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Zeitschrift für Anorganische und Allgemeine Chemie

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Journal:	*Zeitschrift für Anorganische und Allgemeine Chemie*
Manuscript ID:	zaac.201000242.R1
Wiley - Manuscript type:	Article
Date Submitted by the Author:	23-Jul-2010
Complete List of Authors:	Ahmed, Ejaz; Technische Universität Dresden, Fachrichtung Chemie und Lebensmittelchemie Ahrens, Eike; Technische Universität Dresden, Fachrichtung Chemie und Lebensmittelchemie Heise, Martin; Technische Universität Dresden, Fachrichtung Chemie und Lebensmittelchemie Ruck, Michael; Technische Universität Dresden, Fachrichtung Chemie und Lebensmittelchemie
Keywords:	Tellurium, Polycations, Cluster compounds, Ionic Liquids, Magnetic properties

Full Paper

A Facile Route for the Synthesis of Polycationic Tellurium Cluster Compounds: Synthesis in Ionic Liquid Media and Characterization by Single-Crystal X-Ray Crystallography and Magnetic Susceptibility

Ejaz Ahmed,[a] Eike Ahrens,[a] Martin Heise,[a] and Michael Ruck*[a]

Keywords: Tellurium; Polycations; Cluster compounds; Ionic Liquids; Magnetic properties

Abstract. An innovative soft chemical approach has been applied, using ionic liquids as an alternative reaction medium for the synthesis of tellurium polycationic cluster compounds at room temperature. $[Mo_2Te_{12}]I_6$, $Te_6[WOCl_4]_2$, and $Te_4[AlCl_4]_2$ have been isolated from the ionic liquid $[BMIM]Cl/AlCl_3$ ($[BMIM]^+$: 1-*n*-butyl-3-methylimidazolium) and characterized. Black, cube-shaped crystals of $[Mo_2Te_{12}]I_6$, which is not accessible by conventional chemical transport reaction, are obtained by reacting the elements at room temperature in $[BMIM]Cl/AlCl_3$. The monoclinic structure ($P2_1/n$, a = 1138.92(2) pm, b = 1628.13(2) pm, c = 1611.05(2) pm, β = 105.88(1) °) is homeotypic to the triclinic bromide $[Mo_2Te_{12}]Br_6$. In the binulear complex $[Mo_2Te_{12}]^{6+}$, the molybdenum(III) atoms are η^4-coordinated by terminal Te_4^{2+} rings and two bridging η^2-Te_2^{2-} dumbbells. Despite the short Mo···Mo distance of 297.16(5) pm, coupling of the magnetic moments is not observed. The paramagnetic moment of 3.53 μ_B per molybdenum(III) atom corresponds to an electron count of seventeen. Black crystals of monoclinic $Te_6[WOCl_4]_2$ are obtained by the oxidation of tellurium with $WOCl_4$ in $[BMIM]Cl/AlCl_3$. Tellurium and tellurium(IV) synproportionate in the ionic liquid at room temperature yielding violet crystals of orthorhombic $Te_4[AlCl_4]_2$.

Introduction

The formation of polycationic clusters in Lewis acid is a well-known feature of tellurium chemistry. Te_4^{2+}, Te_6^{4+}, Te_6^{2+}, Te_8^{2+}, Te_7^{2+}, and Te_{10}^{2+} have been structurally characterized [1]. There are three different routes for the synthesis of tellurium polycationic clusters in literature: The classical route, performed by *Gillespie* and co-workers, is the oxidation of tellurium by AsF_5 or SbF_5 in liquid SO_2 using a temperature range from –196 °C to 25 °C [2]. High temperature synthesis was carried out by *Corbett et al*. [3] and *Beck et al*. [1]. *Kloo* and co-workers introduced the first room temperature synthesis using GaX_3–benzene media (X = Br, Cl) [4].

Recently, we have reported a new room temperature route for the synthesis of bismuth polycationic clusters from ionic liquids and anticipated that this approach can be extended to other elements of the periodic table, e.g. tellurium [5].

The key factors of this new synthetic route are:

i) Very high solubility of the elements (Bi, Te, Sb, Se, Ga, In) and their halogenides at room temperature allow reactions under mild conditions involving one-pot synthesis.

ii) Low temperature synthesis eliminates the risk of product decomposition.

iii) Easier reproducibility of the products.

iv) Controllable parameters, such as solvent acidity, oxidizing/reducing agent, or halide acceptor, allow modifying the synthesis with the objective to crystallize different products.

