

Crystal structure of *trans*-bis[2-(1*H*-benzotriazol-1-yl)acetato- κ O]bis(ethanolamine- κ^2 N,O)copper(II)

Guloy K. Alieva,^{a*} Jamshid M. Ashurov,^b Shahnoza A. Kadirova,^c Bakhtiyar T. Ibragimov^b and Kasim A. Zakhidov^d

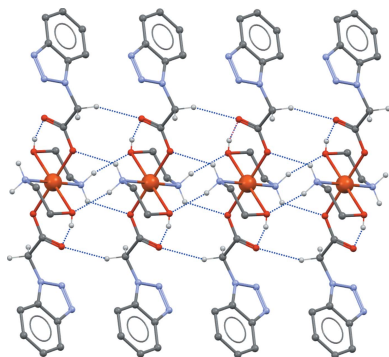
^aInstitute of General and Inorganic Chemistry of Uzbekistan Academy of Sciences, M. Ulugbek Str, 77a, Tashkent,

^bInstitute of Bioorganic Chemistry, Academy of Sciences of Uzbekistan, M. Ulugbek Str 83, Tashkent, ^cChemistry Department, National University of Uzbekistan, Tashkent, and ^dSamarkand State University 140104, University blv. 15, Samarkand, Samarkand region, Uzbekistan. *Correspondence e-mail: x-ray.uz@mail.ru

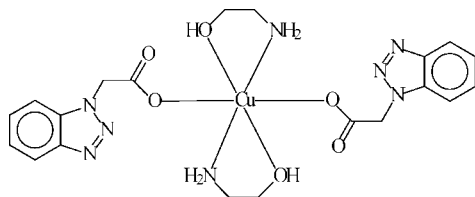
The reaction of 2-(1*H*-benzotriazol-1-yl)acetic acid (HBTA; C₈H₇N₃O₂) and monoethanolamine (MEA; C₂H₇NO) with CuCl₂·2H₂O resulted in the formation of the title complex, [Cu(C₈H₆N₃O₂)₂(C₂H₇NO)₂] or [Cu(BTA)₂(MEA)₂]. Its asymmetric unit comprises one BTA anion coordinating to the Cu²⁺ cation (site symmetry $\bar{1}$) through the carboxyl O atom, and one MEA ligand chelating the metal cation by two heteroatoms (O and N). The equatorial Cu—O and Cu—N bond lengths are similar at 2.029 (1) and 1.980 (2) Å, respectively, while the length of the axial Cu—O bond is considerably greater [2.492 (2) Å], as is typical for Jahn–Teller-distorted systems. An intramolecular hydrogen bond is present between the hydroxy group of the MEA ligand and the non-coordinating O atom of the carboxylate group. Intermolecular hydrogen bonding involving the amino function of the MEA ligand and the carboxylate group results in eight-membered rings with an R₂²(8) graph-set motif. The molecules are further linked by C—H... π interactions involving the triazole rings and methylene groups of MEA, thus generating an overall three-dimensional supramolecular framework.

1. Chemical context

Recently, systematic studies of the structures and metal complex formation features of benzoic acid (Ibragimov *et al.*, 2016*a,b*) and benzotriazole derivatives have been carried out by our group. Benzotriazoles consist of nitrogen-containing bicyclic ring systems and demonstrate many types of biological activities, such as antibacterial (Wan *et al.*, 2010; Suma *et al.*, 2012), antimicrobial (Nanjunda Swamy *et al.*, 2006; Singh *et al.*, 2009; Patel *et al.*, 2012; Ramachandran *et al.*, 2011), antifungal (Khabnadideh *et al.*, 2012; Rezaei *et al.*, 2009; Gaikwad *et al.*, 2012; Rakesh *et al.*, 2010), anticancer, anti-inflammatory, analgesic, antimalarial and antitubercular (Kopańska (née Zastąpiło) *et al.*, 2004; Jamkhandi *et al.*, 2015). Functional groups such as carboxylate, hydroxyl and pyridyl can be introduced to benzotriazole, increasing the coordination possibilities (Stoumpos *et al.*, 2008; Wang *et al.*, 2008*a,b*). The interaction of metal ions with HBTA results in the formation of complexes in which it demonstrates monodentate (Ma *et al.*, 2015; Zeng *et al.*, 2012; Wang *et al.*, 2014*a*) coordination. HBTA also can show bridging (Li *et al.*, 2016; Wang *et al.*, 2014*b*) and *catena*-type (Wang *et al.*, 2011, 2014*b*; Liu *et al.*, 2012) coordination modes. The interaction of metal cations with MEA results in the formation of complexes in which



MEA demonstrates monodentate (Hajji & Guerfel, 2016; Luo *et al.*, 2012; Ren *et al.*, 2014; Heinrich *et al.*, 2012; Guzei *et al.*, 2010*a*) and bidentate (Ibragimov *et al.*, 2017; Seppälä *et al.*, 2013; Yeşilel *et al.*, 2012; Xue *et al.*, 2016; Ashurov *et al.*, 2015) coordination modes. In some complexes, MEA has bridging properties (Shahid *et al.*, 2015; Tudor *et al.*, 2013; Schwarz *et al.*, 2010; Maclaren *et al.*, 2012; Seppälä *et al.*, 2012). In addition, there are metal complexes in which MEA molecules show non-coordinating behaviour (Wang *et al.*, 2013; Lemmerer & Billing, 2010; Calderone *et al.*, 2011; Yadav *et al.*, 2015; Sutradhar *et al.*, 2012; Liu *et al.*, 2011).



