

Supporting Information

Remote deprotometallation-iodolysis of *N,N*-diisopropyl-2-trimethylsilylferrocenecarboxamide: a new route towards 1,1'-disubstituted ferrocenes

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Crystallography

For compound *rac-2*, the X-ray diffraction data were collected using D8 VENTURE Bruker AXS diffractometer at the temperature given in the crystal data. The samples were studied with monochromatized Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). The structure was solved by dual-space algorithm using the *SHELXT* program,¹ and then refined with full-matrix least-square methods based on F^2 (*SHELXL*).² All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions and treated as riding on their parent atom with constrained thermal parameters. The molecular diagram was generated by MERCURY (version 3.9).

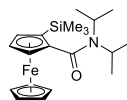
Crystal data for *rac-2*. C₂₀H₃₁FeNOSi, $M = 385.40$, $T = 150 \text{ K}$; Triclinic $P -1$ (I.T.#2), $a = 7.2963(8)$, $b = 10.1529(10)$, $c = 14.3227(17) \text{ \AA}$, $\alpha = 85.787(4)$, $\beta = 88.895(4)$, $\gamma = 72.136(4)^\circ$, $V = 1007.12(19) \text{ \AA}^3$, $Z = 2$, $d = 1.271 \text{ g.cm}^{-3}$, $\mu = 0.814 \text{ mm}^{-1}$. A final refinement on F^2 with 4603 unique intensities and 224 parameters converged at $\omega R(F^2) = 0.1174$ ($R(F) = 0.0423$) for 4202 observed reflections with $I > 2\sigma(I)$. CCDC 2004798.



Figure 1. Molecular structure of compound *rac-2* (thermal ellipsoids shown at the 30% probability level). The two Cp ring are almost coplanar (angle between the Cp ring plans: 1.4°) and are in a staggered conformation (torsion angle C1-Cg1...Cg1'-C1': 9.2° , Cg1 and Cg1' being the centroids of the C1-C5 and C6-C10 rings, respectively). The C=O bond is almost perpendicular to the C1-C5 ring (torsion angle C2-C1-C-O: 87.6°).

Variable temperature ^1H NMR spectra of compound *rac-2*

Spectra were recorded in DMSO-d^6 , at 298 K (FID 10), 308 K (FID 11), 318 K (FID 12), 328 K (FID 13), 338 K (FID 14), 348 K (FID 16), 358 K (FID 18), 368 K (FID 19), 378 K (FID 20) and 383 K (FID 21). Spectra were recorded after 5 min of equilibration for each temperature.



Coalescence of the CH_3 signals is already visible at 298 K while coalescence of the CH signals starts around 338 K.

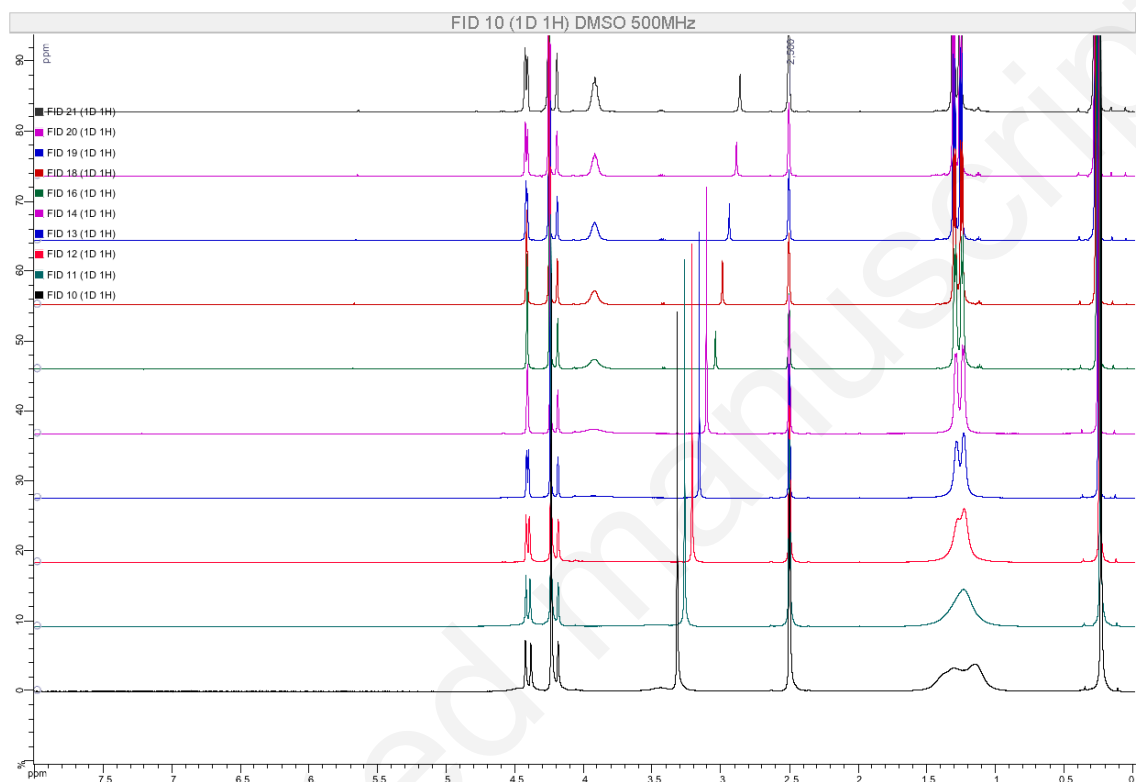
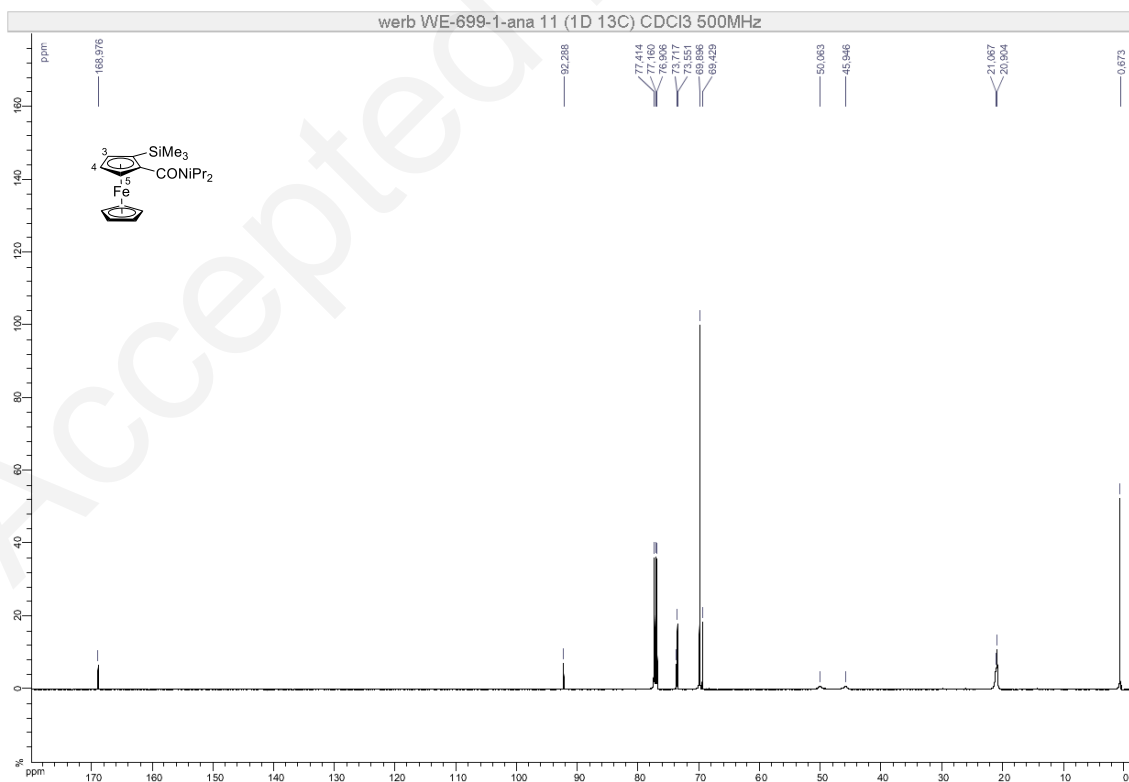
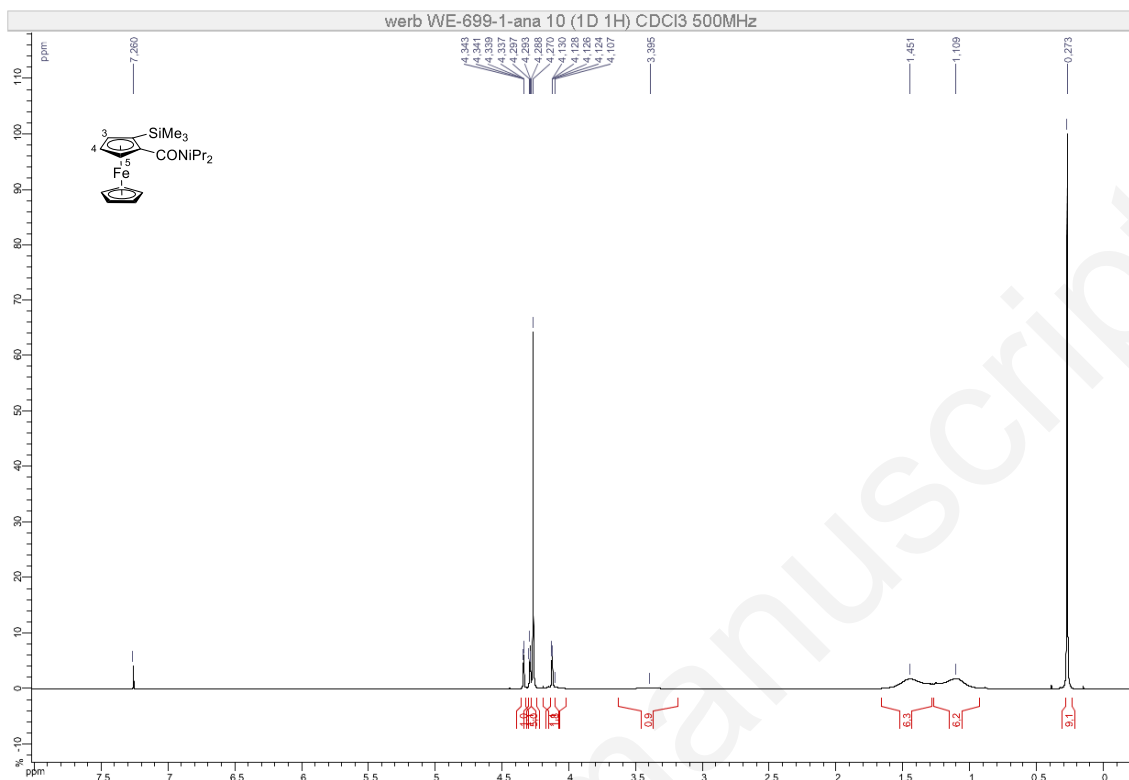
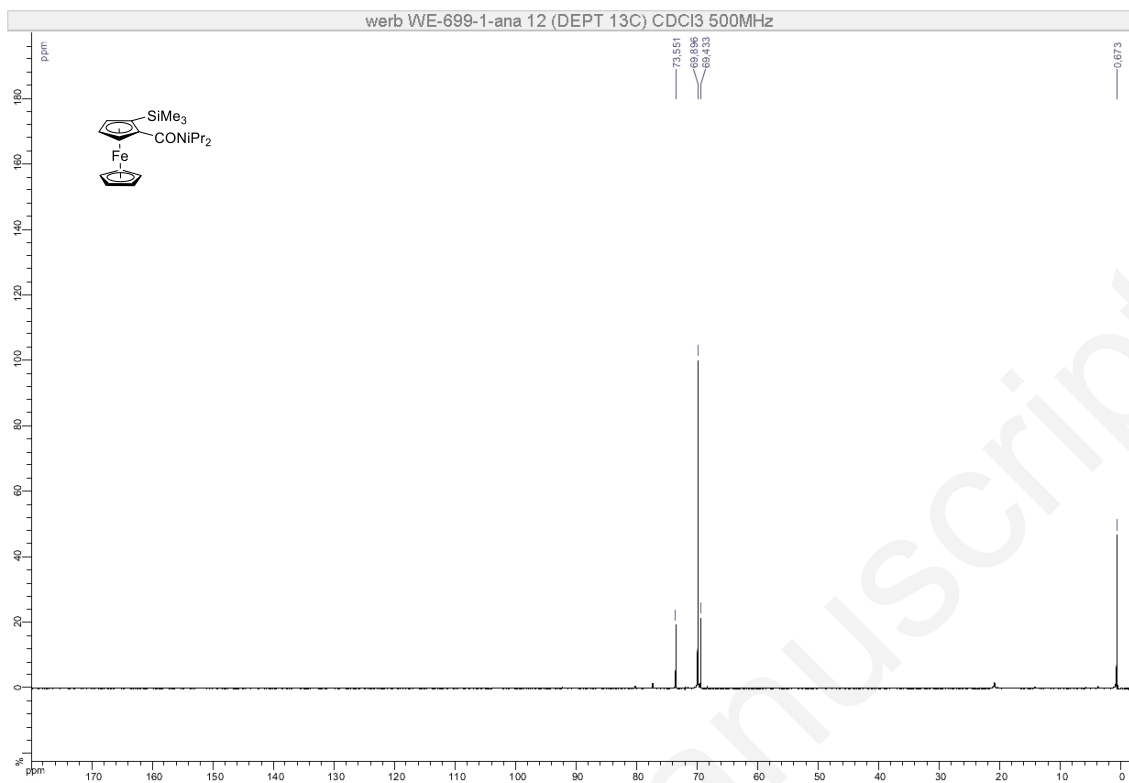


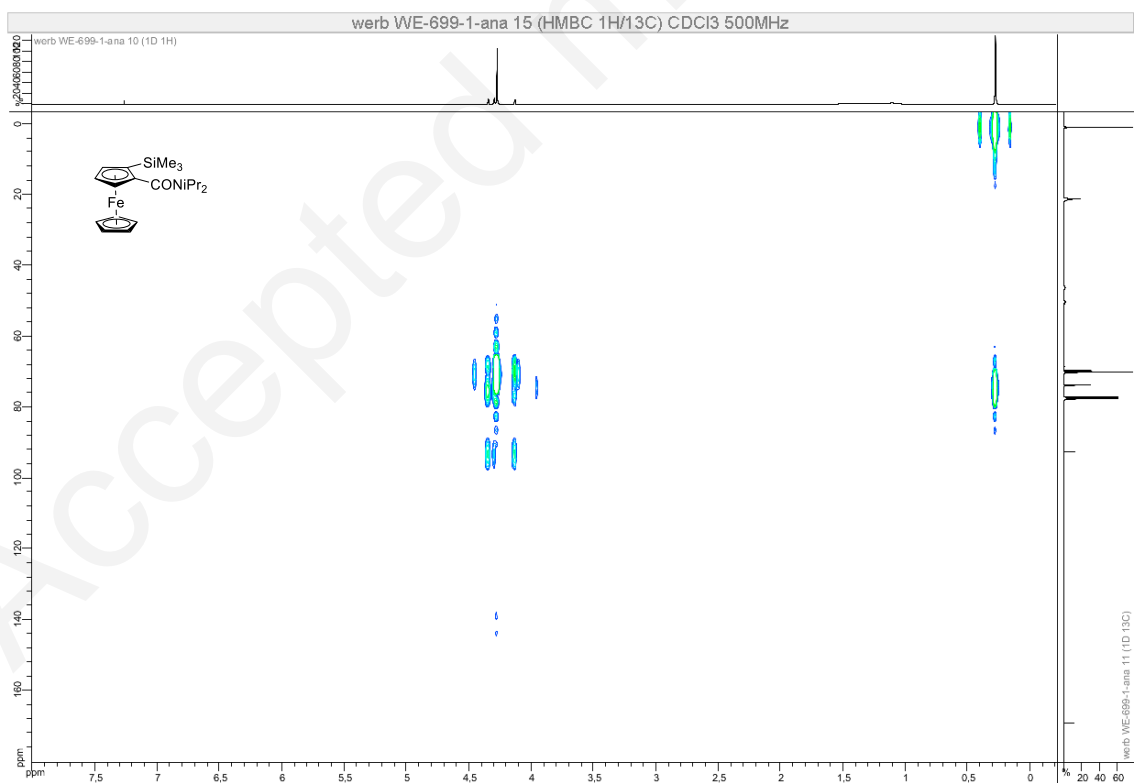
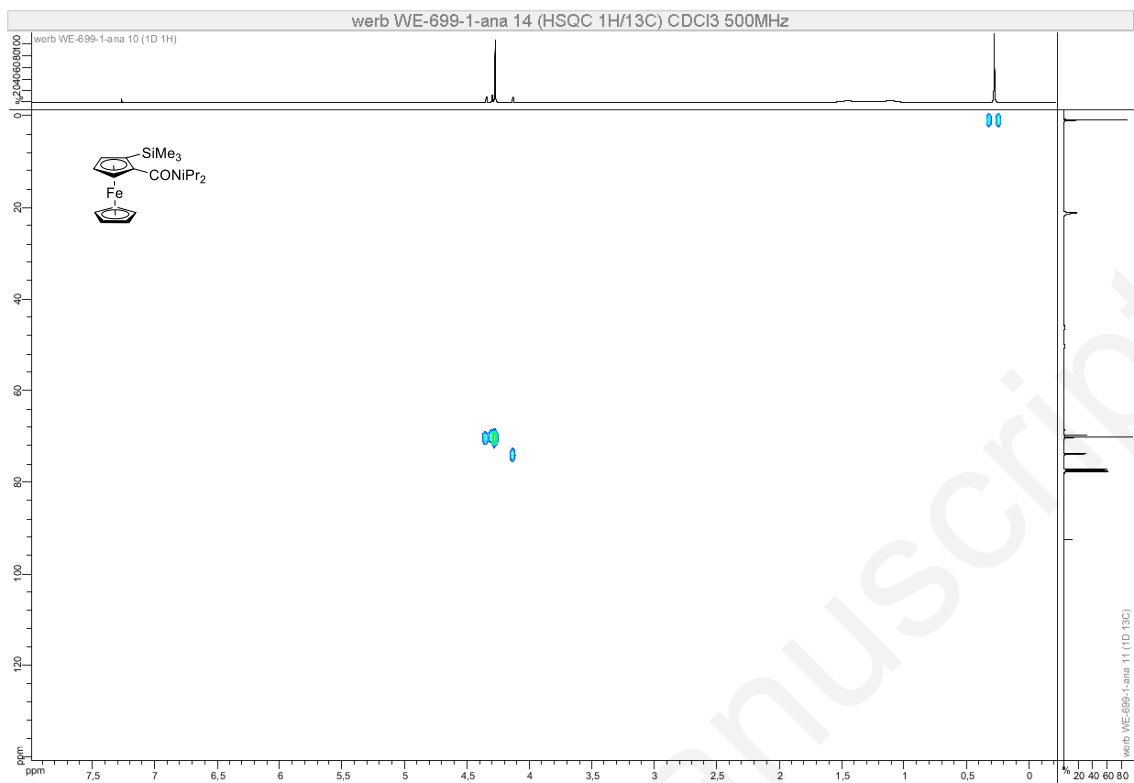
Figure 2. Bottom to top: FID 10 to FID 21.

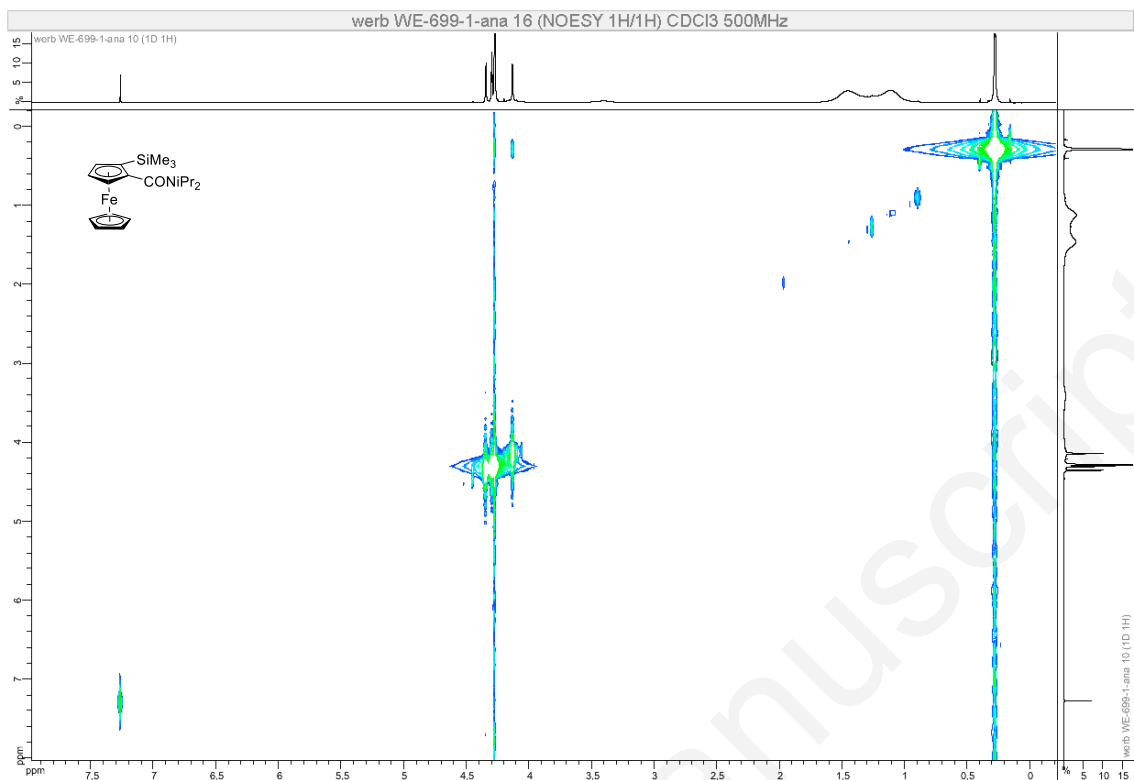
NMR Spectra

Compound *rac-2*

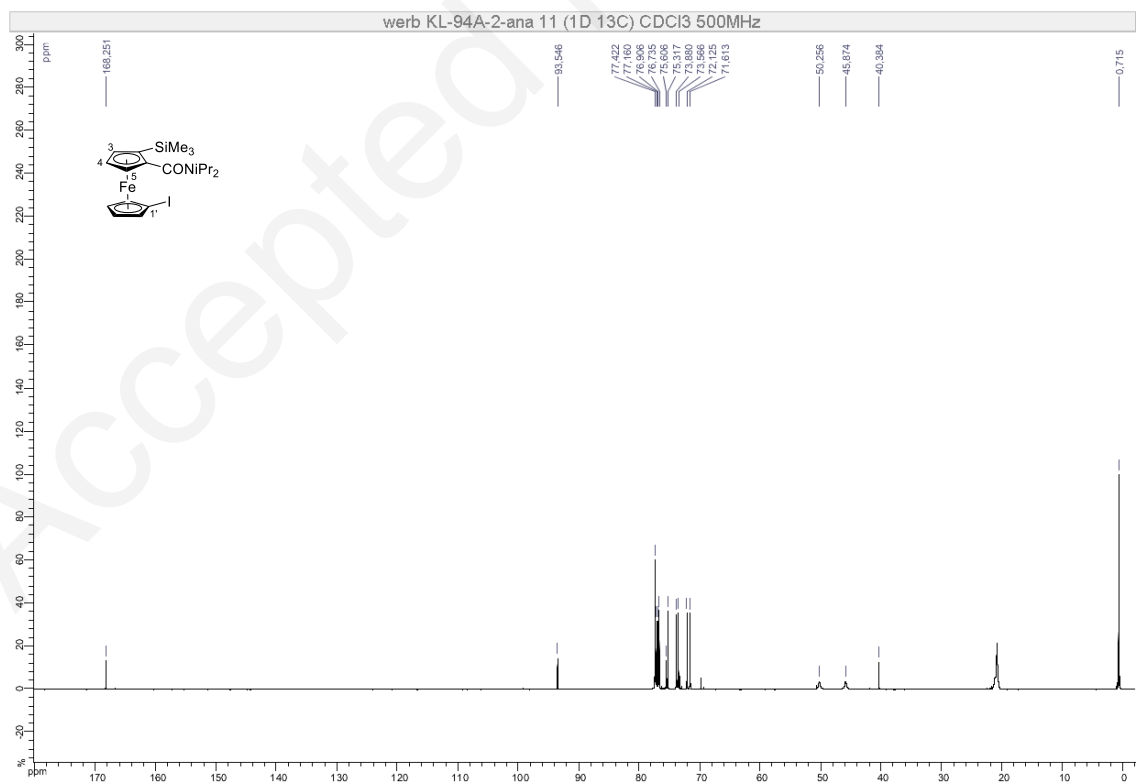
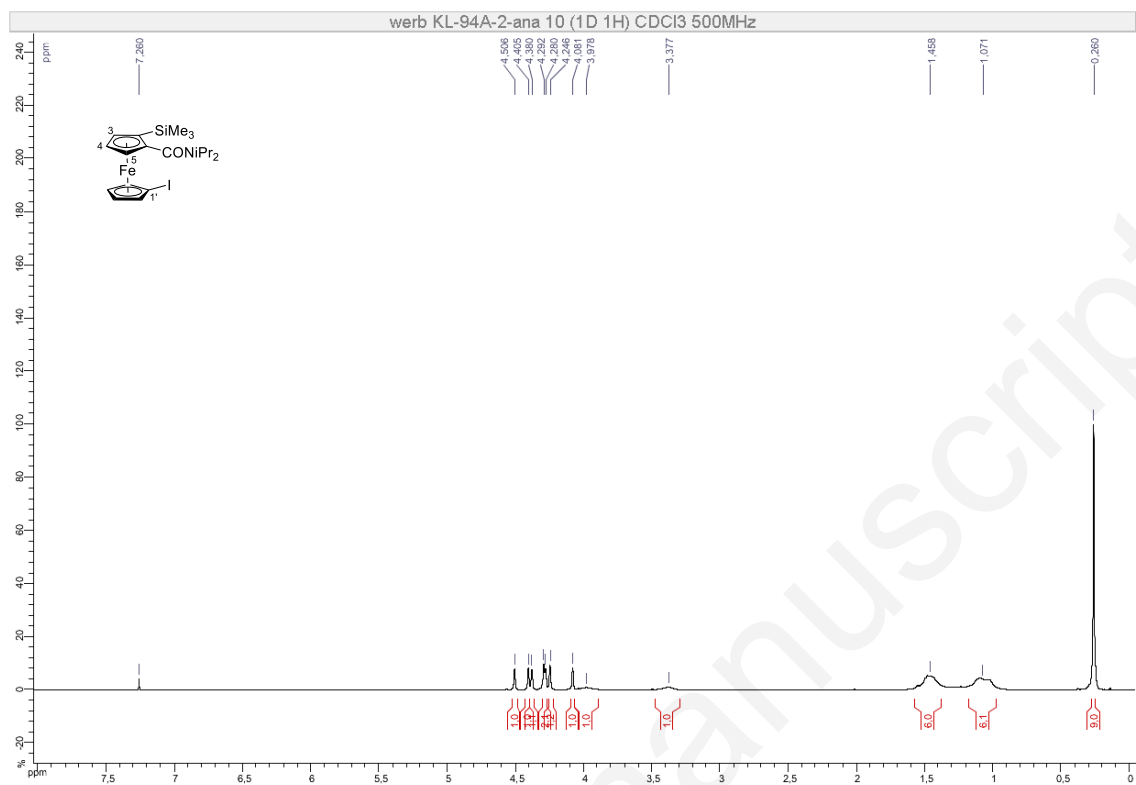


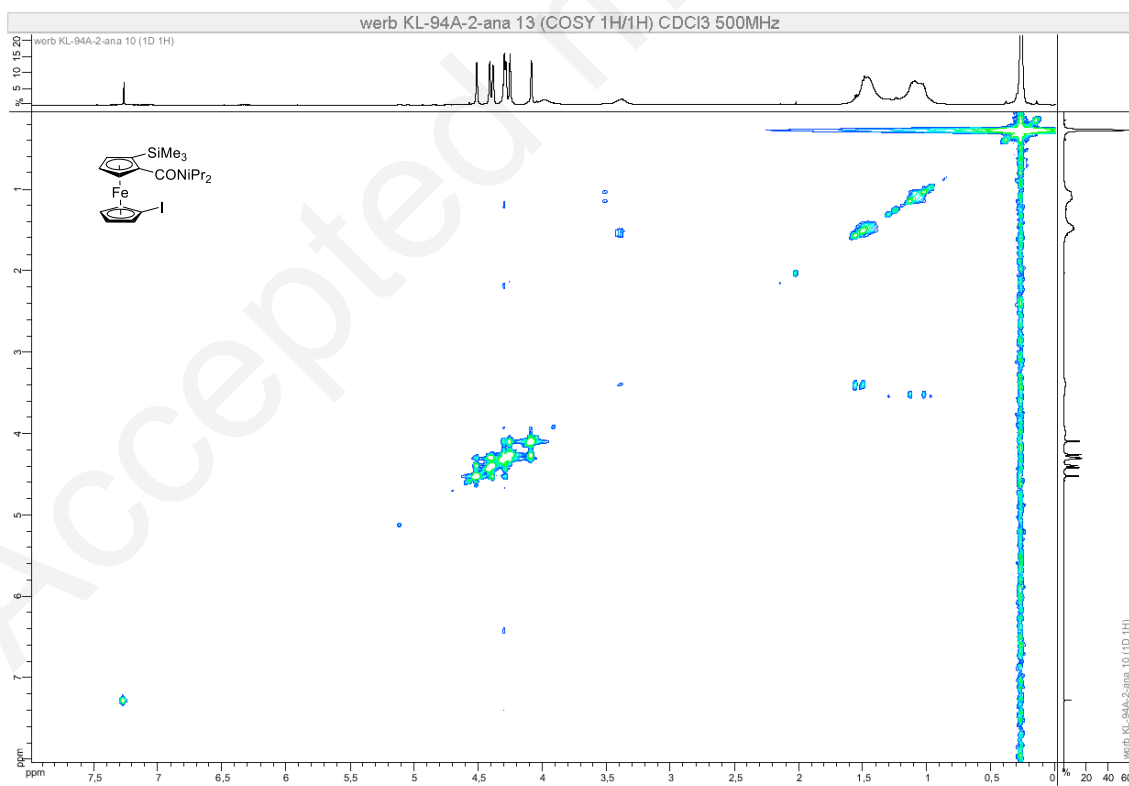
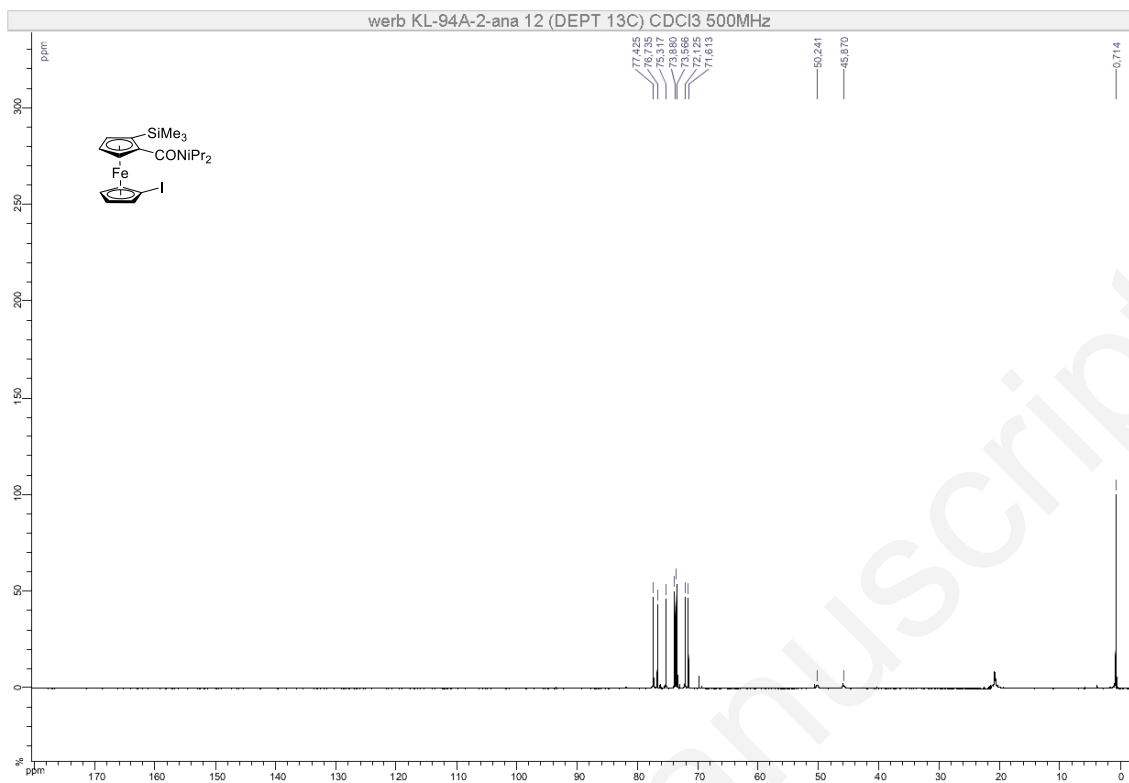