The application of this approach in order to synthesize tellurium cluster compounds at room temperature resulted in the isolation of $[Mo_2Te_{12}]I_6$, $Te_4[AlCl_4]_2$, and $Te_6[WOCl_4]_2$.

* Prof. Dr. Michael Ruck
Fax: (+49) 351-463-37287
E-mail: Michael.Ruck@tu-dresden.de
[a] Professur für Anorganische Chemie II
Technische Universität Dresden
D-01062 Dresden, Germany.

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/zaac201000xxx

Results and Discussions

Synthesis and Structure of $[Mo_2Te_{12}]I_6$

$[Mo_2Te_{12}]I_6$ is homeotypic to $[Mo_2Te_{12}]Br_6$ containing a heteropolycationic $[Mo_2Te_{12}]^{6+}$ cluster. $[Mo_2Te_{12}]Br_6$ was synthesized by *Beck* through chemical vapor transport (CVT) [6a]. He used a temperature gradient of 340 °C to 270 °C to get single-crystals of $[Mo_2Te_{12}]Br_6$ within four weeks. Despite many attempts, the CVT synthesis of the analogous iodide failed [7]. In our studies, $[Mo_2Te_{12}]I_6$ was obtained by reacting Mo, Te and I_2 in the Lewis acidic ionic liquid [BMIM]Cl/$AlCl_3$ at room temperature to give a dark solution within minutes. After overnight stirring and a crystallization period of only three days, single-crystals could be isolated with a yield of 35 %.

The monoclinic cell (space group $P2_1/n$) contains four formula units. The cationic complex $[Mo_2Te_{12}]^{6+}$ has a pair of molybdenum atoms, which are connected by two bridging η^2-Te_2^{2-} groups (Figure 1). In addition, each molybdenum atom is coordinated by a terminal polycationic Te_4^{2+} ligand in η^4 mode. As a result, both molybdenum atoms are in square-antiprismatic environment.

Figure 1. $[Mo_2Te_{12}]^{6+}$ complex cation. The ellipsoids represent 95 % probability at 150(2) K.

The point symmetry of the $[Mo_2Te_{12}]^{6+}$ complex in both compounds, i.e. in monoclinic $[Mo_2Te_{12}]I_6$ and in triclinic $[Mo_2Te_{12}]Br_6$, is close to D_{2h} (*mmm*), although the site symmetry of the cluster is C_i in the bromide and C_s in the iodide. Moreover, there is only one independent complex present in the unit cell of $[Mo_2Te_{12}]I_6$ whereas two independent complexes are found in $[Mo_2Te_{12}]Br_6$.

The distances between molybdenum and tellurium atoms of the Te_2^{2-} groups are slightly shorter (272.35(4) pm to 274.41(4) pm) than those between molybdenum and tellurium atoms of the Te_4^{2+} rings (278.99(5) pm to 285.38(4) pm). The Te_4^{2+} rings show significant distortion (distances: 276.12(5) pm to 286.85(4) pm, angles: 87.19(1) ° to 92.61(1) °) from D_{4h} to butterfly conformation with dihedral angles 5.3 ° and 5.9 ° (Table 1).

The complexes $[(Mo^{3+})_2(Te_2^{2-})_2(Te_4^{2+})_2]^{6+}$ are surrounded by iodide ions, which connect them into layers (Figure 2). The shortest distance between an iodide ion and an atom of the Te_4^{2+} rings is 302.20(4) pm.

Figure 2. Section of the crystal structure of $[Mo_2Te_{12}]I_6$. The ellipsoids represent 95 % probability at 150(2) K.

$[Mo_2Te_{12}]I_6$ is paramagnetic with Weiss temperature θ = +5.8 K (Figure 3). An analysis based on the Curie-Weiss law gives a paramagnetic effective moment of 3.53 μ_B per molybdenum atom, which is slightly lower than the spin-only value of 3.87 μ_B for a d^3 configuration. In low fields some traces of ferromagnetism are found but these are very weak and most probably due to minor impurity phases.

