

Two [Cu₂I₂]-based coordination polymers of 1,3-bis(pyridin-4-yl)propane

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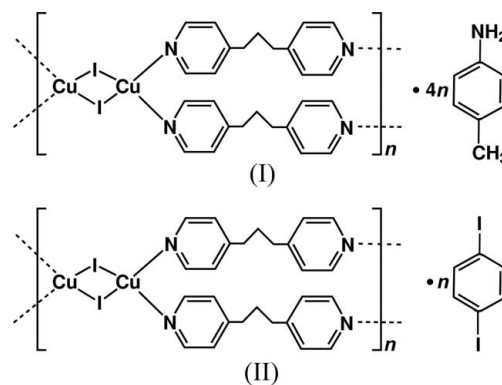
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Solvothermal reactions of Cu₂(OH)₂CO₃ with 1,3-bis(pyridin-4-yl)propane (bpp) in the presence of aqueous ammonia in 4-iodotoluene/CH₃CN or 1,4-diiodobenzene/CH₃CN afforded two [Cu₂I₂]-based coordination polymers, namely *catenapoly*[[[di- μ -iodido-dicopper(I)]-bis[μ -1,3-bis(pyridin-4-yl)propane- κ^2 N:N'] *p*-toluidine tetrasolvate], {[Cu₂I₂(C₁₃H₁₄N₂)₂·4C₇H₉N]_n, (I), and the analogous 1,4-diiodobenzene monosolvate, {[Cu₂I₂(C₁₃H₁₄N₂)₂·C₆H₄I₂]_n, (II). The [Cu₂I₂] unit of (I) lies on a centre of symmetry at the mid-point of the two I atoms, while that of (II) has a twofold axis running through the I··I line. In (I) and (II), each Cu centre is tetrahedrally coordinated by two μ -I and two N atoms from two different bpp ligands. Each rhomboid [Cu₂I₂] unit can be considered as a four-connecting node linked to the symmetry-related [Cu₂I₂] units *via* two pairs of bpp ligands to form a one-dimensional double chain along the *c* axis. The dimensions of the [Cu₂I₂(bpp)₂]₂ rings in (I) and (II) are different, which may be due to the presence of different guest solvent molecules in the structures. In (I), one *p*-toluidine molecule, derived from an Ullmann coupling reaction of 4-iodotoluene with ammonia, interacts with the [Cu₂I₂] cluster fragment through N—H··I hydrogen bonds, while the two *p*-toluidine molecules interact *via* N—H··N hydrogen bonds. In (II), two I atoms of each 1,4-diiodobenzene molecule are linked to the I atoms of the [Cu₂I₂] fragments from a neighbouring chain *via* I··I secondary interactions.

Comment

In recent years, the construction of cluster-based coordination polymers has attracted much interest due to their structural diversity (Fujita, 1998; Blake, Champness *et al.*, 1999; Leininger *et al.*, 2000; Naumov *et al.*, 2002; Tulsy *et al.*, 2003; Wang *et al.*, 2008) and their applications in storage and separation processes (Ma *et al.*, 2007; Cotton *et al.*, 2004; Chatterjee *et al.*, 2004; Rosi *et al.*, 2003), magnetism (Kahn, 2000), catalysis (Fujita *et al.*, 1994; Pan *et al.*, 2003; Hasegawa *et al.*,