We have reported the synthesis of mixed-ligand complexes of Cu and Zn with MEA and α -naphthylacetic acid (NAA) and determined the structures of [Cu(NAA)₂(MEA)₂] and [Zn(NAA)₂(MEA)₂] (Ashurov *et al.*, 2015). A search in the Cambridge Structural Database (CSD Version 5.39, last update August 2018; Groom *et al.*, 2016) revealed that crystal structures have been reported for complexes of HBTA and MEA with many metal ions. However, no mixed-ligand metal complex including HBTA and MEA is documented in the CSD. Here, the synthesis and structure of the title compound, [Cu(BTA)₂(MEA)₂], (I), is described.

2. Structural commentary

The molecular structure of *trans*-bis(ethanolamine- κ^2N,O)bis-[2-(1*H*-benzotriazol-1-yl)acetato- κO]copper(II), (I), is shown in Fig. 1 and consists of isolated [Cu(MEA)₂(BTA)₂] units. The Cu²⁺ cation is located on a center of inversion. Its coordination polyhedron is a distorted N₂O₄ octahedron formed by two oxygen atoms (O2) of the carboxy groups of symmetry-related BTA anions, by two nitrogen atoms (N4) of two symmetry-related MEA ligands in the equatorial plane and by two O atoms (O3) of the same set of MEA ligands in the axial positions. The Cu—O2 and Cu—N4 bond lengths are 2.029 (1) and 1.980 (2) Å, respectively, whereas the length of the axial

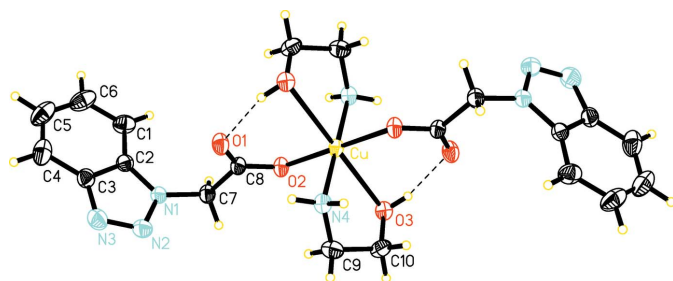


Figure 1
The molecular structure of [Cu(MEA)₂(BTA)₂] with the atom-numbering scheme. Displacement ellipsoids are drawn at the 25% probability level.

Table 1
Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O3—H3···O1 ⁱ	0.80 (1)	1.86 (1)	2.634 (2)	163 (3)
N4—H4A···O2 ⁱⁱ	0.89 (1)	2.41 (2)	3.046 (2)	129 (2)
N4—H4B···O3 ⁱⁱⁱ	0.89 (1)	2.12 (1)	2.973 (2)	161 (2)
C7—H7A···O1 ⁱⁱⁱ	0.97	2.53	3.449 (3)	158
C9—H9A···N3 ^{iv}	0.97	2.58	3.345 (3)	136

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

Cu—O3 bond is 2.492 (2) Å, typical for Jahn–Teller distortions. The MEA ligand is neutral and acts as a bidentate N- and O-donor ligand and forms CuNC₂O five-membered chelate rings which have a twist conformation; the O3—C10—C9—N4 torsion angle is -60.3 (3)°. The planar benzotriazole ring system (N1–N3/C1–C6; r.m.s. deviation = 0.0064 Å) is coplanar with the methyl carbon atom C7 [deviation from the plane of 0.158 (2) Å], whereas the carboxylate group is nearly normal to this plane [88.0 (2)°]. The difference of the C8—O(1,2) distances of the carboxylate group ($\Delta = 0.036$ Å) is due to the monodentate coordination, with the longer C—O distance involving the coordinating O2 atom.

The molecular structure is stabilized by an intramolecular O3—H3···O1 hydrogen bond between the OH group of the MEA ligand and the non-coordinating carboxylate O atom (Fig. 1, Table 1).