The absence of intramolecular coupling of the magnetic cations in $[Mo_2Te_{12}]I_6$ is astonishing, since the short distance of 297.16(5) pm between the molybdenum(III) atoms suggests a chemical bond. In the present case the complex is a diradical with seventeen electrons per central atom. The electron counting scheme adds up the three valence electrons of a molybdenum(III) atom, six electrons of the π-system of the Te_4^{2+} ring, and four electrons (two lone-pairs) of each Te_2^{2-} group (3 + 6 + 4·2 = 17).

In contrast, the closely related *catena*-compound $MoTe_4Br = [(Mo^{3+})_2(Te_2^{2-})_2(Te_2Br^-)_2]$ with a Mo–Mo bond length of 300.5(2) pm shows only weak, nearly temperature-independent paramagnetism with a magnetic moment of 0.4 μ_B at 310 K, indicating a strong coupling and an eighteen electron situation [6a].

Figure 3. Temperature dependence of reciprocal molar susceptibility for $[Mo_2Te_{12}]I_6$ in a magnetic field of H = 35 kOe.

Synthesis and Structure of $Te_4[AlCl_4]_2$

$Te_4[AlCl_4]_2$ was reported by *Corbett* and co-workers through synproportionation at 250 °C [3]. Contrary to higher temperature method, we obtained $Te_4[AlCl_4]_2$ by reacting Te and $TeCl_4$ in the Lewis acidic ionic liquid [BMIM]Cl/$AlCl_3$ at room temperature. Addition of a small amount of

anhydrous NaCl is helpful for getting single crystals in two days (66 % yield). The orthorhombic compound contains planar Te_4^{2+} polycations and tetrahedral $AlCl_4^-$ anions. The atomic coordinates agree within three standard deviations with those published earlier [3].

Synthesis and Structure of $Te_6[WOCl_4]_2$

Crystals of $Te_6[WOCl_4]_2$ have previously been synthesized within two weeks using CVT in the temperature gradient of 230 °C to 210 °C [6b]. We carried out the oxidation of elemental tellurium with $WOCl_4$ in ionic liquids at room temperature. Addition of anhydrous NaCl to the dark coloured filtrate resulted in precipitation of black crystals in three days (yield: 39 %).

The crystal structure consists of boat shaped Te_6^{2+} polycations and one-dimensional square-pyramidal $WOCl_4^-$ anions. The atomic coordinates agree within six standard deviations with those published by *Beck* [6b].

Conclusion

We have developed an ionic-liquid-based room-temperature synthesis of homo- and heteropolyatomic cluster compounds. The significance of the present study is manifold. The redox reactions in ionic liquids provide an efficient, one-step route to the inorganic clusters. Salting out is quite helpful technique to get single crystals in few days. The additional advantages of this synthetic route are:

i) Inclusion of the organic cation has not been observed yet.
ii) Ionic liquids render stability to the ionic intermediates [5].
iii) Reactions are faster and there is no need to wait for weeks as in chemical vapor transport (CVT). Crystal growth requires only few days, and the yields can be rather high.
iv) Moreover, ionic liquids are regarded as economical and environmental friendly solvents. They could be alternatives to cancer causing organic solvents [8].

These features make the soft chemical procedure a versatile and general route towards the synthesis of other innovative materials.

Experimental Section

Chemicals: Starting materials were commercial 1-methylimidazole (Merck, 99 %), 1-chlorobutane (Merck, 98.5 %), $AlCl_3$ (Fluka, anhydrous, 98%), Te (Aldrich, 99.999 %), $WOCl_4$ (Aldrich, 98 %), Mo (Aldrich, 99.95 %), I_2 (Grüssing, 99.5 %), and Cl_2 (Air Liquide, 99.8 %). $TeCl_4$ was synthesized from the elements [9]. $AlCl_3$ was sublimated three times. I_2 was sublimated twice over BaO. Organic reagents were distilled before use. Because of the high moisture sensitivity of the anhydrous metal halides used in this work, all manipulations were performed under dry argon (99.999 %) atmosphere in standard Schlenk tubes.

Synthesis of $[Mo_2Te_{12}]I_6$: The ionic liquid [BMIM]Cl/$AlCl_3$ ($[BMIM]^+$: 1-*n*-butyl-3-methylimidazolium) were prepared according to a literature procedure [10]. The starting materials were added together in the Lewis acidic [BMIM]Cl/$AlCl_3$ ionic liquid (molar ratio = 1:1.3, volume = 1.5 ml) having molar ratio of Mo:Te:I = 1:6:3 (total mass = 355 mg). The reaction mixture was left for stirring overnight at room temperature. After three days, black, cube-shaped crystals were obtained. Yield: 35 %. The energy-dispersive X-ray (EDX) analysis of a single crystal gave the atomic ratio Mo:Te:I = 10:55:35 (expected = 10:60:30). EDX is only a semiquantitative technique, which sometimes cannot give exact estimation for two closely related elements.

