

## The Crystal and Molecular Structure of Bis-(3-amino-1-phenyl-2-buten-1-ono)copper (II)

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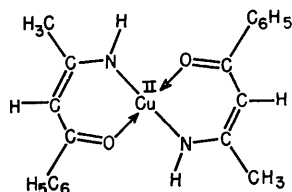
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Olive-green crystals of bis-(3-amino-1-phenyl-2-buten-1-ono)copper(II),  $(C_{10}H_{10}NO)_2Cu$ , are monoclinic with  $a = 6.456$ ,  $b = 13.437$ ,  $c = 12.440$  (Å),  $\beta = 123.31^\circ$ ,  $Z = 2$ , space group  $P2_1/c$ . They are isomorphous with red-brown crystals of the corresponding nickel(II) compound. The structure of the copper complex was determined by the heavy-atom method with use of visually estimated Cu  $K\alpha$  data. Positional and anisotropic thermal parameters for all atoms except H were refined by the full-matrix least-squares method, the final  $R$  index being 0.099 for 1312 observed reflections. The molecule has an internal center of symmetry at the Cu atom, requiring that the 4-coordination of the Cu be planar. The molecules are packed in the cell in a herringbone pattern, with no close intermolecular approaches (all  $> 3.5$  Å). The metal-chelate bond lengths are Cu–N 1.919, Cu–O 1.954 Å.

### Introduction

The inner complex chelates of divalent copper and nickel with 3-amino-1-phenyl-2-buten-1-one have been prepared by N. P. Sweeny of these Laboratories.

Archer (1963) used the trivial name 'acim' (acetylacetone-imine) to denote the ligand bis-(4-amino-3-pentene-2-one). Using the same system of nomenclature bis-(3-amino-1-phenyl-2-buten-1-one) would be 'bacim' from benzoylacetone-imine, and the chelates would be denoted Ni(bacim)<sub>2</sub> or Cu(bacim)<sub>2</sub>. One of the possible resonance forms for Cu(bacim)<sub>2</sub> is:



The Ni and Cu compounds are respectively dia- and paramagnetic and were expected to involve square-planar  $dsp^2$  coordination of the metal atoms. However, in view of the reported resolution of some closely related Cu(II) and Ni(II)  $\beta$ -ketoimines into optical isomers and the inference that these might be tetrahedral or distorted tetrahedral (Hseu, Martin & Moeller, 1963), it seemed desirable to undertake a complete X-ray structure analysis of Cu(bacim)<sub>2</sub> to determine the nature of the metal coordination.

### Experimental

Weissenberg photographs of acicular crystals of olive-green Cu(bacim)<sub>2</sub> and red-brown Ni(bacim)<sub>2</sub> were obtained using both copper and chromium radiation. The crystals are isomorphous, having similar unit cells and an almost identical distribution of intensities.

A standard Weissenberg camera (Supper) was modified in a manner similar to that described by

Herbstein (1963) to allow reflections to be recorded at high angles ( $\theta \leq 86.50^\circ$ ). Cell dimensions of the Cu complex were obtained by full-matrix least-squares refinement (Burnham, 1964) of data measured from  $0kl$  and  $h0l$  zones recorded with Cr radiation. Camera eccentricity and crystal absorption correction terms were refined along with the lattice dimensions. The camera radius was determined with use of Al ( $a_0 = 4.04100$  kX) and NaCl ( $a_0 = 5.62800$  kX) powders, converting these dimensions to Å by using the relationship  $\text{Å} \approx kX/1.00202$ . The quoted standard deviations are those obtained from the full matrix. The X-ray wavelengths used were Cr  $K\alpha_1 = 2.28962$ , Cr  $K\alpha_2 = 2.29351$  and Cr  $K\beta_1 = 2.08480$  Å (*International Tables for X-ray Crystallography*, 1962).

The measured density (mercury penetrometer method) is  $1.411$  g.cm<sup>-3</sup>, while the density calculated for two molecules per unit cell is  $1.414$  g.cm<sup>-3</sup>.

### Crystal data

Bis-(3-amino-1-phenyl-2-buten-1-ono)copper(II)

$(C_{10}H_{10}NO)_2Cu$  M.W. 383.9

Monoclinic

Olive-green acicular crystals elongated along **a**

$$a = 6.456 \pm 0.002, b = 13.437 \pm 0.002, c = 12.440 \pm 0.003 \text{ \AA}, \beta = 123.31 \pm 0.01^\circ, V = 901.8 \pm 0.7 \text{ \AA}^3$$

$$Z = 2, \rho_o = 1.411 \pm 0.01, \rho_c = 1.414 \text{ g.cm}^{-3}.$$

The  $h0l$  reflections were observed only when  $l = 2n$ , the  $0k0$  only when  $k = 2n$ . Space group  $P2_1/c$ .

Complete three-dimensional intensity data for Cu(bacim)<sub>2</sub> were visually estimated from multiple-film equi-inclination Weissenberg photographs recorded with Cu  $K\alpha$  radiation. The observed intensities from layers 0–5 about **a** and 0–11 about **b** were scaled, correlated by least-squares, and reduced to 1312 independent structure amplitudes using the FORTRAN programs of Craven (1962). The unobserved reflections were given an  $|F_o|$  value equal to 0.7 times the

local minimum observable value, giving a total of 1985 recorded reflections. The relatively large number of unobserved reflections is a result of the Cu atoms being in special positions such that they do not contribute to  $hkl$  reflections for which  $(k+l)$  is odd. The reflections 011, 100 and  $\bar{1}11$  were inaccessible. No absorption corrections were applied to the data.

### Structure determination

Since there are only two molecules in a unit cell having four general positions, the molecules must have the same point symmetry as the special positions of the cell, and the centers of symmetry of the molecules must coincide with the special positions of the cell. Thus the molecules have  $\bar{1}$  point symmetry about the copper atoms and hence must have the *trans* configuration (Gurr, 1964). The copper atoms were placed at

the special positions ( $a$ ) of the space group; *i.e.* 0,0,0 and  $0, \frac{1}{2}, \frac{1}{2}$ .