2007; Lin & Wu, 2007) and other potential uses (Beauvais *et al.*, 2000; Horcajada *et al.*, 2006). Cu^I salts are used in reactions with *N*-donor ligands to form cluster-based coordination polymers of various structural motifs, such as rhomboid [Cu₂I₂] dimers (Biradha *et al.*, 2000; Hu *et al.*, 2006; Graham & Pike, 2000; Thébault *et al.*, 2006; Niu *et al.*, 2006; Araki *et al.*, 2005), cubane-like [Cu₄I₄] tetramers (Blake *et al.*, 2001), zigzag [CuI]_n or [Cu₃I₄]_n[−] chains (Graham & Pike, 2000; Thébault *et al.*, 2006; Cheng *et al.*, 2004), double-stranded [Cu₂I₂]_n ladders or ribbons (Graham & Pike, 2000; Thébault *et al.*, 2006; Cheng *et al.*, 2004), and two-dimensional [CuI]_n layers (Thébault *et al.*, 2006; Peng *et al.*, 2005; Blake, Brooks *et al.*, 1999). Many factors like reaction temperature, pH value and the symmetry of the ligands can exert a great influence on the construction and structure of [Cu_xI_x]-based coordination polymers, but solvent effects on the assembly and structure of such polymers have been less well explored (Chen *et al.*, 2008). Herein, we report the crystal structures of two [Cu₂I₂]-based coordination polymers, {[Cu₂I₂(bpp)₂·4(*p*-toluidine)]_n [bpp is 1,3-bis(pyridin-4-yl)propane], (I), and {[Cu₂I₂(bpp)₂·1,4-diiodobenzene]_n, (II), which were obtained from solvothermal reactions of Cu₂(OH)₂CO₃ with bpp and aqueous ammonia in 4-iodotoluene/CH₃CN and 1,4-diiodobenzene/CH₃CN, respectively.



The asymmetric unit of (I) contains half a [Cu₂I₂(bpp)₂] unit and two *p*-toluidine solvent molecules (Fig. 1), while that of (II) consists of half a [Cu₂I₂(bpp)₂] unit and half a 1,4-diiodobenzene solvent molecule (Fig. 2). In (I) and (II), each Cu atom is tetrahedrally coordinated by two μ -I and two N atoms from two different bpp ligands. The [Cu₂I₂] unit of (I) lies on a centre of symmetry at the mid-point of atoms I1 and I1ⁱ [symmetry code: (i) $-x, -y, -z + 1$], while that of (II) has a twofold axis running along the I1··I2 line. Each rhomboid [Cu₂I₂] unit can be viewed as a four-connecting node linking symmetry-equivalent [Cu₂I₂] units *via* two pairs of bpp ligands to afford a one-dimensional double chain along the *c* axis. [Fig. 3 for (I) and Fig. 4 for (II)].

The Cu1··Cu1ⁱ distance in (I) [2.7366 (19) Å] is slightly shorter than that observed in [Cu₂I₂L₄]_n [2.781 (2) Å, *L* = 3-methylpyridine; Rath *et al.*, 1986], but longer than those found in [Cu₂I₂L₄]_n [2.699 (5) Å, *L* = pyridine; Dyason *et al.*, 1984] and [Cu₂I₂L₄]_n [2.687 (3) Å, *L* = 3,5-dimethylpyridine; Healy *et al.*, 1983]. The length of the Cu1··Cu2 contact [3.6543 (11) Å] in (II) precludes any metal–metal interaction.

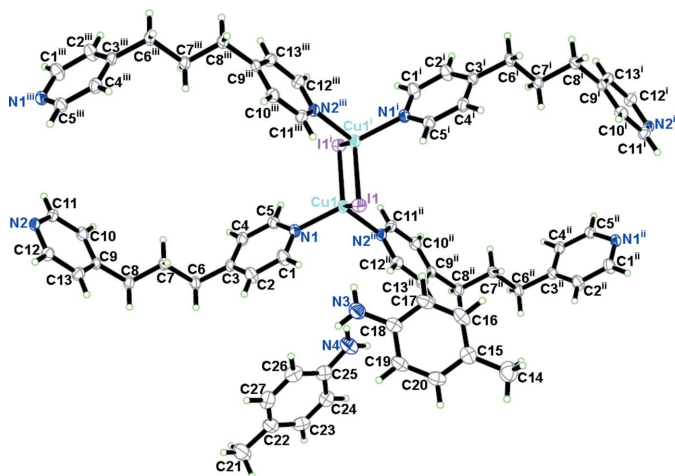


Figure 1
A segment of the chain of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x, -y, -z + 1$; (ii) $x, y, z + 1$; (iii) $-x, -y, -z$.]

