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[*N,N'*-Bis(4-chlorobenzylidene)-2,2-dimethylpropane-1,3-diamine- κ^2 N,*N'*]-iodidocopper(I)

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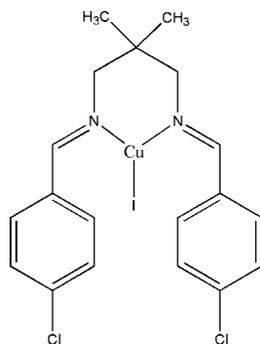
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.024; wR factor = 0.067; data-to-parameter ratio = 39.9.

The molecule of the title compound, $[\text{CuI}(\text{C}_{19}\text{H}_{20}\text{Cl}_2\text{N}_2)]$, lies across a crystallographic mirror plane. The coordination around the copper centre is distorted trigonal planar, with a bite angle of $94.40(7)^\circ$. A six-membered chelate ring is formed by the coordination of iminic N atoms of the bidentate ligand to the Cu^{I} atom, adopting a chair conformation. This conformation is required if the local symmetry of the metal coordination site is in accordance with a mirror plane that passes through the metal atom normal to the line connecting the N atoms. The dihedral angle between the benzene rings is $78.66(5)^\circ$. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\pi$ interactions, which link the molecules into chains along the b axis.

Related literature

For puckering parameters, see: Cremer & Pople (1975). For related literature and the catalytic applications, see, for example: Killian *et al.* (1996); Jung *et al.* (1996); Small *et al.* (1998). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$[\text{CuI}(\text{C}_{19}\text{H}_{20}\text{Cl}_2\text{N}_2)]$	$V = 2125.37(3) \text{ \AA}^3$
$M_r = 537.71$	$Z = 4$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 16.2770(1) \text{ \AA}$	$\mu = 2.74 \text{ mm}^{-1}$
$b = 12.2983(1) \text{ \AA}$	$T = 296(2) \text{ K}$
$c = 10.6255(1) \text{ \AA}$	$0.44 \times 0.31 \times 0.28 \text{ mm}$
$\beta = 92.249(1)^\circ$	

Data collection

Bruker APEXII CCD area-detector diffractometer	39802 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2005)	4869 independent reflections
$T_{\text{min}} = 0.334$, $T_{\text{max}} = 0.463$	4157 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$	122 parameters
$wR(F^2) = 0.067$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.74 \text{ e \AA}^{-3}$
4869 reflections	$\Delta\rho_{\text{min}} = -0.55 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8A}\cdots\text{Cg1}^{\text{i}}$	0.97	2.92	3.7220 (19)	141

Symmetry code: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$. Cg1 is the centroid of the C1‐C6 benzene ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2609).

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[*N,N'*-Bis(4-chlorobenzylidene)-2,2-dimethylpropane-1,3-diamine- κ^2 *N,N'*]iodidocopper(I)

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S1. Comment

In recent years, an increasing amount of research has been focused on the design and preparation of mono- or di-nuclear mixed ligand transition metal complexes containing neutral, chelating nitrogen ligands. Early and late transition metal complexes of this type have extensively been used as catalysts for a wide categories of reactions, including olefin polymerization (Killian *et al.*, 1996) and oxygen activation (Jung *et al.*, 1996). In this context, diverse chelating Schiff base type ligands, amines and pyridine derivatives (Small *et al.*, 1998) have successfully been applied in the preparation of these homogeneous catalysts. Here we report the crystal structure of an aldimine Schiff base ligand with copper(I) iodide. To the best of our knowledge, the title compound is the first tricoordinate complex of an aldimine bis-Schiff base ligand with copper(I) iodide adopting trigonal planar geometry.