The structure was determined from the  $0kl$  and  $h0l$  projections, by the heavy-atom method. Because of the special positions of the copper atoms the  $0kl$  Fourier synthesis based only on the heavy-atom signs showed a false mirror symmetry. By comparing the two projections the correct arrangement of atoms was found, and the false symmetry was eliminated by including the lighter atoms in the structure-factor calculation. Programs written for the IBM 1620 computer by Shiono, Hall & Chu (1963) were used for these calculations. Atomic scattering factors used throughout this study were taken from *International Tables for X-ray Crystallography* (1962).

Using the coordinates obtained from the two-dimensional work, a three-dimensional structure-factor calculation gave an agreement  $R=0.48$  for 1858 reflections. The three-dimensional electron density synthesis based on these calculated signs is shown in Fig. 1. Approximate coordinates obtained from this Fourier synthesis were used in the first cycle of refinement.

### Refinement

A total of eight cycles of structure-factor and least-squares calculations were made. The first six cycles (minimizing  $\sum w(|F_o| - |F_c|)^2$ ) made use of the programs of Jacobson (1963) and his coworkers for the Control Data Corporation 1604 computer. The weighting scheme of Hughes (1941) was used, with  $4|F|_{\min} = 4.0$ , and full calculated shifts were applied. Reflections for which  $|F_c| < 0.7$  were excluded from the least-squares calculation, as were those for which  $|F_o| > 5|F_c|$  and  $|F_o| < 0.01|F_c|$ .

In the first three least-squares cycles the copper atom was allowed anisotropic thermal vibration parameters, while the light atoms were kept isotropic. At this stage ( $R=0.15$ ) a three-dimensional difference

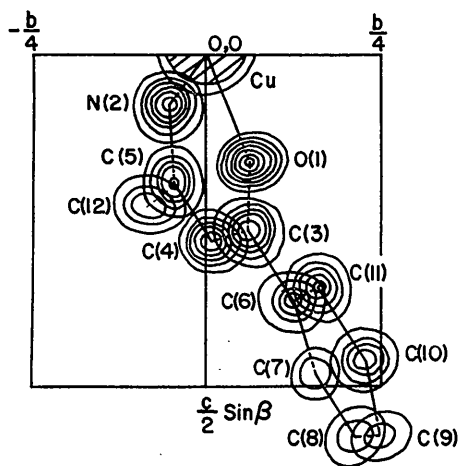


Fig. 1. First 3-D Fourier synthesis for  $\text{Cu}(\text{bacim})_2$ , represented sectionally with respect to the  $a$  axis. One half of the molecule, *i.e.* the asymmetric unit, is shown.

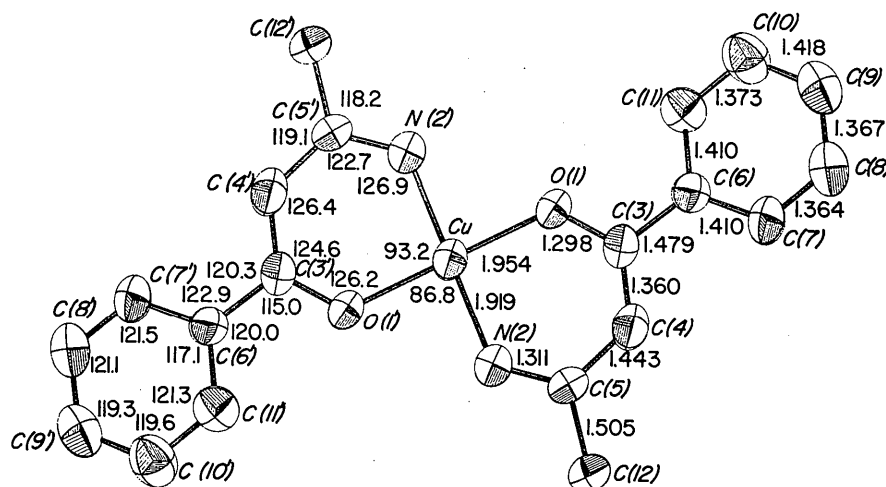


Fig. 2. Bond lengths, bond angles and ellipsoids of thermal vibration for  $\text{Cu}(\text{bacim})_2$ .

Fourier synthesis was calculated using only the reflections with  $\sin \theta < 0.40$ . Regions of positive electron density were found at or near the positions expected for hydrogen atoms. Hydrogen atoms were included in all subsequent structure factor calculations, but were not refined. The coordinates of the hydrogen atoms were obtained graphically by assuming 'normal' values for the C-H (1.08 Å) and N-H (0.99 Å) bond lengths and angles, and the thermal parameters were assumed isotropic and equal to those of the atoms to which the hydrogens were attached.

After a fourth least-squares cycle, a 3-D difference Fourier synthesis was calculated with use of all reflections. This showed the light atoms to be vibrating anisotropically. In the fifth and sixth least-squares cycles all atoms (except hydrogen) were allowed anisotropic thermal parameters. The hydrogen atom parameters were held constant. An empirical correction for secondary extinction of the form  $F_{\text{corrected}} = F_{\text{obs}}(1 + \beta F_{\text{obs}})$  was applied to the six largest structure amplitudes.

In the seventh and eighth least-squares cycles only the observed reflections were used and allowance was made for dispersion by the Cu atom, using the FORTRAN least-squares program *UMLSTSQ* [minimizing  $\sum w(F_o^2 - F_c^2)^2$ ] (Finger, 1965). The final value of  $R = [\sum (|F_o| - |F_c|) / \sum |F_o|]$  is 0.099 for the 1312 observed reflections. Table 1 gives the values of  $F_o$  and  $F_c$  at this stage. Table 2 lists the final atomic coordinates, while Table 3 gives the thermal vibration parameters. Thermal ellipsoid data are listed in Table 4 and depicted in Fig. 2.

