

The Crystal Structure of the Copper(II) Complex with *o*-Hydroxyacetophenone Imine

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The crystal structure of the copper(II) complex with *o*-hydroxyacetophenone imine, $\text{Cu}(\text{C}_8\text{H}_8\text{NO})_2$, has been determined from 5170 independent reflexions, measured with Mo $K\alpha$ radiation by diffractometer, and refined by least squares to $R=0.037$. Crystal data are: $a_0=11.4359$ (10), $b_0=12.3695$ (9), $c_0=9.4460$ (9) Å, $\alpha=103.953$ (10), $\beta=111.168$ (8), $\gamma=102.729$ (5)°, $Z=3$, space group $P\bar{1}$. The two independent molecules have essentially the same bond distances, but markedly different distortions from planarity. Coordination of the copper(II) ion is planar, with mean Cu-N=1.922 (2) Å and mean Cu-O=1.880 (2) Å.

Introduction

N-Substituted *o*-hydroxyacetophenone imines form bis-chelates with copper(II), $[-\text{O}-\text{C}_6\text{H}_4-\text{C}(\text{CH}_3)=\text{N}(\text{R})-]_2\text{Cu}$, which are green or brown in the solid depending on the substituent, R. We have previously reported the structure of the isobutyl compound (Ghilardi & Lingafelter, 1969) and have now completed the determination of the structure of the unsubstituted compound, R = H.

Experimental

The brown crystals were prepared by Professor Forrest D. Thomas II of the University of Montana. A series of oscillation and Weissenberg photographs showed the crystals to be triclinic laths, bounded by {010} and {323} and terminated by several small faces which were not identified. For collection of diffractometer data, a crystal was selected which was 0.35 mm long, 0.18 mm wide, and 0.08 mm thick.

All measurements were made with Mo $K\alpha$ radiation, filtered through 0.001 inch of niobium foil, on a Picker automated diffractometer equipped with a scintillation counter and a pulse-height discriminator. The take-off angle was set at 3.5°.

The cell constants and their estimated standard deviations were obtained by least-squares fit of 32 2θ values (averages of $+2\theta$ and -2θ) in the range $22^\circ < 2\theta < 43^\circ$, measured at 26°C. The final values are $a_0=11.4359$ (10), $b_0=12.3695$ (9), $c_0=9.4460$ (9) Å, $\alpha=103.953$ (10), $\beta=111.168$ (8), $\gamma=102.729$ (5)°. The density measured by flotation, 1.44 g.cm⁻³, agrees well with the value of 1.452 g.cm⁻³ calculated for three molecules per cell. The space group was assumed to be $P\bar{1}$ and this was confirmed by the structure determination.

The intensity data were collected by the ω - 2θ scan method using the formula of Alexander & Smith (1964): scan range = $1.10 + 1.00 \tan \theta$. Stationary background measurements were made (for 22 seconds) at

the start and finish of each scan. After each four hours of data collection the intensities of four standard reflections were remeasured and the weighted mean of these values was used to calculate a scale factor for each set of reflections. No systematic variation of scale factors was observed and the maximum variation was $\pm 1\%$. Intensities were measured for the 5170 unique reflections in the range $0 < 2\theta < 55^\circ$. Measurements were recorded in truncated dekaounts and the intensities and their estimated standard deviations were calculated from

$$I = S - \frac{t_s}{2t_B} (B_1 + B_2) - 0.45 \left(2 \frac{t_s}{2t_B} - 1 \right)$$

$$\sigma_I^2 = \frac{1}{10} \left[S + \left(\frac{t_s}{2t_B} \right)^2 (B_1 + B_2) \right]$$

$$+ k^2 \left[S + \frac{t_s}{2t_B} (B_1 + B_2) \right]^2 + 0.25$$

S = dekaounts recorded during scan time t_s

B_i = dekaounts recorded during background time t_B

k = empirical stability constant

The terms 0.45 and 0.25 are corrections for the truncation of counts to dekaounts.