The title compound, I, Fig. 1, lies across a crystallographic mirror plane. Atoms I1, Cu1, C9, C10 and C11 lies on this mirror plane. The asymmetric unit of (I) is composed of one-half of the molecule. The coordination geometry around copper has a distorted trigonal planar geometry. The deviation of the Cu atom from the N1/N1A/I1 plane is $-0.1213(8)$ Å. A six-membered chelate ring is formed in this case by the coordination of iminic nitrogen atoms of the bidentate ligand which adopts the chair conformation with the ring puckering parameters (Cremer & Pople 1975) of $Q = 0.7001(14)$ Å, $\Theta = 7.72(11)^\circ$, $\Phi = 0.0(9)^\circ$. This conformation is required if the local symmetry of the metal coordination site is in accordance with a mirror plane that passed through the metal atom normal to the line connecting the nitrogen atoms. The dihedral angle between the phenyl rings is $78.66(5)^\circ$. The crystal structure is stabilized by weak intermolecular C—H $\cdots\pi$ interactions ($Cg1$ is the centroid of the C1–C6 benzene ring) which link the molecules into chains along the *b*-axis (Fig. 2 and Table 1).

S2. Experimental

N,N'-Bis(4-chlorobenzylidene)-2,2-dimethylpropane (694 mg, 2 mmol) was added dropwise to a suspension of CuI (380 mg, 2.0 mmol) in 50 ml of THF. After 15 minutes a clear yellowish solution was obtained. The volume of the reaction mixture was reduced until the formation of a yellow precipitate occurred. Single crystals suitable for X-ray diffraction were grown from the acetonitrile solution.

S3. Refinement

All H atoms were positioned geometrically with C—H = 0.93 Å (aromatic), 0.96 Å (methyl), and 0.97 Å (methylene) and refined in the riding model approximation with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. The highest peak (0.74 e. Å $^{-3}$) is located 0.60 Å from I1 and the deepest hole (-0.55 e. Å $^{-3}$) is located 0.59 Å from I1.

