mathrm{shift/e.s.d.})_{\max} = 0{\cdot}6$, $(\mathrm{shift/e.s.d.})_{\mathrm{av.}} = 0{\cdot}05$, maximum peak in the final $\Delta F$ map 0·6 e Å$^{-3}$; scattering factors for non-H atoms taken from Cromer & Waber (1965) and those for H atoms from Stewart, Davidson & Simpson (1965).

**Discussion.** The molecular structure with atomic numbering scheme is shown in Fig. 1. Table 1 lists the atomic parameters.* The bond distances, angles and selected torsion angles for the sulfacetamide molecule are listed in Table 2. Each Ag atom forms normal coordination bonds with N(1) of one molecule and O(1) and O(3) of a symmetry-related molecule ($x + 1$, $y$, $z$), thus forming a long polymeric chain extended along **a**. The Ag—N(1) distance, 2·218 (5) Å, is comparable to the Ag—N distances observed in silver sulfadiazine [2·24 (1), 2·45 (1) Å] (Cook & Turner, 1975) and the $\alpha$-polymorph of silver $N^1$-(3-pyridyl)sulfanilamide [2·25 (1), 2·30 (1) Å] (Cook & Turner, 1976). There is also an

Table 2. *Intermolecular contacts, distances* (Å) *and angles* (°) *and intramolecular bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

