

1140, 1110, 1090*vs*; 1035, 1020*m*; 960, 955, 950*m*; 885*m*, 860*s*, 755*vs*; 635, 625*m*; 410, 400*m*; 275*w*. Among these the band at 860 cm<sup>-1</sup> seems to be specially characteristic of the complex; this is absent from the spectrum of quinoxaline. The 275 cm<sup>-1</sup> band we assign to ν(Cu–N) stretching vibrations.

The IR spectrum of the complex shows the strong band found at 953 cm<sup>-1</sup> in the quinoxaline spectrum (Billing, 1969), obviously in the region 1000–900 cm<sup>-1</sup>, split into a triplet at 960–950 cm<sup>-1</sup> and pointing to quinoxaline coordination. A complex studied earlier, triaquabis(quinoxaline)copper(II) perchlorate, with terminal quinoxaline ligands, has a medium peak at 940 cm<sup>-1</sup> in its IR spectrum (Lumme *et al.*, 1987). The responsibility of this band for the coordination of pyrazine to the central atom has earlier been assumed (Lever, Lewis & Nyholm, 1962). The present complex also shows three intense bands of the bridging bidentate perchlorate in the range 1150–1050 cm<sup>-1</sup> (1140, 1110 and 1090 cm<sup>-1</sup>) owing to ν(Cl–O) stretching vibrations.

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## Structure of {4,4'-[Iminobis(1,3-propanediylnitrilo)]-di-2-pentanonato-N,N',N'',O,O'} copper

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**Abstract.** [Cu(C<sub>16</sub>H<sub>27</sub>N<sub>3</sub>O<sub>2</sub>)], *M<sub>r</sub>* = 356.95, monoclinic, *C2/c*, *a* = 22.234 (5), *b* = 11.753 (5), *c* = 19.005 (4) Å, β = 135.57 (2)°, *V* = 3476.8 Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.364 g cm<sup>-3</sup>, λ(Mo *Kα*) = 0.71073 Å, μ = 12.7 cm<sup>-1</sup>, *F*(000) = 1512, *T* = 168 K, final *R* = 0.036 for 2813 unique observed reflections. The copper coordination is fivefold, approximately trigonal bipyramidal. Imine nitrogens occupy *trans* apical positions (1.96 Å, 175°). Two oxygens (1.98 Å, 142°) and the secondary nitrogen of the pentadentate ligand (2.29 Å, 103°) occupy the trigonal equatorial sites.

**Introduction.** Since initial isolation, neutral five-coordinate Cu<sup>II</sup> complexes containing doubly condensed pentadentate Schiff bases have been described as most probably possessing distorted square-pyramidal

structure (Cummings & Wallis, 1974; Cummings, Chen, Chu, McKinney & Willis, 1981; Cummings, Braydich & Fortman, 1983). The structure of the title compound demonstrates that in the solid state the molecule is more appropriately described as trigonal bipyramidal with a nearly perfect twofold axis along the copper–secondary-amine vector.

**Experimental.** Green crystals were obtained following method *B* of Cummings *et al.* (1981). Nearly cube-shaped specimen, dimensions 0.30 × 0.30 × 0.40 mm. Enraf–Nonius CAD-4 diffractometer with graphite crystal monochromator using ω–2θ, 2θ < 55°, scan speed varying from 2 to 20° min<sup>-1</sup> (in 2θ). Lattice parameters from 25 reflections in θ range of 7–11°. Empirical absorption corrections (Walker & Stuart,

Table 1. Atomic positions and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{1}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + bc(\cos\alpha)B_{23} + ac(\cos\beta)B_{13} + ab(\cos\gamma)B_{12}]$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}$
Cu	0.76143 (1)	0.11077 (3)	0.74667 (2)	2.260 (7)
O(1)	0.65812 (9)	0.1271 (2)	0.6007 (1)	2.63 (4)
O(2)	0.85132 (9)	0.2024 (2)	0.8705 (1)	3.12 (5)
N(2)	0.8360 (1)	0.868 (2)	0.7275 (1)	2.61 (5)
N(3)	0.6868 (1)	0.1196 (2)	0.7670 (1)	2.40 (5)
N(4)	0.7932 (1)	-0.0708 (2)	0.8084 (1)	2.46 (5)
C(1)	0.5714 (2)	0.1552 (3)	0.4288 (2)	3.51 (8)
C(2)	0.6573 (1)	0.1300 (2)	0.5325 (2)	2.57 (6)
C(3)	0.7271 (1)	0.1106 (3)	0.5483 (2)	3.30 (7)
C(4)	0.8122 (1)	0.0867 (3)	0.6419 (2)	3.32 (7)
C(5)	0.8751 (2)	0.0591 (5)	0.6365 (2)	5.8 (1)
C(6)	0.9237 (1)	0.0536 (3)	0.8204 (2)	2.97 (7)
C(7)	0.9275 (1)	-0.0686 (3)	0.8490 (2)	3.33 (7)
C(8)	0.8868 (1)	-0.0907 (3)	0.8858 (2)	3.02 (7)
C(11)	0.9124 (1)	0.3396 (3)	0.9968 (2)	3.41 (7)
C(12)	0.8387 (1)	0.2632 (2)	0.9153 (1)	2.63 (6)
C(13)	0.7657 (1)	0.2619 (3)	0.8946 (1)	2.91 (6)
C(14)	0.6956 (1)	0.1857 (3)	0.8292 (1)	2.72 (6)
C(15)	0.6317 (1)	0.1820 (3)	0.8355 (2)	3.80 (7)
C(16)	0.6235 (1)	0.0279 (3)	0.7136 (2)	2.88 (6)
C(17)	0.6612 (1)	-0.0837 (3)	0.7726 (2)	3.04 (6)
C(18)	0.7587 (1)	-0.0906 (2)	0.8506 (2)	2.94 (7)