3. Supramolecular features

In the crystal structure of (I), molecules are linked by C7—H7A···O1ⁱⁱⁱ, N4—H4A···O2ⁱⁱ and N4—H4B···O3ⁱⁱⁱ hydrogen bonds between the amino function and carboxylate/hydroxy

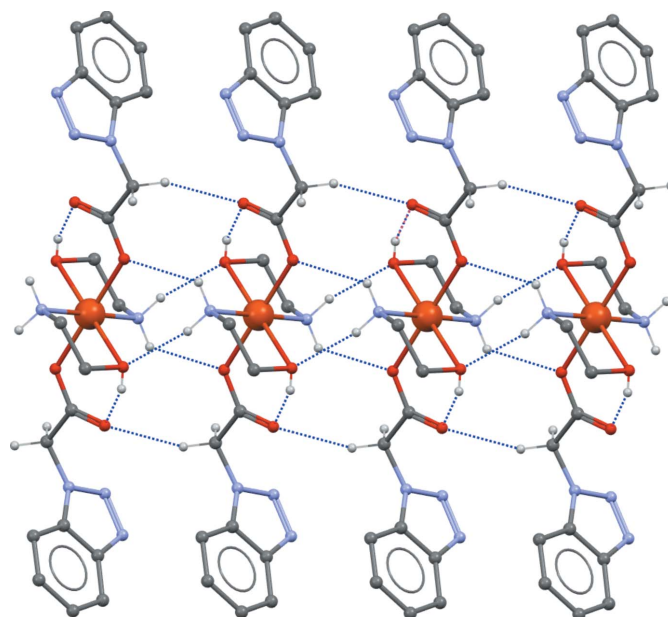


Figure 2
Chain structures formed by hydrogen bonds in the structure of (I). Hydrogen bonds are shown as dashed lines.

O-atom acceptors (Table 2, Fig. 2), forming chains propagating parallel to [010]. Adjacent chains are linked by C9—H9A...N3^{iv} hydrogen bonds into a layered arrangement parallel to (10 $\bar{1}$) (Fig. 3). Additional C—H... π interactions between the triazole rings and methylene groups of MEA (H...Cg = 2.88, C—H...Cg = 140°, $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$) generate a three-dimensional supramolecular framework.

4. Database survey

There are thirty-one structures of coordination compounds that are derived from 2-(1*H*-benzotriazol-1-yl)acetic acid and different metal cations in the CSD (Version 5.39, last update August 2018; Groom *et al.*, 2016). The interaction of metal ions with BTA results in the formation of complexes in which metals demonstrate monodentate [CUYGAG (Ma *et al.*, 2015), DUWQES (Zheng *et al.*, 2010), LAMYUV (Zeng *et al.*, 2012), TIVWOM (Wang *et al.*, 2014*b*), TOBDUK (Hang & Ye, 2008)] and bridging [COHFOW (Ren *et al.*, 2013), DEZHIB (Zeng, 2013), GADVEP (Li *et al.*, 2016), TIVXAZ (Wang *et al.*, 2014*b*)] coordination modes. BTA also can show *catenatype* structures [DEZHOH (Zeng, 2013), DUWQAO (Zheng *et al.*, 2010), DUWQIW (Zheng *et al.*, 2010), GUTZAX (Wang *et al.*, 2009), IPAGIQ (Wang *et al.*, 2011), TIVXED (Wang *et al.*, 2014*b*), TIVXON (Wang *et al.*, 2014*b*), UFETEF (Hu *et al.*, 2008), YATPAM (Liu, 2012), ZIPLOB (Chen *et al.*, 2010) *etc.*]. In most cases, MEA behaves as a chelating ligand; however, there are metal complexes in which non-coordinating MEA molecules are situated in the outer coordination sphere [AXUQAN (Ibragimov *et al.*, 2016*c*), FAFTOV (Spitsin *et al.*, 1986), TIRQEQ (Halvorson *et al.*, 1995), WUZZOH (Guzei *et al.*, 2010*b*) *etc.*]. Mixed-ligand metal complexes including BTA and MEA have not been reported in the CSD up to date.

5. Synthesis and crystallization

To an aqueous solution (2.5 ml) of CuCl₂·2H₂O (0.048 g, 0.282 mmol) was slowly added an ethanol solution (5 ml) containing MEA (0.034 g, 0.565 mmol) and HBTA (0.1 g, 0.565 mmol) under constant stirring. Blue crystals of the product were obtained by solvent evaporation at room