The appropriate value of $k(0.004)$ was determined from the analysis of eight selected reflections, each remeasured 25 times.

1053 reflections, each having a calculated intensity less than twice its estimated standard deviation, were coded as 'unobserved' and assigned intensity values of $I + 2\sigma$ for special treatment during the least-squares refinement. Lorentz and polarization factors were applied and the structure factor and its standard deviation were calculated for each reflection. No correction was made for absorption ($\mu=22.6$ cm⁻¹, range of transmission factors, 0.5 to 0.8) nor for anomalous dispersion.

Structure determination and refinement

All calculations were carried out on an IBM 7040-7094 direct-coupled system, using the set of programs written or adapted by Stewart (1964). Scattering factors for

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copper, oxygen, nitrogen and carbon atoms were taken from Cromer & Waber (1965) and for hydrogen from Table II of Stewart, Davidson & Simpson (1965).

The copper positions were determined from an unsharpened three-dimensional Patterson synthesis. All other non-hydrogen atoms were located by a series of

Table 1. *Positional and thermal parameters*(a) Positional parameters ($\times 10^4$) and thermal parameters ($\times 10^2$)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
Cu ²⁺ (1)	5000 (0)	5000 (0)	5000 (0)	280 (2)	272 (2)	344 (2)	116 (2)	197 (2)	111 (2)
Cu ²⁺ (2)	2067 (0)	7682 (0)	5222 (0)	291 (2)	343 (2)	240 (1)	144 (1)	132 (1)	89 (1)
O(11)	4647 (2)	3374 (2)	4599 (3)	397 (10)	336 (10)	604 (12)	141 (8)	360 (9)	154 (9)
O(21)	3375 (2)	7403 (2)	6871 (2)	485 (11)	552 (12)	278 (9)	328 (10)	163 (8)	106 (8)
O(31)	623 (2)	7792 (2)	3528 (2)	334 (9)	480 (11)	282 (8)	186 (8)	150 (7)	130 (8)
N(11)	3684 (2)	5004 (2)	5815 (3)	357 (12)	302 (11)	432 (12)	129 (9)	243 (10)	125 (10)
N(21)	1558 (2)	8496 (2)	6790 (3)	390 (12)	540 (14)	289 (11)	268 (11)	148 (10)	113 (10)
N(31)	2689 (2)	6981 (2)	3721 (3)	371 (12)	464 (13)	318 (11)	192 (10)	175 (10)	116 (10)
C(11)	3678 (3)	2618 (3)	4659 (3)	288 (14)	310 (14)	284 (13)	106 (11)	134 (11)	103 (11)
C(12)	3504 (3)	1402 (3)	4041 (4)	410 (17)	324 (16)	414 (16)	125 (13)	232 (14)	104 (13)
C(13)	2533 (3)	536 (3)	4041 (4)	425 (17)	294 (16)	396 (16)	78 (14)	165 (14)	89 (13)
C(14)	1691 (3)	846 (3)	4694 (4)	325 (16)	361 (18)	467 (18)	35 (14)	154 (14)	171 (15)
C(15)	1835 (3)	2013 (3)	5321 (4)	279 (15)	430 (17)	363 (15)	119 (13)	149 (13)	154 (13)
C(16)	2803 (3)	2932 (3)	5303 (3)	239 (12)	317 (14)	256 (12)	91 (11)	110 (10)	104 (11)
C(17)	2878 (3)	4156 (3)	5912 (3)	288 (13)	388 (14)	297 (13)	128 (11)	160 (11)	120 (11)
C(18)	1954 (5)	4446 (4)	6642 (7)	713 (28)	442 (22)	884 (30)	231 (20)	644 (26)	207 (21)
C(21)	3866 (3)	7923 (3)	8446 (3)	362 (14)	370 (15)	312 (13)	154 (12)	141 (12)	141 (12)
C(22)	5045 (3)	7784 (3)	9431 (4)	421 (18)	580 (21)	408 (18)	250 (16)	148 (15)	189 (16)
C(23)	5624 (4)	8286 (4)	11082 (5)	474 (20)	705 (24)	436 (19)	234 (18)	47 (16)	260 (18)
C(24)	5059 (4)	8961 (4)	11846 (5)	662 (24)	705 (25)	287 (17)	250 (20)	72 (17)	124 (17)
C(25)	3921 (4)	9116 (3)	10936 (4)	627 (22)	534 (20)	295 (15)	245 (17)	152 (15)	82 (14)
C(26)	3293 (3)	8616 (3)	9233 (3)	394 (15)	359 (14)	258 (13)	153 (12)	136 (11)	93 (11)
C(27)	2096 (3)	8838 (3)	8346 (3)	437 (15)	370 (14)	315 (14)	185 (12)	200 (12)	106 (11)
C(28)	1468 (5)	9496 (4)	9256 (5)	660 (24)	642 (24)	385 (18)	399 (21)	267 (17)	118 (17)
C(31)	281 (3)	7397 (3)	1964 (3)	305 (13)	351 (14)	305 (13)	73 (11)	120 (11)	143 (11)
C(32)	- 878 (3)	7555 (3)	931 (4)	388 (17)	549 (19)	381 (16)	188 (15)	140 (14)	209 (15)
C(33)	- 1346 (4)	7158 (4)	- 720 (5)	490 (20)	731 (25)	387 (19)	192 (18)	77 (16)	273 (18)
C(34)	- 681 (4)	6577 (4)	- 1442 (4)	685 (23)	815 (26)	298 (16)	241 (20)	135 (16)	176 (17)
C(35)	438 (4)	6421 (3)	- 486 (4)	595 (22)	598 (21)	305 (16)	231 (18)	193 (16)	118 (18)
C(36)	966 (3)	6819 (3)	1225 (3)	385 (15)	388 (15)	273 (13)	118 (12)	149 (12)	113 (11)
C(37)	2190 (3)	6649 (3)	2162 (3)	418 (15)	339 (14)	320 (14)	132 (12)	215 (12)	103 (11)
C(38)	2897 (5)	6071 (4)	1305 (5)	629 (24)	578 (23)	444 (19)	307 (20)	349 (19)	153 (18)

