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# The Crystal Structure of Bis(salicylaldoximato)copper(II)

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The crystal structure of bis(salicylaldoximato)copper(II), $[OC_6H_4CHNOH]_2Cu$, has been determined from three-dimensional X-ray diffraction data, $h0l$ through $h3l$. The cell has dimensions $a=13{\cdot}98$, $b=6{\cdot}08$, $c=8{\cdot}00$ Å, $\beta=97°\ 35'$, space group $P2_1/c$, and contains two molecules. The molecule has a *trans*-planar configuration and deviates slightly, but significantly, from complete planarity. The copper(II) ion has a distorted octahedral coordination with Cu–N = 1·94 Å and Cu–O = 1·92 and 2·66 Å.

## Introduction

Cox & Webster (1935) reported bis(salicylaldoximato)-copper(II), $Cu(C_7H_6NO_2)_2$, to crystallize with a monoclinic cell having dimensions

$$a=27{\cdot}61,\ b=6{\cdot}00,\ c=7{\cdot}86\ \text{Å};\ \beta=98°\ 17'$$

and containing four molecules. On the basis of the rather unusual systematic absence of $h0l$ for either $h$ or $l$ odd and of $0k0$ for $k$ odd, they concluded that the space group is $P2_1/a$ with an additional glide plane parallel to **b** with a translation of $c/2$. This led them to decide that the molecules must be centrosymmetric and therefore planar. As a part of a program of investigation of the coordination configurations of transition metal ions, we have determined the crystal structure of the compound from three-dimensional X-ray diffraction data.

## Experimental

Bis(salicylaldoximato)copper(II) was prepared by the addition of an aqueous solution of salicylaldoxime to aqueous copper(II) chloride. Crystals were grown by slow cooling of a solution in a mixture of Methyl Cellosolve and acetone. The crystals were monoclinic plates on {100}, bounded by {001} and {010}.

Cell dimensions were obtained from measurements of rotation and zero-level Weissenberg photographs taken about $b$ with Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å). The films were calibrated by superimposing the rotation pattern of NaCl ($a=5{\cdot}6387$ Å) on each film. The results are:

$$a=13{\cdot}98\pm0{\cdot}03,\ b=6{\cdot}08\pm0{\cdot}01,\ c=8{\cdot}00\pm0{\cdot}01\ \text{Å};\ \beta=97°\ 35'.$$

The cell contains two molecules. Density calculated is 1·65 $g.cm^{-3}$, observed 1·67 $g.cm^{-3}$. Systematic absences of $h0l$ for $l$ odd and of $0k0$ for $k$ odd indicate the space group to be $P2_1/c$. We could find no evidence for doubling of the length of $a$, as reported by Cox & Webster (1935).

The crystal selected for intensity measurements was a plate of dimensions 0·15 × 0·12 × 0·04 mm. Integrated equi-inclination Weissenberg photographs of ($h0l$) through ($h3l$) were obtained with Ni-filtered Cu $K\alpha$ radiation on a Nonius camera. Multiple films and a range of exposure times were used in order to get a satisfactory range of intensities. Camera integration was in one direction only, and each diffraction spot in the linear response range of each film was then scanned normal to the direction of camera integration with a Moll type densitometer feeding into a Leeds & Northrup amplifier and recorder having a logarithmic slide wire. The areas under the densitometer tracings were measured with a planimeter and used as relative intensities. The intensities from the several films for a given level were placed on the same scale by comparison of common spots. For the initial stages of the structure determination the several levels were roughly scaled together on the basis of their exposure times. During the later stages each level was scaled independently by comparison of observed and calculated structure factors.

A total of 905 reflections were examined, of which 356 were too faint to be measured. The range of relative intensities was 1 to 4000.

No correction was made for absorption. Lorentz and polarization factors were applied and observed structure factors calculated using the data reduction program written in this Laboratory for the IBM 650 computer.

The atomic scattering factors used were those of Thomas & Umeda (1957) for copper, Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen, nitrogen,, and carbon, and McWeeny (1951) for hydrogen. No correction was made for anomalous dispersion.

## Structure determination

The two copper atoms must lie in a twofold special position. They may be placed in the positions 0, 0, 0 and 0, $\frac{1}{2}$, $\frac{1}{2}$ without loss of generality.

A three-dimensional Patterson synthesis was first calculated, from which the positions of the oxygen and nitrogen atoms were readily obtained. A three-dimensional Fourier synthesis, using phases calculated from the positions of the copper, oxygen and nitrogen atoms, gave positions for the carbon atoms. A second Fourier synthesis gave small shifts in atomic positions and reduced $R$ to 0·20. $R$ is throughout defined by $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$, where the summation is over observed reflections only. Hydrogen atoms were introduced in calculated positions and assigned temperature factors of the atoms to which they are attached and refinement was continued by the least-squares method using the program of Busing & Levy (1959) on an IBM 709 computer. During the refinement, individual atom isotropic temperature factors were used.

A modified Hughes weighting scheme was used, with $w=0$ for unobserved reflections for which $F_c<F_{\min}$, $w=1$ for unobserved reflections for which $F_c>F_{\min}$, $w=1$ for observed reflections for which $F_o<40$ and $w=40/F_o$ for $F_o>40$. The function minimized was $\Sigma w(F_o-F_c)^2$.

Six cycles of least-squares refinement reduced $R$ to 0·096. In the final cycle, the parameter shifts were $\bar{\delta}_x=0{\cdot}00004$, $\bar{\delta}_y=0{\cdot}00015$, $\bar{\delta}_z=0{\cdot}00006$, $\bar{\delta}_B=0{\cdot}015$ and maximum $\delta_x=0{\cdot}0009$ (for N), $\delta_y=0{\cdot}00028$ (for C(1) and C(3)), $\delta_z=0{\cdot}00020$ (for C(6)), $\delta_B=0{\cdot}046$ (for C(6)).

