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(Pentane-2,4-dionato-κ^2O,O')(pyridin-2-amine-κN^1)copper(II) and (pentane-2,4-dionato-κ^2O,O')(pyrimidin-2-amine-κN^1)copper(II)

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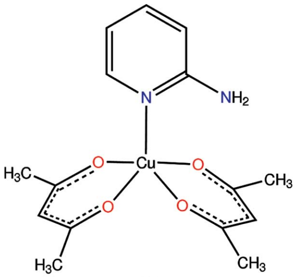
The title compounds, $[Cu(C_5H_7O_2)_2(C_5H_6N_2)]$, (I), and $[Cu(C_5H_7O_2)_2(C_4H_5N_3)]$, (II), were prepared by the reaction of bis(pentane-2,4-dionato-κ^2O,O')copper(II) with pyridin-2-amine and pyrimidin-2-amine, respectively. From a chemical point of view, it is interesting that no Schiff base formation was observed. The compounds are isostructural, with both having a square-pyramidal coordination of the Cu^{II} atom and intramolecular N—H⋯O hydrogen bonding. The additional N atom of the pyrimidin-2-amine ligand is not involved in hydrogen bonding or in metal coordination. In the crystal structure, chelate rings are involved in π–π interactions and molecules of (I) are linked together *via* N—H⋯O hydrogen bonds.

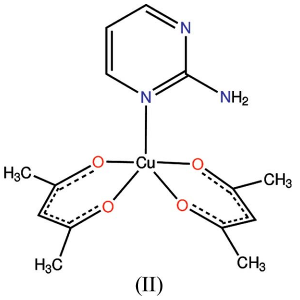
Comment

Metal β-diketonate compounds have attracted great interest, because metal complexes of β-diketonate derivatives can be used as precursors in metal–organic chemical vapour deposition (MOCVD) (Kodas & Hampden-Smith, 1994) or as building blocks for the design of porous and supramolecular materials (Bray *et al.*, 2007; Garibay *et al.*, 2009). β-Diketonates and organic compounds containing an amino group are suitable starting materials for the formation of Schiff bases (Bourget-Merle *et al.*, 2002; Holm *et al.*, 1966). Pyridin-2-amine and pyrimidin-2-amine are close analogues of purine and pyrimidine nucleobases and are thus interesting as model compounds. They can act both as metal-coordinating ligands through the aromatic N atom and as hydrogen-bond donors through the amino group. Furthermore, pyrimidin-2-amine has an additional aromatic N atom that can serve as a second ligation centre or as a strong hydrogen-bond acceptor. When we examined the reactivity of bis(pentane-2,4-dionato-O,O')-copper(II) with pyridin-2-amine and pyrimidin-2-amine, no Schiff base formation was observed under the applied reaction conditions; instead both compounds acted as a nitrogen-donor ligand, occupying the fifth coordination site *via* an aromatic N atom.

The title compounds, namely (pentane-2,4-dionato-κ^2O,O')-(pyridin-2-amine-κN^1)copper(II), (I) (Fig. 1), and (pentane-2,4-dionato-κ^2O,O')(pyrimidin-2-amine-κN^1)copper(II), (II) (Fig. 2), have similar molecular structures. In the equatorial plane, the Cu atom is surrounded by four O atoms of two chelating pentane-2,4-dionate ligands. Selected bond lengths and angles are listed in Tables 1 and 3, and are typical for β-diketonate compounds (Gromilov & Baidina, 2004; Stabnikov *et al.*, 2008; Germán-Acacio *et al.*, 2009). The Cu atom lies above the plane formed by four acetylacetonate O atoms, by 0.219 (1) Å in (I) and 0.174 (1) Å in (II). The fifth coordination site is occupied by the additional ligand in the axial position. The geometry is square pyramidal with only slight distortion. The distortion of a square pyramid can be best described by the structural parameter $\tau = (\beta - \alpha)/60°$, where β and α are the largest angles in the coordination sphere ($\tau = 0$ for a square-pyramid and $\tau = 1$ for a trigonal bipyramid; Addison *et al.*, 1984), which in this case has values of 0.05 and 0.04 for (I) and (II), respectively. For five-coordinate β-dike-



(I)



(II)

