Research Article

X-ray absorption fine structure spectroscopic study of $(NH_4)_2ReF_6$

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Abstract
The $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) salts have been investigated by x-ray absorption fine structure spectroscopy. The Re-F distance determined by EXAFS in $(NH_4)_2ReF_6$ (i.e., 1.95 Å) is in good agreement with the one determined by single crystal x-ray diffraction in A_2ReF_6 salts (A = K, Rb, Cs). The XANES studies of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) indicates that the positions of the absorption edge and of the white line are shifted to higher energy when moving from I to F. These shifts have been explained in terms of the crystal field splitting parameter and covalent charge carried by the Re atoms. Calculations of the XANES spectra of the ReX_6^{2-} (X = F, Cl, Br, I) anions at the Re-L_3 edge have been performed and the calculated shifts and intensity of the white lines reproduce well the experimental observations.

Keywords Rhenium · Halogen · Fluorine · X-ray absorption fine structure spectroscopy

1 Introduction

In compounds containing ReF_6 octahedra, the Re atom can be found in oxidation state +7 (e.g., $ReF_6(Sb_2F_{11})$) [1], +6 (e.g., ReF_6) [2], +5 (e.g., $CsReF_6$) [3], and +4 (e.g., A_2ReF_6; A = K, Rb, Cs) [4]. Among these species, ReF_6 and ReF_6^{2-} have been the most studied. Rhenium hexafluoride has been studied in the solid-state [2], in solution [5], and in the gas phase [6]; it exhibits interesting properties that can be used for chemical vapor deposition [7], and fluorination of organic substrates [8]. Species containing the ReF_6^{2-} anion have been characterized by x-ray diffraction [9], vibrational [10], and electronic spectroscopy [11]. Because technetium is a major fission product of the nuclear industry and rhenium is an often used as a homologue of technetium for studies related to nuclear cycle application, x-ray absorption study on rhenium halides could inform about the behavior of technetium halides in molten salts for nuclear fuel cycle reprocessing [12]. Among the ReF_6^{2-} salts, $(NH_4)_2ReF_6$ has been used as a precursor for the preparation of compounds with interesting magnetic properties (e.g., $[Zn(viz)_4(ReF_6)]$; viz = vinylimidazole) [13]. While $(NH_4)_2ReF_6$ has been characterized by powder x-ray diffraction [14], its single crystal structure has not been determined, and the Re-F distances in $(NH_4)_2ReF_6$ are not reported.

In cases where x-ray quality single crystals cannot be isolated, x-ray absorption spectroscopy has proven to be a powerful technique for the determination of oxidation states and structural parameters. The study of the x-ray absorption near edge structure (XANES) spectra (~ 0–50 eV above the absorption edge) can provide information about the oxidation state of the absorbing atoms while the extended x-ray absorption fine structure (EXAFS) (~ 50–1000 eV above the edge) can provide information on chemical environment and structural parameters (i.e., metal–ligand distances).

X-ray absorption fine structure spectroscopy is a versatile technique for the characterization of Re species; it has been used for the speciation of Re in anhydrous HF, chloride molten salts [12] and borosilicate glass [15]. In a previous study, EXAFS spectroscopy has also been used

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for the characterization of $(NH_4)_2ReX_6$ (X = Cl, Br, I), but the XANES spectra of these salts have not been reported [16]. A XANES study of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) will provide information on the effect of the halogen ligands on the absorption edge. Here we report XAFS measurements on $(NH_4)_2ReX_6$ (X = F, Cl, Br, I), determine the Re-F distance in $(NH_4)_2ReF_6$ and study the effect of the halogen ligand on the XANES spectra of the ReX_6^{2-} anions.

2 Experimental section

2.1 Materials and reagents

Caution! Because of the corrosive nature of molten ammonium bifluoride, the preparation must be performed in a well-ventilated fume hood. All chemicals, solvents and reactants (e.g., $(NH_4)ReO_4$, NH_4HF_2) were purchased from Sigma – Aldrich and used as received.

2.2 Preparation of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) salts

The $(NH_4)_2ReX_6$ salts (X = Cl, Br, I) were prepared from the reduction of NH_4ReO_4 with hypophosphorous acid in concentrated HX in the presence of NH_4X [17].