The final parameters are listed in Table 1. The estimated standard deviations are: copper $\sigma_B=0{\cdot}04$; oxygen, $\sigma_x=0{\cdot}0006$, $\sigma_y=0{\cdot}0019$, $\sigma_z=0{\cdot}0011$, $\sigma_B=0{\cdot}2$; nitrogen, $\sigma_x=0{\cdot}0008$, $\sigma_y=0{\cdot}0023$, $\sigma_z=0{\cdot}0013$, $\sigma_B=0{\cdot}2$; carbon $\sigma_x=0{\cdot}0010$, $\sigma_y=0{\cdot}0029$, $\sigma_z=0{\cdot}0017$, $\sigma_B=0{\cdot}3$. The final values of observed and calculated structure factors are listed in Table 2.

Table 1. *Atomic positions and temperature factors*

| | $x/a$ | $y/b$ | $z/c$ | $B$ |
|---|---|---|---|---|
| Cu | 0·0000 | 0·5000 | 0·5000 | 3·88 |
| O(1) | 0·0060 | 0·9118 | 0·6759 | 3·36 |
| O(2) | 0·1187 | 0·3472 | 0·4884 | 4·89 |
| N | 0·0678 | 0·7440 | 0·6199 | 3·83 |
| C(1) | 0·2100 | 0·4139 | 0·5429 | 3·05 |
| C(2) | 0·2877 | 0·2842 | 0·5123 | 4·40 |
| C(3) | 0·3806 | 0·3518 | 0·5680 | 4·98 |
| C(4) | 0·4007 | 0·5451 | 0·6501 | 5·12 |
| C(5) | 0·3261 | 0·6758 | 0·6811 | 4·81 |
| C(6) | 0·2276 | 0·6164 | 0·6234 | 3·72 |
| C(7) | 0·1560 | 0·7732 | 0·6632 | 4·31 |
| H(1) | 0·2751 | 0·1287 | 0·4501 | 4·40 |
| H(2) | 0·4383 | 0·2498 | 0·5426 | 4·98 |
| H(3) | 0·4746 | 0·5936 | 0·6940 | 5·12 |
| H(4) | 0·3411 | 0·8276 | 0·7463 | 4·81 |
| H(5) | 0·1790 | 0·9221 | 0·7295 | 4·31 |
| H(6) | 0·0421 | 0·1889 | 0·3874 | 3·36 |

## Discussion

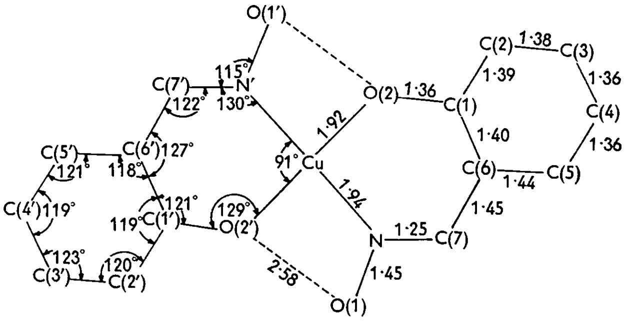

Fig. 1. Bond distances and angles.

Bond lengths and angles in the molecule of bis-(salicylaldoximato)copper(II) are listed in Table 3 with their estimated standard deviations and shown in Fig. 1.

The coordination configuration of the copper(II) ion is the usual distorted octahedral configuration. Ordinarily one finds the polyhedron for copper to deviate only very slightly from tetragonal symmetry. However, in the present case, the line between the two apical oxygen atoms makes an angle of 15° with the normal to the plane of the other ligand atoms.

The Cu–O and Cu–N distances appear to be normal. The Cu–O(2) distance of 1·92 Å may be compared with those found in similar compounds: 1·90 Å in bis-(*N*-methylsalicylaldiminato)copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961); 1·91 Å in bis-(5-chlorosalicylaldoximato)copper (II) (Orioli, Lingafelter & Brown, 1964); and 1·88 Å in bis-(*N*-phenylsalicylaldiminato)copper(II) (Wei & Lingafelter, to be published). The Cu–N distance of 1·94 Å agrees well with the distance of 1·96 Å found in bis-(5-chlorosalicylaldoximato)copper(II) and may be compared with (although it may be expected to differ somewhat from) those found in such compounds as bis-(*N*-methylsalicylaldiminato)copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961) 1·99 Å, and bis-(*N*-phenylsalicylaldiminato)copper(II), 1·99 Å (Wei, Stogsdill & Lingafelter, 1964). The 'long bond' in copper(II) coordination compounds is found to vary widely in length and the value of 2·66 Å reported here is quite reasonable.

The hydrogen bond formed between the two salicylaldoxime residues, with O–H · · · O distance of 2·58 Å, is only slightly longer than the 2·52 Å bond in bis(salicylaldoximato)nickel(II) reported by Merritt, Guare & Lessor (1956).

The bond distances within the salicylaldoxime residues do not differ significantly from the distances reported for similar molecules.

Although the molecule is nearly planar, there appear to be small but significant deviations from complete planarity. These deviations are best illustrated by the distances listed in Table 4, and by the observation that the perpendicular distance between the two parallel planes defined by the two phenyl groups is 0·13 Å. The least squares plane for each