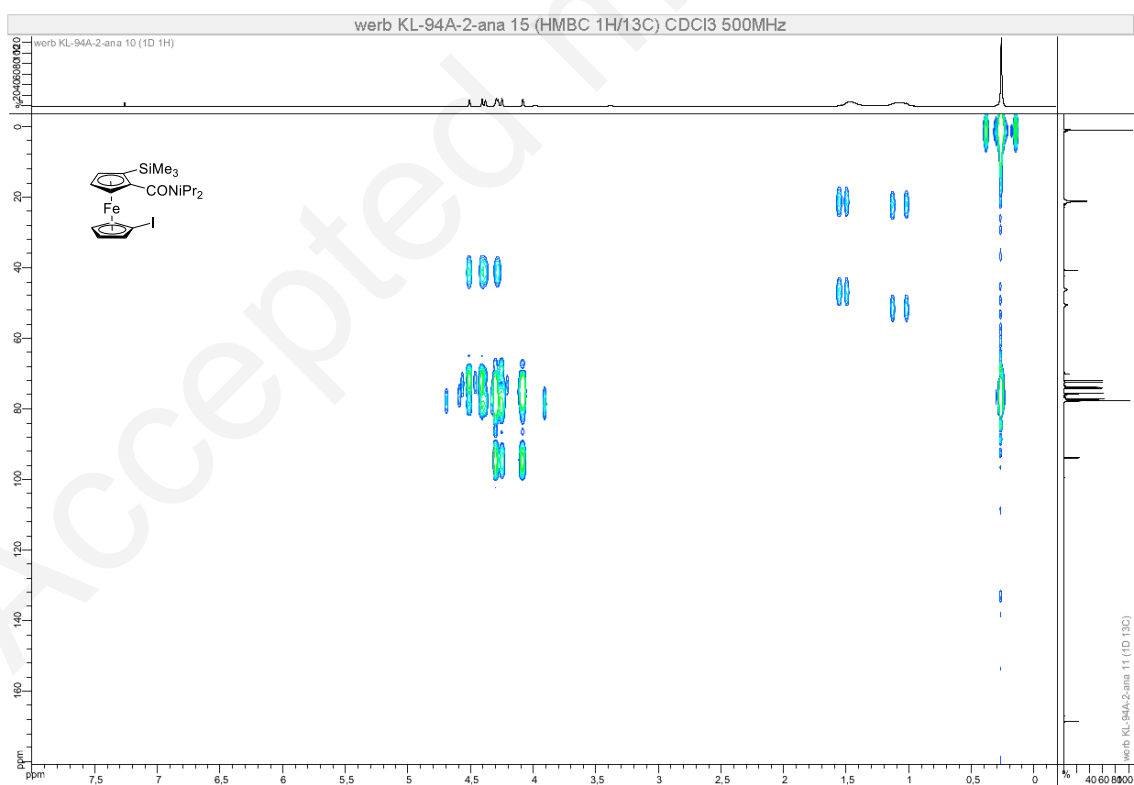


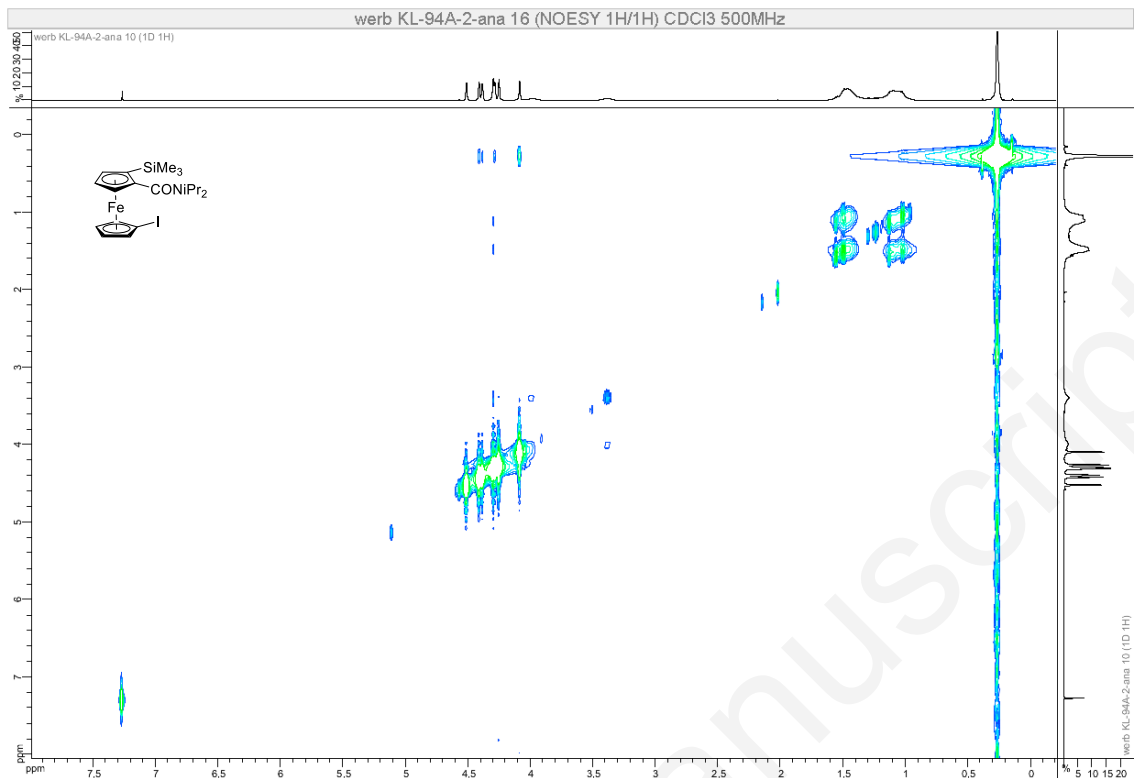


Compound *rac-3*

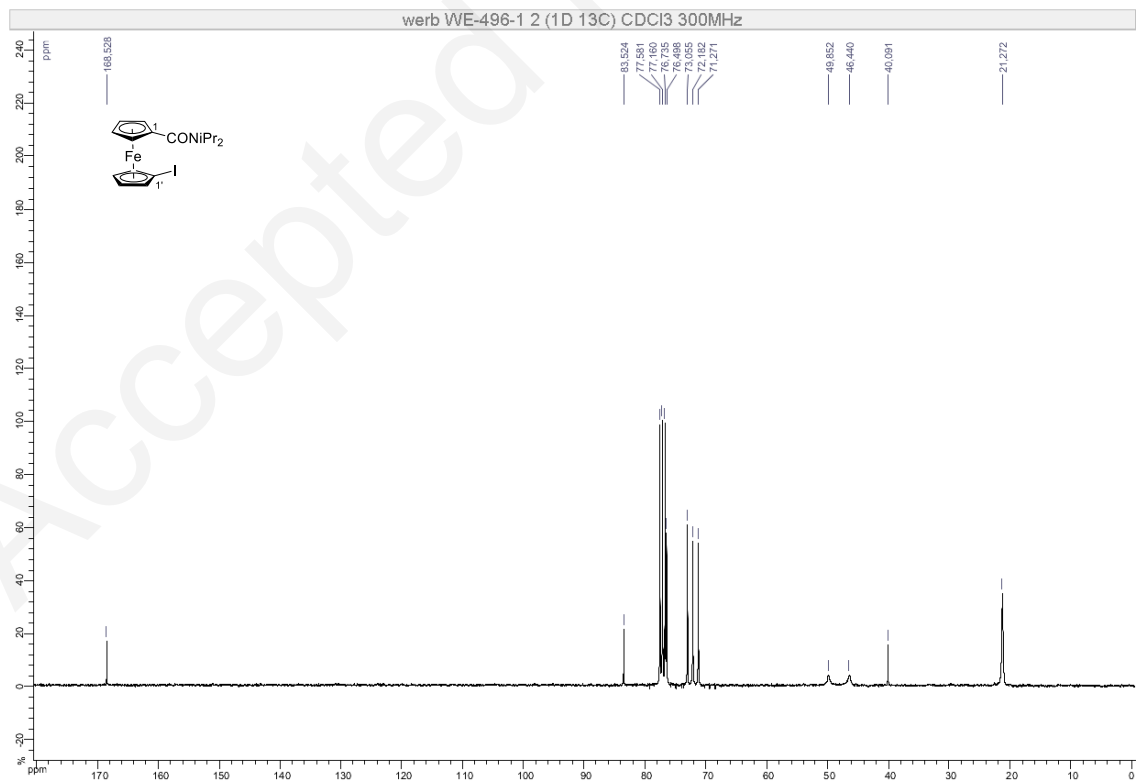
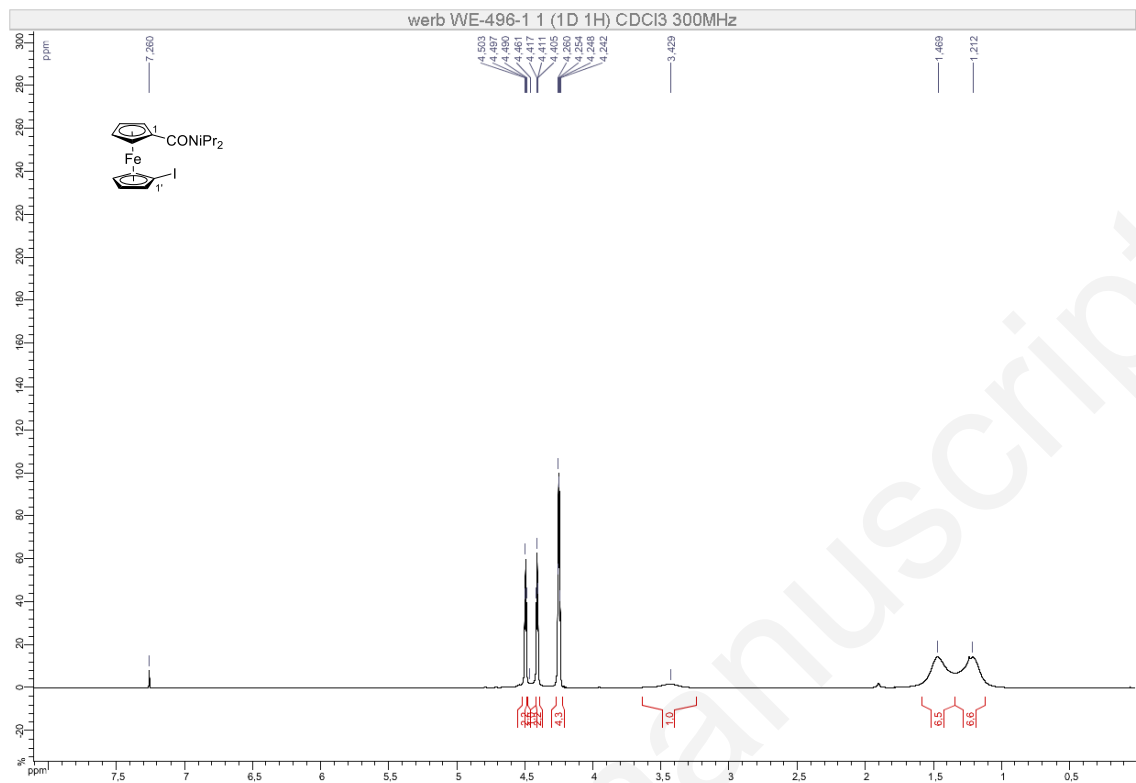


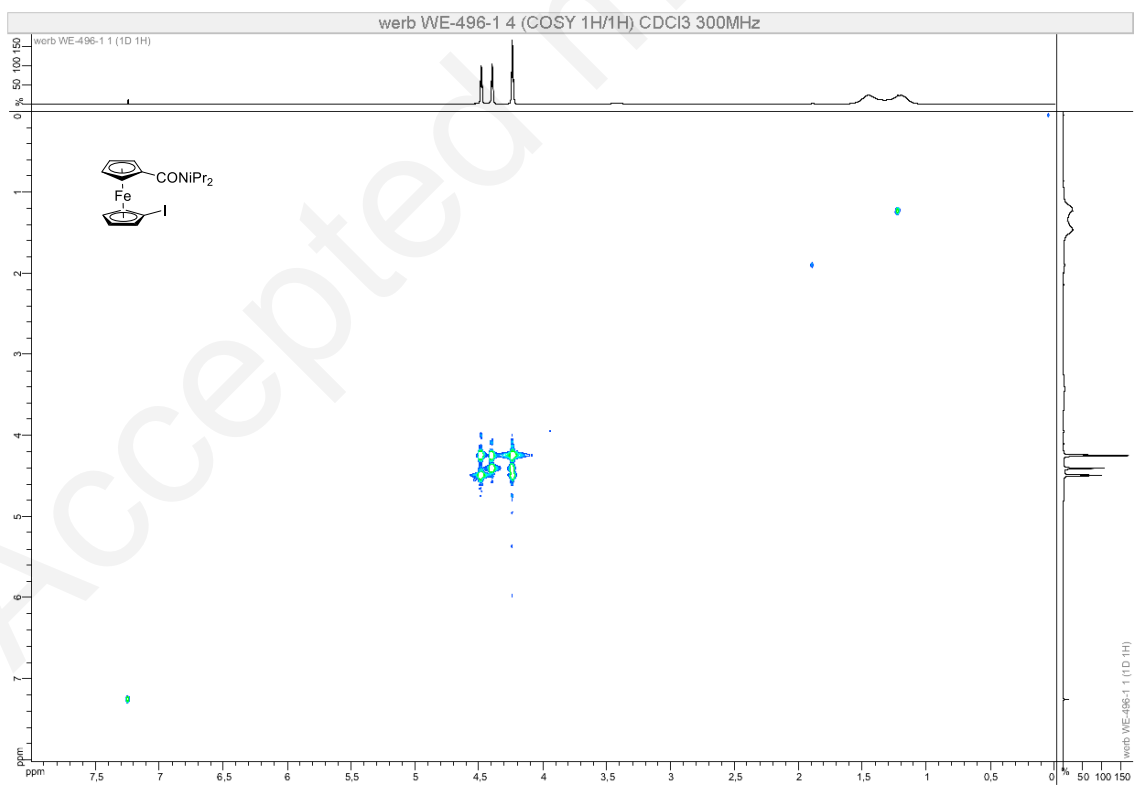
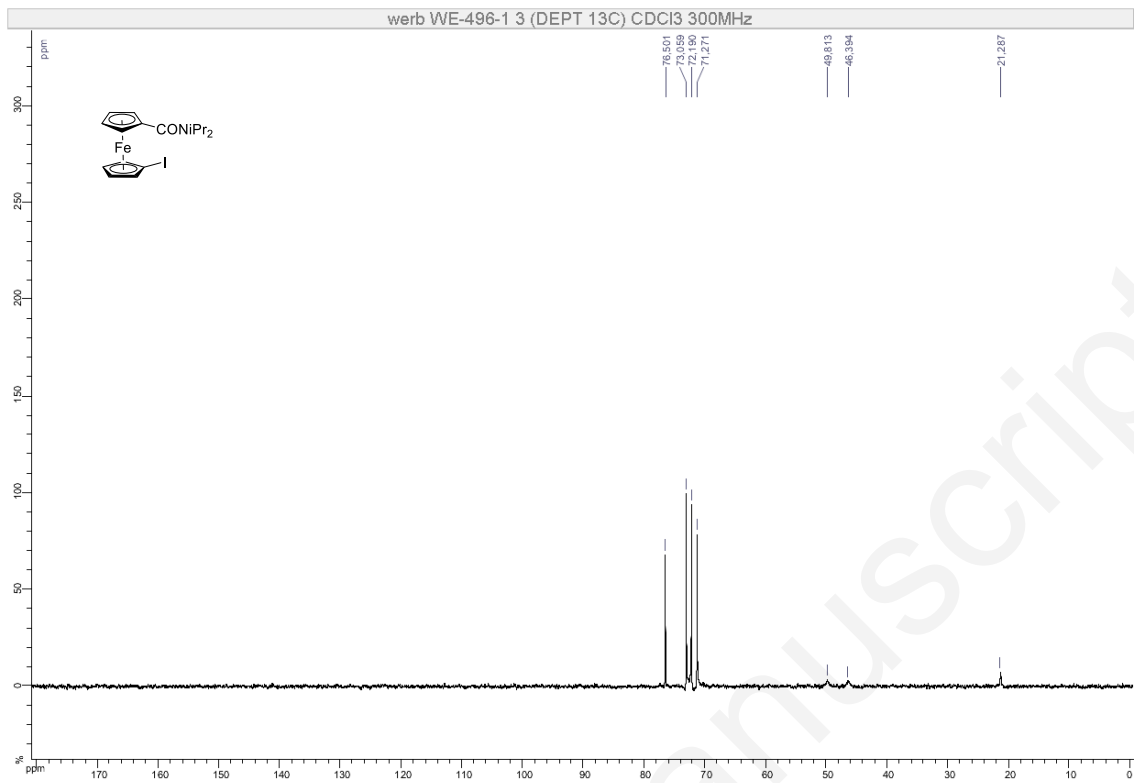


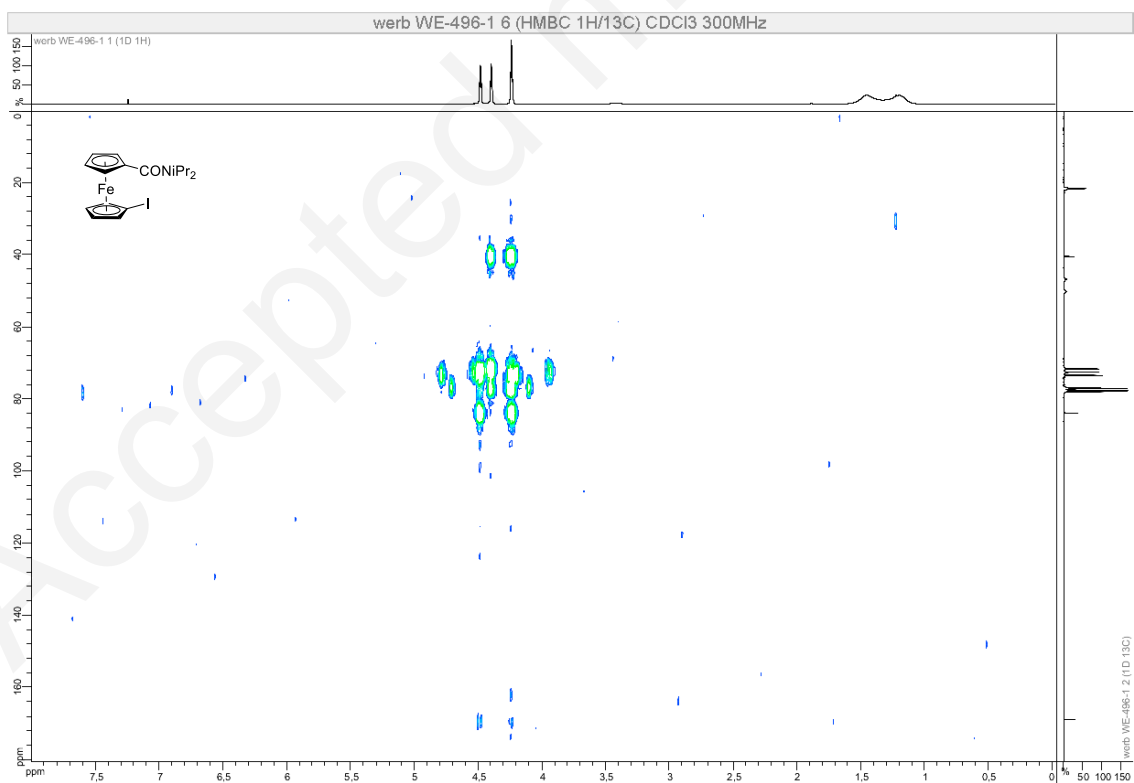
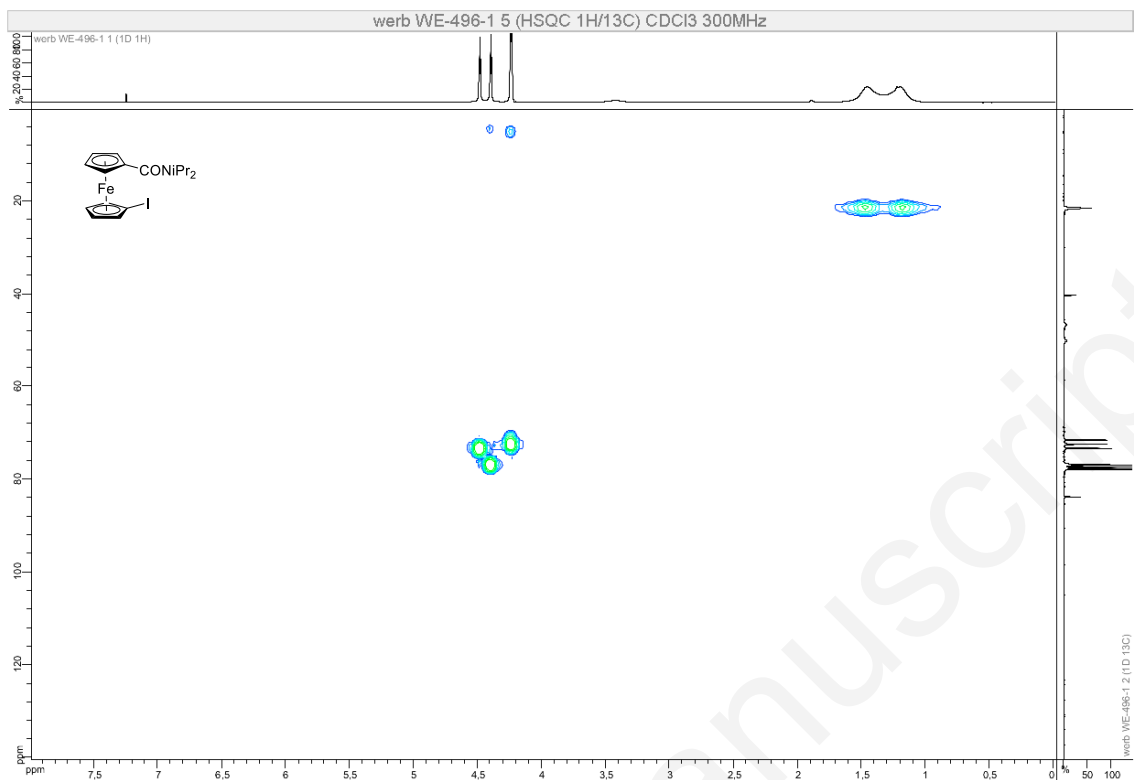




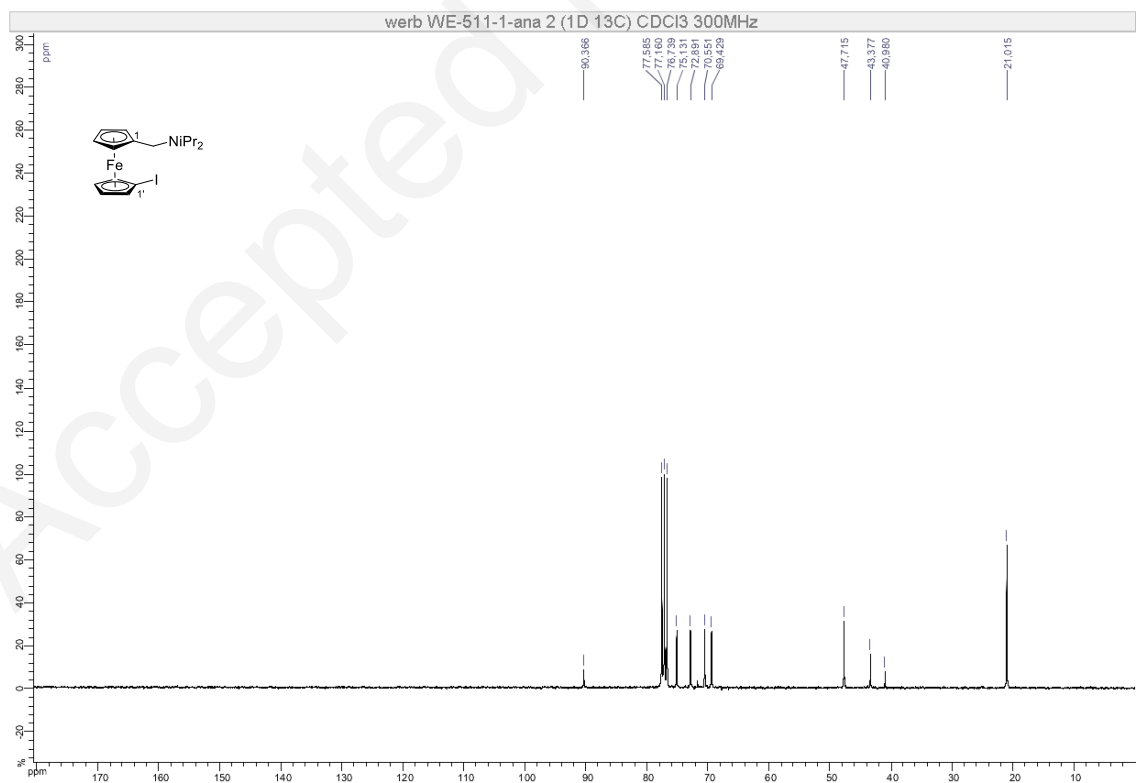
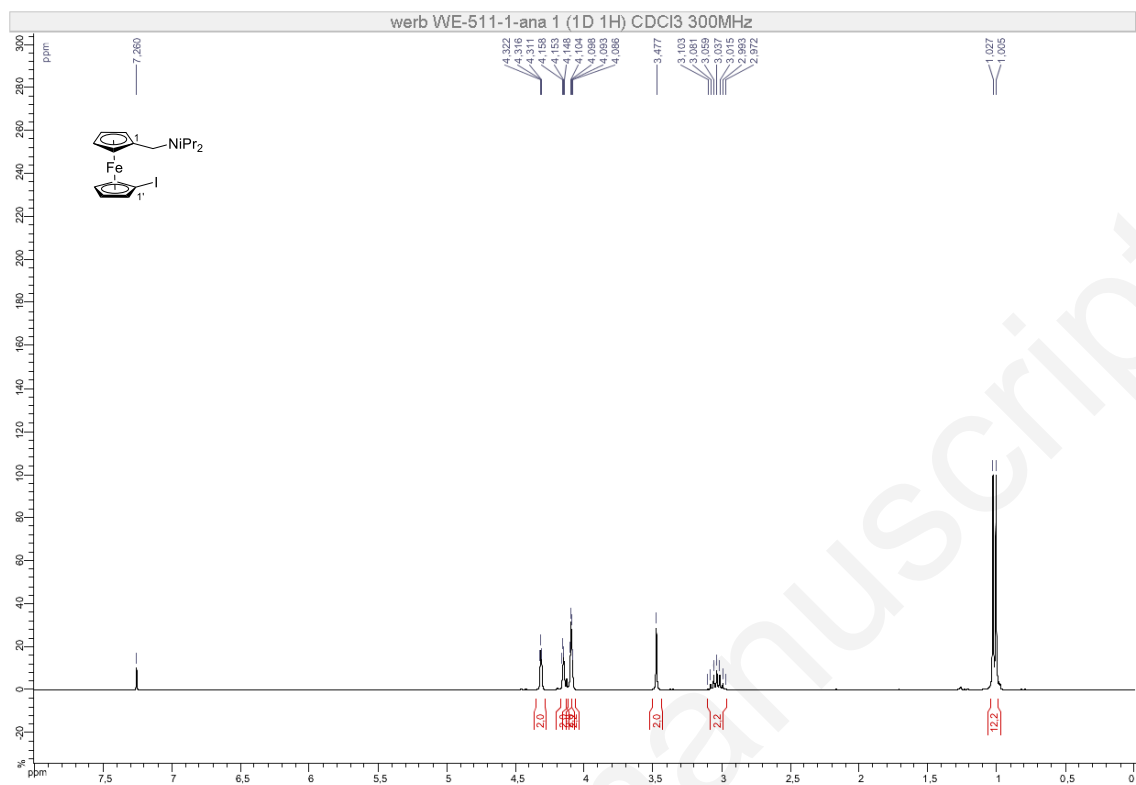
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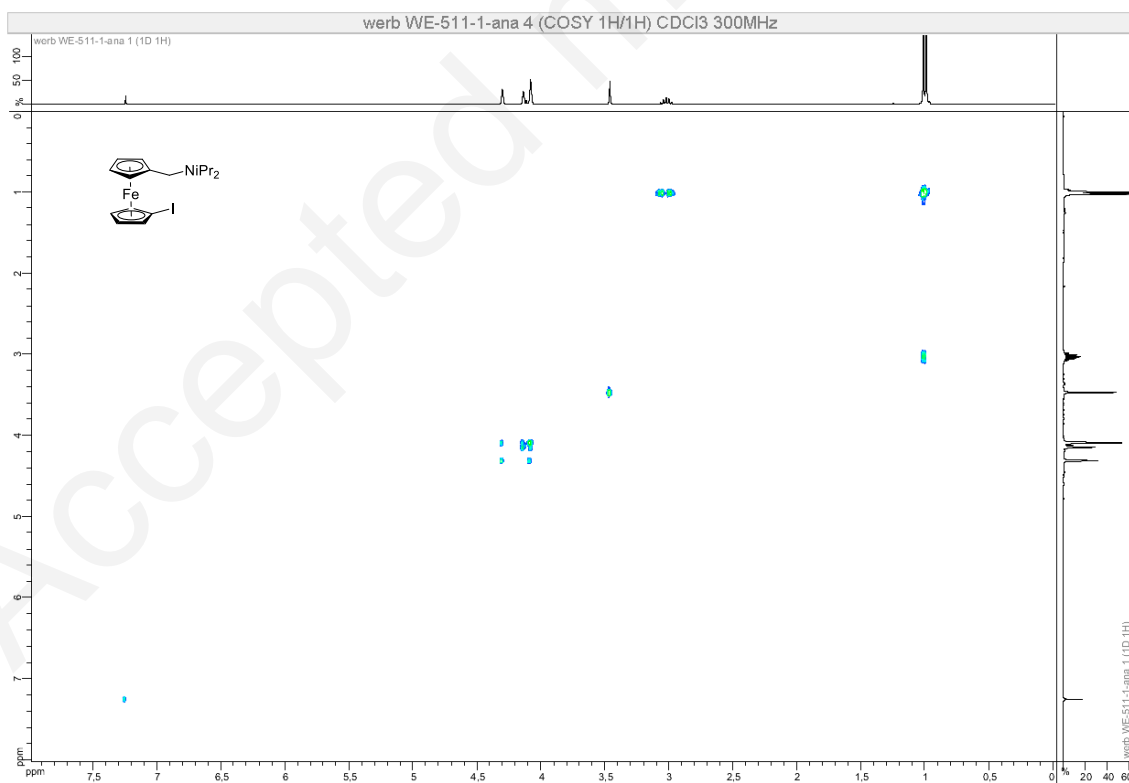
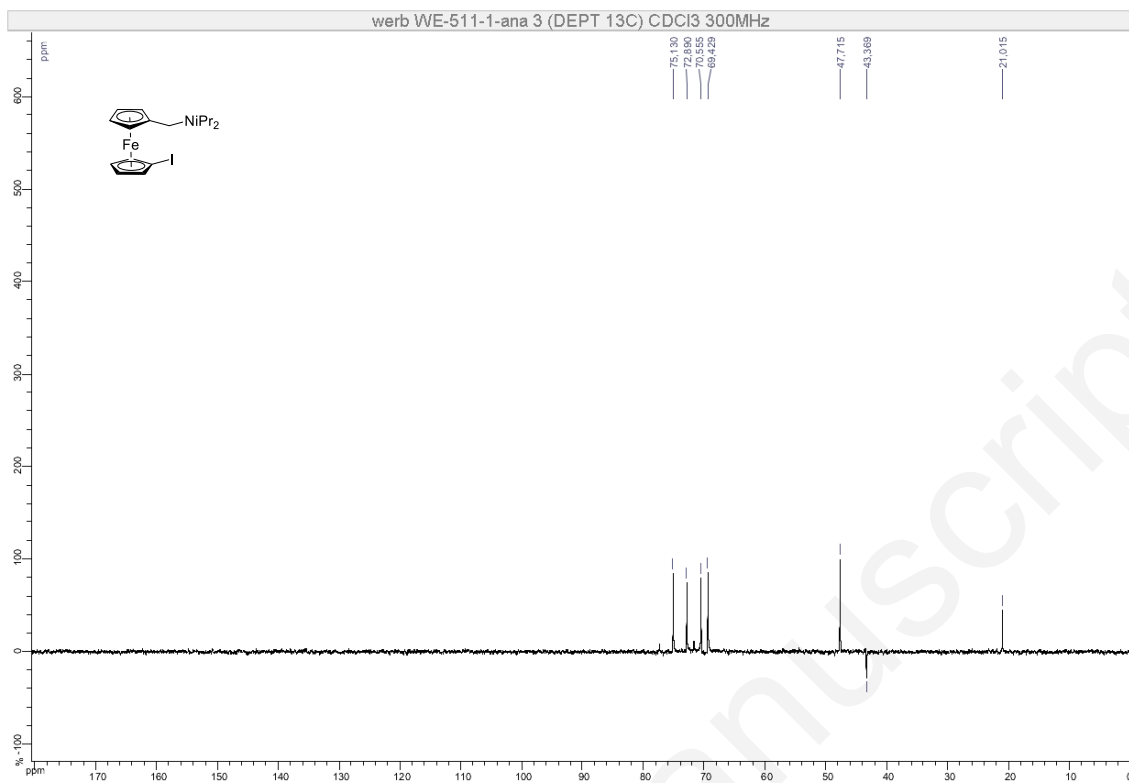