Accuracy

An analysis of the errors of this determination was made by using the FORTRAN program *UMBADTEA* (Finger, 1966), which uses the full matrix to calculate estimated standard deviations. Molecular dimensions and their standard deviations are shown in Table 5. The standard deviation of the electron density was calculated to be 0.25 e.Å<sup>3</sup>. After the sixth least-squares

Table 1.  $10|F_o|$ ,  $10|F_c|$  and phase angle (different from 0° or 180° because of anomalous dispersion) for Cu (bacim)<sub>2</sub>. The atomic parameters used are those given in Table 2 and Table 3. An \* denotes the six reflections corrected for extinction.

6 58 52 2.0	7 21 26 0.3	8 71 92 2.0	9 34 45 4.8	10 83 68 4.3	11 83 68 4.3	12 281 268 17.0	13 92 56 2.9	14 190 170 2.2	15 100 100 2.2	16 82 45 2.0	17 92 56 2.9	18 100 100 2.2	19 92 56 2.9	20 82 45 2.0	21 92 56 2.9	22 100 100 2.2	23 92 56 2.9	24 82 45 2.0	25 92 56 2.9	26 100 100 2.2	27 92 56 2.9	28 82 45 2.0	29 92 56 2.9	30 100 100 2.2	31 92 56 2.9	32 82 45 2.0	33 92 56 2.9	34 100 100 2.2	35 92 56 2.9	36 82 45 2.0	37 92 56 2.9	38 100 100 2.2	39 92 56 2.9	40 82 45 2.0	41 92 56 2.9	42 100 100 2.2	43 92 56 2.9	44 82 45 2.0	45 92 56 2.9	46 100 100 2.2	47 92 56 2.9	48 82 45 2.0	49 92 56 2.9	50 100 100 2.2	51 92 56 2.9	52 82 45 2.0	53 92 56 2.9	54 100 100 2.2	55 92 56 2.9	56 82 45 2.0	57 92 56 2.9	58 100 100 2.2	59 92 56 2.9	60 82 45 2.0	61 92 56 2.9	62 100 100 2.2	63 92 56 2.9	64 82 45 2.0	65 92 56 2.9	66 100 100 2.2	67 92 56 2.9	68 82 45 2.0	69 92 56 2.9	70 100 100 2.2	71 92 56 2.9	72 82 45 2.0	73 92 56 2.9	74 100 100 2.2	75 92 56 2.9	76 82 45 2.0	77 92 56 2.9	78 100 100 2.2	79 92 56 2.9	80 82 45 2.0	81 92 56 2.9	82 100 100 2.2	83 92 56 2.9	84 82 45 2.0	85 92 56 2.9	86 100 100 2.2	87 92 56 2.9	88 82 45 2.0	89 92 56 2.9	90 100 100 2.2	91 92 56 2.9	92 82 45 2.0	93 92 56 2.9	94 100 100 2.2	95 92 56 2.9	96 82 45 2.0	97 92 56 2.9	98 100 100 2.2	99 92 56 2.9	100 82 45 2.0	101 92 56 2.9	102 100 100 2.2	103 92 56 2.9	104 82 45 2.0	105 92 56 2.9	106 100 100 2.2	107 92 56 2.9	108 82 45 2.0	109 92 56 2.9	110 100 100 2.2	111 92 56 2.9	112 82 45 2.0	113 92 56 2.9	114 100 100 2.2	115 92 56 2.9	116 82 45 2.0	117 92 56 2.9	118 100 100 2.2	119 92 56 2.9	120 82 45 2.0	121 92 56 2.9	122 100 100 2.2	123 92 56 2.9	124 82 45 2.0	125 92 56 2.9	126 100 100 2.2	127 92 56 2.9	128 82 45 2.0	129 92 56 2.9	130 100 100 2.2	131 92 56 2.9	132 82 45 2.0	133 92 56 2.9	134 100 100 2.2	135 92 56 2.9	136 82 45 2.0	137 92 56 2.9	138 100 100 2.2	139 92 56 2.9	140 82 45 2.0	141 92 56 2.9	142 100 100 2.2	143 92 56 2.9	144 82 45 2.0	145 92 56 2.9	146 100 100 2.2	147 92 56 2.9	148 82 45 2.0	149 92 56 2.9	150 100 100 2.2	151 92 56 2.9	152 82 45 2.0	153 92 56 2.9	154 100 100 2.2	155 92 56 2.9	156 82 45 2.0	157 92 56 2.9	158 100 100 2.2	159 92 56 2.9	160 82 45 2.0	161 92 56 2.9	162 100 100 2.2	163 92 56 2.9	164 82 45 2.0	165 92 56 2.9	166 100 100 2.2	167 92 56 2.9	168 82 45 2.0	169 92 56 2.9	170 100 100 2.2	171 92 56 2.9	172 82 45 2.0	173 92 56 2.9	174 100 100 2.2	175 92 56 2.9	176 82 45 2.0	177 92 56 2.9	178 100 100 2.2	179 92 56 2.9	180 82 45 2.0	181 92 56 2.9	182 100 100 2.2	183 92 56 2.9	184 82 45 2.0	185 92 56 2.9	186 100 100 2.2	187 92 56 2.9	188 82 45 2.0	189 92 56 2.9	190 100 100 2.2	191 92 56 2.9	192 82 45 2.0	193 92 56 2.9	194 100 100 2.2	195 92 56 2.9	196 82 45 2.0	197 92 56 2.9	198 100 100 2.2	199 92 56 2.9	200 82 45 2.0	201 92 56 2.9	202 100 100 2.2	203 92 56 2.9	204 82 45 2.0	205 92 56 2.9	206 100 100 2.2	207 92 56 2.9	208 82 45 2.0	209 92 56 2.9	210 100 100 2.2	211 92 56 2.9	212 82 45 2.0	213 92 56 2.9	214 100 100 2.2	215 92 56 2.9	216 82 45 2.0	217 92 56 2.9	218 100 100 2.2	219 92 56 2.9	220 82 45 2.0	221 92 56 2.9	222 100 100 2.2	223 92 56 2.9	224 82 45 2.0	225 92 56 2.9	226 100 100 2.2	227 92 56 2.9	228 82 45 2.0	229 92 56 2.9	230 100 100 2.2	231 92 56 2.9	232 82 45 2.0	233 92 56 2.9	234 100 100 2.2	235 92 56 2.9	236 82 45 2.0	237 92 56 2.9	238 100 100 2.2	239 92 56 2.9	240 82 45 2.0	241 92 56 2.9	242 100 100 2.2	243 92 56 2.9	244 82 45 2.0	245 92 56 2.9	246 100 100 2.2	247 92 56 2.9	248 82 45 2.0	249 92 56 2.9	250 100 100 2.2	251 92 56 2.9	252 82 45 2.0	253 92 56 2.9	254 100 100 2.2	255 92 56 2.9	256 82 45 2.0	257 92 56 2.9	258 100 100 2.2	259 92 56 2.9	260 82 45 2.0	261 92 56 2.9	262 100 100 2.2	263 92 56 2.9	264 82 45 2.0	265 92 56 2.9	266 100 100 2.2	267 92 56 2.9	268 82 45 2.0	269 92 56 2.9	270 100 100 2.2	271 92 56 2.9	272 82 45 2.0	273 92 56 2.9	274 100 100 2.2	275 92 56 2.9	276 82 45 2.0	277 92 56 2.9	278 100 100 2.2	279 92 56 2.9	280 82 45 2.0	281 92 56 2.9	282 100 100 2.2	283 92 56 2.9	284 82 45 2.0	285 92 56 2.9	286 100 100 