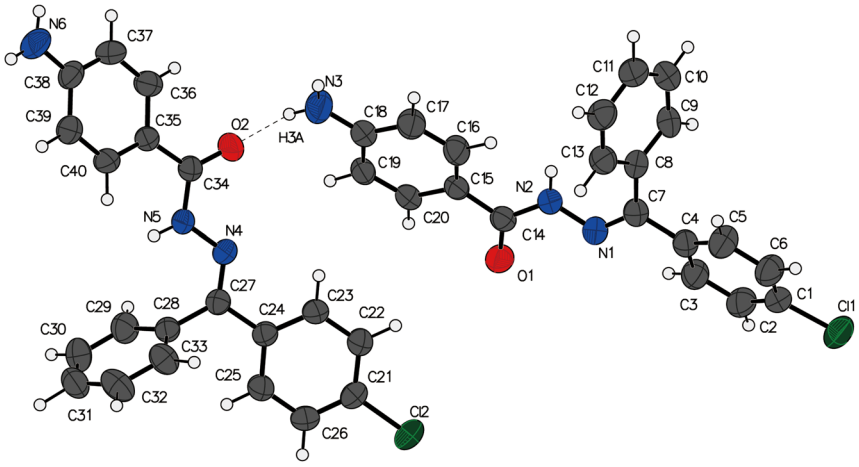
Yanli Song, Mo Du, Zhoujing Zhu, Wenqiang Tang and Hongjuan Tong*

Crystal structure of (*Z*)-4-amino-*N*′-((4-chlorophenyl)(phenyl)methylene) benzohydrazide, $C_{20}H_{16}ClN_3O$



https://doi.org/10.1515/ncrs-2024-0327
Received July 31, 2024; accepted September 27, 2024;
published online October 9, 2024

Abstract

$C_{20}H_{16}ClN_3O$, monoclinic, *I*2/*a* (no. 15), *a* = 16.3816(6) Å, *b* = 15.3173(5) Å, *c* = 27.7833(14) Å, *β* = 92.800(2)°, *V* = 6,963.1(5) Å^3, *Z* = 8, $R_{gt}(F)$ = 0.0375, w$R_{ref}(F^2)$ = 0.1061, T = 299 K.

CCDC no.: 2387239

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.64 × 0.61 × 0.57 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	2.04 mm^{-1}
Diffractometer, scan mode:	Bruker P4, *ω*
θ_{max}, completeness:	68.3°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	65,999, 6,370, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5,881
$N(param)_{refined}$:	579
Programs:	Bruker[1], SHELX[2,3], Olex2[4]

1 Source of materials

In a 25 mL screw-cap vial, 4-aminobenzohydrazide (0.151 g, 1 mmol), 4-chlorobenzophenone (0.216 g, 1 mmol), 5 drops of acetic acid, and 20 mL of anhydrous ethanol were combined. The solution was stirred at 50 °C for 10 min, resulting in the formation of a precipitate. Stirring was continued for an additional 5 h. The vial was then removed and allowed to cool. The cap was opened, and the solution was left to evaporate slowly in a fume hood to yield the crude product. To obtain crystals, 0.05 g of the crude product was dissolved in the minimum amount of hot ethanol. The solution was then allowed to slowly cool to room temperature in an open environment, leading to solvent evaporation and the formation of single crystals.

2 Experimental details

Hydrogen atoms were positioned in idealized locations and refined as riding atoms, with U_{iso} values set to 1.2 times U_{eq} of the parent atoms. The structure was solved using

***Corresponding author: Hongjuan Tong**, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China, E-mail: dearthj@126.com. https://orcid.org/0000-0001-7219-8018
Yanli Song, Mo Du, Zhoujing Zhu and Wenqiang Tang, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China. https://orcid.org/0000-0001-5150-4549 (W. Tang)

Open Access. © 2024 the author(s), published by De Gruyter. This work is licensed under the Creative Commons Attribution 4.0 International License.