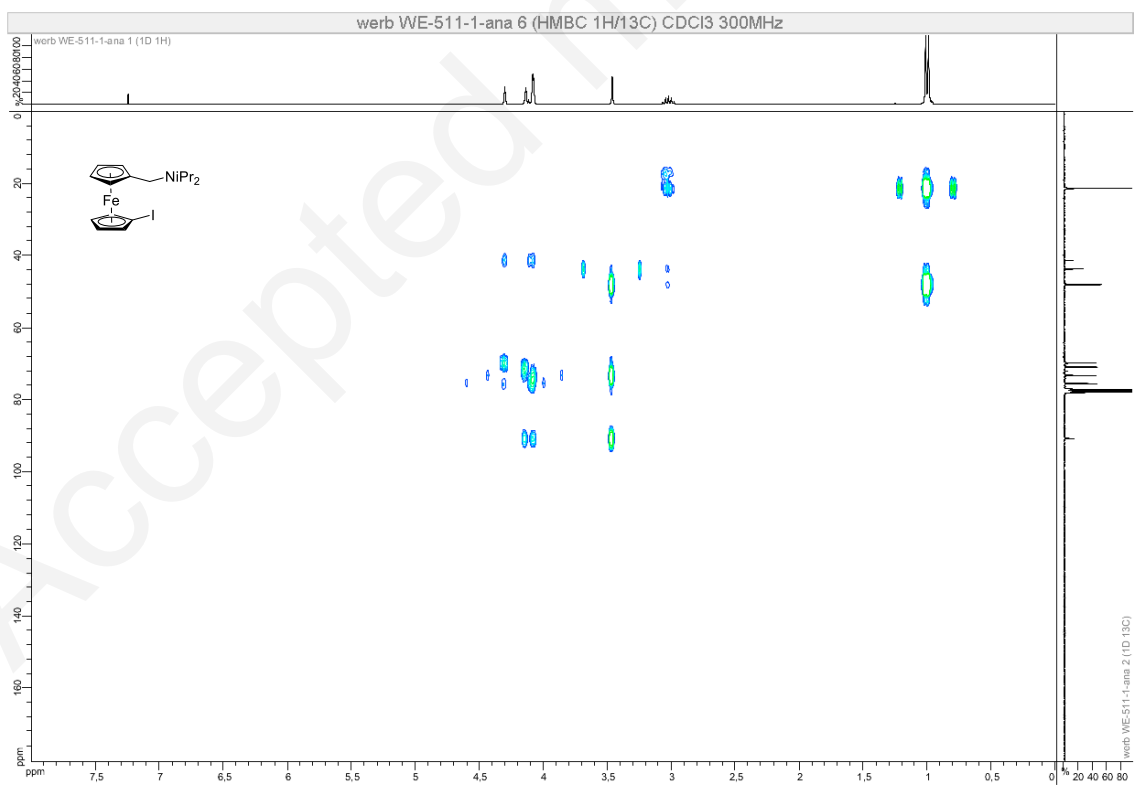
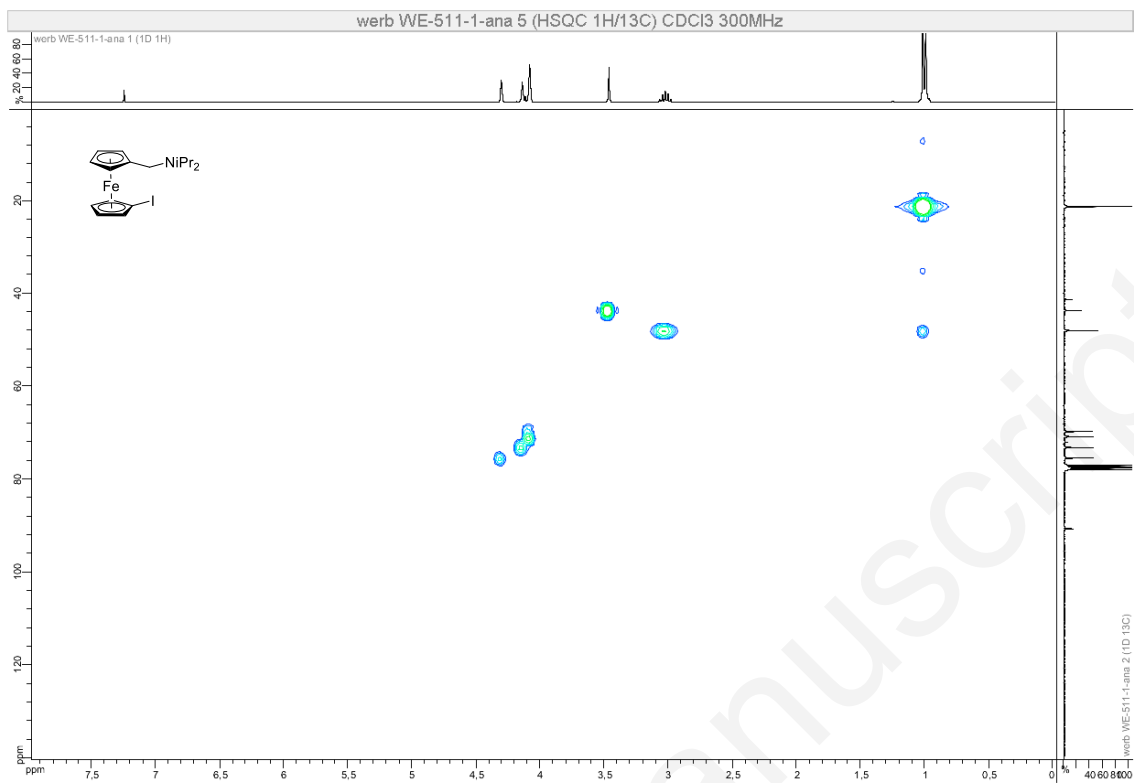


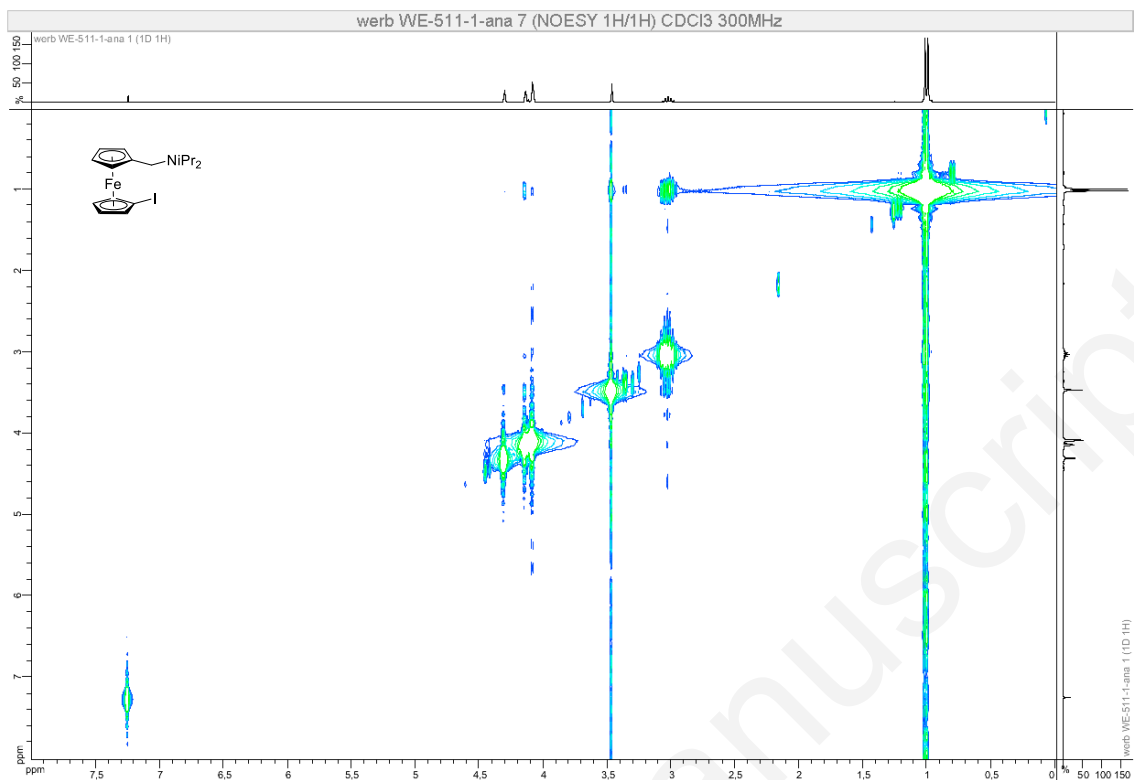


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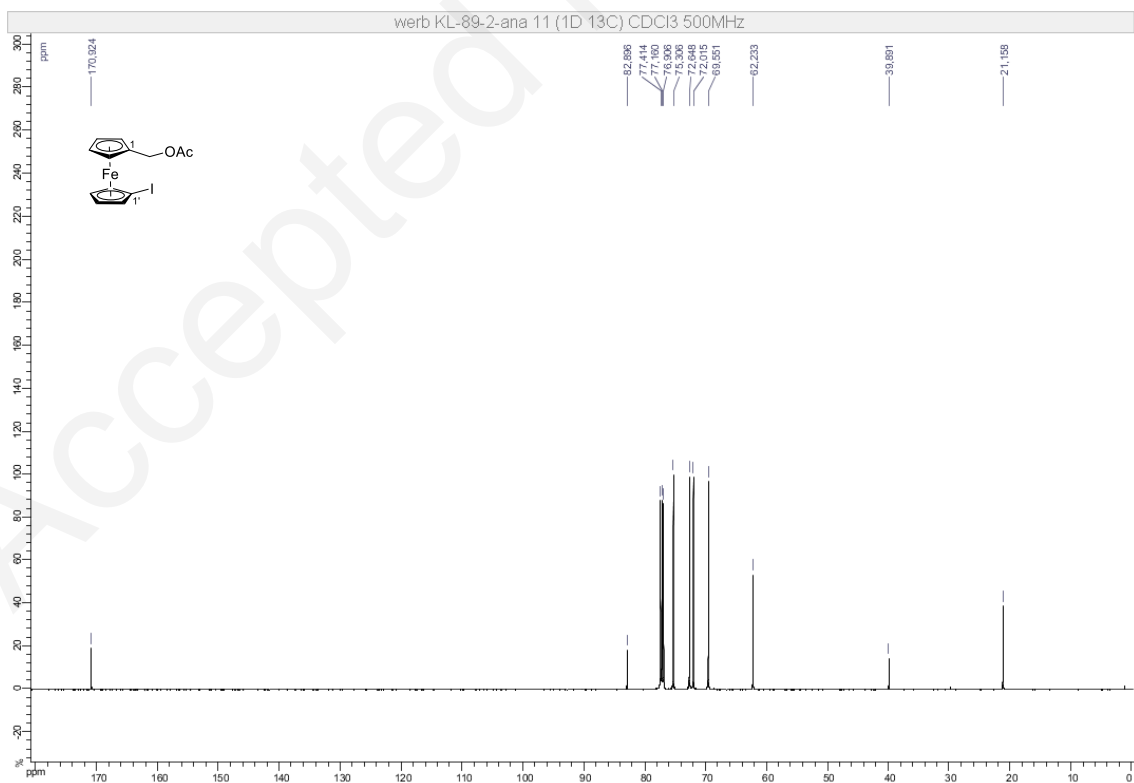
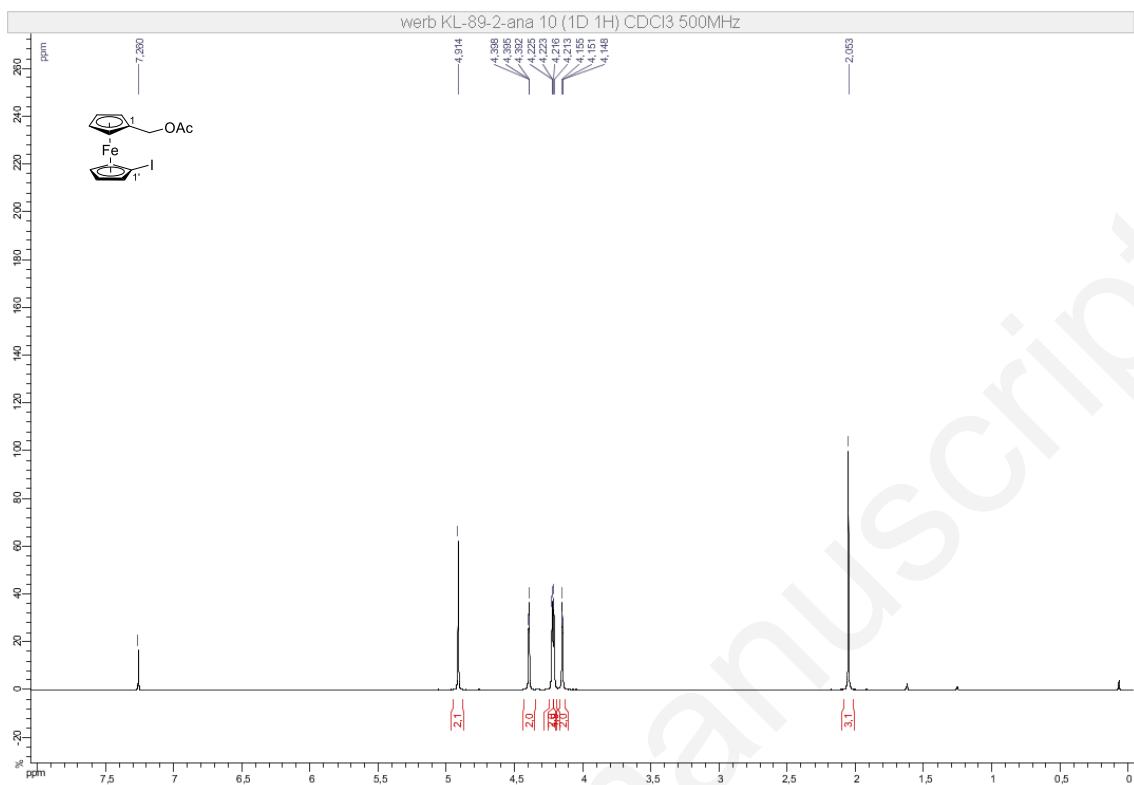


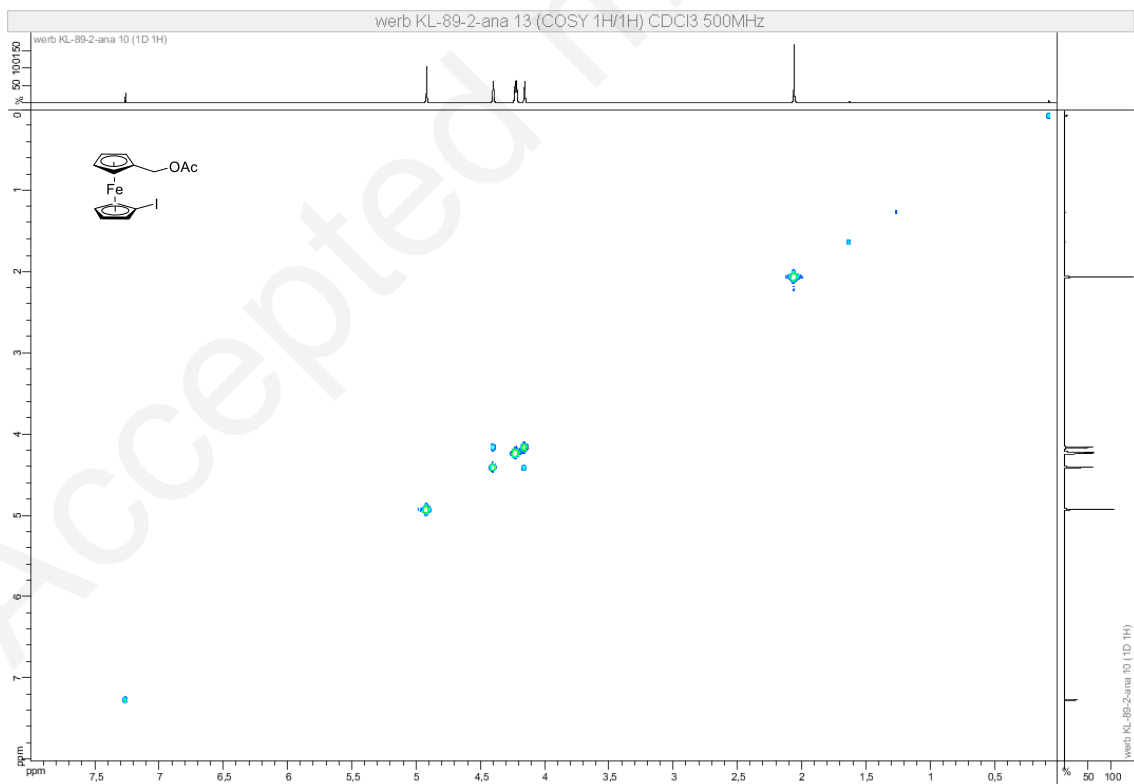
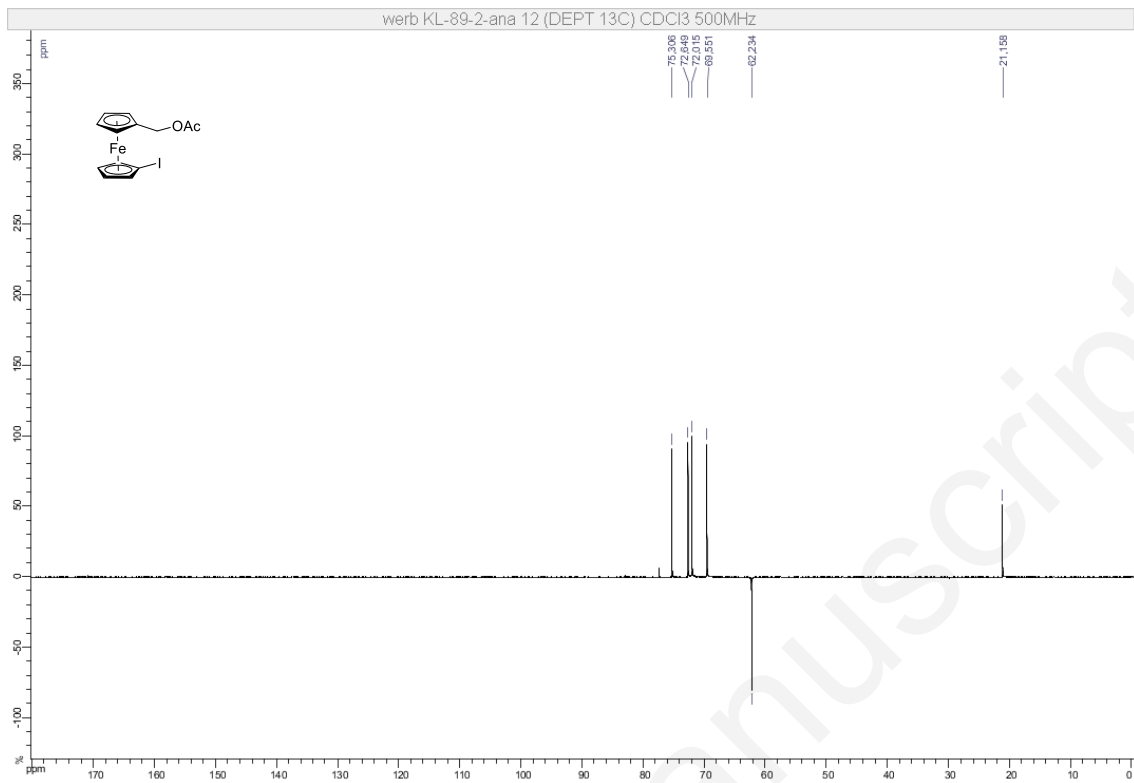


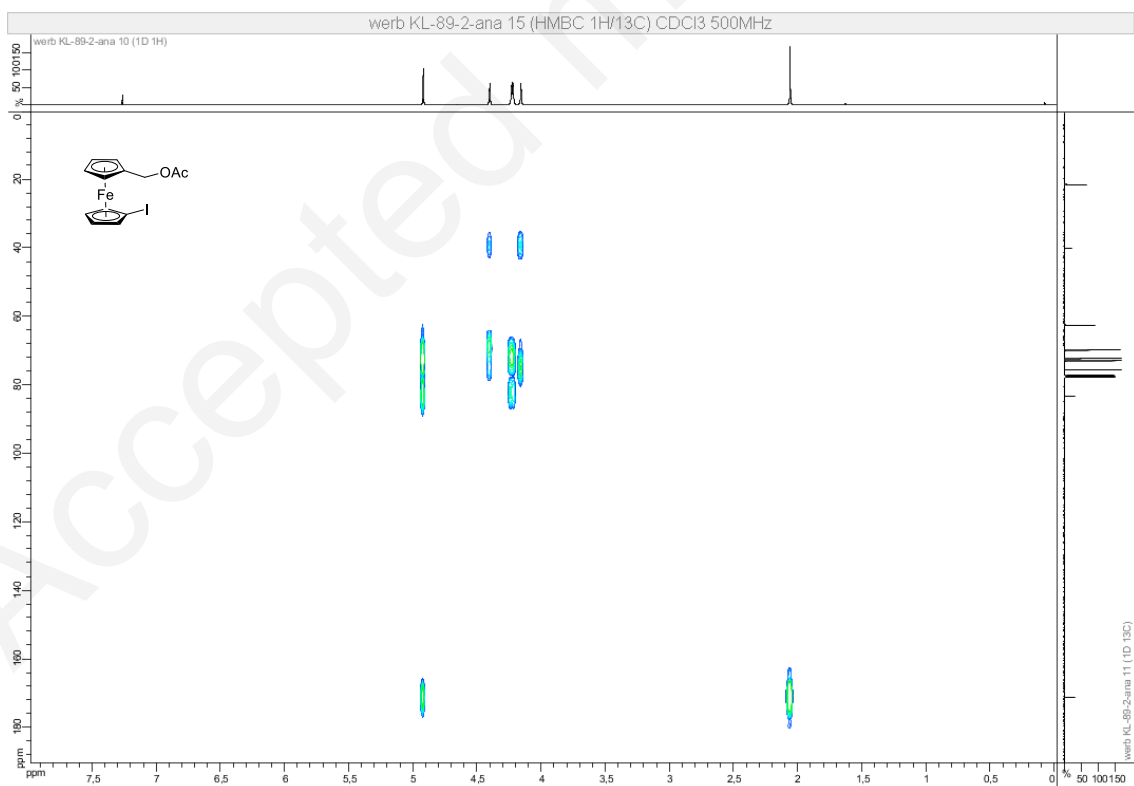
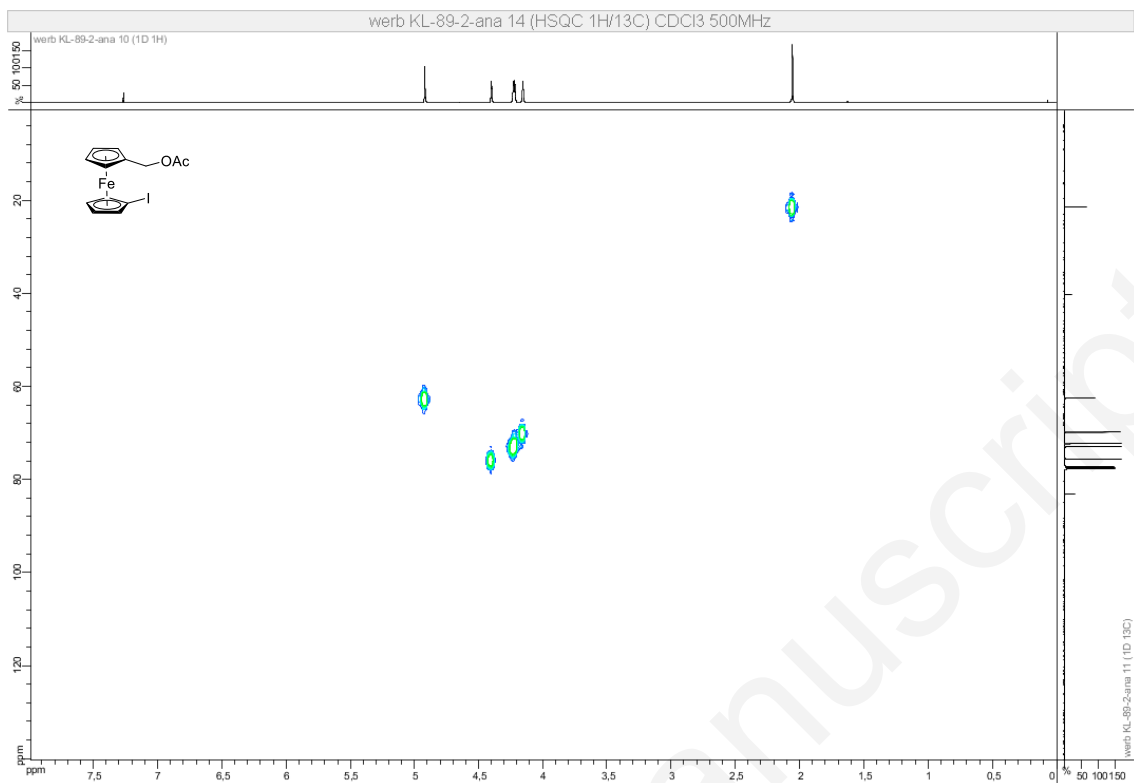




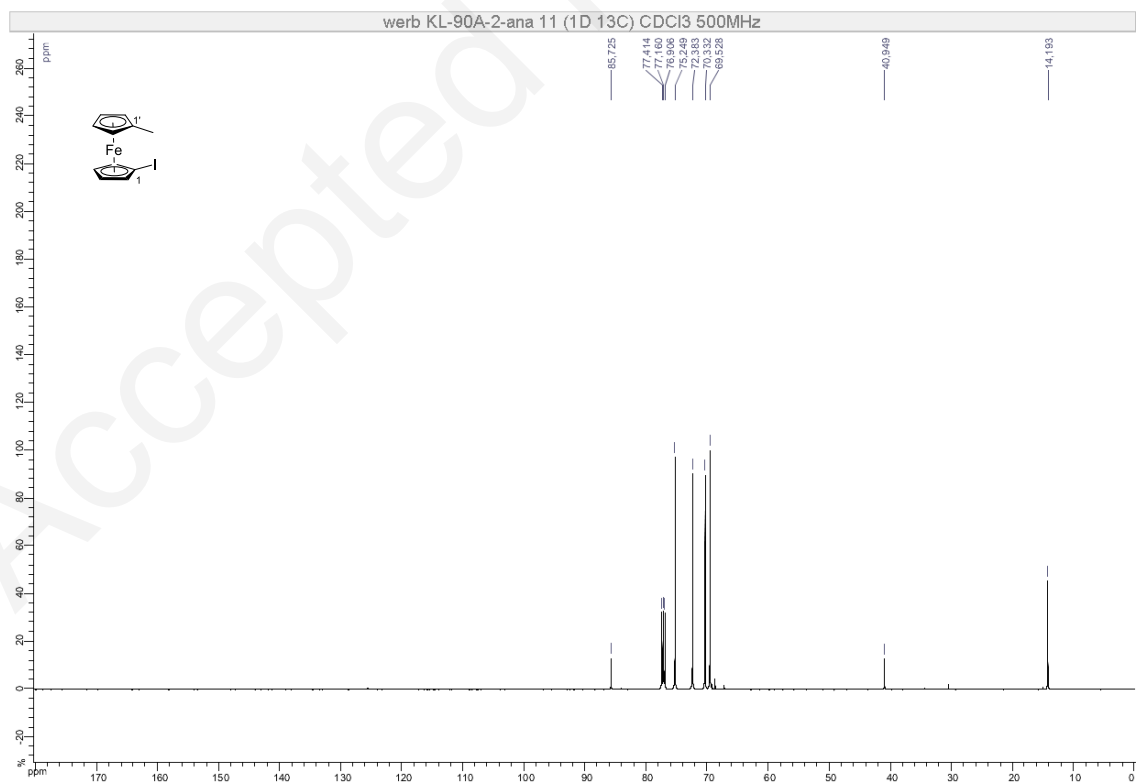
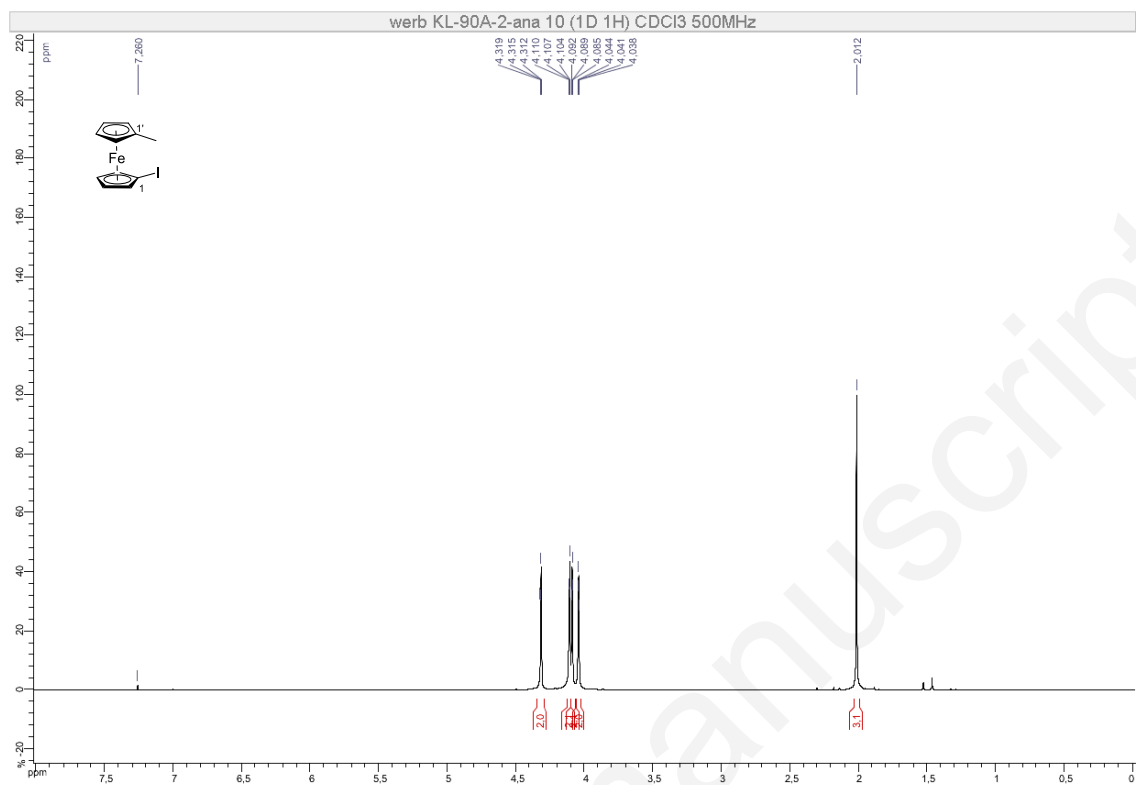
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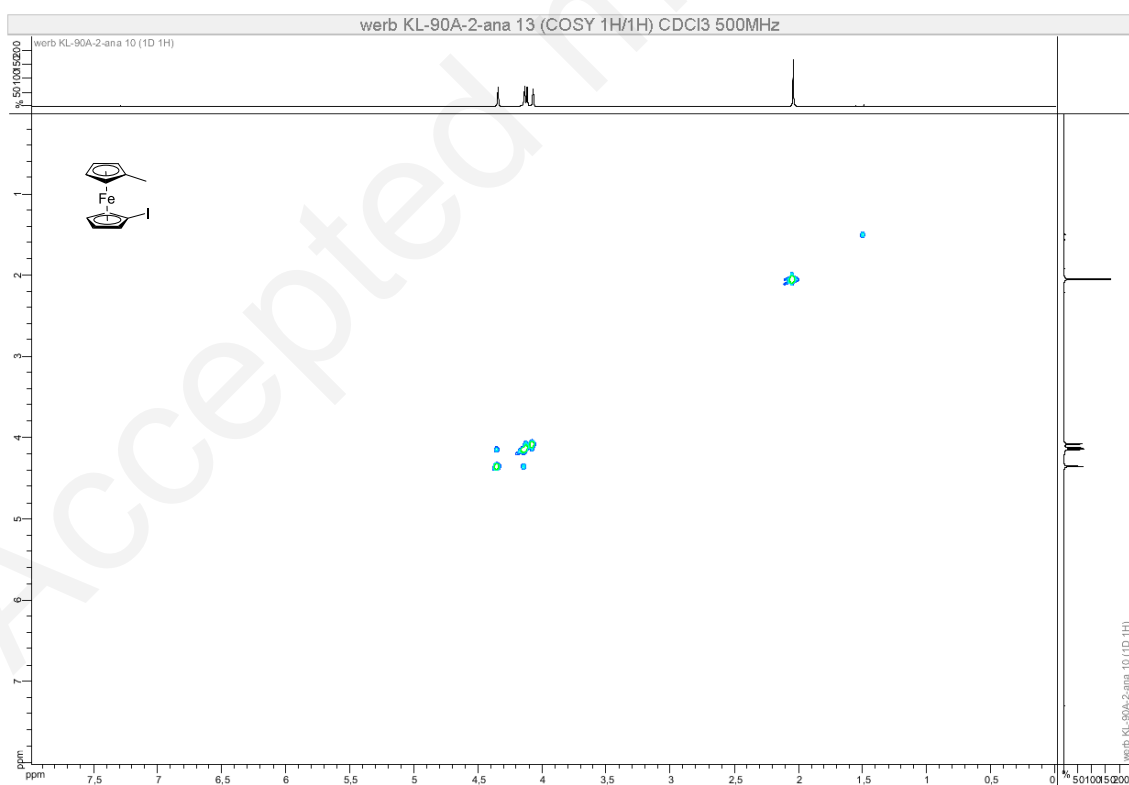
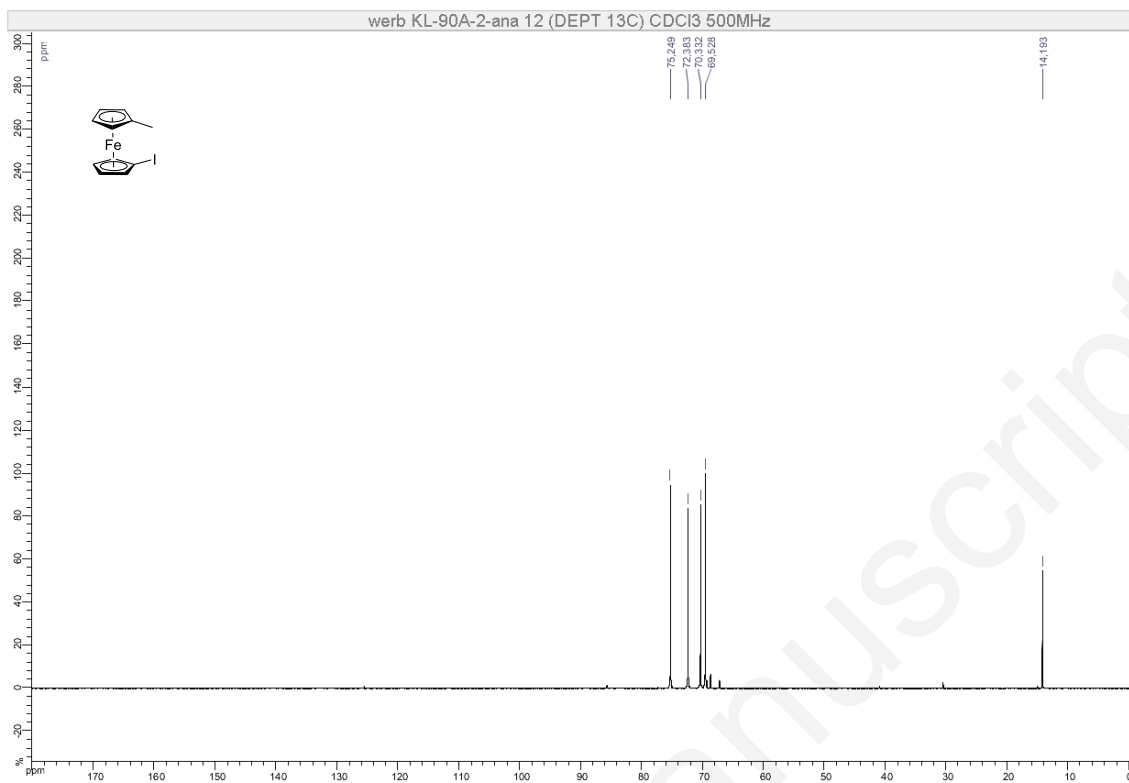