2.2	287 92 56 2.9	288 82 45 2.0	289 92 56 2.9	290 100 100 2.2	291 92 56 2.9	292 82 45 2.0	293 92 56 2.9	294 100 100 2.2	295 92 56 2.9	296 82 45 2.0	297 92 56 2.9	298 100 100 2.2	299 92 56 2.9	300 82 45 2.0	301 92 56 2.9	302 100 100 2.2	303 92 56 2.9	304 82 45 2.0	305 92 56 2.9	306 100 100 2.2	307 92 56 2.9	308 82 45 2.0	309 92 56 2.9	310 100 100 2.2	311 92 56 2.9	312 82 45 2.0	313 92 56 2.9	314 100 100 2.2	315 92 56 2.9	316 82 45 2.0	317 92 56 2.9	318 100 100 2.2	319 92 56 2.9	320 82 45 2.0	321 92 56 2.9	322 100 100 2.2	323 92 56 2.9	324 82 45 2.0	325 92 56 2.9	326 100 100 2.2	327 92 56 2.9	328 82 45 2.0	329 92 56 2.9	330 100 100 2.2	331 92 56 2.9	332 82 45 2.0	333 92 56 2.9	334 100 100 2.2	335 92 56 2.9	336 82 45 2.0	337 92 56 2.9	338 100 100 2.2	339 92 56 2.9	340 82 45 2.0	341 92 56 2.9	342 100 100 2.2	343 92 56 2.9	344 82 45 2.0	345 92 56 2.9	346 100 100 2.2	347 92 56 2.9	348 82 45 2.0	349 92 56 2.9	350 100 100 2.2	351 92 56 2.9	352 82 45 2.0	353 92 56 2.9	354 100 100 2.2	355 92 56 2.9	356 82 45 2.0	357 92 56 2.9	358 100 100 2.2	359 92 56 2.9	360 82 45 2.0	361 92 56 2.9	362 100 100 2.2	363 92 56 2.9	364 82 45 2.0	365 92 56 2.9	366 100 100 2.2	367 92 56 2.9	368 82 45 2.0	369 92 56 2.9	370 100 100 2.2	371 92 56 2.9	372 82 45 2.0	373 92 56 2.9	374 100 100 2.2	375 92 56 2.9	376 82 45 2.0	377 92 56 2.9	378 100 100 2.2	379 92 56 2.9	380 82 45 2.0	381 92 56 2.9	382 100 100 2.2	383 92 56 2.9	384 82 45 2.0	385 92 56 2.9	386 100 100 2.2	387 92 56 2.9	388 82 45 2.0	389 92 56 2.9	390 100 100 2.2	391 92 56 2.9	392 82 45 2.0	393 92 56 2.9	394 100 100 2.2	395 92 56 2.9	396 82 45 2.0	397 92 56 2.9	398 100 100 2.2	399 92 56 2.9	400 82 45 2.0	401 92 56 2.9	402 100 100 2.2	403 92 56 2.9	404 82 45 2.0	405 92 56 2.9	406 100 100 2.2	407 92 56 2.9	408 82 45 2.0	409 92 56 2.9	410 100 100 2.2	411 92 56 2.9	412 82 45 2.0	413 92 56 2.9	414 100 100 2.2	415 92 56 2.9	416 82 45 2.0	417 92 56 2.9	418 100 100 2.2	419 92 56 2.9	420 82 45 2.0	421 92 56 2.9	422 100 100 2.2	423 92 56 2.9	424 82 45 2.0	425 92 56 2.9	426 100 100 2.2	427 92 56 2.9	428 82 45 2.0	429 92 56 2.9	430 100 100 2.2	431 92 56 2.9	432 82 45 2.0	433 92 56 2.9	434 100 100 2.2	435 92 56 2.9	436 82 45 2.0	437 92 56 2.9	438 100 100 2.2	439 92 56 2.9	440 82 45 2.0	441 92 56 2.9	442 100 100 2.2	443 92 56 2.9	444 82 45 2.0	445 92 56 2.9	446 100 100 2.2	447 92 56 2.9	448 82 45 2.0	449 92 56 2.9	450 100 100 2.2	451 92 56 2.9	452 82 45 2.0	453 92 56 2.9	454 100 100 2.2	455 92 56 2.9	456 82 45 2.0	457 92 56 2.9	458 100 100 2.2	459 92 56 2.9	460 82 45 2.0	461 92 56 2.9	462 100 100 2.2	463 92 56 2.9	464 82 45 2.0	465 92 56 2.9	466 100 100 2.2	467 92 56 2.9	468 82 45 2.0	469 92 56 2.9	470 100 100 2.2	471 92 56 2.9	472 82 45 2.0	473 92 56 2.9	474 100 100 2.2	475 92 56 2.9	476 82 45 2.0	477 92 56 2.9	478 100 100 2.2	479 92 56 2.9	480 82 45 2.0	481 92 56 2.9	482 100 100 2.2	483 92 56 2.9	484 82 45 2.0	485 92 56 2.9	486 100 100 2.2	487 92 56 2.9	488 82 45 2.0	489 92 56 2.9	490 100 100 2.2	491 92 56 2.9	492 82 45 2.0	493 92 56 2.9	494 100 100 2.2	495 92 56 2.9	496 82 45 2.0	497 92 56 2.9	498 100 100 2.2	499 92 56 2.9	500 82 45 2.0	501 92 56 2.9	502 100 100 2.2	503 92 56 2.9	504 82 45 2.0	505 92 56 2.9	506 100 100 2.2	507 92 56 2.9	508 82 45 2.0	509 92 56 2.9	510 100 100 2.2	511 92 56 2.9	512 82 45 2.0	513 92 56 2.9	514 100 100 2.2	515 92 56 2.9	516 82 45 2.0	517 92 56 2.9	518 100 100 2.2	519 92 56 2.9	520 82 45 2.0	521 92 56 2.9	522 100 100 2.2	523 92 56 2.9	524 82 45 2.0	525 92 56 2.9	526 100 100 2.2	527 92 56 2.9	528 82 45 2.0	529 92 56 2.9	530 100 100 2.2	531 92 56 2.9	532 82 45 2.0	533 92 56 2.9	534 100 100 2.2	535 92 56 2.9	536 82 45 2.0	537 92 56 2.9	538 100 100 2.2	539 92 56 2.9	540 82 45 2.0	541 92 56 2.9	542 100 100 2.2	543 92 56 2.9	544 82 45 2.0	545 92 56 2.9	546 100 100 2.2	547 92 56 2.9	548 82 45 2.0	549 92 56 2.9	550 100 100 2.2	551 92 56 2.9	552 82 45 2.0	553 92 56 2.9	554 100 100 2.2	555 92 56 2.9	556 82 45 2.0	557 92 56 2.9	558 100 100 2.2	559 92 56 2.9	560 82 45 2.0	561 92 56 2.9	562 100 100 2.2	563 92 56 2.9	564 82 45 2.0	565 92 56 2.9	566 100 100 2.2	567 92 56 2.9	568 82 45 2.0	569 92 56 2.9	570 100 100 2.2	571 92 56 2.9	572 82 45 2.0	573 92 56 2.9	574 100 100 2.2	575 92 56 2.9	576 82 45 2.0	577 92 56 2.9	578 100 100 2.2	579 92 56 2.9	580 82 45 2.0	581 92 56 2.9	582 100 100 2.2	583 92 56 2.9	584 82 45 2.0	585 92 56 2.9	586 100 100 2.2	587 92 56 2.9	588 82 45 2.0	589 92 56 2.9	590 100 100 2.2	591 92 56 2.9	592 82 45 2.0	593 92 56 2.9	594 100 100 2.2	595 92 56 2.9	596 82 45 2.0	5
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Table 2. Fractional atomic coordinates in  $\text{Cu}(\text{bacim})_2$ 

