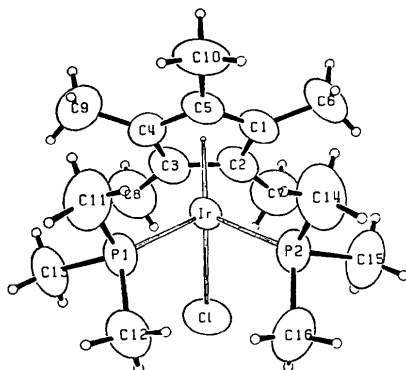


Table 2. Selected bond distances (Å) and angles (°)

Ir—Cl	2.410 (1)	P(2)—C(15)	1.808 (6)
Ir—P(1)	2.289 (1)	P(2)—C(16)	1.801 (7)
Ir—P(2)	2.300 (1)	C(1)—C(2)	1.414 (7)
Ir—C <sub>5</sub> , Me <sub>3</sub> centroid	1.875 (1)	C(2)—C(3)	1.459 (7)
Ir—C(1)	2.258 (4)	C(3)—C(4)	1.397 (7)
Ir—C(2)	2.233 (4)	C(4)—C(5)	1.444 (7)
Ir—C(3)	2.236 (5)	C(1)—C(5)	1.431 (6)
Ir—C(4)	2.255 (5)	C(1)—C(6)	1.497 (7)
Ir—C(5)	2.188 (4)	C(2)—C(7)	1.488 (7)
P(1)—C(11)	1.798 (6)	C(3)—C(8)	1.510 (8)
P(1)—C(12)	1.785 (6)	C(4)—C(9)	1.489 (8)
P(1)—C(13)	1.805 (6)	C(5)—C(10)	1.495 (7)
P(2)—C(14)	1.791 (7)		
P(1)—Ir—Cl	85.83 (5)	C(15)—P(2)—C(16)	99.5 (4)
P(1)—Ir—P(2)	96.40 (5)	C(16)—P(2)—C(14)	101.7 (4)
P(1)—Ir—C <sub>5</sub> , Me <sub>3</sub> centroid	126.37 (3)	C(1)—C(2)—C(3)	107.4 (4)
Cl—Ir—P(2)	86.69 (5)	C(2)—C(3)—C(4)	108.7 (4)
C(1)—Ir—C <sub>5</sub> , Me <sub>3</sub> centroid	122.10 (4)	C(3)—C(4)—C(5)	107.5 (4)
P(2)—Ir—C <sub>5</sub> , Me <sub>3</sub> centroid	126.88 (3)	C(4)—C(5)—C(1)	108.2 (4)
C(11)—P(1)—Ir	116.18 (23)	C(5)—C(1)—C(2)	107.9 (4)
C(12)—P(1)—Ir	121.03 (22)	C(6)—C(1)—C(5)	125.5 (5)
C(13)—P(1)—Ir	111.18 (21)	C(6)—C(1)—C(2)	125.9 (5)
C(11)—P(1)—C(12)	99.6 (3)	C(7)—C(2)—C(1)	127.5 (5)
C(12)—P(1)—C(13)	100.9 (3)	C(7)—C(2)—C(3)	125.0 (5)
C(13)—P(1)—C(11)	105.9 (3)	C(8)—C(3)—C(2)	124.1 (5)
C(14)—P(2)—Ir	116.42 (25)	C(8)—C(3)—C(4)	127.0 (5)
C(15)—P(2)—Ir	109.85 (23)	C(9)—C(4)—C(3)	127.0 (5)
C(16)—P(2)—Ir	121.51 (23)	C(9)—C(4)—C(5)	124.8 (5)
C(14)—P(2)—C(15)	105.7 (3)	C(10)—C(5)—C(4)	124.8 (5)

Fig. 1. View of the  $[\text{IrCl}(\text{C}_{10}\text{H}_{15})\{\text{P}(\text{CH}_3)_3\}_2]^+$  cation along the pseudo mirror plane with atomic labeling (ORTEP, Johnson, 1965).

thermal parameters for all non-H atoms; function minimized  $\sum w(F_o - F_c)^2$  where  $w = 1/\sigma^2(F_o^2)$  and  $\sigma(F_o^2) = [(\sigma_F)^2 + (0.035F_o^2)^2]^{1/2}$ , based on counting statistics; final  $R = 0.021$ ,  $wR = 0.027$ ,  $S = 1.36$ ;  $(\Delta/\sigma)_{\text{max}} = 0.07$  on final cycle; difference Fourier height max. =  $0.49 \text{ e } \text{Å}^{-3}$ .

