Synthesis and Characterization of Novel Re(BIAN)$(CO)_3$Cl Derivatives Including the First Example of a Water-soluble Tricarbonyl Rhenium(I) Complex with Bis(imino)acenaphthene Ligands

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Supporting Information

Crystallographic Data of $3 \cdot (H_2O)_x$

Refinement of $\mathbf{3} \cdot (H_2O)_x$

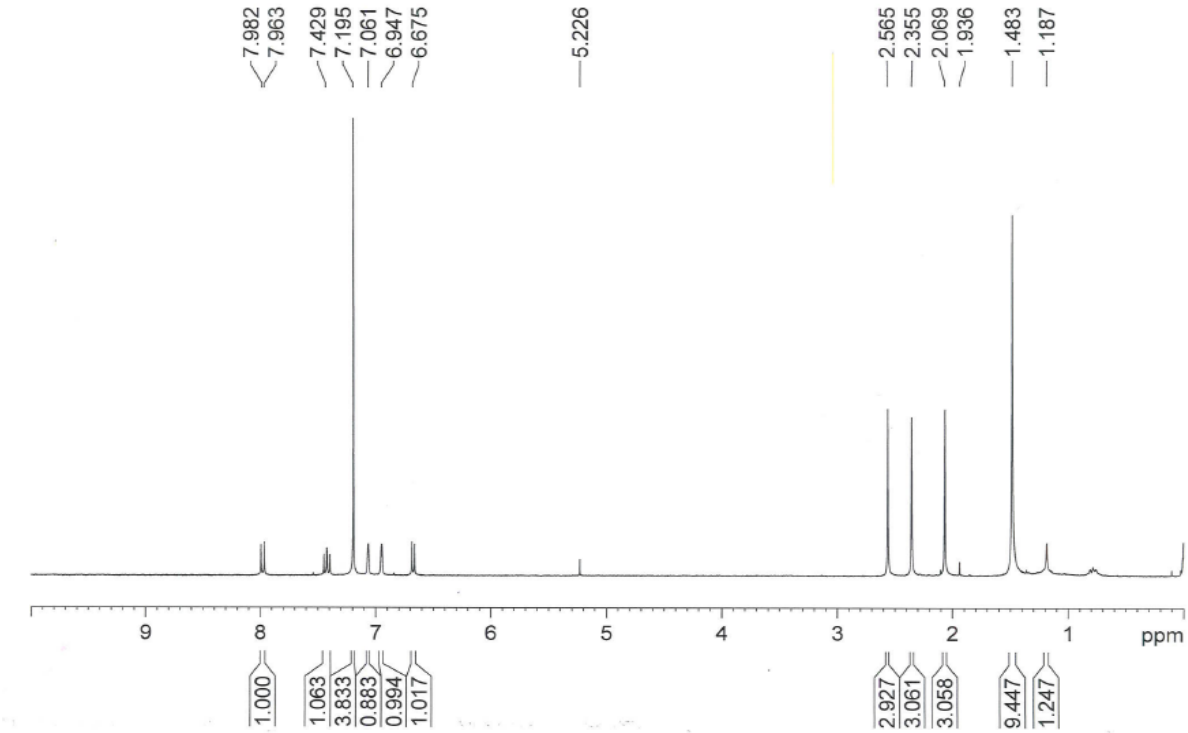
The structure could be solved but the refinement was hampered by the under-occupied positions of the coordinating water molecules (the positions of the oxygen atoms of the water molecule are refined with an occupancy of 75 %). The unusual displacement parameters suggest a potential problem in the determination of the unit cell. However, all attempts to refine the structure in different unit cells result in unstable refinement procedures. Therefore, the structure was eventually refined in the highest symmetric unit cell yielding good *R* values but very disappointing displacement factors. Therefore, the structural data are presented here as preliminary results for information and as a connectivity proof of the complex. Most crystallographic problems alerted in the checkcif routine are related to the described peculiarity of the investigated crystals.

Table S1. Crystal structure data for **3** · $(H_2O)_x$.