(b) Positional parameters ($\times 10^3$) and thermal parameters ($\times 10$)

H(11)	358 (3)	574 (3)	609 (3)	36 (7)
H(12)	412 (3)	119 (3)	361 (3)	40 (7)
H(13)	240 (3)	- 35 (3)	355 (3)	46 (8)
H(14)	106 (3)	31 (3)	468 (4)	41 (8)
H(15)	126 (3)	228 (2)	575 (3)	34 (7)
H(181)	210 (3)	534 (3)	699 (4)	56 (8)
H(182)	201 (4)	406 (3)	737 (4)	77 (13)
H(183)	99 (4)	400 (4)	590 (5)	106 (15)
H(21)	86 (3)	867 (2)	641 (3)	44 (7)
H(22)	540 (3)	732 (3)	889 (3)	43 (8)
H(23)	644 (3)	818 (3)	1176 (4)	52 (8)
H(24)	550 (4)	940 (3)	1303 (4)	82 (11)
H(25)	350 (3)	964 (3)	1145 (4)	57 (9)
H(281)	70 (3)	958 (3)	853 (4)	56 (9)
H(282)	211 (3)	1030 (3)	992 (4)	68 (10)
H(283)	132 (4)	910 (3)	1005 (5)	83 (9)
H(31)	348 (3)	687 (2)	422 (3)	53 (7)
H(32)	- 131 (3)	801 (3)	145 (3)	50 (8)
H(33)	- 214 (3)	730 (3)	- 143 (4)	66 (10)
H(34)	- 103 (3)	625 (3)	- 261 (4)	76 (10)
H(35)	88 (3)	598 (3)	- 95 (4)	48 (8)
H(381)	373 (4)	605 (3)	207 (4)	77 (12)
H(382)	231 (4)	527 (4)	58 (5)	85 (13)
H(383)	311 (3)	656 (3)	58 (4)	71 (10)

Anisotropic temperature factors are calculated as

$$\exp \left\{ - \frac{1}{4} \sum_{i=1}^3 \sum_{j=1}^3 h_i h_j a_i^* a_j^* \right\}$$

Table 2. Observed and calculated structure factors

Columns are k, 10F_o and 10F_c. 'Unobserved' reflections are marked with *.