1983). Data collected by Molecular Structure Corporation using *I*-centered cell ( $a = 15.877$ ,  $b = 11.753$ ,  $c = 19.005$   $\text{\AA}$ ,  $\beta = 101.36^\circ$ ) and reindexed to *C*-centered cell.  $h = -23$  to  $28$ ,  $k = -15$  to  $0$ ,  $l = -24$  to  $24$ . Three representative reflections measured every 41 min,  $-1.3\%$  loss in intensity over 29.7 h collection time, 4321 unique reflections; 2813 with  $I > 3\sigma(I)$ , 948 with  $I < \sigma(I)$ . Structure solved by combination of direct methods and difference Fourier synthesis. Some H atoms found in difference maps, others placed at calculated positions. Refinement by full-matrix least squares, minimizing  $w\Delta F^2$ ,  $w^{-1} = \sigma^2(F_o)$ , of 308 parameters including scale factor, secondary-extinction coefficient, position, anisotropic displacement for non-H atoms, and isotropic displacement for H atoms. Final  $R = 0.036$ ,  $wR = 0.048$ ,  $S = 1.37$ . Ratio of greatest parameter shift to e.s.d. in final cycle 0.28 for H and 0.14 for non-H with the majority of parameters  $< 0.05$ . Density in final difference Fourier synthesis did not exceed  $0.27 \text{ e } \text{\AA}^{-3}$ . Secondary-extinction coefficient  $5.38 \times 10^{-7}$ . Atom scattering factors ( $f$ ,  $f'$ ,  $f''$ ) from *International Tables for X-ray Crystallography* (1974). Computer programs described by Frenz (1983). The structure is defined in Tables 1 and 2 and Fig. 2.\* There appear to be no intermolecular interactions stronger than van der Waals.

\* Lists of H-atom parameters, structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44122 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** Addison, Rao, Reedijk, van Rijn & Verschoor (1984) have described the 'trigonality' of five-coordinate geometries using the angular parameter

$$\tau = (\text{largest } L-M-L' \text{ angle} - \text{next largest } L''-M-L''' \text{ angle})/60.$$

$L$ ,  $L'$ ,  $L''$ ,  $L'''$  represent ligand electron-pair donor atoms chosen pairwise to form angle arms with the central atom  $M$ .  $\tau = 1$  represents the ideal trigonal-bipyramidal geometry,  $\tau = 0$  the ideal square-pyramidal geometry. For the title compound  $\tau = 0.55$ .

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Cu—O(1)	1.976 (2)	Cu—N(4)	2.292 (2)
Cu—O(2)	1.994 (2)	Cu—N(2)	1.956 (2)
C(1)—C(2)	1.498 (4)	Cu—N(3)	1.955 (2)
C(2)—O(1)	1.286 (3)	C(11)—C(12)	1.505 (4)
C(2)—C(3)	1.372 (4)	C(12)—O(2)	1.286 (3)
C(3)—C(4)	1.420 (4)	C(12)—C(13)	1.371 (4)
C(4)—N(2)	1.303 (3)	C(13)—C(14)	1.418 (4)
C(4)—C(5)	1.507 (4)	C(14)—N(3)	1.308 (3)
C(6)—N(2)	1.468 (3)	C(14)—C(15)	1.509 (4)
C(6)—C(7)	1.516 (4)	C(16)—N(3)	1.461 (3)
C(7)—C(8)	1.509 (4)	C(16)—C(17)	1.529 (4)
C(8)—N(4)	1.478 (3)	C(17)—C(18)	1.523 (4)
O(1)—Cu—O(2)	141.72 (7)	C(18)—N(4)	1.471 (3)
O(1)—Cu—N(2)	92.48 (8)	Cu—O(2)—C(12)	124.9 (2)
O(1)—Cu—N(3)	88.41 (8)	N(3)—C(14)—C(13)	122.0 (2)
Cu—N(2)—C(4)	126.2 (2)	N(3)—C(14)—C(15)	121.0 (3)
Cu—N(2)—C(6)	112.9 (2)	N(3)—C(16)—C(17)	118.8 (2)
Cu—O(1)—C(2)	125.7 (2)	O(2)—C(12)—C(11)	114.9 (2)
N(2)—C(4)—C(3)	123.6 (3)	O(2)—C(12)—C(13)	125.3 (2)
N(2)—C(4)—C(5)	121.1 (3)	C(14)—N(3)—C(16)	120.9 (2)
N(2)—C(6)—C(7)	111.6 (2)	C(11)—C(12)—C(13)	119.8 (3)
O(1)—C(2)—C(1)	114.7 (3)	C(12)—C(13)—C(14)	126.3 (3)
O(1)—C(2)—C(3)	125.1 (2)	C(13)—C(14)—C(15)	117.0 (3)
C(4)—N(2)—C(6)	120.5 (2)	O(2)—Cu—N(4)	102.55 (8)
C(1)—C(2)—C(3)	120.2 (3)	N(2)—Cu—N(4)	88.17 (8)
C(2)—C(3)—C(4)	126.5 (3)	N(3)—Cu—N(4)	86.74 (8)
C(3)—C(4)—C(5)	115.3 (3)	Cu—N(4)—C(8)	110.8 (2)
O(1)—Cu—N(4)	115.60 (7)	Cu—N(4)—C(18)	109.6 (2)
O(2)—Cu—N(2)	91.68 (8)	N(4)—C(8)—C(7)	113.3 (2)
O(2)—Cu—N(3)	90.85 (8)	N(4)—C(18)—C(17)	113.3 (2)
N(2)—Cu—N(3)	174.71 (9)	C(8)—N(4)—C(18)	110.1 (2)
Cu—N(3)—C(14)	127.6 (2)	C(6)—C(7)—C(8)	115.5 (2)
Cu—N(3)—C(16)	110.9 (2)	C(16)—C(17)—C(18)	114.3 (2)

