Zhu Jiaping, Wang Gang and Wu Dang*

The cocrystal structure of 1'-hydroxy-1*H*,1'*H*-[5,5'-bitetrazol]-1-olate and 1,10-phenanthroline-1-ium, C₁₄H₁₀N₁₀O₂

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker-AXS SMART APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9417, 3263, 0.047
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2913
$N(\text{param})_{\text{refined}}$:	235
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.33900(15)	0.50833(15)	0.46701(8)	0.0256(3)
O2	0.38976(16)	0.72023(16)	0.55757(9)	0.0302(4)
H2	0.3693	0.6439	0.5312	0.036*
N1	0.36559(17)	0.55329(17)	0.39340(10)	0.0204(3)
N2	0.31312(19)	0.48673(19)	0.31947(10)	0.0277(4)
N3	0.3614(2)	0.5577(2)	0.26166(11)	0.0324(4)
N4	0.4424(2)	0.66820(19)	0.29706(10)	0.0286(4)
N5	0.62572(18)	0.84444(18)	0.44086(10)	0.0248(4)
N6	0.66177(18)	0.91214(19)	0.51716(11)	0.0271(4)
N7	0.58173(18)	0.87123(18)	0.56821(10)	0.0251(4)
N8	0.49148(16)	0.77746(17)	0.52303(10)	0.0210(4)
N9	0.20923(17)	0.49282(18)	0.61644(9)	0.0215(4)
N10	0.11072(16)	0.33939(16)	0.46999(9)	0.0184(3)
H10	0.1701	0.4061	0.4720	0.022*
C1	0.4440(2)	0.6639(2)	0.38011(11)	0.0204(4)
C2	0.5177(2)	0.7598(2)	0.44529(11)	0.0203(4)
C3	0.2518(2)	0.5676(2)	0.68718(12)	0.0243(4)
H3	0.3261	0.6342	0.6895	0.029*
C4	0.1936(2)	0.5543(2)	0.75901(12)	0.0251(4)
H4	0.2266	0.6126	0.8076	0.030*
C5	0.0893(2)	0.4568(2)	0.75854(12)	0.0236(4)
H5	0.0491	0.4463	0.8067	0.028*
C6	0.0420(2)	0.3719(2)	0.68530(11)	0.0201(4)
C7	0.10518(19)	0.39631(19)	0.61598(11)	0.0183(4)
C8	0.05505(19)	0.31538(19)	0.53900(11)	0.0181(4)
C9	−0.0504(2)	0.2118(2)	0.53371(12)	0.0201(4)
C10	−0.1079(2)	0.1868(2)	0.60695(12)	0.0233(4)

<https://doi.org/10.1515/ncrs-2019-0633>

Received August 28, 2019; accepted October 16, 2019; available online November 2, 2019

Abstract

C₁₄H₁₀N₁₀O₂, monoclinic, *P*2₁/*n* (no. 14), *a* = 9.6366(19) Å, *b* = 9.5098(19) Å, *c* = 16.095(3) Å, β = 103.59(3)°, *V* = 1433.7(5) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0579, *wR*_{ref}(*F*²) = 0.1309, *T* = 153(2) K.

CCDC no.: 1561086

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Wu Dang**, Department of Applied Chemistry, School of Materials Science and Engineering, Guangdong University of Petrochemical Technology, Maoming, Guangdong, China; and Department of Polymers, School of Materials Science and Engineering, Guangdong University of Petrochemical Technology, Maoming, Guangdong, China, e-mail: wudangxtu@163.com

Zhu Jiaping and Wang Gang: Department of Applied Chemistry, School of Chemistry, Guangdong University of Petrochemical Technology, Maoming, Guangdong, China. <https://orcid.org/0000-0002-7395-8477> (Z. Jiaping)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H10A	−0.1772	0.1151	0.6049	0.028*
C11	−0.0642(2)	0.2647(2)	0.67914(12)	0.0227(4)
H11	−0.1050	0.2476	0.7265	0.027*
C12	−0.0942(2)	0.1367(2)	0.45632(12)	0.0233(4)
H12	−0.1657	0.0664	0.4509	0.028*
C13	−0.0339(2)	0.1648(2)	0.38886(12)	0.0238(4)
H13	−0.0632	0.1137	0.3369	0.029*
C14	0.0704(2)	0.2686(2)	0.39704(12)	0.0215(4)
H14	0.1125	0.2887	0.3506	0.026*

