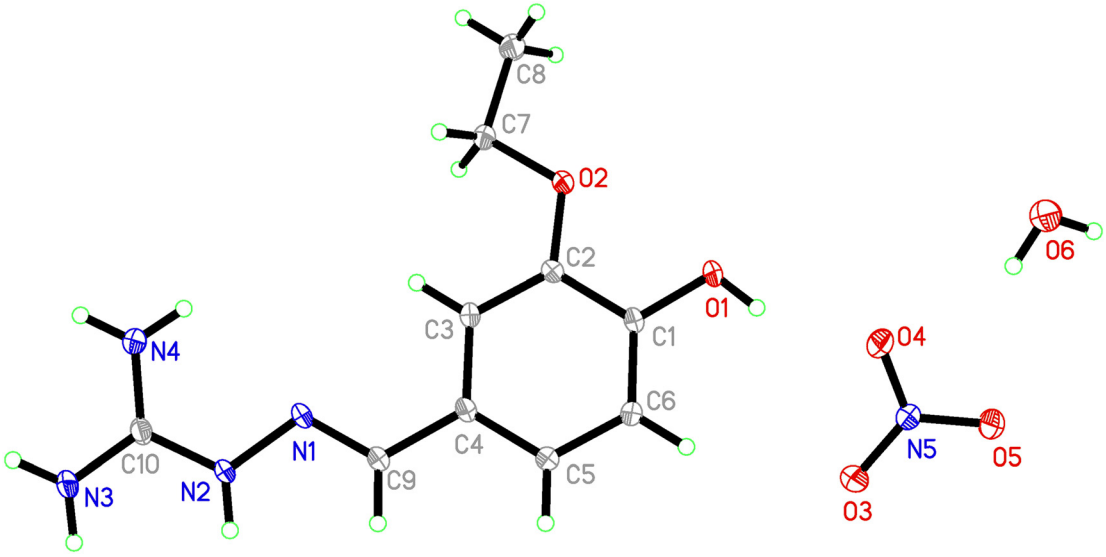
Ze-Sen Jin, E. Liu, Xiao-Jing Liu, Fang-Fang Jian* and Tongling Liang

Crystal structure of (*E*)-amino(2-(3-ethoxy-4-hydroxybenzylidene)hydrazineyl)methaniminium nitrate hemihydrate $C_{10}H_{16}N_5O_{5.5}$



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Abstract

$C_{10}H_{16}N_5O_{5.5}$, monoclinic, *C*2/*c* (no. 15), a = 17.7836(8) Å, b = 9.4796(4) Å, c = 15.8740(8) Å, β = 97.093(5)°, V = 2655.6(2) Å^3, Z = 8, $R_{gt}(F)$ = 0.0469, $wR_{ref}(F^2)$ = 0.1289, T = 170 K.

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.32 × 0.24 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.12 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC10 (RCD3), ω
θ_{max}, completeness:	30.9°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,999, 3563, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2866
$N(param)_{refined}$:	188
Programs:	CrysAlis^{PRO} [1], SHELX [2], Olex2 [3]

***Corresponding author: Fang-Fang Jian**, School of Chemical Engineering and Pharmaceutics, Henan University of Science and Technology, Luoyang Hekeda Recycling Energy Co.LTD, Luoyang, Henan 471003, P. R. China, E-mail: FangfJian@126.com
Ze-Sen Jin and Xiao-Jing Liu, School of Chemical Engineering and Pharmaceutics, Henan University of Science and Technology, Luoyang, Henan 471003, P. R. China. https://orcid.org/0000-0003-2287-4166 (Z.-S. Jin). https://orcid.org/0000-0002-5358-3479 (X.-J. Liu)
E. Liu, School of Chemical Engineering and Pharmaceutics, Henan University of Science and Technology, Luoyang Hekeda Recycling Energy Co.LTD, Luoyang, Henan 471003, P. R. China
Tongling Liang, Institute of Chemistry Chinese Academy of Sciences, Zhongguancun, Beijing 100190, P. R. China

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

Source of material

3-Ethoxy-4-hydroxybenzaldehyde (1.66 g, 0.01 mol) was placed in 15 ml ethanol at room temperature; then added to the aminoguanidine nitrate (1.37 g, 0.01 mol) solution containing 10 ml water and 8 ml ethanol. The mixture was