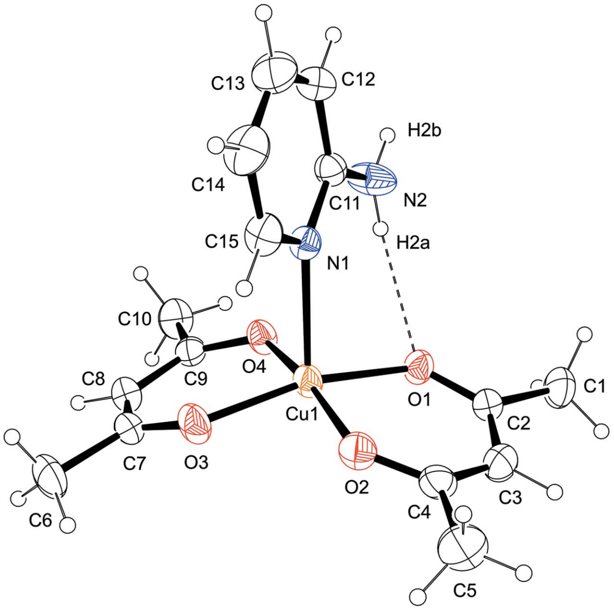


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

tonates, square-pyramidal geometry is more common (Bray *et al.*, 2007; Garibay *et al.*, 2009) than trigonal bipyramidal. But still, the three closely related compounds (4-dimethylaminopyridine-κ*N*)bis(pentane-2,4-dionato-κ^2O,O')copper(II), (4-aminopyridine-κ*N*)bis(pentane-2,4-dionato-κ^2O,O')copper(II) and (4-dimethylaminopyridine-κ*N*)bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato-κ^2O,O')copper(II) have trigonal–bipyramidal geometry (Lindoy *et al.*, 2006; Stabnikov *et al.*, 2011). The Cu—N bond length is 2.3287 (17) Å in (I) and 2.3672 (19) Å in (II), significantly greater than in the above-mentioned trigonal–bipyramidal compounds [2.119 (2), 2.008 (2) and 1.978 (3) Å, respectively; Lindoy *et al.*, 2006; Stabnikov *et al.*, 2011]. However, they are similar to those in square-pyramidal copper(II) pentane-2,4-dionate complexes with adenine (Zaworotko *et al.*, 2009), quinoline (Jose *et al.*, 1969) and isonicotinamide (Germán-Acacio *et al.*, 2009), where the bond lengths are 2.328 (5), 2.36 (1) and 2.384 (2) Å, respectively. The molecular structure is by no means easy to predict. Coordination geometry can be influenced by electronic, steric and packing effects. Even small changes in noncovalent bonding, such as hydrogen-bonding and π–π interactions, might lead to different coordination geometries. Quantum-mechanical studies would be needed to understand the differences in geometries.

In both (I) and (II), the two acetylacetonate ligands are not coplanar. The angle formed between the Cu1/O1/C2–C4/O2 and Cu1/O3/C6–C8/O4 rings is 30.84 (8)° in (I) and 26.89 (8)° in (II). The pyridine and pyrimidine rings are not perpendicular to the O1/O2/O3/O4 plane, but form an angle of 81.72 (10)° in (I) and 81.35 (10)° in (II). Furthermore, the pyridine and pyrimidine rings are slightly rotated towards the O1 atom, as can be seen by comparing the O1—Cu1—N1—C11 and O4—Cu1—N1—C11 torsion angles [42.55 (16) and −44.05 (16)°, respectively, in (I), and 42.65 (19) and −44.10 (18)°, respectively, in (II)]. Owing to this rotation, the amino groups in both compounds form an intramolecular hydrogen bond with the O1 atom and not with the O4 atom (Tables 2 and 4). The graph-set motif is *S*(6) (Bernstein *et al.*, 1995). In compound (I), a chain is formed due to the intermolecular bifurcated hydrogen bonds N2—H2*B*···O2($x+1, y, z$) and N2—H2*B*···O3($x+1, y, z$); the graph-set motif is $C_1^2(6)[R_1^2(4)]$ (Fig. 3). Although the packing in (II) is similar, no intermolecular hydrogen bonding was observed owing to a somewhat larger separation between atom N2 and atoms O2 and O3 of an adjacent molecule, *viz.* 3.248 (3) and 3.301 (3) Å in (I), and 3.339 (3) Å and 3.322 (3) Å in (II). The H2*B*···O2($x+1, y, z$) and H2*B*···O3($x+1, y, z$) hydrogen-bond separations in (I) are 2.48 and 2.56 Å, respectively; the separations are, however, slightly longer in (II), where both contacts are 2.60 Å. The degree of similarity between crystals can be defined, for example, by the unit-cell similarity index (Π). In the event of great similarity of two unit cells, the index Π is practically zero (Kálmán *et al.*, 1993), as can be observed also for (I) and (II) with $\Pi = 0.016$. Since the compounds are

