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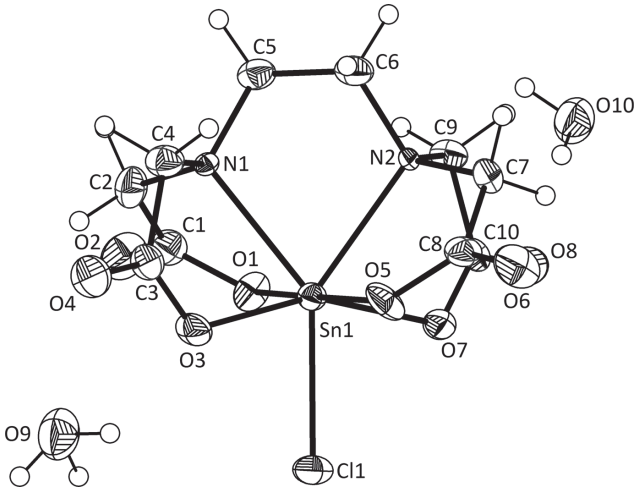
The crystal structure of oxonium chlorido-ethylenediaminetetraactetotin(IV) hydrate, $C_{10}H_{17}ClN_2O_{10}Sn$



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Abstract

$C_{10}H_{17}ClN_2O_{10}Sn$, triclinic, $P\bar{1}$ (no. 1), $a = 6.6666(3)$ Å, $b = 6.6892(4)$ Å, $c = 8.8779(6)$ Å, $\alpha = 101.340(5)°$, $\beta = 99.624(5)°$, $\gamma = 91.279(4)°$, $V = 382.08(4)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0318$, $wR_{ref}(F^2) = 0.0726$, $T = 294$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Plate, clear light colourless
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.91 mm^{-1}
Diffractometer, scan mode:	*CrysAlis* PRO, φ and ω-scans
$2\theta_{max}$, completeness:	29.2°, 88% (>99% up to θ_{max} 25.2°)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5137, 3181, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3180
$N(param)_{refined}$:	232
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3], SIR2004 [4], PLATON [5]

Source of material

Ethylenediaminetetraacetic acid (EDTA, 0.277 g, 0.95 mmol) was added to an aqueous solution (10 mL) of potassium perrhenate (2.60 mg, 0.09 mmol) under stirring. Then an aqueous solution of $SnCl_2$ (170 mg, 0.90 mmol) dissolved in 1.5 mL concentrated hydrochloric acid was added dropwise, and the pH was adjusted to 3.0 by adding ammonium hydroxide (about 0.5 mL) with stirring at room temperature (RT) for 20 h. After filtering and adding excess ethanol into the brown-yellow filtrate the solution was kept at RT for about 1 week and colorless crystals were obtained. The crystals were washed with ethanol.

Experimental details

H atoms bonded to O atoms were located in a difference electron density map and refined with distance restraints of O—H = 0.85 Å, and with $U_{iso}(H) = 1.5\,U_{eq}(O)$. Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 Å and with $U_{iso}(H) = 1.2\,U_{eq}(C)$. The modified Flack parameter was refined to −0.02(2) based on 1368 quotients.

Discussion

Ethylenediaminetetraacetic acid contains four carboxylic acid groups and two amine groups with lone-pair electrons that chelate metal ions. EDTA has value in the application of

