Crystal structure of dicesium hexachloromolybdate(IV), $\text{Cs}_2[\text{MoCl}_6]$

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Abstract

$\text{Cl}_6\text{Cs}_2\text{Mo}$, cubic, $Fm\bar{3}m$ (no. 225), $a = 10.2121(6)$ Å, $V = 1065.0$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.025$, $wR_{\text{ref}}(F^2) = 0.067$, $T = 293$ K.

Source of material

The reaction of CsCl (0.61 g, 3.62 mmol), MoCl_5 (0.49 g, 1.79 mmol) in a sealed silica tube at a temperature gradient of

925K/915K for four weeks afforded a small amount of octahedral crystals in the low-temperature zone. Mo(VI) was formed by disproportionation of Mo(V) in Mo(IV) and Mo(VI).

Discussion

The title compound consists of Cs^+ cations and the $[\text{MoCl}_6]^{2-}$ anions and crystallizes with the well-known K_2PtCl_6 structure type. The Mo atoms are each octahedrally coordinated by six Cl atoms, $d(\text{Mo}-\text{Cl}) = 2.355(2)$ Å, and the Cs^+ cations are each cuboctahedrally surrounded by twelve Cl atoms, $d(\text{Cs}-\text{Cl}) = 3.6160(2)$ Å.

Differently to the title compound, the colors of crystals of $\text{Cs}_2[\text{MoCl}_{6-n}\text{O}_n]$ had been green ($n = 0$ [1], $n = 1$ [2]). An explanation could not be found. Crystals of the same crimson color were obtained after a second preparation. The crystal structures of $\text{Tl}_2[\text{MoCl}_6]$ [3] and $\text{Cs}_2[\text{WCl}_6]$ [4] are isotypic with the structure of the title compound.

Table 1. Data collection and handling.

Crystal:	crimson octahedron, size $0.26 \times 0.28 \times 0.29$ mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	94.00 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II, ω/ϕ
$2\theta_{\text{max}}$:	60.88°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	561, 115
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 109
$N(\text{param})_{\text{refined}}$:	7
Program:	SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs(1)	8c	¼	¼	¼	0.0371(4)	U_{11}	U_{11}	0	0	0
Mo(1)	4a	0	0	0	0.0575(7)	U_{11}	U_{11}	0	0	0
Cl(1)	24e	0	0.2306(2)	0	0.0448(7)	0.037(1)	U_{11}	0	0	0

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