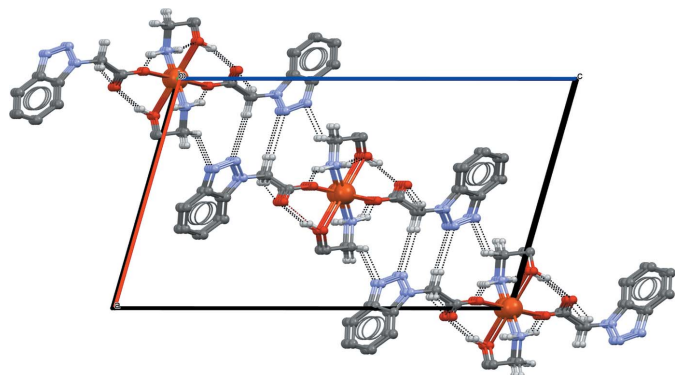


Figure 3
A partial view along the *b* axis of the crystal packing of compound (I). Intermolecular hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Cu(C ₈ H ₆ N ₃ O ₂) ₂ (C ₂ H ₇ NO) ₂]
<i>M_r</i>	538.03
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	12.4283 (4), 4.84866 (9), 20.6944 (5)
β (°)	105.823 (3)
<i>V</i> (Å ³)	1199.80 (5)
<i>Z</i>	2
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	1.75
Crystal size (mm)	0.36 × 0.22 × 0.12
Data collection	
Diffractionmeter	Rigaku Xcalibur Ruby
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.558, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8833, 2444, 2152
<i>R_{int}</i>	0.031
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.629
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.033, 0.096, 1.06
No. of reflections	2444
No. of parameters	173
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.24, -0.31

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2006 and *publCIF* (Westrip, 2010).

temperature after one week. Yield: 70%. Elemental analysis: Calc. for C₂₀H₂₆CuN₈O₆ (538.04): C, 44.65; H, 4.87 N, 20.83%. Found: C, 44.73; H, 4.93; N, 20.88%.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound hydrogen atoms were placed in calculated positions and refined as riding atoms with C—H = 0.93 and 0.97 Å for aromatic and methylene hydrogen atoms, respectively, and with *U*_{iso}(H) = 1.2*U*_{eq}(C). The positions of the O- and N bound H atoms were located from a difference-Fourier map and were refined with soft distance restraints, 0.82 Å for the hydroxyl group and 0.95 Å for the primary amine group.

Funding information

This work was supported by a Grant for Fundamental Research of the Center of Science and Technology, Uzbekistan (No. BA-FA-F7-004).

References

Ashurov, J. M., Mukhamedov, N. S. & Ibragimov, A. B. (2015). *Russ. J. Coord. Chem.* **41**, 207–211.