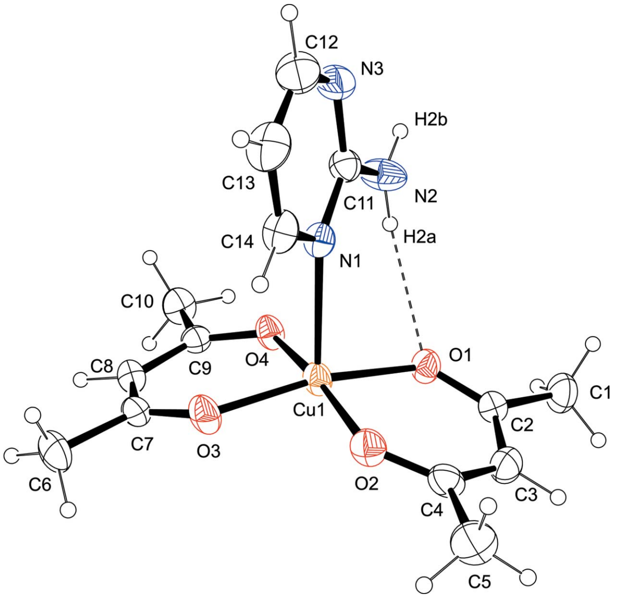


Figure 2
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

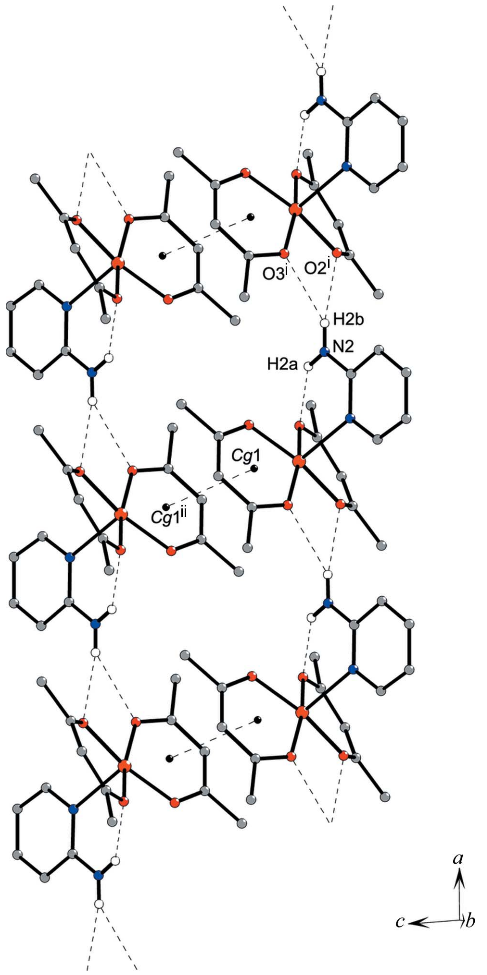


Figure 3
A packing diagram for (I). Dashed lines indicate hydrogen bonds and π–π interactions. For the sake of clarity, H atoms not involved in the motif shown have been omitted. [Symmetry codes: (i) $x + 1, y, z$; (ii) $-x, -y, -z + 1$.]

isostructural, the interactions should also be similar in both cases. The crystal packing in both compounds is similar also, because pyrimidine atom N3 in compound (II), which could act as an electron donor, is not involved in hydrogen bonding or in metal coordination. For comparison, both N atoms of the pyrimidine ring are involved in the coordination to the copper metal centres in bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato)copper(II) adducts with pyrimidine and 4-methylpyrimidine (Yasui *et al.*, 2001).