The mean Cu–I bond length in (I) [2.6608 (9) Å; Table 1] is comparable with that in $\{[\text{Cu}_2\text{I}_2(\text{bpp})_2]\cdot 2(\text{aniline})\}_n$ [2.6674 (19) Å; Chen *et al.*, 2008], but shorter than those in (II) [2.7392 (8) Å] or $\{[\text{Cu}_2\text{I}_2(\text{bpe})_2]\cdot \text{Am}\}_n$ [Am = aniline, 2.7939 (15) Å; Am = *p*-toluidine, 2.7415 (11) Å; Yang *et al.*, 2011]. The average Cu–N bond length of 2.051 (2) Å in (I) is longer than those observed in (II) [2.027 (4) Å; Table 3] or $\{[\text{Cu}_2\text{I}_2(\text{bpe})_2]\cdot \text{Am}\}_n$ [Am = aniline, 2.006 (2) Å; Am = *p*-toluidine, 2.014 (3) Å], but slightly shorter than those in $\{[\text{Cu}_2\text{I}_2(\text{bpp})_2]\cdot 2(\text{aniline})\}_n$ [2.064 (7) Å] and $[\text{Cu}_2\text{I}_2L_4]_n$ [2.06 (3) Å, $L = 3,5$ -dimethylpyridine; Healy *et al.*, 1983].

Each bpp ligand in (I) and (II) adopts a *trans*–*trans* configuration (Chen *et al.* 2008). However, as the pyridine rings are rotated around the Cu–N direction, the bpp ligand in (I) is not planar, with a dihedral angle of 70.64 (15)° between the two pyridine rings, while that of (II) is approxi-

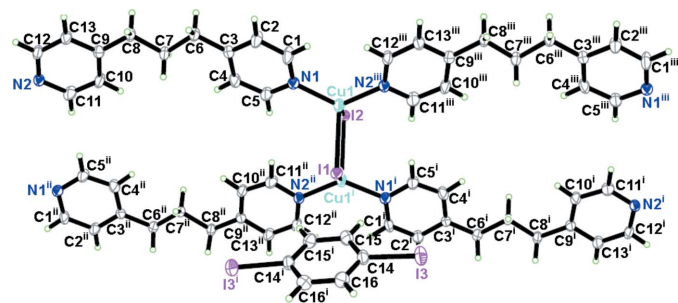


Figure 2
A segment of the chain of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x, y, -z + \frac{1}{2}$; (ii) $-x, y, -z + \frac{3}{2}$; (iii) $x, y, z - 1$.]

mately planar, with a dihedral angle of 12.5 (2)° between the two pyridine rings. The N1···N2 separation [9.595 (4) Å] in (I) is shorter than that in (II) [10.179 (6) Å]. On the other hand, the one-dimensional chains in (I) and (II) may be viewed as a sequence of 28-membered $[\text{Cu}_2\text{I}_2(\text{bpp})_2]$ metallomacrocyclic rings (Perera *et al.*, 2010; Chan *et al.*, 2009), which are constructed of two $[\text{Cu}_2\text{I}_2]$ and two bpp ligands. The dimensions of these metallomacrocyclic rings in (I) and (II) are roughly 12.96×8.27 and 13.76×7.79 Å, respectively. These differences may be ascribed to solvent effects, which greatly influence the structures of $[\text{Cu}_2\text{I}_2]$ -based coordination polymers (Blake *et al.*, 2001; Chen *et al.*, 2008; Yang *et al.*, 2011).

In the formation of (I) and (II), Cu^{II} may be reduced by bpp and/or iodide and the resulting Cu^{I} may combine iodides to produce the $[\text{Cu}_2\text{I}_2]$ species (Yang *et al.* 2011). The latter have then been captured by bpp ligands to yield the $[\text{Cu}_2\text{I}_2]$ -based frameworks of (I) and (II). In (I), the *p*-toluidine guest molecules have been generated *in situ* through an Ullmann coupling reaction of 4-iodotoluene with ammonia (Chen *et al.* 2008).