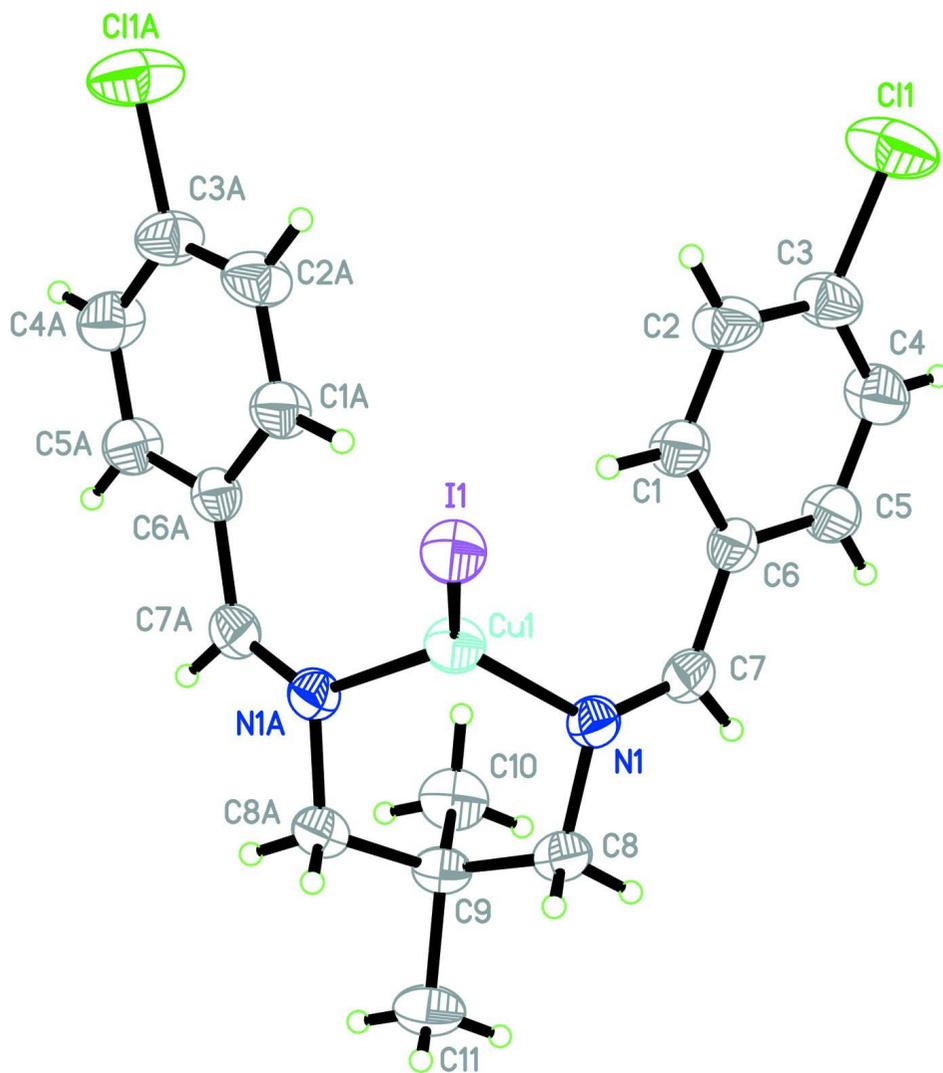


Figure 1

The molecular structure of (I), showing 40% probability displacement ellipsoids and the atomic numbering. Symmetry code for A atoms; $X, -Y, Z$.

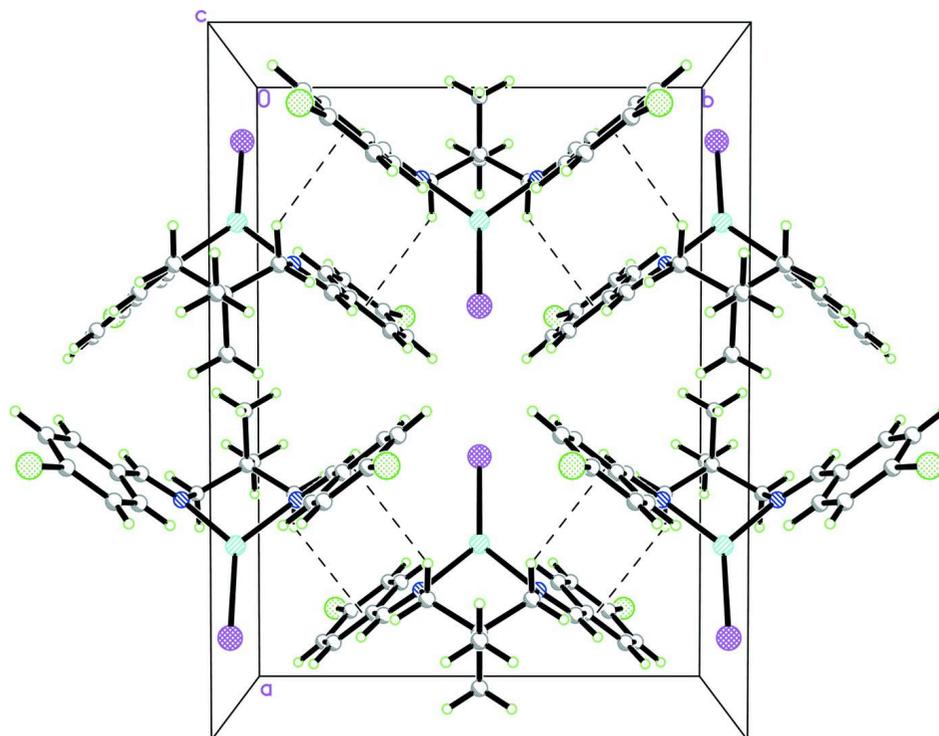


Figure 2

The crystal packing of (I), viewed down the *c*-axis, showing C—H... π interactions linking the molecules into chains along the *b*-axis.

