

All H atoms refined  
 $w = 1/[\sigma^2(F_o) + 0.00001|F_o|^2]$   
 $(\Delta/\sigma)_{\max} = 0.01$

Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

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Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Sn—C11	2.436 (1)	N1—C	1.314 (5)
Sn—C12	2.429 (1)	N2—C	1.316 (5)
Sn—C13	2.422 (1)	N3—C	1.312 (4)
C11—Sn—C11 <sup>1</sup>	88.35 (5)	C12—Sn—C13	89.90 (4)
C11—Sn—C12	91.05 (3)	C12—Sn—C13 <sup>1</sup>	90.21 (4)
C11—Sn—C12 <sup>2</sup>	88.84 (3)	C13—Sn—C13 <sup>1</sup>	91.24 (6)
C11—Sn—C13	90.21 (3)	N1—C—N2	120.3 (3)
C11—Sn—C13 <sup>2</sup>	178.07 (4)	N1—C—N3	120.0 (4)
C12—Sn—C12 <sup>2</sup>	179.84 (6)	N2—C—N3	119.7 (4)

Symmetry code: (i)  $-x, y, \frac{1}{2} - z$ .

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 $\cdots$ C12	0.81 (3)	2.63 (4)	3.414 (4)	164 (4)
N1—H2 $\cdots$ C11 <sup>1</sup>	0.83 (3)	2.63 (3)	3.429 (4)	162 (3)
N2—H3 $\cdots$ C13 <sup>1</sup>	0.84 (4)	2.63 (4)	3.456 (4)	170 (4)
N2—H4 $\cdots$ C12 <sup>11</sup>	0.87 (3)	2.56 (4)	3.413 (4)	168 (4)
N3—H5 $\cdots$ C11 <sup>11</sup>	0.77 (3)	2.66 (3)	3.421 (4)	166 (3)
N3—H6 $\cdots$ C13	0.84 (4)	2.62 (4)	3.439 (4)	167 (3)

Symmetry codes: (i)  $\frac{1}{2} + x, \frac{1}{2} + y, z$ ; (ii)  $x, 1 + y, z$ .

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN for Windows*. Software used to prepare material for publication: *TEXSAN for Windows*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1486). Services for accessing these data are described at the back of the journal.

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## Diaquabis(hydrogen phthalato)copper(II), a new phase

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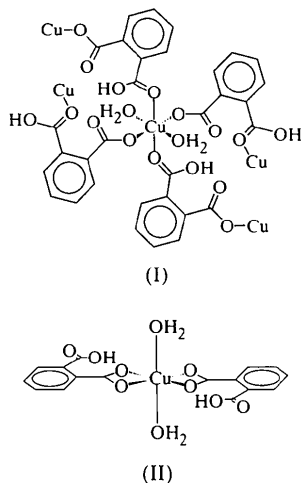
## Abstract

In the title compound,  $\beta$ -[Cu(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]<sub>n</sub>, the Cu atom lies on an inversion centre and has a tetragonal distortion from octahedral symmetry. Each hydrogen phthalate ligand is coordinated to two metals, forming a polymeric structure. The aromatic ring and the carboxylate group form an approximate plane, with the carboxylic acid group plane almost perpendicular to it. An intermolecular hydrogen bond between hydrogen phthalate groups is observed, with an O $\cdots$ O distance of 2.614 (2)  $\text{\AA}$ .