	<i>x</i>	<i>y</i>	<i>z</i>		<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> †
O(1)	0.0963 (9)	0.0671 (4)	0.1604 (5)	H(N2*)	0.346	-0.099	0.008	3.6
N(2)	0.3229 (12)	-0.0568 (5)	0.0713 (6)	H(C4)	0.685	0.010	0.386	3.4
C(3)	0.3124 (13)	0.0632 (4)	0.2677 (6)	H(C12-1)	Not located			
C(4)	0.5098 (15)	0.0116 (5)	0.2851 (7)	H(C12-2)	0.767	-0.170	0.270	3.8
C(5)	0.5158 (12)	-0.0470 (5)	0.1896 (7)	H(C12-3)	0.760	-0.120	0.146	3.8
C(6)	0.3365 (13)	0.1228 (4)	0.3739 (6)	H(C7)	0.739	0.129	0.490	3.9
C(7)	0.5676 (15)	0.1524 (6)	0.4821 (8)	H(C8)	0.764	0.234	0.648	5.1
C(8)	0.5833 (19)	0.2123 (7)	0.5748 (9)	H(C9)	0.390	0.293	0.639	4.4
C(9)	0.3749 (21)	0.2486 (6)	0.5641 (8)	H(C10)	-0.019	0.240	0.462	4.6
C(10)	0.1388 (20)	0.2163 (6)	0.4607 (9)	H(C11)	-0.051	0.133	0.290	3.4
C(11)	0.1225 (14)	0.1555 (5)	0.3676 (7)					
C(12)	0.7535 (14)	-0.0976 (5)	0.2254 (7)					
Cu	0	0	0					

\* The symbol in parenthesis denotes the atom to which the hydrogen atom is bonded.

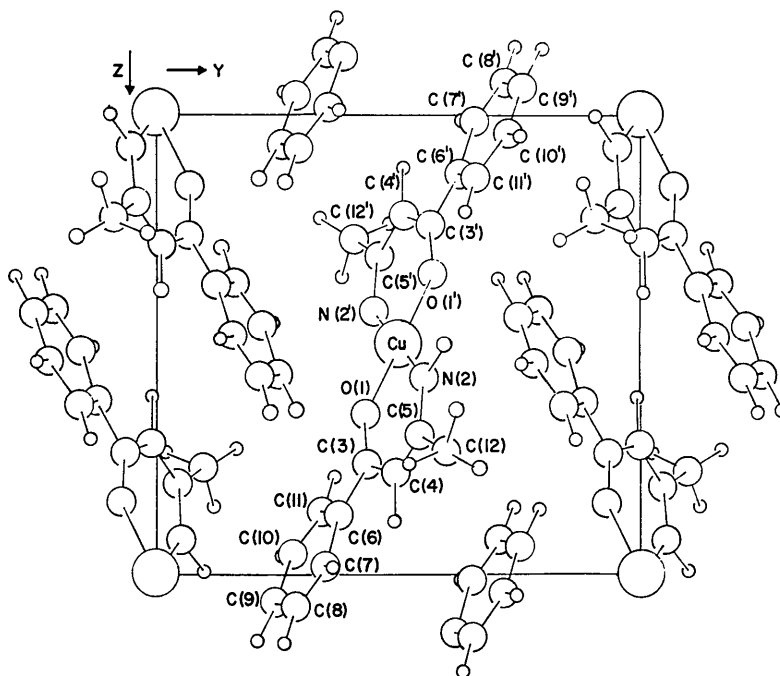
† Assumed value of isotropic temperature factor.