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.67160 (11)	−0.02043 (10)	0.44575 (6)	0.0569 (4)
C2	0.61451 (12)	0.01201 (11)	0.47584 (6)	0.0635 (4)
H2	0.5850 (14)	−0.0290 (15)	0.4947 (8)	0.083 (6)*
C3	0.60501 (11)	0.10138 (11)	0.47954 (6)	0.0580 (4)
H3	0.5648 (13)	0.1274 (14)	0.4989 (8)	0.080 (6)*
C4	0.65166 (9)	0.15804 (9)	0.45313 (5)	0.0475 (3)
C5	0.70863 (11)	0.12306 (11)	0.42345 (7)	0.0624 (4)
H5	0.7408 (14)	0.1607 (14)	0.4052 (8)	0.083 (6)*
C6	0.71933 (12)	0.03382 (11)	0.41983 (7)	0.0652 (4)
H6	0.7577 (14)	0.0112 (15)	0.3988 (8)	0.088 (7)*
C7	0.64137 (9)	0.25393 (9)	0.45668 (5)	0.0471 (3)
C8	0.66250 (9)	0.30961 (9)	0.41478 (5)	0.0458 (3)
C9	0.62229 (10)	0.29926 (10)	0.36994 (5)	0.0528 (3)
H9	0.5819 (12)	0.2551 (13)	0.3656 (7)	0.067 (5)*
C10	0.63931 (12)	0.35384 (11)	0.33215 (6)	0.0602 (4)
H10	0.6094 (12)	0.3482 (13)	0.3027 (7)	0.068 (5)*
C11	0.69873 (11)	0.41759 (11)	0.33794 (6)	0.0613 (4)
H11	0.7151 (11)	0.4592 (12)	0.3073 (6)	0.061 (5)*
C12	0.74079 (11)	0.42663 (11)	0.38182 (6)	0.0589 (4)
H12	0.7814 (14)	0.4713 (14)	0.3864 (8)	0.084 (6)*
C13	0.72262 (9)	0.37386 (10)	0.42012 (6)	0.0519 (3)
H13	0.7514 (11)	0.3796 (12)	0.4521 (7)	0.063 (5)*
C14	0.58237 (8)	0.41127 (9)	0.54079 (5)	0.0442 (3)
C15	0.58645 (8)	0.50787 (9)	0.53996 (4)	0.0430 (3)
C16	0.62441 (11)	0.55616 (11)	0.50485 (6)	0.0581 (4)
H16	0.6510 (13)	0.5249 (13)	0.4767 (8)	0.078 (6)*
C17	0.62919 (13)	0.64565 (11)	0.50703 (6)	0.0641 (4)
H17	0.6555 (14)	0.6764 (14)	0.4808 (8)	0.084 (6)*
C18	0.59637 (10)	0.69173 (10)	0.54480 (5)	0.0509 (3)
C19	0.55971 (9)	0.64396 (10)	0.58069 (5)	0.0478 (3)
H19	0.5393 (11)	0.6729 (12)	0.6089 (7)	0.065 (5)*
C20	0.55464 (9)	0.55443 (10)	0.57802 (5)	0.0455 (3)
H20	0.5295 (11)	0.5205 (12)	0.6025 (7)	0.062 (5)*
Cl1	0.68145 (4)	−0.13304 (3)	0.43825 (2)	0.08771 (19)
N1	0.61418 (8)	0.28312 (8)	0.49651 (4)	0.0496 (3)
N2	0.60332 (8)	0.37180 (8)	0.49907 (5)	0.0485 (3)
H2A	0.6064 (11)	0.4022 (13)	0.4741 (7)	0.063 (5)*
N3	0.60002 (12)	0.78074 (10)	0.54676 (6)	0.0728 (4)
H3A	0.5807 (13)	0.8080 (14)	0.5714 (8)	0.076 (6)*
H3B	0.6330 (18)	0.812 (2)	0.5255 (10)	0.118 (9)*
O1	0.56531 (7)	0.37052 (7)	0.57673 (4)	0.0575 (3)