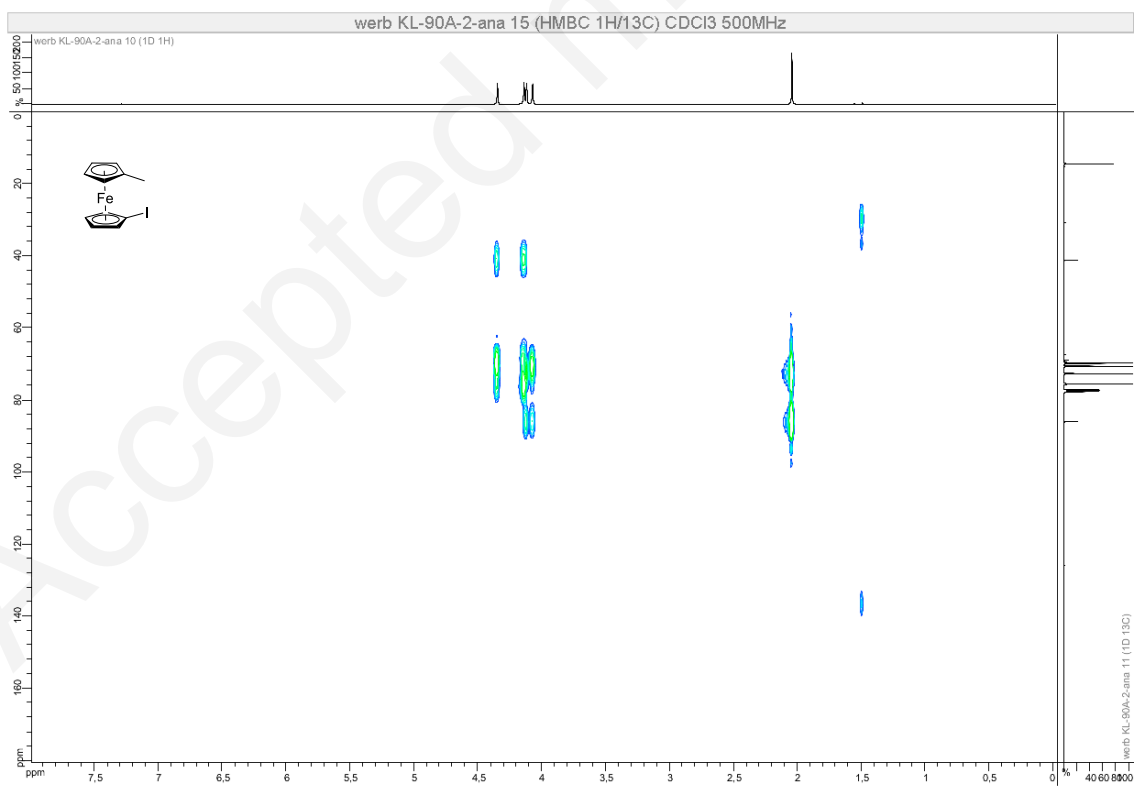
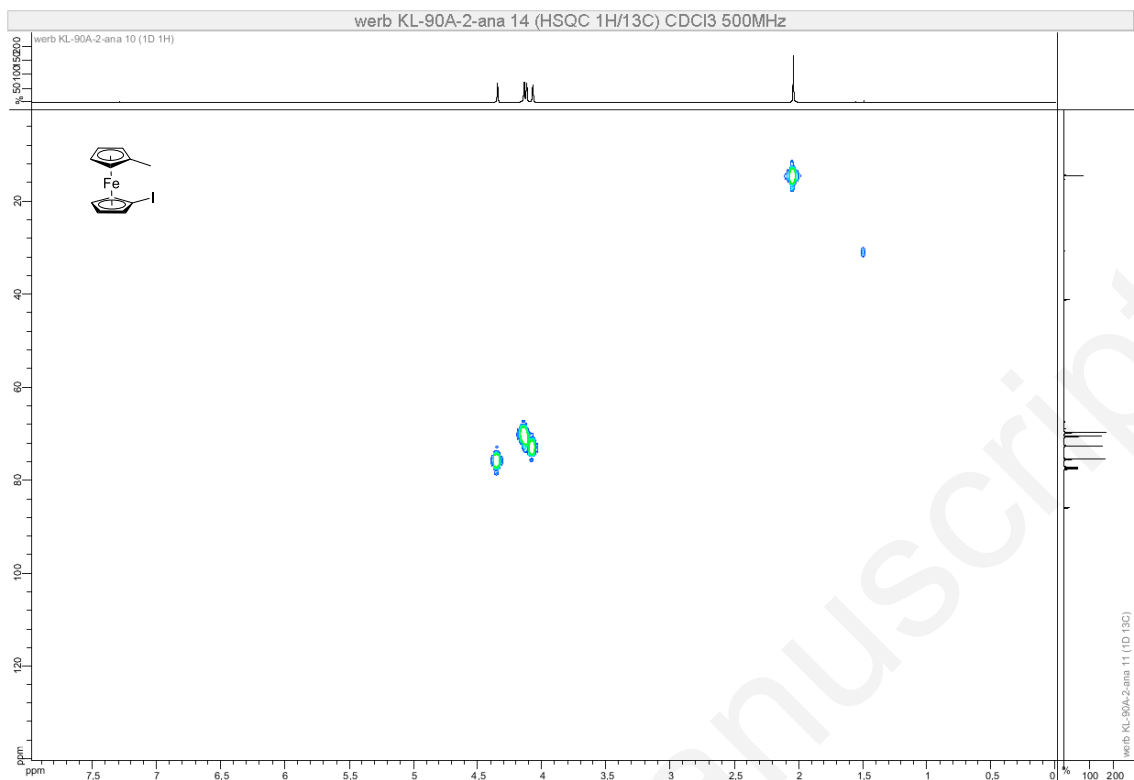


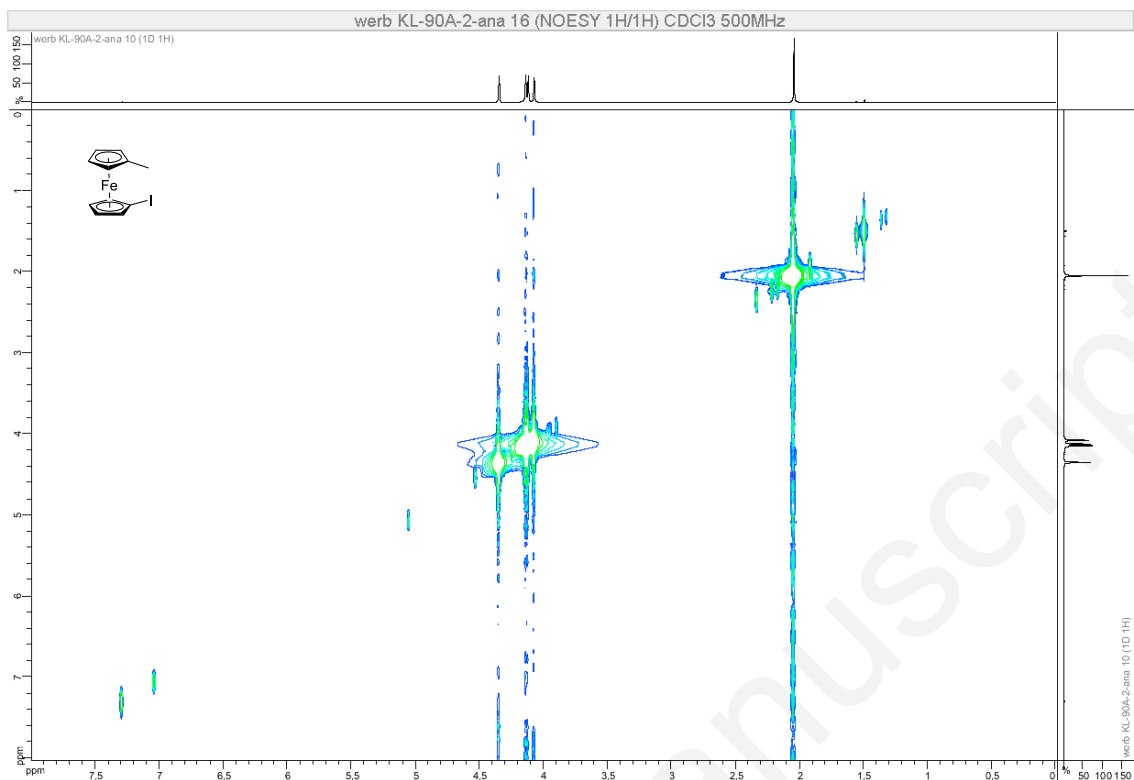


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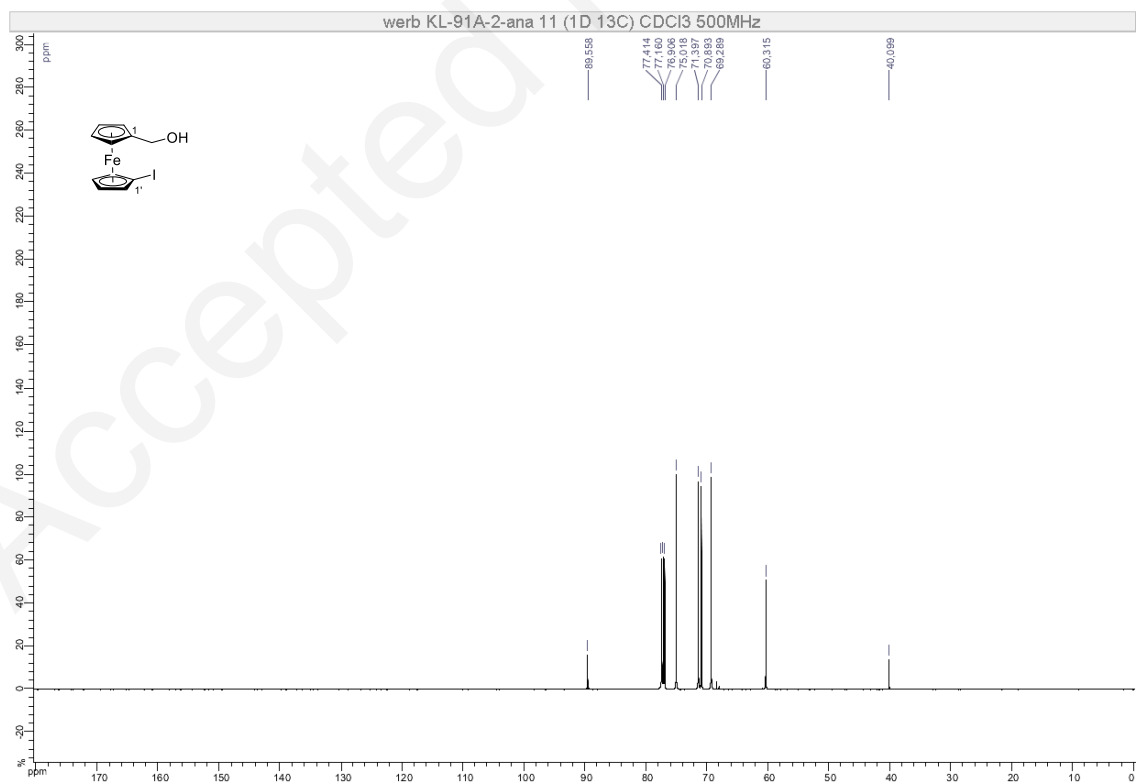
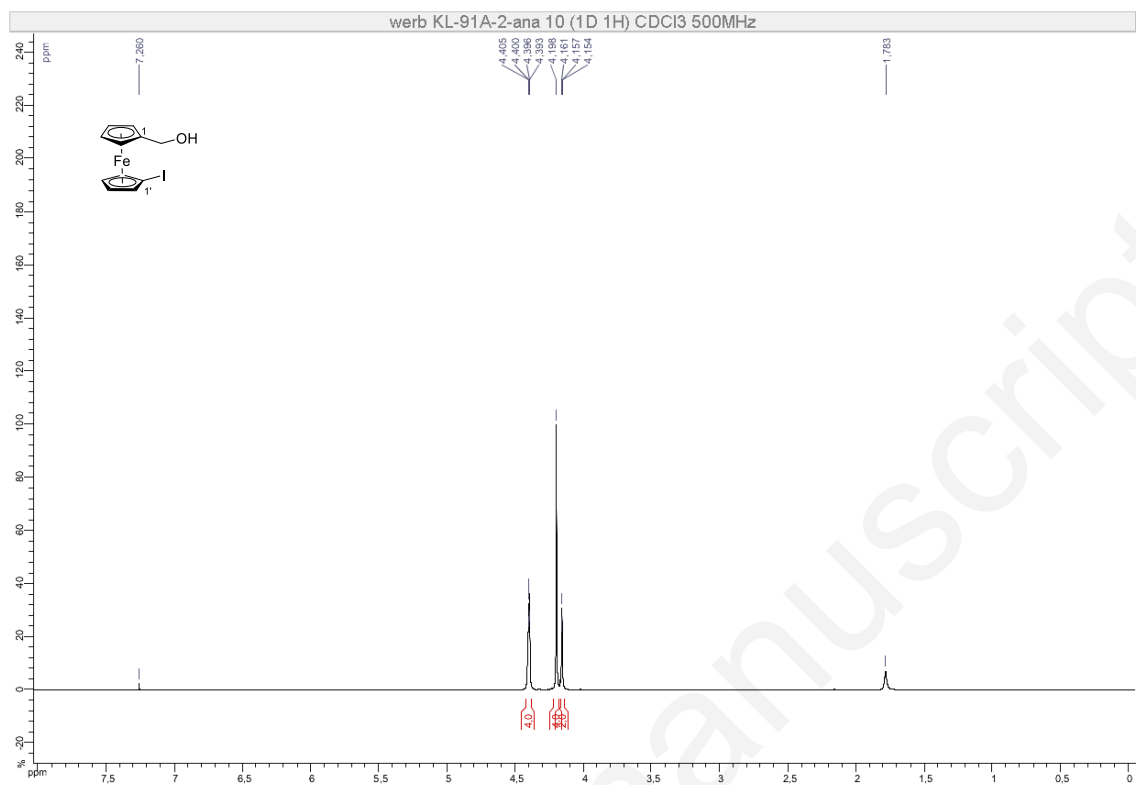


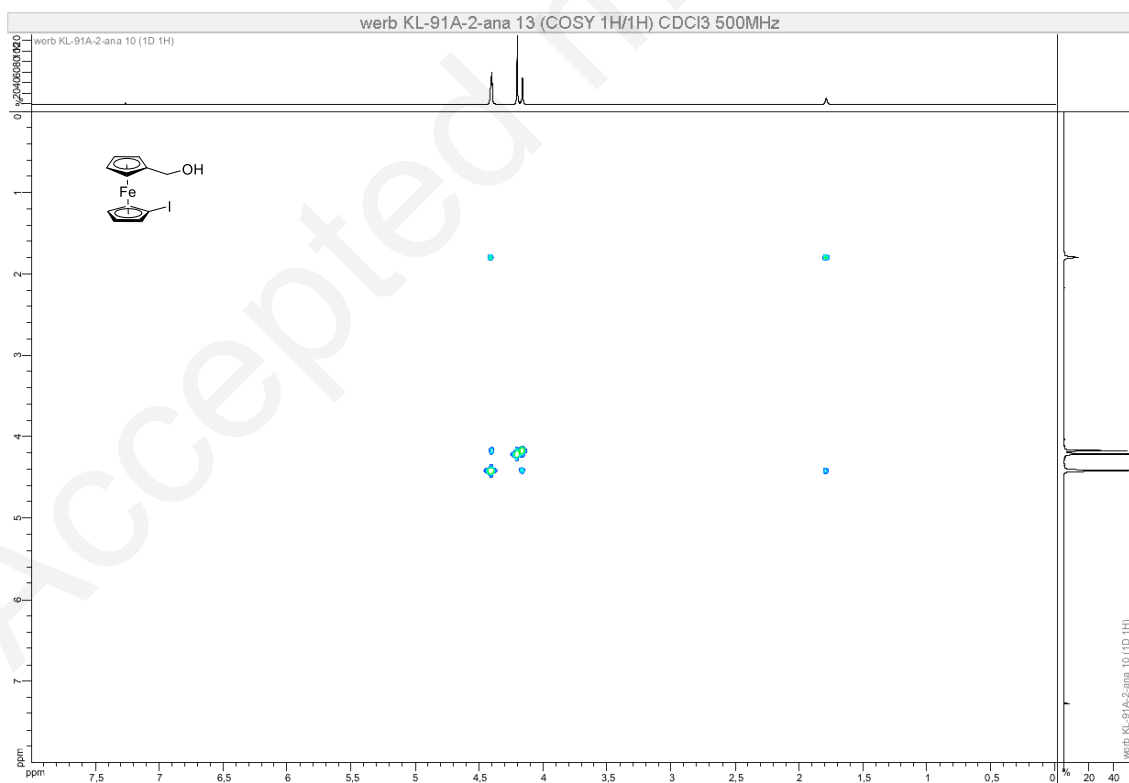
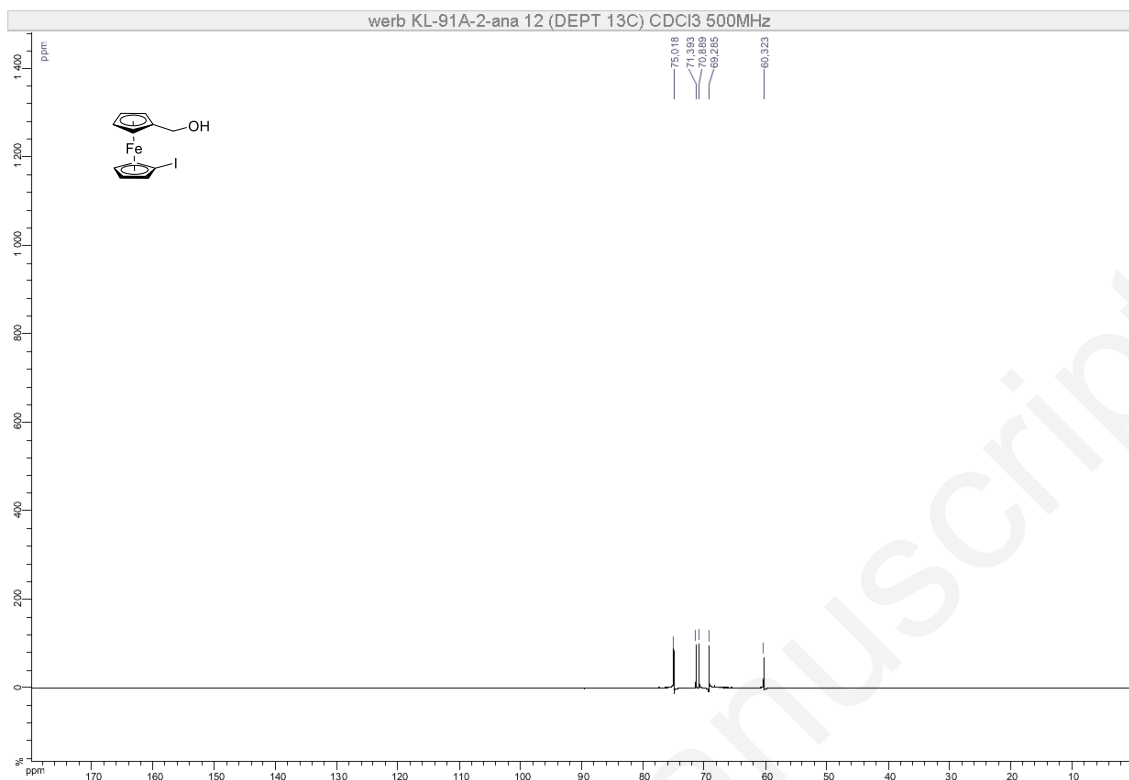


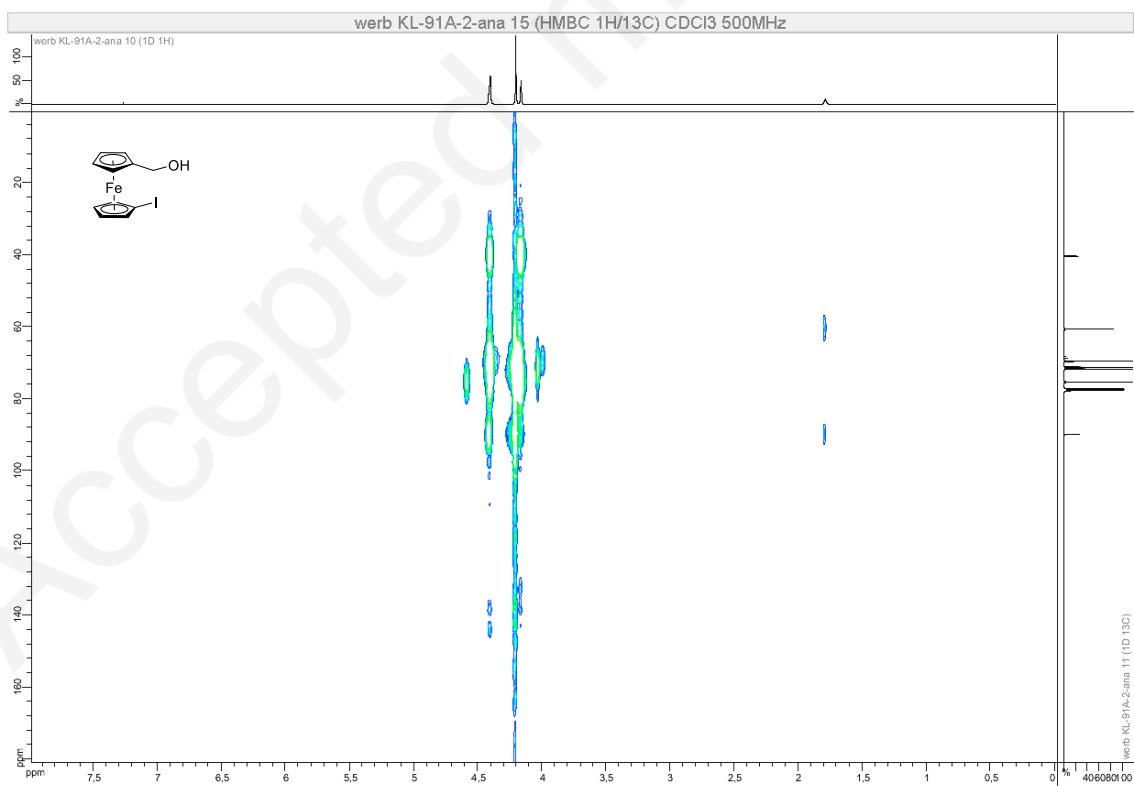
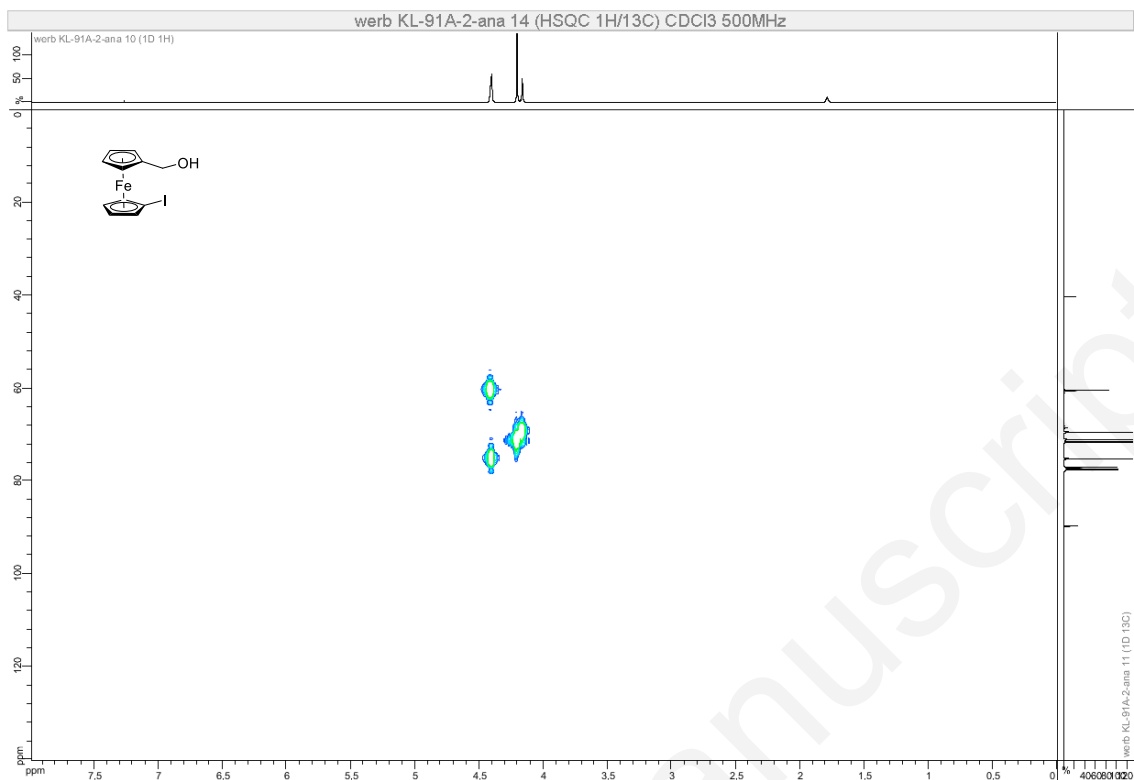