Table 2. *Observed and calculated structure factors*

Columns are $h$, $10F_o$, $10F_c$. Unobserved reflections marked with *

| h | 10Fo | 10Fc |
|---|---|---|
| H,0,0 | | |
| 1 | 929 | 997 |
| 2 | 307 | 331 |
| 3 | 773 | 765 |
| 4 | 338 | 348 |
| 5 | 610 | 645 |
| 6 | 358 | 413 |
| 7 | 388 | 398 |
| 8 | 438 | 422 |
| 9 | 347 | 342 |
| 10 | 378 | 370 |
| 11 | 143 | 130 |
| 12 | 142 | 134 |
| 13 | 233 | 235 |
| 14 | 183 | 175 |
| 15 | 176 | 139 |
| 16 | 77 | 66 |
| 17 | 43* | 70 |
| H,0,2 | | |
| 0 | 827 | 772 |
| 1 | 332 | -275 |
| -1 | 1417 | 1292 |
| 2 | 209 | 204 |
| -2 | 78 | 122 |
| 3 | 39* | 29 |
| -3 | 409 | 434 |
| 4 | 279 | 305 |
| -4 | 199 | 238 |
| 5 | 113 | 121 |
| -5 | 241 | 257 |
| 6 | 191 | -174 |
| -6 | 341 | 358 |
| 7 | 245 | 225 |
| -7 | 251 | 269 |
| 8 | 305 | 277 |
| -8 | 415 | 332 |
| 9 | 241 | 229 |
| -9 | 228 | 190 |
| 10 | 227 | 228 |
| -10 | 298 | 288 |
| 11 | 142 | 153 |
| -11 | 182 | 149 |
| 12 | 84 | 77 |
| -12 | 171 | -166 |
| 13 | 73 | 50 |
| -13 | 84 | 66 |
| 14 | 93 | 67 |
| -14 | 145 | 167 |
| 15 | 56* | 20 |
| -15 | 104 | 107 |
| 16 | 46* | 15 |
| -16 | 134 | 96 |
| -17 | 45* | 41 |
| H,0,4 | | |
| 0 | 213 | 220 |
| 1 | 318 | 314 |
| -1 | 225 | 253 |
| 2 | 199 | 225 |
| -2 | 98 | 138 |
| 3 | 294 | 296 |
| -3 | 153 | 144 |
| 4 | 295 | 288 |
| -4 | 180 | 178 |
| 5 | 59* | -5 |
| -5 | 179 | 182 |
| 6 | 176 | 161 |
| -6 | 457 | 462 |
| 7 | 383 | 360 |
| -7 | 289 | 280 |
| 8 | 284 | 264 |
| -8 | 174 | 178 |
| 9 | 239 | 228 |
| -9 | 219 | 206 |
| 10 | 214 | 225 |
| -10 | 173 | 159 |
| 11 | 113 | 94 |
| -11 | 270 | 248 |
| 12 | 65* | 11 |
| -12 | 70* | 16 |
| 13 | 86 | 66 |
| -13 | 96 | -98 |
| 14 | 50* | 57 |
| -14 | 87 | 67 |
| 15 | 38* | 41 |
| -15 | 69 | 54 |
| -16 | 48* | 48 |
| -17 | 35* | 35 |
| H,0,6 | | |
| 0 | 425 | 424 |
| 1 | 284 | 289 |
| -1 | 305 | 303 |
| 2 | 278 | 275 |
| -2 | 115 | 127 |
| 3 | 281 | 278 |
| -3 | 135 | 153 |
| 4 | 183 | 187 |
| -4 | 132 | 164 |
| 5 | 181 | 175 |
| -5 | 95 | 104 |
| 6 | 184 | 202 |
| -6 | 283 | 272 |
| 7 | 179 | 185 |
| -7 | 383 | 387 |
| 8 | 150 | 132 |
| -8 | 200 | 196 |
| 9 | 112 | 131 |
| -9 | 155 | 171 |
| 10 | 111 | 102 |
| -10 | 153 | 173 |
| 11 | 53* | 22 |
| -11 | 171 | 158 |
| 12 | 44* | 46 |
| -12 | 178 | 166 |
| -13 | 58* | 77 |
| -14 | 51* | 73 |
| -15 | 41* | 81 |
| H,0,8 | | |
| 0 | 106 | 106 |
| 1 | 72 | 65 |
| -1 | 191 | 177 |
| 2 | 87 | 79 |
| -2 | 97 | 69 |
| 3 | 91 | 71 |
| -3 | 68* | 31 |
| 4 | 123 | 102 |
| -4 | 99 | 55 |
| 5 | 59* | 19 |
| -5 | 67* | 18 |
| 6 | 55* | -19 |
| -6 | 66* | 16 |
| 7 | 50* | 23 |
| -7 | 77 | 86 |
| 8 | 43* | 20 |
| -8 | 87 | 84 |
| -9 | 58* | 29 |
| -10 | 54* | 50 |
| -11 | 48* | 45 |
| -12 | 41* | 20 |
| H,0,10 | | |
| 0 | 35* | 37 |
| -1 | 37* | 67 |
| -2 | 38* | 71 |
| -3 | 38* | 42 |
| -4 | 37* | 51 |
| H,1,0 | | |
| 1 | 20* | 37 |
| 2 | 142 | 170 |
| 3 | 60 | 80 |
| 4 | 85 | 74 |
| 5 | 59 | 44 |
| 6 | 184 | 193 |
| 7 | 234 | 236 |
| 8 | 64* | -34 |
| 9 | 69* | -12 |
| 10 | 74* | 9 |
| 11 | 167 | -207 |
| 12 | 138 | -107 |
| 13 | 81* | 75 |
| 14 | 78* | 33 |
| 15 | 72* | 26 |
| 16 | 62* | 23 |
| 17 | 48* | 7 |
| H,1,1 | | |
| 0 | 1296 | 1087 |
| 1 | 69 | 93 |
| -1 | 711 | 643 |
| 2 | 411 | 428 |
| -2 | 78 | -52 |
| 3 | 220 | 275 |
| -3 | 327 | 311 |
| 4 | 275 | 312 |
| -4 | 108 | 151 |
| 5 | 347 | 407 |
| -5 | 496 | 496 |
| 6 | 84 | 102 |
| -6 | 258 | 280 |
| 7 | 269 | 266 |
| -7 | 68 | 89 |
| 8 | 344 | 339 |
| -8 | 272 | 263 |