C21	0.61219 (9)	0.43664 (9)	0.75520 (5)	0.0490 (3)
C22	0.59571 (12)	0.47970 (10)	0.71232 (6)	0.0594 (4)
H22	0.5913 (13)	0.4476 (14)	0.6811 (8)	0.079 (6)*
C23	0.59014 (11)	0.56959 (10)	0.71270 (6)	0.0572 (4)
H23	0.5833 (13)	0.6012 (15)	0.6832 (8)	0.084 (6)*
C24	0.60126 (8)	0.61717 (9)	0.75533 (5)	0.0423 (3)
C25	0.61635 (11)	0.57096 (10)	0.79797 (5)	0.0546 (4)
H25	0.6242 (12)	0.6018 (13)	0.8279 (7)	0.075 (6)*
C26	0.62150 (11)	0.48085 (10)	0.79791 (6)	0.0579 (4)
H26	0.6323 (13)	0.4495 (14)	0.8267 (8)	0.078 (6)*
C27	0.60083 (8)	0.71391 (9)	0.75490 (5)	0.0412 (3)
C28	0.63291 (8)	0.76137 (8)	0.79893 (5)	0.0420 (3)
C29	0.58192 (11)	0.80748 (12)	0.82796 (6)	0.0599 (4)
H29	0.5247 (12)	0.8106 (12)	0.8183 (7)	0.065 (5)*
C30	0.61457 (14)	0.84975 (13)	0.86883 (7)	0.0733 (5)
H30	0.5808 (16)	0.8784 (17)	0.8882 (9)	0.101 (8)*
C31	0.69656 (13)	0.84653 (12)	0.88054 (6)	0.0656 (4)
H31	0.7243 (11)	0.8784 (12)	0.9122 (7)	0.061 (5)*
C32	0.74757 (12)	0.79955 (14)	0.85218 (6)	0.0668 (5)
H32	0.8096 (15)	0.7963 (15)	0.8595 (8)	0.092 (7)*
C33	0.71551 (10)	0.75643 (12)	0.81185 (5)	0.0560 (4)
H33	0.7519 (12)	0.7215 (13)	0.7930 (7)	0.074 (6)*
C34	0.56439 (8)	0.88071 (9)	0.66940 (5)	0.0427 (3)
C35	0.58366 (8)	0.97461 (8)	0.66574 (5)	0.0422 (3)
C36	0.54619 (10)	1.02080 (10)	0.62724 (6)	0.0540 (4)
H36	0.5076 (12)	0.9918 (13)	0.6068 (7)	0.064 (5)*
C37	0.56245 (11)	1.10742 (11)	0.61981 (6)	0.0576 (4)
H37	0.5364 (13)	1.1384 (14)	0.5942 (8)	0.081 (6)*
C38	0.61831 (10)	1.15239 (9)	0.65019 (5)	0.0509 (3)
C39	0.65547 (11)	1.10688 (10)	0.68894 (6)	0.0571 (4)
H39	0.6933 (12)	1.1364 (13)	0.7111 (7)	0.071 (5)*
C40	0.63830 (10)	1.01986 (9)	0.69642 (5)	0.0514 (3)
H40	0.6685 (12)	0.9870 (13)	0.7223 (7)	0.073 (5)*
Cl2	0.62282 (3)	0.32379 (2)	0.75548 (2)	0.06899 (14)
N4	0.57762 (7)	0.75098 (7)	0.71499 (4)	0.0437 (3)
N5	0.58464 (8)	0.84024 (7)	0.71229 (4)	0.0446 (3)
H5A	0.6014 (11)	0.8691 (12)	0.7365 (7)	0.054 (5)*
N6	0.63755 (13)	1.23736 (10)	0.64134 (7)	0.0708 (4)
H6A	0.6132 (14)	1.2601 (16)	0.6174 (9)	0.086 (7)*
H6B	0.6723 (16)	1.2633 (17)	0.6614 (9)	0.091 (8)*
O2	0.53179 (8)	0.84003 (7)	0.63579 (4)	0.0605 (3)

