

Bis(acetato)(4-benzyl-1,7-diphenyl-2,4,6-triazahepta-2,5-diene- $\kappa^2 N^2, N^6$)copper(II)

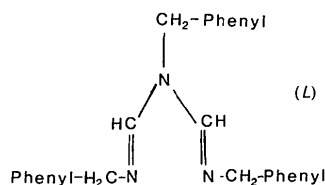
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Abstract. $[\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{23}\text{H}_{23}\text{N}_3)]$, $M_r = 523.09$, monoclinic, $P2_1/c$, $a = 11.03$ (1), $b = 10.29$ (1), $c = 25.03$ (2) Å, $\beta = 116.8$ (1)°, $V = 2535.7$ Å³, $Z = 4$, $D_x = 1.370$ g cm⁻³, $F(000) = 1092$, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 8.94$ cm⁻¹, $T = 290$ K, $R = 0.048$ for 1305 unique observed reflexions. Coordination around Cu is approximately square planar. Cu is in the plane of the bidentate amidine group with Cu—N 1.991 (7) and 1.991 (9) Å; O atoms of each acetate group are respectively 0.512 (9) and 0.925 (8) Å above and below this plane, with Cu—O 1.972 (7) and 1.960 (6) Å. Cu—O contacts to the other O atoms of the acetate groups are 2.531 (8) and 2.483 (7) Å.

Experimental. The ligand (*L*) 4-benzyl-1,7-diphenyl-2,4,6-triazahepta-2,5-diene was prepared by the reaction of ethyl orthoformate and benzylamine on *N,N'*-dibenzylformamide.* By reacting the ligand (*L*) with copper(II) acetate in methanol, the title



compound was obtained as bright-blue needles elongated parallel to *b*. Cell dimensions, initially from Weissenberg photographs [$\lambda(\text{Cu } K\alpha) = 1.542$ Å], refined from setting angles of 20 reflexions in the range $10 < 2\theta < 40^\circ$ on STADI-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$). This was also used for measurement of 3297 intensities (2926 unique, $R_{\text{int}} = 0.022$) from a crystal $0.06 \times 0.08 \times 0.30$ mm for the layers $h0l$ to $h10l$, standard measured every 20 reflexions (overall decrease $< 10\%$, allowed for by linear interpolation), $h - 10$ to 9, $k 0$ to 10, $l 0$ to 24, max. $(\sin\theta)/\lambda = 0.50$ Å⁻¹; variable ω

* Full details of the preparation of the ligand (*L*) and lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53614 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

scan, $2\theta'$ fixed, stationary background count. Lp but not absorption correction made. 1306 reflexions for which $I > 3\sigma(I)$ were used in the structure determination. Cu-atom position obtained from Patterson maps, other non-H positions from subsequent ΔF maps. Refinement, using *F*, of non-H-atom positions (the C atoms in the phenyl groups being constrained as rigid groups with C—C = 1.395 Å) and U_{ij} . H atoms in calculated positions (C—H = 1.08 Å) with common isotropic *U* refined in CH, CH₂, CH₃ and phenyl groups respectively. At an intermediate stage interlayer scale factors were refined. 299 parameters refined in two blocks alternately until $\Delta/\sigma < 0.05$, and largest features on ΔF map were $+0.29$ to -0.29 e Å⁻³, $R = 0.048$, $wR = 0.043$, $S = 1.318$, $Q = 4.36$ and $w = 1.273/[\sigma^2(F) + 0.000131F^2]$. SHELX76 (Sheldrick, 1976) programs were used throughout.