In both square-pyramidal compounds (I) and (II), there are π–π interactions between two parallel neighbouring Cu1/O3/C7–C9/O4 chelate rings, with a $Cg1\cdots Cg1(-x, -y, -z + 1)$ centroid–centroid distance of 3.7106 (11) Å, a perpendicular distance from the centroid *Cg*1 to the plane of the other ring of 3.5682 (8) Å and a slippage between the centroids of 1.018 Å in compound (I). In compound (II), these values are even smaller, since two parallel neighbouring Cu1/O3/C7–C9/O4 rings have a $Cg1\cdots Cg1(-x, -y + 1, -z + 2)$ centroid–centroid distance of 3.5310 (11) Å, a perpendicular distance from the centroid *Cg*1 to the plane of the other ring of 3.4535 (8) Å and a slippage between the centroids of 0.736 Å. Such interactions would be consistent with well defined π–π stacking interactions in organic aromatic compounds (Hunter, 1994; Choudhury & Chitra, 2010; Malathy Sony & Ponnuswamy, 2006; Perdih & Perdih, 2011), although the interplanar separations are somewhat greater than the graphite spacing of 3.35 Å (Bacon, 1951). Janiak (2000) classified π–π interactions in nitrogen-containing heteroaromatic compounds where such values would indicate strong interactions, since strong interactions exhibit rather short centroid–centroid contacts ($Cg\cdots Cg$ < 3.8 Å), small slip angles (<25°) and vertical displacements (<1.5 Å), which translate into a sizeable overlap of the aromatic planes. In comparison, medium-to-weak interactions exhibit rather long centroid–centroid distances (>4.0 Å) together with large slip angles (>30°) and vertical displacements (>2.0 Å) (Janiak, 2000; Yang *et al.*, 2005; Dorn *et al.*, 2005). It is arguable whether aromatic π–π interactions are comparable with those observed in (I) and (II), since π-delocalized β-diketonate chelate rings have little or no aromatic character (Cotton *et al.*, 1999). The necessity of the aromaticity for π–π interactions was also questioned by Bloom & Wheller (2011). Detailed analysis of X-ray diffraction data on copper β-diketonates and quantum-mechanical studies would be needed in order to study the nature as well as the factors influencing the π–π interactions in β-diketonates.

Strong π–π interactions are very common in square-planar bis(β-diketonato)copper(II) compounds. For example, in *trans*-bis(benzoylacetonato)copper(II), bis(pentane-2,4-dionato)copper(II) and bis(3-benzylpentane-2,4-dionato)copper(II), centroid–centroid contacts are 3.13, 3.14 and 3.2198 (13) Å, respectively, with parallel metallacycles [or deviating from planarity by 0.03 (7)°] (Hon *et al.*, 1966; Lebrun *et al.*, 1986; Judaš & Kaitner, 2006*a*). Strong π–π interactions are also present in some square-pyramidal compounds of copper(II) β-diketonates. The shortest interactions were found in *catena*-[3-cyanopentane-2,4-dionato-*O*,*O*′,*N*)(3-cyanopentane-2,4-dionato-*O*,*O*′)copper(II), with centroid–centroid contacts between two parallel metallacycles of 3.16 Å (Angelova *et al.*, 1989). Centroid–centroid distances in the range 3.23–3.49 Å with parallel metallacycles (or deviating from planarity by up to 0.03°) were observed in several examples (Lee *et al.*, 2001; Zaworotko *et al.*, 2009; Delgado *et al.*, 2007; Jose *et al.*, 1969; Stabnikov *et al.*, 2008; Caneschi *et al.*, 1988; Song & Iyoda, 2009; Judaš & Kaitner, 2006*b*; Yoshida *et al.*, 2008; Atienza *et al.*, 2008). Interestingly, when searching for centroid–centroid distances in the range 3.50–4.00 Å, as observed in compounds (I) and (II), we found only two structures, *viz.* a tricopper complex where the centroid–centroid distance is 3.66 Å and the angle between metallacycles is 0.02° (Maxim *et al.*, 2010), and (isonicotinamide-*N*)bis(pentane-2,4-dionato-*O*,*O*′)copper(II), with a centroid–centroid distance of 3.90 Å and an angle between metallacycles of 6.97° (Germán-Acacio *et al.*, 2009).