| | | | |
|---|---|---|---|
| Ag—*X* distances | | | |
| Ag$^{\mathrm{i}}$—N(1)$^{\mathrm{i}}$ | 2·218 (5) | Ag$^{\mathrm{i}}$—O(1)$^{\mathrm{ii}}$ | 2·408 (4) |
| Ag$^{\mathrm{i}}$—O(2)$^{\mathrm{i}}$ | 2·893 (5) | Ag$^{\mathrm{i}}$—O(3)$^{\mathrm{ii}}$ | 2·336 (5) |
| Ag$^{\mathrm{i}}$—Ag$^{\mathrm{iii}}$ | 3·102 (1) | | |
| *X*—Ag—*X* angles | | | |
| N(1)$^{\mathrm{i}}$—Ag$^{\mathrm{i}}$—O(2)$^{\mathrm{i}}$ | 54·9 (2) | N(1)$^{\mathrm{i}}$—Ag$^{\mathrm{i}}$—O(1)$^{\mathrm{ii}}$ | 133·4 (1) |
| N(1)$^{\mathrm{i}}$—Ag$^{\mathrm{i}}$—O(3)$^{\mathrm{i}}$ | 130·7 (2) | O(1)$^{\mathrm{ii}}$—Ag$^{\mathrm{i}}$—O(2)$^{\mathrm{i}}$ | 137·9 (1) |
| O(1)$^{\mathrm{ii}}$—Ag$^{\mathrm{i}}$—O(3)$^{\mathrm{ii}}$ | 68·9 (1) | O(2)$^{\mathrm{i}}$—Ag$^{\mathrm{i}}$—O(3)$^{\mathrm{ii}}$ | 144·5 (1) |
| Ag$^{\mathrm{iii}}$—Ag$^{\mathrm{i}}$—O(3)$^{\mathrm{ii}}$ | 146·6 (0) | Ag$^{\mathrm{iii}}$—Ag$^{\mathrm{i}}$—O(1)$^{\mathrm{ii}}$ | 113·2 (0) |
| | | Ag$^{\mathrm{iii}}$—Ag$^{\mathrm{i}}$—N(1)$^{\mathrm{i}}$ | 60·6 (1) |
| Hydrogen-bond distances | | | |
| N(2)$^{\mathrm{i}}$···O(2)$^{\mathrm{iv}}$ | 3·067 (9) | N(2)$^{\mathrm{i}}$—H(41)$^{\mathrm{i}}$ | 0·74 (10) |
| H(41)$^{\mathrm{i}}$···O(2)$^{\mathrm{iv}}$ | 2·37 (10) | | |
| N(2)$^{\mathrm{i}}$···O(3)$^{\mathrm{v}}$ | 3·019 (8) | N(2)$^{\mathrm{i}}$—H(42)$^{\mathrm{i}}$ | 0·85 (8) |
| H(42)$^{\mathrm{I}}$···O(3)$^{\mathrm{v}}$ | 2·22 (9) | | |
| Hydrogen-bond angles | | | |
| N(2)$^{\mathrm{i}}$—H(41)$^{\mathrm{i}}$···O(2)$^{\mathrm{iv}}$ | 158 (10) | N(2)$^{\mathrm{i}}$—H(42)$^{\mathrm{i}}$···O(3)$^{\mathrm{v}}$ | 158 (9) |
| Other short contacts < 3·8 Å | | | |
| Ag$^{\mathrm{i}}$—S$^{\mathrm{iii}}$ | 3·409 (2) | Ag$^{\mathrm{i}}$—S$^{\mathrm{i}}$ | 3·189 (2) |
| Ag$^{\mathrm{i}}$—N(1)$^{\mathrm{iii}}$ | 2·792 (5) | Ag$^{\mathrm{i}}$—S$^{\mathrm{ii}}$ | 3·403 (2) |
| Ag$^{\mathrm{i}}$—C(7)$^{\mathrm{iii}}$ | 3·484 (6) | Ag$^{\mathrm{i}}$—O(1)$^{\mathrm{iii}}$ | 3·517 (4) |
| Ag$^{\mathrm{i}}$—C(7)$^{\mathrm{i}}$ | 2·238 (6) | Ag$^{\mathrm{i}}$—O(2)$^{\mathrm{iii}}$ | 3·396 (5) |
| Ag$^{\mathrm{i}}$—C(8)$^{\mathrm{i}}$ | 3·397 (7) | Ag$^{\mathrm{i}}$—C(7)$^{\mathrm{ii}}$ | 3·191 (6) |
| S—O(1) | 1·443 (5) | S—O(2) | 1·445 (5) |
| S—N(1) | 1·631 (5) | S—C(1) | 1·758 (7) |
| N(1)—C(7) | 1·368 (8) | N(2)—C(4) | 1·350 (9) |
| C(1)—C(2) | 1·382 (9) | C(1)—C(6) | 1·388 (9) |
| C(2)—C(3) | 1·375 (9) | C(3)—C(4) | 1·395 (10) |
| C(4)—C(5) | 1·400 (9) | C(5)—C(6) | 1·364 (9) |
| C(7)—C(8) | 1·492 (9) | C(7)—O(3) | 1·249 (7) |
| O(1)—S—C(1) | 108·3 (3) | O(2)—S—C(1) | 108·7 (3) |
| O(1)—S—O(2) | 114·1 (3) | N(1)—S—C(1) | 109·1 (3) |
| N(1)—S—O(1) | 112·3 (3) | N(1)—S—O(2) | 104·2 (3) |
| S—N(1)—C(7) | 119·8 (4) | S—C(1)—C(2) | 119·5 (5) |
| S—C(1)—C(6) | 120·1 (5) | C(2)—C(1)—C(6) | 119·9 (6) |
| C(1)—C(2)—C(3) | 120·0 (6) | C(2)—C(3)—C(4) | 121·1 (6) |
| C(3)—C(4)—C(5) | 117·6 (6) | C(4)—C(5)—C(6) | 121·6 (7) |
| C(1)—C(6)—C(5) | 119·7 (6) | N(2)—C(4)—C(3) | 121·0 (7) |
| N(2)—C(4)—C(5) | 121·4 (7) | N(1)—C(7)—C(8) | 114·9 (5) |
| N(1)—C(7)—O(3) | 123·9 (6) | O(3)—C(7)—C(8) | 121·1 (6) |
| O(1)—S—C(1)—C(2) | 22·5 (6) | O(2)—S—C(1)—C(2) | −102·0 (6) |
| O(1)—S—C(1)—C(6) | −165·3 (5) | O(2)—S—C(1)—C(6) | 70·2 (6) |
| N(1)—S—C(1)—C(2) | 145·0 (5) | N(1)—S—C(1)—C(6) | −42·8 (6) |
| O(1)—S—N(1)—C(7) | 55·2 (5) | O(2)—S—N(1)—C(7) | 179·2 (5) |
| C(1)—S—N(1)—C(7) | −64·9 (5) | S—C(1)—C(2)—C(3) | 171·5 (5) |
| S—N(1)—C(7)—O(3) | 0·2 (9) | C(2)—C(3)—C(4)—N(2) | −179·2 (7) |
| S—C(1)—C(6)—C(5) | −172·6 (5) | S—N(1)—C(7)—C(8) | 179·2 (4) |
| N(2)—C(4)—C(5)—C(6) | 178·0 (7) | | |

