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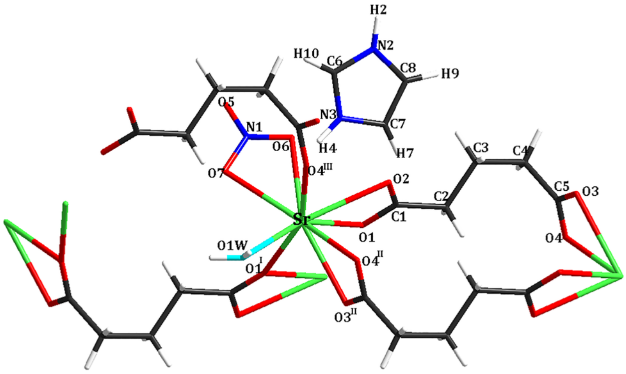
Rawiya Dridi, Amira Bouhali*, Chaouki Boudaren, Abdelmalek Bouraiou and Hocine Merazig

Crystal structure of 1*H*-imidazol-3-ium poly[aqua-(μ$_4$-glutarato-κ^6*O,O′:O′:O″,O‴:O‴*)-(nitrato-κ^2*O,O′*)strontium(II)], $C_8H_{13}N_3O_8Sr$



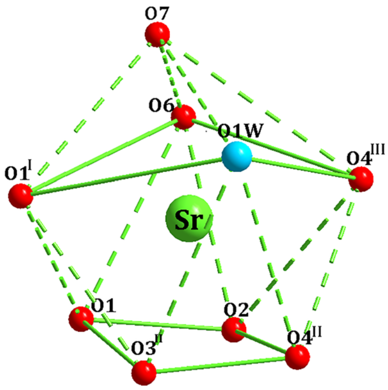


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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.12 \times 0.10 \times 0.08$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	4.19 mm^{-1}
Diffractometer, scan mode:	Bruker APEXII
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,520, 2983, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2823
$N(param)_{refined}$:	189
Programs:	Bruker [1], SHELX [2], WinGX/ ORTEP [3], Diamond [4]

Abstract

$C_8H_{13}N_3O_8Sr$, monoclinic, $C2/c$ (no. 15), $a = 8.0945(2)$ Å, $b = 15.7091(5)$ Å, $c = 20.6169(6)$ Å, $\beta = 96.807(2)°$, $V = 2603.11(13)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.021$, $wR_{ref}(F^2) = 0.0494$, $T = 273(2)$ K.

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A part of 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

Glutaric acid (0.13 g, 1 mmol) was added to NaOH (0.04 g, 1 mmol) dissolved in 5 ml distilled water. Strontium nitrate (0.26 g, 1 mmol) was mixed with the stirred aqueous solution and finally 0.06 g of imidazole (1 mmol) was added to the incolor solution by continuous stirring at room temperature overnight. After a few days, several colorless block crystals were obtained and filtered, washed with cold distilled water and dried in a vacuum desiccator containing calcium chloride to a constant weight (yield 33.6%).

Experimental details

All H atoms were placed in idealized positions using the standard riding models of the SHELX System [2]. The

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***Corresponding author: Amira Bouhali**, Unité de recherche de Chimie de l'Environnement et Moléculaire Structurale, CHEMS, Université des frére Mentouri Constantine 1, 25000, Constantine, Algeria, E-mail: bouhali_amira@yahoo.fr. https://orcid.org/0000-0003-1301-5431
Rawiya Dridi, Chaouki Boudaren, Abdelmalek Bouraiou and Hocine Merazig, Unité de recherche de Chimie de l'Environnement et Moléculaire Structurale, CHEMS, Université des frére Mentouri Constantine 1, 25000, Constantine, Algeria, E-mail: dridirawiya@gmail.com (R. Dridi), cboudaren@yahoo.com (C. Boudaren), bouraiou.abdelmalek@yahoo.fr (A. Bouraiou), hmerazig@gmail.com (H. Merazig)

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

Atom	x	y	z	$U_{iso}*/U_{eq}$
C1	0.1018 (2)	0.38255 (10)	0.43721 (8)	0.0151 (3)
C2	0.1449 (2)	0.47344 (12)	0.42628 (10)	0.0221 (4)
H21	0.2621	0.4769	0.4209	0.027*
H22	0.1275	0.506	0.4649	0.027*
N2	0.1885 (2)	0.32133 (14)	0.20577 (9)	0.0346 (4)
H2	0.1363	0.3216	0.1669	0.042*
C3	0.0431 (2)	0.51473 (11)	0.36632 (10)	0.0230 (4)
H31	−0.0721	0.5196	0.3745	0.028*
H32	0.0466	0.478	0.3287	0.028*
C4	0.1091 (2)	0.60442 (12)	0.35033 (9)	0.0223 (4)
H41	0.2271	0.6006	0.3462	0.027*
H42	0.0527	0.6234	0.3087	0.027*
N3	0.2905 (2)	0.27889 (12)	0.30059 (9)	0.0287 (4)
H4	0.3156	0.2466	0.334	0.034*
C5	0.0827 (2)	0.66847 (10)	0.40181 (8)	0.0147 (3)
C7	0.3358 (3)	0.36130 (16)	0.29630 (12)	0.0368 (5)
H7	0.3989	0.393	0.3283	0.044*
N1	0.06789 (19)	0.10379 (10)	0.37074 (8)	0.0245 (3)
C8	0.2711 (3)	0.38874 (17)	0.23611 (13)	0.0419 (6)
H9	0.2811	0.443	0.2189	0.05*
C6	0.2020 (3)	0.25568 (15)	0.24578 (11)	0.0316 (4)
H10	0.1566	0.202	0.2368	0.038*
O1	0.21403 (14)	0.33205 (8)	0.45999 (7)	0.0199 (3)
O2	−0.04789 (14)	0.35761 (8)	0.42307 (7)	0.0202 (3)
O1W	−0.11563 (17)	0.10539 (9)	0.56795 (7)	0.0215 (3)
O3	−0.06482 (15)	0.69278 (8)	0.40604 (7)	0.0213 (3)
O4	0.20262 (15)	0.69467 (8)	0.44076 (7)	0.0216 (3)
O5	0.1037 (3)	0.06317 (12)	0.32359 (9)	0.0540 (5)
O6	0.03955 (16)	0.18226 (8)	0.36632 (7)	0.0242 (3)
O7	0.05833 (18)	0.06854 (8)	0.42545 (7)	0.0278 (3)
Sr1	0.00025 (2)	0.21191 (2)	0.49004 (2)	0.01294 (6)
H1W	−0.094 (3)	0.0520 (18)	0.5708 (13)	0.037 (7)*
H2W	−0.215 (4)	0.1113 (19)	0.5702 (15)	0.053 (9)*