- Calderone, P. J., Forster, P. M., Borkowski, L. A., Teat, S. J., Feyngenson, M., Aronson, M. C. & Parise, J. B. (2011). *Inorg. Chem.* **50**, 2159–2167.
- Chen, Y., Li, C. & Li, J.-M. (2010). *Hecheng Huaxue*, **18**, 328–331.
- Gaikwad, N. D., Patil, S. V. & Bobade, V. D. (2012). *Bioorg. Med. Chem. Lett.* **22**, 3449–3454.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Guzei, I. A., Spencer, L. C., Ainooson, M. K. & Darkwa, J. (2010a). *Acta Cryst.* **C66**, m89–m96.
- Guzei, I. A., Spencer, L. C., Yankey, M. & Darkwa, J. (2010b). *Acta Cryst.* **E66**, m1551–m1552.
- Hajji, M. & Guerfel, T. (2016). *J. Clust. Sci.* **27**, 1395–1417.
- Halvorson, K. E., Patylek, B. & Willett, R. D. (1995). *J. Chem. Crystallogr.* **25**, 537–542.
- Hang, T. & Ye, Q. (2008). *Acta Cryst.* **E64**, m758.
- Heinrich, F., Kessler, M. T., Dohmen, S., Singh, M., Prechtel, M. H. G. & Mathur, S. (2012). *Eur. J. Inorg. Chem.* pp. 6027–6033.
- Hu, T.-L., Du, W.-P., Hu, B.-W., Li, J.-R., Bu, X.-H. & Cao, R. (2008). *CrystEngComm*, **10**, 1037–1043.
- Ibragimov, A., Ashurov, J., Ibragimov, B., Wang, A., Mouhib, H. & Englert, U. (2016a). *Acta Cryst.* **C72**, 566–571.
- Ibragimov, A. B., Ashurov, J. M., Ibragimov, A. B. & Tashpulatov, Zh. Zh. (2017). *Russ. J. Coord. Chem.* **43**, 380–388.
- Ibragimov, A. B., Ashurov, J. M. & Zakirov, B. (2016b). *J. Chem. Crystallogr.* **46**, 352–363.
- Ibragimov, A. B., Ashurov, Zh. M. & Zakirov, B. C. (2016c). *Doklad. Akad. Nauk Uzbek. SSR*, **1**, 622.
- Jamkhandi, C. M., Kumbhar, P. S., Disouza, J. I. & Patil, S. M. (2015). *Eur. J. Pharm. Med. Res.* **2**, 1004–1010.
- Khabnadideh, S., Rezaei, Z., Pakshir, K., Zomorodian, K. & Ghafari, N. (2012). *Res. Pharm. Sci.* **7**, 65–72.
- Kopańska (née Zastąpiło), K., Najda, A., Żebrowska, J., Chomicz, L., Piekarczyk, J., Myjak, P. & Bretner, M. (2004). *Bioorg. Med. Chem.* **12**, 2617–2624.
- Lemmerer, A. & Billing, D. G. (2010). *CrystEngComm*, **12**, 1290–1301.
- Li, J.-M., Shi, Z.-F. & He, K.-H. (2016). *Chin. J. Struct. Chem.* **35**, 135–139.
- Liu, B., Zheng, H.-B., Wang, Z.-M. & Gao, S. (2011). *CrystEngComm*, **13**, 5285–5288.
- Liu, Q. (2012). *Acta Cryst.* **E68**, m379.
- Luo, W., Mu, W.-Q., Zhang, X., Zhang, X., Pu, Y. Y., Zhu, Q. Y. & Dai, J. (2012). *Inorg. Chem.* **51**, 1489–1494.
- Ma, Y., Zhang, Y.-X. & Tang, G.-M. (2015). *J. Chem. Res. (S)*, **39**, 233–237.
- Maclaren, J. K., Sanchiz, J., Gili, P. & Janiak, C. (2012). *New J. Chem.* **36**, 1596–1609.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Nanjunda Swamy, S., Basappa, Sarala, G., Priya, B. S., Gaonkar, S. L., Shashidhara Prasad, J. & Rangappa, K. S. (2006). *Bioorg. Med. Chem. Lett.* **16**, 999–1004.
- Patel, J. S., Garg, C. S. & Sen, D. (2012). *Int. J. Drug Dev. Res.* **4**, 322–329.
- Rakesh, S., Saurabh, Ch., Achyut, N. K. & Swatrantra, K. (2010). *Pharma Chem.* **2**, 297–302.
- Ramachandran, R., Rani, M., Senthana, S., Jeong, Y. T. & Kabilan, S. (2011). *Eur. J. Med. Chem.* **46**, 1926–1934.
- Ren, N., Zhang, D.-H., Dong, C.-H., Zhang, J.-J. & Liu, Y. (2013). *Huaxue Shiji*, **35**, 353–355.
- Ren, X., Pan, Q., Liu, J., Li, Y., Wang, F., Li, J., Xu, Y. & Li, L. (2014). *CrystEngComm*, **16**, 9545–9554.
- Rezaei, Z., Khabnadideh, S., Pakshir, K., Hossaini, Z., Amiri, F. & Assadpour, E. (2009). *Eur. J. Med. Chem.* **44**, 3064–3067.
- Rigaku OD (2015). *CrysAlis PRO*, Rigaku Corporation, Tokyo, Japan.
- Schwarz, A. D., Chu, Z. & Mountford, P. (2010). *Organometallics*, **29**, 1246–1260.
- Seppälä, P., Colacio, E., Mota, A. J. & Sillanpää, R. (2012). *Dalton Trans.* **41**, 2648–2658.
- Seppälä, P., Colacio, E., Mota, A. J. & Sillanpää, R. (2013). *Inorg. Chem.* **52**, 11096–11109.
- Shahid, M., Siddique, A., Ansari, I. A., Sama, F., Chibber, S., Khalid, M., Siddiqi, Z. A. & Faizi, S. H. (2015). *J. Coord. Chem.* **68**, 848–862.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Singh, R. J. (2009). *Rasayan J. Chem.* **2**, 598–601.
- Spitsin, V. I., Kazin, P. E., Subbotin, M. Yu., Aslanov, L. A., Zelentsov, V. V., Zhironov, A. I. & Felin, M. G. (1986). *Doklad. Akad. Nauk SSSR*, **287**, 134.
- Stoumpos, C. C., Diamantopoulou, E., Raptopoulou, C. P., Terzis, A., Perlepes, S. P. & Lalioti, N. (2008). *Inorg. Chim. Acta*, **361**, 3638–3645.
- Suma, B. V., Natesh, N. N., Venkataramana, C. H. S., Jays, J. & Madhavan, V. (2012). *Int. J. Pharm. Pharm. Sci.* **4**, 169–173.
- Sutradhar, M., Barman, T. R., Mukherjee, G., Drew, M. G. B. & Ghosh, S. (2012). *Polyhedron*, **34**, 92–101.
- Tudor, V., Mocanu, T., Tuna, F., Madalan, A. M., Maxim, C., Shova, S. & Andruh, M. (2013). *J. Mol. Struct.* **1046**, 164–170.
- Wan, J., Lv, P.-C., Tian, N. N. & Zhu, H. L. (2010). *J. Chem. Sci.* **122**, 597–606.
- Wang, G.-M., Li, J.-H., Li, Z.-X., Xue, Sh.-Y. & Li, H. (2008b). *Z. Anorg. Allg. Chem.* **634**, 1149–1153.
- Wang, J., Huang, M., Liu, P. & Cheng, W. (2008a). *J. Mol. Struct.* **875**, 22–26.
- Wang, J.-H., Tang, G.-M., Qin, T.-X., Yan, S.-C., Wang, Y.-T., Cui, Y.-Z. & Weng Ng, S. (2014a). *J. Solid State Chem.* **219**, 55–66.
- Wang, J.-H., Tang, G.-M., Wang, Y.-T., Qin, T.-X. & Ng, S.-W. (2014b). *CrystEngComm*, **16**, 2660–2683.
- Wang, W., Fu, D.-W., Xu, X.-B. & Ye, Q. (2011). *Z. Anorg. Allg. Chem.* **637**, 467–471.
- Wang, Y.-L., Feng, M.-L., Wang, K.-Y., Li, J.-R., Wang, Z.-P., Zou, G.-D. & Huang, X.-Y. (2013). *Inorg. Chem. Commun.* **33**, 10–14.
- Wang, Y.-T., Tang, G.-M., Wei, Y.-Q., Qin, T.-X., Li, T.-D., Ling, J.-B. & Long, X.-F. (2009). *Inorg. Chem. Commun.* **12**, 1164–1167.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Xue, H., Zhang, Z., Yang, B.-F., Liu, H.-S. & Yang, G.-Y. (2016). *J. Clust. Sci.* **27**, 1439–1449.
- Yadav, M., Mereacre, V., Lebedkin, S., Kappes, M. M., Powell, A. K. & Roesky, P. W. (2015). *Inorg. Chem.* **54**, 773–781.
- Yeşilel, O. Z., Mutlu, A., Günay, G., Caner, N., Ölmez, H. & Büyükgüngör, O. (2012). *J. Chem. Crystallogr.* **42**, 519–523.
- Zeng, L. (2012). *Acta Cryst.* **E68**, m329.
- Zeng, L. (2013). *Wuji Huaxue Xuebao*, **29**, 1149.
- Zheng, Z., Wu, R., Li, J., Han, Y. & Lu, J. (2010). *J. Coord. Chem.* **63**, 1118–1129.