Roman numeral superscripts denote the following equivalent positions: (i) $x, y, z$; (ii) $x+1, y, z$; (iii) $-x, -y, -z$; (iv) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x-\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

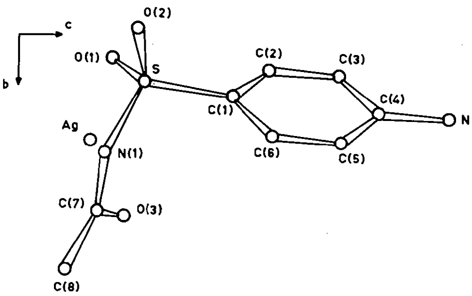

Fig. 1. Diagram of the molecule with numbering scheme, viewed down the *a* axis.

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* Lists of structure factors, anisotropic thermal parameters, and H-atom parameters, bond distances and angles involving H atoms and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52676 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Ag—O(2) distance of 2·893 (5) Å, but this is probably outside the sphere of coordination. Each sulfacetamide molecule chelates one Ag atom through O(1) and O(3) forming a six-membered ring. The interesting feature is that Ag atoms of each polymeric unit interact with Ag atoms of other polymeric units showing four coordination with N(1), O(1), O(3) and Ag at alternate centres of inversion. The Ag—Ag distance [3·102 (1) Å] in the present structure is similar to that in silver sulfadiazine [2·916 Å], silver metal [2·889 Å] and phenylethynyl(trimethylphosphine)silver(I) [3·033 Å] (Corfield & Shearer, 1966). As in silver sulfadiazine the amino nitrogen N(2) is not coordinated to the Ag atom.

*The sulfacetamide moiety.* The geometry of the ligand molecule is not modified by the Ag atom. The S atom has a distorted-tetrahedral configuration. Both the S—O distances are almost equal though one of the sulfonyl oxygens, O(1), does not participate in hydrogen bonding. The angle O(1)—S—O(2) [114·1 (2)°] is comparable to the value observed in sodium sulfacetamide [115·3 (1)°] but is less than those found in sulfacetamide [118·3 (1)°], 1/1 sulfacetamide–caffeine [119·2 (1·8)°] (Leger, Alberola & Carpy, 1977), sulfamerazine [118·8 (3)°] (Rabindra Acharya, Kuchela & Kartha, 1982), 2-[4-(acetylaminosulfonyl)phenylcarbamoyl]benzoic acid [118·1 (7)°] (Singh, Patel & Haridas, 1984) and 4-phthalimido-*N*-(1,3-thiazol-2-yl)benzenesulfonamide [118·8 (2)°] (Ghosh, Basak, Roy, Mazumdar & Sheldrick, 1987).

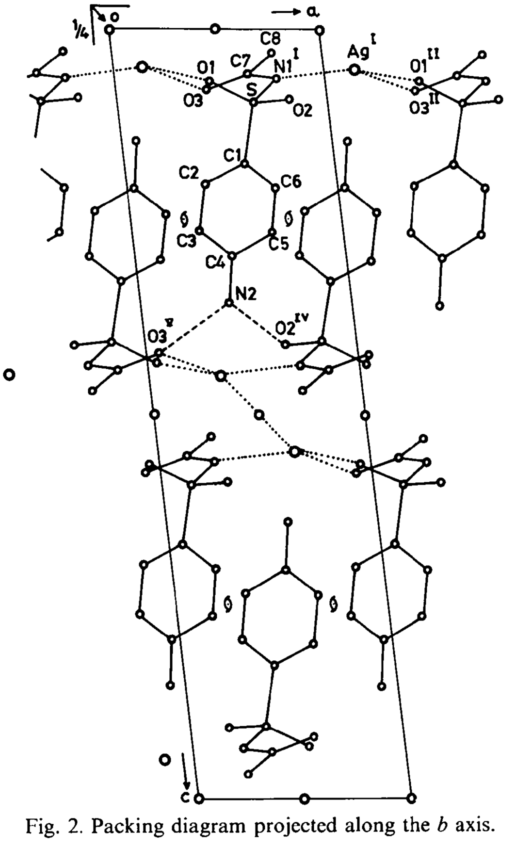

Fig. 2. Packing diagram projected along the *b* axis.