Synthesis of $Te_4[AlCl_4]_2$: The stoichiometric amount of Te and $TeCl_4$ (total mass = 260 mg) were added to the Lewis acidic [BMIM]Cl/$AlCl_3$ (molar ratio = 1:1.3, volume = 1.5 ml) ionic liquid. The reaction mixture was left for stirring overnight at room temperature then filtered to separate unreacted material. Addition of a small amount of anhydrous NaCl into the dark violet filtrate led to the precipitation of violet, column-like crystals in two days. Yield: 66 %. EDX: Te:Al:Cl = 27:15:58 (expected = 29:14:57).

Synthesis of $Te_6[WOCl_4]_2$: The same procedure was used as described for $Te_4[AlCl_4]_2$, starting from Te and $WOCl_4$ (total mass = 288 mg). After three days, black, irregular shaped crystals were obtained. Yield: 39 %. Because of the high moisture sensitivity of $Te_6[WOCl_4]_2$, it was not yet possible to carry out EDX analysis.

X-Ray Crystallography: Since all compounds are sensitive towards hydrolysis by moist air, crystals were selected in a glove box (concentration of O_2 < 0.1 ppm and H_2O < 0.1 ppm) and filled in glass capillaries of 0.2 mm diameter, which were sealed by a hot filament in the argon atmosphere. Intensity data of a single-crystal of $[Mo_2Te_{12}]I_6$ were recorded using a Bruker Kappa CCD diffractometer and graphite-monochromatized Mo-*K*α radiation (λ = 71.073 pm) at 150 K. Data sets of single-crystals of $Te_4[AlCl_4]_2$ and $Te_6[WOCl_4]_2$ were collected on an imaging-plate diffraction system IPDS-I (Stoe) using graphite-monochromatized Mo-Kα radiation at room temperature. The raw data were corrected for background, polarization and the Lorentz factor. The microscopic description of the crystal shapes, which was later used in the numerical absorption corrections [11], was optimized using sets of reflections that are equivalent in the monoclinic Laue class for $[Mo_2Te_{12}]I_6$ and $Te_6[WOCl_4]_2$, orthorhombic for $Te_4[AlCl_4]_2$ [12]. The structures were solved with direct methods and refined using *SHELX97* [13]. After refinement of the structure model of $[Mo_2Te_{12}]I_6$, we found a residual electron density of +5.08 $e \cdot 10^{-6}$ pm^{-3} which can be interpreted as a second orientation of the Te_4^{2+} rings with weight of a few percents. This disorder could not be refined neither in a split atom model nor in a twin model. Slow cooling of the crystals did not lead to ordering. Graphics of the structures were developed using the program Diamond 3.2e [14]. The results of the structure determinations are summarized in Tables 2 and 3. Further crystal structure data have been deposited at the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany (Fax: +49-7247-808-666, E-Mail: crysdata@fiz-karlsruhe.de). Inquiries should be accompanied by the depository number CSD-421881 ($[Mo_2Te_{12}]I_6$), CSD-421883 ($Te_4[AlCl_4]_2$), and CSD-421882 ($Te_6[WOCl_4]_2$).

Magnetic Susceptibility Measurements: The magnetic susceptibility of $[Mo_2Te_{12}]I_6$ was measured with a SQUID magnetometer (MPMS-XL7, Quantum Design) between 1.8 K and 400 K of a 50 mg sample in four different magnetic fields between 0.10 and 70 kOe.

Supporting information: Structural parameters for $Te_4[AlCl_4]_2$ and $Te_6[WOCl_4]_2$, as well as data of the temperature dependence of the reciprocal molar susceptibility for $[Mo_2Te_{12}]I_6$ in different magnetic fields are available on internet http://dx.doi.org/10.1002/zaac.

Acknowledgments. We thank *Dr. W. Schnelle* (Max Planck Institute for Chemical Physics of Solids, Dresden) for performing magnetic susceptibility measurements and *Prof. Dr. J. Beck* (University of Bonn) for helpful discussions. The authors also gratefully acknowledge the Higher Education Commission of Pakistan and the German Academic Exchange Service (DAAD) for financial support.