Table with multiple columns containing numerical data for structure factors. The columns are labeled with k, 10F_o, and 10F_c. The data is organized in a grid-like format with various sub-headers and numerical values. Some values are marked with an asterisk to indicate unobserved reflections.

three-dimensional Fourier syntheses. Refinement was by full-matrix least squares, minimizing $\sum w(F_o - F_c)^2$. In each cycle, those unobserved reflexions having $F_c < F_o$ were given $w=0$. The discrepancy factors

$$\text{quoted are } R = \frac{\sum ||F_o| - |F_c||}{\sum F_o} \text{ and } R_w = \left\{ \frac{\sum w(F_o - F_c)^2}{\sum w F_o^2} \right\}^{1/2}.$$

Two cycles of isotropic refinement with all $w=1$ reduced R to 0.093 and one cycle of anisotropic refinement reduced R to 0.050. A three-dimensional difference Fourier synthesis revealed the positions of the hydrogen atoms with peak heights ranging from 0.80 to 0.34 e.Å⁻³. Inclusion of the hydrogen atoms reduced R to 0.043. Weights were then assigned equal to the reciprocals of the variances of the F_o 's and refinement was continued using anisotropic thermal parameters for the non-hydrogen atoms and isotropic for the hydrogen atoms. Since the number of parameters being refined, 382, greatly exceeded the capacity of the computer, each cycle of refinement was carried out in three blocks (sub-cycles). Each block involved refinement of the scale factor, parameters of both copper atoms, and the parameters of one of the three independent ligands.

Two complete cycles gave final values of $R=0.037$ and $R_w=0.030$. During the final cycle, the mean and maximum shifts were 0.06σ and 0.60σ, respectively. Examination of the final values of the structure factors showed no evidence of secondary extinction effects.

The final values of the parameters and their estimated standard deviations are given in Table 1, and observed and calculated structure factors are in Table 2.

Discussion

The unit cell contains three molecules, one centric molecule with the copper atom Cu(1) at a center of symmetry (chosen for convenience to be at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$), and the other pair, Cu(2), in a set of general positions, as illustrated in Fig. 1. A similar situation has been reported by Kilbourn, Powell & Darbyshire (1963) for bis(benzylidiphenylphosphine)nickel(II)bromide.

The coordination of both copper atoms is fourfold, the nearest other neighbors being hydrogen atoms from adjacent molecules, Cu(1)-H(31)=3.26 Å and Cu(2)-H(13)=3.22 Å. Thus the color and the coordination agree with the suggestion of Waters & Hall