The shortening of the S—C(1) [1·758 (7) Å] and C(4)—N(2) [1·350 (9) Å] distances from the single-bond distances indicates the partial quinoidal structure analogous to nitroanilines (O'Connell & Maslen, 1977). But the bond character of the S—O(1), S—O(2) and S—N(1) bonds is similar to that in sulfonamide structures. The length of the N(1)—C(7) bond [1·368 (8) Å] agrees well with that [1·363 (4) Å] found in sulfacetamide.

The phenyl ring is almost planar but the ring-attached atoms N(2) and S deviate by −0·029 (8) and −0·217 (2) Å from this plane. The fragment consisting of S, N(1), C(7), C(8) and O(3) atoms is also planar but with significant deviations of O(1) [1·093 (4) Å] and O(2) [0·018 (5) Å] from the plane. The dihedral angle between these two planes is 87·5 (2)°.

*Conformation.* In most of the substituted sulfonamides the torsion angles $|\varepsilon_1|$ [C($X$)—C(1)—S—N(1), where $X = 2,6$] and $|\varepsilon_2|$ [C(1)—S—N(1)—C(7)] lie in the clustering range 70–120° and 60–90°, respectively (Kálmán, Czugler & Argay, 1981). No deviation from this rule is observed in sulfacetamide, 1/1 sulfacetamide–caffeine or sodium sulfacetamide monohydrate. In the present structure the $|\varepsilon_1|$ values [145·0 (5) and −42·8 (6)°] do not agree with the values observed in $\alpha$- and $\beta$-polymorphs of silver $N^1$-(3-pyridyl)sulfanilamide. However, in silver sulfadiazine the $|\varepsilon_1|$ [C($X$)—C(1)—S—N(1), where $X = 2,6$] and $|\varepsilon_2|$ values are −65·4 (1·4), 106·9 (1·3) and −53·4 (1·1)°, respectively.

*Hydrogen bonding and molecular packing.* The amino nitrogen N(2) participates in two hydrogen bonds of the type N—H···O involving sulfonyl oxygen O(2) and keto oxygen O(3) of two separate molecules. The hydrogen-bonding geometry is given in Table 2. Fig. 2 clearly shows alternate hydrophobic and hydrophilic regions extending along **c**.

We thank Mr Amit Kumar Das for very useful discussions.

## References

Basak, A. K., Mazumdar, S. K. & Chaudhuri, S. (1982). *Cryst. Struct. Commun.* **11**, 1609–1616.

Cook, D. S. & Turner, M. F. (1975). *J. Chem. Soc. Perkin Trans.* 2, pp. 1021–1025.

Cook, D. S. & Turner, M. F. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 1379–1383.

Corfield, P. W. R. & Shearer, H. M. (1966). *Acta Cryst.* **20**, 502–508.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104–109.

Ghosh, M., Basak, A. K. & Mazumdar, S. K. (1987). *J. Crystallogr. Spectrosc. Res.* **17**, 739–749.

GHOSH, M., BASAK, A. K., ROY, P. N., MAZUMDAR, S. K. & SHELDRICK, B. (1987). *Acta Cryst.* C**43**, 732–734.

KÁLMÁN, A., CZUGLER, M. & ARGAY, GY. (1981). *Acta Cryst.* B**37**, 868–877.

LEGER, J. M., ALBEROLA, S. & CARPY, A. (1977). *Acta Cryst.* B**33**, 1455–1459.

O'CONNELL, A. M. & MASLEN, E. N. (1977). *Acta Cryst.* **22**, 134–145.