## Comment

The structure of  $\alpha$ -[Cu(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]<sub>n</sub> (II), has been reported by Goeta *et al.* (1993), Bartl & Küppers (1980) (neutron data), Cingi *et al.* (1969) and Cingi & Magnano (1959). In that phase, as well as in structures such as [Co(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>] $\cdot$ 6H<sub>2</sub>O (Küppers, 1990), [Mg(H<sub>2</sub>O)<sub>6</sub>](C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub> $\cdot$ 2H<sub>2</sub>O (Kariuki & Jones, 1989), [Li(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)] $\cdot$ H<sub>2</sub>O (Küppers *et al.*, 1985) and [Li(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)] $\cdot$ CH<sub>3</sub>OH (Küppers *et al.*, 1981), the ligand shows a planar configuration, having a short intramolecular hydrogen bond between the two carboxylate groups, with O $\cdots$ O distances of around 2.4  $\text{\AA}$ . On the other hand, crystals of K(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>) $\cdot$ 0.5H<sub>2</sub>O (Kariuki & Jones, 1995), [Co(H<sub>2</sub>O)<sub>6</sub>](C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub> (Kariuki & Jones, 1993) and [N(CH<sub>3</sub>)<sub>4</sub>](C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>) (Jessen, 1990) do exhibit an intermolecular hydrogen bond once the carboxylate and carboxylic acid groups form two planes approximately perpendicular to each other. The structure reported here,  $\beta$ -[Cu(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]<sub>n</sub> (I), belongs to the latter

group, *i.e.* hydrogen phthalate anions are linked through a moderate hydrogen bond, with the two O atoms separated by 2.614 (2) Å. This structure is isomorphous with the manganese compound (Bats *et al.*, 1978). Therefore, diaquabis(hydrogen phthalato)copper(II) has at least two phases in the solid state, one presenting a short and the other a long hydrogen bond. The metal alone is probably not responsible for the type of hydrogen bonding.



The Cu atom lies on an inversion centre coordinated to six O atoms. The arrangement is a tetragonal dis-

tortion from octahedral symmetry, as shown in Fig. 1 and Table 1. The aromatic ring has similar C—C distances and angles, with mean values of 1.382 (2) Å and 120.0 (2)°, respectively. As can be seen in Table 1, the carboxylate group has two C—O bond distances with the same value. On the other hand, it is easy to distinguish between the single and double bonds in the carboxylic acid group. The aromatic ring forms angles of 7.19 (9) and 89.74 (5)° with the carboxylate and carboxylic acid groups, respectively. Only O4 acts as an acceptor of all three hydrogen bonds (Fig. 1 and Table 2); one of them is weak and involves water, while the other two are of moderate strength, with one forming a six-membered ring (Cu, O3, C7, O4, HW1 and O5) and the other approximately in the plane of this ring (Fig. 1).

It is helpful to make a comparison between the two phases of [Cu(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]. In the  $\alpha$ -phase (previous work, short hydrogen bond), the whole ligand is planar. In the  $\beta$ -phase (present work, long hydrogen bond), the carboxylate group and the aromatic ring are in a plane perpendicular to the plane defined by the carboxylic acid group. However, it is interesting to note that these different configurations do not result in changes in the interatomic distances of equivalent bonds in the ligand, except in the hydrogen bond. In the  $\alpha$ -phase, the metal is coordinated to six O atoms in a highly distorted octahedral geometry; Cu—O<sub>axial</sub> bond