[*N,N'*-Bis(4-chlorobenzylidene)-2,2-dimethylpropane-1,3-diamine- κ^2N,N']iodidocopper(I)

Crystal data

[CuI(C₁₉H₂₀Cl₂N₂)]

M_r = 537.71

Monoclinic, *C2/m*

Hall symbol: -C 2y

a = 16.2770 (1) Å

b = 12.2983 (1) Å

c = 10.6255 (1) Å

β = 92.249 (1)°

V = 2125.37 (3) Å³

Z = 4

F(000) = 1056

D_x = 1.674 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 9801 reflections

θ = 2.5–35.7°

μ = 2.74 mm⁻¹

T = 296 K

Block, yellow

0.44 × 0.31 × 0.28 mm

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

T_{min} = 0.334, *T_{max}* = 0.463

39802 measured reflections

4869 independent reflections

4157 reflections with *I* > 2 σ (*I*)

R_{int} = 0.026

θ_{\max} = 35.0°, θ_{\min} = 1.9°

h = -26→26

k = -19→19

l = -17→17

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.024$ $wR(F^2) = 0.067$ $S = 1.02$

4869 reflections

122 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 1.3061P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.74 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.55 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00458 (18)

*Special details***Experimental.** The low-temperature data were collected with the Oxford Cyrosystem Cobra low-temperature attachment**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.**Refinement.** Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.116357 (8)	0.0000	0.351624 (13)	0.04474 (5)
Cu1	0.250035 (16)	0.0000	0.46811 (2)	0.04325 (7)
Cl1	0.38586 (4)	0.32893 (7)	-0.03450 (5)	0.0900 (2)
N1	0.31811 (7)	0.11994 (10)	0.54747 (11)	0.0410 (2)
C1	0.32263 (11)	0.17020 (14)	0.27282 (15)	0.0515 (4)
H1A	0.2889	0.1119	0.2920	0.062*
C2	0.32963 (11)	0.20089 (16)	0.14902 (16)	0.0567 (4)
H2A	0.3009	0.1637	0.0851	0.068*
C3	0.37946 (11)	0.28682 (17)	0.12088 (16)	0.0545 (4)
C4	0.42404 (12)	0.34113 (16)	0.21435 (18)	0.0603 (4)
H4A	0.4593	0.3974	0.1940	0.072*
C5	0.41577 (11)	0.31124 (14)	0.33842 (16)	0.0519 (3)
H5A	0.4444	0.3493	0.4017	0.062*
C6	0.36540 (9)	0.22526 (11)	0.37029 (13)	0.0411 (3)
C7	0.35971 (9)	0.19805 (12)	0.50364 (14)	0.0440 (3)
H7A	0.3889	0.2415	0.5614	0.053*
C8	0.32286 (11)	0.10423 (13)	0.68508 (14)	0.0486 (3)
H8A	0.2675	0.1019	0.7156	0.058*
H8B	0.3505	0.1664	0.7237	0.058*
C9	0.36849 (14)	0.0000	0.72732 (19)	0.0456 (4)
C10	0.45648 (16)	0.0000	0.6822 (3)	0.0618 (6)
H10A	0.4554	0.0000	0.5918	0.093*

H10B	0.4847	-0.0637	0.7132	0.093*
C11	0.3698 (2)	0.0000	0.8725 (2)	0.0717 (8)
H11A	0.3144	0.0000	0.9003	0.108*
H11B	0.3978	0.0637	0.9038	0.108*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.03731 (7)	0.05062 (8)	0.04559 (8)	0.000	-0.00706 (5)	0.000
Cu1	0.03802 (12)	0.05125 (14)	0.04003 (12)	0.000	-0.00415 (9)	0.000
Cl1	0.0925 (4)	0.1280 (6)	0.0491 (2)	-0.0450 (4)	-0.0017 (2)	0.0181 (3)
N1	0.0443 (6)	0.0406 (5)	0.0381 (5)	0.0015 (4)	-0.0004 (4)	-0.0020 (4)
C1	0.0583 (9)	0.0507 (8)	0.0454 (7)	-0.0190 (7)	0.0027 (6)	-0.0049 (6)
C2	0.0583 (9)	0.0682 (10)	0.0433 (7)	-0.0221 (8)	0.0003 (6)	-0.0066 (7)
C3	0.0507 (8)	0.0683 (10)	0.0444 (7)	-0.0135 (7)	0.0022 (6)	0.0042 (7)
C4	0.0625 (10)	0.0636 (10)	0.0548 (9)	-0.0268 (8)	0.0007 (7)	0.0043 (8)
C5	0.0539 (8)	0.0520 (8)	0.0492 (8)	-0.0170 (7)	-0.0044 (6)	-0.0023 (6)
C6	0.0416 (6)	0.0381 (6)	0.0435 (6)	-0.0026 (5)	-0.0005 (5)	-0.0035 (5)
C7	0.0496 (7)	0.0396 (6)	0.0425 (6)	-0.0034 (5)	-0.0019 (5)	-0.0060 (5)
C8	0.0578 (8)	0.0509 (8)	0.0372 (6)	0.0027 (6)	0.0037 (6)	-0.0045 (6)
C9	0.0502 (11)	0.0543 (11)	0.0320 (8)	0.000	0.0000 (7)	0.000
C10	0.0463 (12)	0.0775 (17)	0.0611 (14)	0.000	-0.0050 (10)	0.000
C11	0.102 (2)	0.0794 (19)	0.0336 (10)	0.000	-0.0029 (12)	0.000