2.2.1 Preparation of $(NH_4)_2[ReF_6]$

The $(NH_4)_2ReF_6$ salt was prepared from the treatment of $(NH_4)_2[ReBr_6]$ (1.0 g, 1.42 mmol) with excess NH_4HF_2 (0.81 g, 14.2 mmol) in a nickel crucible at 300 °C for 20 min in a box furnace. After thermal treatment, the resulting solid product was allowed to cool to room temperature and was washed with MeOH (4 × 10 mL). Subsequently, the product was washed with several aliquots of H_2O-MeOH mixture (3 × 5 mL, 1:4 volume ratios) and centrifuged. The resulting solid was dissolved in warm H_2O (5 mL) and evaporated slowly at room for 2 weeks; after this time, white $(NH_4)_2ReF_6$ was obtained (300.0 mg, yield = 64%). The $(NH_4)_2ReF_6$ salt was characterized in water by UV–Visible spectroscopy and in the solid-state by powder x-ray diffraction [18].

2.2.2 Preparation of $(NH_4)_2[ReCl_6]$

NH_4ReO_4 (1.003 g, 3.740 mmol) was dissolved in 10 mL of concentrated HCl and 1 mL of H_3PO_2 was added to the HCl solution. The resulting solution was refluxed at 90 °C while stirring for 30 min. After cooling, NH_4Cl (200.0 mg, 3.739 mmol) dissolved in 0.5 mL of H_2O was added. Reflux was continued for an additional 20 min at 90 °C and green precipitate was observed. The mixture was cooled in an ice bath. The resulting green precipitate was filtered in vacuum and washed (3 × 3 mL) with isopropanol followed by (3 x 3 mL) with diethyl ether. Yield: 1.479 g (90.9%).

2.2.3 Preparation of $(NH_4)_2[ReBr_6]$

$(NH_4)_2[ReBr_6]$ was prepared similarly to $(NH_4)_2[ReCl_6]$. NH_4ReO_4 (1.064 g, 3.966 mmol) was dissolved in 10 mL of concentrated HBr and NH_4Br (391.2 mg, 3.990 mmol) was used. A dark red precipitate ($(NH_4)_2[ReBr_6]$) was observed. Yield: 2.028 g (72.8%).

2.2.4 Preparation of $(NH_4)_2[ReI_6]$

NH_4ReO_4 (1.088 g, 4.057 mmol) was dissolved in 10 mL of concentrated HI and NH_4I (588.0 mg, 4.057 mmol) dissolved in 1 mL of H_2O and a dark purple precipitate was observed. The precipitate was filtered in vacuum and washed (3 x 3 mL) with acetone. Yield: 3.101 g (77.7%).

2.3 XAFS measurements

XAFS measurements were performed at the Advanced Photon Source at the BESSRC-CAT 12 BM station at Argonne National Laboratory. The Re salts were diluted in BN (5% by mass) and sandwiched between layers of Kapton tape. XAFS spectra were recorded at the Re-L_3 edge (10 533 eV) in fluorescence mode at room temperature using a 13-element germanium detector. A double crystal of Si [1 1 1] was used as a monochromator. Energy calibration was performed using a Ge foil (K edge = 11 103 eV). Several XAFS spectra were recorded over the k range [0–16 Å]$^{-1}$ and averaged. The background contribution was removed using Athena software [19]. The Fourier transform and data analysis were performed using WINXAS [20]. For the fitting procedure, scatterings wave functions were calculated by FEFF8.2 [21]; the input files were generated by ATOMS [22]. The XANES calculations were performed using the FDMNES code [23].

3 Results and discussion

3.1 EXAFS analysis

The extracted EXAFS spectrum was k^3-weighted and the Fourier Transform (FT) performed in the k-range [2.5–14.5] Å^{-1}. The FT exhibits one peak centered at R + Δ ~ 1.60 Å.

