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Crystal structure of bis(2-((*E*)-5-chloro-2-hydroxybenzylidene)hydrazineyl)methaniminium trifluoroacetate dihydrate, $C_{34}H_{36}Cl_4N_{10}O_{12}$

<https://doi.org/10.1515/ncrs-2021-0150>

Received April 21, 2021; accepted May 10, 2021;
published online August 2, 2021

Abstract

$C_{34}H_{36}Cl_4N_{10}O_{12}$, triclinic, $P\bar{1}$ (no. 2), $a = 10.3071(3)$ Å, $b = 13.4696(4)$ Å, $c = 16.1893(4)$ Å, $\alpha = 81.467(2)^\circ$, $\beta = 77.726(2)^\circ$, $\gamma = 82.055(2)^\circ$, $V = 2158.64(11)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0539$, $wR_{ref}(F^2) = 0.1609$, $T = 170$ K.

CCDC no.: 2082670

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.17 × 0.13 × 0.12 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ :	3.37 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Synergy R, ω
θ_{max} , completeness:	75.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	28,700, 8586, 0.030
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 7352
$N(param)_{refined}$:	633
Programs:	CRYALIS ^{PRO} [1], SHELX [2], OLEX2 [3]

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}
Cl1	−0.05533 (10)	0.33439 (6)	0.37696 (5)	0.0661 (2)
Cl2	0.14210 (7)	1.26659 (5)	−0.34515 (4)	0.04321 (17)
O1	0.3043 (2)	0.62940 (15)	0.38860 (12)	0.0504 (5)
H1	0.333580	0.617187	0.432774	0.076*
O2	−0.0586 (2)	0.88453 (14)	−0.18364 (11)	0.0430 (4)
H2	−0.031111	0.863916	−0.139548	0.065*
N1	0.12845 (19)	0.66670 (14)	0.18500 (11)	0.0302 (4)
N2	0.16555 (19)	0.74812 (14)	0.12622 (11)	0.0315 (4)
H2A	0.232408	0.778377	0.129051	0.038*
N3	0.0020 (2)	0.72751 (15)	0.05418 (12)	0.0340 (4)
H3A	−0.041282	0.748089	0.013808	0.041*
H3B	−0.015719	0.673196	0.087653	0.041*
N4	0.12725 (19)	0.86369 (14)	0.01494 (11)	0.0303 (4)
H4	0.181767	0.899164	0.027694	0.036*
N5	0.07320 (19)	0.89460 (14)	−0.05701 (11)	0.0298 (4)
C1	0.0520 (3)	0.42164 (19)	0.37996 (16)	0.0398 (5)
C2	0.1145 (3)	0.4123 (2)	0.44901 (16)	0.0430 (6)
H2B	0.098845	0.359706	0.492841	0.052*
C3	0.1990 (3)	0.4806 (2)	0.45245 (15)	0.0406 (6)
H3	0.240857	0.474352	0.498814	0.049*
C4	0.2232 (3)	0.55987 (19)	0.38667 (15)	0.0370 (5)
C5	0.1621 (2)	0.56839 (17)	0.31608 (14)	0.0317 (5)
C6	0.0749 (2)	0.49875 (18)	0.31357 (14)	0.0356 (5)
H6	0.032434	0.504091	0.267583	0.043*
C7	0.1904 (2)	0.65063 (17)	0.24722 (14)	0.0325 (5)
H7	0.254656	0.692112	0.248668	0.039*
C8	0.0951 (2)	0.77892 (16)	0.06471 (13)	0.0284 (4)
C9	0.1150 (2)	0.97549 (16)	−0.10113 (13)	0.0293 (4)
H9	0.175378	1.007361	−0.081872	0.035*
C10	0.0721 (2)	1.01930 (17)	−0.17971 (13)	0.0298 (4)
C11	0.1193 (2)	1.11005 (17)	−0.22061 (14)	0.0318 (5)
H11	0.177857	1.139608	−0.197751	0.038*
C12	0.0797 (2)	1.15596 (18)	−0.29444 (14)	0.0350 (5)
C13	−0.0083 (3)	1.1138 (2)	−0.32933 (15)	0.0413 (6)
H13	−0.035914	1.145922	−0.378597	0.050*
C14	−0.0545 (3)	1.0235 (2)	−0.29023 (16)	0.0435 (6)
H14	−0.113802	0.995202	−0.313421	0.052*
C15	−0.0137 (2)	0.97451 (19)	−0.21672 (14)	0.0345 (5)
Cl1A	0.86785 (9)	1.10630 (6)	0.47209 (5)	0.0644 (2)
Cl2A	0.64381 (7)	0.18672 (5)	1.20335 (4)	0.04497 (17)
O1A	0.5085 (2)	0.81325 (15)	0.45436 (13)	0.0521 (5)
