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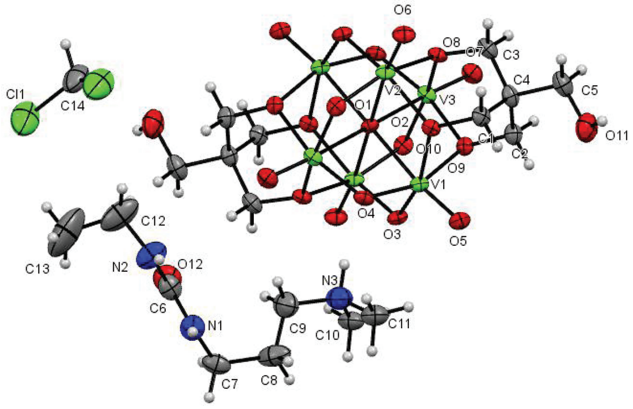
# Crystal structure of bis(3-(3-ethylureido)-*N*,*N*-dimethylpropan-1-aminium) bis ($\mu_3$-2-(hydroxymethyl)-2-(oxidomethyl)propane-1,3-bis(olato))-($\mu_6$-oxo)-hexakis($\mu_2$-oxo)-hexaoxo-hexavanadium(V) – dichloromethane (1/1), $C_{27}H_{60}Cl_2N_6O_{23}V_6$

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## Abstract

$C_{27}H_{60}Cl_2N_6O_{23}V_6$, $C2/c$ (no. 15), $a = 26.1482(12)$ Å, $b = 10.6096(4)$ Å, $c = 17.4426(6)$ Å, $\beta = 90.932(4)°$, $V = 4838.3(3)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0565$, $wR_{ref}(F^2) = 0.1477$, $T = 106$ K.

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A part 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.

## Source of materials

The title compound was synthesized by dissolving 0.632 g (0.5 mmol) $(Bu_4N)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]$, which was prepared according to the literature [1], into 30 mL acetonitrile. Then 0.192 g (1 mmol) 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI) and 0.135 g (1 mmol) 1-hydroxybenzotriazole (Hobt) was added to the solution. The mixture was heated at 50 °C for 48 h. Precipitations appear on slow evaporation after heating and precipitates were collected by filtration. The crystals of the title compound were obtained by recrystalization from dichloromethane as red platelet crystals. IR spectrum (cm$^{-1}$, KBr pellet): 1650 (vs), 1520 (s), 1252 (w), 1060 (w), 957 (vs), 789 (m), 702 (m).

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Red platelet |
| Size: | 0.15 × 0.12 × 0.03 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.30 mm$^{-1}$ |
| Diffractometer, scan mode: | Xcalibur, Eos, Gemini, $\omega$ |
| $\theta_{max}$, completeness: | 26.0°, 99.71% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 10699, 4756, 0.037 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3735 |
| $N(param)_{refined}$: | 295 |
| Programs: | Olex2 [6], CryAlis$^{PRO}$ [7] |

## Experimental details

The hydrogen atoms were generated geometrically and refined using the riding model.

## Comment

Polyoxovanadates (POVs) have attracted much attention because of their excellent catalytic, magnetic and biochemical properties [2–4]. However, the application of POVs still faces numerous challenges such as their strong oxidative properties and poor process ability. So it is significant to develop new derivatives of POVs [5].

Herein, we report a new POV salt, which consists of a polyoxoanion $[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]^{2-}$ and two 3-(3-ethylureido)-N,N-dimethylpropan-1-aminium cationic ligands (cf. the figure). The polyoxoanion $[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]^{2-}$ exhibits a well-known Lindqvist-type structure which consists of a $\{V_6O_{19}\}$ moiety. The bond lengths of the terminal V-O bonds are around 1.6 Å while the bond lengths of the bridging V-O bonds are 1.8 Å. Two $[HOCH_2C(OCH_2)_3]^{3-}$ subunits occupy opposite faces of the octahedron and were connected to the $V_6$ cluster through three $\mu_2$-O-V bonds of which the bond lengths are 2.0 Å. The couterions were obtained from the reaction between EDCI and water in the solvent. In the cation structure, the bond lengths