| 9 | 324 | 312 |
| -9 | 231 | 227 |
| 10 | 249 | 232 |
| -10 | 293 | 286 |
| 11 | 85 | 77 |
| -11 | 96 | 101 |
| 12 | 115 | 117 |
| -12 | 81* | -12 |
| 13 | 86 | 111 |
| -13 | 96 | 108 |
| 14 | 105 | 102 |
| -14 | 79* | 81 |
| 15 | 103 | 84 |
| -15 | 73* | 74 |
| 16 | 58* | 43 |
| -16 | 63* | 43 |
| 17 | 42* | 58 |
| -17 | 50* | 18 |
| H,1,2 | | |
| 0 | 722 | 589 |
| 1 | 217 | -187 |
| -1 | 1285 | 951 |
| 2 | 67 | 63 |
| -2 | 83 | 106 |
| 3 | 46* | -15 |
| -3 | 91 | 109 |
| 4 | 61 | -30 |
| -4 | 120 | 118 |
| 5 | 80 | 81 |
| -5 | 191 | -179 |
| 6 | 137 | 159 |
| -6 | 91 | -89 |
| 7 | 65* | 19 |
| -7 | 60* | -28 |
| 8 | 96 | -79 |
| -8 | 65* | -21 |
| 9 | 75* | 16 |
| -9 | 70* | 32 |
| 10 | 100 | -77 |
| -10 | 75* | -13 |
| 11 | 115 | -115 |
| -11 | 79* | 41 |
| 12 | 91 | 76 |
| -12 | 81* | 105 |
| 13 | 100 | 81 |
| -13 | 81* | 45 |
| 14 | 73* | 27 |
| -14 | 78* | 30 |
| 15 | 64* | 12 |
| -15 | 72* | 33 |
| 16 | 51* | -2 |
| -16 | 63* | 11 |
| -17 | 50* | 9 |
| H,1,3 | | |
| 0 | 588 | 564 |
| 1 | 534 | 527 |
| -1 | 1087 | 965 |
| 2 | 539 | 524 |
| -2 | 503 | 470 |
| 3 | 368 | 403 |
| -3 | 320 | 335 |
| 4 | 522 | 503 |
| -4 | 326 | 352 |
| 5 | 274 | 288 |
| -5 | 377 | 426 |
| 6 | 251 | 252 |
| -6 | 624 | 618 |
| 7 | 344 | 359 |
| -7 | 324 | 340 |
| 8 | 277 | 305 |
| -8 | 301 | 322 |
| 9 | 259 | 259 |
| -9 | 274 | 299 |
| 10 | 154 | 190 |
| -10 | 246 | 263 |
| 11 | 119 | 155 |
| -11 | 257 | 289 |
| 12 | 105 | 106 |
| -12 | 82* | 78 |
| 13 | 105 | 104 |
| -13 | 81* | 87 |
| 14 | 85 | 98 |
| -14 | 133 | 159 |
| 15 | 55* | 58 |
| -15 | 89 | 111 |
| -16 | 90 | 96 |
| -17 | 46* | 44 |
| H,1,4 | | |
| 0 | 335 | -316 |
| 1 | 303 | -282 |
| -1 | 363 | 343 |
| 2 | 134 | -143 |
| -2 | 240 | 242 |
| 3 | 157 | -151 |
| -3 | 75 | -35 |
| 4 | 85 | -68 |
| -4 | 81 | 95 |
| 5 | 150 | -155 |
| -5 | 68 | -61 |
| 6 | 73* | -75 |
| -6 | 196 | -198 |
| 7 | 77* | -3 |
| -7 | 86 | -90 |
| 8 | 80* | -14 |
| -8 | 74* | -19 |
| 9 | 81* | 14 |
| -9 | 77* | -39 |
| 10 | 81* | -32 |
| -10 | 80* | -29 |
| 11 | 75* | -8 |
| -11 | 82* | 1 |
| 12 | 74* | 21 |
| -12 | 81* | -67 |
| 13 | 67* | -4 |
| -13 | 79* | -97 |
| 14 | 56* | -14 |
| -14 | 73* | -14 |
| -15 | 65* | 9 |
| -16 | 54* | 2 |
| H,1,5 | | |
| 0 | 222 | 224 |
| 1 | 200 | 209 |
| -1 | 209 | 213 |
| 2 | 134 | 145 |
| -2 | 221 | 238 |
| 3 | 200 | 207 |
| -3 | 121 | 127 |
| 4 | 127 | 128 |
| -4 | 92 | 125 |
| 5 | 82 | 75 |
| -5 | 76 | 94 |
| 6 | 93 | 112 |
| -6 | 209 | 225 |
| 7 | 103 | 102 |
| -7 | 206 | 232 |
| 8 | 108 | 101 |
| -8 | 115 | 112 |
| 9 | 90 | 97 |
| -9 | 121 | 150 |
| 10 | 103 | 97 |
| -10 | 87 | 106 |
| 11 | 72* | 38 |
| -11 | 118 | 124 |
| 12 | 65* | 23 |
| -12 | 92 | 98 |
| 13 | 54* | 45 |
| -13 | 74* | 4 |
| -14 | 67* | 62 |
| -15 | 58* | 80 |
| -16 | 44* | 63 |
| H,1,6 | | |
| 0 | 108 | -108 |
| 1 | 77* | -10 |
| -1 | 76* | -37 |
| 2 | 78* | -16 |
| -2 | 76* | 83 |
| 3 | 80* | 31 |
| -3 | 77* | -8 |
| 4 | 81* | -66 |
| -4 | 78* | 19 |
| 5 | 82* | -77 |
| -5 | 79* | 23 |
| 6 | 108 | 119 |
| -6 | 80* | 15 |
| 7 | 95 | 100 |
| -7 | 81* | 35 |
| 8 | 78* | 45 |
| -8 | 82* | 19 |
| 9 | 74* | 47 |
| -9 | 82* | 19 |
| 10 | 68* | 4 |
| -10 | 80* | 2 |
| 11 | 60* | -10 |
| -11 | 77* | 48 |
| 12 | 49* | -0 |
| -12 | 73* | 50 |
| -13 | 66* | -71 |
| -14 | 58* | -64 |
| -15 | 46* | -6 |
| H,1,7 | | |
| 0 | 153 | 169 |
| 1 | 108 | 112 |
| -1 | 147 | 163 |
| 2 | 132 | 146 |
| -2 | 86 | 99 |
| 3 | 125 | 122 |
| -3 | 91 | 106 |
| 4 | 95 | 113 |