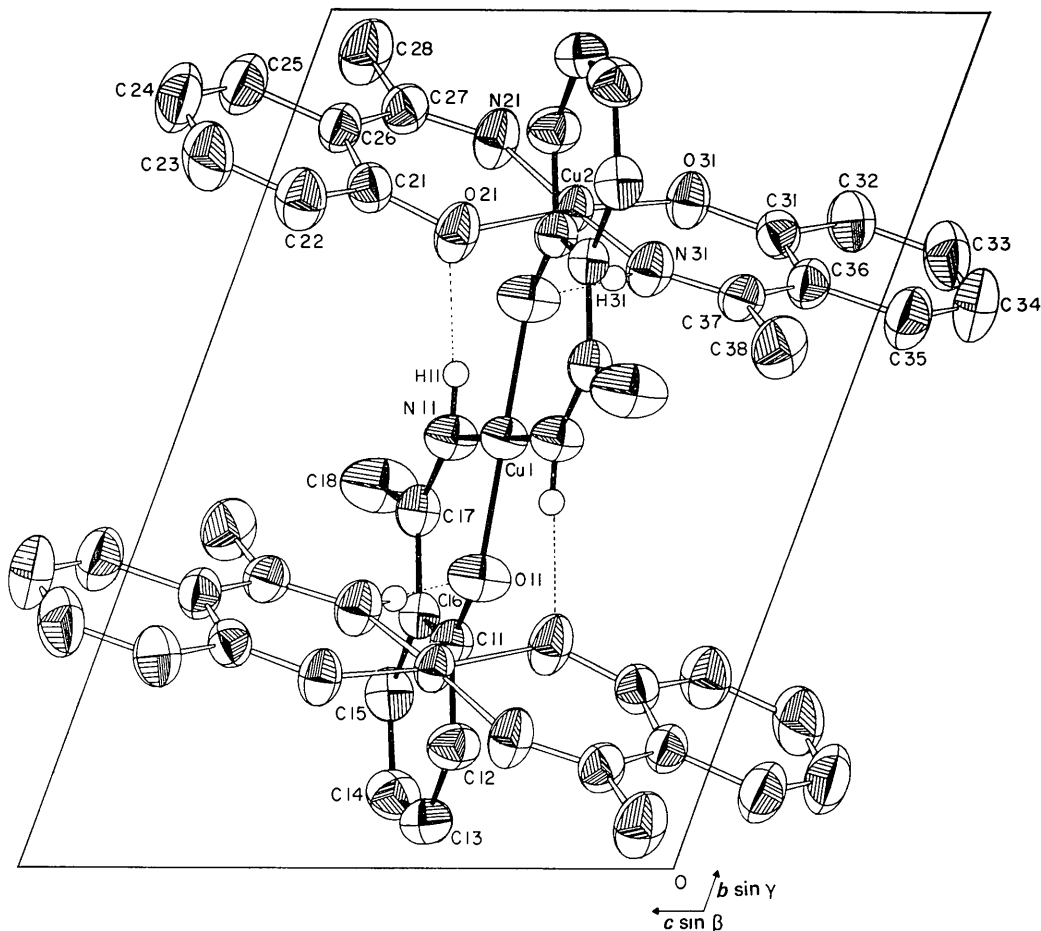


Fig. 1. Projection of structure along [100]. Suggested hydrogen bonds are shown by dashed lines.

(1959). The centrosymmetric site of Cu(1) requires its coordination configuration to be exactly planar, while that of Cu(2) is nearly planar, its distortion from planarity corresponding to a twist of 7.75° of one CuNO group with respect to the other.

Interatomic distances and angles are given in Table 3. The Cu(1)–O(11) bond distance of 1.876 (2) Å appears to be significantly short in comparison with Cu(2)–O(21), 1.894 (2) Å and Cu(2)–O(31), 1.898 (2) Å, with the Cu–O distance of 1.891 (2) Å in the isobutyl compound (Ghilardi & Lingafelter, 1970) or with the weighted mean value of 1.893 Å from 13 substituted salicylaldimine–copper(II) chelates. The three Cu–N bond distances agree among themselves, mean value 1.922 (2) Å, but are significantly shorter than the Cu–N distance of 2.003 (2) Å in the isobutyl compound or the weighted mean value of 1.969 Å from 13 substituted salicylaldimine–copper(II) chelates. This

shortening of the Cu–N distance has been also observed in the two chelates of the unsubstituted salicylaldimine with nickel(II) (Stewart & Lingafelter, 1959) and copper(II) (Baker, Hall & Waters, 1966).

The bond distances in the three ligands are in complete agreement with one another and are in satisfactory agreement with the comparable distances in the isobutyl compound (Ghilardi & Lingafelter, 1970) and agree with the mean values for salicylaldimine chelates reported by Lingafelter & Braun (1966) except for C(J6)–C(J7), which is slightly ($\sim 5\sigma$) long.

