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Key indicators
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.019
wR factor = 0.045
Data-to-parameter ratio = 18.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

A second polymorph of cyclo-tetrakis[μ_2-[(dimethylamino)methyl]phenyl-κ^3 C^2:N]copper tetrahydrofuran solvate

The structure of the title compound, [Cu_4(C_9H_12N)_4]·C_4H_8O, has previously been reported in the monoclinic space group C2/c. We report here the structure of a second polymorph which crystallizes in the orthorhombic space group P2_1_2_1_2_1. The copper complex is located at a general position but exhibits local S_4 symmetry.

Comment
The structure of the title compound, (I), has previously been reported crystallizing in the monoclinic space group C2/c, (II) (Janssen et al., 1996). The asymmetric unit of (II) contains one half of the copper complex, the other half being identical by a twofold rotation, and one half-weight tetrahydrofuran (THF) molecule. The THF was found to be disordered about the twofold rotation axis. The solvent-free metal complex has also been reported, in space group P2_1/c, (III) [Spek & van Koten, 2004; Cambridge Structural Database (Version 5.26) and Conquest (Version 1.7) (Allen, 2002) refcode IQOTOX].

The title compound, (I), is a second polymorph of the C2/c structure (Fig. 1) and crystallizes in the orthorhombic space group P2_1_2_1_2_1. The asymmetric unit consists of one complete copper complex and a single disordered THF molecule. The overall crystal structure is quite different from that of the previous polymorph, (II). In (II), the complex and the THF lie on a twofold rotation axis, with the THF molecules lying in the cavities of adjacent copper complexes (Fig. 2). In (I), however, whilst the complexes now line up roughly parallel to the a axis, the THF no longer sits between pairs of complexes and is instead displaced to the side (Fig. 3).

The structure of the copper complex is essentially the same as previously reported, with the Cu atoms forming a ‘butterfly’ arrangement. The Cu⋯C and Cu⋯N bond lengths are comparable with those in (II), as are the Cu⋯C⋯Cu bond angles, which are once again very acute, at between 70.33 (8) and 72.14 (9). The Cu⋯·Cu distances in (II) are very short, at...
Polymorphs (I) and (III) also have very short Cu\(\cdots\)Cu distances for Cu atoms that are adjacent in the metallocycle, at between 2.3773 (4) and 2.4118 (4) Å for (I), and between 2.372 (2) and 2.396 (2) Å for (III). However, in both cases, opposite Cu atoms have longer Cu\(\cdots\)Cu distances, with one distinctly longer than the other [3.0065 (4) and 3.3810 (4) Å in (I), and 3.040 (2) and 3.380 (3) Å in (III)].

As a consequence of the different packing, the intermolecular Cu\(\cdots\)Cu distances are different for the three crystal structures: in (I), the shortest Cu\(\cdots\)Cu distance is 8.1780 (4) Å, in (II) it is 8.0630 (6) Å, and in (III) it is 6.508 (2) Å.

**Experimental**

The title compound was obtained unintentionally during an attempt to synthesize a mixed Cu–Li complex. It was recrystallized from THF, yielding pale-yellow crystals of (I) suitable for X-ray diffraction.

![Figure 1](image1.png)

**Figure 1**
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Both disorder components are shown.

![Figure 2](image2.png)

**Figure 2**
A packing diagram for the \(P2_12_12_1\) polymorph, (I), viewed along the \(a\) axis. The THF molecule is disordered over two positions, with relative occupancies of 73% and 27%; dashed lines indicate the minor disorder component.

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References