The atomic coordinates are listed in Table 1 and bond distances and angles are presented in Table 2.\* A view of the molecule along the pseudo mirror plane and the numbering scheme are shown in Fig. 1.

The crystal-structure analysis was performed by the authors at the U. C. Berkeley X-ray Diffraction Facility (CHEXRAY) under the supervision of Dr F. J. Hollander, Professor D. H. Templeton and Mr B. Borgias as part of a requirement for Chemistry 208.

\* Lists of structure factors, anisotropic thermal parameters, positional and isotropic thermal parameters for calculated H atoms and selected least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42642 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A Five-Coordinate Mononuclear Copper Complex\*

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**Abstract.** Dibromo(1-methyl-2-imidazolecarbaldehyde azine)copper(II),  $\text{C}_{10}\text{H}_{12}\text{Br}_2\text{CuN}_6$ ,  $M_r = 439.6$ , mono-

clinic,  $Cc$ ,  $a = 7.540 (1)$ ,  $b = 13.613 (2)$ ,  $c = 13.537 (3) \text{ Å}$ ,  $\beta = 95.69 (2)^\circ$ ,  $V = 1382.6 \text{ Å}^3$ ,  $Z = 4$ ,  $D_x = 2.112 \text{ g cm}^{-3}$ ,  $\text{Cu K}\alpha_1$ ,  $\lambda = 1.54056 \text{ Å}$ ,  $\mu = 90.4 \text{ cm}^{-1}$ ,  $F(000) = 852$ ,  $T = 296 \text{ K}$ , final  $R = 0.038$

\* NRCC No. 25323.

for 1030 unique observed reflections. The copper coordination is fivefold, approximately square pyramidal, with three nitrogen atoms of the tridentate organic ligand and one bromine atom comprising the base, and with the other bromine atom at the apex. The Cu—Br distances are: apical, 2.609 (2) Å; basal, 2.436 (2) Å.

Table 1. Atomic coordinates (e.s.d.'s) and equivalent isotropic temperature factors for the non-hydrogen atoms

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cu	0.31190 (21)	0.99658 (10)	0.60369 (14)	2.9
Br(1)	0.00127*	1.00468 (8)	0.50034*	3.8
Br(2)	0.28350 (24)	0.88537 (8)	0.74218 (15)	4.0
N(1)	0.2767 (11)	1.1156 (6)	0.6824 (7)	2.9
N(2)	0.4931 (12)	1.1811 (6)	0.5086 (8)	3.4
N(3)	0.4650 (11)	1.0821 (6)	0.5168 (8)	3.0
N(4)	0.4176 (12)	0.8915 (6)	0.5257 (8)	3.2
N(5)	0.5858 (11)	0.8556 (6)	0.4048 (8)	3.1
N(6)	0.2646 (12)	1.2714 (6)	0.7187 (7)	3.2
C(1)	0.3214 (14)	1.2068 (7)	0.6551 (8)	2.8
C(2)	0.2856 (18)	1.3819 (8)	0.7185 (9)	3.5
C(3)	0.1718 (17)	1.2221 (9)	0.7860 (9)	3.6
C(4)	0.1797 (14)	1.1243 (8)	0.7599 (10)	3.5
C(5)	0.4232 (15)	1.2346 (8)	0.5755 (8)	3.3
C(6)	0.5458 (13)	1.0330 (7)	0.4522 (8)	2.5
C(7)	0.5153 (13)	0.9259 (7)	0.4573 (8)	2.8
C(8)	0.7032 (15)	0.8671 (9)	0.3256 (10)	4.1
C(9)	0.5316 (15)	0.7689 (8)	0.4448 (10)	3.9
C(10)	0.4271 (16)	0.7932 (8)	0.5179 (11)	4.1

\* Held constant during refinement.

Table 2. Bond lengths and angles and their e.s.d.'s (Å, °)

