

trans-Bis[2-(2-bromobenzamido)-1,3-oxazoline(1-)-κ²N¹,O]copper(II)

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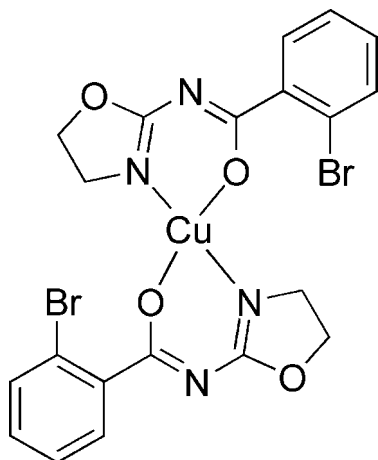
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 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.046; wR factor = 0.105; data-to-parameter ratio = 14.1.

In the mononuclear title complex, $[\text{Cu}(\text{C}_{10}\text{H}_8\text{BrN}_2\text{O}_2)_2]$, the Cu atom, which lies on a centre of symmetry, is in a square-planar geometry defined by a *trans*- N_2O_2 donor set.

Related literature

For related literature, see: Douglass & Dains (1934); Jiang *et al.* (2006a, 2006b); Kato *et al.* (2004); Paintner *et al.* (2005); Wipf & Miller (1992); Zhang *et al.* (2003).



Experimental

Crystal data

$[\text{Cu}(\text{C}_{10}\text{H}_8\text{BrN}_2\text{O}_2)_2]$
 $M_r = 599.73$
 Orthorhombic, *Pbca*
 $a = 11.4637$ (9) Å
 $b = 11.9265$ (9) Å
 $c = 14.9016$ (12) Å

$V = 2037.4$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 5.03$ mm⁻¹
 $T = 291$ (2) K
 $0.27 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{\min} = 0.31$, $T_{\max} = 0.45$

10306 measured reflections
 2006 independent reflections
 1580 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.105$
 $S = 1.08$
 2006 reflections

142 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.45$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ e Å⁻³

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2176).

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supplementary materials

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Comment

Oxazolines are important structural entities in natural products, can be used as protecting groups for carboxylic acids, and, as well, can be used as building blocks in organic synthesis (Wipf *et al.*, 1992). In addition, oxazolines have been proven to be powerful in transition metal-catalyzed organic synthesis (Paintner *et al.*, 2005; Kato *et al.*, 2004). Despite this interest, there are only two relevant structures that have been reported (Zhang *et al.*, 2003; Jiang *et al.*, 2006a,b). In order to extend the work on these complexes, the title mononuclear copper(II) complex, (I), is described herein.

The 2-bromo-*N*-(4,5-dihydrooxazol-2-yl)benzamide anion serves as a bidentate ligand and ligates the Cu atom through the carbonyl-O2 and oxazoline-N1 atoms. The Cu atom, which lies at a centre of inversion, adopts a square planar geometry within a *trans*-N₂O₂ donor set.

Experimental

1-(2-Bromobenzoyl)-3-(2-hydroxyethyl)thiourea was prepared according to literature (Douglass & Dains, 1934). This compound (2 mmol, 0.61 g) was dissolved in acetonitrile (20 ml). Dicyclohexylcarbodiimide (2 mmol, 0.22 g) and pyridine (0.02 mmol, 0.16 g), were added and the mixture was stirred for 12 h. The solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel to afford desired 2-bromo-*N*-(4,5-dihydrooxazol-2-yl)benzamide (yield 83%). To an ethanol solution (20 ml) of 2-bromo-*N*-(4,5-dihydrooxazol-2-yl)benzamide (1 mmol, 0.27 g) was added an ethanol solution (10 ml) of cupric(II) nitrate trihydrate (0.5 mmol, 0.12 g) with stirring. Black block-shaped crystals were formed at the bottom of the vessel upon slow evaporation of the solvent. The crystals were isolated, washed three times with ethanol and dried in a vacuum desiccator (yield 90%). Elemental analysis found: C 40.10, H 2.68, N 9.39%; C₂₀H₁₆Br₂CuN₄O₄ requires: C 40.05, H 2.69, N 9.37%.

Refinement

The H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances = 0.93–0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

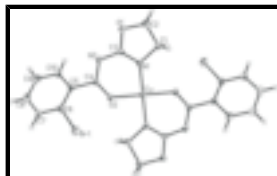


Fig. 1. The structure of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Unlabelled atoms are related to labelled atoms by $1 - x, -y, 2 - z$.