Table 1. Fractional atomic coordinates ($\times 10^4$; Cu $\times 10^5$) and U_{eq} (Å² $\times 10^3$; Cu $\times 10^4$) values

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cu	15910 (11)	24418 (14)	55527 (5)	379 (10)
C(1)	1382 (9)	4461 (87)	4658 (4)	46 (10)
N(1)	623 (8)	3427 (7)	4791 (3)	37 (9)
C(2)	-635 (10)	3397 (10)	4444 (4)	42 (12)
N(2)	-1531 (8)	2553 (10)	4492 (3)	47 (8)
C(3)	-1248 (10)	1661 (10)	4939 (4)	43 (12)
N(3)	-119 (8)	1503 (7)	5391 (3)	40 (9)
C(4)	-73 (9)	443 (8)	5810 (4)	46 (10)
C(5)	-2993 (8)	2739 (11)	4053 (3)	50 (10)
C(25)	5133 (9)	1673 (11)	5511 (5)	86 (14)
C(24)	3725 (12)	1709 (13)	5492 (5)	56 (14)
O(1)	3403 (6)	2836 (6)	5619 (3)	47 (7)
O(2)	3032 (7)	731 (7)	5384 (3)	68 (9)
C(27)	2576 (9)	3288 (9)	7272 (3)	54 (10)
C(26)	2181 (10)	3203 (11)	6614 (4)	42 (12)
O(3)	2352 (7)	2096 (6)	6413 (3)	41 (7)
O(4)	1687 (7)	4135 (6)	6278 (3)	63 (8)
C(7)	3237 (7)	4733 (6)	4376 (3)	52 (11)
C(8)	3973 (7)	4373 (6)	4069 (3)	66 (15)
C(9)	3630 (7)	3244 (6)	3724 (3)	78 (15)
C(10)	2551 (7)	2476 (6)	3684 (3)	79 (13)
C(11)	1815 (7)	2836 (6)	3991 (3)	58 (13)
C(6)	2158 (7)	3965 (6)	4337 (3)	39 (10)
C(13)	-3584 (7)	3178 (5)	2988 (3)	59 (12)
C(14)	-3941 (7)	2775 (5)	2403 (3)	71 (15)
C(15)	-4024 (7)	1453 (5)	2269 (3)	67 (14)
C(16)	-3750 (7)	533 (5)	2718 (3)	68 (14)
C(17)	-3393 (7)	936 (5)	3302 (3)	56 (12)
C(12)	-3310 (7)	2259 (5)	3437 (3)	43 (11)
C(19)	-1097 (7)	2086 (7)	6265 (3)	56 (13)
C(20)	-1350 (7)	2467 (7)	6740 (3)	69 (12)
C(21)	-825 (7)	1742 (7)	7268 (3)	79 (16)
C(22)	-47 (7)	637 (7)	7321 (3)	73 (16)
C(23)	207 (7)	257 (7)	6845 (3)	60 (12)
C(18)	-318 (7)	981 (7)	6318 (3)	40 (10)

Table 2. Bond distances (Å) and angles (°)

Cu—N(1)	1.991 (7)	N(2)—C(5)	1.499 (10)
Cu—N(3)	1.991 (9)	C(3)—N(3)	1.260 (11)
Cu—O(1)	1.972 (7)	N(3)—C(4)	1.498 (12)
Cu—O(2)	2.531 (8)	C(4)—C(18)	1.519 (13)
Cu—O(3)	1.960 (6)	C(5)—C(12)	1.503 (12)
Cu—O(4)	2.483 (7)	C(25)—C(24)	1.532 (18)
C(1)—N(1)	1.482 (13)	C(24)—O(1)	1.294 (15)
C(1)—C(6)	1.503 (14)	C(24)—O(2)	1.218 (15)
N(1)—C(2)	1.262 (12)	C(27)—C(26)	1.505 (13)
C(2)—N(2)	1.361 (15)	C(26)—O(3)	1.291 (14)
N(2)—C(3)	1.371 (14)	C(26)—O(4)	1.230 (12)
N(1)—Cu—N(3)	90.1 (3)	C(12)—C(5)—N(2)	112.7 (8)
N(1)—Cu—O(1)	93.5 (3)	N(2)—C(3)—N(3)	126.0 (10)
N(3)—Cu—O(3)	91.9 (3)	C(3)—N(3)—C(4)	115.4 (9)
O(1)—Cu—O(3)	91.7 (3)	N(3)—C(4)—C(18)	110.9 (7)
C(6)—C(1)—N(1)	113.2 (7)	C(25)—C(24)—O(1)	113.6 (10)
C(1)—N(1)—C(2)	114.8 (8)	C(25)—C(24)—O(2)	120.8 (12)
N(1)—C(2)—N(2)	124.9 (9)	O(1)—C(24)—O(2)	125.5 (13)
C(2)—N(2)—C(5)	116.0 (8)	C(27)—C(26)—O(3)	117.1 (9)
C(2)—N(2)—C(3)	126.3 (7)	C(27)—C(26)—O(4)	121.6 (10)
C(5)—N(2)—C(3)	117.2 (9)	O(3)—C(26)—O(4)	121.2 (10)