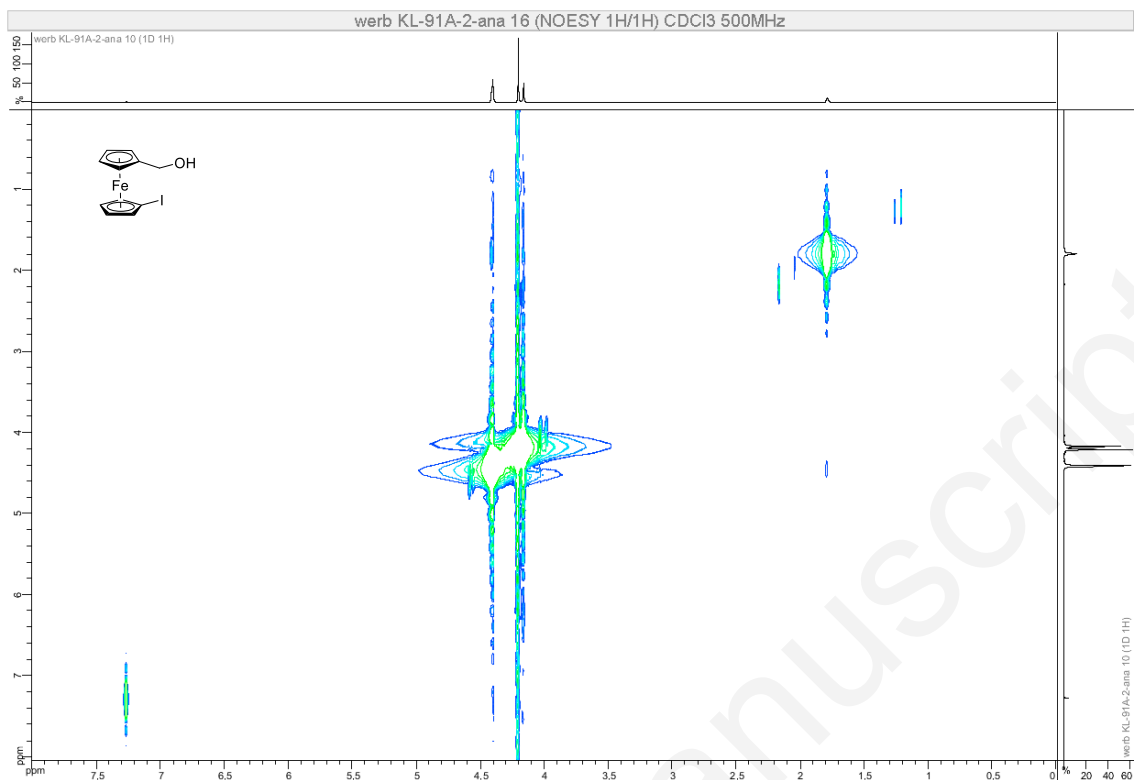


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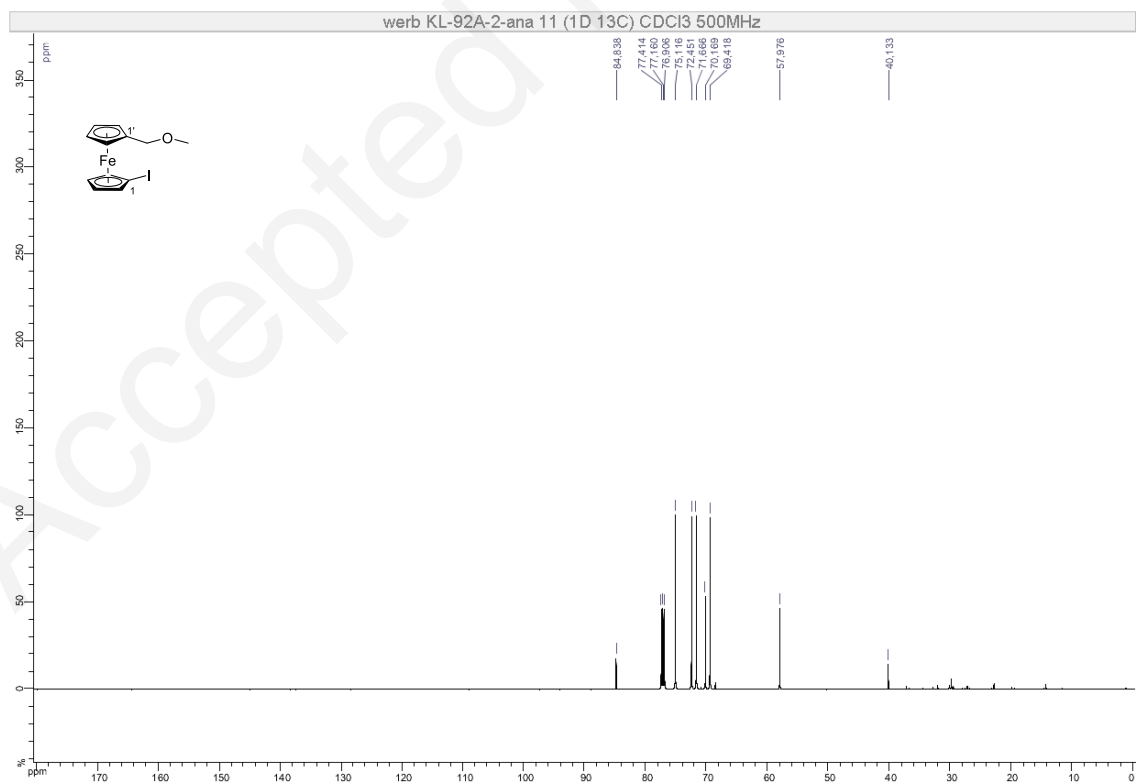
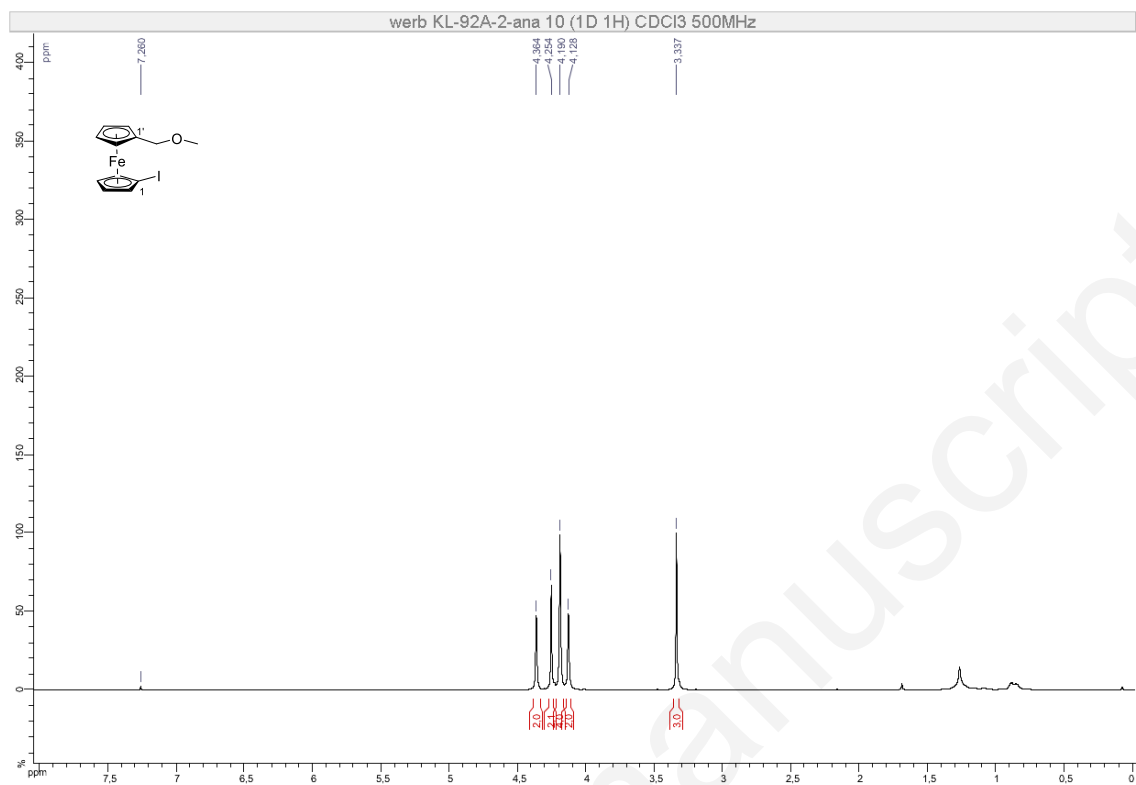


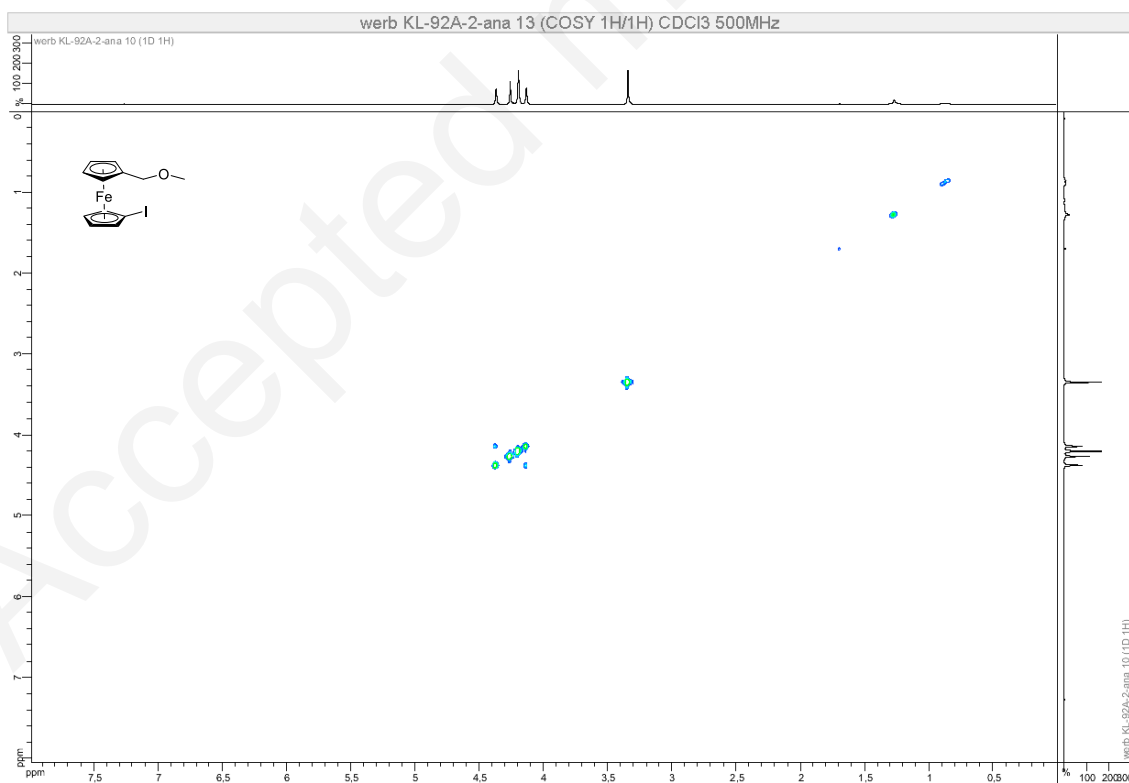
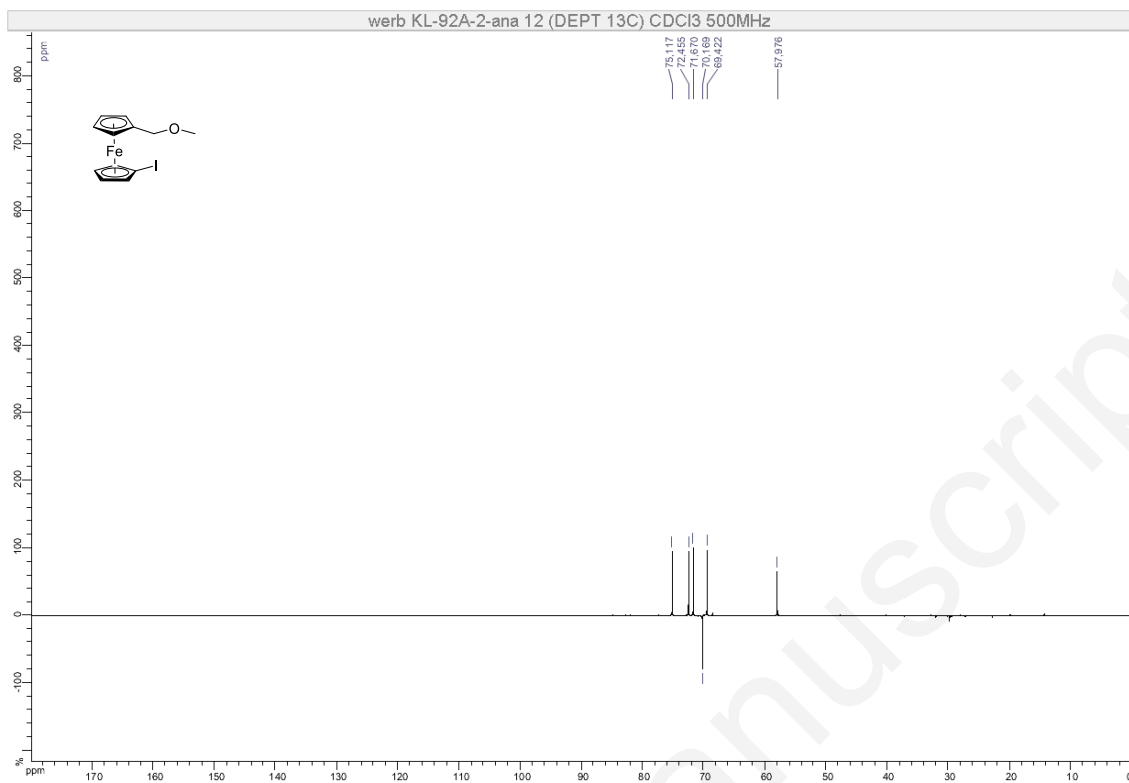


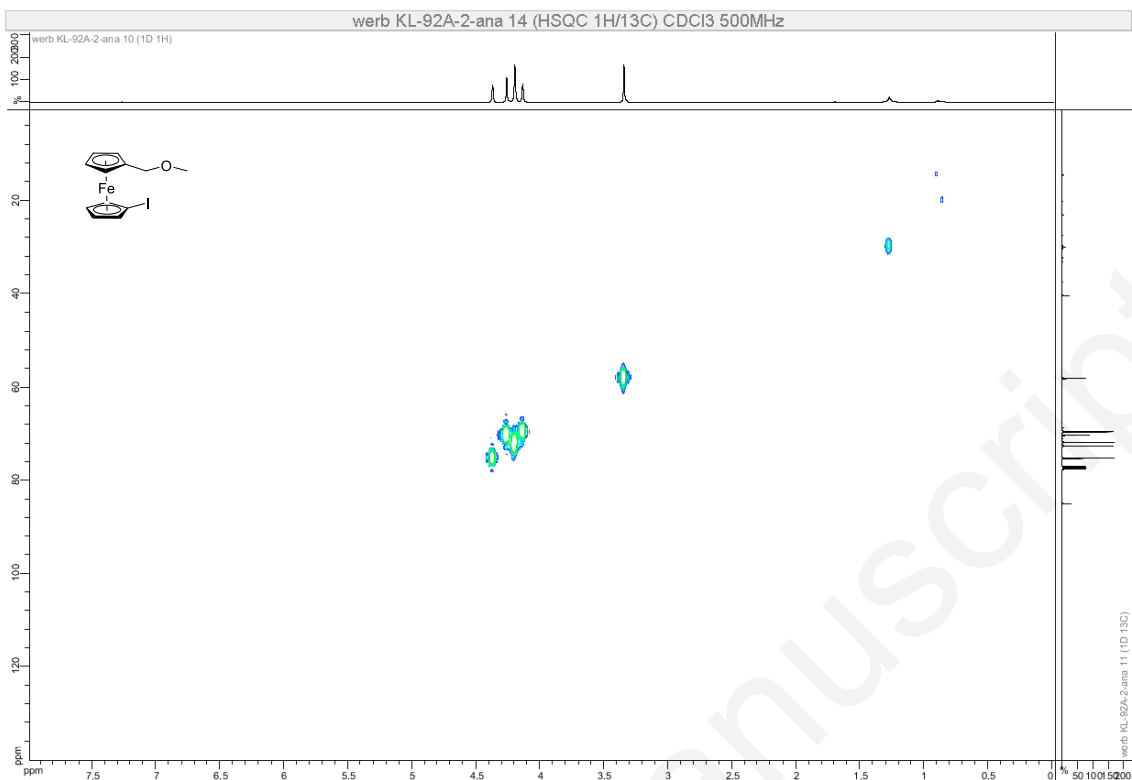


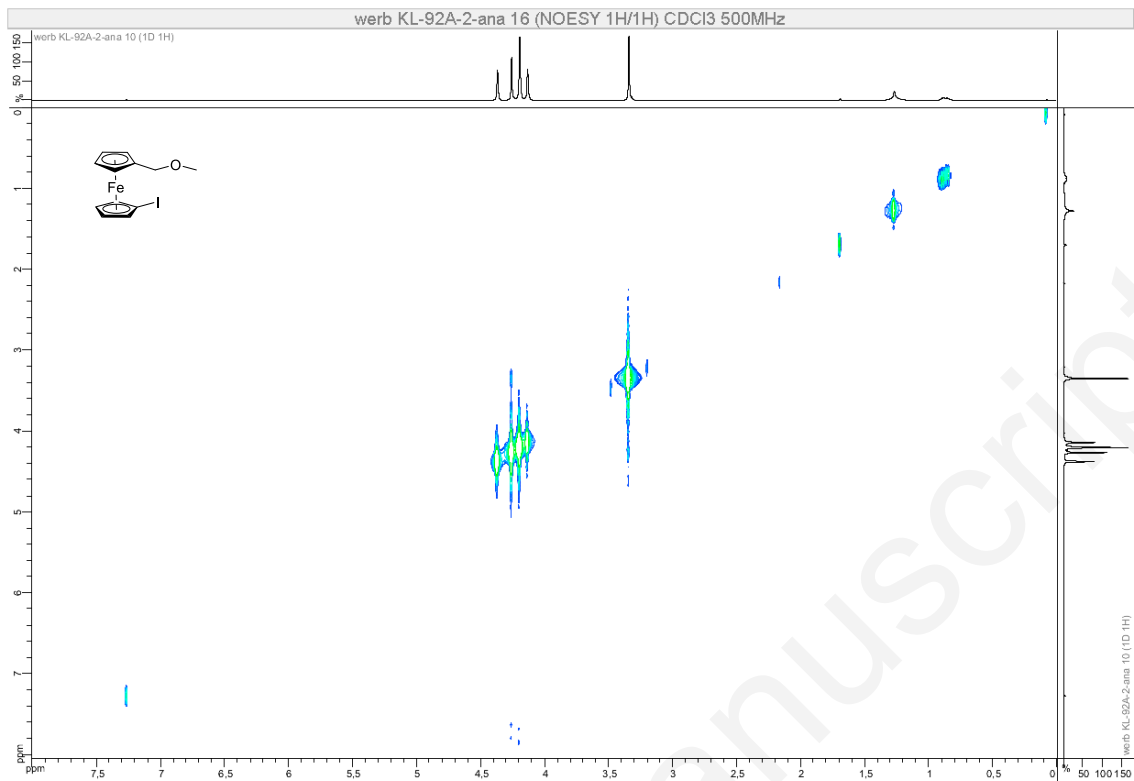


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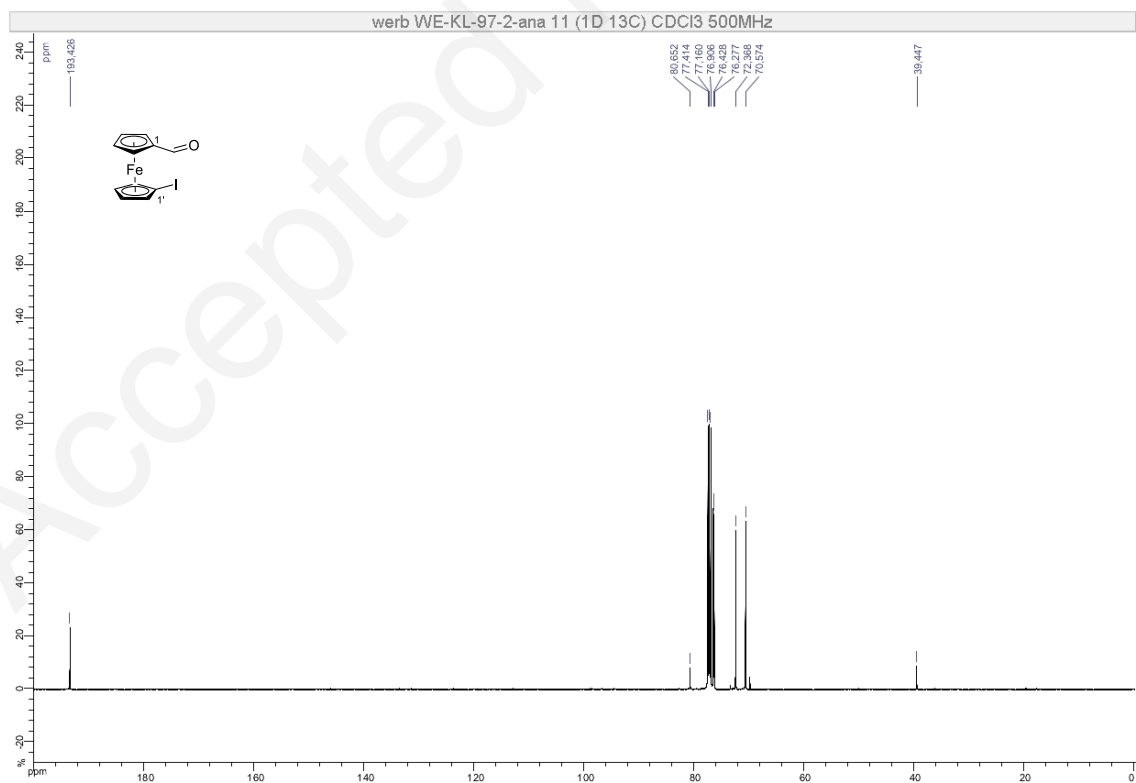
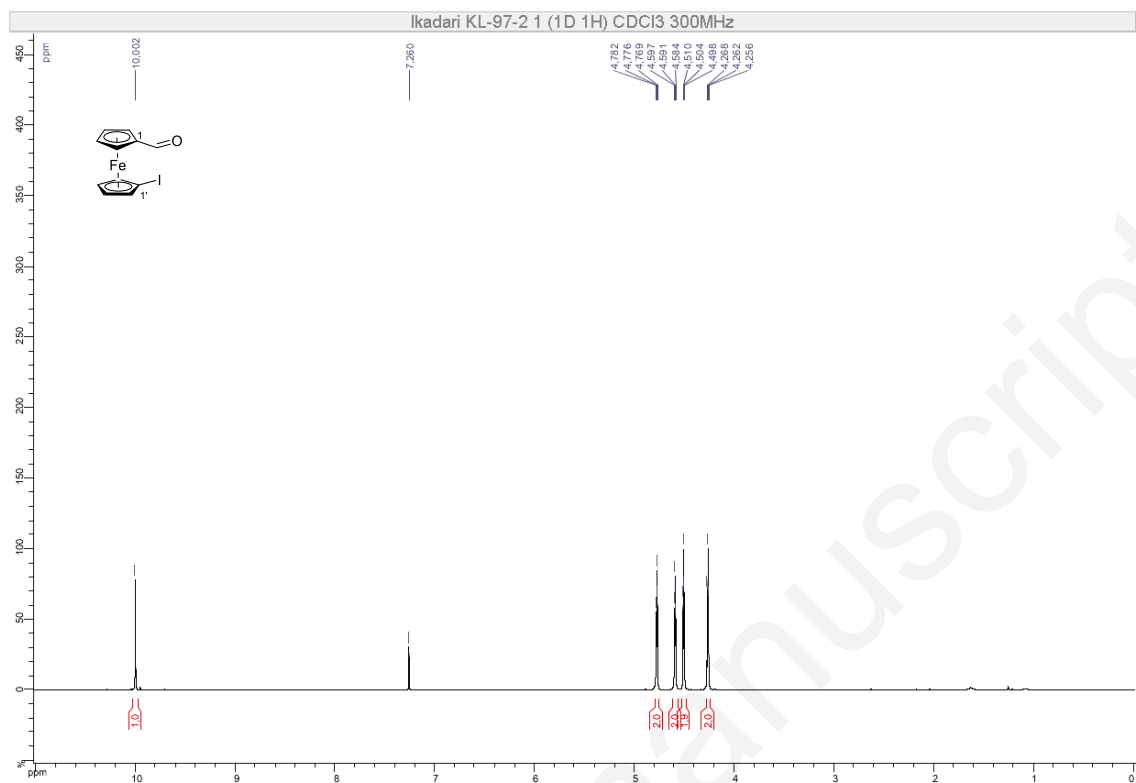


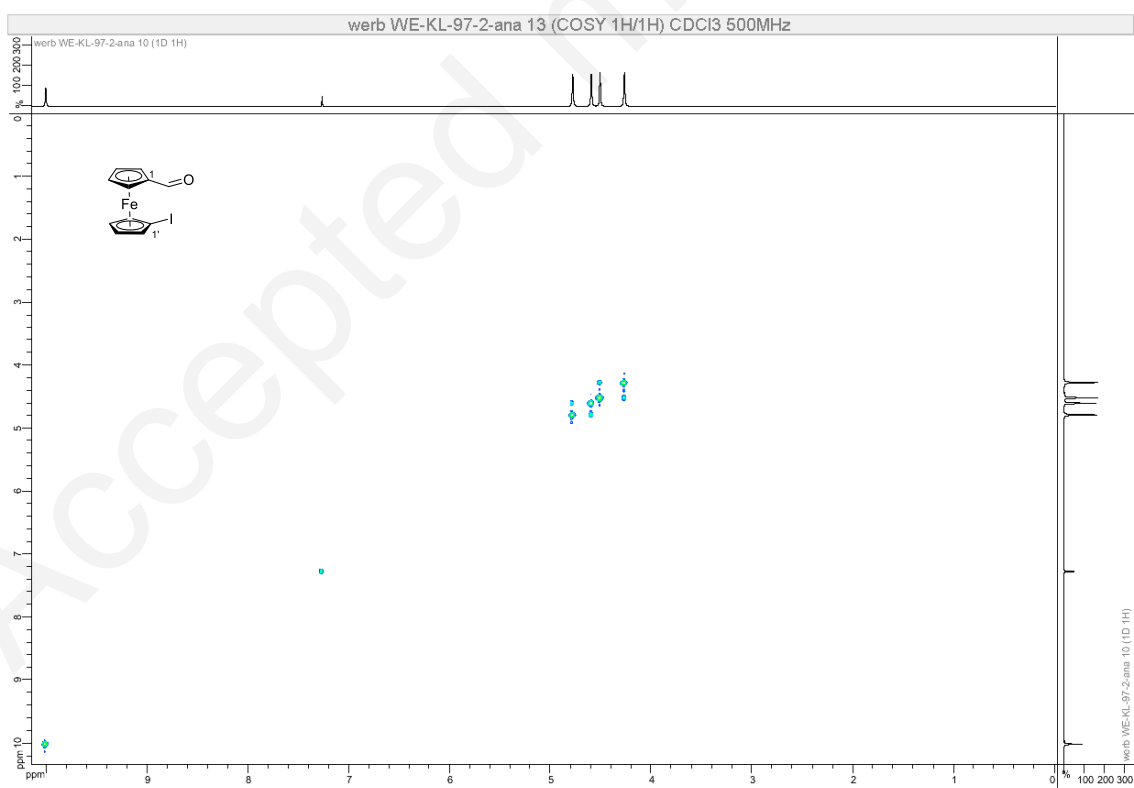
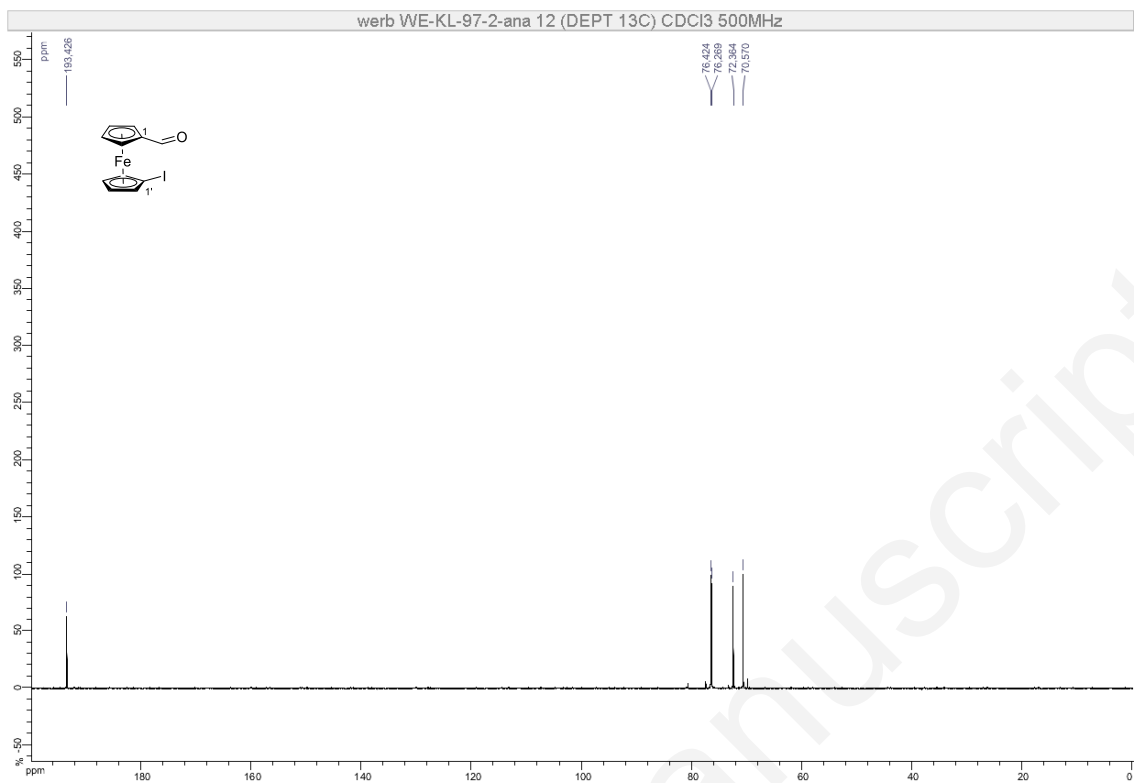


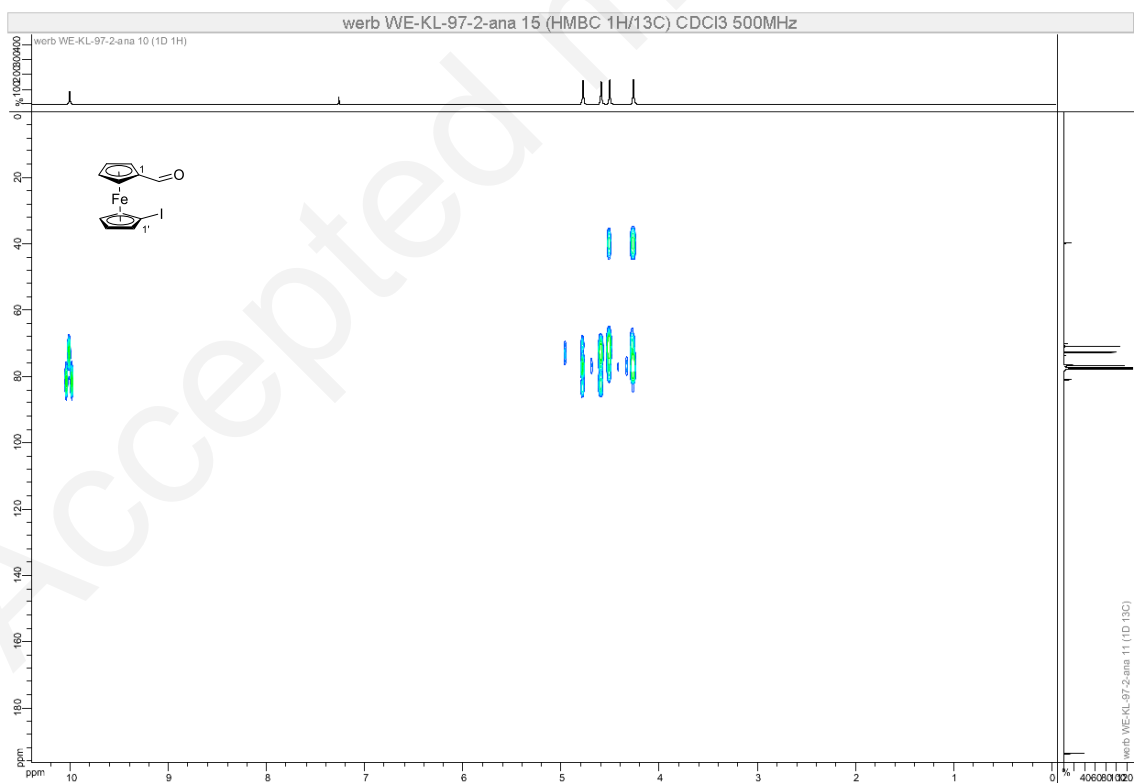
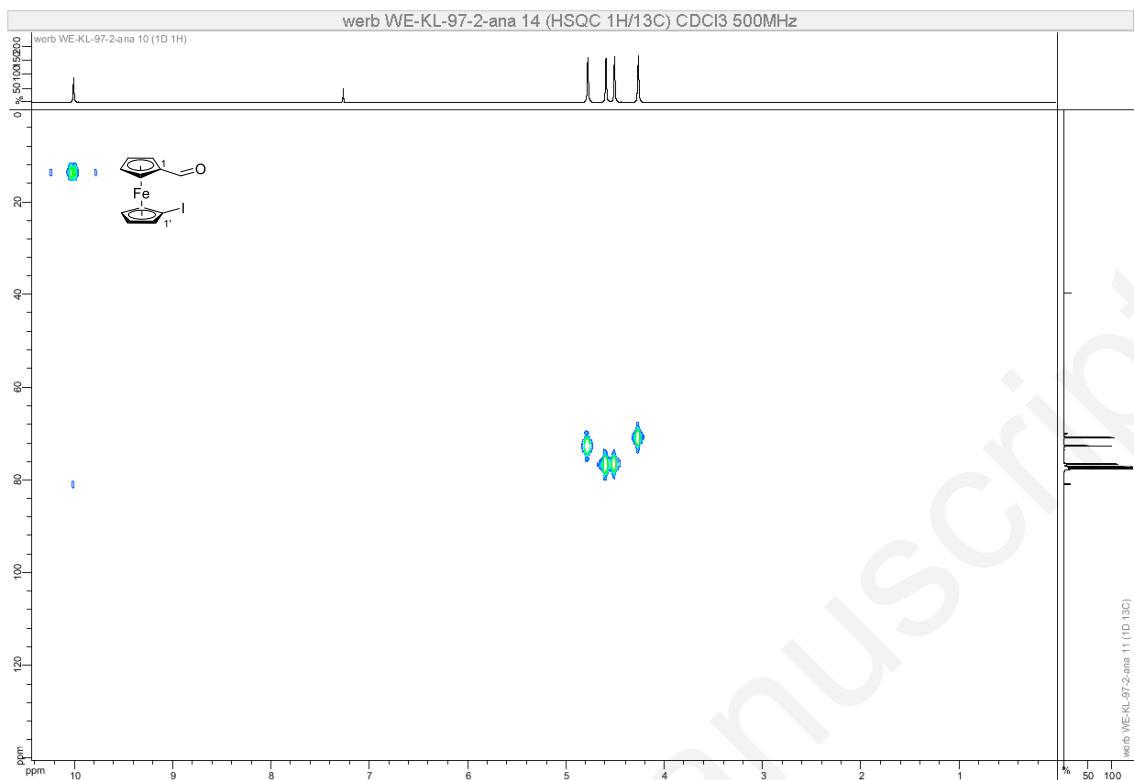