Cu—Br(1)	2.609 (2)	N(4)—C(10)	1.35 (2)
Cu—Br(2)	2.436 (2)	N(5)—C(7)	1.33 (1)
Cu—N(1)	1.972 (8)	N(5)—C(8)	1.46 (2)
Cu—N(3)	2.083 (10)	N(5)—C(9)	1.38 (2)
Cu—N(4)	1.991 (10)	N(6)—C(1)	1.33 (1)
N(1)—C(1)	1.35 (1)	N(6)—C(2)	1.51 (1)
N(1)—C(4)	1.34 (2)	N(6)—C(3)	1.38 (2)
N(2)—N(3)	1.37 (1)	C(1)—C(5)	1.43 (2)
N(2)—C(5)	1.31 (2)	C(3)—C(4)	1.38 (2)
N(3)—C(6)	1.30 (1)	C(6)—C(7)	1.48 (2)
N(4)—C(7)	1.32 (2)	C(9)—C(10)	1.37 (2)
Br(1)—Cu—Br(2)	107.2 (1)	C(7)—N(4)—C(10)	105 (1)
Br(1)—Cu—N(1)	95.4 (3)	C(7)—N(5)—C(8)	128 (1)
Br(1)—Cu—N(3)	101.1 (3)	C(7)—N(5)—C(9)	105 (1)
Br(1)—Cu—N(4)	97.5 (3)	C(8)—N(5)—C(9)	127 (1)
Br(2)—Cu—N(1)	93.9 (3)	C(1)—N(6)—C(2)	128 (1)
Br(2)—Cu—N(3)	151.4 (3)	C(1)—N(6)—C(3)	109 (1)
Br(2)—Cu—N(4)	91.8 (3)	C(2)—N(6)—C(3)	123 (1)
N(1)—Cu—N(3)	87.6 (4)	N(1)—C(1)—N(6)	109 (1)
N(1)—Cu—N(4)	163.6 (4)	N(1)—C(1)—C(5)	128 (1)
N(3)—Cu—N(4)	80.1 (4)	N(6)—C(1)—C(5)	123 (1)
Cu—N(1)—C(1)	123.9 (8)	N(6)—C(3)—C(4)	105 (1)
Cu—N(1)—C(4)	127.5 (7)	N(1)—C(4)—C(3)	109 (1)
C(1)—N(1)—C(4)	108 (1)	N(2)—C(5)—C(1)	131 (1)
N(3)—N(2)—C(5)	114 (1)	N(3)—C(6)—C(7)	113 (1)
Cu—N(3)—N(2)	134.0 (8)	N(4)—C(7)—N(5)	113 (1)
Cu—N(3)—C(6)	114.6 (7)	N(4)—C(7)—C(6)	119 (1)
N(2)—N(3)—C(6)	111 (1)	N(5)—C(7)—C(6)	128 (1)
Cu—N(4)—C(7)	113.4 (7)	N(5)—C(9)—C(10)	107 (1)
Cu—N(4)—C(10)	141.6 (9)	N(4)—C(10)—C(9)	110 (1)

**Experimental.** Specimen crystal green, prismatic [110] with terminal faces [001], dimensions 0.10 × 0.12 × 0.14 mm. Picker four-circle diffractometer with Ni-filtered Cu  $K\alpha$  radiation and  $\theta$ - $2\theta$  scan,  $2\theta < 130^\circ$ . Cell constants from 28 reflections in  $\theta$  range 53 to 65°. Absorption corrections (Gaussian integration) ranged from 1.88 to 3.18. Max.  $(\sin\theta)/\lambda = 0.588 \text{ Å}^{-1}$ ,  $h = -8$  to 8,  $k = 0$  to 16,  $l = 0$  to 15. 1 standard reflection (020) measured every 60 reflections, mean intensity 111 305(493) counts; no indication of specimen decay. Background counts accumulated for 0.1 times the scan period at beginning and end of each scan. 1180 unique reflections measured, of which 150 unobserved [ $I_{net} < 3\sigma(I_{net})$ ;  $\sigma$  from counting statistics]. Structure solved by inspection of Patterson synthesis, refined by block-diagonal least squares, minimizing  $\sum w\Delta F^2$ ,  $w^{-1} = \sigma^2(F_o) + 0.001F_o^2$ . Where possible, H atoms assigned reasonable parameters (C—H = 1.08 Å,  $U = 0.06 \text{ Å}^2$ ), not refined; methyl H atoms disregarded. Parameters refined were scale factor, secondary-extinction coefficient (7930 Å; Larson, 1970), coordinates and anisotropic temperature parameters for non-H atoms. Final  $R = 0.038$  (0.046 including unobserved reflections);  $wR = 0.051$ ,  $S = 1.29$ . Ratio of greatest parameter shift to e.s.d. in final cycle 0.02. Density in final difference-Fourier synthesis did not exceed  $-0.7$  to  $0.6 \text{ e Å}^{-3}$ . Atomic scattering factors ( $f, f', f''$ ) from *International Tables for X-ray Crystallography* (1974). (Refinement with the sense of the  $b$  axis reversed converged at  $R = 0.039$ ; the correct sense is not considered to be established.) Computer program system described by Larson & Gabe (1978).