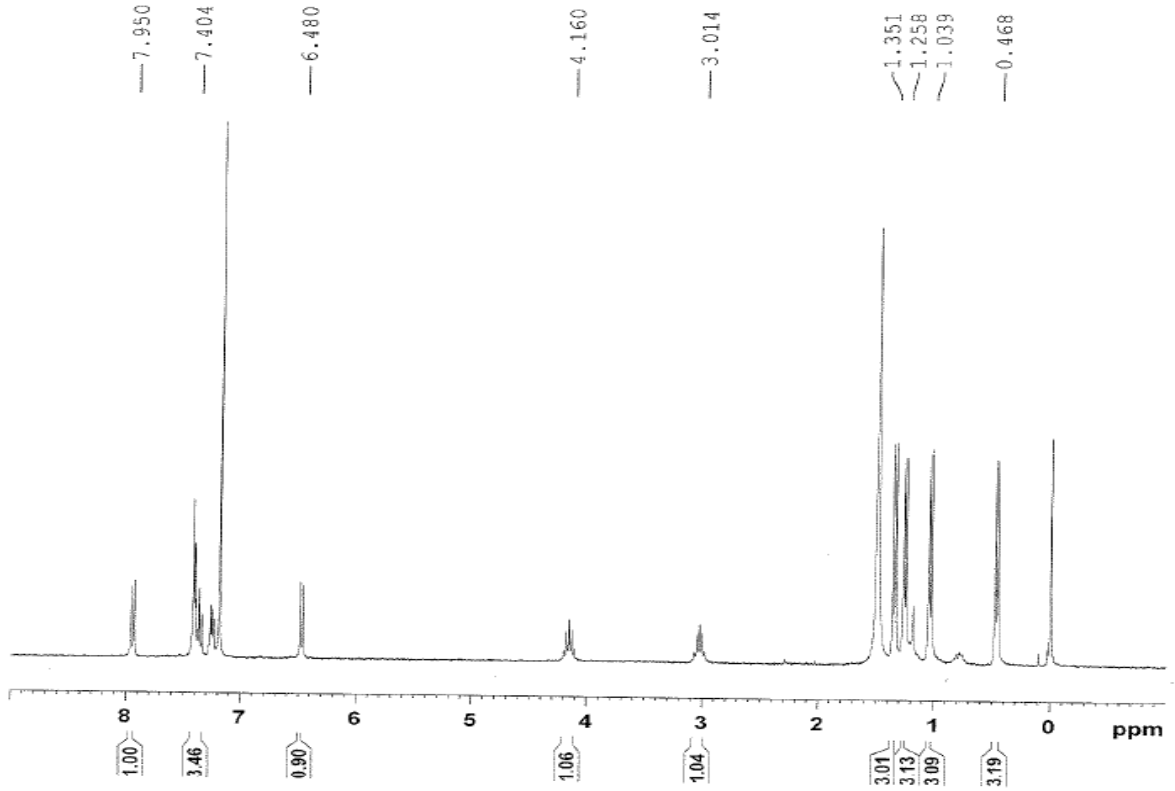
Compound	**3** · $(H_2O)_x$
Empirical formula	$C_{156}H_{152}Cl_4N_8Na_8O_{45}Re_4S_8$
Molecular weight, g mol^{-1}	4185.86
Size, mm^3	0.35 × 0.24 × 0.13
Crystal system	monoclinic
Space group	$P2_1/n$
a, Å	10.218(2)
b, Å	17.966(3)
c, Å	25.104(4)
β, deg	96.00(1)
V, Å^3	4583(1)
$\rho_{calcd.}$, g cm^{-3}	1.52
Z	1
μ(MoK_α), mm^1	2.9
T, K	230
θ range, deg	2.3–15.0
Measured reflections	27501
Independent reflections	1918
Reflections with $I > 2\ \sigma(I)$	1673
Restraints/ refined parameters	0/504
Absorption correction	multi-scan
T_{min} / T_{max}	0.42 / 0.71
$R1$ [$F^2 > 2\ \sigma(F^2)$]	0.034
$wR2$(F^2)	0.072
$\Delta\rho_{fin}$ (max / min), e Å^{-3}	0.39 / −0.28
CCDC number	973349