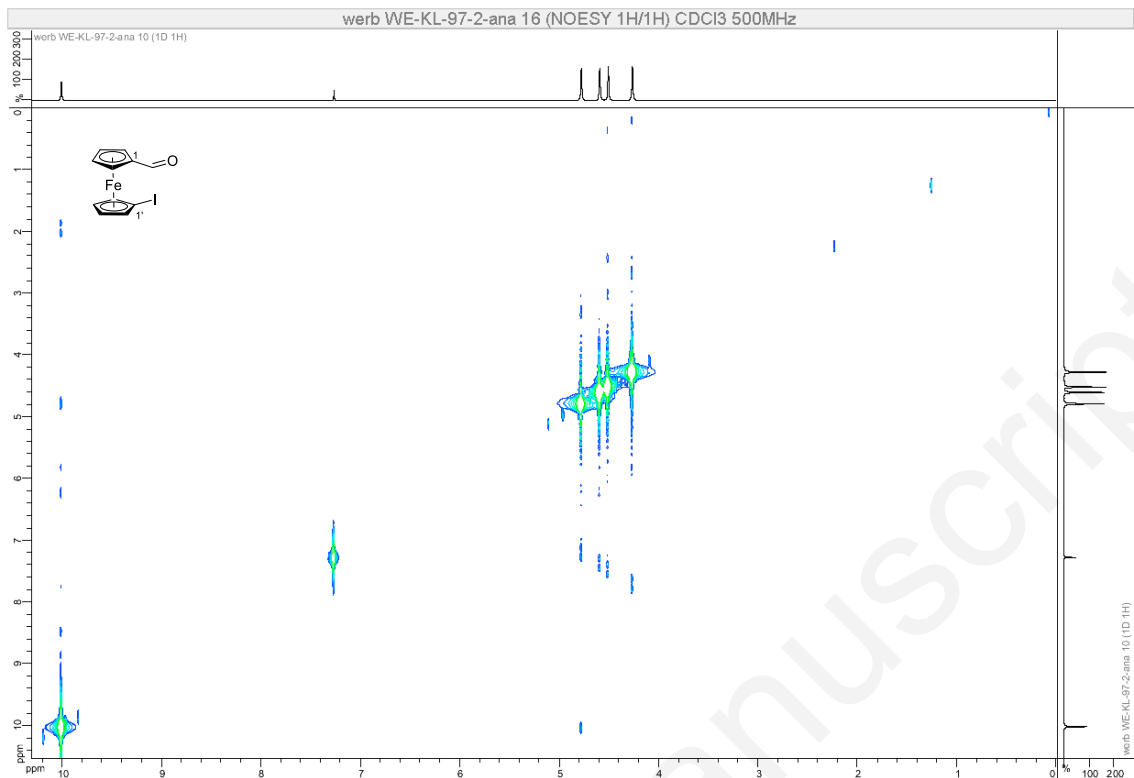


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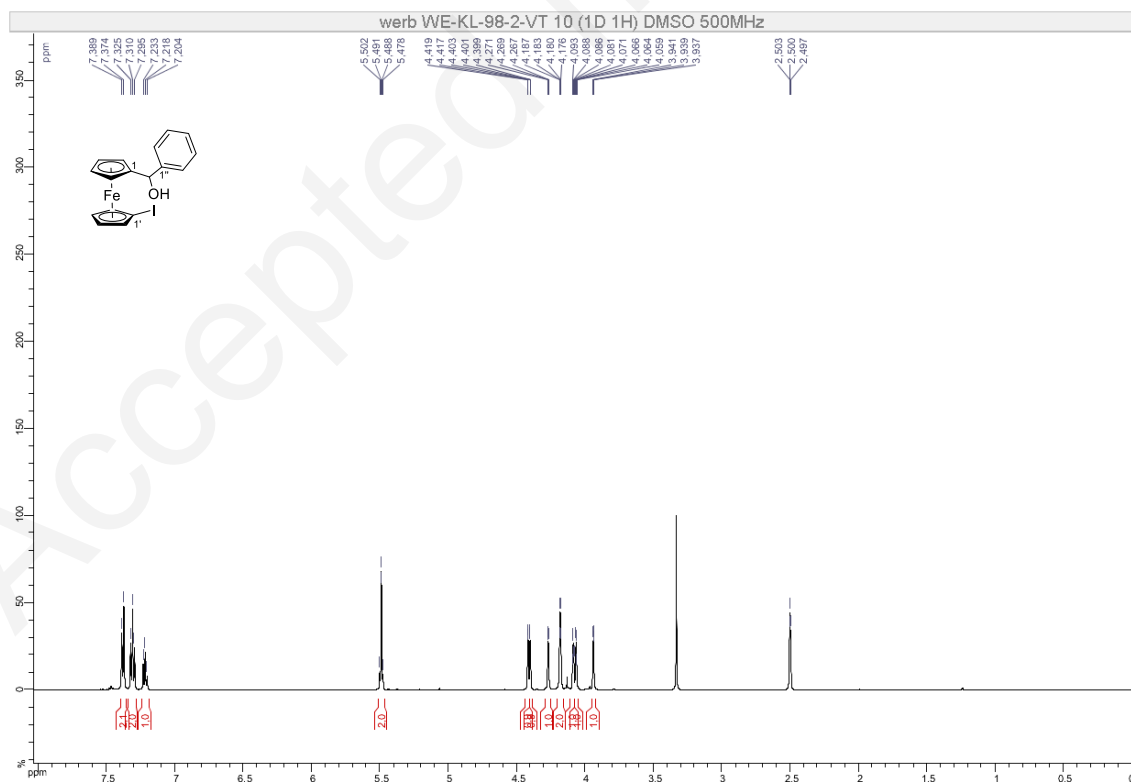
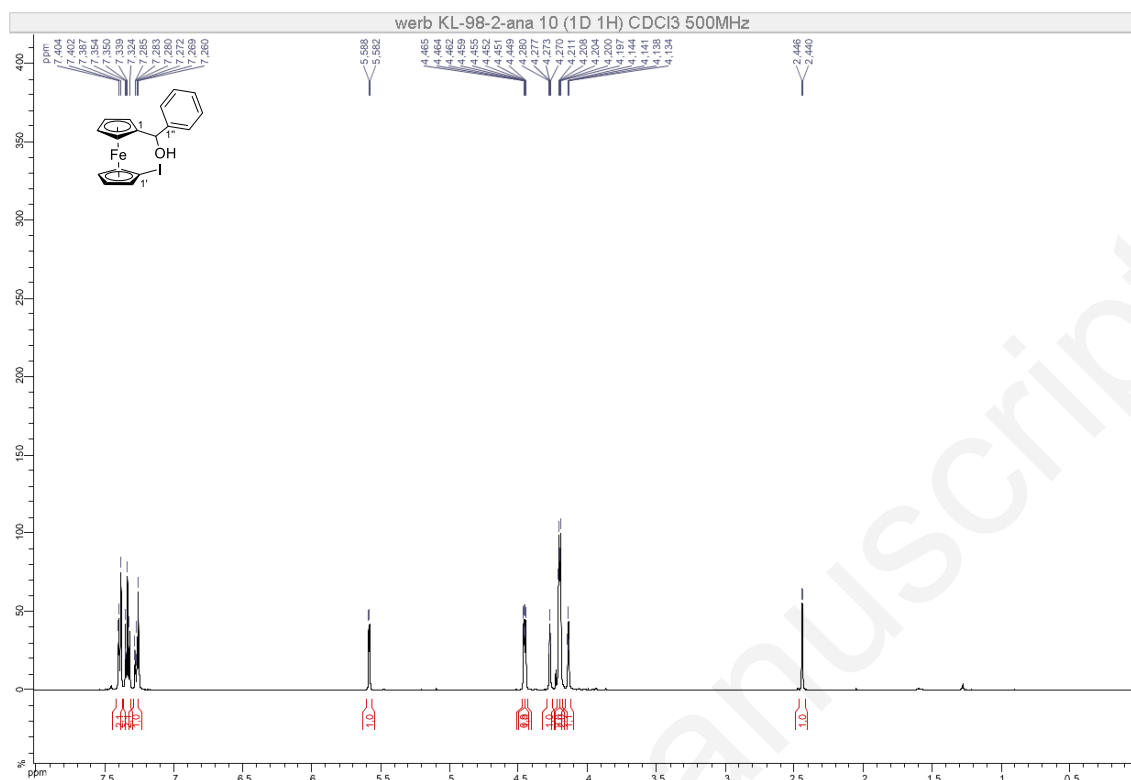


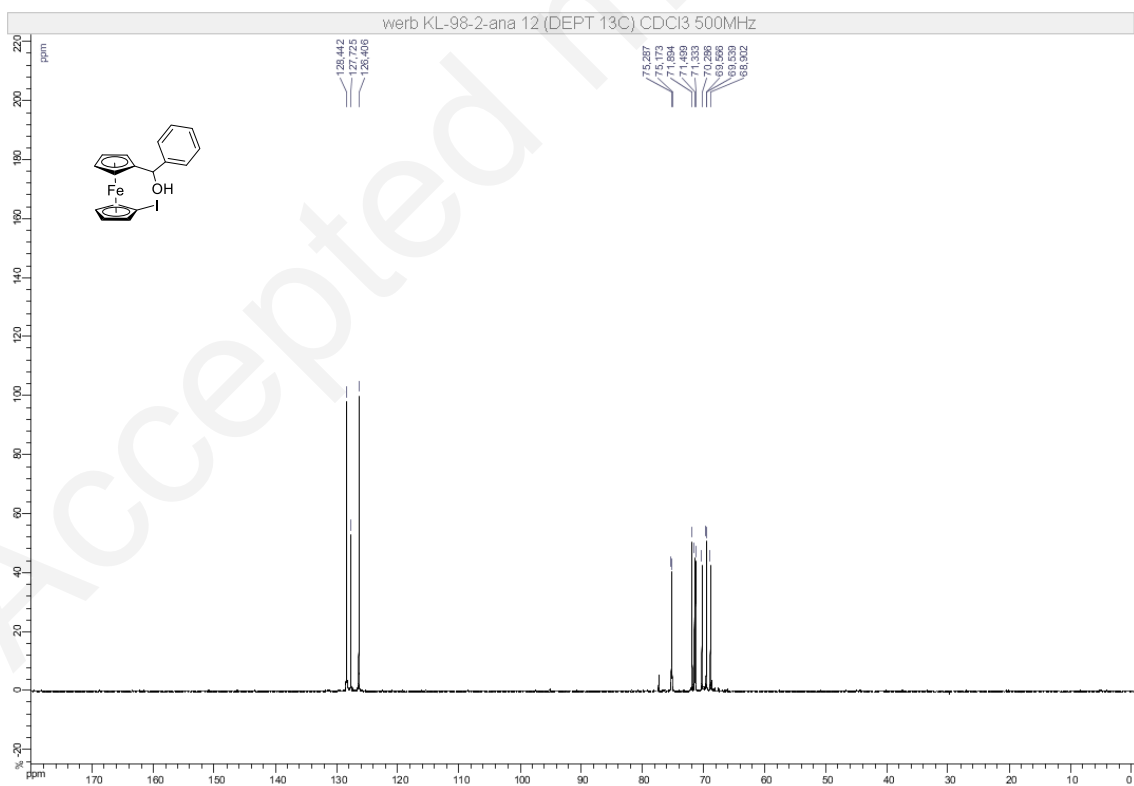
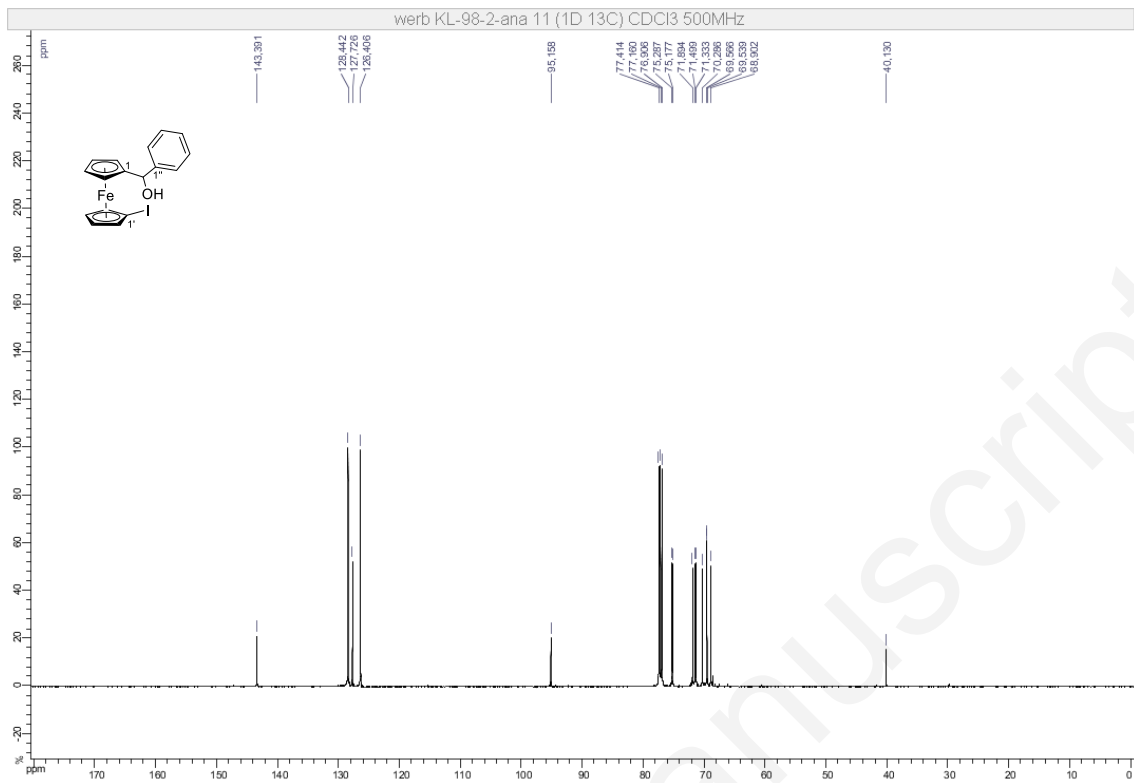


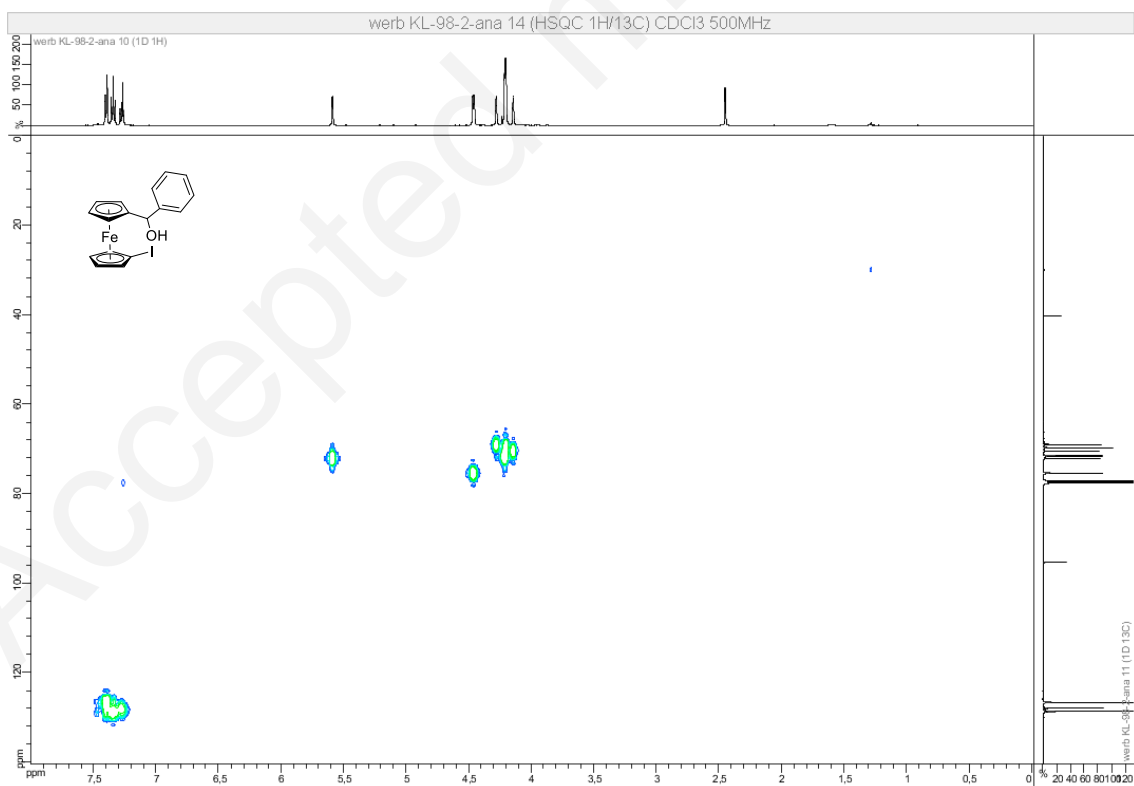
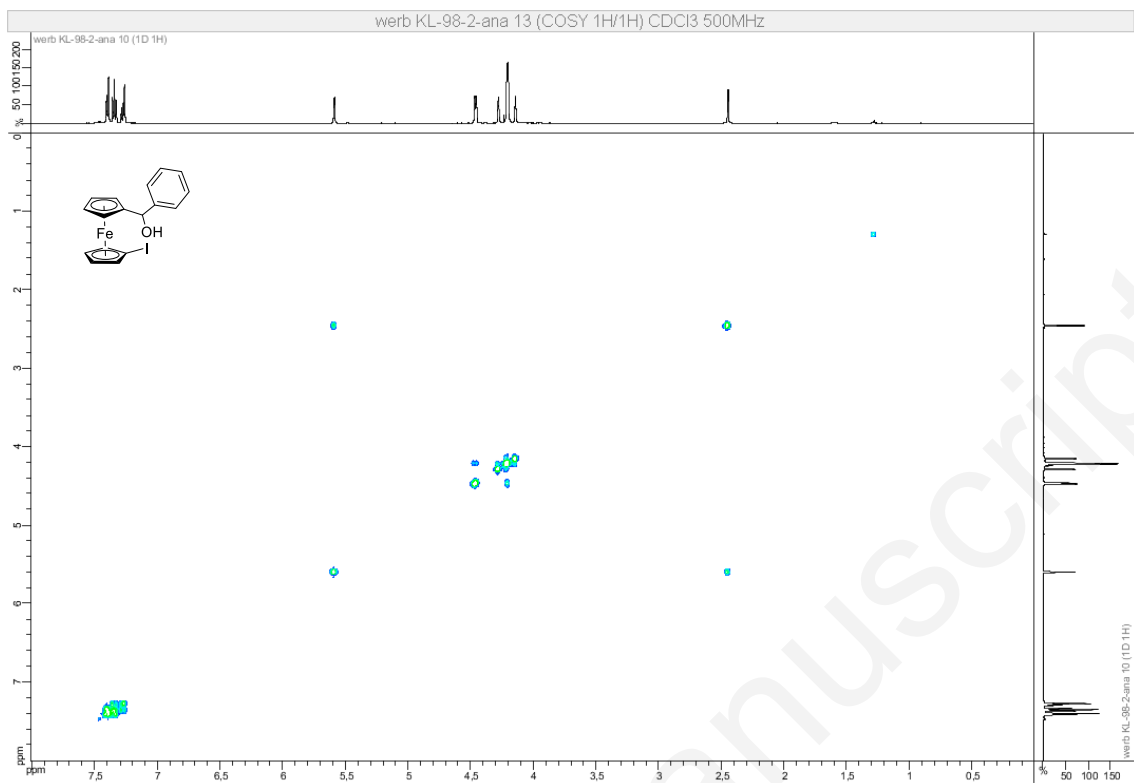


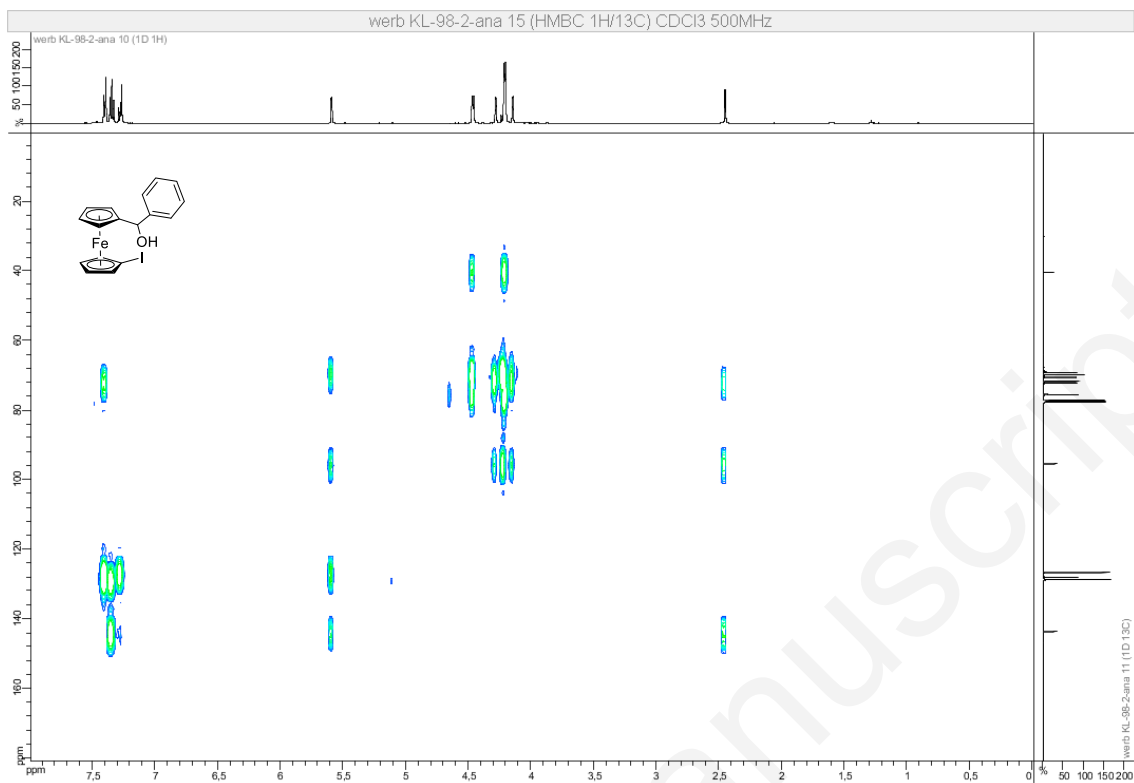


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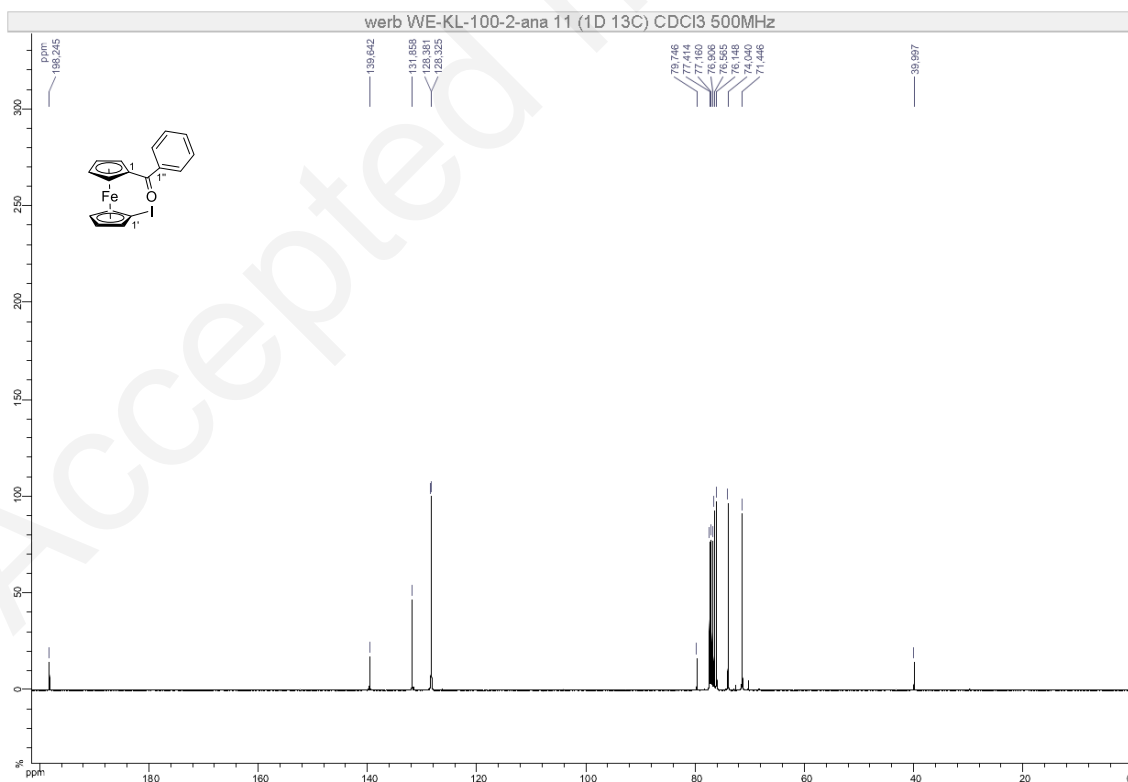
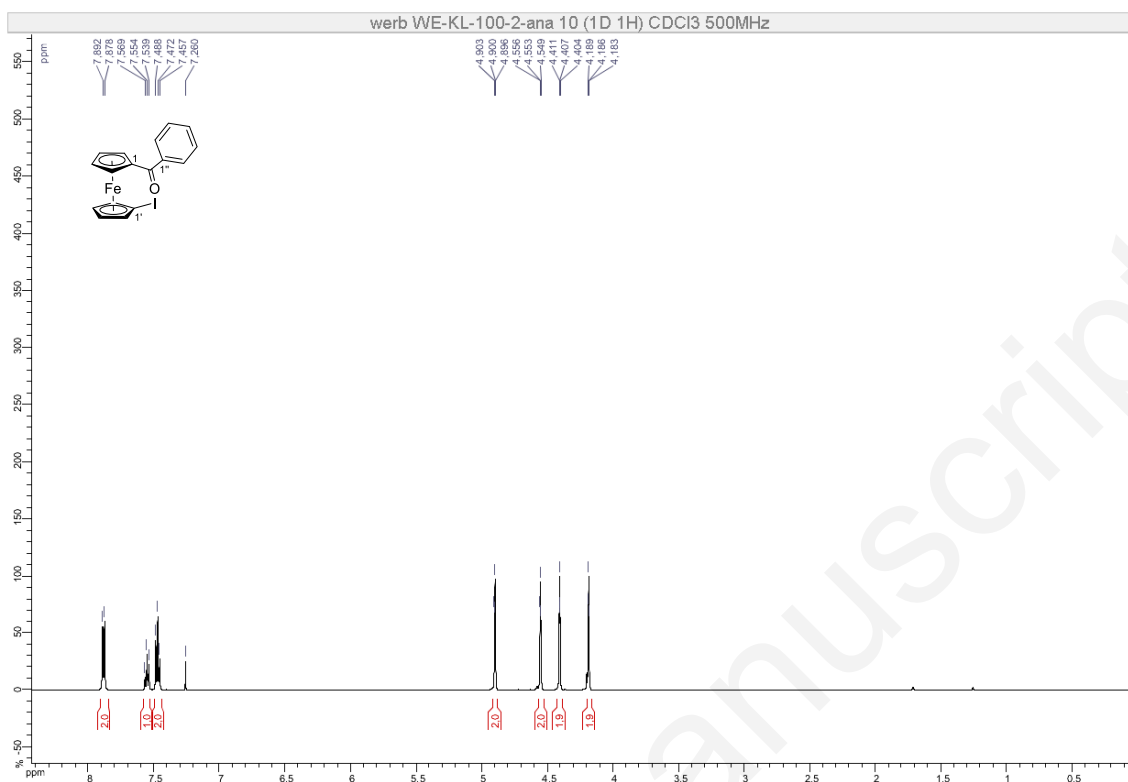


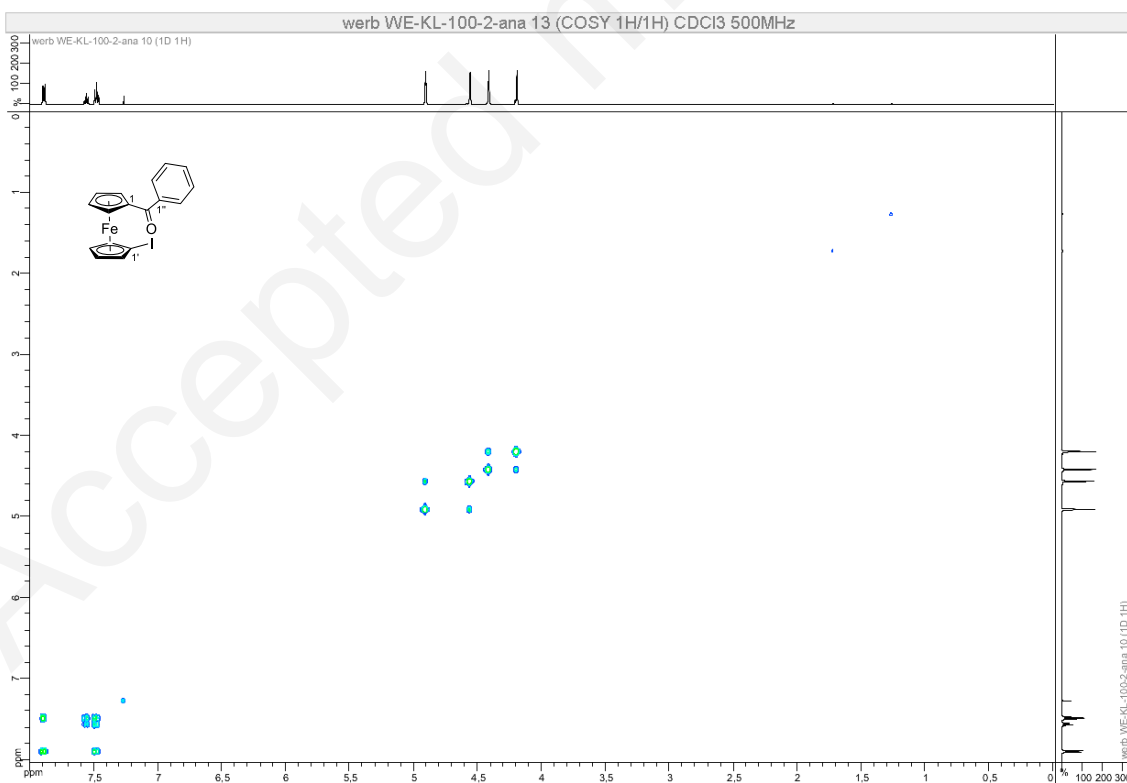
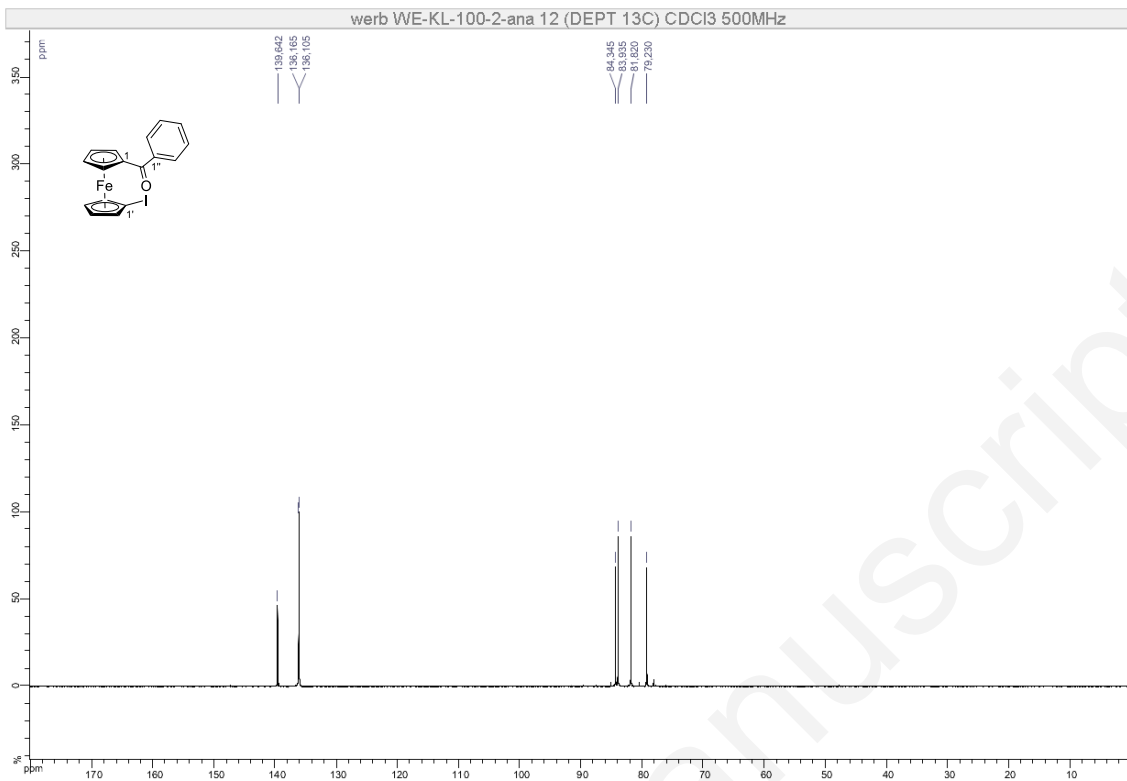