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Table 1. Selected interatomic distances (pm) in $[Mo_2Te_{12}]I_6$.

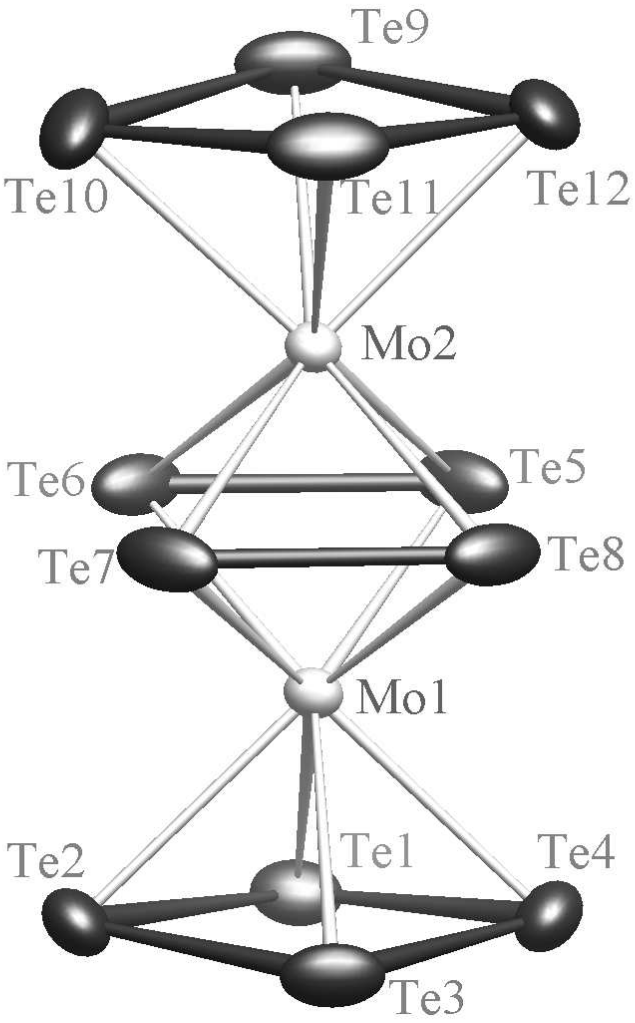
atoms		distance	atoms		distance
Te1–	Te2	286.85(4)	Te7–	Te8	276.81(4)
	Te4	276.97(4)		Mo1	272.73(4)
	Mo1	284.54(4)		Mo2	273.71(4)
Te2–	Te3	276.93(4)	Te8–	Mo1	273.10(4)
	Mo1	280.18(4)		Mo2	273.49(4)
	I2	302.20(4)	Te9–	Te10	283.07(5)
Te3–	Te4	280.66(4)		Te12	276.12(5)
	Mo1	284.54(4)		Mo2	285.38(4)
Te4–	Mo1	280.24(4)	Te10–	Te11	276.91(5)
Te5–	Te6	276.71(4)		Mo2	279.97(5)
	Mo1	274.05(4)		I6	314.77(5)
	Mo2	274.41(4)	Te11–	Te12	281.18(5)
Te6–	Mo1	273.22(4)		Mo2	284.43(5)
	Mo2	272.35(4)	Te12–	Mo2	278.99(5)
			Mo1–	Mo2	297.16(5)

Table 2. Crystallographic data and details of the structure determinations for $[Mo_2Te_{12}]I_6$, $Te_4[AlCl_4]_2$, and $Te_6[WOCl_4]_2$.