| -4 | 100 | 105 |
| 5 | 84 | 101 |
| -5 | 96 | 102 |
| 6 | 77* | 51 |
| -6 | 91 | 100 |
| 7 | 81 | 82 |
| -7 | 131 | 142 |
| 8 | 79 | 92 |
| -8 | 80* | 96 |
| 9 | 107 | 94 |
| -9 | 77* | 60 |
| 10 | 51* | 62 |
| -10 | 74* | 81 |
| -11 | 69* | 58 |
| -12 | 62* | 58 |
| -13 | 54* | 34 |
| -14 | 41* | 17 |
| H,1,8 | | |
| 0 | 78* | 22 |
| 1 | 77* | 13 |
| -1 | 79* | 16 |
| 2 | 76* | 27 |
| -2 | 79* | 13 |
| 3 | 74* | 6 |
| -3 | 79* | -21 |
| 4 | 71* | -49 |
| -4 | 78* | -17 |
| 5 | 68* | 44 |
| -5 | 77* | -37 |
| 6 | 63* | 83 |
| -6 | 76* | -46 |
| 7 | 56* | 8 |
| -7 | 73* | 59 |
| 8 | 48* | -2 |
| -8 | 70* | 62 |
| -9 | 66* | 14 |
| -10 | 61* | 13 |
| -11 | 54* | 14 |
| -12 | 45* | 51 |
| H,1,9 | | |
| 0 | 73 | 78 |
| 1 | 95 | 81 |
| -1 | 117 | 123 |
| 2 | 71 | 78 |
| -2 | 94 | 92 |
| 3 | 78 | 83 |
| -3 | 70 | 63 |
| 4 | 76 | 91 |
| -4 | 73 | 72 |
| 5 | 46* | 27 |
| -5 | 81 | 83 |
| -6 | 86 | 102 |
| -7 | 79 | 85 |
| -8 | 77 | 77 |
| -9 | 47* | 60 |
| H,1,10 | | |
| -2 | 41* | 13 |
| -3 | 41* | -7 |
| H,2,0 | | |
| 1 | 235 | 275 |
| 2 | 530 | 549 |
| 3 | 333 | 396 |
| 4 | 534 | 575 |
| 5 | 691 | 733 |
| 6 | 280 | 320 |
| 7 | 267 | 278 |
| 8 | 347 | 367 |
| 9 | 323 | 329 |
| 10 | 302 | 288 |
| 11 | 152 | 157 |
| 12 | 127 | 133 |
| 13 | 102 | 88 |
| 14 | 88 | 69 |
| 15 | 86 | 83 |
| 16 | 48* | 51 |
| H,2,1 | | |
| 0 | 68 | -51 |
| 1 | 466 | -452 |
| -1 | 60 | -25 |
| 2 | 356 | -378 |
| -2 | 97 | -44 |
| 3 | 224 | -243 |
| -3 | 72 | 59 |
| 4 | 86 | -64 |
| -4 | 129 | -121 |
| 5 | 44* | -28 |
| -5 | 272 | -297 |
| 6 | 85 | -91 |
| -6 | 203 | -193 |
| 7 | 76 | -42 |
| -7 | 199 | -199 |
| 8 | 88 | -93 |
| -8 | 80 | -79 |
| 9 | 63* | -14 |
| -9 | 61* | 27 |
| 10 | 66* | -46 |
| -10 | 65* | -48 |
| 11 | 185 | -204 |
| -11 | 68* | 15 |
| 12 | 69* | -51 |
| -12 | 69* | 40 |
| 13 | 67* | 28 |
| -13 | 68* | -67 |
| 14 | 62* | -25 |
| -14 | 65* | -51 |
| 15 | 55* | -15 |
| -15 | 58* | -17 |
| 16 | 44* | -34 |
| -16 | 49* | -4 |
| -17 | 36* | 5 |
| H,2,2 | | |
| 0 | 217 | 245 |
| 1 | 461 | 489 |
| -1 | 234 | 265 |
| 2 | 418 | 456 |
| -2 | 159 | 141 |
| 3 | 279 | 328 |
| -3 | 174 | 171 |
| 4 | 466 | 448 |
| -4 | 148 | 152 |
| 5 | 293 | 316 |
| -5 | 407 | 412 |
| 6 | 279 | 286 |
| -6 | 361 | 406 |
| 7 | 229 | 221 |
| -7 | 78 | 60 |
| 8 | 135 | 146 |
| -8 | 172 | 178 |
| 9 | 223 | 196 |
| -9 | 236 | 244 |
| 10 | 84 | 35 |
| -10 | 194 | 192 |
| 11 | 69* | 6 |
| -11 | 213 | 211 |
| 12 | 102 | 92 |
| -12 | 209 | 196 |
| 13 | 91 | 70 |
| -13 | 161 | 135 |
| 14 | 85 | 74 |
| -14 | 88 | 66 |
| 15 | 50* | 58 |
| -15 | 84 | 73 |
| 16 | 36* | 41 |
| -16 | 49* | 43 |
| -17 | 35* | 12 |
| H,2,3 | | |
| 0 | 43* | -34 |
| 1 | 221 | -209 |
| -1 | 431 | 421 |
| 2 | 46* | 19 |
| -2 | 53 | 35 |
| 3 | 69 | -53 |
| -3 | 66 | -28 |
| 4 | 63 | 55 |
| -4 | 73 | 60 |
| 5 | 55* | -2 |
| -5 | 76 | -62 |
| 6 | 59* | -0 |
| -6 | 69 | -45 |
| 7 | 119 | 118 |
| -7 | 81 | -61 |
| 8 | 106 | 105 |
| -8 | 92 | -81 |
| 9 | 93 | 77 |
| -9 | 64* | 10 |
| 10 | 101 | -77 |
| -10 | 82 | 52 |
| 11 | 97 | -61 |
| -11 | 69* | 39 |
| 12 | 80 | 68 |
| -12 | 69* | -3 |
| 13 | 60* | 33 |
| -13 | 67* | -27 |
| 14 | 52* | 23 |
| -14 | 62* | -33 |
| 15 | 41* | 2 |
| -15 | 56* | -28 |
| -16 | 46* | -9 |
| H,2,4 | | |
| 0 | 273 | 276 |
| 1 | 306 | 333 |
| -1 | 456 | 462 |
| 2 | 205 | 229 |
| -2 | 425 | 428 |
| 3 | 243 | 247 |
| -3 | 141 | 155 |
| 4 | 234 | 238 |
| -4 | 189 | 215 |
| 5 | 243 | 271 |
| -5 | 153 | 160 |
| 6 | 196 | 212 |
