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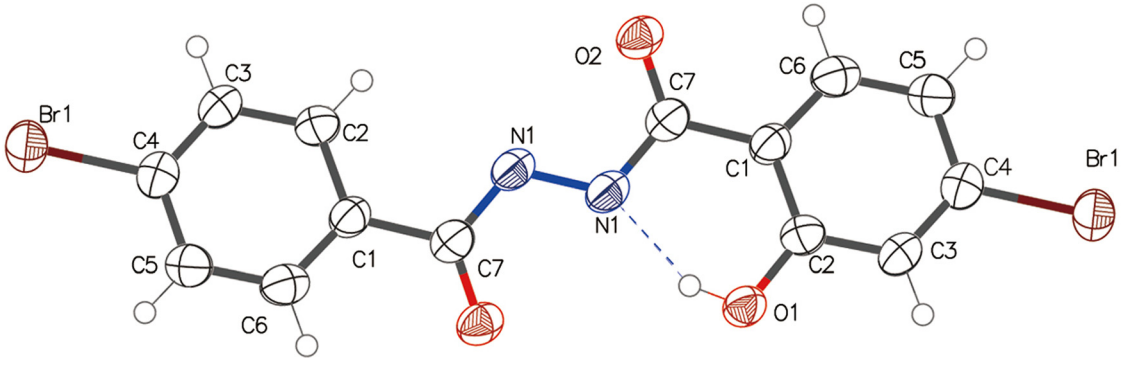
Crystal structure of 4-bromo-*N*′-[(3-chloro-2-hydroxyphenyl)methylidene]benzohydrazide, $C_{14}H_7Br_2N_2O_2$



https://doi.org/10.1515/ncrs-2020-0586
Received November 9, 2020; accepted November 29, 2020; published online December 16, 2020

Abstract

$C_{14}H_7Br_2N_2O_2$, triclinic, $P2_1/n$ (no. 14), a = 4.8308(2) Å, b = 6.1438(2) Å, c = 23.0836(9) Å, β = 91.0784(14)°, V = 684.99(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0477, $wR_{ref}(F^2)$ = 0.1108, T = 150.0 K.

CCDC No.: 2043231

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

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.20 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	5.92 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7475, 1392, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1256
$N(param)_{refined}$:	101
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.19186 (15)	0.44226 (11)	0.29563 (3)	0.0497 (2)
O1[a]	0.3352 (16)	−0.0676 (15)	0.4698 (3)	0.0398 (19)
H1[a]	0.259304	−0.160050	0.491143	0.060*
O2[a]	−0.3915 (17)	−0.3968 (13)	0.4479 (4)	0.042 (2)
N1	0.0380 (10)	−0.4121 (8)	0.4838 (2)	0.0395 (12)
C2	0.1643 (12)	−0.0134 (10)	0.4274 (3)	0.0401 (14)
C6	−0.2070 (12)	−0.1013 (10)	0.3593 (3)	0.0428 (15)
H6	−0.357737	−0.191717	0.347766	0.051*
C1	−0.0621 (12)	−0.1478 (10)	0.4104 (3)	0.0369 (13)
C4	0.0867 (12)	0.2048 (10)	0.3430 (3)	0.0378 (13)
C7	−0.1447 (13)	−0.3393 (11)	0.4451 (3)	0.0439 (15)
C5	−0.1371 (12)	0.0729 (11)	0.3250 (3)	0.0418 (14)
H5	−0.237255	0.103173	0.290092	0.050*
C3	0.2328 (12)	0.1653 (10)	0.3933 (3)	0.0390 (13)
H3	0.379983	0.259160	0.404846	0.047*

[a]Occupancy: 0.5.

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idealized positions and refined using a riding model, with O–H = 0.84 (phenolic hydroxyl), 0.95 Å (benzene), and with U_{iso}(H) = 1.2 U_{eq}(C) for H atoms on phenolic hydroxyl and benzene.

Experimental details

4-Bromo-2-hydroxybenzaldehyde (0.201 g, 1.0 mmol) was dissolved in 30 mL methanol forming a colorless solution. Additionally 20 mL methanol containing 0.215 g 4-bromobenzohydrazide (1 mmol) was added to above solution. The obtained mixture stirred for 12 h at room temperature. Then the yellow filtrate was sealed in a beaker with cling film and kept undisturbed at room temperature. The yellow square crystals of the title compound were obtained after 1 week.

Comment

Hydrazones characterized by the –NHN=CH-group represent an important class of compounds in the field of medical science and ion recognition [5, 6]. As a part of our current research interest in exploring the relationship between molecular structure and physicochemical properties of halogenated Schiff-base compounds [7, 8], in this work, we report a new bromohydrazone.

X-ray diffraction analysis reveals that the compound contains a planar hydrazone molecule, as shown in the figure. The five non-hydrogen conjugated atoms [C(=O)N–N=C] constitute the central chromophore and over all exhibit an E configuration. The dihedral angles formed between the central plane and the least-squares planes through the flanking phenyl ring is 17.92(15)°, indicating the twisted nature of the whole molecule. In addition, the central chromophore and bromo-phenol ring generates an intramolecular hydroxy N–H···O hydrogen bond. [O1–H1···N1: O1–H1 = 0.84(1) Å, H1···N1 = d 1.888(5) Å, O1···N1 = 2.5813(10) Å with angle at H1 = 138.9]. Bond lengths and angles are in the expected ranges [9].

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Fund for Less Developed Regions of the National Natural Science Foundation of China (No. 31760257); Joint Basic Research Program (partial) of Yunnan Local Undergraduate Universities (2017FH001–002). The reserve academic and technical leaders of Yunnan Province (2019HB098).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Agilent Technologies. *CrysAlis^{PRO} Software System*; Agilent Technologies UK Ltd: Oxford, UK, 2015.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Cryst.* 2010, *42*, 339–341.
3. Sheldrick G. A short history of *SHELX*. *Acta Crystallogr.* 2008, *A64*, 112–122.
4. Sheldrick G. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Li Y., Wu Q., Lecren L., Clérac R. Synthesis, structure and magnetism of new polynuclear transition metal aggregates assembled with Schiff-base ligand and anionic *N*-donor ligands. *J. Mol. Struct.* 2008, *890*, 339–345.
6. Bedia K.-K., Elçin O., Seda U., Fatma K., Nathaly S., Sevim R., Dimoglo A. Synthesis and characterization of novel hydrazide-hydrazones and the study of their structure-antituberculosis activity. *Eur. J. Med. Chem.* 2006, *41*, 1253–1261.
7. Wu Q., Xiao J.-C., Zhou C., Sun J.-R., Huang M.-F., Xu X., Li T., Tian H. Crystal structure and supramolecular architecture of inorganic ligand-coordinated Salen-type Schiff base complex: insights into halogen bond from theoretical analysis and 3D energy framework calculations. *Crystals* 2020, *10*, 334.
8. Fang J., Yan L., Liu T., Xiongzi Y., Qiong W. Crystal structure of 6,6′-((cyclohexane-1,2-diylbis(azanylylidene))bis(methanylylidene)) bis(2- bromo-4-chlorophenolato-κ^4N,N',O,O')nickel(II), $C_{20}H_{16}Br_2Cl_2NiN_2O_2$. *Z. Kristallogr. NCS* 2020, *235*, 863–864.
9. Bart J. C. J., Schmidt G. M. J. Topochemistry. Part XXXVII. The crystal structure of dibenzoyldiimide. *Isr. J. Chem.* 1975, *13*, 23–34.