Equations for the least-squares planes defined by the benzene rings and the distances of various atoms from these planes are given in Table 4. All three of the independent chelate groups show significant distortions from planarity, but the nature and magnitude of the distortion is different for all three groups. These distortions are presumably controlled by packing effects.

Table 3. *Interatomic distances (Å) and angles (°) with estimated standard deviations*

The three corresponding values in the asymmetric unit are given under columns $J=1,2,3$.

	$J=1$	$J=2$	$J=3$
Cu—O(J1)	1.876 (2)	1.894 (2)	1.898 (2)
Cu—N(J1)	1.922 (3)	1.926 (3)	1.917 (3)
O(J1)—C(J1)	1.310 (4)	1.311 (3)	1.314 (4)
N(J1)—C(J7)	1.281 (4)	1.284 (4)	1.289 (4)
C(J1)—C(J2)	1.416 (5)	1.409 (5)	1.421 (5)
C(J2)—C(J3)	1.363 (5)	1.365 (5)	1.368 (5)
C(J3)—C(J4)	1.390 (6)	1.386 (7)	1.393 (7)
C(J4)—C(J5)	1.369 (5)	1.360 (6)	1.361 (6)
C(J5)—C(J6)	1.409 (5)	1.406 (4)	1.415 (5)
C(J6)—C(J1)	1.422 (6)	1.418 (6)	1.419 (6)
C(J6)—C(J7)	1.456 (5)	1.449 (5)	1.453 (5)
C(J7)—C(J8)	1.515 (8)	1.512 (7)	1.506 (7)
O(J1)—N(J1)	2.715 (4)	2.706 (4)	2.728 (4)
O(J1)—Cu—N(J1)	91.3 (1)	90.2 (1)	91.3 (1)
Cu—O(J1)—C(J1)	128.5 (2)	128.0 (2)	127.9 (2)
Cu—N(J1)—C(J7)	130.8 (2)	131.4 (3)	131.3 (3)
O(J1)—C(J1)—C(J6)	124.9 (3)	124.6 (3)	125.4 (3)
O(J1)—C(J1)—C(J2)	117.1 (3)	118.0 (3)	117.1 (3)
C(J2)—C(J1)—C(J6)	118.0 (3)	117.5 (3)	117.6 (3)
C(J1)—C(J2)—C(J3)	112.4 (4)	122.1 (4)	122.3 (4)
C(J2)—C(J3)—C(J4)	119.5 (4)	120.2 (4)	120.0 (4)
C(J3)—C(J4)—C(J5)	120.0 (4)	119.5 (4)	119.1 (4)
C(J4)—C(J5)—C(J6)	122.2 (4)	122.2 (4)	123.0 (4)
C(J1)—C(J6)—C(J5)	117.9 (3)	118.6 (3)	118.0 (3)
C(J1)—C(J6)—C(J7)	121.8 (3)	122.4 (3)	122.5 (3)
C(J5)—C(J6)—C(J7)	120.3 (3)	119.0 (3)	119.6 (3)
N(J1)—C(J7)—C(J6)	121.9 (3)	121.3 (3)	121.4 (3)
N(J1)—C(J7)—C(J8)	118.4 (3)	118.9 (3)	118.8 (3)
C(J6)—C(J7)—C(J8)	119.7 (3)	119.9 (3)	119.8 (3)
O(21)—Cu(2)—N(31)	89.0 (1)	O(21)—Cu(2)—O(31)	173.4 (1)
O(31)—Cu(2)—N(21)	90.0 (1)	N(21)—Cu(2)—N(31)	175.6 (1)
O(11)—Cu(1)—N(11)	88.7 (1)		
N(J1)—H(J1)	0.93 (3)	0.85 (3)	0.91 (3)
C(J2)—H(J2)	0.98 (4)	0.94 (4)	0.99 (4)
C(J3)—H(J3)	1.05 (3)	0.98 (3)	1.01 (4)
C(J4)—H(J4)	0.86 (3)	0.99 (4)	0.98 (4)
C(J5)—H(J5)	0.96 (4)	1.02 (4)	0.96 (4)
C(J8)—H(J81)	1.04 (4)	0.94 (4)	0.97 (4)
C(J8)—H(J82)	0.92 (5)	0.98 (5)	0.98 (3)
C(J8)—H(J83)	1.00 (4)	1.03 (5)	1.07 (4)