Source of material

1*H*,1'*H*-[5,5'-bitetrazole]-1,1'-diol (BTO) was synthesized according to the method using the procedure reported in the literature TM [5]. BTO (1.00 g) and 1,10-phenanthroline (Phen) (1.06 g) were dissolved in 30 mL of C₂H₅OH, heated to reflux 30 min, and then cooled to room temperature. The colourless precipitate formed was filtered and dried under vacuum, yielding 2.01 g (97.6%) of product. IR (KBr, ν/cm^{−1}): 3496, 3399, 3059, 2739, 2082, 1989, 1635, 1616, 1595, 1544, 1502, 1473, 1450, 1406, 1382, 1318, 1238, 1219, 1191, 1142, 1095, 1050, 1004, 957, 884, 858, 787, 736, 719; **Elemental Analysis** (%) calcd. for C₁₄H₁₀N₁₀O₂ (BTO.Phen, 350.10): C, 48.00; H, 2.88; N, 39.99; found: C, 47.98; H, 2.90; N, 40.00.

Experimental details

A crystal of dimension 0.24 × 0.23 × 0.20 mm³ of the title compound (BTO[−]Phen⁺) was mounted on a MiTeGen MicroMesh using a small amount of Cargille immersion oil. Data were collected on a Bruker three-circle diffractometer equipped with a Bruker-AXS SMART APEX II CCD detector. The crystal was irradiated using graphite monochromated MoK α radiation ($\lambda = 0.71073$). An Oxford Cobra low-temperature device was used to keep the crystals at a constant 153(2) K during data collection. The structure was refined by the full-matrix-block least-squares method against *F*² with anisotropic thermal parameters for all non-hydrogen atoms. H-atoms bonded to C-atoms were placed geometrically and refined using a riding model with common isotropic

displacement factors *U*_{iso}(H) = 1.2 (parent atom). H-atoms bonded to O- and N-atoms were located in a Fourier difference map and later refined using the SHELXL AFIX 3 command with *U*_{iso}(H) = 1.2 (parent atom).

Comment

The ionic nature of the co-crystal was confirmed by single-crystal X-ray diffraction. All bond lengths and angles are within normal ranges. An intramolecular hydrogen bond is formed between O(1) and O(2) in the anion [$\angle$ DHA = 170.4° and d(D···A) = 2.468(2)]. BTA[−] and Phen⁺ are hydrogen bonded via N(10)–H(10)···O(1) [$\angle$ DHA = 161.7° and d(D···A) = 2.734(2)]. Further C–H···N contacts contribute the stability of the structure. The structures of other BTO salts have been reported in the literature [6]. BTO.Phen crystallizes in the monoclinic space group *P*2₁/*n*. The two five-membered rings in the anion are not coplanar [torsion angles of N(8)–C(2)–C(1)–N(4) = 162.19° and N(5)–C(2)–C(1)–N(1) = 159.87°].

Acknowledgements: We gratefully acknowledge support by the Scientific Research Fund of Guangdong University of Petrochemical Technology (Nos. 518044 and 519015), and the Program for Innovative Research Team of Guangdong University of Petrochemical Technology (No. 519124).

References

1. Bruker. SMART, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. Fischer, N.; Klapötke, T. M.; Reymann, M.; Stierstorfer, J.: Nitrogen-rich salts of 1*H*,1'*H*-5,5'-bitetrazole-1,1'-diol: energetic materials with high thermal stability. *Chem. Eur. J.* **18** (2012) 4051–4062.
6. Shang, Y.; Jin, B.; Peng, R.; Guo, Z.; Liu, Q.; Zhao, J.; Zhang, Q.: Nitrogen-rich energetic salts of 1*H*,1'*H*-5,5'-bistetrazole-1,1'-diolate: synthesis, characterization, and thermal behaviors. *RSC Adv.* **6** (2016) 48590–48598.