RABINDRA ACHARYA, K., KUCHELA, K. N. & KARTHA, G. (1982). *J. Crystallogr. Spectrosc. Res.* **12**, 369–376.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SINGH, T. P., PATEL, U. & HARIDAS, M. (1984). *Acta Cryst.* C**40**, 2088–2091.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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*Acta Cryst.* (1990). C**46**, 1226–1228

# Structure of Dinuclear Bis(di-2-pyridylmethanone oximato)copper(II) Dihydrate

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(*Received* 6 *February* 1989; *accepted* 13 *September* 1989)

**Abstract.** Bis-$\mu$-(di-2-pyridyl ketone oximato-*O*,*N*)-bis(di-2-pyridyl ketone oximato)dicopper(II) dihydrate, $[Cu(C_{11}H_8N_3O)_2]_2.2H_2O$, $M_r = 956{\cdot}0$, monoclinic, $C2/c$, $a = 23{\cdot}619$ (4), $b = 11{\cdot}280$ (2), $c = 19{\cdot}155$ (3) Å, $\beta = 125{\cdot}24$ (2)°, $V = 4168$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}523$ (2) g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu = 10{\cdot}85$ cm$^{-1}$, $F(000) = 1960$, $T = 295$ (1) K, final $R = 0{\cdot}038$ for 1171 observed reflections $[F_o^2 > 2\sigma(F_o^2)]$. This structure consists of dinuclear complexes with twofold internal symmetry. The copper(II) coordination geometry is slightly distorted trigonal bipyramidal with each copper coordinated by two pyridine N atoms [av. Cu—N = 2·031 (13) Å], two oxime N atoms [av. Cu—N = 1·964 (6) Å], and one oxime O atom [Cu—O = 2·133 (5) Å]. One pyridine nitrogen of each ligand as well as the oxime oxygen of one ligand on each Cu atom are not involved in copper coordination. The angles of the trigonal plane are 100·4 (2), 123·1 (2) and 136·4 (2)° while the angle between axial ligand atoms is 179·0 (3)°. The water of hydration bridges two molecules by hydrogen bonding to the free oxime oxygen in one molecule and to one of the free pyridine nitrogens of the second molecule.

**Introduction.** Although metal complexes of the ligand di-2-pyridylmethanone oxime have been known for some time and used for analytical purposes (*e.g.* Holland & Bozic, 1968; Holland, Bozic & Gerard, 1968), no crystal-structure studies have been reported. Because of the four possible coordinating atoms of the ligand (two pyridine nitrogens, an oxime nitrogen and an oxime oxygen), we decided to investigate the structure of the complex isolated by reaction with $CuCl_2$ in aqueous media. This complex is different to the Cu$^{II}$ complex from acetone media reported earlier (Blackmore, Sceney, O'Neill & Magee, 1974).

**Experimental.** The ligand was obtained by reaction of the commercial ketone with $NH_2OH.HCl$ in a fashion similar to that reported (Niemers & Hiltmann, 1976). Reaction of the ligand (1:2 $M$:$L$ ratio) with $CuCl_2$(aq.) in slightly basic solution produced green crystals on slow evaporation of the solvent.

The crystal chosen for the X-ray study had approximate dimensions 0·1 × 0·15 × 0·2 mm and was placed in random orientation on an Enraf–Nonius CAD-4 diffractometer. After optical centering, 25 reflections ($2\theta = 18$–35°) were located by automatic search of reciprocal space and recentered twice. The setting angles of these reflections were used to determine the cell dimensions. $\theta$–$2\theta$ scan. Three standard reflections were measured after every 7200 s of X-ray exposure (no significant variation), three orientation standards after every 200 reflections with recentering of all 25 if any significant angular error, three $\psi$ scans to obtain an empirical absorption curve (relative transmission = 88·9–99·6%), *hkl* range (0,0,−19 to 22,10,19 and −13,−6,−11 to 0,0,11), agreement of Friedel pairs, statistics, and successful refinement confirmed the choice of space group $C2/c$. 2700 reflections from $2\theta = 4$–40°, 1945 independent reflections (internal agreement on $F_o^2 = 0{\cdot}035$), 1171 reflections with $F_o^2 > 2\sigma(F_o^2)$ used in structure solution and refinement: structure solved by Patterson and difference Fourier methods, full-matrix least squares minimized $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/[\sigma^2_{\rm counting} + (0{\cdot}040F_o^2)^2]$, H atoms from difference Fourier map and calculation were not refined, all other atoms