H1A	0.467667	0.832969	0.415630	0.078*
O2A	0.86160 (19)	0.55755 (13)	1.02652 (12)	0.0426 (4)
H2AA	0.825472	0.581364	0.986355	0.064*
N1A	0.6830 (2)	0.76981 (15)	0.65886 (12)	0.0342 (4)
N2A	0.6516 (2)	0.68375 (15)	0.71339 (12)	0.0352 (4)
H2AB	0.605857	0.641864	0.700136	0.042*
N3A	0.7739 (2)	0.72526 (16)	0.80528 (13)	0.0395 (5)
H3AA	0.800238	0.775575	0.769243	0.047*
H3AB	0.799824	0.712970	0.853246	0.047*
N4A	0.6527 (2)	0.58653 (15)	0.84019 (12)	0.0345 (4)
H4A	0.593090	0.554086	0.829610	0.041*
N5A	0.7067 (2)	0.55601 (14)	0.91194 (11)	0.0316 (4)
C1A	0.7602 (3)	1.0183 (2)	0.46772 (17)	0.0447 (6)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C2A	0.6945 (3)	1.0323 (2)	0.40016 (16)	0.0460 (6)
H2AC	0.707172	1.087641	0.358648	0.055*
C3A	0.6104 (3)	0.9639 (2)	0.39486 (15)	0.0431 (6)
H3AC	0.566273	0.972849	0.349382	0.052*
C4A	0.5906 (3)	0.88072 (19)	0.45724 (15)	0.0385 (5)
C5A	0.6574 (3)	0.86759 (19)	0.52585 (14)	0.0369 (5)
C6A	0.7432 (3)	0.9371 (2)	0.53007 (15)	0.0409 (6)
H6A	0.788777	0.928783	0.574837	0.049*
C7A	0.6336 (3)	0.78105 (19)	0.59150 (15)	0.0380 (5)
H7A	0.581489	0.733235	0.584011	0.046*
C8A	0.6939 (2)	0.66707 (17)	0.78704 (14)	0.0317 (5)
C9A	0.6667 (2)	0.47441 (17)	0.95583 (14)	0.0321 (5)
H9A	0.604119	0.443120	0.938191	0.039*
C10A	0.7162 (2)	0.42932 (17)	1.03222 (13)	0.0304 (4)
C11A	0.6661 (2)	0.34053 (17)	1.07574 (14)	0.0328 (5)
H11A	0.601869	0.313081	1.056275	0.039*
C12A	0.7117 (2)	0.29395 (18)	1.14721 (15)	0.0358 (5)
C13A	0.8109 (3)	0.3317 (2)	1.17592 (15)	0.0407 (5)
H13A	0.842800	0.298549	1.223452	0.049*
C14A	0.8613 (3)	0.4187 (2)	1.13319 (16)	0.0413 (6)
H14A	0.928559	0.443606	1.151652	0.050*
C15A	0.8129 (2)	0.46981 (18)	1.06268 (15)	0.0345 (5)
F1	0.47681 (19)	1.09688 (13)	0.07935 (12)	0.0594 (5)
F2	0.3957 (2)	1.05030 (16)	0.21052 (12)	0.0675 (5)
F3	0.56648 (18)	0.96438 (14)	0.14418 (14)	0.0675 (5)
O3	0.35856 (19)	0.86359 (14)	0.14786 (13)	0.0463 (4)
O4	0.28491 (19)	0.99482 (13)	0.06061 (11)	0.0420 (4)
C16	0.3551 (2)	0.95224 (18)	0.11146 (15)	0.0355 (5)
C17	0.4502 (3)	1.0172 (2)	0.13598 (17)	0.0427 (6)
F1A ^a	0.2742 (10)	0.4634 (6)	0.6658 (7)	0.091 (3)
F2A ^a	0.4168 (6)	0.3360 (8)	0.6865 (8)	0.096 (3)
F3A ^a	0.2720 (8)	0.3850 (6)	0.7859 (3)	0.074 (2)
O3A	0.49305 (19)	0.45855 (14)	0.80113 (11)	0.0421 (4)
O4A	0.4722 (2)	0.56097 (14)	0.68117 (11)	0.0443 (4)
C16A	0.4501 (3)	0.48571 (19)	0.73381 (15)	0.0375 (5)
C17A	0.3566 (3)	0.4142 (2)	0.71528 (17)	0.0430 (6)
O5	0.3568 (3)	0.83840 (18)	0.33704 (14)	0.0655 (6)
H5A	0.333023	0.779206	0.350328	0.098*
H5B	0.371613	0.847176	0.282908	0.098*
O6	0.4274 (5)	0.6164 (3)	0.5145 (2)	0.1205 (16)
H6B	0.420793	0.578805	0.561957	0.181*
H6C	0.457248	0.668912	0.523276	0.181*
O7	0.4485 (2)	0.29175 (15)	0.92910 (12)	0.0472 (4)
H7B	0.434924	0.339481	0.890105	0.071*
H7C	0.497284	0.245631	0.902656	0.071*
O8	0.22076 (16)	0.21592 (13)	1.02692 (10)	0.0364 (4)
H8A	0.288524	0.235935	0.991673	0.055*
H8B	0.242274	0.153155	1.039693	0.055*
F1B ^b	0.3559 (16)	0.4219 (8)	0.6330 (5)	0.082 (4)
F2B ^b	0.4121 (12)	0.3187 (7)	0.7278 (7)	0.076 (3)
F3B ^b	0.2436 (10)	0.4172 (12)	0.7605 (11)	0.105 (5)