Table 4. *Least-squares planes defined by the benzene rings referred to the cell axes*

$$J=1 \quad 3.889X - 4.193Y + 7.105Z = 3.639$$

$$J=2 \quad 5.079X + 9.583Y - 4.700Z = 5.589$$

$$J=3 \quad 4.573X + 9.943Y - 3.692Z = 6.765$$

X, Y, Z are fractional coordinates

Displacements of atoms from planes

	<i>J</i> =1	<i>J</i> =2	<i>J</i> =3
C(<i>J</i> 1)	0.003 Å	-0.002 Å	-0.006 Å
C(<i>J</i> 2)	0.007	0.001	0.002
C(<i>J</i> 3)	-0.008	0.000	0.003
C(<i>J</i> 4)	-0.001	0.001	-0.004
C(<i>J</i> 5)	0.011	-0.001	-0.000
C(<i>J</i> 6)	-0.011	0.001	0.005
C(<i>J</i> 7)	-0.062	0.023	0.050
C(<i>J</i> 8)	-0.025	-0.093	0.115
O(<i>J</i> 1)	0.021	-0.010	-0.034
N(<i>J</i> 1)	-0.173	0.153	0.033
Cu	-0.239	0.368	-0.109

The molecules appear to interact through NH---O hydrogen bonds, the two unique bonds being N(11)–H(11)---O(21) (N–O distance, 3.04 Å, N–H–O angle, 176°) and N(31)–H(31)---O(11) at 1–*x*, 1–*y*, 1–*z* (N–O distance, 3.07 Å; N–H–O angle, 179°). No other intermolecular contact distances are unusual.

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Hydrogen Bond Studies. XLVII. The Crystal Structure of the Intermolecular Complex 2-Pyridone: 6-Chloro-2-hydroxypyridine

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The crystal structure of a 1:1 complex between 2-pyridone and 6-chloro-2-hydroxypyridine has been determined from three-dimensional X-ray diffraction data recorded on a Stoë-Philips two-circle Weissenberg diffractometer. The crystals are monoclinic, space group *C2/c*, with eight formula units in a cell of dimensions *a* = 10.012, *b* = 10.999, *c* = 19.840 Å, β = 105.74°. The pyridone molecule occurs in the keto form, the chlorohydroxypyridine in the enol form. The hydrogen atoms have been located from a difference synthesis, and their coordinates allowed to vary in the final cycles of refinement. The molecules are linked to form dimers by O–H...O(2.57 Å) and N–H...N(2.90 Å) hydrogen bonds. No hydrogen bonding occurs between individual dimers. Semi-empirical molecular orbital calculations for the dimer and the monomers have also been carried out, and the results of these compared.

Introduction

This work is part of a systematic investigation of hydrogen bonding in simple organic compounds which can serve as model substances for biologically important molecules. The structure of 2-pyridone (denoted by *P*) has been studied earlier by Penfold (1953), and that of 6-chloro-2-hydroxypyridine (ClP) by Kwick & Olovsson (1968).

Experimental

The melting point diagram for the binary system *P*–ClP was determined in order to investigate the existence of intermediate phases. Commercial *P* and ClP were purified by repeated recrystallization from ethanol and benzene, respectively. Small amounts of the components were weighed carefully and were then mixed. The mixtures were sealed in glass tubes and were then melted to obtain as homogenous samples as possible. The melting-point interval for each sample was determined, and the endpoints of the interval

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