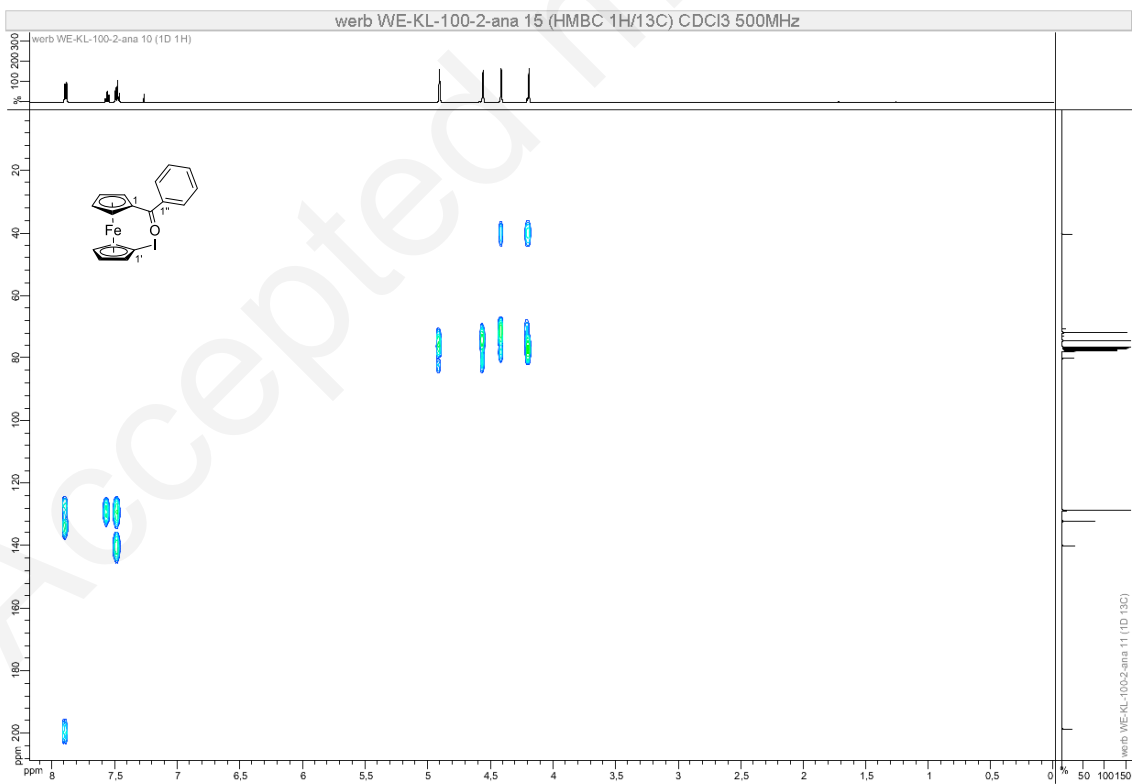




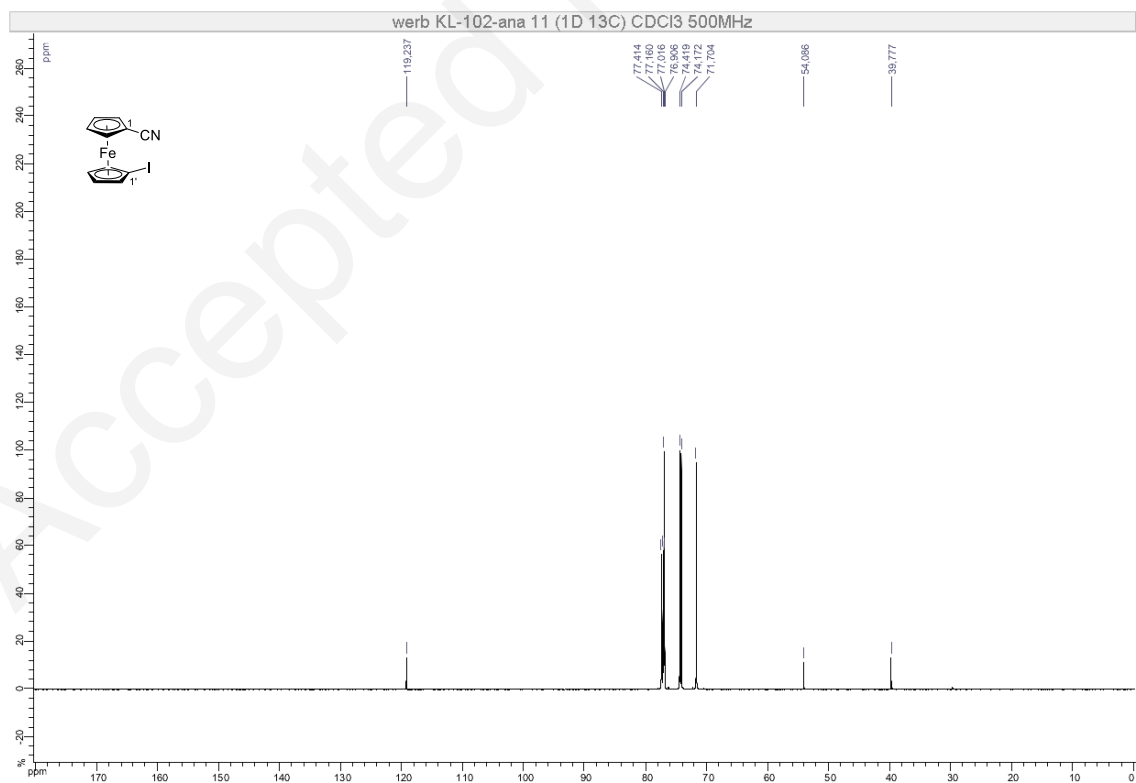
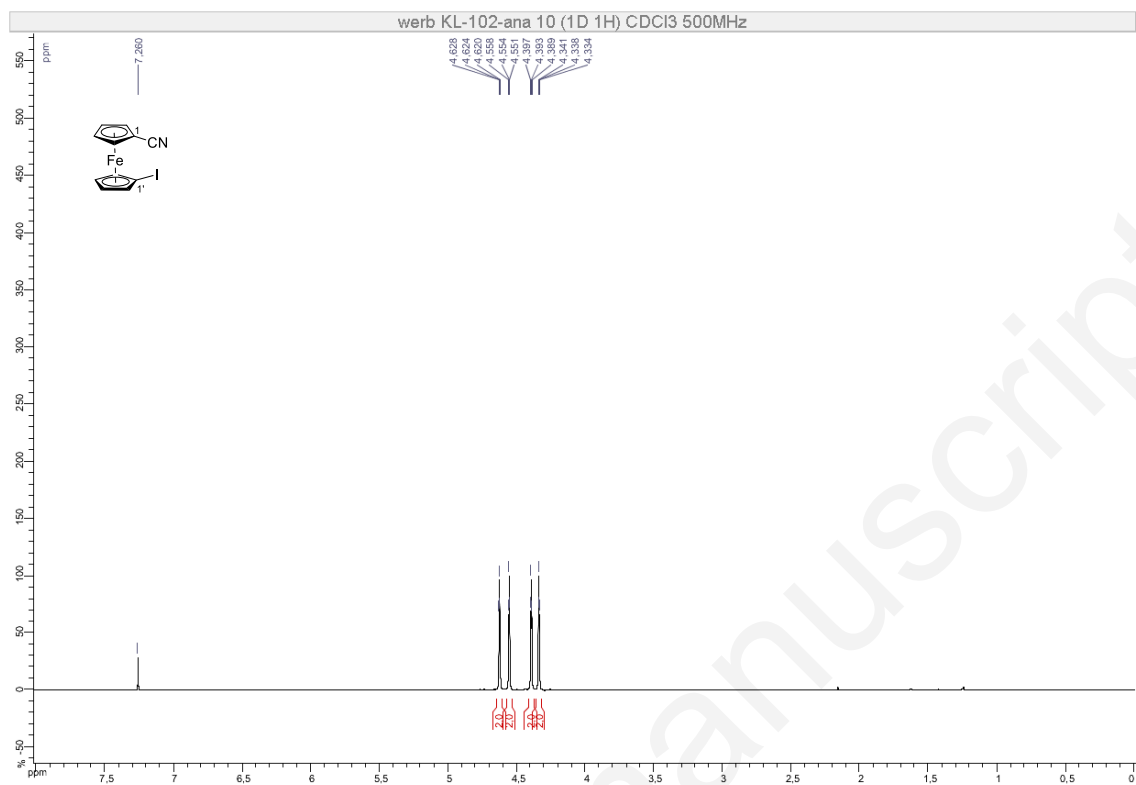
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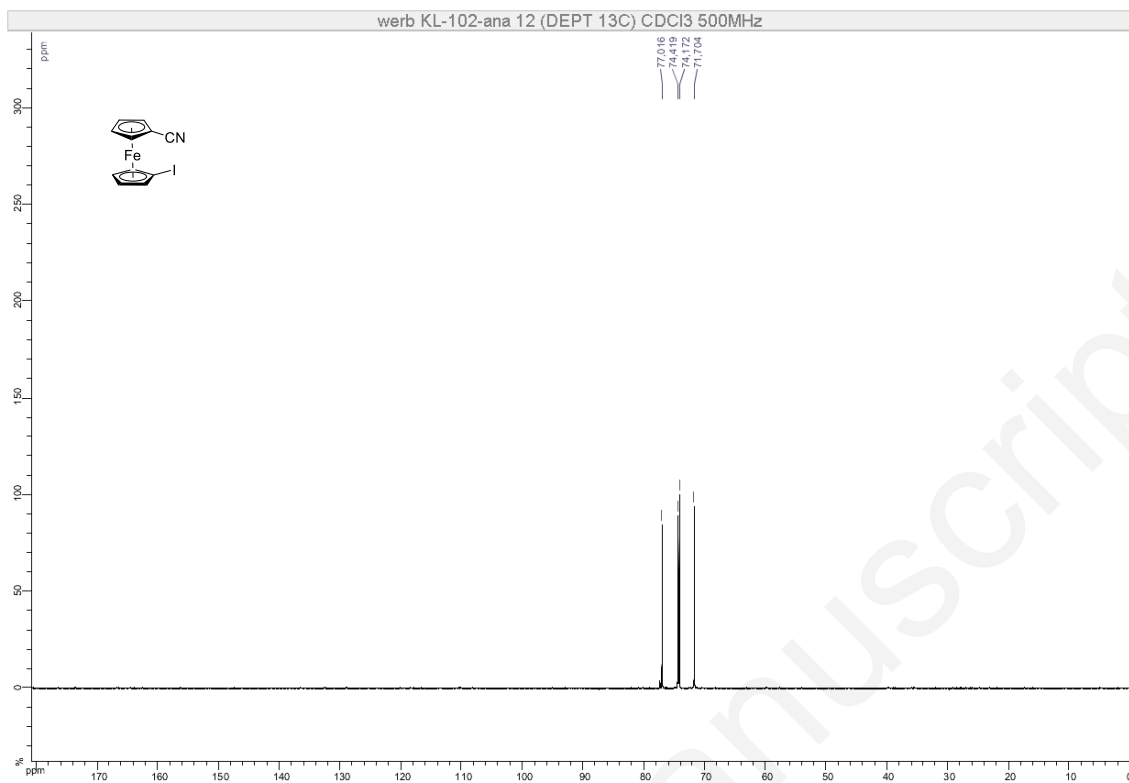


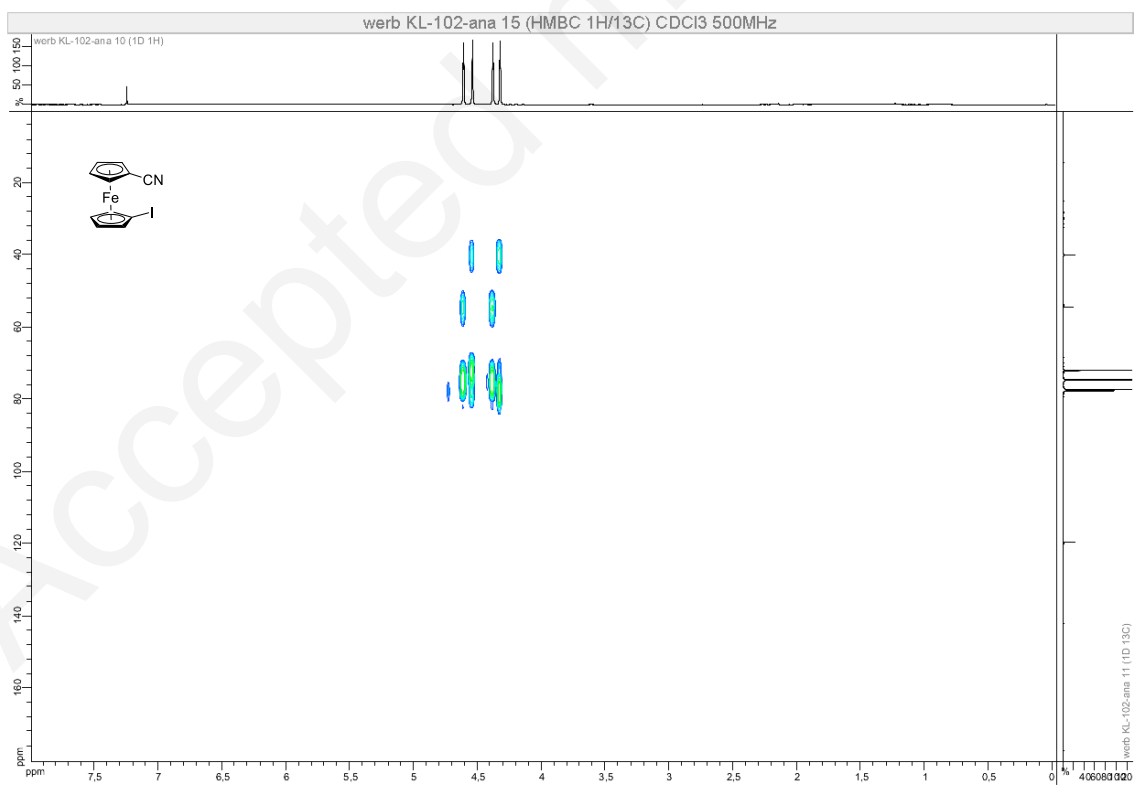
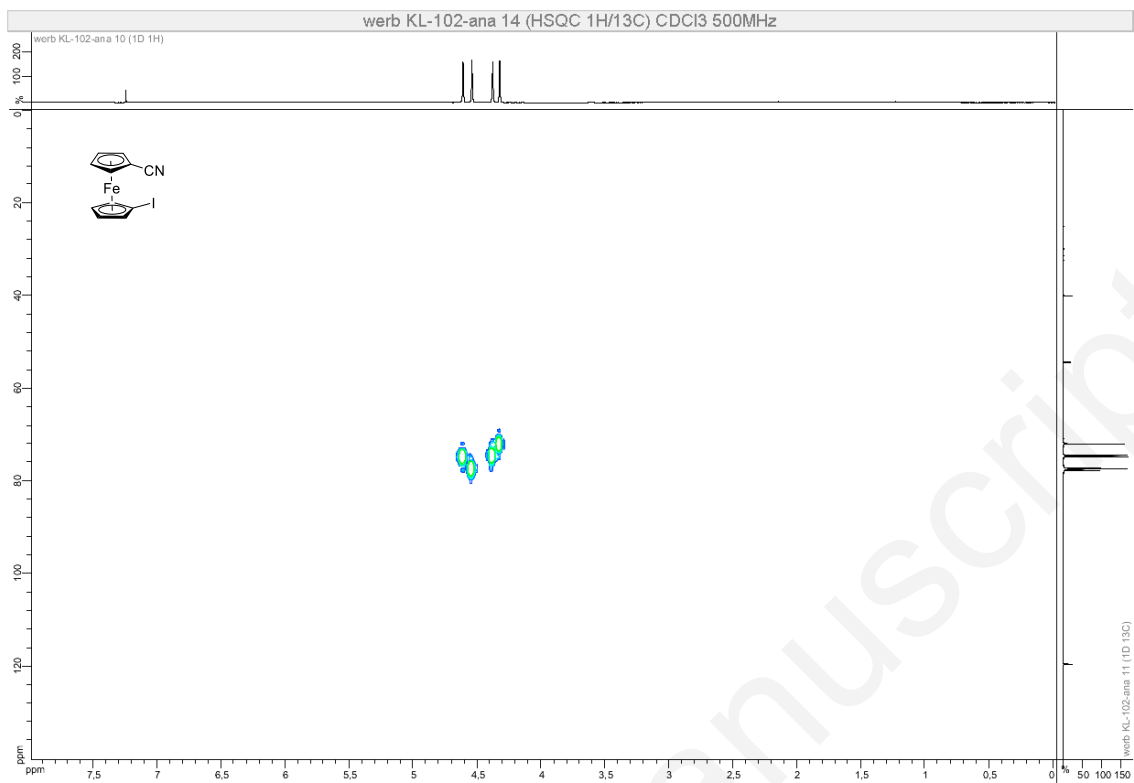




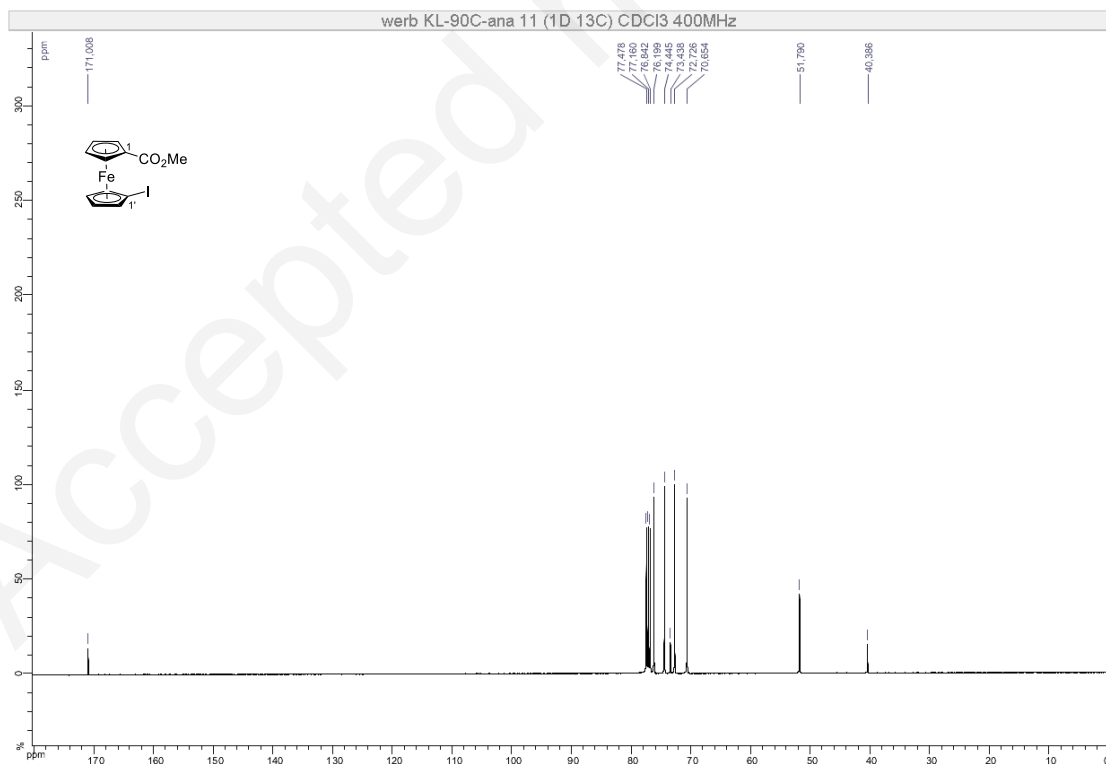
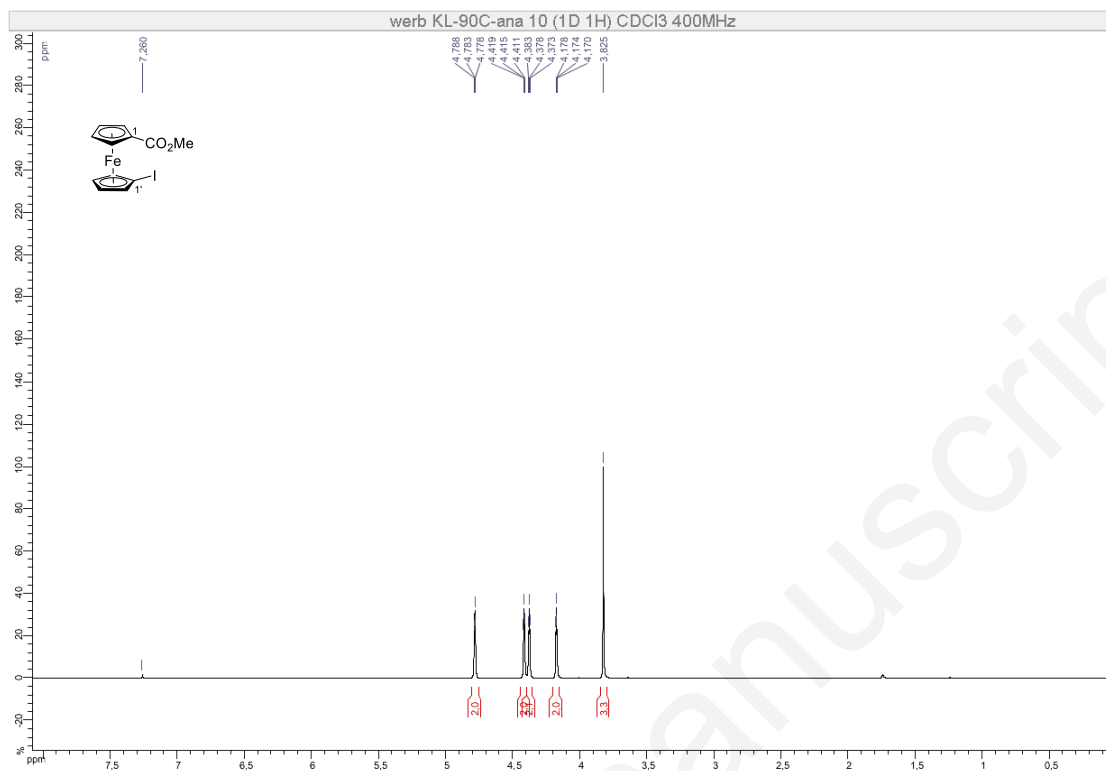
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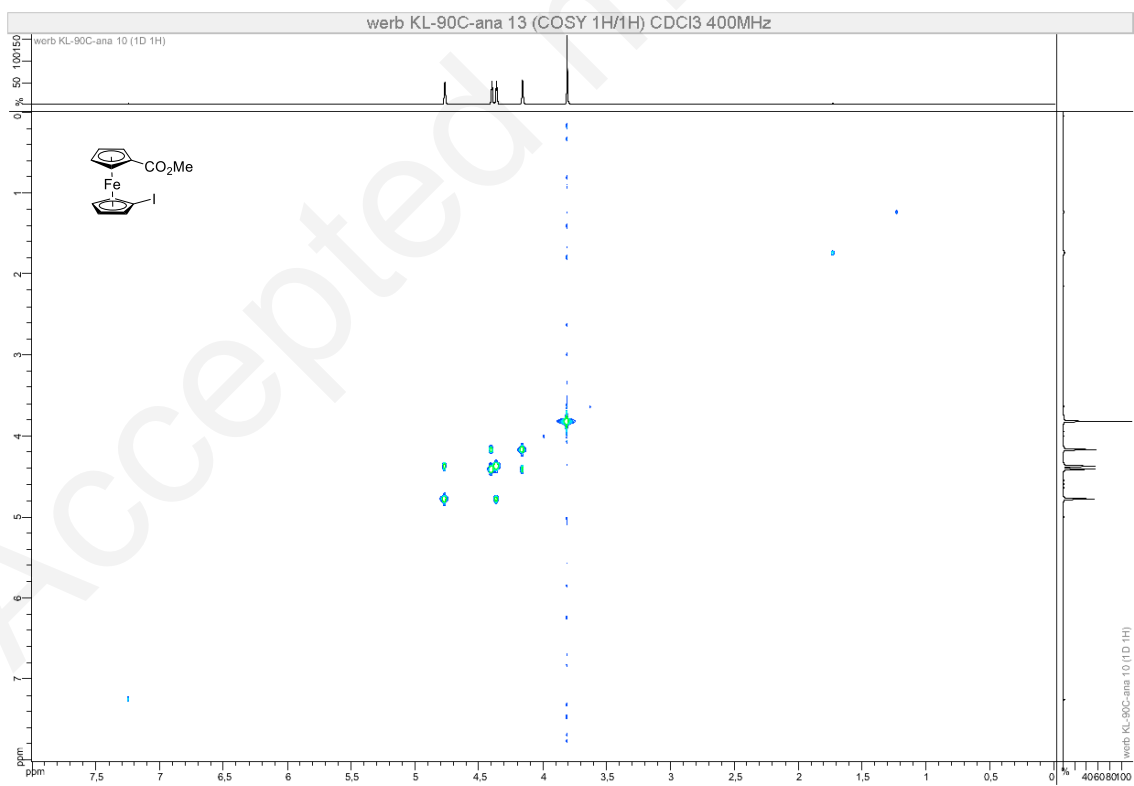
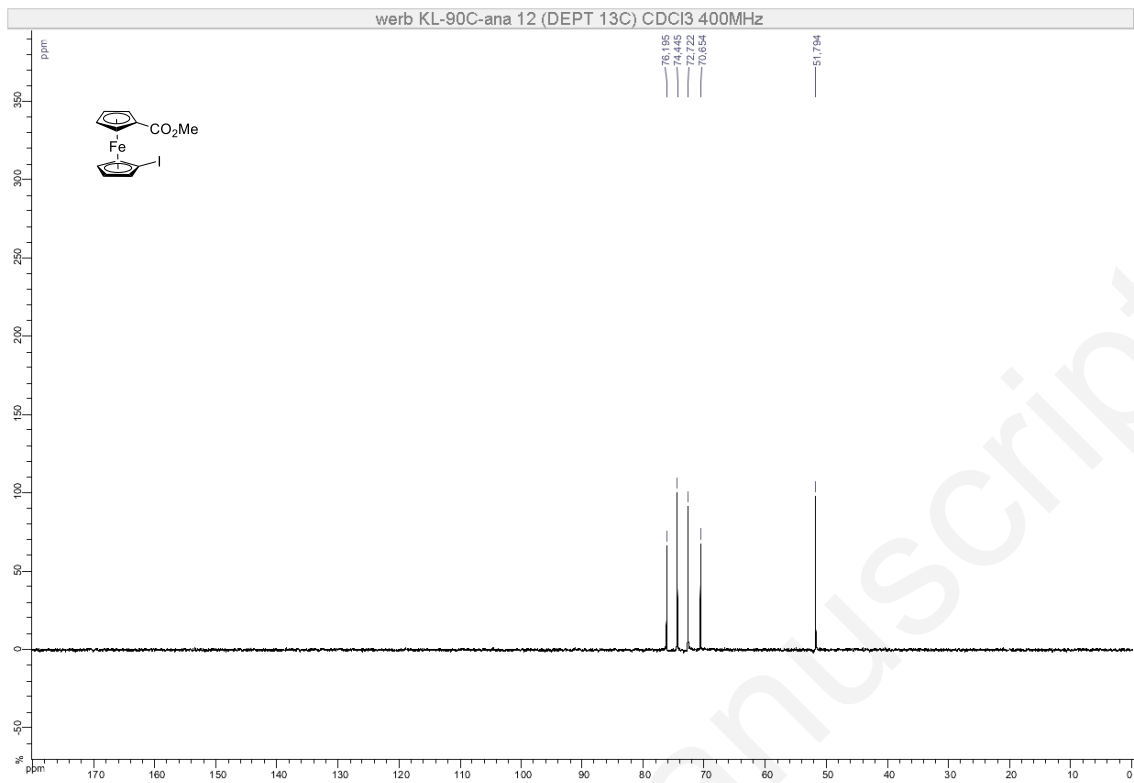


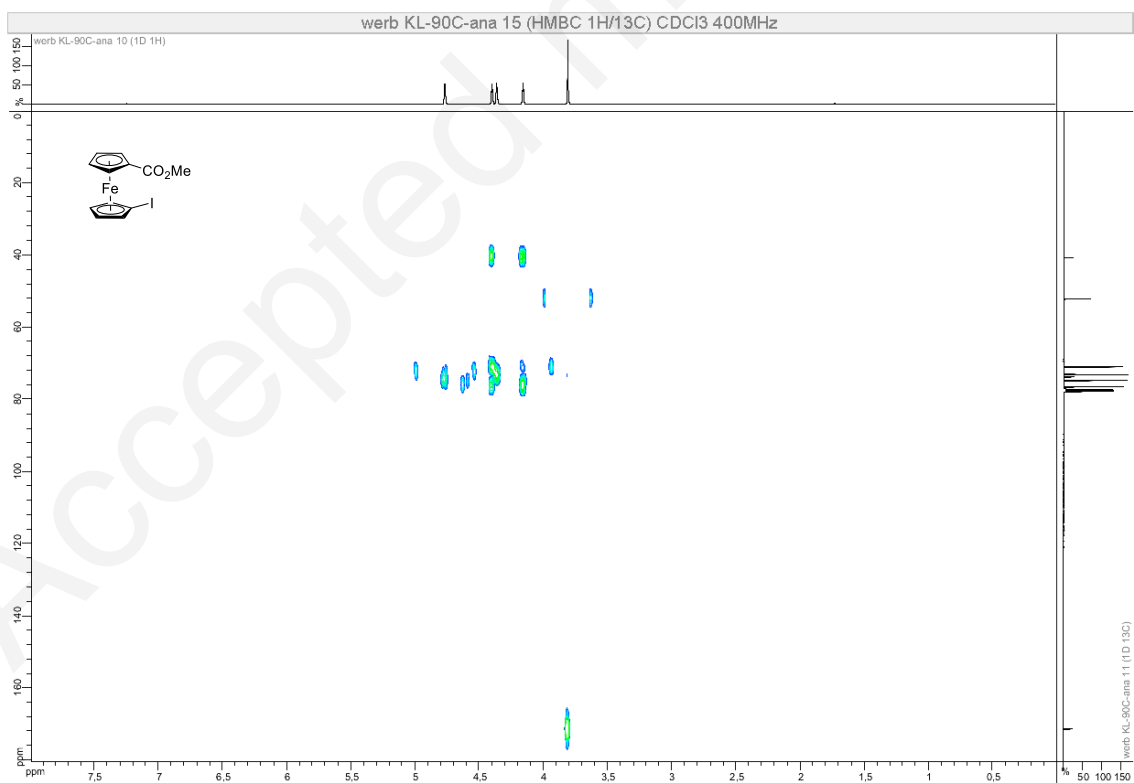
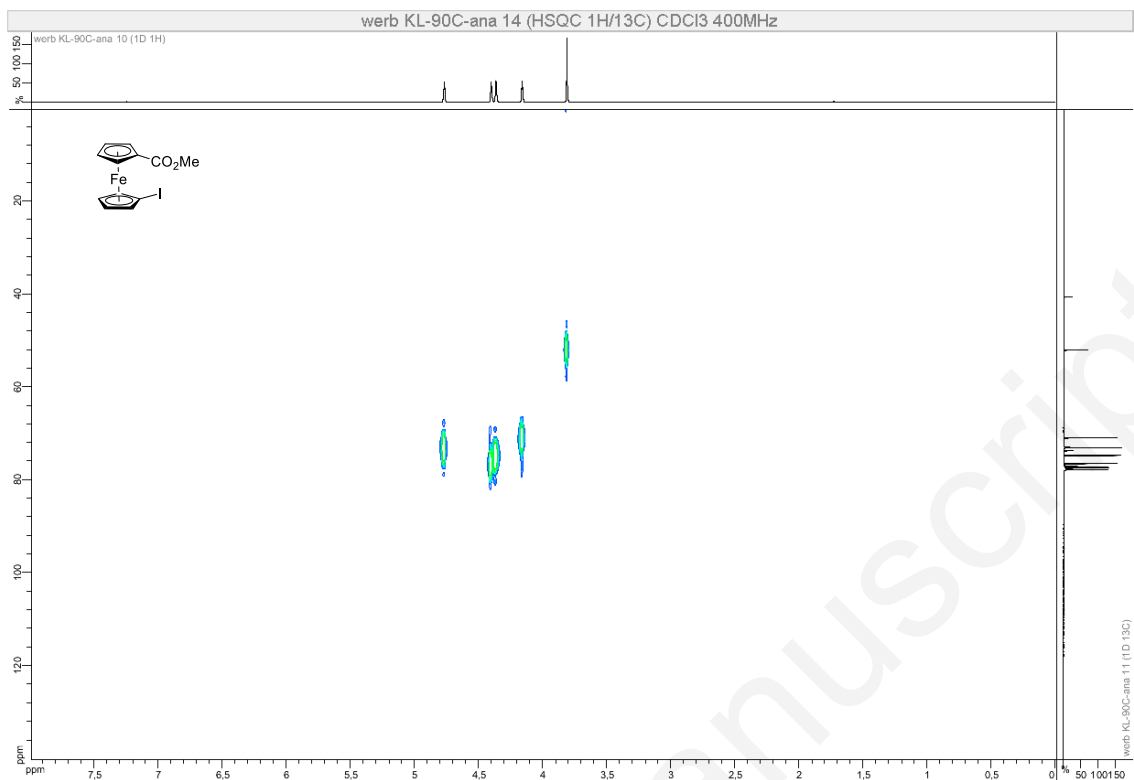