Because of our interest in determining the Re-F distance in $(NH_4)_2ReF_6$, we focused our EXAFS study on the first coordination shell around the Re atom. In this context, a window filter was performed on the first peak of the FT between R + ΔR = [1–2.1] Å. The FT was back-transformed and the corresponding EXAFS spectra fitted in the k-range

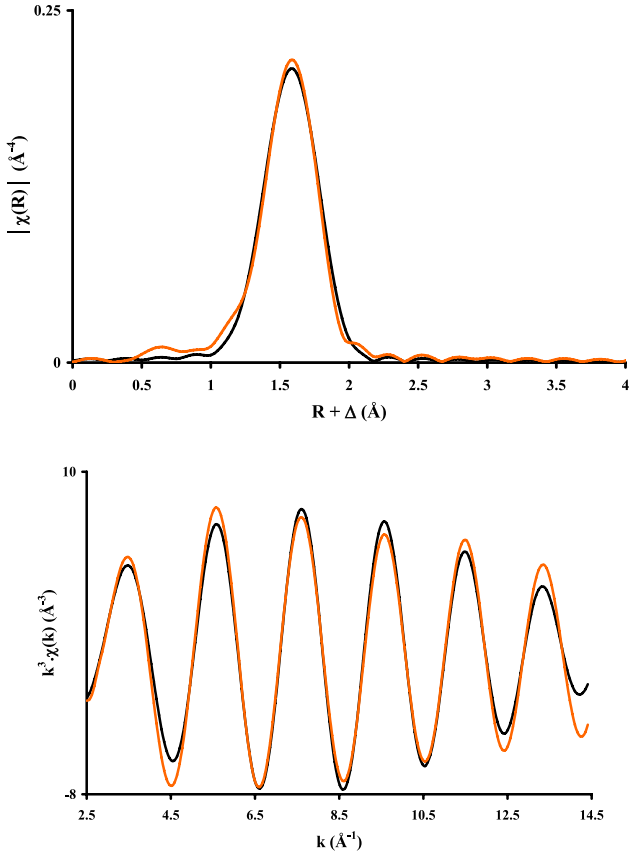

Fig. 1 Adjustment of filtered FT (top) and back transformed k^3-EXAFS (bottom) spectra of $(NH_4)_2ReF_6$. Fourier filtering between R+Δ R=[1–2.1] Å; adjustment between k=[2.5–14.5] $Å^{-1}$. Experimental data in black and fit in orange

Table 1 Structural parameters obtained from the adjustment of the k^3-EXAFS spectrum of $(NH_4)_2ReF_6$

	C.N.	R (Å)	σ^2 ($Å^2$)
Re-F	5.5 ± 1.1	1.95(2)	0.0016(1)

ΔE_0 = 5.53 eV. Residual = 4.82%

[2.5–14.5] $Å^{-1}$ using the Re-F scattering factor calculated for K_2ReF_6 [24].

The adjustment of the EXAFS spectrum was performed under the constraints S_0^2 = 0.9. In the fitting procedure, all the parameters (coordination number, σ^2, R and ΔE_0) were allowed to vary. Usually, the uncertainty on the coordination number (C.N.) and the uncertainty on the distance (R) determined by EXAFS spectroscopy are respectively 20% and 1%.

The fitted k^3-EXAFS spectrum and FT are presented in Fig. 1 The structural parameters are presented in Table 1.

The adjustment indicates that the environment around the Re atom is constituted by 5.5 ± 1.1 fluorine atoms at 1.95(2) Å. The EXAFS analysis is consistent with the presence of the ReF_6^{2-} species. The Re-F distance found in $(NH_4)_2ReF_6$ by EXAFS is comparable to the one found by single crystal x-ray diffraction (SCXRD) in A_2ReF_6 (Table 2).

Table 2 Average Re-F distances (Å) in Re(+7), Re(+6), Re(+5) and Re(+4) fluorides species determined by x-ray diffraction and XAFS spectroscopy (in italic)

Compounds	Re-F	Re oxidation state	References
$CsReF_8$	1.879	7	[25]
ReF_7	1.842	7	[26]
$(NO_2)_2ReF_8$	1.928	6	[27]
ReF_6	1.824; *1.817*	6	[2]
$CsReF_6$	1.863	5	[3]
K_2ReF_6	1.948; 1.953	4	[4], [9]
Cs_2ReF_6	1.959	4	[4]
Rb_2ReF_6	1.945	4	[4]
$(NH_4)_2ReF_6$	*1.95*	4	This work

Table 3 Structural parameters obtained from the adjustment of the k^3-EXAFS spectrum of $(NH_4)_2ReF_6$. In italic are the distances found by EXAFS in [16]

	C.N.	R (Å)	σ^2 ($Å^2$)	ΔE_0	Residual
Re-Cl	5.6	2.36(2) *2.36*	0.0019	0.95	2.87
Re-Br	6.1	2.50(2) *2.48*	0.0041	0.22	3.20
Re-I	5.4	2.74(3) *2.73*	0.0052	2.33	2.10

The EXAFS analysis of the $(NH_4)_2ReX_6$ (X = Cl, Br, I) have also been performed (Table 3) and the Re-X distances by EXAFS match well the one previously fund by EXAFS in those salts [16].