supporting information

Acta Cryst. (2019). E75, 233-236 [https://doi.org/10.1107/S2056989019000744]

Crystal structure of *trans*-bis[2-(1*H*-benzotriazol-1-yl)acetato- κ O]bis(ethanolamine- κ^2 N,O)copper(II)

Guloy K. Alieva, Jamshid M. Ashurov, Shahnoza A. Kadirova, Bakhtiyar T. Ibragimov and Kasim A. Zakhidov

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2006; software used to prepare material for publication: *publCIF* (Westrip, 2010).

trans-Bis[2-(1*H*-benzotriazol-1-yl)acetato- κ O]bis(ethanolamine- κ^2 N,O)copper(II)

Crystal data

[Cu(C₈H₆N₃O₂)₂(C₂H₇NO)₂]

$M_r = 538.03$

Monoclinic, $P2_1/n$

$a = 12.4283$ (4) Å

$b = 4.84866$ (9) Å

$c = 20.6944$ (5) Å

$\beta = 105.823$ (3)°

$V = 1199.80$ (5) Å³

$Z = 2$

$F(000) = 558$

$D_x = 1.489$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 3723 reflections

$\theta = 3.7\text{--}75.7^\circ$

$\mu = 1.75$ mm⁻¹

$T = 293$ K

Block, blue

$0.36 \times 0.22 \times 0.12$ mm

Data collection

Rigaku Xcalibur Ruby
diffractometer

Radiation source: fine-focus sealed X-ray tube

Detector resolution: 10.2576 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Rigaku OD, 2015)

$T_{\min} = 0.558$, $T_{\max} = 1.000$

8833 measured reflections

2444 independent reflections

2152 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 75.9^\circ$, $\theta_{\min} = 3.8^\circ$

$h = -15 \rightarrow 15$

$k = -5 \rightarrow 5$

$l = -20 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.096$

$S = 1.06$

2444 reflections

173 parameters

3 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.364P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.24$ e Å⁻³

