Crystal structure of (*E*)-1-((1-(3,4-dimethoxyphenyl)ethyl)imino) guanidinium perchlorate, $C_{11}H_{17}ClN_4O_6$

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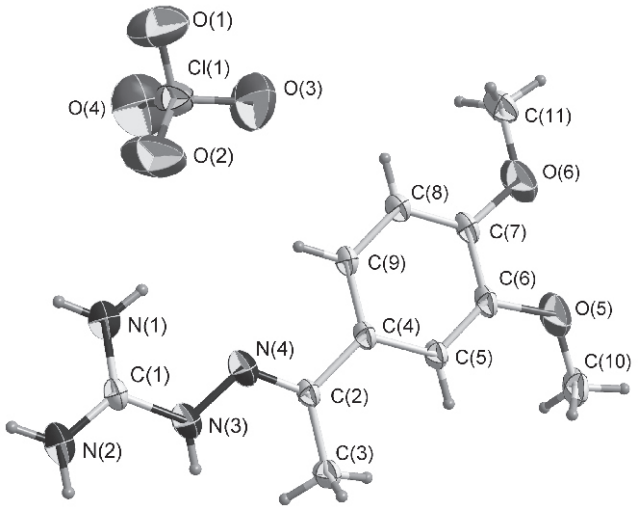

Abstract

$C_{11}H_{17}ClN_4O_6$, monoclinic, *C*2/*c* (no. 15), a = 17.870(6) Å, b = 7.885(2) Å, c = 23.105(8) Å, β = 108.139(4)°, V = 3093.9 Å^3, Z = 8, $R_{gt}(F)$ = 0.0614, $wR_{ref}(F^2)$ = 0.1961, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless rods, size 0.12×0.26×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.81 cm^{-1}
Diffractometer, scan mode:	Bruker Smart APEXII CCD, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11294, 3465
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2836
$N(param)_{refined}$:	202
Programs:	SHELX, PLATON, DIAMOND [7–9]

Source of material

All reagents and solvents obtained from commercial sources were used without further purification. Aminoguanidine hydrochloride (1.1 g, 10 mmol) was slowly added to a solution of 1-(3,4-dimethoxyphenyl)ethanone (1.8 g, 10 mmol) in 60 ml absolute ethanol. The reaction mixture was stirred for 10 hours under reflux, and then filtered after cooled down. The resulted white powder crystal was obtained as the product in yielding 88 %. After the filtrate was allowed to stand for three days, colourless, tabular crystals suitable for X-ray analysis were obtained. **IR** (KBr pellet, cm^{-1}): 3540vs, 3409vs, 3082b, 1624s, 1520m, 1426s, 1227m, 1114m, 877w, 851s, 781w, 743w, 719s, 655m, 522w, 505w, 433w. **Elemental analysis**: found - C, 55.89%; H, 6.85%; N, 23.73% (VarioEL); calcd. for $C_{11}H_{17}N_4O_6$ (336.74) - C, 55.92%; H, 6.83%; N, 23.71%.

Experimental details

All the H atoms were treated as riding-model, with N–H = 0.86 Å, C–H = 0.93-0.98 Å, and their U_{iso} = 1.2U_{eq}(carrier atom).