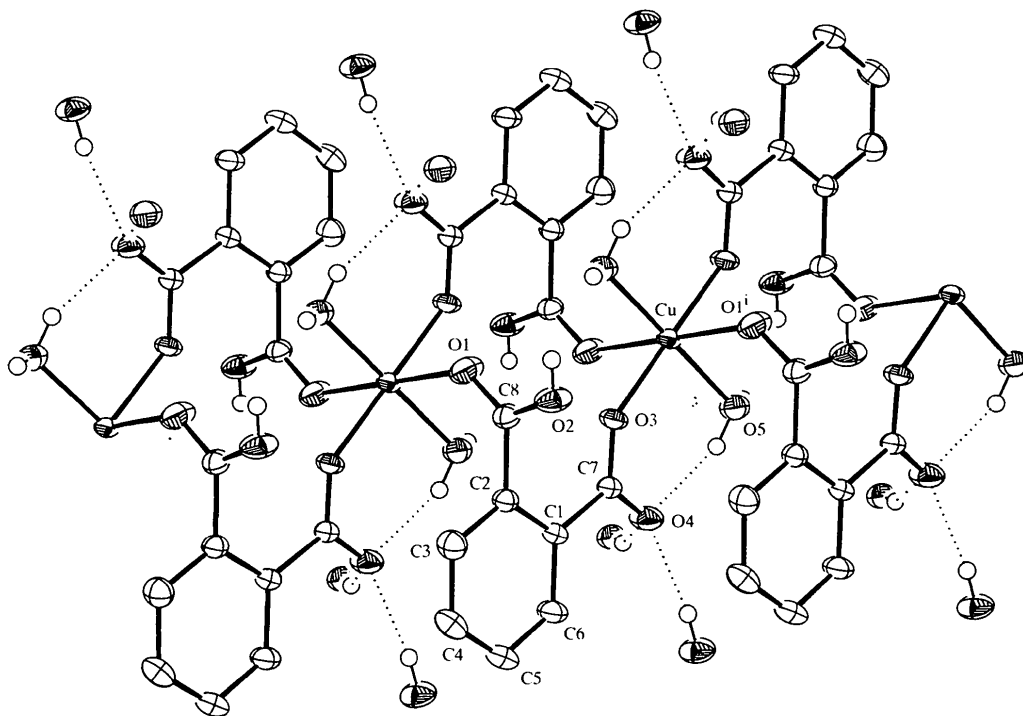


Fig. 1. ORTEP (Burnett & Johnson, 1996) drawing of the crystal packing in (I), with displacement ellipsoids at the 50% probability level. Hydrogen bonds are drawn as dotted lines. [Symmetry code: (i)  $x, y - 1, z$ .]

lengths are 2.636 Å, as determined by Bartl & Küppers (1980). The angle between these long Cu—O bonds and the equatorial plane is 54.3°. On the other hand, in the  $\beta$ -phase, all O—Cu—O angles are close to 90° and the axial distortion is less pronounced (Fig. 1 and Table 1). It is also important to emphasize that in both phases, the bidentate ligands form polymeric structures. However, only in the  $\alpha$ -phase is the ligand a chelate, meaning that, in the  $\alpha$ -phase, the polymeric structure is maintained only by moderate hydrogen bonds, while in the  $\beta$ -phase, the metal—oxygen bonds also contribute to the polymeric structure (Fig. 1).

## Experimental

Blue single crystals of the title compound were obtained by addition of copper carbonate to an aqueous solution of phthalic acid in such a way that the resulting solution had a 20% excess of the acid. Prussian blue crystals of the  $\alpha$ -phase were also formed in the solution.

### Crystal data

[Cu(C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]

$M_r = 429.83$

Monoclinic

$P2_1/c$

$a = 13.177(2) \text{ \AA}$

$b = 5.111(1) \text{ \AA}$

$c = 12.972(2) \text{ \AA}$

$\beta = 116.63(1)^\circ$

$V = 780.9(2) \text{ \AA}^3$

$Z = 2$

$D_x = 1.828 \text{ Mg m}^{-3}$

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 37 reflections

$\theta = 5.0\text{--}14.0^\circ$

$\mu = 1.46 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prismatic

$0.28 \times 0.25 \times 0.14 \text{ mm}$

Blue

$R_{\text{int}} = 0.022$

$\theta_{\text{max}} = 30^\circ$

$h = -18 \rightarrow 17$

$k = -7 \rightarrow 1$

$l = -1 \rightarrow 18$

3 standard reflections

every 100 reflections

intensity decay: 1.1%

### Data collection

Siemens P4 diffractometer

$\theta$ - $2\theta$  scans

Absorption correction:

analytical (JANA98;

Petricek & Dusek, 1998)

$T_{\text{min}} = 0.68$ ,  $T_{\text{max}} = 0.85$

3065 measured reflections

2281 independent reflections

2000 reflections with

$I > 2\sigma(I)$

### Refinement

Refinement on  $F^2$

$R(F) = 0.029$

$wR(F^2) = 0.092$

$S = 1.11$

2193 reflections

146 parameters

Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$ ,  
where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$

