Ionic-Liquid-Based Synthesis of Tellurium-Rhenium Carbonyls with Specific Reaction Control

Silke Wolf[a], **Claus Feldmann**[a,*]

[a] *Institut für Anorganische Chemie, Karlsruhe Institute of Technology (KIT), Engesserstrasse 15, D-76131 Karlsruhe, Germany*
**claus.feldmann@kit.edu*

Supplementary Information

Content

Structure analysis of [{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$]

[{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$] was obtained with yellow needles as by-phase of [TeI$_2$Re(CO)$_5$][AlCl$_4$] (**1**) (*see main paper*). The presence of [{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$] was validated by crystal structure analysis. Cation and anion are well-known individually in the literature (e.g., [{Re(CO)$_5$}$_2$I]$^+$ in [{Re(CO)$_5$}$_2$I][AsF$_6$], *see main paper*). Here, the compound was only identified to complete and understand the underlying synthesis of the title compound **1**. Therefore, the structural data of [{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$] are briefly summarized but not discussed in detail. The crystallographic data are shown in Table S1, unit cell and structure of cation are illustrated in Figures S1 and S2).

Table S1. Crystallographic data of [{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$].

Compound	**[{Re(CO)$_5$}$_2$I][Al$_2$Cl$_7$]**
Sum formula	$C_{10}O_{10}Cl_7I_1Al_2Re_2$
Formula weight	1081.51 g mol^{-1}
Crystal system / space group	monoclinic / *P2$_1$/n*
Lattice parameters	*a* = 1094.8(2) pm *b* = 1181.7(2) pm *c* = 1268.7(5) pm *β* = 125.4(1)° V = 1338.0 × 10^6 pm^3
Formula units per cell, Z	2
Density (calculated)	2.684 g cm^{-3}
Absorption correction	numerical
Absorption coefficient	10.998 mm^{-1}
Measurement conditions	Stoe IPDS II diffractometer (Stoe) *λ*(Mo-*K*α) = 71.073 pm, (T = 200 K)
Measurement limits	–14≤ h < 15; -16 ≤ k ≤14 15; -17 ≤ l ≤ 15, 2Θ$_{max}$ = 29.21°
Number of reflections	13039 (11045 independent)
Refinement method	Full-matrix least-squares on F^2
Merging	R_{int} = 0.081
Total number of least squares parameters	146
Largest diff. peak and hole	2.65/ –2.68e × 10^{-6} pm^3
Figures of merit	*R1* = 0.033 [F$_o$ > 4*σ*(F$_o$)] *R1* (all data) = 0.045 wR2 = 0.074 GooF = 0.985

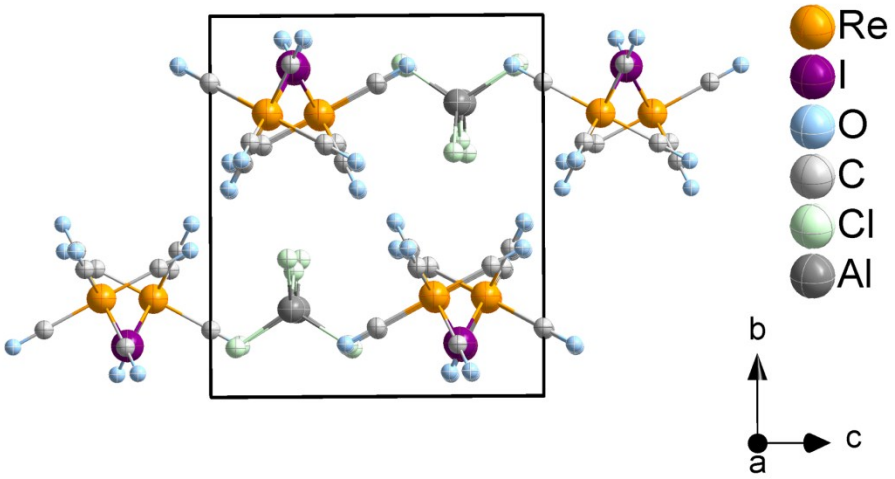

Figure S1. Unit cell of $[\{Re(CO)_5\}_2I][Al_2Cl_7]$.

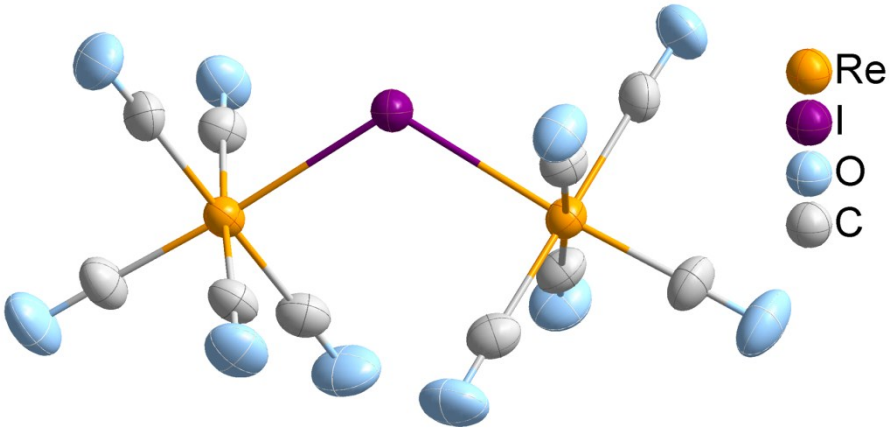

Figure S2. Structure of the $[\{Re(CO)_5\}_2I]^+$ cation in $[\{Re(CO)_5\}_2I][Al_2Cl_7]$.