In Re fluorides, the Re-F distance depends on the oxidation state of the Re atom, as well as the number of F atoms coordinated to the Re center (Table 2). For ReF_6^n (n = 0, − 1, − 2) species, the Re-F distance increases as the oxidation state decreases; an increase of ~ 0.04 Å is observed between Re(+6) and Re(+5) and an increase of 0.08 Å between Re(+5) and Re(+4). A Re(+3) species containing the ReF_6^{3-} anion has not been reported and the ReF_6^+ cation has not been structurally characterized. For Re fluorides with the Re atom in the same oxidation state, the Re-F distance increases with the number of coordinating ligands; for Re(+6), the average Re-F distance in ReF_8^{2-} is ~ 0.1 Å longer than in ReF_6, while for Re(+7), the average Re-F distance in ReF_8^- is ~ 0.03 Å longer than in ReF_7.

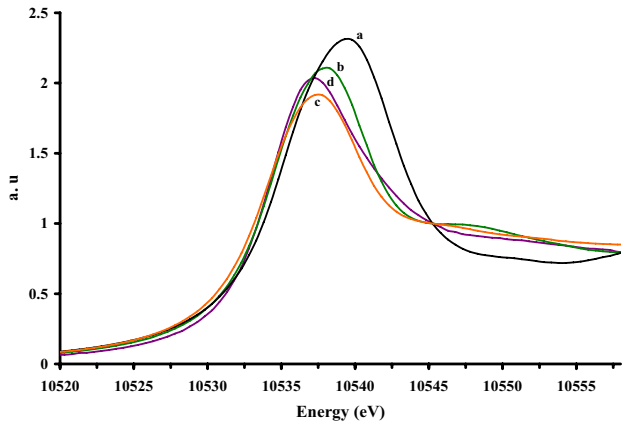

Fig. 2 Normalized L_3-edge spectra of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I). **a** X = F (in black); **b** X = Cl in green; **c** X = Br in orange and d) X = I in purple

Table 4 Position (eV) of the L_3-edge and white line for $(NH_4)_2ReX_6$ (X = F, Cl, Br, I), as well as the calculated Re covalent charge and the magnitude of the crystal field splitting parameter (Δ_0, cm^{-1}) for ReX_6^{2-} (X = F, Cl, Br, I)

Compounds	L_3-edge (eV)	White line position (eV)	Covalent charge	Δ_0 (cm^{-1})
$(NH_4)_2ReF_6$	10535.3	10539.5	+2.87	32,800
$(NH_4)_2ReCl_6$	10534.6	10538.0	+1.76	29,000
$(NH_4)_2ReBr_6$	10534.5	10537.5	+1.48	28,000
$(NH_4)_2ReI_6$	10534.4	10537.1	+1.11	26,000

3.2 XANES analysis

For third row transition metals, the main electronic transition at the L_3-edge is $2p_{3/2} \rightarrow 5d$ and the corresponding absorption peak in the XANES spectra is called the white line. On the XANES spectra, the position of the Re-L_3 absorption edge was determined at the first inflection point of the spectrum by using the maximum of the first derivative, while the position of the white line was taken from the first node of the first derivative.

The Re-L_3 edge XANES spectra of the $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) salts are presented in Fig. 2, and the position of the absorption edge and white line are listed in Table 4. A shift of the absorption edge and white line to higher energy is observed when moving in the series I, Br, Cl and F.

Usually, the position of the white line and absorption edge can be affected by several factors, including the crystal field splitting parameter [28], oxidation state and covalent charge of the absorbing atom, the bonding between the metal and the ligand and finally the bond length and the nature of the ligand.

Earlier XANES studies of Re compounds have focused primarily on the effect of oxidation state on the position of L_3-edge and white line [29, 30]. Previous XANES studies on Re oxides and chlorides have shown that the position of the L_3-edge and white line were affected by the oxidation state of the Re atom. For Re oxides, a difference of ~ 3 eV was observed between the L_3-edge of $(NH_4)ReO_4$ and ReO_2 and a difference of ~ 4 eV between ReO_2 and Re metal. For Re chlorides, a difference of 0.2 eV was observed between the white line of $ReCl_3$ and K_2ReCl_6 and a difference of 0.9 eV between K_2ReCl_6 and $ReCl_5$.