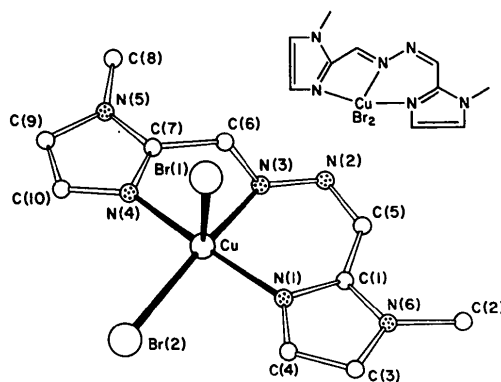


Fig. 1. A view of the molecule.

The structure is defined in Tables 1 and 2 and Fig. 1.\* There appear to be no intermolecular forces stronger than van der Waals.

**Related literature.** A preliminary description of the molecular structure (attributed to Hanson, unpublished) has been given by Woon, Thompson & Robichaud

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42654 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(1984). Although they describe the material as a hydrate, there is no evidence of hydration in the crystal.

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## The Structure of a Trichothecene from *Fusarium roseum*\*

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**Abstract.** 3,15-Diacetoxy-7,8-dihydroxy-12,13-epoxytrichothec-9-ene (12,13-epoxytrichothec-9-ene-3,7,8,15-tetrol 3,15-diacetate),  $C_{19}H_{26}O_8$ ,  $M_r = 382.4$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.972$  (1),  $b = 16.340$  (1),  $c = 10.212$  (1) Å,  $V = 1830.8$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.37$  (1),  $D_x = 1.387$  g cm<sup>-3</sup>,  $Cu K\alpha_1$ ,  $\lambda = 1.54056$  Å,  $\mu = 8.65$  cm<sup>-1</sup>,  $F(000) = 816$ ,  $T = 296$  K, final  $R = 0.042$  for 1420 unique observed reflections. The compound is a toxic metabolite present in substantial amounts in cultures of the fungus *Fusarium roseum*. The analysis confirms the formulation and establishes the stereochemistry. The torsion angle (O–C–C–O) of the hydroxyl-bearing 7,8 linkage is  $47.1$  (4)°. The hydroxyl group at C(8) acts as a proton donor and acceptor, forming an intermolecular hydrogen bond with the carbonyl group of the acetoxy group at C(15), and an intramolecular hydrogen bond with the adjacent hydroxyl group at C(7).

**Experimental.** Tabular crystal [010],  $0.13 \times 0.35 \times 0.35$  mm,  $D_m$  by flotation in carbon tetrachloride and cyclohexane solution. Picker four-circle diffractometer with Ni-filtered  $Cu K\alpha$  radiation,  $\theta$ - $2\theta$  scan,  $2\theta < 130^\circ$ . Cell constants from 30 reflections,  $56 < \theta < 64^\circ$ .

Absorption corrections (Gaussian integration) ranged from 1.117 to 1.312. Max.  $(\sin\theta)/\lambda = 0.588$  Å<sup>-1</sup>,  $h = 0$  to 12,  $k = 0$  to 19,  $l = 0$  to 12. 1 standard reflection (103) measured every 60 reflections, mean intensity 48 750(288) counts; no indication of specimen decay. 1798 unique reflections scanned, of which 378 unobserved [ $I_{net} < 3\sigma(I_{net})$ ;  $\sigma$  evaluated from counting statistics]. Net intensities derived by analysis of individual reflection profiles (Grant & Gabe, 1978). Structure solved by direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refined by block-diagonal least squares, minimizing  $\sum w\Delta F^2$ ,  $w^{-1} = \sigma^2(F_o) + 0.0003F_o^2$ . Where possible, H atoms placed in reasonable positions (C–H = 1.08 Å); otherwise (hydroxyl and methyl groups) located in difference Fourier synthesis. Reasonable isotropic temperature factors assigned:  $U = 0.08$  Å<sup>2</sup> for methyl H,  $0.06$  Å<sup>2</sup> otherwise. Parameters refined were scale factor, extinction coefficient ( $7.27 \times 10^4$ ; Larson, 1970), atomic coordinates and anisotropic temperature factors for C and O. Final  $R = 0.042$  (0.056 including unobserved reflections);  $wR = 0.057$ ,  $S = 2.10$ . Ratio of the max. shift to e.s.d. in final cycle 0.1. Density in final difference Fourier synthesis did not exceed  $\pm 0.2$  e Å<sup>-3</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

\* NRCC No. 25174.