Spectroscopic Data

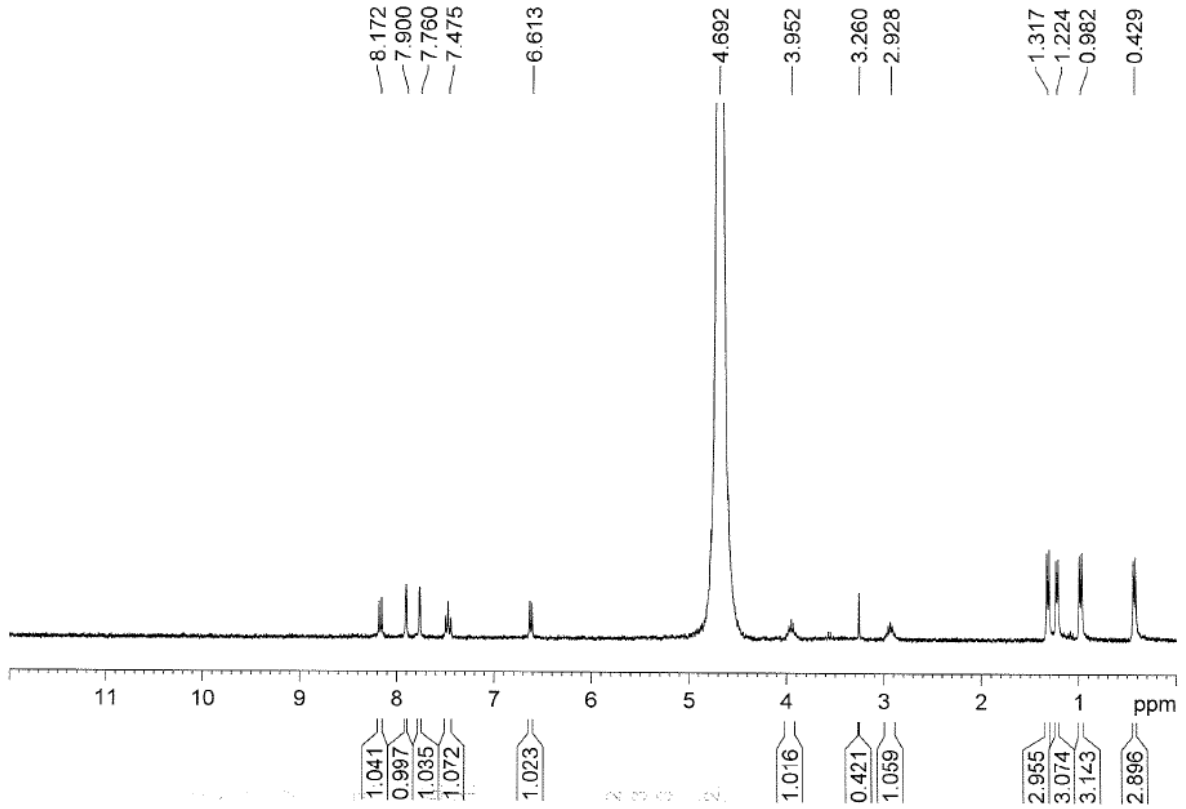
^{1}H NMR spectrum of complex **1** in $CDCl_3$.



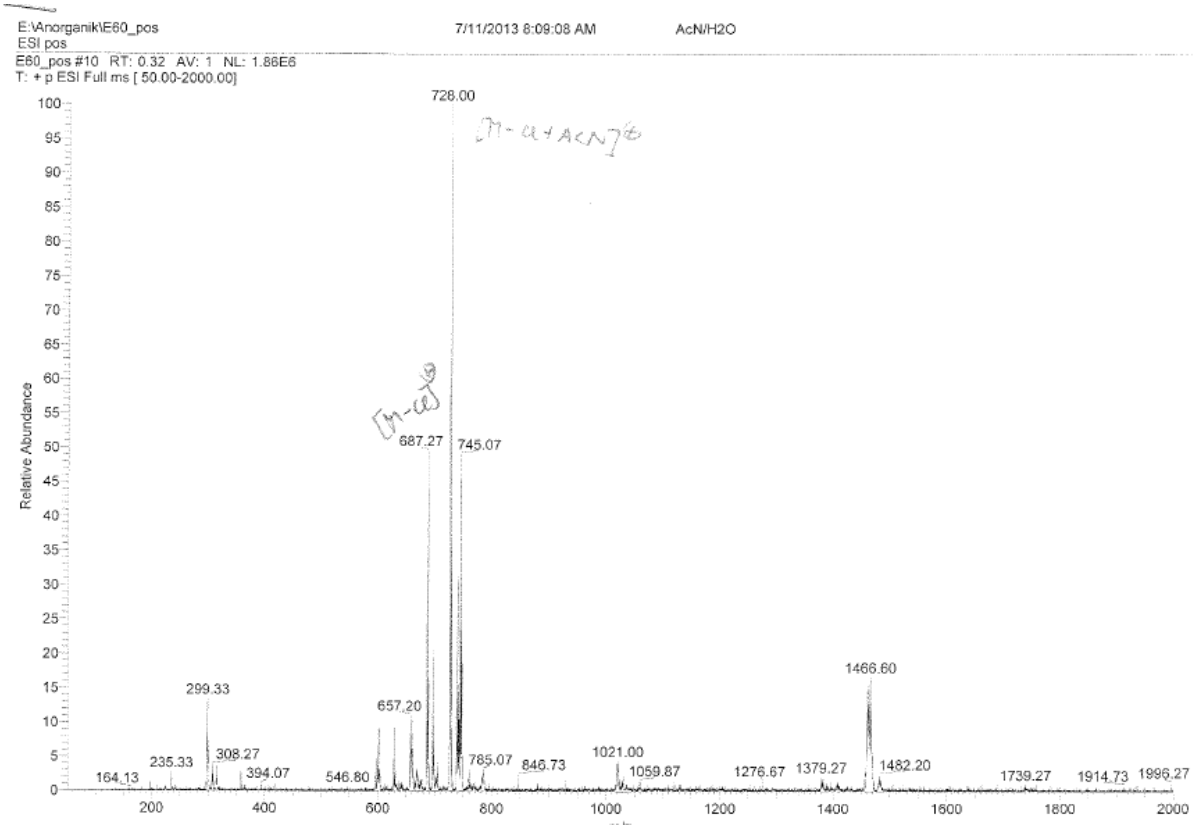
^{1}H NMR spectrum of complex **2** in $CDCl_3$.