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Crystal data

[Cu(C ₁₀ H ₈ BrN ₂ O ₂) ₂]	$F_{000} = 1180$
$M_r = 599.73$	$D_x = 1.955 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
Hall symbol: -P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 11.4637 (9) \text{ \AA}$	Cell parameters from 4720 reflections
$b = 11.9265 (9) \text{ \AA}$	$\theta = 2.2\text{--}27.6^\circ$
$c = 14.9016 (12) \text{ \AA}$	$\mu = 5.03 \text{ mm}^{-1}$
$V = 2037.4 (3) \text{ \AA}^3$	$T = 291 (2) \text{ K}$
$Z = 4$	Block, blue
	$0.27 \times 0.20 \times 0.16 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2006 independent reflections
Radiation source: sealed tube	1580 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.059$
$T = 291(2) \text{ K}$	$\theta_{\text{max}} = 26.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -14 \rightarrow 14$
$T_{\text{min}} = 0.31$, $T_{\text{max}} = 0.45$	$k = -12 \rightarrow 14$
10306 measured reflections	$l = -18 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
2006 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
142 parameters	$\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.55307 (4)	0.37304 (4)	0.91935 (4)	0.04221 (18)
C1	0.6284 (4)	-0.1319 (4)	1.1432 (4)	0.0517 (15)
H1A	0.5970	-0.2010	1.1188	0.062*
H1B	0.5843	-0.1113	1.1962	0.062*
C2	0.7570 (4)	-0.1433 (4)	1.1642 (4)	0.0394 (11)
H2A	0.7695	-0.1511	1.2283	0.047*
H2B	0.7900	-0.2079	1.1340	0.047*
C3	0.7293 (4)	0.0068 (3)	1.0760 (3)	0.0273 (8)
C4	0.7008 (4)	0.1421 (3)	0.9690 (3)	0.0285 (9)
C5	0.7599 (4)	0.2323 (3)	0.9150 (3)	0.0284 (9)
C6	0.7067 (4)	0.3302 (4)	0.8842 (3)	0.0315 (9)
C7	0.7635 (4)	0.4066 (4)	0.8315 (3)	0.0395 (11)
H7	0.7256	0.4713	0.8125	0.047*
C8	0.8791 (5)	0.3862 (5)	0.8064 (4)	0.0469 (13)
H8	0.9185	0.4371	0.7700	0.056*
C9	0.9347 (4)	0.2900 (5)	0.8359 (4)	0.0457 (12)
H9	1.0115	0.2757	0.8195	0.055*
C10	0.8756 (4)	0.2159 (4)	0.8896 (3)	0.0356 (10)
H10	0.9143	0.1521	0.9098	0.043*
Cu	0.5000	0.0000	1.0000	0.0329 (2)
N1	0.6269 (3)	-0.0407 (3)	1.0754 (3)	0.0360 (9)
N2	0.7688 (3)	0.0945 (3)	1.0295 (3)	0.0322 (8)
O1	0.8085 (3)	-0.0406 (3)	1.1313 (2)	0.0365 (7)
O2	0.5968 (3)	0.1175 (2)	0.9485 (2)	0.0373 (8)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0329 (3)	0.0295 (3)	0.0642 (4)	0.00341 (19)	0.0062 (2)	-0.0014 (2)
C1	0.035 (3)	0.048 (3)	0.073 (4)	-0.013 (2)	-0.015 (3)	0.030 (3)
C2	0.037 (3)	0.028 (2)	0.053 (3)	0.0020 (18)	-0.007 (2)	0.008 (2)
C3	0.0254 (19)	0.026 (2)	0.031 (2)	0.0027 (17)	-0.0070 (16)	-0.0004 (17)
C4	0.023 (2)	0.0236 (19)	0.039 (2)	-0.0005 (16)	-0.0003 (18)	0.0049 (16)
C5	0.026 (2)	0.027 (2)	0.033 (2)	-0.0025 (16)	-0.0076 (18)	-0.0024 (16)
C6	0.029 (2)	0.028 (2)	0.037 (2)	-0.0023 (17)	0.0001 (19)	0.0004 (18)
C7	0.037 (2)	0.027 (2)	0.055 (3)	-0.0020 (19)	-0.001 (2)	0.013 (2)
C8	0.037 (3)	0.052 (3)	0.052 (3)	-0.011 (2)	0.010 (2)	0.010 (2)