Extinction correction:

*SHELXL93* (Sheldrick, 1993)

Extinction coefficient:

0.019 (3)

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Cu—O1'	2.461 (1)	C1—C6	1.396 (2)
Cu—O3	1.944 (1)	C1—C7	1.496 (2)
Cu—O5	1.974 (1)	C2—C3	1.392 (2)
O1—C8	1.210 (2)	C2—C8	1.497 (2)
O2—C8	1.312 (2)	C3—C4	1.387 (2)
O3—C7	1.256 (2)	C4—C5	1.375 (3)
O4—C7	1.256 (2)	C5—C6	1.383 (2)
C1—C2	1.393 (2)		
O1'—Cu—O3	91.57 (5)	C1—C2—C3	120.4 (1)
O1'—Cu—O5	94.05 (5)	C1—C2—C8	123.2 (1)
O3—Cu—O5	92.52 (5)	C2—C3—C4	119.9 (2)
Cu—O1'—C8'	130.7 (1)	C3—C4—C5	120.1 (1)
Cu—O3—C7	131.8 (1)	C4—C5—C6	120.1 (2)
C8—O2—HO2	108 (3)	C1—C6—C5	120.9 (2)
Cu—O5—HW1	108 (3)	O3—C7—O4	124.5 (1)
Cu—O5—HW2	117 (3)	O4—C7—C1	119.5 (2)
HW1—O5—HW2	108 (3)	O3—C7—C1	116.0 (3)
C2—C1—C6	118.6 (1)	O1—C8—C2	124.8 (1)
C2—C1—C7	121.3 (1)	O2—C8—C2	111.2 (1)
C6—C1—C7	120.1 (1)	O1—C8—O2	123.8 (1)
C3—C2—C8	116.4 (1)		

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

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O2—HO2...O4'	0.70 (4)	1.92 (4)	2.614 (2)	171 (4)
O5—HW1...O4	0.79 (4)	1.90 (4)	2.635 (2)	155 (4)
O5—HW2...O4''	0.76 (4)	2.34 (4)	3.060 (2)	159 (4)

Symmetry codes: (i)  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (ii)  $-x, y - \frac{1}{2}, -\frac{1}{2} - z$ .

Refined C—H distances are in the range 0.82 (4)–0.96 (4) Å.

Data collection: XSCANS (Siemens, 1991). Cell refinement: XSCANS. Data reduction: JANA98 (Petricek & Dusek, 1998). Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPIII (Burnett & Johnson, 1996). Software used to prepare material for publication: PLATON (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1480). Services for accessing these data are described at the back of the journal.

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## A rhenium(V)–arylimide species incorporating pyridine-2-alimine chelation

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### Abstract

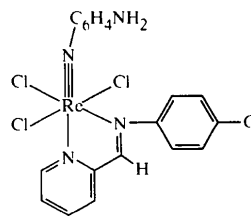
The title compound, (4-aminophenylimido)trichloro[2-(4-chlorophenyliminomethyl)pyridine]rhenium(V), [Re(NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>)Cl<sub>3</sub>(CIA)], where CIA is *N*-(*p*-chlorophenyl)pyridine-2-alimine (*p*-ClC<sub>6</sub>H<sub>4</sub>N=CHC<sub>5</sub>H<sub>4</sub>N), was synthesized by reacting the corresponding oxo complex, [ReOCl<sub>3</sub>(CIA)], or the corresponding phosphine oxide complex, [Re(OPPh<sub>3</sub>)Cl<sub>3</sub>(CIA)], with *p*-phenylenediamine in toluene. The structure determination revealed that the ReCl<sub>3</sub>N<sub>3</sub> coordination sphere is severely distorted from octahedral geometry and that it has a meridional ReCl<sub>3</sub> fragment. The Re—N—C angle in the imide part is nearly linear and the Re—N<sub>imide</sub> bond can be considered to be triple bond in character. The Re atom is distorted towards the imide N atom by 0.29 Å from the equatorial Cl<sub>3</sub>N<sub>imine</sub> plane. The Re—N<sub>pyridine</sub> bond is longer by 0.15 Å than the Re—N<sub>imine</sub> bond due to the *trans* influence of the imide fragment.