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

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| V3 | 0.22663(3) | 0.94559(7) | −0.03617(4) | 0.0224(2) |
| V1 | 0.19750(3) | 0.65452(7) | −0.08353(4) | 0.0218(2) |
| V2 | 0.19540(3) | 0.73886(7) | 0.09804(4) | 0.0217(2) |
| O2 | 0.22495(11) | 0.5859(3) | 0.11013(16) | 0.0242(7) |
| O10 | 0.15363(11) | 0.6693(3) | 0.01120(16) | 0.0215(6) |
| O4 | 0.22589(11) | 0.5127(3) | −0.03708(16) | 0.0237(7) |
| O3 | 0.25051(11) | 0.6764(3) | −0.14707(16) | 0.0244(7) |
| O8 | 0.17740(11) | 0.9079(3) | 0.04864(16) | 0.0222(6) |
| O1 | 0.2500 | 0.7500 | 0.0000 | 0.0191(8) |
| O5 | 0.15354(12) | 0.5946(3) | −0.13795(17) | 0.0284(7) |
| O6 | 0.15154(12) | 0.7417(3) | 0.16115(17) | 0.0296(7) |
| O9 | 0.17821(11) | 0.8347(3) | −0.09769(16) | 0.0220(6) |
| O7 | 0.20248(12) | 1.0774(3) | −0.06229(19) | 0.0307(7) |
| C4 | 0.09876(17) | 0.8497(4) | −0.0243(3) | 0.0277(10) |
| O12 | 0.07480(15) | 1.2386(3) | 0.0301(2) | 0.0497(10) |
| O11 | 0.01704(14) | 0.8127(4) | −0.0888(2) | 0.0513(10) |
| H11 | −0.0118 | 0.7915 | −0.0728 | 0.077* |
| C5 | 0.04256(19) | 0.8882(5) | −0.0322(3) | 0.0398(12) |
| H5A | 0.0256 | 0.8779 | 0.0177 | 0.048* |
| H5B | 0.0403 | 0.9782 | −0.0470 | 0.048* |
| C1 | 0.10150(16) | 0.7117(4) | 0.0013(3) | 0.0267(10) |
| H1A | 0.0834 | 0.7021 | 0.0504 | 0.032* |
| H1B | 0.0839 | 0.6581 | −0.0374 | 0.032* |
| C2 | 0.12441(17) | 0.8667(5) | −0.1016(3) | 0.0297(10) |
| H2A | 0.1071 | 0.8125 | −0.1403 | 0.036* |
| H2B | 0.1206 | 0.9554 | −0.1184 | 0.036* |
| C3 | 0.12362(18) | 0.9353(4) | 0.0355(3) | 0.0308(11) |
| H3A | 0.1200 | 1.0239 | 0.0185 | 0.037* |
| H3B | 0.1053 | 0.9261 | 0.0844 | 0.037* |
| Cl1 | 0.05355(7) | 1.0512(2) | −0.27121(10) | 0.0828(6) |
| N1 | 0.12680(17) | 1.4031(4) | 0.0620(3) | 0.0406(11) |
| H1 | 0.1447 | 1.4670 | 0.0447 | 0.049* |
| N3 | 0.22148(19) | 1.1043(5) | 0.1674(3) | 0.0508(12) |
| H3 | 0.2275 | 1.0401 | 0.1324 | 0.061* |
| N2 | 0.1083(2) | 1.3571(5) | −0.0639(3) | 0.0533(13) |
| H2 | 0.1323 | 1.4117 | −0.0758 | 0.064* |
| C10 | 0.2026(2) | 1.0449(5) | 0.2358(3) | 0.0405(13) |
| H10A | 0.2278 | 0.9835 | 0.2548 | 0.061* |
| H10B | 0.1703 | 1.0017 | 0.2239 | 0.061* |
| H10C | 0.1969 | 1.1090 | 0.2752 | 0.061* |
| C7 | 0.1256(2) | 1.3819(5) | 0.1438(3) | 0.0429(13) |
| H7A | 0.0951 | 1.3312 | 0.1563 | 0.051* |
| H7B | 0.1229 | 1.4638 | 0.1706 | 0.051* |
| C11 | 0.2728(2) | 1.1680(5) | 0.1834(3) | 0.0452(14) |
| H11A | 0.2686 | 1.2339 | 0.2222 | 0.068* |
| H11B | 0.2854 | 1.2059 | 0.1360 | 0.068* |
| H11C | 0.2975 | 1.1053 | 0.2022 | 0.068* |
| C6 | 0.1015(2) | 1.3290(5) | 0.0106(3) | 0.0398(12) |
| C14 | 0.0000 | 0.9557(11) | −0.2500 | 0.066(3) |
| H14A | 0.0083 | 0.9010 | −0.2056 | 0.079* |
| H14B | −0.0083 | 0.9010 | −0.2944 | 0.079* |
| C9 | 0.1826(3) | 1.1913(6) | 0.1302(4) | 0.0548(16) |
| H9A | 0.1936 | 1.2113 | 0.0776 | 0.066* |
| H9B | 0.1493 | 1.1470 | 0.1262 | 0.066* |
| C8 | 0.1757(3) | 1.3102(6) | 0.1730(3) | 0.0589(18) |
| H8A | 0.2059 | 1.3650 | 0.1660 | 0.071* |
| H8B | 0.1728 | 1.2916 | 0.2284 | 0.071* |
| C12 | 0.0780(3) | 1.3011(8) | −0.1248(4) | 0.082(3) |
| H12A | 0.0706 | 1.2121 | −0.1121 | 0.099* |
| H12B | 0.0975 | 1.3026 | −0.1730 | 0.099* |
| C13 | 0.0277(4) | 1.3730(13) | −0.1361(6) | 0.140(5) |
| H13A | 0.0041 | 1.3227 | −0.1678 | 0.210* |
| H13B | 0.0344 | 1.4535 | −0.1617 | 0.210* |
| H13C | 0.0125 | 1.3890 | −0.0861 | 0.210* |

of C-N bonds in urea group are 1.3 Å and the angle of $N_1$-$C_6$-$N_2$ is 116.0°. There is a dichloromethane solvent molecule in the crystal structure in which the bond lengths of C-Cl bond are 1.77 Å and the Cl-C-Cl angle is 110.3°.

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