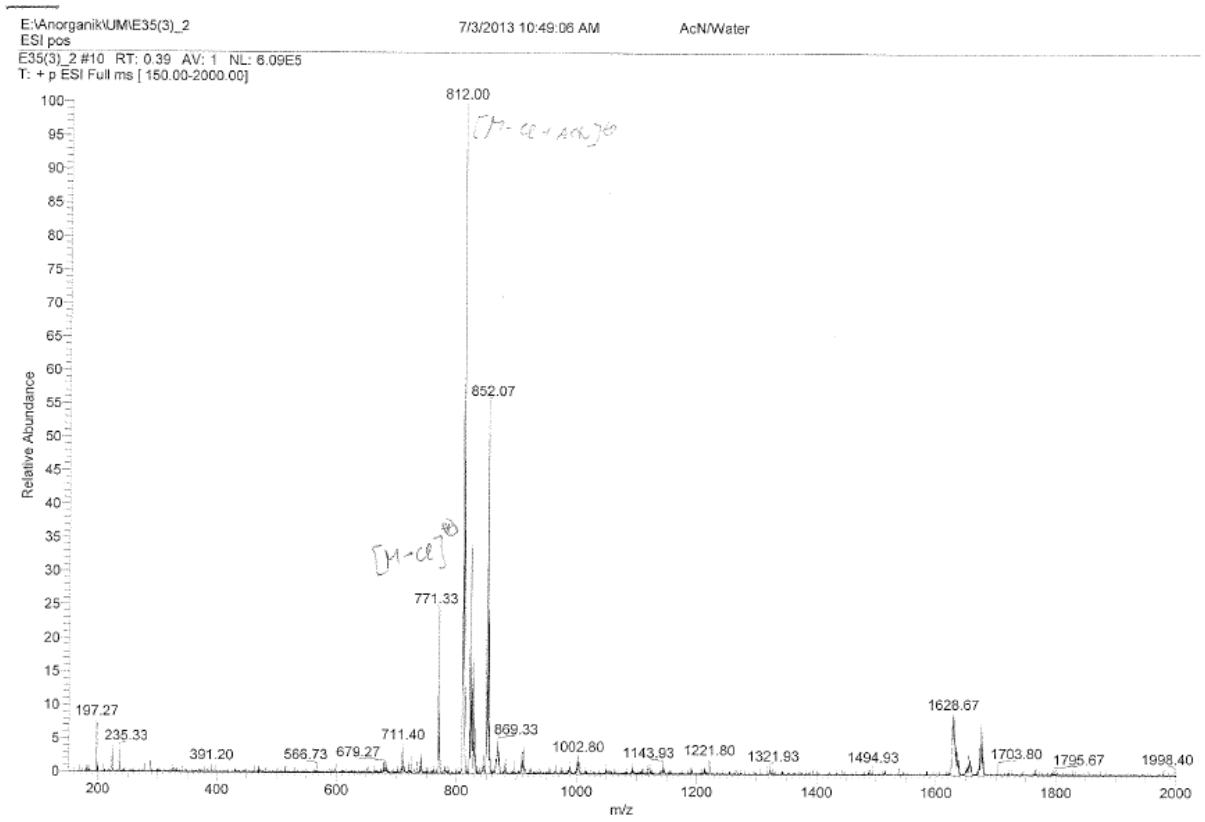
^{1}H NMR spectrum of complex **3** in D_2O.