### Comment

High-valent rhenium complexes with metal–ligand multiple bonds, *e.g.* imido or oxo complexes, have been of increasing interest in inorganic chemistry (Wang *et al.*, 1993; Nugent & Mayer, 1988). In the course of our search for new and reactive oxorhenium(V) species, we isolated a pyridine-2-alimine-chelated Re<sup>V</sup>O species, (2), which undergoes O-atom transfer to

PPh<sub>3</sub>, affording an Re<sup>III</sup>OPPh<sub>3</sub> species, (3) (Dirghangi, Menon, Pramanik & Chakravorty, 1997). Both (2) and (3) were found to react with ArNH<sub>2</sub>, furnishing Re<sup>V</sup>NAr species which undergo inward O-atom transfer from water followed by metal oxidation to give rare Re<sup>V</sup>NAr species incorporating 2-picolinamide chelation (Dirghangi, Menon, Banerjee & Chakravorty, 1997; Banerjee *et al.*, 1997). This work forms part of a program on the synthesis and characterization of new Re<sup>V</sup>NAr compounds.

The species of concern to us here is [Re(NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>)Cl<sub>3</sub>(CIA)], (1), where CIA is *N*-(*p*-chlorophenyl)pyridine-2-alimine (*p*-ClC<sub>6</sub>H<sub>4</sub>N=CHC<sub>5</sub>H<sub>4</sub>N). This was



(1)

synthesized from the oxo complex [ReOCl<sub>3</sub>(CIA)], (2), or from the phosphine oxide complex [Re(OPPh<sub>3</sub>)Cl<sub>3</sub>(CIA)], (3), with *p*-phenylenediamine in toluene. A view of the molecular structure of (1) is shown in Fig. 1. The ReCl<sub>3</sub>N<sub>3</sub> coordination sphere is severely distorted from octahedral geometry. The ReCl<sub>3</sub> fragment is meridionally disposed. The chelate ring [bite angle 73.8 (2)°] along with the pyridine ring constitute a good plane (mean deviation 0.05 Å) with which the chlorophenyl group makes a dihedral angle of 61.6 (3)°. The aryl group of the imide fragment makes a dihedral angle of 39.5 (4)° with the chlorophenyl group.

An interesting feature of the structure is that the pyridine and imide N atoms (N1 and N3, respectively) bend towards each other in the direction of the Re/Cl<sub>2</sub>/N<sub>2</sub> plane. Additionally, the metal atom is displaced from the N<sub>2</sub>/Cl<sub>1</sub>/Cl<sub>2</sub>/Cl<sub>3</sub> mean plane by 0.29 Å towards N<sub>3</sub>. This distortion and bending is necessarily reflected in deviations of the *trans* angles from 180° [range 162.8 (1)–165.2 (2)°] and in deviations of the *cis* angles with respect to N1 and N3 from 90° to the lower and higher side, respectively. Other *cis* angles are very close to 90°. This type of metal deviation is common to multiple-bonded metal–ligand complexes (Dirghangi, Menon, Banerjee & Chakravorty, 1997; Shivakumar *et al.*, 1998; Bélanger & Beauchamp, 1999; Banerjee *et al.*, 1997; Lahiri *et al.*, 1987; Bakir & Sullivan, 1995).

Among hexacoordinated Re<sup>V</sup>NX (X is alkyl or aryl) structures (Bakir *et al.*, 1992; Masood *et al.*, 1994; Fung *et al.*, 1995; Wang *et al.*, 1993; Yan *et al.*, 1995; Rossi *et al.*, 1993; Dirghangi, Menon, Banerjee & Chakravorty, 1997; Banerjee *et al.*, 1997; Lahiri *et al.*, 1987), the