Discussion

The aminoguanidine derivatives with aldehyde or keto group have been employed to prepare metal complexes for its biological activities in the research area of bioinorganic chemistry [1–4]. The title compound is a new aminoguanidine derivative and has been synthesized in a convenient approach in our work. It was expected to be used as a ligand to obtain vanadium complexes, which have the potential insulin-mimetic effect [5, 6]. The title compound is a perchlorate salt, the structure of the cation moiety will be discussed in this work. The 1-(3,4-dimethoxy)phenyl-1-methane imino guanidine molecule was protonated to obtain the title salt. The bond length of C2–N4 is 1.291(3) Å, showing a double bond formed by the condensation of the ketone and amino groups. However, the bond distance of C1–N1 (1.309(3) Å) and C1–N2 (1.315(3) Å) are not significantly different, which may be considered that the two bonds were delocalized. The cation moieties were linked each to other form an infinite chain by the N–H···O hydrogen bonds by the amino and methoxyl groups. The distances and angles of bond N2–H2A···O5 and N2–H2A···O6 were 2.975(3) Å, 2.989(3) Å and 107.5°, 130.0°, respectively. In the chain, the neighbouring cations were not coplanar, their intersection angle was 51°. Sequentially, perchlorate anions are connected to the infinite chains constructing a two-dimensional layer by the N–H···O hydrogen bonds through the guandine groups and perchlorate anions. The distances and angles of bond N1–H1B···O2, N2–H2B···O1, and N3–H3···O1 were 2.991(3) Å, 2.906(4) Å, 3.034(3) Å and 130.4°, 150.6°, 140.1°, respectively. Further, the weaker hydrogen bonds between the N1, N2 atoms of the guandine groups and O2, O1 atoms of the perchlorate anions enlarge the two-dimentional network into a three-dimentional network. The distances and angles of bond N1–H1A···O2 and N2–H2A···O1 were 3.105(4) Å, 3.161(4) Å and 165.5°, 144.5°, respectively.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3C)	8*f*	0.6187	0.8220	–0.0272	0.082
H(3B)	8*f*	0.6771	0.7848	0.0378	0.082
H(3A)	8*f*	0.6328	0.9590	0.0242	0.082
H(5A)	8*f*	0.5070	0.8497	–0.0833	0.056
H(8A)	8*f*	0.3082	0.6076	–0.0218	0.062
H(9A)	8*f*	0.4322	0.6397	0.0469	0.059
H(10C)	8*f*	0.4697	0.8177	–0.1908	0.123
H(10B)	8*f*	0.4538	0.9994	–0.1697	0.123
H(10A)	8*f*	0.4073	0.9360	–0.2353	0.123

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11C)	8*f*	0.2151	0.5036	–0.1057	0.129
H(11B)	8*f*	0.1531	0.6088	–0.1558	0.129
H(11A)	8*f*	0.1884	0.6822	–0.0898	0.129
H(1B)	8*f*	0.5454	0.5866	0.1691	0.084
H(1A)	8*f*	0.5924	0.5622	0.2343	0.084
H(2B)	8*f*	0.7546	0.7454	0.2242	0.082
H(2A)	8*f*	0.7213	0.6600	0.2682	0.082
H(3)	8*f*	0.6814	0.7784	0.1244	0.060

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.6488(1)	0.6741(4)	0.1885(1)	0.046(1)	0.067(2)	0.034(1)	–0.002(1)	0.0000(9)	0.0033(9)
C(2)	8*f*	0.5628(1)	0.7668(3)	0.03442(9)	0.039(1)	0.048(1)	0.0298(9)	0.0008(8)	0.0037(8)	–0.0025(8)
C(3)	8*f*	0.6287(1)	0.8396(4)	0.0157(1)	0.045(1)	0.080(2)	0.037(1)	–0.006(1)	0.0102(9)	–0.001(1)
C(4)	8*f*	0.4831(1)	0.7475(3)	–0.01038(8)	0.039(1)	0.050(1)	0.0280(9)	0.0031(8)	0.0020(8)	–0.0005(8)
C(5)	8*f*	0.4671(1)	0.8014(3)	–0.07073(9)	0.042(1)	0.067(2)	0.0292(9)	0.004(1)	0.0058(8)	0.0029(9)
C(6)	8*f*	0.3924(1)	0.7834(3)	–0.11191(9)	0.048(1)	0.068(2)	0.0269(9)	0.011(1)	0.0016(8)	0.0005(9)
C(7)	8*f*	0.3323(1)	0.7088(3)	–0.0938(1)	0.042(1)	0.058(1)	0.036(1)	0.003(1)	–0.0039(9)	–0.0046(9)
C(8)	8*f*	0.3480(1)	0.6564(3)	–0.0344(1)	0.043(1)	0.064(2)	0.042(1)	–0.007(1)	0.0029(9)	0.005(1)
C(9)	8*f*	0.4226(1)	0.6757(3)	0.0069(1)	0.044(1)	0.064(2)	0.032(1)	–0.003(1)	0.0019(8)	0.0069(9)
C(10)	8*f*	0.4302(2)	0.9026(5)	–0.1936(1)	0.073(2)	0.134(3)	0.039(1)	0.023(2)	0.017(1)	0.027(2)
C(11)	8*f*	0.1994(2)	0.6156(5)	–0.1210(2)	0.049(2)	0.107(3)	0.080(2)	–0.020(2)	–0.012(1)	0.006(2)
N(1)	8*f*	0.5887(1)	0.5991(4)	0.1984(1)	0.049(1)	0.107(2)	0.044(1)	–0.016(1)	0.0001(9)	0.024(1)
N(2)	8*f*	0.7160(1)	0.6956(4)	0.23199(9)	0.052(1)	0.105(2)	0.0334(9)	–0.016(1)	–0.0066(9)	0.011(1)
N(3)	8*f*	0.6422(1)	0.7327(3)	0.13262(8)	0.0369(9)	0.075(1)	0.0299(8)	–0.0086(8)	–0.0016(7)	0.0034(8)
N(4)	8*f*	0.5693(1)	0.7165(3)	0.08895(8)	0.0362(9)	0.064(1)	0.0305(8)	–0.0052(8)	–0.0019(7)	0.0010(8)
O(1)	8*f*	0.2981(2)	0.4205(4)	0.1724(1)	0.088(2)	0.121(2)	0.102(2)	–0.019(1)	0.062(2)	–0.017(2)
O(2)	8*f*	0.4172(2)	0.5230(4)	0.1712(1)	0.073(2)	0.149(3)	0.104(2)	–0.057(2)	0.026(1)	–0.044(2)
O(3)	8*f*	0.3217(2)	0.4565(5)	0.0817(1)	0.119(2)	0.157(3)	0.053(1)	–0.029(2)	0.010(1)	0.001(2)
O(4)	8*f*	0.3843(2)	0.2393(4)	0.1448(2)	0.134(3)	0.095(2)	0.130(3)	0.023(2)	0.040(2)	–0.011(2)
O(5)	8*f*	0.3708(1)	0.8354(3)	–0.17136(7)	0.054(1)	0.124(2)	0.0292(8)	0.009(1)	0.0003(7)	0.0170(9)
O(6)	8*f*	0.2612(1)	0.6928(3)	–0.13805(8)	0.0468(9)	0.094(2)	0.0431(9)	–0.0023(9)	–0.0105(7)	–0.0028(9)
Cl(1)	8*f*	0.35726(3)	0.40765(8)	0.14355(2)	0.0442(3)	0.0687(5)	0.0423(3)	–0.0118(2)	0.0156(2)	–0.0089(2)