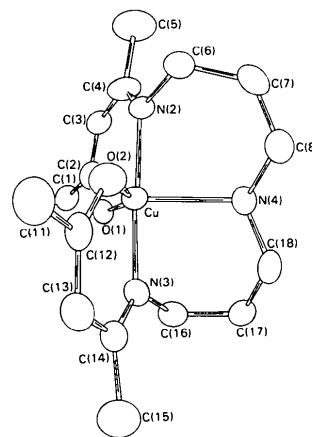


Fig. 1. Molecular conformation of the title compound showing atom identification. Displacement ellipsoids drawn at 50% probability.

Characterization of the coordinated ligand moiety as 'en...ate' is supported by comparison of the C(2)—C(3), C(12)—C(13) distances with those of C(3)—C(4), C(13)—C(14). However both C—O distances are decidedly carbonyl. C(4)—N(2) and C(14)—N(3) appear to be imine bonds relative to the amine bonds of C(6)—N(2) and C(8)—N(4) or C(16)—N(3) and C(18)—N(4). The nearly perfect twofold axis down the N(4)—Cu vector is noteworthy.

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### Structure of Tricarbonyl( $\eta^4$ -*syn*-1,4-diacetoxy-1,3-butadiene)iron(0)\*

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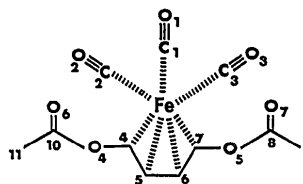
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**Abstract.** [Fe(CO)<sub>3</sub>(C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>)], *M*<sub>r</sub> = 310.0, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 11.093 (4), *b* = 10.680 (3), *c* = 11.763 (5) Å,  $\beta$  = 101.45 (3)°, *V* = 1366 (1) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.51 Mg m<sup>-3</sup>, *Mo* *K*α,  $\lambda$  = 0.7107 Å,  $\mu$  = 1.124 mm<sup>-1</sup>, *F*(000) = 632, *T* = 293 K, *R* = 0.046 for 1888 observed reflections. The molecular structure is determined from the X-ray data and confirms the structure previously assigned on the basis of IR and NMR spectra. The Fe(CO)<sub>3</sub> group is symmetrically bonded to the butadiene moiety formed by C(4) to C(7) of the ligand. Both ester groups at C(4) and C(7) are planar and *cis* to each other about the central C(5)—C(6) bond. The structure consists of discrete molecules held in the crystal by van der Waals forces.

**Introduction.** As part of our investigations of complexes between Fe(CO)<sub>3</sub> and vinyl ester ligands, the title compound (1) was synthesized. Several complexes between Fe(CO)<sub>3</sub> and the vinyl ligand with various substituents including phenyl, ester and acyl groups

have been prepared in order to study their molecular conformation and chemical reactivity. The structure determination was undertaken to elucidate the stereochemistry of (1).



(1)

**Experimental.** The title compound (1) was obtained from the reaction of *trans,trans*-1,4-diacetoxybutadiene (1.7 g, 10 mmol) (prepared as described by Hill & Carlson, 1964) and Fe(CO)<sub>5</sub> (9.7 g, 50 mmol) which was irradiated with an ultraviolet lamp of 200 W at room temperature. The product is purified by chromatographic methods and recrystallized from ethyl acetate to give yellow air-stable crystals with a composition of Fe(C<sub>11</sub>H<sub>10</sub>O<sub>7</sub>).

\* Contribution No. 855 of the Instituto de Química, UNAM.

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