ShelXT[2] and refined with ShelXL[3] in the Olex2 software[4] employing full-matrix least-squares refinement.

3 Comment

Hydrazide compounds have recently garnered significant attention from both theoreticians and experimentalists due to their biological significance in medicinal and enzyme chemistry. Investigating the crystal structure of benzohydrazide derivatives is essential for the development of new drugs.

In the crystal structure, as illustrated, two orientations of molecules are arranged in a head-to-tail crossed manner, forming the asymmetric unit. All bond lengths and bond angles are within reasonable ranges, comparable to previously reported structures.[5–11] The molecular structure contains three types of benzene rings: para-aminobenzene group, para-chlorobenzene group, and phenyl group. The carbon atoms in the three benzene rings lie within their respective planes. Notable torsions exist between them: the dihedral angle between the para-aminobenzene group and

the para-chlorobenzene group planes is 18.5°, between the para-chlorobenzene group and the phenyl group planes is 76.8°, and between the para-aminobenzene group and the phenyl group planes is 60.3°.

There is a hydrogen bond N3–H3A···O2 between molecules, with a bond length of 2.06(3) Å and a bond angle of 165(3)°. The short bond length and the near 180° bond angle indicate high bond energy.

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

Research funding: This work was financially supported by the 2023 research and development project of the Xianyang Science and Technology Bureau (L2023-ZDYF-SF-030), Natural Science Foundation Project of Shaanxi Province (2024JC-YBMS-733), the Key Laboratory of Molecular Imaging and Drug Synthesis of Xianyang City (2021QXNL-PT-0008) and the youth innovation team of Shaanxi university 2023 "Key technology development and application innovation team of the industrialization of traditional Chinese medicine with northwest characteristics.

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

References

1. Bruker. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2012.
2. Sheldrick, G. M. SHELXT – 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. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A.; Puschmann, H. OLEX2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.
5. Li, Y.; Chen, C.; Yang, R.; Zhao, R. *N′*-(Diphenylmethylene)-2-Hydroxybenzohydrazide. *Acta Crystallogr.* **2009**, *E65*, o659.
6. Chang, J.-G. *N′*-[(1*E*)-(3,5–Dichloro-2-Hydroxyphenyl)(Phenyl) Methylene]-4-Methoxybenzohydrazide. *Acta Crystallogr.* **2008**, *E64*, o198.
7. Chang, J.-G.; Cheng, Y.; He, X.-T. *N′*-[(1*E*)-(5–Chloro-2-Hydroxyphenyl) (Phenyl)Methylene]-2-Hydroxybenzohydrazide. *Acta Crystallogr.* **2007**, *E63*, o474–o475.
8. Chang, J.-G.; Yang, R.-D. *N′*-[(1*E*)-(5-Chloro-2-Hydroxyphenyl)(Phenyl) Methylene]-4-Methoxybenzohydrazide. *Acta Crystallogr.* **2007**, *E63*, o4022.
9. Zheng, C.-Z.; Ji, C.-Y.; Chang, X.-L. *N′*-[(*E*)-(5-Bromo-2-Hydroxyphenyl)(Phenyl)Methylene] Benzohydrazide. *Acta Crystallogr.* **2009**, *E65*, o641.
10. Zheng, C.-Z.; Ji, C.-Y.; Chang, X.-L. *N′*-[(*E*)-(5-Bromo-2-Hydroxyphenyl)(Phenyl)Methylidene]-4-Chlorobenzohydrazide. *Acta Crystallogr.* **2009**, *E65*, o1944.
11. Ton, Q. C.; Bolte, M.; Egert, E. Structural Similarities Among Eight Benzoylhydrazones. *Acta Crystallogr.* **2014**, *C70*, 912–919.