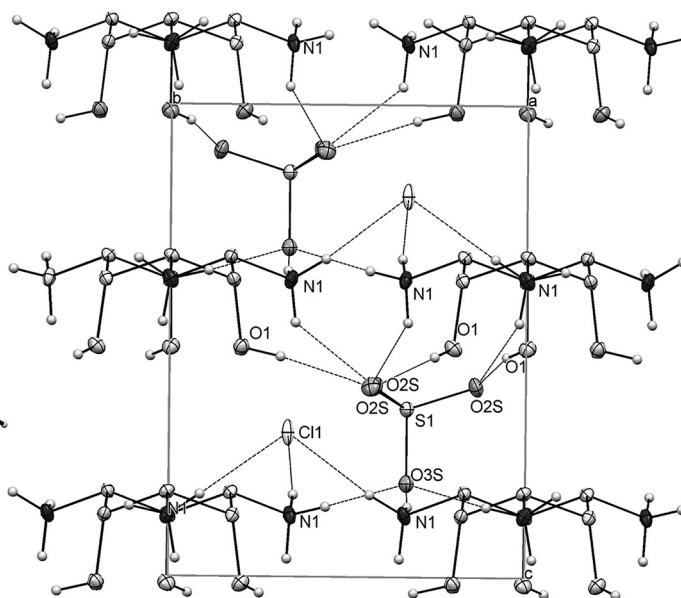
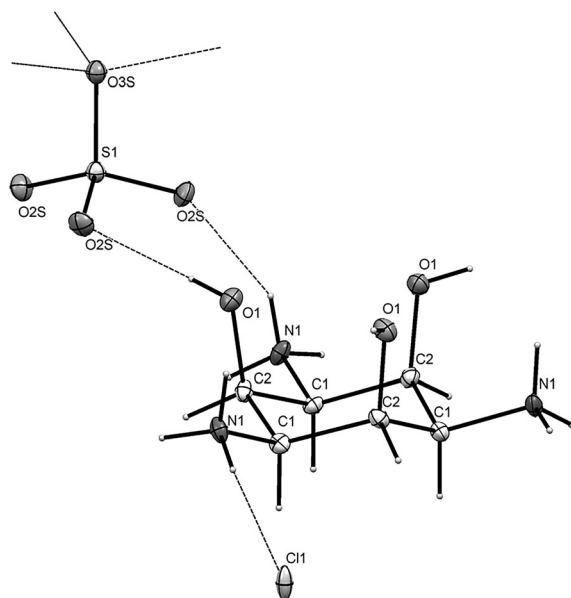


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Crystal structure of all-*cis*-2,4,6-trihydroxycyclohexane-1,3,5-triaminium chloride sulfate, $C_6H_{18}ClN_3O_7S$



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Abstract

$C_6H_{18}ClN_3O_7S$, trigonal, $P31c$ (no. 159), $a = 8.3990(14)$ Å, $b = 8.3990(14)$ Å, $c = 9.6208(17)$ Å, $V = 587.76(17)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1404$, $T = 300.15$ K.

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The molecular structure of the title compound and 3D packing along a -axis are presented in the Figure. Table 1 contains crystallographic data and Table 2 contains the list

Table 1: Data collection and handling.

Crystal:	Colourless prism
size:	0.45 × 0.07 × 0.05 mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	0.54 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	1923, 753, 0.046
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 648
$N(param)_{refined}$:	57
Programs:	Bruker [1, 2], SHELX [3, 4], Olex2 [5], Mercury [6]

of atoms, including atomic coordinates and displacement parameters.

Source of material

Single crystals of the title compound were obtained during the interaction of 2,4,6-triaminocyclohexane-1,3,5-triol·1.5H₂SO₄ [7] (0.1 g, 0.32 mmol) and K₂PtI₆

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²). The sign (*) distinguishes the isotropic parameters (U_{iso}*) from equivalent isotropic parameters (U_{eq})

Atom	X	Y	Z	U _{iso} /U _{eq}
Cl1	0.666667	0.333333	0.1949 (5)	0.0319 (11)
S1	0.666667	1.333333	0.6444 (3)	0.0215 (8)
O1	0.7954 (8)	0.9966 (7)	0.5153 (7)	0.0272 (14)
H1	0.743824	1.054749	0.534496	0.041*
O2S	0.5753 (8)	1.1402 (6)	0.5971 (7)	0.0306 (13)
O3S	0.666667	1.333333	0.8028 (10)	0.026 (2)
N1	0.6599 (8)	0.6608 (8)	0.3708 (7)	0.0237 (15)
H1A	0.651569	0.563658	0.326896	0.028*
H1B	0.560576	0.669403	0.352483	0.028*
H1C	0.667730	0.648046	0.461927	0.028*
C2	0.8202 (10)	0.9990 (11)	0.3714 (10)	0.0207 (15)
H2	0.716462	1.000265	0.325758	0.025*
C1	0.8266 (11)	0.8302 (11)	0.3223 (8)	0.0206 (16)
H1D	0.825454	0.829876	0.220471	0.025*

(molar ratio: Pt:taci = 1) at pH 3 in the presence of diluted HCl (0.002 mol/L) and heating at $T = 60$ °C for 3 h. Colorless crystals were separated from the reaction system a few hours later.