$\Delta\rho_{\min} = -0.31$ e Å⁻³

Extinction correction: SHELXL2014
 (Sheldrick, 2015b),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0009 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{iso}^*/U_{eq}
Cu	0.5000	0.5000	0.5000	0.03515 (14)
O2	0.53163 (12)	0.2852 (3)	0.58733 (6)	0.0434 (3)
O3	0.66203 (12)	0.2720 (3)	0.47288 (7)	0.0449 (3)
O1	0.46322 (14)	0.5886 (3)	0.64711 (7)	0.0535 (4)
N1	0.56782 (14)	0.3490 (4)	0.76528 (8)	0.0420 (4)
N4	0.62260 (16)	0.7586 (3)	0.54159 (8)	0.0432 (4)
N3	0.60695 (19)	0.6560 (5)	0.84410 (10)	0.0659 (6)
C7	0.57879 (18)	0.2195 (5)	0.70430 (9)	0.0458 (5)
H7A	0.5484	0.0342	0.7013	0.055*
H7B	0.6574	0.2054	0.7061	0.055*
N2	0.64071 (17)	0.5465 (5)	0.79544 (10)	0.0582 (5)
C8	0.51797 (16)	0.3819 (4)	0.64159 (9)	0.0383 (4)
C3	0.50966 (19)	0.5287 (5)	0.84514 (11)	0.0494 (5)
C2	0.48288 (17)	0.3334 (4)	0.79457 (10)	0.0430 (4)
C10	0.74518 (19)	0.4794 (5)	0.49439 (13)	0.0568 (6)
H10A	0.7391	0.6123	0.4585	0.068*
H10B	0.8187	0.3956	0.5041	0.068*
C9	0.73262 (19)	0.6246 (5)	0.55560 (12)	0.0557 (5)
H9A	0.7405	0.4930	0.5919	0.067*
H9B	0.7910	0.7621	0.5696	0.067*
C1	0.3878 (2)	0.1733 (5)	0.78222 (14)	0.0628 (6)
H1	0.3704	0.0434	0.7479	0.075*
C4	0.4385 (3)	0.5704 (7)	0.88710 (14)	0.0763 (8)
H4	0.4549	0.7005	0.9214	0.092*
C6	0.3200 (3)	0.2185 (7)	0.82423 (19)	0.0817 (9)
H6	0.2551	0.1146	0.8180	0.098*
C5	0.3452 (3)	0.4120 (7)	0.87504 (19)	0.0849 (10)
H5	0.2967	0.4345	0.9018	0.102*
H4A	0.609 (2)	0.836 (6)	0.5773 (10)	0.078 (9)*
H4B	0.623 (2)	0.895 (4)	0.5130 (11)	0.063 (7)*
H3	0.6329 (19)	0.295 (5)	0.4336 (6)	0.055 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.0477 (2)	0.0285 (2)	0.0312 (2)	0.00425 (15)	0.01401 (15)	0.00123 (13)
O2	0.0614 (9)	0.0372 (7)	0.0338 (6)	0.0079 (6)	0.0165 (6)	0.0036 (5)
O3	0.0546 (8)	0.0376 (7)	0.0420 (7)	0.0017 (6)	0.0123 (6)	0.0019 (6)
O1	0.0741 (10)	0.0485 (8)	0.0375 (7)	0.0204 (8)	0.0144 (7)	0.0023 (6)
N1	0.0441 (9)	0.0473 (10)	0.0336 (8)	-0.0002 (7)	0.0088 (6)	0.0049 (7)
N4	0.0584 (10)	0.0309 (9)	0.0406 (9)	0.0015 (7)	0.0143 (8)	-0.0008 (7)
N3	0.0681 (13)	0.0681 (14)	0.0559 (11)	-0.0137 (11)	0.0077 (10)	-0.0147 (10)
C7	0.0558 (12)	0.0469 (11)	0.0350 (9)	0.0103 (9)	0.0130 (8)	0.0053 (8)
N2	0.0507 (11)	0.0671 (13)	0.0535 (11)	-0.0122 (9)	0.0085 (8)	-0.0018 (9)
C8	0.0472 (10)	0.0361 (10)	0.0322 (8)	0.0001 (8)	0.0119 (7)	0.0025 (7)
C3	0.0562 (12)	0.0505 (13)	0.0401 (10)	0.0043 (10)	0.0109 (9)	0.0045 (9)
C2	0.0446 (10)	0.0441 (11)	0.0385 (9)	0.0040 (8)	0.0082 (8)	0.0103 (8)
C10	0.0449 (11)	0.0573 (14)	0.0721 (15)	-0.0031 (10)	0.0227 (11)	-0.0066 (11)
C9	0.0502 (12)	0.0472 (13)	0.0611 (13)	-0.0009 (10)	0.0009 (10)	-0.0049 (10)
C1	0.0580 (14)	0.0556 (15)	0.0722 (15)	-0.0095 (11)	0.0133 (12)	0.0092 (12)
C4	0.105 (2)	0.0715 (18)	0.0614 (15)	0.0215 (17)	0.0369 (16)	0.0050 (13)
C6	0.0641 (16)	0.0718 (19)	0.121 (3)	0.0015 (14)	0.0447 (17)	0.0307 (19)
C5	0.087 (2)	0.082 (2)	0.105 (2)	0.0210 (18)	0.060 (2)	0.0341 (19)

Geometric parameters (\AA , $^\circ$)