In (I), two *p*-toluidine molecules interact with each other *via* intermolecular hydrogen bonds between two amino groups

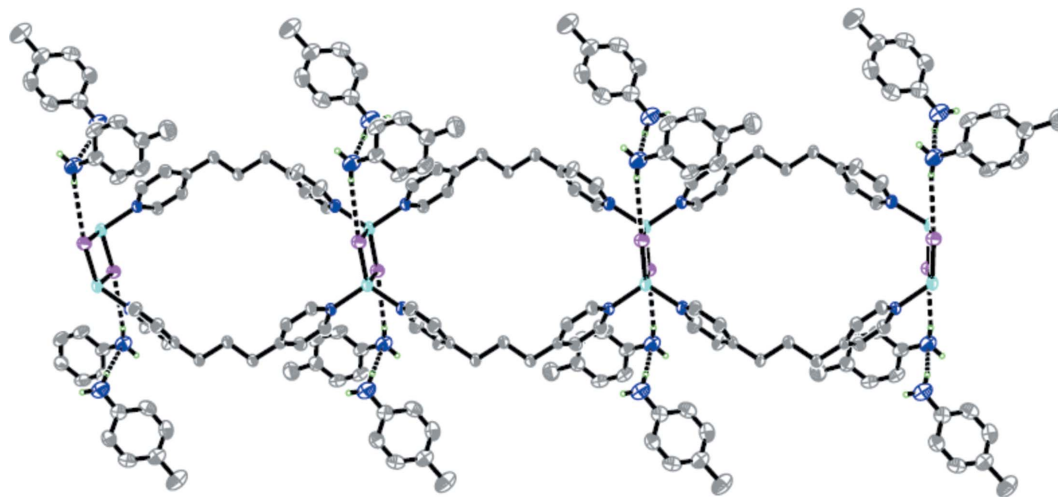


Figure 3
A view of the one-dimensional chain of (I), extending along the *c* axis. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate N–H···N and N–H···I hydrogen bonds. (In the electronic version of the paper, atom colour codes are: Cu cyan, I pink, N blue, C grey and H green.)

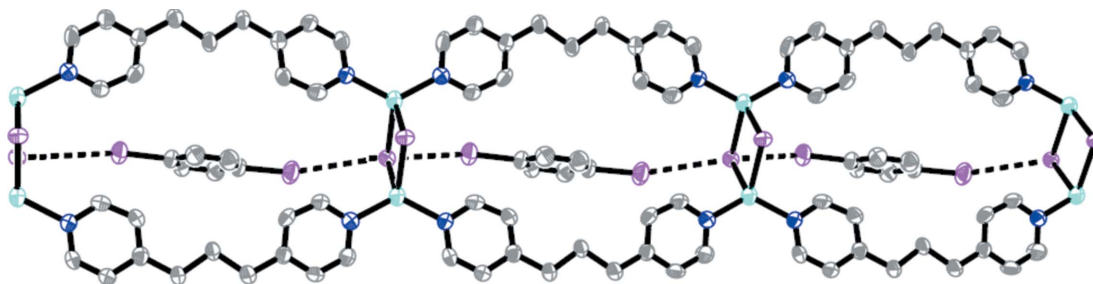


Figure 4
A view of the one-dimensional chain of (II), extending along the *c* axis. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate I...I secondary interactions. (In the electronic version of the paper, atom colour codes are: Cu cyan, I pink, N blue, C grey and H green.)

(N4—H4...N3; Table 2). The NH₂ group of one *p*-toluidine molecule binds the I atom of the [Cu₂I₂] fragment through an intermolecular hydrogen bond (N3—H3...I1) (Ciunik & Desiraju, 2001) (Fig. 3).

In (II), the two I atoms of each 1,4-diiodobenzene molecule interact with the I atoms of the [Cu₂I₂] fragments from an adjacent chain via an I...I(−*x*, −*y* + 1, −*z*) secondary interaction [3.6200 (9) Å] (Fig. 4).