| -6 | 197 | 202 |
| 7 | 67* | 25 |
| -7 | 141 | 145 |
| 8 | 106 | 113 |
| -8 | 79 | 61 |
| 9 | 110 | 100 |
| -9 | 124 | 137 |
| 10 | 68* | 29 |
| -10 | 97 | 97 |
| 11 | 115 | 109 |
| -11 | 101 | 82 |
| 12 | 108 | 109 |
| -12 | 102 | 120 |
| 13 | 89 | 87 |
| -13 | 109 | 111 |
| 14 | 42* | 67 |
| -14 | 98 | 90 |
| -15 | 77 | 77 |
| -16 | 40* | 66 |
| H,2,5 | | |
| 0 | 100 | -98 |
| 1 | 61* | 40 |
| -1 | 205 | 205 |
| 2 | 62* | 8 |
| -2 | 294 | 287 |
| 3 | 64* | 28 |
| -3 | 61* | 6 |
| 4 | 66* | 33 |
| -4 | 62* | 0 |
| 5 | 82 | -86 |
| -5 | 63* | -14 |
| 6 | 87 | 81 |
| -6 | 65* | 52 |
| 7 | 107 | 131 |
| -7 | 67* | 79 |
| 8 | 69* | 36 |
| -8 | 68* | 35 |
| 9 | 67* | -10 |
| -9 | 69* | 36 |
| 10 | 81 | -48 |
| -10 | 69* | 33 |
| 11 | 58* | 12 |
| -11 | 86 | 91 |
| 12 | 51* | 28 |
| -12 | 65* | 57 |
| 13 | 40* | 7 |
| -13 | 60* | -27 |
| -14 | 53* | 8 |
| -15 | 44* | 22 |
| H,2,6 | | |
| 0 | 140 | 135 |
| 1 | 149 | 149 |
| -1 | 187 | 172 |
| 2 | 168 | 153 |
| -2 | 375 | 358 |
| 3 | 163 | 163 |
| -3 | 262 | 249 |
| 4 | 129 | 144 |
| -4 | 198 | 205 |
| 5 | 149 | 162 |
| -5 | 262 | 273 |
| 6 | 69* | 55 |
| -6 | 135 | 143 |
| 7 | 67* | 62 |
| -7 | 127 | 114 |
| 8 | 124 | 133 |
| -8 | 117 | 114 |
| 9 | 101 | 96 |
| -9 | 113 | 109 |
| 10 | 95 | 93 |
| -10 | 100 | 111 |
| 11 | 46* | 74 |
| -11 | 74 | 75 |
| -12 | 75 | 76 |
| -13 | 69 | 47 |
| -14 | 44* | 43 |
| H,2,7 | | |
| 0 | 81 | -72 |
| 1 | 73 | -36 |
| -1 | 73 | -74 |
| 2 | 73 | -37 |
| -2 | 69* | 54 |
| 3 | 68* | -10 |
| -3 | 69* | 10 |
| 4 | 100 | -112 |
| -4 | 69* | -57 |
| 5 | 65* | -61 |
| -5 | 112 | -119 |
| 6 | 62* | 53 |
| -6 | 105 | -92 |
| 7 | 58* | -8 |
| -7 | 67* | 23 |
| 8 | 53* | -31 |
| -8 | 66* | -15 |
| 9 | 47* | -28 |
| -9 | 63* | -26 |
| 10 | 37* | -26 |
| -10 | 59* | -28 |
| -11 | 55* | -19 |
| -12 | 48* | 30 |
| -13 | 40* | -31 |
| H,2,8 | | |
| 0 | 64* | 48 |
| 1 | 74 | 43 |
| -1 | 65* | 39 |
| 2 | 79 | 69 |
| -2 | 76 | 61 |
| 3 | 73 | 46 |
| -3 | 88 | 53 |
| 4 | 67 | 52 |
| -4 | 82 | 50 |
| 5 | 71 | 58 |
| -5 | 118 | 120 |
| 6 | 69 | 45 |
| -6 | 119 | 132 |
| 7 | 42* | 68 |
| -7 | 59* | 55 |
| -8 | 56* | 58 |
| -9 | 52* | 54 |
| -10 | 47* | 53 |
| -11 | 40* | 52 |
| H,2,9 | | |
| 0 | 51* | 18 |
| 1 | 49* | 28 |
| -1 | 52* | -6 |
| 2 | 47* | 28 |
| -2 | 52* | 14 |
| 3 | 43* | -10 |
| -3 | 52* | 27 |
| 4 | 38* | -26 |
| -4 | 51* | 23 |
| -5 | 49* | -3 |
| -6 | 47* | -57 |
| -7 | 44* | 10 |
| -8 | 39* | 46 |
| H,3,0 | | |
| 1 | 54 | -74 |
| 2 | 234 | -258 |
| 3 | 142 | -121 |
| 4 | 31 | -12 |
| 5 | 82 | 89 |
| 6 | 58 | -45 |
| 7 | 48 | -43 |
| 8 | 69 | 77 |
| 9 | 49* | 35 |
| 10 | 51* | 51 |
| 11 | 52* | 59 |
| 12 | 52* | 36 |
| 13 | 49* | 50 |
| 14 | 45* | 28 |
| 15 | 38* | 8 |
| 16 | 29* | -23 |
| H,3,1 | | |
| 0 | 31 | 61 |
| 1 | 409 | 371 |
| -1 | 212 | 226 |
| 2 | 365 | 348 |
| -2 | 509 | 481 |
| 3 | 236 | 233 |
| -3 | 382 | 367 |
| 4 | 483 | 462 |
| -4 | 318 | 312 |
| 5 | 382 | 367 |
| -5 | 382 | 364 |
| 6 | 304 | 290 |
| -6 | 280 | 256 |
| 7 | 260 | 248 |
| -7 | 219 | 199 |
| 8 | 48* | 60 |
| -8 | 194 | 200 |
| 9 | 94 | 113 |
| -9 | 104 | 129 |
| 10 | 101 | 134 |
| -10 | 89 | 92 |
| 11 | 95 | 174 |
| -11 | 111 | 141 |
| 12 | 89 | 136 |
| -12 | 110 | 174 |
| 13 | 48* | 82 |
| -13 | 50* | 94 |
| 14 | 43* | 74 |
| -14 | 46* | 71 |
| 15 | 36* | 50 |
| -15 | 39* | 78 |
| -16 | 30* | 34 |
| H,3,2 | | |
| 0 | 29* | 13 |
| 1 | 88 | -88 |
| -1 | 29* | -24 |
| 2 | 32* | -4 |
| -2 | 132 | 106 |