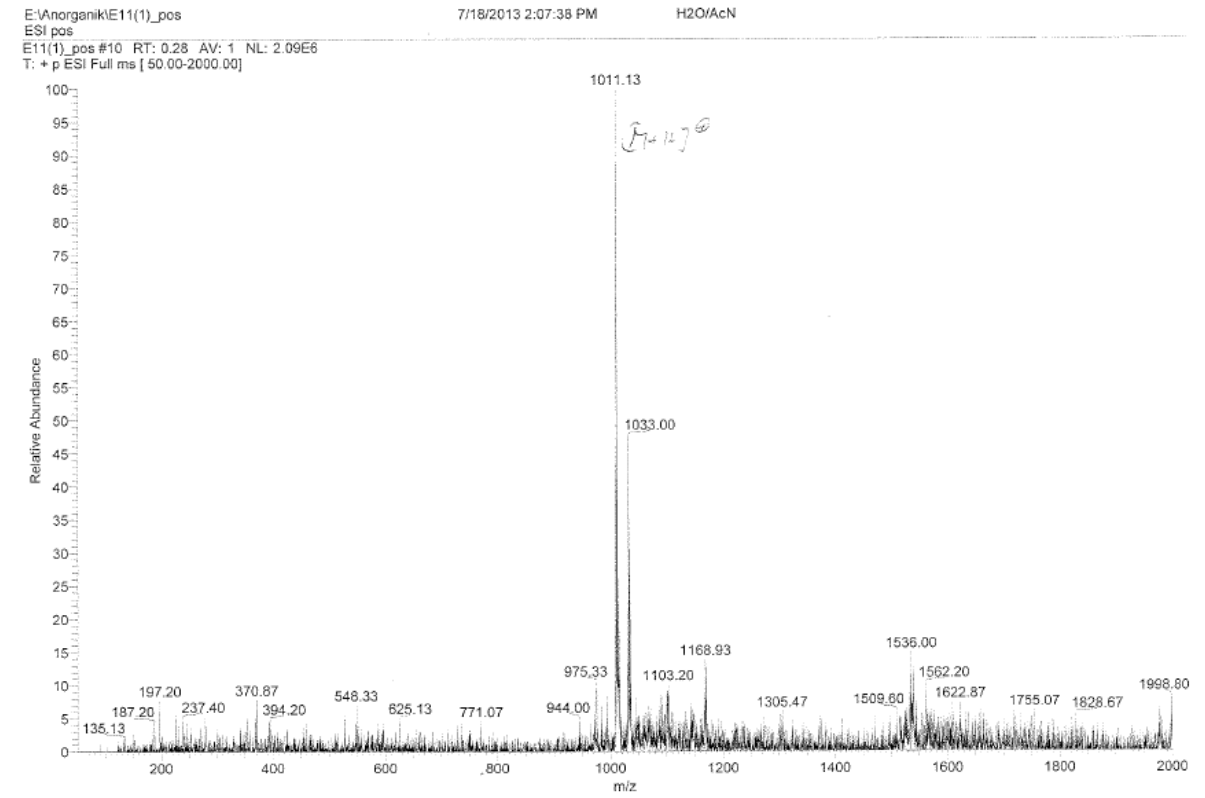
Positive ESI mass spectrum of complex **1**.



Positive ESI mass spectrum of complex **2**.

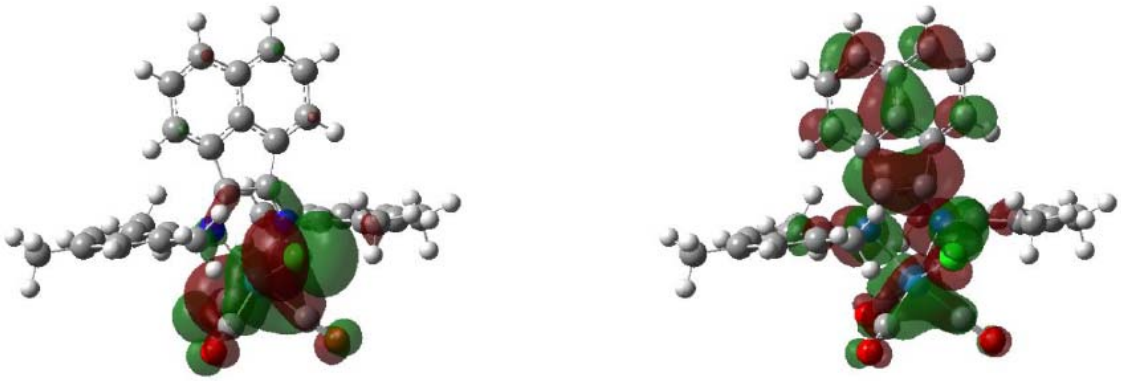


Positive ESI mass spectrum of complex **3**.