Experimental details

All hydrogen atoms are placed on their calculated positions and constrained to ride on their parent atoms. The absolute structure was established by anomalous dispersion [8], Flack parameter is -0.08(11).

Comment

The title compound is the triprotonated form of the ligand all-*cis*-2,4,6-triaminocyclohexane-1,3,5-triol, which is known by a trivial name taci. The remarkable coordination ability of taci to various metal cations and the structure of most of its complexes, some of which with a proven bioactivity profile, have been studied in detail [9]. The determination of the crystal structure of the neutral compound as taci·2H₂O [10] and of those compounds in which taci participates as protonated cations [11–13] has been previously reported. In this study, the crystal structure of the triprotonated taci balanced with sulfate and chloride counter anions is presented.

The asymmetric unit of the structure contains 1/3 of [H₃taci]³⁺ and 1/3 of each counter ion. The [H₃taci]³⁺ cations adopt a chair conformation with three hydroxy groups (O–C - 1.398 Å) in axial and three azaniumyl groups (N–C - 1.486 Å) in equatorial position (left part of the

figure). The sulfate ions possess typical tetrahedral geometry as the S and O3S atoms occupy a special position on a 3-fold axis while O2S-atom resides in a general position, forming three crystallographically equivalent bonds of all four sulfur oxygen bonds in the anion. The 3D packing of the title compound is constructed of alternating planes parallel to the *ab*-plane of the crystal cell where the [H₃taci]³⁺ cations are disposed. The distance between adjacent planes is 4.810 Å. The sulfate and chloride anions are located between the planes. In detail, the sulfate anions are located almost in the middle of a channel, forming nine hydrogen bonds with surrounding [H₃taci]³⁺ cations (right part of the figure): 3 with hydroxy groups (O··O2S - 2.779(9) Å) and 3 with azaniumyl groups (N··O2S_1 - 2.780(9) Å, 1 = -x + y, -x + 1, z) from the upper plane; and 3 H-bonds with azaniumyl groups from the bottom plane (N··O3S_2 - 2.844(6) Å; 2 = +y - 1, x, z - 0.5). The Cl⁻ anions are located closer to the plane here noted as bottom, forming hydrogen bonds only with azaniumyl groups (N··Cl1 = 3.254(7) Å). To our point of view, the higher symmetry of the structure is shaped by the H-bonding network.

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References

1. Bruker. APEX2 and SAINT; Bruker AXS Inc.: Madison, Wisconsin, USA, 2013.
2. Bruker. SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2014.
3. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
6. Macrae C. F., Bruno I. J., Chisholm J. A., Edgington P. R., McCabe P., Pidcock E., Rodriguez-Monge L., Taylor R., van de Streek J., Wood P. A. Mercury CSD 2.0 – new features for the visualization and investigation of crystal structures. *J. Appl. Crystallogr.* 2008, *41*, 466–470.
7. Hegetschweiler K., Erni I., Schneider W., Schmalte H. 9. Preparation, characterisation, and structure of N-methylated

- derivatives of 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol: polyalcohols with unusual acidity. *Helv. Chim. Acta* 1990, 73, 97–105.
8. Parsons S., Flack H. D., Wagner T. Use of intensity quotients and differences in absolute structure refinement. *Acta Crystallogr* 2013, B69, 249–259.
 9. Hegetschweiler K. Polyalcohols and polyaminopolyalcohols as selective receptors for metal ions. *Bol. Soc. Chil. Quim.* 1997, 42, 257–279.
 10. Hegetschweiler K., Robert J., Hancock D., Chisletta M., Kradolfer T., Gramlich V., Schmalte H. W. 5. 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol, a ligand with remarkable versatility for metal ions. complex formation with magnesium(II), calcium(II), strontium(II), barium (II) and cadmium(II). *Inorg. Chem.* 1993, 32, 5273–5284.
 11. Neis C., Hegetschweiler K. Hydrogen bonding in polyaminopolyalcohols: a monohydrate cocrystal of 1,3-diamino-5-azaniumyl-1,3,5-trideoxy-*cis*-inositol iodide and 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol. *Acta Crystallogr.* 2014, C70, 369–399.
 12. Gencheva G., Bontchev P. R., Sandler J., Hegetschweiler K. Crystal structure of tetrakis (1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol) decachloride hexachloroplatinate hexahydrate, $(H_3taci)_4[PtCl_6]Cl_{10} \cdot 6H_2O$, two different forms of $(H_3taci)^{3+}$ in one crystal. *Z. Kristallogr. N. Cryst. Struct.* 2000, 215, 183–185.
 13. Ghisletta M., Jalett H., Gerfin T., Gramlich V., Hegetschweiler K. 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol, a new ligand with a remarkable versatility for metal ions. safe and efficient ligand preparation and structure of the free ligand and the Co^{III} complex. *Helv. Chim. Acta* 1992, 75, 2233–2242.