^aOccupancy: 0.578 (16), ^bOccupancy: 0.422 (16).

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

Source of material

5-Chlorosalicylaldehyde (3.12 g, 0.02 mol) was placed in 15 mL ethanol at room temperature, added into *N,N'*-diaminoguanidine hydrochloride (1.25 g, 0.01 mol) solution containing 10 mL water and 8 mL ethanol, then dripped TFA (1.14 g), heated and stirred for 4 h, cooled to room temperature, then the precipitate was removed, and the filtrate was left standing to precipitate light yellow crystals.

Experimental details

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

Comment

Schiff bases, especially aromatic Schiff bases, are widely used as catalysts for acetaldehyde polymerization and oxidation of ascorbic acid and cysteine [4], as stabilizers for gasoline [5], as antiviral and antibacterial bioactive agents, and as modifiers for polymerization reaction [6]. In recent years, it has also obtained important applications in the field of materials [7]. At the same time, it is also an important ligand in organic synthesis [8]. The asymmetric unit of the crystal structure of the title compound is shown in the Figure. The cationic Schiff bases, the trifluoro acetate anions and the water molecules are linked by hydrogen bonds. The bond lengths and angles range in a regular distance [9, 10].

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

Research funding: Henan University of Science and Technology Distinguished Professor Open Fund (Grant No. 135100001).

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

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