Table 3. Anisotropic thermal parameters in  $\text{Cu}(\text{bacim})_2$ 

The temperature factor expression used was:

$$\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$$

	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
O(1)	0.0241 (16)	0.0084 (3)	0.0074 (4)	0.0015 (5)	0.0049 (7)	-0.0006 (3)
N(2)	0.0301 (22)	0.0089 (4)	0.0086 (6)	0.0015 (7)	0.0060 (10)	-0.0014 (4)
C(3)	0.0299 (22)	0.0061 (3)	0.0070 (5)	-0.0012 (7)	0.0063 (9)	-0.0002 (3)
C(4)	0.0334 (23)	0.0083 (4)	0.0074 (5)	-0.0003 (9)	0.0056 (9)	-0.0004 (4)
C(5)	0.0248 (20)	0.0066 (4)	0.0092 (6)	0.0019 (7)	0.0072 (9)	0.0001 (4)
C(6)	0.0316 (23)	0.0061 (3)	0.0079 (5)	0.0015 (7)	0.0085 (10)	0.0003 (3)
C(7)	0.0323 (26)	0.0095 (5)	0.0095 (7)	-0.0029 (10)	0.0061 (12)	-0.0030 (5)
C(8)	0.0494 (42)	0.0106 (6)	0.0103 (8)	-0.0016 (13)	0.0089 (16)	-0.0032 (6)
C(9)	0.0612 (44)	0.0080 (4)	0.0107 (8)	-0.0010 (12)	0.0153 (17)	-0.0010 (5)
C(10)	0.0549 (41)	0.0092 (5)	0.0131 (10)	-0.0038 (12)	0.0182 (18)	-0.0017 (6)
C(11)	0.0411 (29)	0.0064 (3)	0.0115 (7)	-0.0020 (8)	0.0145 (13)	0.0000 (4)
C(12)	0.0298 (23)	0.0073 (4)	0.0105 (7)	0.0034 (8)	0.0072 (11)	-0.0006 (4)
Cu	0.0261 (5)	0.0083 (1)	0.0070 (1)	0.0001 (2)	0.0055 (2)	-0.0010 (1)

Fig. 3. Packing of molecules of  $\text{Cu}(\text{bacim})_2$  in the crystal. The view shown is a parallel projection down the *a* axis.

atomic distances of approach less than 4.0 Å were calculated with the FORTRAN program of Shiono *et al.* (1963). No atoms of one molecule (except hydrogen) approach within 3.5 Å of the atoms (except hydrogen) of any surrounding molecules. Close copper-copper approach is not observed. Hydrogen bonding is not possible. The molecules pack in a herringbone pattern (Fig. 3) that fills space very effectively (Kitaigorodskii, 1957). Van der Waals forces are the only type of intermolecular interaction that stabilize the structure. One might speculate that the deviations from planarity

observed in similar molecules are the result of molecular interactions in the crystalline state, rather than being an inherent property of the molecules.

The benzene rings are inclined at an angle of 18.1° to the least-squares plane through the copper atom and both chelate rings.

#### Anisotropic thermal motion

An attempt was made to analyze the thermal motion in terms of rigid-body libration and translation, using

Table 4. Principal axes of thermal ellipsoids for Cu (bacim)<sub>2</sub>

	<i>B</i> *	<i>U</i> †	$\theta_a$ ‡	$\theta_b$	$\theta_c$		<i>B</i>	<i>U</i>	$\theta_a$	$\theta_b$	$\theta_c$
O(1)	4.30	0.181 0.224 0.283	52 43 108	93 110 159	71 153 72	C(8)	6.37	0.215 0.291 0.333	86 156 114	67 67 146	44 65 57
N(2)	4.87	0.201 0.232 0.301	60 41 115	87 121 149	63 139 62	C(9)	5.73	0.226 0.274 0.303	110 76 25	72 19 97	21 109 100
C(3)	3.85	0.192 0.225 0.242	73 37 122	82 59 32	51 135 71	C(10)	5.81	0.231 0.262 0.314	140 118 64	89 135 135	17 101 77
C(4)	4.74	0.195 0.256 0.276	73 18 95	86 96 173	51 140 83	C(11)	4.51	0.205 0.246 0.262	148 80 60	116 141 118	43 127 71
C(5)	4.01	0.186 0.233 0.251	33 68 114	106 112 153	94 156 66	C(12)	4.73	0.196 0.239 0.291	39 83 127	115 126 133	92 138 48
C(6)	3.85	0.207 0.212 0.242	83 26 65	103 113 26	42 132 96	Cu	4.24	0.186 0.220 0.279	65 27 101	84 105 164	58 144 74
C(7)	5.33	0.192 0.260 0.313	65 28 102	70 111 150	62 137 60						

\* Value of equivalent isotropic temperature factor.

† r.m.s. amplitudes of vibration (Å) along the minor, semi-major and major directions respectively of the vibration ellipsoid.

‡ Angles of inclination (°) of the corresponding ellipsoid axes to the *a*, *b* and *c* crystallographic axes respectively.