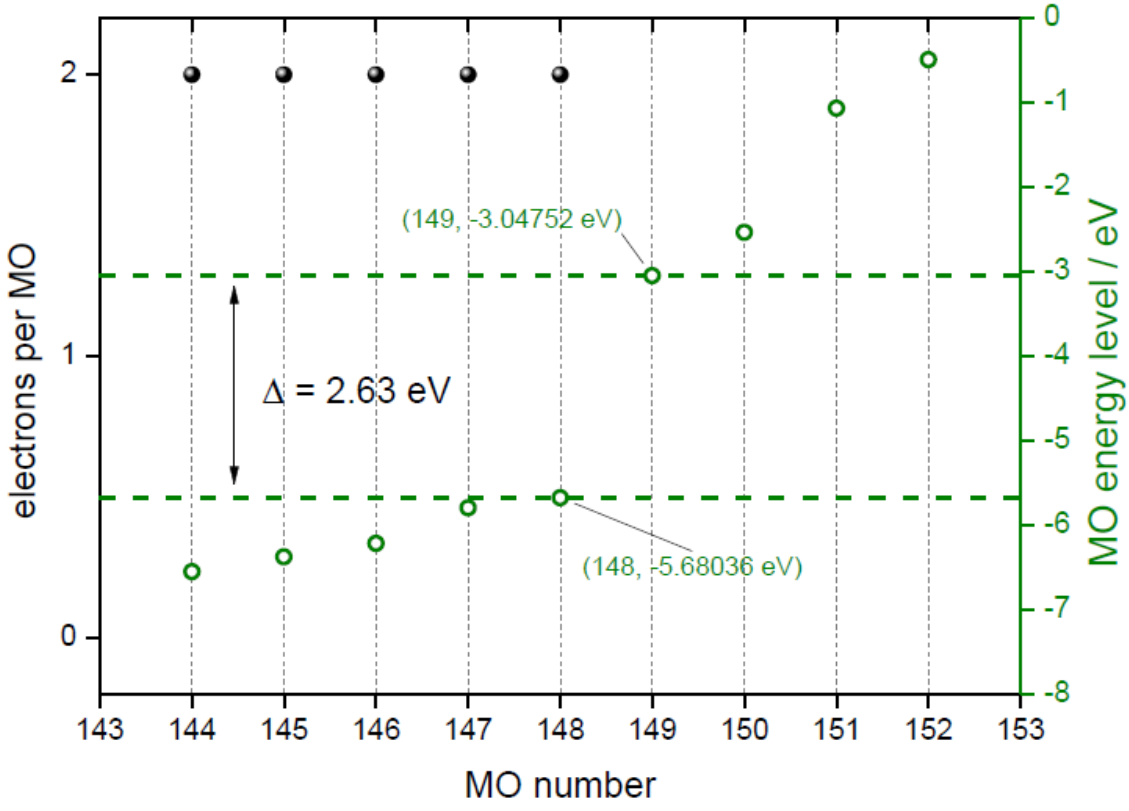


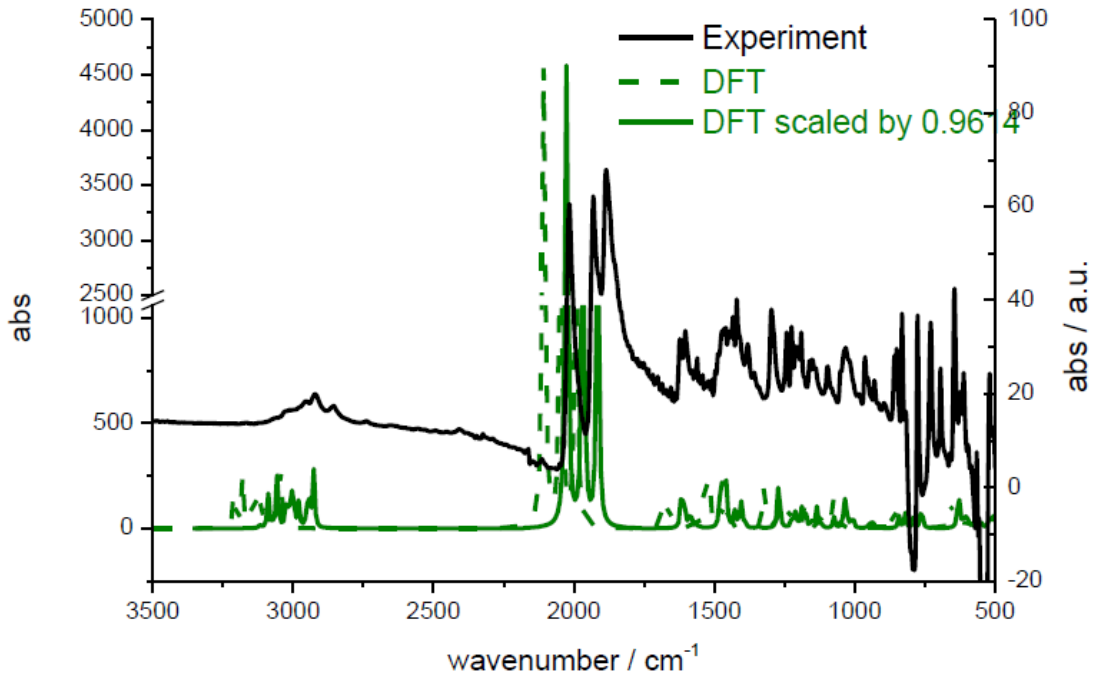
Computational Data

DFT results for complex **1**.



HOMO LUMO