supplementary materials

C9	0.028 (2)	0.057 (3)	0.052 (3)	-0.001 (2)	0.002 (2)	0.012 (3)
C10	0.025 (2)	0.032 (2)	0.050 (3)	-0.0009 (17)	-0.0042 (19)	0.0025 (19)
Cu	0.0224 (3)	0.0276 (4)	0.0486 (5)	-0.0066 (3)	-0.0093 (3)	0.0110 (3)
N1	0.0219 (18)	0.0317 (19)	0.054 (3)	-0.0098 (15)	-0.0098 (16)	0.0111 (18)
N2	0.0247 (18)	0.0276 (17)	0.044 (2)	-0.0064 (15)	-0.0043 (16)	0.0052 (16)
O1	0.0267 (15)	0.0387 (16)	0.0442 (18)	-0.0036 (13)	-0.0134 (14)	0.0090 (14)
O2	0.0254 (16)	0.0268 (16)	0.060 (2)	-0.0065 (12)	-0.0076 (15)	0.0156 (14)

Geometric parameters (Å, °)

Br1—C6	1.907 (4)	C5—C10	1.393 (6)
C1—N1	1.484 (6)	C5—C6	1.395 (6)
C1—C2	1.513 (6)	C6—C7	1.368 (6)
C1—H1A	0.9700	C7—C8	1.399 (7)
C1—H1B	0.9700	C7—H7	0.9300
C2—O1	1.446 (5)	C8—C9	1.385 (8)
C2—H2A	0.9700	C8—H8	0.9300
C2—H2B	0.9700	C9—C10	1.371 (7)
C3—N1	1.304 (6)	C9—H9	0.9300
C3—N2	1.333 (5)	C10—H10	0.9300
C3—O1	1.350 (5)	Cu—N1 ⁱ	1.901 (4)
C4—O2	1.265 (5)	Cu—N1	1.901 (4)
C4—N2	1.319 (5)	Cu—O2	1.945 (3)
C4—C5	1.504 (6)	Cu—O2 ⁱ	1.945 (3)
N1—C1—C2	102.6 (4)	C6—C7—C8	119.3 (5)
N1—C1—H1A	111.2	C6—C7—H7	120.4
C2—C1—H1A	111.2	C8—C7—H7	120.4
N1—C1—H1B	111.2	C9—C8—C7	119.7 (5)
C2—C1—H1B	111.2	C9—C8—H8	120.2
H1A—C1—H1B	109.2	C7—C8—H8	120.2
O1—C2—C1	104.6 (4)	C10—C9—C8	119.5 (4)
O1—C2—H2A	110.8	C10—C9—H9	120.2
C1—C2—H2A	110.8	C8—C9—H9	120.2
O1—C2—H2B	110.8	C9—C10—C5	122.6 (4)
C1—C2—H2B	110.8	C9—C10—H10	118.7
H2A—C2—H2B	108.9	C5—C10—H10	118.7
N1—C3—N2	130.0 (4)	N1 ⁱ —Cu—N1	180.000 (1)
N1—C3—O1	115.3 (4)	N1 ⁱ —Cu—O2	91.11 (15)
N2—C3—O1	114.7 (4)	N1—Cu—O2	88.89 (15)
O2—C4—N2	128.4 (4)	N1 ⁱ —Cu—O2 ⁱ	88.89 (15)
O2—C4—C5	117.4 (4)	N1—Cu—O2 ⁱ	91.11 (15)
N2—C4—C5	114.0 (4)	O2—Cu—O2 ⁱ	180.00 (12)
C10—C5—C6	116.4 (4)	C3—N1—C1	107.7 (4)
C10—C5—C4	118.3 (4)	C3—N1—Cu	125.5 (3)
C6—C5—C4	125.3 (4)	C1—N1—Cu	126.8 (3)
C7—C6—C5	122.5 (4)	C4—N2—C3	119.5 (4)
C7—C6—Br1	114.7 (3)	C3—O1—C2	106.7 (3)

C5—C6—Br1

122.6 (3)

C4—O2—Cu

127.6 (3)

Symmetry codes: (i) $-x+1, -y, -z+2$.

Fig. 1