| 3 | 81 | 75 |
| -3 | 177 | 166 |
| 4 | 232 | 217 |
| -4 | 69 | 83 |
| 5 | 84 | 104 |
| -5 | 166 | 163 |
| 6 | 66 | -80 |
| -6 | 156 | 150 |
| 7 | 108 | 126 |
| -7 | 62 | 90 |
| 8 | 187 | 181 |
| -8 | 88 | 73 |
| 9 | 82 | 96 |
| -9 | 111 | 106 |
| 10 | 52* | 28 |
| -10 | 100 | 130 |
| 11 | 52* | -33 |
| -11 | 53* | 62 |
| 12 | 50* | 14 |
| -12 | 52* | 28 |
| 13 | 45* | 9 |
| -13 | 45* | 7 |
| 14 | 39* | -1 |
| -14 | 45* | -24 |
| 15 | 31* | -10 |
| -15 | 39* | -4 |
| -16 | 30* | 24 |
| H,3,3 | | |
| 0 | 157 | 136 |
| 1 | 281 | 251 |
| -1 | 164 | 151 |
| 2 | 197 | 170 |
| -2 | 282 | 265 |
| 3 | 334 | 295 |
| -3 | 366 | 338 |
| 4 | 365 | 313 |
| -4 | 391 | 366 |
| 5 | 264 | 235 |
| -5 | 260 | 217 |
| 6 | 292 | 277 |
| -6 | 227 | 182 |
| 7 | 66 | 54 |
| -7 | 119 | 119 |
| 8 | 97 | 76 |
| -8 | 101 | 92 |
| 9 | 180 | 175 |
| -9 | 170 | 179 |
| 10 | 82 | 54 |
| -10 | 117 | 116 |
| 11 | 115 | 86 |
| -11 | 87 | 81 |
| 12 | 123 | 96 |
| -12 | 102 | 151 |
| 13 | 98 | 60 |
| -13 | 68 | 113 |
| 14 | 33* | 64 |
| -14 | 43* | 41 |
| -15 | 37* | 54 |
| H,3,4 | | |
| 0 | 128 | -119 |
| 1 | 87 | -70 |
| -1 | 82 | -53 |
| 2 | 83 | -48 |
| -2 | 140 | -123 |
| 3 | 46* | -25 |
| -3 | 178 | -144 |
| 4 | 48* | 18 |
| -4 | 256 | -220 |
| 5 | 129 | -131 |
| -5 | 185 | -155 |
| 6 | 51* | -67 |
| -6 | 48* | 45 |
| 7 | 74 | 67 |
| -7 | 50* | -38 |
| 8 | 52* | 9 |
| -8 | 85 | -104 |
| 9 | 82 | -73 |
| -9 | 52* | -43 |
| 10 | 161 | -143 |
| -10 | 52* | 7 |
| 11 | 61 | -60 |
| -11 | 51* | 31 |
| 12 | 41* | 1 |
| -12 | 49* | 7 |
| 13 | 34* | -26 |
| -13 | 45* | 1 |
| -14 | 40* | -27 |
| -15 | 32* | -36 |
| H,3,5 | | |
| 0 | 141 | 126 |
| 1 | 199 | 163 |
| -1 | 181 | 153 |
| 2 | 176 | 140 |
| -2 | 284 | 244 |
| 3 | 214 | 178 |
| -3 | 153 | 128 |
| 4 | 167 | 145 |
| -4 | 287 | 245 |
| 5 | 196 | 201 |
| -5 | 332 | 270 |
| 6 | 129 | 125 |
| -6 | 154 | 101 |
| 7 | 74 | 55 |
| -7 | 140 | 117 |
| 8 | 164 | 148 |
| -8 | 83 | 71 |
| 9 | 76 | 61 |
| -9 | 91 | 82 |
| 10 | 49 | 26 |
| -10 | 130 | 127 |
| 11 | 68 | 75 |
| -11 | 77 | 78 |
| 12 | 32* | 55 |
| -12 | 76 | 90 |
| -13 | 61 | 95 |
| -14 | 34* | 55 |
| H,3,6 | | |
| 0 | 52* | -5 |
| 1 | 52* | 59 |
| -1 | 58 | 48 |
| 2 | 52* | 35 |
| -2 | 143 | 148 |
| 3 | 52* | 56 |
| -3 | 64 | 52 |
| 4 | 52* | 15 |
| -4 | 69 | -49 |
| 5 | 52* | -24 |
| -5 | 139 | -127 |
| 6 | 50* | 31 |
| -6 | 52* | -12 |
| 7 | 48* | 29 |
| -7 | 52* | 60 |
| 8 | 45* | 3 |
| -8 | 51* | -34 |
| 9 | 50 | -47 |
| -9 | 50* | -32 |
| 10 | 35* | -20 |
| -10 | 47* | -16 |
| -11 | 44* | 3 |
| -12 | 39* | 20 |
| -13 | 33* | 5 |
| H,3,7 | | |
| 0 | 89 | 93 |
| 1 | 81 | 61 |
| -1 | 68 | 67 |
| 2 | 80 | 70 |
| -2 | 186 | 166 |
| 3 | 93 | 92 |
| -3 | 118 | 137 |
| 4 | 83 | 86 |
| -4 | 57 | 45 |
| 5 | 105 | 102 |
| -5 | 141 | 152 |
| 6 | 71 | 69 |
| -6 | 96 | 105 |
| 7 | 90 | 72 |
| -7 | 59 | 46 |
| 8 | 34* | 51 |
| -8 | 73 | 79 |
| 9 | 27* | 17 |
| -9 | 62 | 53 |
| -10 | 67 | 65 |
| -11 | 36* | 60 |
| -12 | 29* | 54 |
| H,3,8 | | |
| 0 | 45* | 5 |
| 1 | 44* | 37 |
| -1 | 46* | -40 |
| 2 | 43* | 21 |
| -2 | 46* | 61 |
| 3 | 41* | 0 |
| -3 | 102 | 105 |
| 4 | 38* | -28 |
| -4 | 45* | 46 |
| 5 | 35* | -14 |
| -5 | 44* | -0 |
| 6 | 30* | -2 |
| -6 | 42* | -16 |
| -7 | 40* | 38 |
| -8 | 37* | 26 |
| -9 | 33* | -3 |
| -10 | 28* | 1 |
| H,3,9 | | |
| 0 | 32* | 32 |
| 1 | 30* | 26 |
| -1 | 33* | 42 |
| 2 | 27* | 59 |
| -2 | 33* | 48 |
| -3 | 33* | 90 |
| -4 | 32* | 50 |
| -5 | 30* | 34 |
| -6 | 28* | 54 |