For Re compounds, the only report on the effect of the halogen ligand on the XANES spectra was performed on $Re_2X_8^{2-}$ (X = F, Cl) [31]. In this study, a shift to higher energy of the white line and the edge between $Re_2Cl_8^{2-}$ and $Re_2F_8^{2-}$ was observed, but the numerical value was not reported; it was shown that the XANES spectrum of $Re_2F_8^{2-}$ exhibits a much more intense white line than the one of $Re_2Cl_8^{2-}$. The authors indicated that the main electronic transition of the white line was the $2p_{3/2} \rightarrow \delta^*$ and that this transition was much more polarized for F than for Cl; this larger polarizability was due to the higher electronegativity, shorter metal–ligand distance and better π donating ability of the fluoride than the chloride ligand.

As mentioned earlier, the position of the white line can be influenced by the magnitude of the crystal field splitting parameter Δ_0 or 10 *Dq* [28]. In this study, it was shown that an increase of the crystal field splitting parameter lead to a shift of the white line to higher energy. Previous UV–Visible studies provided the value of Δ_0 in ReX_6^{2-} complexes (X = F, Cl, Br, I) [18]. The value of Δ_0 decreases in the order ReF_6^{2-} (32,800 cm^{-1}) > $ReCl_6^{2-}$ (29,000 cm^{-1}) > $ReBr_6^{2-}$ (28,000 cm^{-1}) > ReI_6^{2-} (26,000 cm^{-1}). Here, the position of the white line correlates reasonably well with the magnitude of Δ in ReX_6^{2-} (Fig. 3).

Concerning the absorption edge, an element to consider to explain the observed shift is the covalent charge (η) which is the charge appearing at the periphery of the atom due to the presence of ligands. Previous studies have shown that complexes with higher covalent charge have their absorption edge shifted to higher energy [32, 33].

The covalent charge depends on factors such as electronegativity, coordination number and valence of the metal and can be calculated using the formula,

$$\eta = ZI \quad (1)$$

where Z is the formal valence of the metal atom and I is the bond ionicity defined as:

$$I = 1 - e^{\left[\left(\frac{1}{4}(\chi^M - \chi^L)\right)^2\right]} \quad (2)$$

where χ_M and χ_L are the electronegativities of the metal and the ligand, respectively [33].

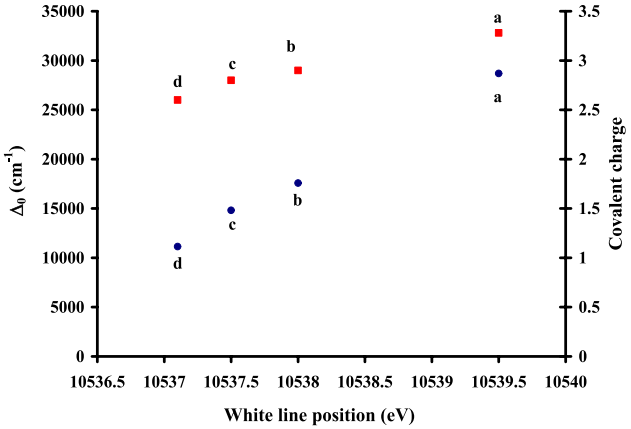

Fig. 3 Representation of the calculated covalent charge (blue dot) and magnitude of Δ_0 (cm^{-1}) (red square) as a function of the white line position (eV) in $(NH_4)_2ReX_6$ (X = F, Cl, Br, I). **a** X = F; **b** X = Cl **c** X = Br and **d** X = I

Using Eq. 1, we calculated the covalent charge carried by the Re atoms in ReX_6^{2-}. The value of the charge follows the order ReF_6^{2-} (+2.87) > $ReCl_6^{2-}$ (+1.76) > $ReBr_6^{2-}$ (+1.48) > ReI_6^{2-} (+1.11). The position of the absorption edge is consistent with the increase of the covalent charge on the metal atoms (Fig. 4) and reflects the electronegativity of the coordinating halogen ligands.