position and U_{iso} of H atoms were freely refined. Reflections, whose intensities were obstructed or shadowed by the beamstop, were omitted during refinement.

Comment

Coordination polymers and in particular metal–organic frameworks (MOFs) [5, 6] are a relatively new classes of compounds showing promising properties for the chemical industry [7, 8]. Synthesis and study of new salts of dicarboxylic acids with metals reveal new types of structures, which are interesting from the viewpoint of crystal engineering and practical applications [9–13]. The glutarate anion contains four potential donor atoms, coming from two carboxylate groups. For this anion, the aliphatic carbon backbone may show in three conformations: anti–anti, anti–*gauche* and *gauche–gauche*.

The asymmetric unit of the title compound contains one Sr^{2+} ion, one coordinated nitrate anion, a $(C_5H_6O_4)^{2-}$ anion, one aqua ligand and one protonated imidazole molecule. The crystal structure is linked into a polymeric chain running parallel to the (010) by a bridging glutarate ligand (μ_4, $-k^6$, η^1:η^2:η^1, η^2). Pairs of these bridging adjacent strontium atoms form $[Sr_2(glu)_2]$ dimeric units with a Sr···Sr distance of 9.0606 (3) Å. The layers are stacked into a three-dimensional structure and linked *via* a weak π–π interaction between imidazole rings in neighboring layers. The strontium atom is coordinated by the O atoms from four glutarate anions involved in a mono- and bidentate mode, one bidentate nitrate anion and a water molecule. The nine-fold coordination polyhedron of Sr can be described as a distorted monocapped square antiprism where the cap position is occupied by O7, which is separated from Sr by 2.6881 Å and from the first square face by 1.7368 Å. First, the mean standard deviation from planarity of the face capped by O7 [O1^I, O4^{III}, O6, O1w] is 0.3394 Å *versus* 0.1024 Å for the second face [O1, O2, O3^{II}, O4^{II}] (for symmetry operation (I) $1/2-x$, $1/2-y$, $1-z$ (II) $-x$, $1-y$, $1-z$ and (III) $-1/2+x$, $-1/2+y$, z). The deformation of two squares is also perceptible from the angles around the oxygen atoms. In particular, the comparison between the two sets of torsion angles confirms a twist more marked for the square face capped by O7 (±22 against ±9.0° for the other plane). Also, the distances from the central Sr atom to the center of each square face differ from each other [0.8004 Å against 1.8271 Å for the second plane]. The dihedral angle between the two planes is equal to 3.536°. Nevertheless, such a distortion into the $SrO_7(H_2O)$ antiprism can be related with the constraints induced by both the capping position and the two opposite pyramidal O7–O6 and O7–O1w edges [2.1607 and 3.4624 Å, respectively]. The Sr–O distances fall in the range 2.5142(1)–2.7258(1) Å (av. = 2.628 Å), in good accordance with the value calculated with the bond valence program VALENCE [13, 14] for ninefold-coordinated Sr^{2+} cation, *i.e.*, 2.672 Å. The bond valences are close to 0.340, 0.214, 0.218, 0.259 and 0.336 valence units (v.u.) for Sr–O1, Sr– O1^I, Sr–O2, Sr–O3^{II} and Sr–O4^{III}, respectively. The bond valences are 0.192, 0.234, 0.212 and 0.289 v.u. for Sr–O4^{II}, Sr–O6, Sr–O7 and Sr–O1w, respectively. Then, the sum of the bond valences around the strontium atom, *i.e.*, 2.2 v.u. must be compared with the +2 oxidation state of Sr. The glutarate ligands adopt the *anti-gauche* conformation, illustrated by the C1–C2–C3–C4 [171.408(2)°] and C2–C3–C4–C5 [67.666(2)°] torsion angles. The dihedral angle between the C1/O1/O2 and C5/O3/O4 carboxylate group at each end of the ion is 62.281°. The carboxylate group binds four metal cations through bidentate and monodentate chelation. The three C_{sp3}–C_{sp3}–C_{sp3} angles of the glutarate anion C1–C2–C3, C2–C3–C4 and C3–C4–C5 are 114.144(2)°, 112.823(2)° and 112.189(1)° but they are considerably greater than the tetrahedral angle 109.28° (mainly due to the presence of intermolecular hydrogen bonding).

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

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