Experimental

Cu₂(OH)₂CO₃ (22 mg, 0.1 mmol), bpp (20 mg, 0.1 mmol), 4-iodotoluene (150 mg, 0.7 mmol), aqueous ammonia (25%, 1.5 ml) and CH₃CN (0.5 ml) were added to a Pyrex glass tube (15 cm long × 7 mm inner diameter). The tube was sealed and heated in an oven at 423 K for 70 h, and then cooled to room temperature at a rate of 5 K per 100 min to form yellow prisms of {[Cu₂I₂(bpp)₂]·4(*p*-toluidine)}_n, (I), which were collected by filtration, washed with CH₃CN and dried in air (yield: 21 mg, 30% based on bpp). Analysis found: C 53.59, H 5.29, N 9.20%; calculated for C₂₇H₃₂CuIN₄: C 53.78, H 5.35, N 9.29%. IR (KBr, ν, cm^{−1}): 3427 (*w*), 3380 (*w*), 3301 (*w*), 3209 (*w*), 3022 (*w*), 2926 (*w*), 2860 (*w*), 1608 (*s*), 1514 (*s*), 1421(*m*), 1262 (*m*), 1220 (*m*), 1011 (*w*), 809 (*s*), 610 (*w*), 512 (*m*).

Cu₂(OH)₂CO₃ (22 mg, 0.1 mmol), bpp (20 mg, 0.1 mmol), 1,4-diiodobenzene (33 mg, 0.1 mmol), aqueous ammonia (25%, 0.2 ml) and CH₃CN (1.5 ml) were added to a Pyrex glass tube (15 cm long × 7 mm inner diameter). The tube was sealed and heated in an oven at 423 K for 70 h, and then cooled to room temperature at a rate of 5 K per 100 min to form orange blocks of {[Cu₂I₂(bpp)₂]·1,4-diiodobenzene}_n (II), which were collected by filtration, washed with CH₃CN and dried in air (yield: 0.021 g, 30% based on bpp). Analysis found: C 34.69, H 2.89, N 5.10%; calculated for C₁₆H₁₆CuI₂N₂: C 34.71, H 2.91, N 5.06%. IR (KBr, ν, cm^{−1}): 3425 (*w*), 3378 (*w*), 3206 (*w*), 3021 (*w*), 2923 (*w*), 2856 (*w*), 1602 (*s*), 1509 (*s*), 1417 (*s*), 1258 (*m*), 1218 (*m*), 1008 (*w*), 805 (*s*), 613 (*w*), 512 (*m*).

Compound (I)

Crystal data

[Cu ₂ I ₂ (C ₁₃ H ₁₄ N ₂) ₂]·4C ₇ H ₉ N	γ = 109.30 (3)°
<i>M_r</i> = 1206.02	<i>V</i> = 1313.4 (5) Å ³
Triclinic, <i>P</i> 1̄	<i>Z</i> = 1
<i>a</i> = 10.119 (2) Å	Mo <i>K</i> α radiation
<i>b</i> = 12.370 (3) Å	μ = 2.03 mm ^{−1}
<i>c</i> = 12.962 (3) Å	<i>T</i> = 223 K
α = 103.19 (3)°	0.60 × 0.60 × 0.40 mm
β = 111.06 (3)°	

Data collection

Rigaku Saturn 724+ CCD area-detector diffractometer	12464 measured reflections
Absorption correction: multi-scan (Jacobson, 1998)	5891 independent reflections
<i>T</i> _{min} = 0.376, <i>T</i> _{max} = 0.498	4782 reflections with <i>I</i> > 2σ(<i>I</i>)
	<i>R</i> _{int} = 0.035

Refinement

<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.030	300 parameters
<i>wR</i> (<i>F</i> ²) = 0.069	H-atom parameters constrained
<i>S</i> = 0.95	Δρ _{max} = 0.59 e Å ^{−3}
5891 reflections	Δρ _{min} = −0.77 e Å ^{−3}