Compound	$[Mo_2Te_{12}]I_6$	$Te_4[AlCl_4]_2$	$Te_6[WOCl_4]_2$
Crystal system	monoclinic	orthorhombic	monoclinic
Space group	$P2_1/n$	$Pbca$	$P2_1/c$
Temperature (K)	150(2)	293(2)	293(2)
a, b, c (pm), β (°)	1138.92(2), 1628.13(2), 1611.05(2), 105.88(1)	1072.1(1), 1412.8(1), 1183.2(2), 90	917.07(8), 1631.9(2), 3077.5(3), 117.008(7)
V (10^6 pm^3)	2873.42(7)	1792.1(4)	4103.4(7)
Formula units per cell	4	4	8
Calculated density (g·cm^{-3})	5.74	3.14	4.69
Measurement device	CCD Kappa (Bruker)	imaging plate diffractometer (IPDS-I, Stoe)	
Radiation	graphite-monochromated Mo-$K\alpha$ radiation (λ = 71.073 pm)		
Measurement range	$-17 \le h \le 17$	$-11 \le h \le 11$	$-9 \le h \le 9$
	$-27 \le k \le 26$	$-14 \le k \le 14$	$-18 \le k \le 18$
	$-27 \le l \le 26$	$-12 \le l \le 12$	$-34 \le l \le 34$
μ(Mo-$K\alpha$) (mm^{-1})	19.25	7.70	20.60
Measured reflections	72507	8267	23195
Unique reflections $F_o > 4\sigma(F_o)$	13529, 9970	1106, 868	5789, 3911
R(int), $R(\sigma)$	0.041, 0.039	0.079, 0.038	0.086, 0.066
R_1 [$F_o > 4\sigma(F_o)$]	0.035	0.033	0.063
R_1 (all F_o)	0.060	0.050	0.103
wR_2 (all F_o^2)	0.060	0.066	0.070
No. of parameters	182	64	305
Goodness of fit	1.27	1.20	1.27
$\Delta\rho$(max, min) (e·10^{-6} pm^{-3})	+5.08, –4.31	+0.86, –0.76	+1.33, –1.23

Table 3. Coordinates and equivalent isotropic displacement parameters for $[Mo_2Te_{12}]I_6$ at 150(2) K. All atoms occupy Wyckoff positions 4*e*. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq} / pm^2
Te1	0.31709(3)	0.37043(2)	0.17595(2)	131.6(6)
Te2	0.36271(3)	0.38843 (2)	0.35915(2)	146.1(6)
Te3	0.27428(3)	0.54712 (2)	0.32742(2)	155.6(6)
Te4	0.20828(3)	0.52383(2)	0.14798(2)	155.9(6)
Te5	–0.02771(3)	0.34316(2)	0.10664(2)	155.7(6)
Te6	0.07955(3)	0.24667(2)	0.24851(2)	145.1(6)
Te7	0.03788(3)	0.41154(2)	0.39261(2)	164.7(6)
Te8	–0.07073(3)	0.50746(2)	0.25047(2)	160.4(6)
Te9	–0.26539(3)	0.20622(2)	0.17358(2)	176.9(6)
Te10	–0.19369(3)	0.23018(2)	0.35450(2)	207.7(7)
Te11	–0.30688(3)	0.38207(2)	0.32504(2)	188.6(7)
Te12	–0.35434(3)	0.36452(2)	0.14525(2)	188.0(6)
Mo1	0.12620(3)	0.41140(2)	0.25141(2)	73.4(6)
Mo2	–0.11637(3)	0.34246(2)	0.24880(2)	71.9(6)
I1	0.42135(3)	0.20062(2)	0.28829(2)	171.5(6)
I2	0.36168(3)	0.43821(2)	0.53966(2)	227.0(7)
I3	0.16105(3)	0.44977(2)	–0.04292(2)	235.9(7)
I4	–0.33000(3)	0.29032(2)	–0.04388(2)	191.8(6)
I5	–0.07943(3)	0.05472(2)	0.29289(2)	172.1(6)
I6	–0.14299(4)	0.32255(2)	0.53245(2)	282.7(8)



$[Mo_2Te_{12}]^{6+}$ complex cation. The ellipsoids represent 95 % probability at 150(2) K.
265x281mm (96 x 96 DPI)