Experimental

Cu(acac)$_2$ (acac is acetylacetonate; 0.25 mmol) was dissolved in warm chloroform (5 ml) and pyridin-2-amine (0.25 mmol) was added for the preparation of (I) and pyrimidin-2-amine (0.25 mmol) was added for the preparation of (II). The reaction mixture was stirred for 5 min and then allowed to stand at room temperature. Crystals suitable for X-ray analysis were obtained after slow evaporation of the solvent (yields: 50–55%).

Compound (I)

Crystal data

$[Cu(C_5H_7O_2)_2(C_5H_6N_2)]$
$M_r = 355.87$
Monoclinic, $P2_1/n$
$a = 7.5686$ (1) Å
$b = 8.2944$ (2) Å
$c = 26.4250$ (7) Å
$\beta = 93.944$ (1)°
$V = 1654.95$ (6) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 1.34$ mm^{-1}
$T = 293$ K
0.6 × 0.6 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.501$, $T_{max} = 0.731$
6316 measured reflections
3572 independent reflections
3238 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.106$
$S = 1.06$
3572 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Compound (II)

Crystal data

$[Cu(C_5H_7O_2)_2(C_4H_5N_3)]$
$M_r = 356.86$
Monoclinic, $P2_1/n$
$a = 7.5686$ (3) Å
$b = 8.3504$ (3) Å
$c = 25.6871$ (10) Å
$\beta = 93.081$ (1)°
$V = 1621.10$ (11) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 1.37$ mm^{-1}
$T = 293$ K
0.55 × 0.38 × 0.13 mm

Table 1
Selected geometric parameters (Å, °) for (I).

Cu1—O3	1.9382 (15)	Cu1—O1	1.9460 (14)
Cu1—O4	1.9385 (15)	Cu1—N1	2.3287 (17)
Cu1—O2	1.9433 (15)		
O3—Cu1—O4	92.98 (6)	O2—Cu1—O1	92.49 (7)
O3—Cu1—O2	85.42 (7)	O3—Cu1—N1	98.10 (7)
O4—Cu1—O2	168.65 (7)	O4—Cu1—N1	93.37 (6)
O3—Cu1—O1	165.46 (7)	O2—Cu1—N1	97.98 (7)
O4—Cu1—O1	86.24 (6)	O1—Cu1—N1	96.45 (6)

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	2.29	3.054 (3)	149
N2—H2*B*···O2^{i}	0.86	2.48	3.248 (3)	149
N2—H2*B*···O3^{i}	0.86	2.56	3.301 (3)	144

Symmetry code: (i) $x + 1, y, z$.

Table 3
Selected geometric parameters (Å, °) for (II).

Cu1—O3	1.9317 (16)	Cu1—O2	1.9428 (16)
Cu1—O4	1.9352 (15)	Cu1—N1	2.3672 (19)
Cu1—O1	1.9397 (15)		
O3—Cu1—O4	93.31 (7)	O1—Cu1—O2	92.61 (7)
O3—Cu1—O1	168.59 (8)	O3—Cu1—N1	95.35 (7)
O4—Cu1—O1	86.48 (6)	O4—Cu1—N1	92.69 (7)
O3—Cu1—O2	85.76 (7)	O1—Cu1—N1	96.05 (7)
O4—Cu1—O2	170.76 (7)	O2—Cu1—N1	96.55 (7)

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	2.35	3.116 (3)	149

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.520$, $T_{\rm max} = 0.842$
6985 measured reflections
3658 independent reflections
3131 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.093$
$S = 1.1$
3658 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.38$ e Å^{-3}

Due to the chosen measurement strategy, 182 strong reflections for (I) and 48 for (II) are missing due to detector overflow. All H atoms were initially located in difference Fourier maps and were subsequently treated as riding atoms in geometrically idealized positions, with C—H = 0.93 (aromatic and alkenyl) or 0.96 Å (CH_3), and N—H = 0.86 Å, and with $U_{\rm iso}({\rm H}) = kU_{\rm eq}({\rm C,N})$, where $k = 1.5$ for amino and methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. To improve the refinement results, two reflections in the case of (I) and eight reflections in the case of (II) with too high values of $\delta(F^2)$/e.s.d. and with $F_o^2 < F_c^2$ were deleted from the refinement.

For both compounds, data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KU3061). Services for accessing these data are described at the back of the journal.

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