Cu—N4 ⁱ	1.9798 (18)	C7—H7A	0.9700
Cu—N4	1.9798 (18)	C7—H7B	0.9700
Cu—O2	2.0292 (12)	C3—C2	1.383 (3)
Cu—O2 ⁱ	2.0293 (12)	C3—C4	1.413 (4)
Cu—O3 ⁱ	2.4917 (15)	C2—C1	1.378 (3)
O2—C8	1.270 (2)	C10—C9	1.494 (3)
O3—C10	1.424 (3)	C10—H10A	0.9700
O3—H3	0.802 (10)	C10—H10B	0.9700
O1—C8	1.234 (2)	C9—H9A	0.9700
N1—N2	1.349 (3)	C9—H9B	0.9700
N1—C2	1.355 (3)	C1—C6	1.383 (4)
N1—C7	1.449 (2)	C1—H1	0.9300
N4—C9	1.470 (3)	C4—C5	1.356 (5)
N4—H4A	0.885 (10)	C4—H4	0.9300
N4—H4B	0.887 (10)	C6—C5	1.380 (5)
N3—N2	1.305 (3)	C6—H6	0.9300
N3—C3	1.363 (3)	C5—H5	0.9300
C7—C8	1.530 (3)		
N4 ⁱ —Cu—N4	180.0	N3—C3—C4	130.7 (3)
N4 ⁱ —Cu—O2	90.08 (6)	C2—C3—C4	120.1 (2)
N4—Cu—O2	89.92 (6)	N1—C2—C3	104.13 (18)
N4 ⁱ —Cu—O2 ⁱ	89.92 (6)	N1—C2—C1	132.9 (2)
N4—Cu—O2 ⁱ	90.08 (6)	C3—C2—C1	122.9 (2)

O2—Cu—O2 ⁱ	180.0	O3—C10—C9	111.30 (18)
C8—O2—Cu	123.91 (12)	O3—C10—H10A	109.4
C10—O3—H3	107.8 (19)	C9—C10—H10A	109.4
N2—N1—C2	109.84 (17)	O3—C10—H10B	109.4
N2—N1—C7	120.00 (17)	C9—C10—H10B	109.4
C2—N1—C7	129.44 (18)	H10A—C10—H10B	108.0
C9—N4—Cu	111.72 (13)	N4—C9—C10	110.35 (18)
C9—N4—H4A	113.6 (19)	N4—C9—H9A	109.6
Cu—N4—H4A	110 (2)	C10—C9—H9A	109.6
C9—N4—H4B	106.4 (17)	N4—C9—H9B	109.6
Cu—N4—H4B	109.1 (18)	C10—C9—H9B	109.6
H4A—N4—H4B	106 (3)	H9A—C9—H9B	108.1
N2—N3—C3	107.5 (2)	C2—C1—C6	115.6 (3)
N1—C7—C8	111.94 (16)	C2—C1—H1	122.2
N1—C7—H7A	109.2	C6—C1—H1	122.2
C8—C7—H7A	109.2	C5—C4—C3	116.9 (3)
N1—C7—H7B	109.2	C5—C4—H4	121.5
C8—C7—H7B	109.2	C3—C4—H4	121.5
H7A—C7—H7B	107.9	C5—C6—C1	122.4 (3)
N3—N2—N1	109.23 (18)	C5—C6—H6	118.8
O1—C8—O2	126.26 (17)	C1—C6—H6	118.8
O1—C8—C7	119.79 (16)	C4—C5—C6	122.0 (3)
O2—C8—C7	113.94 (17)	C4—C5—H5	119.0
N3—C3—C2	109.3 (2)	C6—C5—H5	119.0
N2—N1—C7—C8	-86.8 (2)	C7—N1—C2—C1	8.5 (4)
C2—N1—C7—C8	82.4 (3)	N3—C3—C2—N1	0.9 (2)
C3—N3—N2—N1	-0.6 (3)	C4—C3—C2—N1	-179.9 (2)
C2—N1—N2—N3	1.2 (2)	N3—C3—C2—C1	-178.9 (2)
C7—N1—N2—N3	172.31 (19)	C4—C3—C2—C1	0.3 (3)
Cu—O2—C8—O1	16.3 (3)	Cu—N4—C9—C10	50.9 (2)
Cu—O2—C8—C7	-162.68 (14)	O3—C10—C9—N4	-60.3 (3)
N1—C7—C8—O1	-3.5 (3)	N1—C2—C1—C6	179.8 (2)
N1—C7—C8—O2	175.61 (17)	C3—C2—C1—C6	-0.4 (3)
N2—N3—C3—C2	-0.2 (3)	N3—C3—C4—C5	179.0 (3)
N2—N3—C3—C4	-179.3 (3)	C2—C3—C4—C5	-0.1 (4)
N2—N1—C2—C3	-1.2 (2)	C2—C1—C6—C5	0.4 (4)
C7—N1—C2—C3	-171.29 (19)	C3—C4—C5—C6	0.0 (5)
N2—N1—C2—C1	178.6 (2)	C1—C6—C5—C4	-0.1 (5)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O1 ⁱ	0.80 (1)	1.86 (1)	2.634 (2)	163 (3)
N4—H4A \cdots O2 ⁱⁱ	0.89 (1)	2.41 (2)	3.046 (2)	129 (2)
N4—H4B \cdots O3 ⁱⁱ	0.89 (1)	2.12 (1)	2.973 (2)	161 (2)

C7—H7A···O1 ⁱⁱⁱ	0.97	2.53	3.449 (3)	158
C9—H9A···N3 ^{iv}	0.97	2.58	3.345 (3)	136

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, y+1, z$; (iii) $x, y-1, z$; (iv) $-x+3/2, y-1/2, -z+3/2$.