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Bis(dicyclohexylammonium) tungstate dihydrate, $[NH_2(C_6H_{11})_2]_2[WO_4]\cdot 2H_2O$

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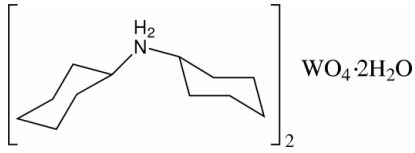
The crystal structure of the title complex is built of dicyclohexylammonium cations, $[NH_2(C_6H_{11})_2]^+$, orthotungstate dianions, $[WO_4]^{2-}$, and lattice water molecules. The cations and coordinated water molecules occupy special positions on twofold axes; the tetrahedral orthotungstate dianion is located on a $\bar{4}$ axis. Hydrogen bonds involving all N- and O-bound H atoms link the cations, anions and water molecules into an infinite three-dimensional aggregate. The crystal is isostructural with its molybdenum analog [Thiele & Fuchs (1979). *Z. Naturforsch. Teil B*, **34**, 145–154].



(I)

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 93%
R factor = 0.020
wR factor = 0.046
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Experimental

$Na_2WO_4.2H_2O$ (0.099 g, 0.3 mmol) was dissolved in a 20 ml mixture of ethanol and water (1:1) and the solution was placed in the bottom of a tube. Then a solution of dicyclohexylamine (0.1 ml, 0.5 mmol) in 10 ml methanol was carefully layered on top and the tube was kept in the dark. After about 10 d, colorless crystals were obtained in 66% yield.

Crystal data

$(C_{12}H_{24}N)_2[WO_4]\cdot 2H_2O$
M_r = 648.54
Tetragonal, $I\bar{4}2d$
a = 12.7053 (8) Å
c = 17.6737 (15) Å
V = 2853.0 (4) Å^3
Z = 4
D_x = 1.500 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 109 reflections
θ = 2.0–25.0°
μ = 4.09 mm^{-1}
T = 293 (2) K
Prism, colorless
0.45 × 0.15 × 0.10 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.756, T_{max} = 1.000
3260 measured reflections
1243 independent reflections
1106 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°
$h = -9 \rightarrow 15$
$k = -14 \rightarrow 9$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.046
S = 1.05
1243 reflections
77 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0129P)^2 + 8.2983P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.00374 (15)
Absolute structure: Flack (1983), 535 Friedel pairs
Flack parameter = 0.020 (19)

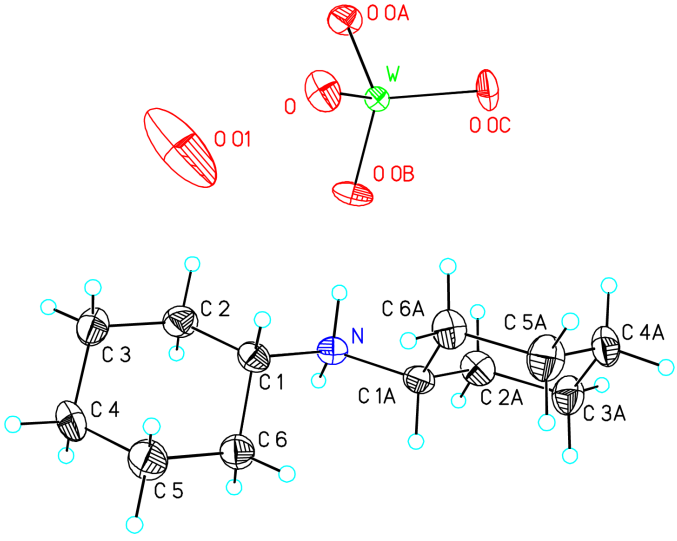


Figure 1
The cation, anion and water molecule of the title complex, shown with 30% probability displacement ellipsoids.

All H atoms (with the exception of water H atoms) were generated geometrically and allowed to ride on their parent atoms. The unique water H atom was not located.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Siemens, 1996); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Siemens (1994). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *SHELXTL*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Thiele, A. & Fuchs, J. (1979). *Z. Naturforsch. Teil B*, **34**, 145–154.