Finally, the effect of the nature of the ligand and the metal–ligand distance on the chemical shifts was investigated by theoretical methods. It is well known that those parameters affect the edge position (Natoli rule) [34]. Here we calculated the XANES spectra of ReX_6^{2-} (X = F, Cl, Br, I) at the Re-L_3 edge using the FDMNES code. The calculation has been performed without taking in account the covalent charge parameter for the input parameters. The calculated XANES spectra (Fig. 5) well reproduce the experimental value: the calculated while line position and maximum as well as the position of the Re-L_3 edge follow well the experimental data.

4 Conclusion

In summary, the XAFS spectra of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) have been measured. Analysis of the EXAFS spectrum of $(NH_4)_2ReF_6$ allows for the first time a determination of the Re-F distance in this salt. The Re-F distance determined by EXAFS is in good agreement with the one determined by x-ray diffraction techniques in other A_2ReF_6 salts (A = K, Rb, Cs), and this study shows that the variation of the Re-F distance follows the order $ReF_6^{2-} > ReF_6^{-} > ReF_6$. In this context, the determination of Re-F distance by EXAFS can be used as a method to evaluate the oxidation state of the Re atom in ReF_6^n (n = 0, -1, -2) species. The XANES studies of $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) indicate that the positions of both the absorption edge and the white line are shifted to higher energy when moving from I^- to F^-. These shifts have been explained in terms of the crystal field splitting parameter and covalent charge carried by the Re atoms. For Re halogen species, our study on $(NH_4)_2ReX_6$ (X = F, Cl, Br, I) also shows that when considering the measure of Re oxidation state using XANES spectroscopy that the nature of the coordinating ligand needs to be considered. In closing, we note that the $(NH_4)_2TcF_6$ salt has been studied by x-ray diffraction and UV–visible spectroscopy but not by XAFS spectroscopy. Current work is focused on the preparation on $(NH_4)_2TcX_6$ (X = F, Cl, Br, I) and their measurement by XAFS spectroscopy.

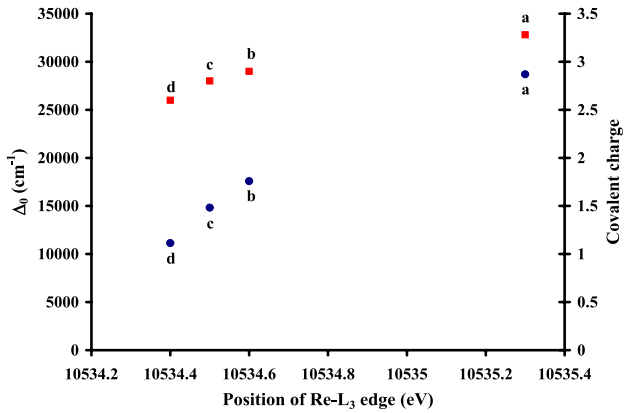

Fig. 4 Representation of the calculated covalent charge (blue dot) and magnitude of Δ_0 (cm^{-1}) (red square) as a function of the L_3-edge position (eV) in $(NH_4)_2ReX_6$ (X = F, Cl, Br, I). **a** X = F; **b** X = Cl **c** X = Br and **d** X = I

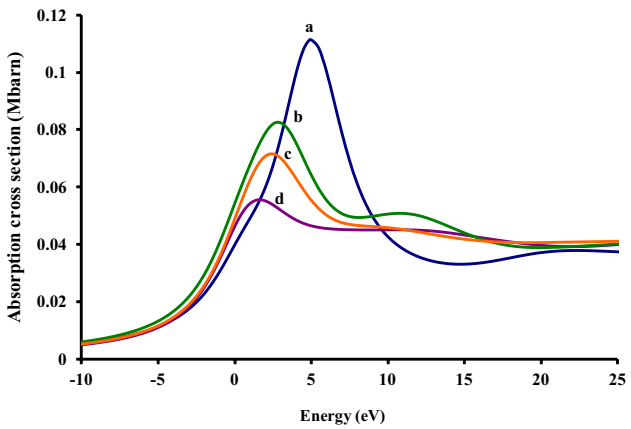

Fig. 5 Calculated XANES spectra at the Re-L_3 edge for the ReX_6^{2-} anions (X = F, Cl, Br, I). **a** X = F (in black); **b** X = Cl in green; **c** X = Br in orange and **d** X = I in purple

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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