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Redetermination of the crystal structure of (*E*)-5-bromo-2-hydroxybenzaldehyde oxime, $C_7H_6BrNO_2$

Table 1: Data collection and handling.

Crystal:	block
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	5.23 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6653, 1378, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1259
$N(\text{param})_{\text{refined}}$:	107
Programs:	Bruker [1], SHELX [2, 3]

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Abstract

$C_{11}H_{14}ClNO$, monoclinic, $P2_1/c$ (no. 14), $a = 14.48(2)$ Å, $b = 3.956(6)$ Å, $c = 13.765(19)$ Å, $\beta = 99.266(15)^\circ$, $V = 778.4(19)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0236$, $wR_{\text{ref}}(F^2) = 0.0584$, $T = 296(2)$ K.

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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.

Source of material

5-Bromosalicylaldehyde (1.61 g, 8 mmol), hydroxylamine hydrochloride (0.61 g, 8.8 mmol) and ethanol (30 mL) were

heated and stirred at 373.15 K for 8 h, then the solvent was evaporated under reduced pressure. The residue was dissolved by ethanol and the crystals of the title compound were obtained by slow evaporation within 3 days.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the hydrogen atoms of phenolic hydroxyl groups were set to $1.5U_{\text{eq}}(\text{C})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(\text{C})$.

Comment

Oximes are a class of Schiff base compounds synthesized by simple reactions of aldehydes or ketones with hydroxylamine. Salicylaldehyde has a good ability to coordinate metal ions. It is one of the most widely used high-efficiency and high-selective extractants in the field of mineral processing and metallurgy [4–6]. It also has important applications in the biopharmaceutical industry and organic analysis [7, 8]. Salicylaldehyde has attracted scientists' interest in its structure because of the presence of intramolecular and intermolecular hydrogen bonds [9–11].

The title structure has been determined decades ago [12]. This older determination didn't contain the hydrogen atom positions. This redetermination provides the hydrogen atoms in this molecule and verifies a typical intramolecular hydrogen bond (see the Figure) [12] between the phenolic

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.58290 (2)	0.81760 (9)	0.59251 (2)	0.06301 (16)
C1	0.81912 (16)	0.7944 (6)	0.80740 (17)	0.0390 (5)
C2	0.78838 (16)	0.9590 (6)	0.88658 (16)	0.0416 (5)
C3	0.69726 (18)	1.0747 (7)	0.87766 (19)	0.0498 (6)
H3	0.677011	1.180994	0.930708	0.060*
C4	0.63643 (17)	1.0337 (7)	0.79082 (19)	0.0494 (6)
H4	0.575276	1.112314	0.784981	0.059*
C5	0.66677 (16)	0.8750 (6)	0.71246 (18)	0.0441 (6)
C6	0.75665 (16)	0.7547 (6)	0.71984 (18)	0.0419 (5)
H6	0.775802	0.646778	0.666445	0.050*
C7	0.91388 (16)	0.6612 (6)	0.81297 (17)	0.0419 (5)
H7	0.929444	0.537401	0.760486	0.050*
N1	0.97537 (14)	0.7119 (5)	0.88841 (15)	0.0449 (5)
O1	0.84654 (13)	1.0063 (6)	0.97403 (13)	0.0563 (5)
H1	0.896 (2)	0.932 (10)	0.975 (3)	0.084*
O2	1.06152 (12)	0.5634 (6)	0.87855 (14)	0.0575 (5)
H2	1.097 (3)	0.628 (9)	0.927 (3)	0.086*

hydroxyl group and the nitrogen atom of the imine form. The molecule adopts a *E*-configuration about the C(7)–N(1) double bond (1.270(3) Å). The bond length and bond angle in the molecular structure are within the expected range. The torsion angle of C(1)–C(7)–N(1)–O(2) is 179.8(2) and the bond angle of C(7)–N(1)–O(2) is 111.8(2).

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References

1. Bruker. *APEX3, SAINT-Plus, XPREP*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2016.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Hoang A. S., Tran T. H., Nguyen H. N., Vu H. S., Vo T. P., Phan C., Nguyen T. V. Synthesis of oxime from a renewable resource for metal extraction. *Kor. J. Chem. Eng.* 2015, *32*, 1598–1605.
5. Naik P. U., Mcmanus G. J., Zaworotko M. J., Singer R. D. Salicylaldoxime and salen containing imidazolium ionic liquids for biphasic catalysis and metal extractions. *Dalton Trans* 2008, 36, 4834–4836.
6. Lasheen T. A., Ibrahim M. E., Hassib H. B., Helal A. S. Recovery of molybdenum from uranium bearing solution by solvent extraction with 5-Nonylsalicylaldoxime. *Hydrometallurgy* 2014, *146*, 175–182.
7. Tuccinardi T., Bertini S., Granchi C., Ortore G., Macchia M., Minutolo F., Martinelli A., Supuran C. T. Salicylaldoxime derivatives as new leads for the development of carbonic anhydrase inhibitors. *Bioorg. Med. Chem. Lett.* 2013, *21*, 1511–1515.
8. Gao H.-P., Su P., Shi Y.-L., Shen X.-X., Zhang Y.-M., Dong J.-Y., Zhang J. Discovery of novel VEGFR-2 inhibitors. part II: biphenyl urea incorporated with salicylaldoxime. *Eur. J. Med. Chem.* 2015, *90*, 232–240.
9. Forgan R. S., Roach B. D., Wood P. A., White F. J., Campbell J., Henderson D. K., Kamenetzky E., McAllister F. E., Parsons S., Pidcock E., Richardson P., Swart R. M., Tasker P. A. Using the outer coordination sphere to tune the strength of metal extractants. *Inorg. Chem.* 2011, *50*, 4515–4522.
10. Forgan R. S., Wood P. A., Campbell J., Henderson D. K., McAllister F. E., Parsons S., Pidcock E., Swart R. M., Tasker P. A. Supramolecular chemistry in metal recovery; H-bond buttressing to tune extractant strength. *Chem. Commun.* 2007, *46*, 4940–4942.
11. Peng Y.-D., Zhang Y., Ren Z.-L., Wang L., Dong W.-K. Crystal structure of (*E*)-2-hydroxybenzaldehyde O-(2-(((*E*)-(4-(dimethylamino)benzylidene)amino)oxy)ethyl)oxime, $\text{C}_{18}\text{H}_{21}\text{N}_3\text{O}_3$. *Z. Kristallogr. N. Cryst. Struct.* 2018, *233*, 635–636.
12. Simonsen S. H., Pfluger C. E., Thompson C. M. The crystal structures of some metallo-organic chelate compounds. I. the ligand 5-chlorosalicylaldoxime. *Acta Crystallogr* 1961, *14*, 269–272.