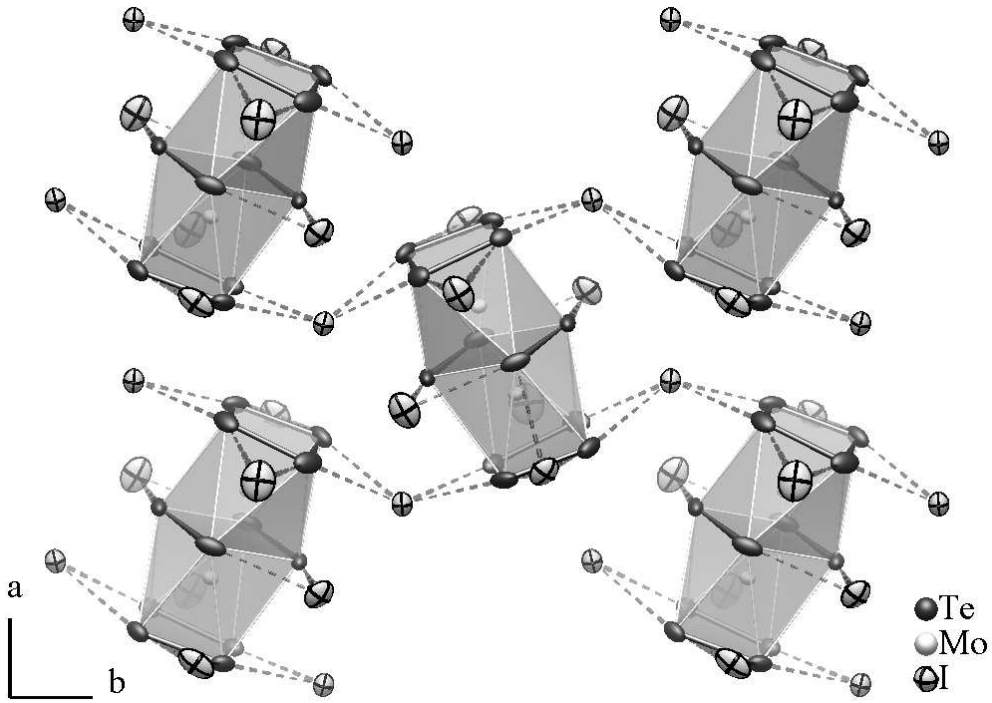
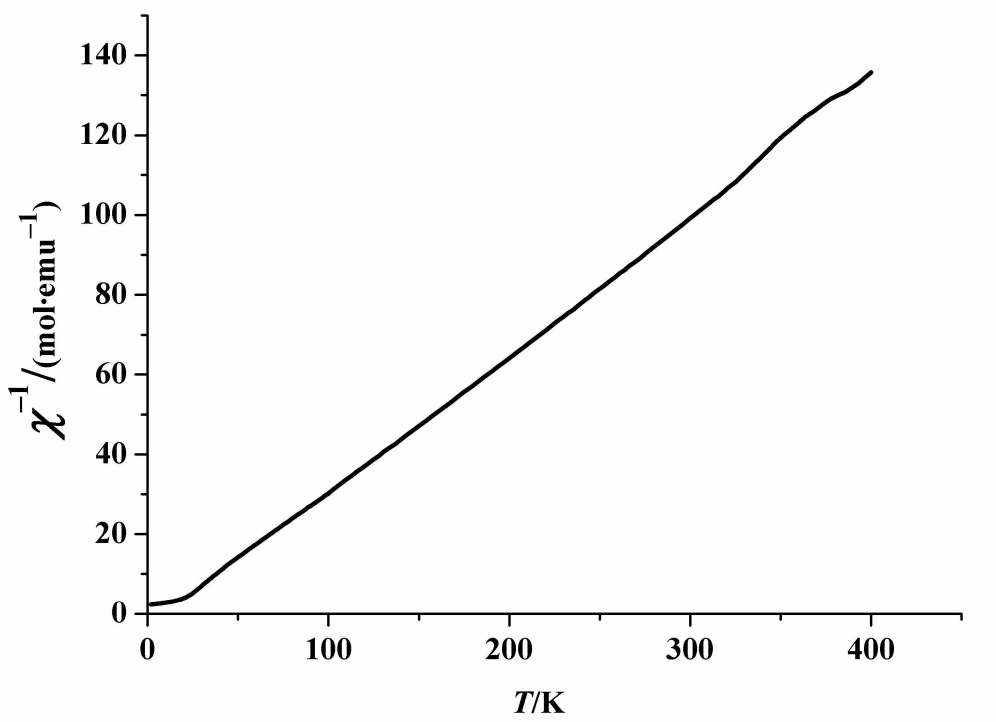


Figure 2. Section of the crystal structure of $[Mo_2Te_{12}]I_6$. The ellipsoids represent 95 % probability at 150(2) K.
265x187mm (96 x 96 DPI)



Temperature dependence of reciprocal molar susceptibility for $[Mo_2Te_{12}]I_6$ in a magnetic field of H = 35 kOe.
242x175mm (300 x 300 DPI)