Geometric parameters (Å, °)

II—Cu1	2.4607 (3)	C5—C6	1.388 (2)
Cu1—N1	2.0104 (12)	C5—H5A	0.9300
Cu1—N1 ⁱ	2.0104 (12)	C6—C7	1.462 (2)
Cl1—C3	1.7373 (17)	C7—H7A	0.9300
N1—C7	1.2739 (19)	C8—C9	1.540 (2)
N1—C8	1.4739 (18)	C8—H8A	0.9700
C1—C2	1.378 (2)	C8—H8B	0.9700
C1—C6	1.3998 (19)	C9—C10	1.528 (3)
C1—H1A	0.9300	C9—C8 ⁱ	1.540 (2)
C2—C3	1.372 (2)	C9—C11	1.542 (3)
C2—H2A	0.9300	C10—H10A	0.9600
C3—C4	1.379 (2)	C10—H10B	0.9601
C4—C5	1.380 (2)	C11—H11A	0.9600
C4—H4A	0.9300	C11—H11B	0.9600
N1—Cu1—N1 ⁱ	94.40 (7)	C1—C6—C7	123.89 (13)
N1—Cu1—II	132.30 (3)	N1—C7—C6	125.56 (13)
N1 ⁱ —Cu1—II	132.30 (3)	N1—C7—H7A	117.2
C7—N1—C8	116.98 (13)	C6—C7—H7A	117.2
C7—N1—Cu1	133.79 (10)	N1—C8—C9	113.84 (13)
C8—N1—Cu1	109.06 (10)	N1—C8—H8A	108.8
C2—C1—C6	121.10 (14)	C9—C8—H8A	108.8

C2—C1—H1A	119.4	N1—C8—H8B	108.8
C6—C1—H1A	119.4	C9—C8—H8B	108.8
C3—C2—C1	119.40 (15)	H8A—C8—H8B	107.7
C3—C2—H2A	120.3	C10—C9—C8 ⁱ	110.85 (12)
C1—C2—H2A	120.3	C10—C9—C8	110.85 (12)
C2—C3—C4	120.98 (16)	C8 ⁱ —C9—C8	112.74 (19)
C2—C3—C11	119.64 (13)	C10—C9—C11	109.8 (2)
C4—C3—C11	119.38 (13)	C8 ⁱ —C9—C11	106.21 (13)
C3—C4—C5	119.38 (15)	C8—C9—C11	106.21 (13)
C3—C4—H4A	120.3	C9—C10—H10A	109.5
C5—C4—H4A	120.3	C9—C10—H10B	109.5
C4—C5—C6	121.10 (14)	H10A—C10—H10B	109.5
C4—C5—H5A	119.5	C9—C11—H11A	109.4
C6—C5—H5A	119.5	C9—C11—H11B	109.5
C5—C6—C1	118.00 (14)	H11A—C11—H11B	109.5
C5—C6—C7	118.11 (13)		

Symmetry code: (i) $x, -y, z$.

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8A ⁱⁱ ⋯Cg1 ⁱⁱ	0.97	2.92	3.7220 (19)	141

Symmetry code: (ii) $-x+1/2, -y+1/2, -z+1$.