Table 5. Molecular dimensions in Cu(bacim)<sub>2</sub>

Bond distances			Bond angles			
Cu	O(1)	1.954 (5*) Å	O(1)	Cu	N(2)†	93.2 (3*)°
Cu	N(2)	1.919 (7)	O(1)	Cu	N(2)	86.8 (3)
O(1)	C(3)	1.298 (8)	Cu	O(1)	C(3)	126.2 (5)
N(2)	C(5)	1.311 (9)	Cu	N(2)	C(5)	126.9 (6)
C(3)	C(4)	1.360 (10)	O(1)	C(3)	C(4)	124.6 (7)
C(4)	C(5)	1.443 (10)	N(2)	C(5)	C(4)	122.7 (7)
C(5)	C(12)	1.505 (9)	C(3)	C(4)	C(5)	126.4 (7)
C(3)	C(5)	1.379 (8)	C(4)	C(5)	C(12)	119.1 (7)
C(6)	C(7)	1.410 (10)	N(2)	C(5)	C(12)	118.2 (7)
C(7)	C(8)	1.364 (11)	O(1)	C(3)	C(6)	115.0 (7)
C(8)	C(9)	1.367 (14)	C(4)	C(3)	C(6)	120.3 (7)
C(9)	C(10)	1.418 (13)	C(6)	C(7)	C(8)	121.5 (9)
C(10)	C(11)	1.373 (10)	C(7)	C(8)	C(9)	121.1 (10)
C(6)	C(11)	1.410 (10)	C(8)	C(9)	C(10)	119.3 (8)
			C(9)	C(10)	C(11)	119.6 (10)
			C(10)	C(11)	C(6)	121.3 (8)
			C(11)	C(6)	C(7)	117.1 (7)

\* Figures in parentheses are the estimated standard deviations and refer to the last decimal position.

† 'Bite' angle of the chelate ring.

Table 6. *Least-squares planes in Cu(bacim)<sub>2</sub>*

Phenyl ring

$$0.30539X + 0.80428Y - 0.59351Z = -0.80956^*$$

Atoms forming the plane:

C(6)	0.020†	C(9)	0.032
C(7)	-0.006	C(10)	-0.017
C(8)	-0.006	C(11)	-0.009

Plane of Cu and both chelate rings

$$0.51516X + 0.81079Y - 0.51752Z = 0^*$$

Atoms forming the plane		Deviation of other atoms from the plane	
Cu	0.000†	C(6)	0.037
O(1), O(1')	0.010	C(7)	0.432
N(2), N(2')	0.011	C(8)	0.532
C(3), C(3')	-0.001	C(9)	0.293
C(4), C(4')	-0.007	C(10)	-0.180
C(5), C(5')	-0.001	C(11)	-0.288
		C(12)	0.020

\* The *X*, *Y* and *Z* coordinates refer to the crystal axial system, the units being Å.

† Deviation from the best plane (Å) of those atoms used in the calculation of the plane equation. For the second plane the sign of the deviation applies to the unprimed atoms (coordinates given in Table 2).

the FORTRAN program of Gantzel, Coulter & Trueblood (1961). Assuming the centroid of libration of the phenyl ring to be at C(3), its point of attachment to the chelate ring, gives reasonably good agreement between measured and calculated mean square amplitudes of vibration (r.m.s.  $\Delta U^2 = 0.0059 \text{ \AA}^2$ ). Agreement is worse if the centroid is assumed to be at the center of the phenyl ring (r.m.s.  $\Delta U^2 = 0.0102 \text{ \AA}^2$ ). If the chelate ring is assumed to be a rigid body librating around a centroid at the copper atom the agreement is good (r.m.s.  $\Delta U^2 = 0.0043 \text{ \AA}^2$ ).

The above leads one to picture the thermal motion as being comprised of motion of the two rigid chelate

rings about their center of gravity at the copper atom, and the independent motion of the rigid phenyl rings 'flapping' about the point at which they are attached to the chelate rings. However, in view of the relatively low accuracy of this determination, no strong emphasis should be placed on this description.

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## References

- ARCHER, R. D. (1963). *Inorg. Chem.* **2**, 292.  
 BARCLAY, G. A., HARRIS, C. M., HOSKINS, B. F. & KOKOT, E. (1961). *Proc. Chem. Soc.* p. 264.  
 BEVAN, J. A., GRADDON, D. P. & MCCONNELL, J. F. (1963). *Nature, Lond.* **199**, 373.  
 BRYAN, R. F., POLJAK, R. J. & TOMITA, K. (1961). *Acta Cryst.* **14**, 1125.  
 BURNHAM, C. W. (1964). *Ann. Rep. of the Director, Geophys. Lab., Carnegie Inst.*, p. 200.  
 CRAVEN, B. M. (1962). Private communication.  
 FACKLER, J. P. & COTTON, F. A. (1963). *Inorg. Chem.* **2**, 102.  
 FINGER, L. W. (1965). Private communication.  
 FINGER, L. W. (1966). Private communication.  
 GANTZEL, P. K., COULTER, C. L. & TRUEBLOOD, K. N. (1961). *World List of Crystallographic Computer Programs*, Prog. 232.  
 GURR, G. E. (1964). *Inorg. Chem.* **3**, 614.  
 HALL, D., MCKINNON, A. J., WATERS, J. M. & WATERS, T. N. (1964). *Nature, Lond.* **201**, 607.  
 HALL, D., RAE, A. D. & WATERS, T. N. (1963). *J. Chem. Soc.* p. 5897.  
 HALL, D. & WATERS, T. N. (1960). *J. Chem. Soc.* p. 2644.  
 HERBSTEIN, F. H. (1963). *Acta Cryst.* **16**, 255.  
 HOLM, R. H. & COTTON, F. A. (1958). *J. Amer. Chem. Soc.* **80**, 5658.