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.25543 (6)	0.39177 (10)	0.39196 (7)	0.0318 (3)
H1	0.229669	0.462203	0.379500	0.048*
O2	0.34455 (6)	0.18510 (9)	0.45047 (6)	0.0284 (2)
N1	0.54638 (6)	0.42068 (11)	0.66818 (7)	0.0232 (2)
N2	0.60827 (6)	0.46825 (12)	0.72163 (7)	0.0249 (3)
H2	0.619101	0.556643	0.725127	0.030*
N3	0.71269 (7)	0.41788 (13)	0.81569 (7)	0.0296 (3)
H3A	0.741245	0.358649	0.845643	0.036*
H3B	0.724219	0.506026	0.816807	0.036*
N4	0.63138 (7)	0.23972 (12)	0.76434 (8)	0.0317 (3)
H4A	0.658947	0.178319	0.793673	0.038*
H4B	0.591003	0.213595	0.732737	0.038*
C1	0.31426 (7)	0.42586 (14)	0.45138 (8)	0.0238 (3)
C2	0.36255 (7)	0.31581 (13)	0.48346 (8)	0.0226 (3)
C3	0.42383 (7)	0.34506 (14)	0.54301 (8)	0.0235 (3)
H3	0.455604	0.272354	0.564714	0.028*
C4	0.43850 (7)	0.48401 (14)	0.57102 (8)	0.0223 (3)
C5	0.39083 (8)	0.59158 (14)	0.53827 (9)	0.0243 (3)
H5	0.400507	0.683902	0.556244	0.029*
C6	0.32881 (8)	0.56270 (14)	0.47890 (9)	0.0258 (3)
H6	0.296932	0.635487	0.457544	0.031*
C7	0.39926 (9)	0.07587 (14)	0.47193 (10)	0.0312 (3)
H7A	0.405204	0.058470	0.532605	0.037*
H7B	0.448020	0.103260	0.455700	0.037*
C8	0.37056 (10)	−0.05443 (16)	0.42450 (12)	0.0413 (4)
H8A	0.362942	−0.034757	0.364724	0.062*
H8B	0.323398	−0.082812	0.442902	0.062*
H8C	0.406991	−0.128946	0.435556	0.062*
C9	0.50415 (7)	0.51750 (14)	0.63196 (8)	0.0232 (3)
H9	0.515566	0.611410	0.644769	0.028*
C10	0.65093 (7)	0.37333 (14)	0.76780 (8)	0.0238 (3)
O3	0.21323 (6)	0.76008 (11)	0.32610 (7)	0.0364 (3)
O4	0.14713 (7)	0.56968 (11)	0.33254 (9)	0.0454 (3)
O5	0.10398 (8)	0.73893 (12)	0.25133 (9)	0.0528 (4)
N5	0.15564 (7)	0.69121 (12)	0.30281 (8)	0.0307 (3)
O6	0.000000	0.4579 (2)	0.250000	0.0815 (9)
H6A[a]	0.032720	0.516237	0.272680	0.122*
H6B[a]	−0.021220	0.500368	0.206200	0.122*

[a]Occupancy: 0.5.

heated and stirred for 8 h, cooled to room temperature. The precipitate was removed, and the filtrate was left standing to precipitate brown crystals.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

In the field of coordination chemistry, Schiff bases are important organic ligands [4]. Because they contain the >C=N– group, the N atom contains lone electron pairs, and all kinds of functional atomic groups can be introduced around the >C=N– group, which makes it widely studied in the fields of chemistry and biology [5, 6]. Chemists use a variety of amines, aldehydes and ketones for different coordination to form Schiff base compounds with different shapes to meet their different needs for materials in different fields such as materials [7], optics [8] and so on. As an intermediate of medicine and organic synthesis, aminoguanidine has always been a research hotspot in the medical field [9]. Therefore, according to previous studies [10, 11], we synthesized the title compound.

The title compound is formed by dehydration and condensation of amine aldehyde and consists of a positively charged $[C_{10}H_{15}N_4O_2]^+$, a nitrate anion and a water. The C1=N9 bond length of the compound is 1.2767 Å, which is within the normal range. Other bond lengths and angles are also within the normal range.

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

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