Compound (II)

Crystal data

[Cu ₂ I ₂ (C ₁₃ H ₁₄ N ₂) ₂]·C ₆ H ₄ I ₂	<i>V</i> = 3351.4 (12) Å ³
<i>M_r</i> = 1107.30	<i>Z</i> = 4
Monoclinic, <i>C</i> 2/ <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 17.700 (4) Å	μ = 4.98 mm ^{−1}
<i>b</i> = 14.604 (3) Å	<i>T</i> = 223 K
<i>c</i> = 13.757 (3) Å	0.54 × 0.40 × 0.38 mm
β = 109.53 (3)°	

Data collection

Rigaku Saturn 724+ CCD area-detector diffractometer	15742 measured reflections
Absorption correction: multi-scan (Jacobson, 1998)	3058 independent reflections
<i>T</i> _{min} = 0.174, <i>T</i> _{max} = 0.253	2902 reflections with <i>I</i> > 2σ(<i>I</i>)
	<i>R</i> _{int} = 0.025

Refinement

<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.032	191 parameters
<i>wR</i> (<i>F</i> ²) = 0.080	H-atom parameters constrained
<i>S</i> = 1.12	Δρ _{max} = 0.84 e Å ^{−3}
3058 reflections	Δρ _{min} = −1.67 e Å ^{−3}

All H atoms were placed in geometrically idealized positions, with C—H = 0.98 Å for methyl groups, 0.99 Å for methylene groups and 0.95 Å for phenyl groups, or N—H = 0.85 Å for amino groups, and constrained to ride on their parent atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C,N) for phenyl and amino groups, and *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl groups.

For both compounds, data collection: *CrystalClear* (Rigaku/MS, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Table 1

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

Cu1—N1	2.050 (2)	Cu1—II ⁱⁱ	2.6746 (9)
Cu1—N2 ⁱ	2.051 (2)	Cu1—Cu1 ⁱⁱ	2.7366 (19)
Cu1—I1	2.6470 (7)	I1—Cu1 ⁱⁱ	2.6746 (9)
N1—Cu1—N2 ⁱ	110.41 (9)	N1—Cu1—Cu1 ⁱⁱ	125.17 (7)
N1—Cu1—I1	107.30 (7)	N2 ⁱ —Cu1—Cu1 ⁱⁱ	124.41 (7)
N2 ⁱ —Cu1—I1	107.55 (7)	I1—Cu1—Cu1 ⁱⁱ	59.55 (3)
N1—Cu1—I1 ⁱⁱ	107.16 (6)	I1 ⁱⁱ —Cu1—Cu1 ⁱⁱ	58.56 (3)
N2 ⁱ —Cu1—I1 ⁱⁱ	106.24 (7)	Cu1—I1—Cu1 ⁱⁱ	61.89 (4)
I1—Cu1—I1 ⁱⁱ	118.11 (4)		

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

Table 2

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3A \cdots I1	0.85	2.99	3.793 (3)	158
N4—H4B \cdots N3	0.85	2.41	3.228 (4)	160

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: YF3014). Services for accessing these data are described at the back of the journal.

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Table 3

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

Cu1—N1	2.023 (4)	Cu1—I2	2.7412 (8)
Cu1—N2 ⁱ	2.030 (4)	I1—Cu1 ⁱⁱ	2.7371 (8)
Cu1—I1	2.7371 (8)	I2—Cu1 ⁱⁱ	2.7412 (8)
N1—Cu1—N2 ⁱ	124.21 (15)	N2 ⁱ —Cu1—I2	108.25 (10)
N1—Cu1—I1	110.57 (10)	I1—Cu1—I2	96.32 (3)
N2 ⁱ —Cu1—I1	108.22 (10)	Cu1—I1—Cu1 ⁱⁱ	83.76 (3)
N1—Cu1—I2	105.60 (10)	Cu1 ⁱⁱ —I2—Cu1	83.60 (3)

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

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