Table 7. *Previously reported Cu-O and Cu-N bond lengths (Å)*

	Cu-O	Cu-N
Bis-( $\beta$ -aminobutyrate)copper(II) dihydrate Bryan, Poljak & Tomita (1961)	2.00	1.99
<i>N,N'</i> -Ethylenebis(acetylacetonimine)copper(II) Hall, Rae & Waters (1963)	1.96	1.95
<i>N,N'</i> -Disalicylidene-1,2-diaminecopper monohydrate Llewellyn & Waters (1960)	1.92	1.99
<i>N,N'</i> -Disalicylidene-1,2-diaminecopper monohydrate Llewellyn & Waters (1960)	1.94	1.78
Bis-(8-hydroxyquinolinato)copper(II) Bevan, Graddon & McConnell (1963)	1.88	1.95
<i>N,N'</i> -Disalicylidene-ethylenediaminecopper Hall & Waters (1960)	2.00	1.94
<i>N,N'</i> -Disalicylidene-ethylenediaminecopper Hall & Waters (1960)	2.03	2.08
<i>Cu</i> <sub>11</sub> <i>H</i> <sub>11</sub> <i>NO</i> <sub>2</sub> <i>Cu</i> Barclay, Harris, Hoskins & Kokot (1961)	1.91	1.94
	1.97	2.00
	1.99	1.97
	1.91	
	2.02	
	1.91	
	1.89	
$\alpha$ -Copper 8-hydroxyquinolate Hoy & Moriss (1967)	1.939	1.984
$\beta$ -Copper 8-hydroxyquinolate Palenik (1964)	1.935	1.974
Average values	1.95	1.96 Å

- HOY, R. C. & MORRIS, R. H. (1967). *Acta Cryst.* **22**, 476.  
 HSEU, T., MARTIN, D. F. & MOELLER, T. (1963). *Inorg. Chem.* **2**, 587.  
 HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737.  
*International Tables for X-ray Crystallography* (1962). Vol. II. Birmingham: Kynoch Press.  
 JACOBSON, R. A. (1963). Technical Reports 1-7, Frick Chemical Lab., Princeton Univ.  
 KITAIGORODSKII, A. I. (1957). *Organic Chemical Crystallography*. New York: Consultants Bureau.  
 LLEWELLYN, F. J. & WATERS, T. N. (1960). *J. Chem. Soc.* p. 2639.  
 PALENIK, G. J. (1964). *Acta Cryst.* **17**, 687.  
 SHONO, R., HALL, D. & CHU, S. C. (1963). Technical Report 43, The Crystallography Lab., Univ. of Pittsburgh.

*Acta Cryst.* (1968). **B24**, 1518

## The Crystal Structure of Grandidierite, (Mg, Fe)Al<sub>3</sub>SiBO<sub>9</sub>

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Grandidierite, (Mg<sub>0.9</sub>Fe<sub>0.1</sub>)Al<sub>3</sub>SiBO<sub>9</sub>,  $a = 10.335 \pm 0.002$ ,  $b = 10.978 \pm 0.002$ ,  $c = 5.760 \pm 0.002$  Å, *Pbnm*,  $Z = 4$ , has Al-O octahedra, Al-O and Mg-O trigonal bipyramids, Si-O tetrahedra, and B-O triangles. Two independent sets of edge-linked Al-O octahedral chains run parallel to *c* [001] and are connected along *a* [100] by the B-centered triangles and Al-centered trigonal bipyramids and along *b* [010] by the Mg-centered trigonal bipyramids and silicate tetrahedra. The crystal structure of grandidierite is related to andalusite. Average M-O distances are Al<sup>VI</sup>-O 1.897 and 1.910, Al<sup>V</sup>-O 1.838, Mg<sup>V</sup>-O 2.042, Si<sup>IV</sup>-O 1.619, and B<sup>III</sup>-O 1.358 Å.

### 1. Introduction and description of the material

Grandidierite was described and named as a new mineral by Lacroix (1902). Its type locality is Andrahomana, near Fort Dauphin, in the southern extremity of Madagascar, where the mineral occurs as elongated anhedral individuals up to 8 cm in length associated with quartz, orthoclase, almandine, spinel, biotite, and andalusite, often poikilistically enclosing grains of these minerals. Our specimen label simply gave the location as Fort Dauphin, the bluish-green grandidierite occurring as small rounded grains and aggregates associated with pale blue spinel and distributed through platy masses of phlogopite.

The precise chemistry of grandidierite was not revealed until recently. The original analysis of Pisani in Lacroix appears in Table 1 along with a recent analysis by Scoon in McKie (1965). Our sample was qualitatively examined for Mg, Fe, Al, and Si by X-ray emission microanalysis and it gave good agreement with the element ratios cited in McKie's paper. Thus, we elected the composition (Mg<sub>0.90</sub>Fe<sub>0.10</sub>)Al<sub>3</sub>SiBO<sub>9</sub> for our structure analysis.

Prior to the appearance of McKie's article, we obtained crystal data for grandidierite which proved to be in very good agreement with his data. These data are given in Table 2. It was then considered that grandidierite may be structurally related to sillimanite,

Table 1. *Grandidierite. Chemical analyses*

	Pisani in Lacroix (1902)	Scoon in McKie (1965)
SiO <sub>2</sub>	20.46	20.39
TiO <sub>2</sub>	—	0.13
Al <sub>2</sub> O <sub>3</sub>	51.75	52.12
B <sub>2</sub> O <sub>3</sub>	2.81	11.57
Fe <sub>2</sub> O <sub>3</sub>	7.29	0.80
FeO	4.11	2.87
MnO	—	0.04
MgO	7.91	12.04
CaO	0.86	nil
Na <sub>2</sub> O	2.94	0.04
K <sub>2</sub> O	0.38	0.09
H <sub>2</sub> O <sup>+</sup>	1.64	—
H <sub>2</sub> O <sup>-</sup>	—	nil
	100.15	100.09

Table 2. *Grandidierite. Crystal data*

	This paper*	McKie†
<i>a</i>	10.34 Å	10.335 Å
<i>b</i>	10.99	10.978
<i>c</i>	5.75	5.760
Space group	<i>Pbnm</i>	
<i>Z</i>	4	
Formula	(Mg <sub>0.9</sub> Fe <sub>0.1</sub> )Al <sub>3</sub> SiBO <sub>9</sub>	
<i>q</i> <sub>obs</sub>	2.99	2.97 <sub>6</sub>
<i>q</i> <sub>calc</sub>		2.96
$\alpha$		1.590
$\beta$		1.618
$\gamma$		1.623
$2V$		30°

\* Estimated standard errors  $\pm 0.01$  Å.

† Estimated standard errors  $\pm 0.002$  Å. These data were used in the final refinement.

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