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References

1. He, C.; Lippard, S. J.: Aminoguanidinium Hydrolysis Effected by a Hydroxo-Bridged Dicobalt(II) Complex as a Functional Model for Arginase and Catalyzed by Mononuclear Cobalt(II) Complexes. J. Am. Chem. Soc. **120** (1998) 105–113.
2. Bambagioni, V.; Bani, D.; Bencini, A.; Biver, T.; Cantore, M.; Chelli, R.; Cinci, L.; Failli, P.; Ghezzi, L.; Giorgi, C.; Nappini, S.; Secco, F.; Tine, M. R.; Valtancoli, B.; Venturini, M.: Polyamine-Polycarboxylate Metal Complexes with Different Biological Effectiveness as Nitric Oxide Scavengers. Clues for Drug Design. J. Med. Chem. **51** (2008) 3250–3260.
3. Leovac, V. M.; Joksovic, M. D.; Divjakovic, V.; Jovanovic, L. S.; Saranovic, Z.; Pevec, A.: Synthesis, spectroscopic and X-ray characterization of a copper(II) complex with the Schiff base derived from pyridoxal and aminoguanidine: NMR spectral studies of the ligand. J. Inorg. Biochem. **101** (2007) 1094–1097.
4. Lalovic, M. M.; Vojinovic-Jesic, L. S.; Jovanovic, L. S.; Leovac, V. M.; Cesljevic, V. I.; Divjakovic, V.: Synthesis, characterization and crystal structure of square-pyramidal copper(II) complexes with pyridoxylidene aminoguanidine. Inorg. Chim. Acta **388** (2012) 157–162.
5. Thompson, K. H.; Orvig, C.: Coordination chemistry of vanadium in metallopharmaceutical candidate compounds. Coordin. Chem. Rev. **219–221** (2001) 1033–1053.
6. Thompson, K. H.; McNeil, J. H.; Orvig, C.: Vanadium Compounds as Insulin Mimics. Chem. Rev. **99** (1999) 2561–2671.
7. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
8. Spek, A. L.: Structure validation in chemical crystallography. Acta Crystallogr. **D65** (2009) 148–155.
9. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany 2012.