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.90742(2)	0.00015(2)	0.11347(2)	0.01672(12)
Cl1	0.6437(3)	−0.2518(3)	0.1138(2)	0.0310(4)
O1	0.7394(12)	0.2261(12)	0.2136(10)	0.0239(18)
O2	0.7114(9)	0.4607(9)	0.4221(8)	0.0392(14)
O3	1.0443(8)	−0.1335(8)	0.3034(6)	0.0254(11)
O4	1.3224(10)	−0.1655(10)	0.4671(8)	0.0364(15)
O5	1.0620(14)	−0.2233(12)	−0.0078(11)	0.029(2)
O6	1.1797(10)	−0.3412(9)	−0.2226(7)	0.0392(14)
O7	0.6904(9)	0.0262(8)	−0.0913(6)	0.0232(11)
O8	0.5633(9)	0.2497(9)	−0.2306(7)	0.0312(14)
O9	0.7593(11)	−0.0538(12)	0.5253(9)	0.0458(16)
H9C	0.613(16)	−0.053(13)	0.494(11)	0.03(2)*
H9D	0.74(2)	−0.13(3)	0.589(15)	0.21(11)*
H9E	0.790(19)	−0.123(17)	0.442(9)	0.07(4)*
O10	0.5617(10)	0.6386(9)	−0.3069(7)	0.0405(15)
H10A	0.6076	0.7306	−0.2271	0.061*
H10B	0.5889	0.5220	−0.2871	0.061*
N1	1.1478(8)	0.2400(8)	0.2829(7)	0.0186(11)
N2	1.0677(8)	0.1690(8)	−0.0452(6)	0.0176(11)
C1	0.8158(12)	0.3488(11)	0.3485(9)	0.0268(16)
C2	1.0391(12)	0.3388(11)	0.4064(9)	0.0284(16)
H2A	1.0986	0.4762	0.4488	0.034*
H2B	1.0570	0.2635	0.4902	0.034*
C3	1.2237(12)	−0.0727(11)	0.3773(9)	0.0233(15)
C4	1.3129(11)	0.1221(11)	0.3485(10)	0.0253(15)
H4A	1.3870	0.2024	0.4457	0.030*
H4B	1.4075	0.0907	0.2763	0.030*
C5	1.2202(12)	0.3919(10)	0.2015(9)	0.0250(15)
H5A	1.3462	0.4609	0.2619	0.030*
H5B	1.1199	0.4935	0.1917	0.030*
C6	1.2551(11)	0.2897(11)	0.0425(9)	0.0250(15)
H6A	1.2909	0.3919	−0.0138	0.030*
H6B	1.3672	0.2005	0.0524	0.030*
C7	1.1156(12)	0.0092(11)	−0.1742(8)	0.0230(14)
H7A	1.2461	0.0463	−0.1988	0.028*
H7B	1.0130	0.0045	−0.2664	0.028*
C8	1.1236(11)	−0.2005(11)	−0.1347(9)	0.0241(15)
C9	0.9111(11)	0.3009(10)	−0.1076(9)	0.0233(15)
H9A	0.9467	0.3396	−0.2000	0.028*
H9B	0.9069	0.4245	−0.0302	0.028*
C10	0.7048(11)	0.1877(11)	−0.1482(9)	0.0203(14)

drug carrier owing to its good chelating ability [6–8]. ^{99m}Tc-EDTA is widely used for radiodiagnostics and radiotherapy in oncology and nuclear medicine [9–11], which was prepared by reducing pertechnetate-99m with an excess of $SnCl_2$ in aqueous EDTA solutions. Thus, a large amount of tin-EDTA compounds can also be present in the reaction. We would like to investigate the morphology and structure of tin-EDTA compounds. For this purpose, one step in this direction is the reaction of perrhenate with an excess of $SnCl_2$ in aqueous EDTA solutions, using Re as an analogue for Tc.

The Sn metal center is coordinated to seven atoms, which comes from four oxygen atoms and two nitrogen atoms of EDTA as well as one chlorido ligand, forming a distorted capped trigonal prism geometry. The bond lengths Sn—O are 2.165 Å, 2.052 Å, 2.142 Å and 2.064 Å, respectively. And the Sn—N bond lengths are 2.353 Å and 2.340 Å, respectively. The bond length between Sn and Cl (2.4087 Å) is slightly longer than in tin(IV) chloride (2.280 Å). The angles of N1—Sn—Cl1 (144.39°) and N1—Sn—Cl1 (141.83°) are in accordance with those in a similar Sn complex with the EDTA ligand [12]. Intermolecular hydrogen bonds both exist in the title structure.

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