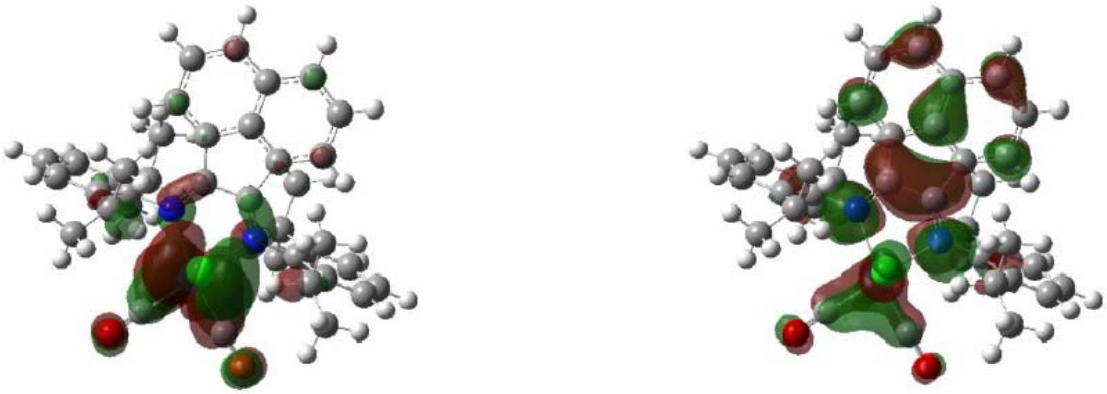




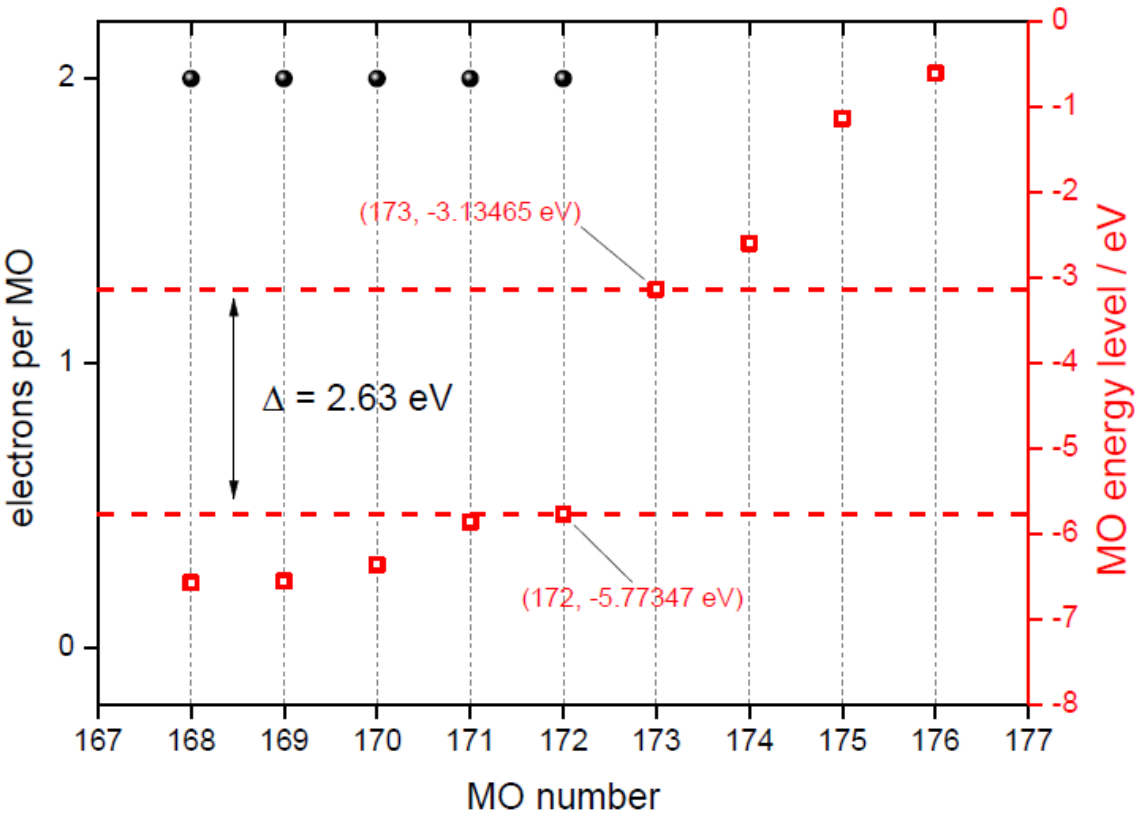
IR data have been scaled by a constant factor of 0.9614 according to:

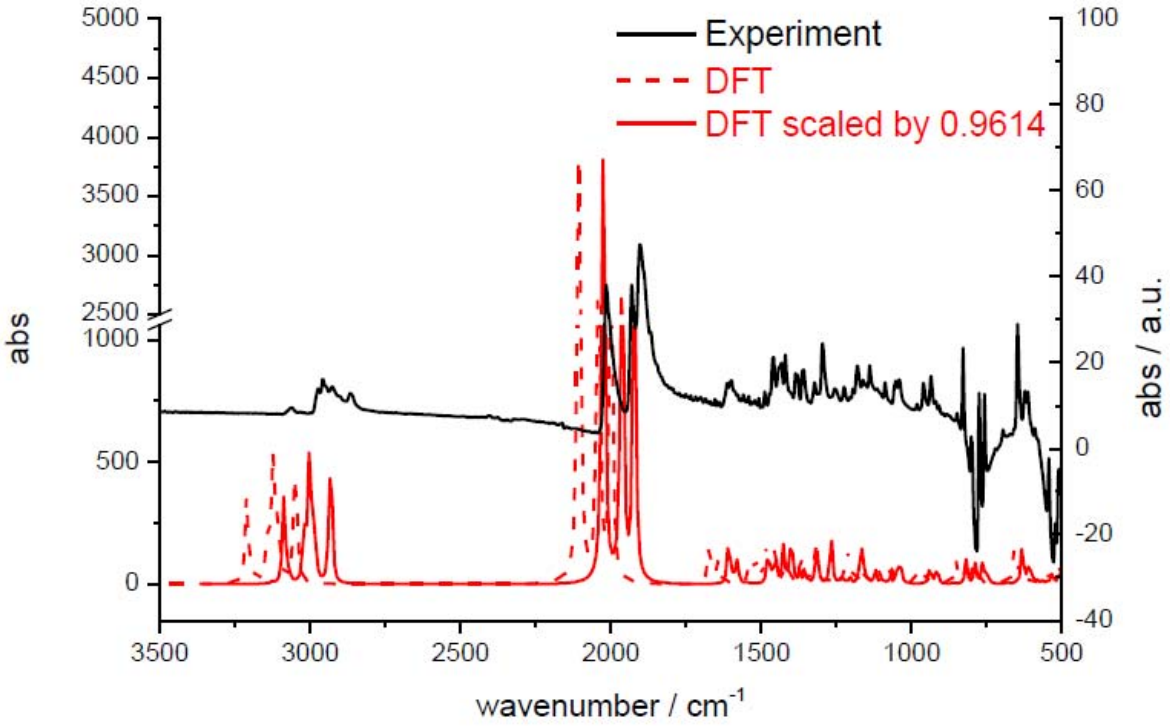
Scott, A. P. & Radom, L. Harmonic Vibrational Frequencies: An Evaluation of Hartree−Fock, Møller−Plesset, Quadratic Configuration Interaction, Density Functional Theory, and Semiempirical Scale Factors. *J. Phys. Chem.* **100,** 16502–16513 (1996).

DFT results for complex **2**.



HOMO LUMO





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