column in Table 4 is defined by the atoms listed above the horizontal line. The equations of the planes, in a Cartesian coordinate system with $x$, $y$ and $z$ axes parallel to **a**, **b** and **c*** are:

$$\begin{array}{ll} A & 0{\cdot}07091x+0{\cdot}20604y-0{\cdot}39364z+1=0 \\ B & 0{\cdot}06913x+0{\cdot}20016y-0{\cdot}38859z+1=0 \\ C & 0{\cdot}07123x+0{\cdot}19917y-0{\cdot}38960z+1=0 \\ D & 0{\cdot}08129x+0{\cdot}22165y-0{\cdot}41131z+1=0\,. \end{array}$$

Intermolecular contact distances are all reasonable. The shortest distances observed between the several types of atoms are: O–O, 3·0; O–N, 2·9; O–C, 3·5; N–C, 4·2; and C–C, 3·6 Å.

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Table 3. *Bond lengths and angles and their e.s.d.'s*

| | |
|---|---|
| Cu–O(2) | 1·92(0·01) |
| Cu–N | 1·94(0·01) |
| Cu–O(1″) | 2·66(0·01) |
| N–O(1) | 1·45(0·02) |
| C(7)–N | 1·25(0·02) |
| C(6)–C(7) | 1·45(0·02) |
| C(1)–O(2) | 1·36(0·02) |
| C(1)–C(2) | 1·39(0·02) |
| C(2)–C(3) | 1·38(0·02) |
| C(3)–C(4) | 1·36(0·03) |
| C(4)–C(5) | 1·36(0·02) |
| C(5)–C(6) | 1·44(0·02) |
| C(6)–C(1) | 1·40(0·02) |
| O(1)–O(2′) | 2·58(0·02) |

| | |
|---|---|
| O(2)–Cu–N | 91·4(0·5) |
| Cu–O(2)–C(1) | 128·6(1·1) |
| Cu–N–O(1) | 114·8(0·8) |
| Cu–N–C(7) | 130·1(1·4) |
| O(1)–N–C(7) | 115·0(1·5) |
| N–C(7)–C(6) | 122·1(1·9) |
| O(2)–C(1)–C(6) | 120·9(1·7) |
| O(2)–C(1)–C(2) | 119·9(1·7) |
| C(6)–C(1)–C(2) | 119·1(1·8) |
| C(1)–C(2)–C(3) | 119·9(1·9) |
| C(2)–C(3)–C(4) | 122·8(2·2) |
| C(3)–C(4)–C(5) | 118·6(2·1) |
| C(4)–C(5)–C(6) | 121·3(2·0) |
| C(5)–C(6)–C(1) | 118·2(1·7) |
| C(5)–C(6)–C(7) | 115·1(1·7) |
| C(1)–C(6)–C(7) | 126·6(2·0) |
| O(2)–Cu–N′ | 88·6(0·5) |
| N–Cu–O(1″) | 104·2(0·5) |
| O(2)–Cu–O(1″) | 85·1(0·5) |
| O(2′)–Cu–O(1″) | 94·9(0·5) |
| N′–Cu–O(1″) | 75·8(0·5) |

Table 4. *Distances of atoms from least squares planes*
(Å × $10^3$)

| | *A* | *B* | *C* | *D* |
|---|---|---|---|---|
| C(1) | −006 | −001 | −005 | −031 |
| C(2) | −004 | +006 | +009 | −022 |
| C(3) | +009 | +013 | +020 | +014 |
| C(4) | −003 | −008 | −004 | +022 |
| C(5) | −007 | −015 | −019 | +012 |
| C(6) | $\underline{+011}$ | +008 | −000 | +005 |
| C(7) | +020 | +012 | −004 | +011 |
| O(2) | −025 | $\underline{-014}$ | −022 | −074 |
| N | +048 | +044 | $\underline{+024}$ | +018 |
| O(1) | −036 | −046 | −074 | −065 |
| Cu | +062 | +070 | +052 | 000 |
| O(1′) | +160 | +187 | +178 | +065 |
| N′ | +077 | +097 | +080 | −018 |
| O(2′) | +150 | +154 | +126 | +074 |
| C(7′) | +105 | +129 | +108 | −011 |
| C(6′) | +114 | +133 | +104 | −005 |
| C(5′) | +132 | +156 | +126 | −012 |
| C(4′) | +128 | +149 | +108 | −022 |
| C(3′) | +116 | +128 | +084 | −014 |
| C(2′) | +128 | +135 | +095 | +022 |
| C(1′) | +131 | +142 | +109 | $\underline{+031}$ |

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