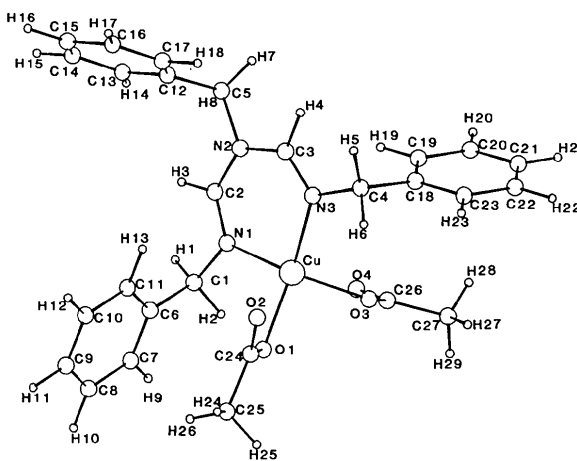


Fig. 1. Molecule of title compound with atom labelling.

Atomic scattering factors: for Cu, Cromer & Mann (1968), other atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters in Table 1, bond distances and angles in Table 2, the molecule with atomic labelling is shown in Fig. 1.

Related literature. No other complexes of copper(II) acetate with a bidentate N-donor ligand have been reported. Some square planar Cu^{II} complexes in which the four coordination involves a bidentate N-donor ligand and two O atoms from other groups have been reported. Amongst these are the following: dinitrato(*N,N,N',N'*-tetramethylethylenediamine)-copper(II) (Pavkovic, Miller & Brown, 1977); bis(*N*-acetyl-glycinato)(1,10-phenanthroline)copper(II) (Battaglia, Bonamartini, Corradi & Marcotrigiano,

1977); bis(nitrito)(2,2'-bipyridyl)copper(II) (Stephens, 1969); (2,2'-bipyridyl)bis(*N*-acetyl-*dl*-leucinato-*O*)copper(II) (Antolini, Menabue, Saladini, Battaglia & Corradi, 1984).

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Structure of Di(quinoxalinium) Tetrachlorocobaltate(II) Monohydrate

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Abstract. (C₈H₇N₂)₂[CoCl₄].H₂O, *M*_r = 481.06, triclinic, *P* $\bar{1}$, *a* = 7.439 (2), *b* = 7.561 (2), *c* = 20.104 (5) Å, α = 94.11 (2), β = 90.95 (2), γ = 117.57 (2)°, *V* = 998.3 (5) Å³, *Z* = 2, *D*_m = 1.604 (8), *D*_x = 1.60 g cm⁻³, λ (Mo *K*α) = 0.71069 Å, μ = 14.3 cm⁻¹, *F*(000) = 486, *T* = 296 K, 4587 unique

reflections measured, final *R* = 0.067 over 3054 reflections having *F*_o > 6σ(*F*_o). The structure consists of discrete CoCl₄²⁻ anions and two quinoxalinium cations. The tetrachlorocobaltate anion is a nearly regular tetrahedron, with average Co—Cl 2.266 (19) Å and Cl—Co—Cl 109.4 (3.5)°. Both quinoxalinium cations are planar. One of them is disordered, having two orientations with a dihedral

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