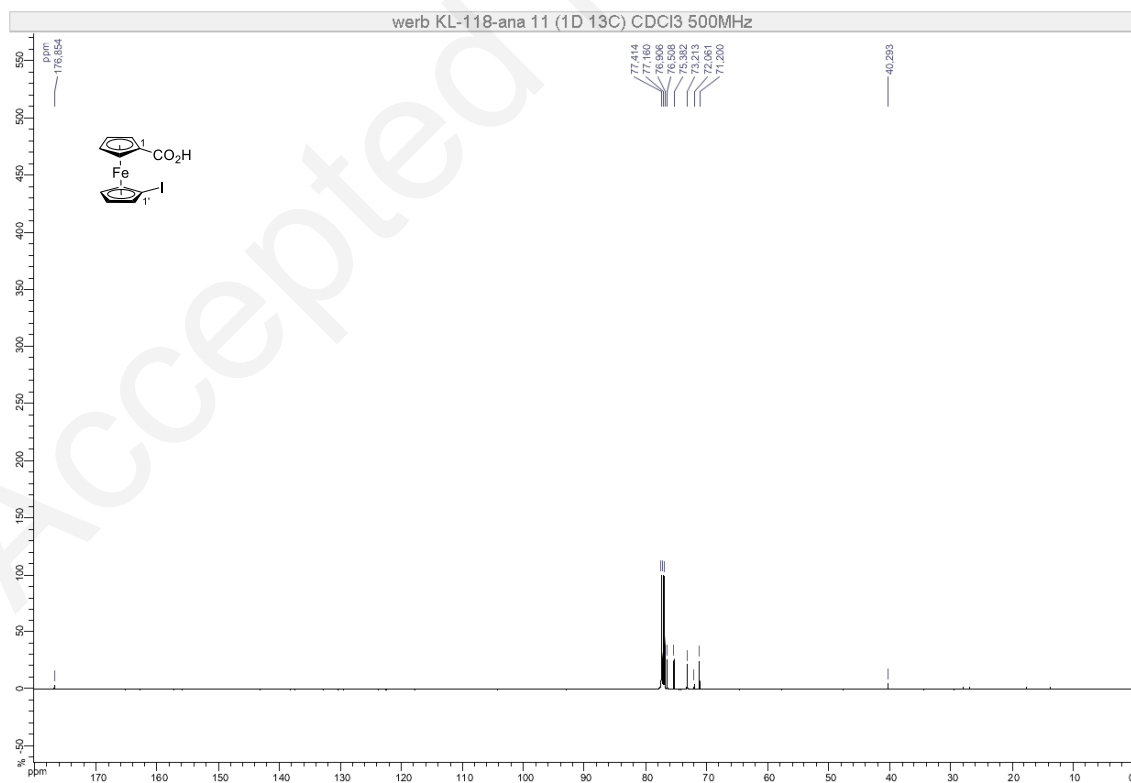
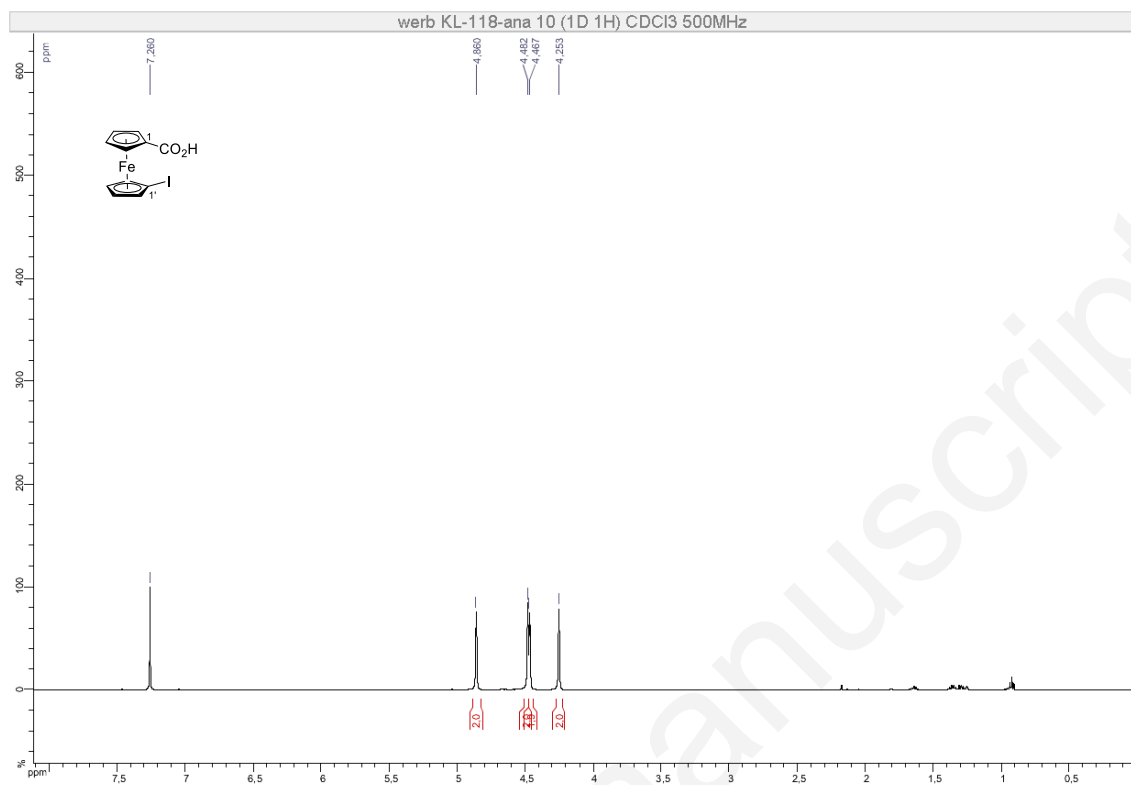
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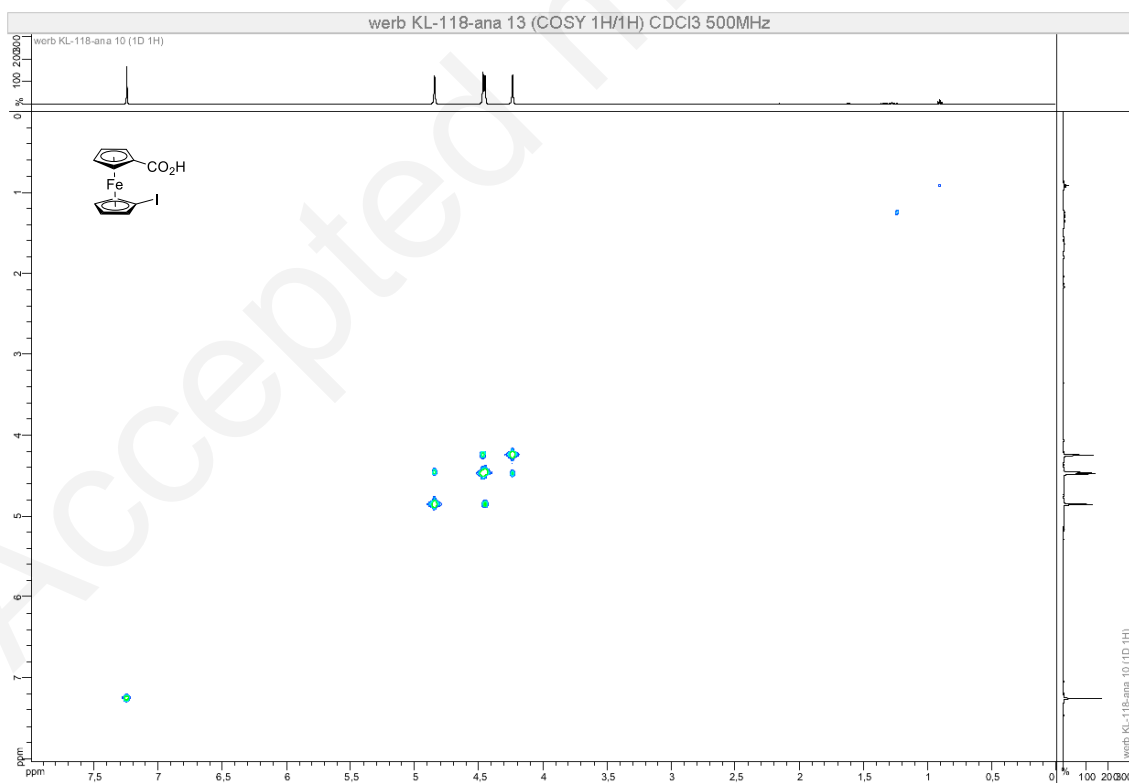
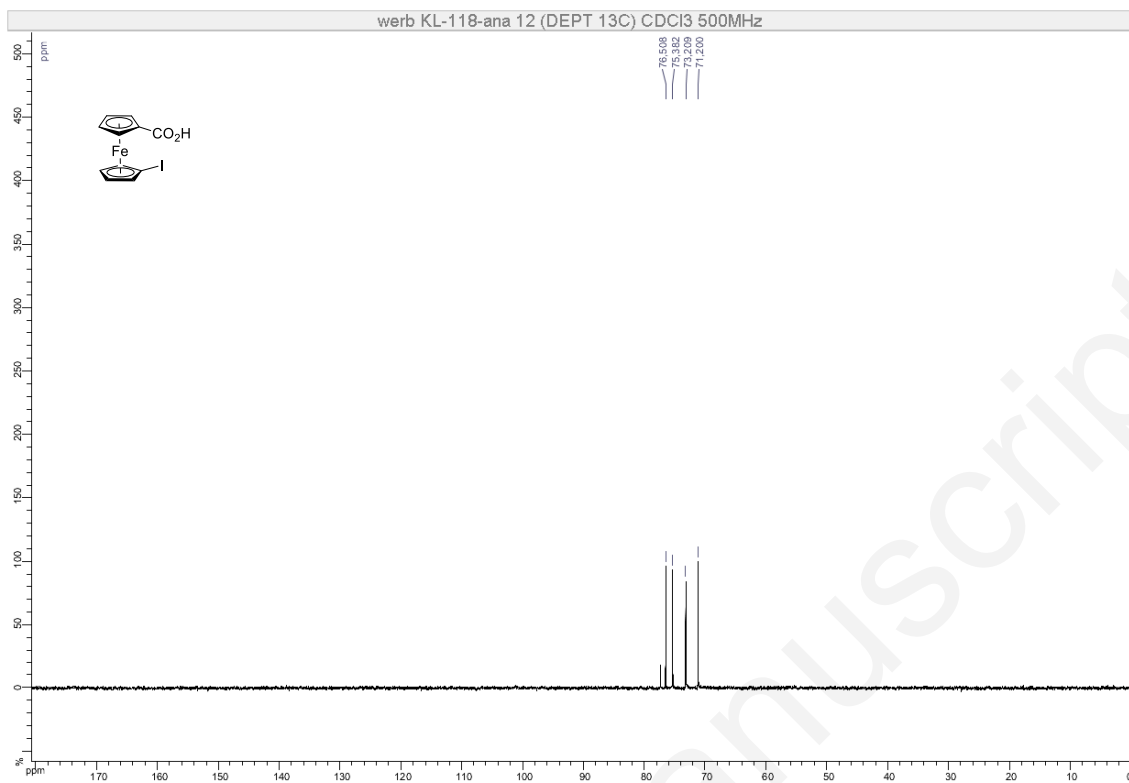


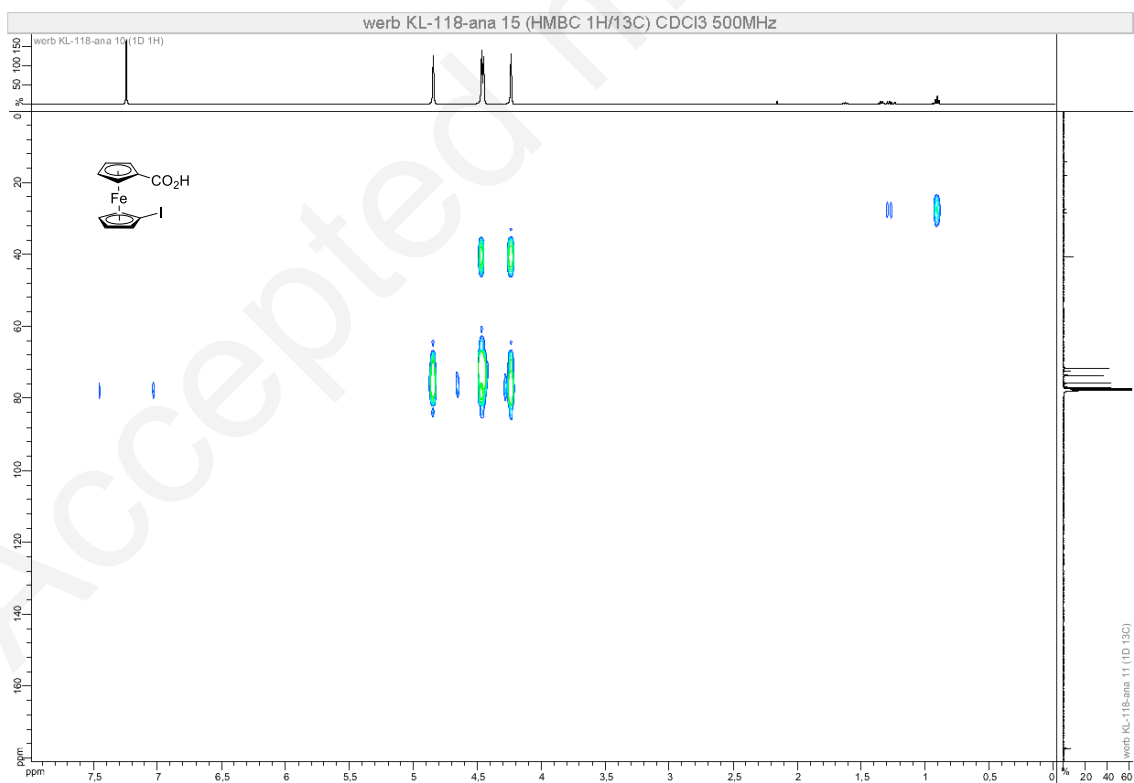




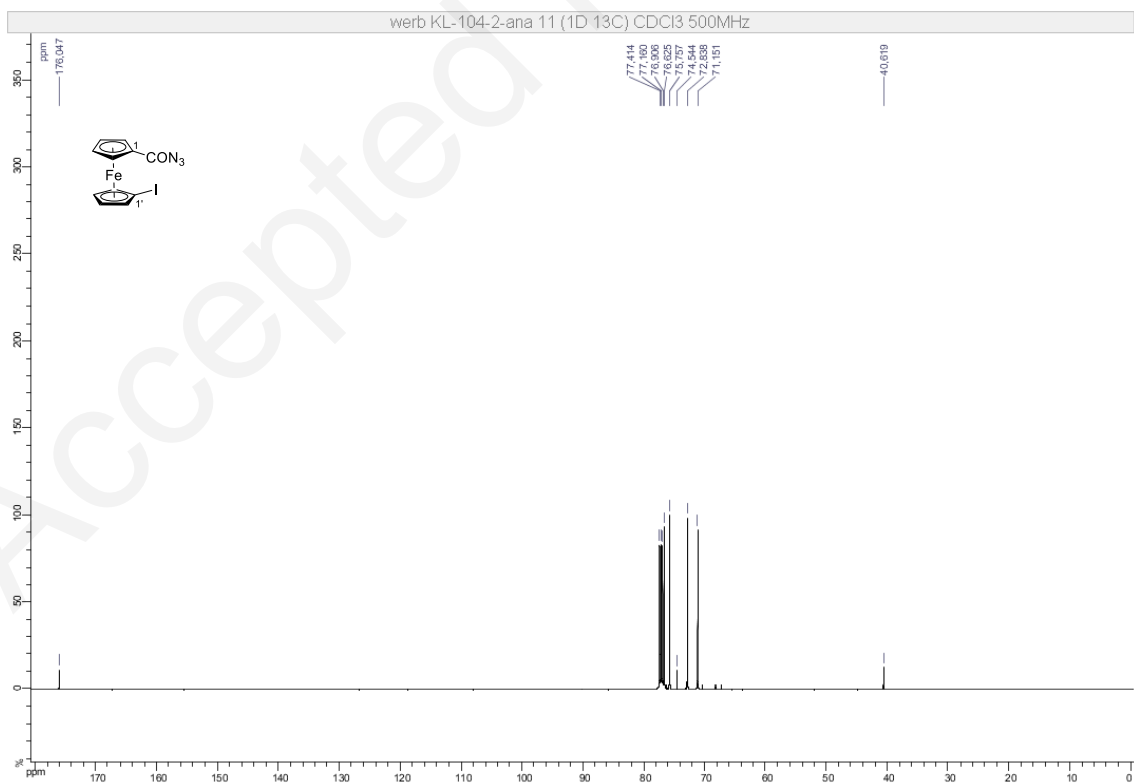
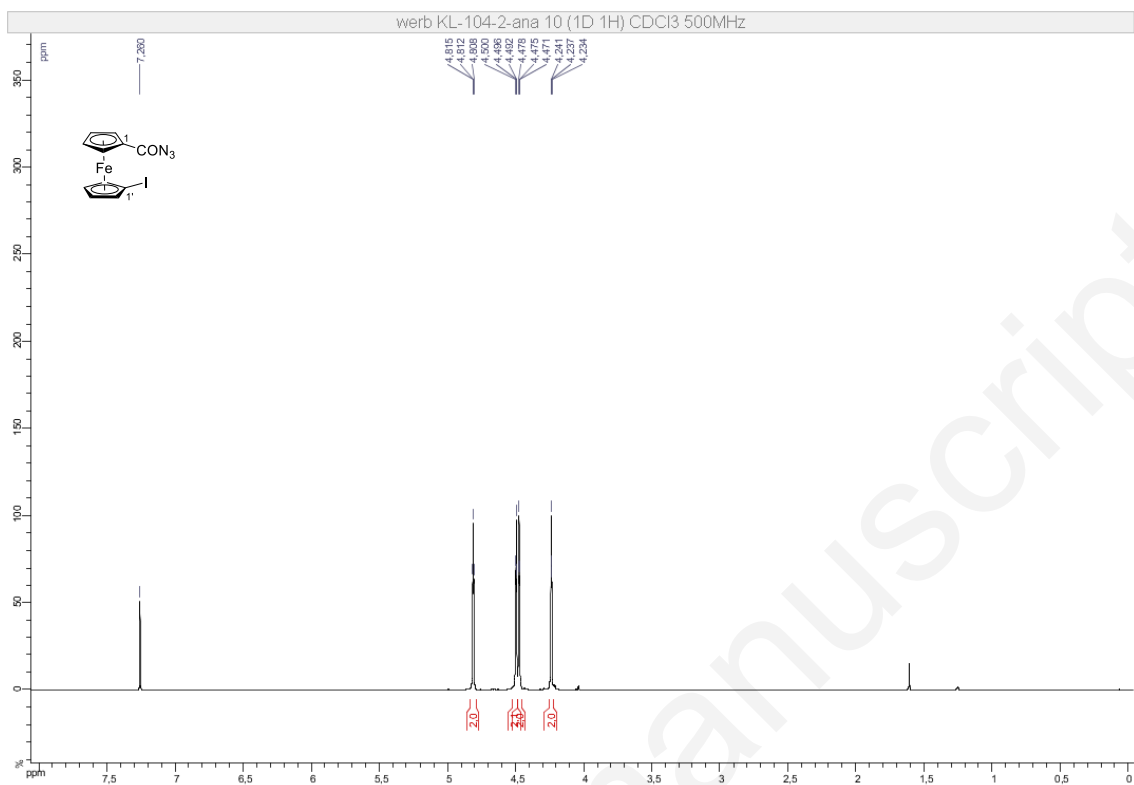
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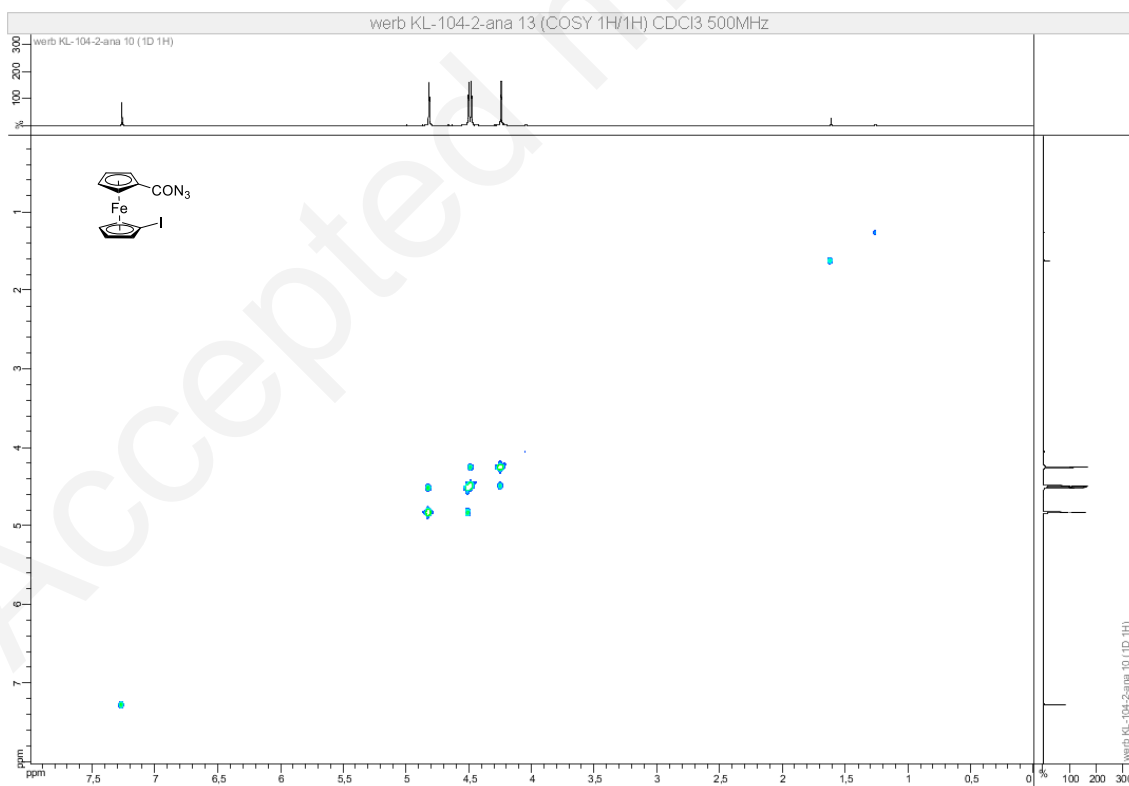
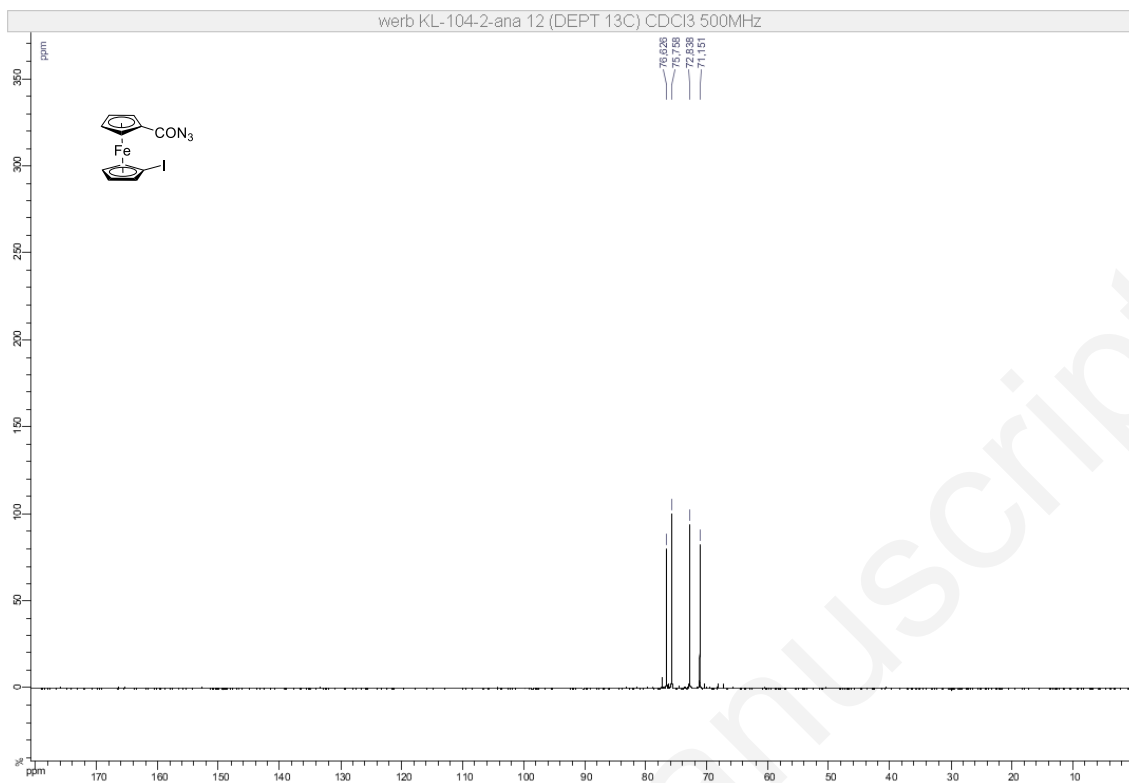


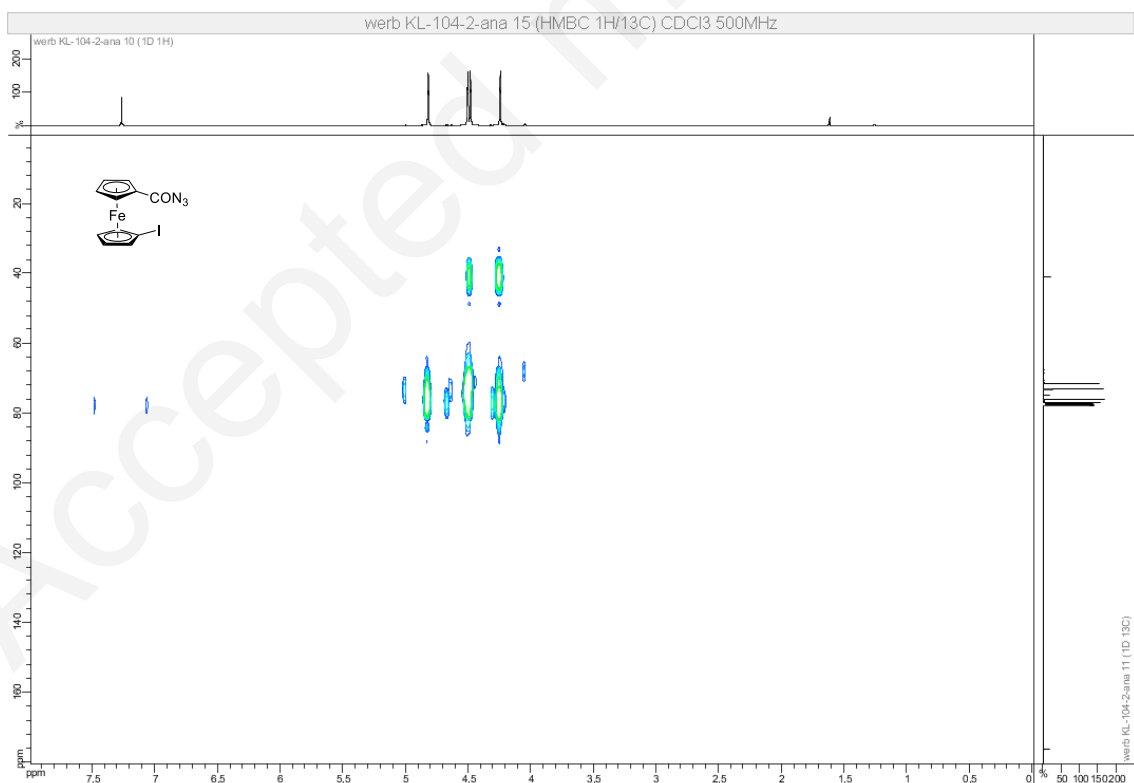
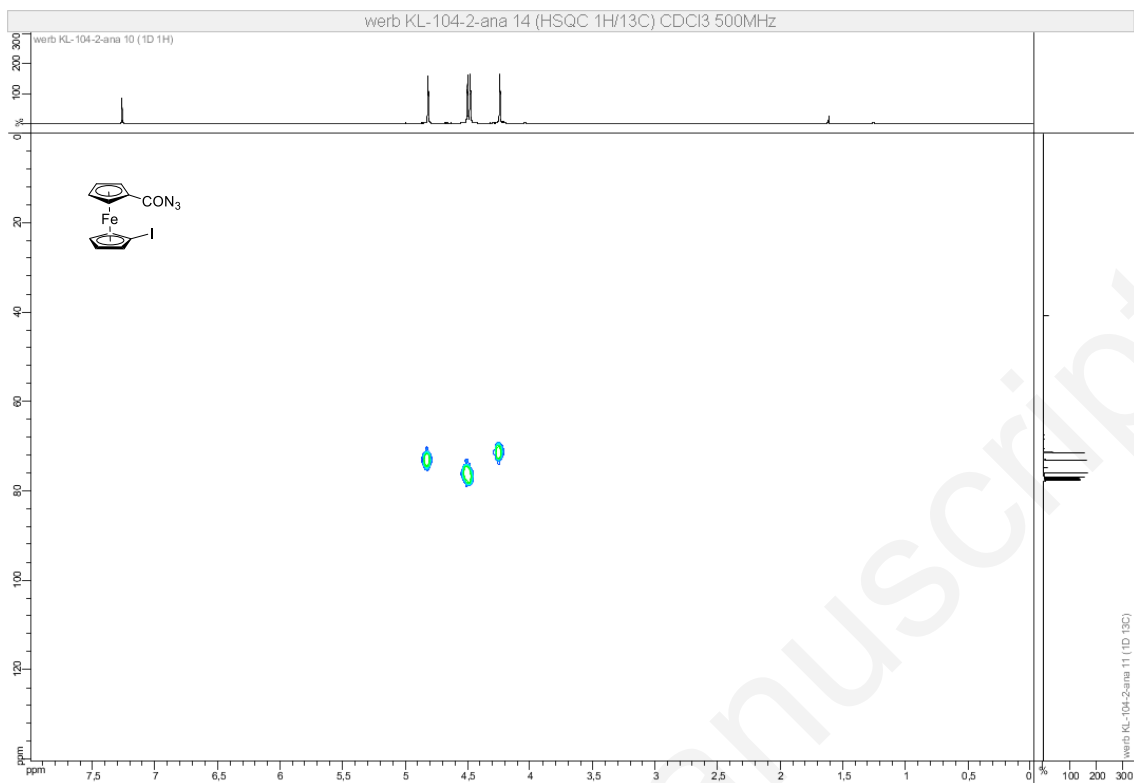




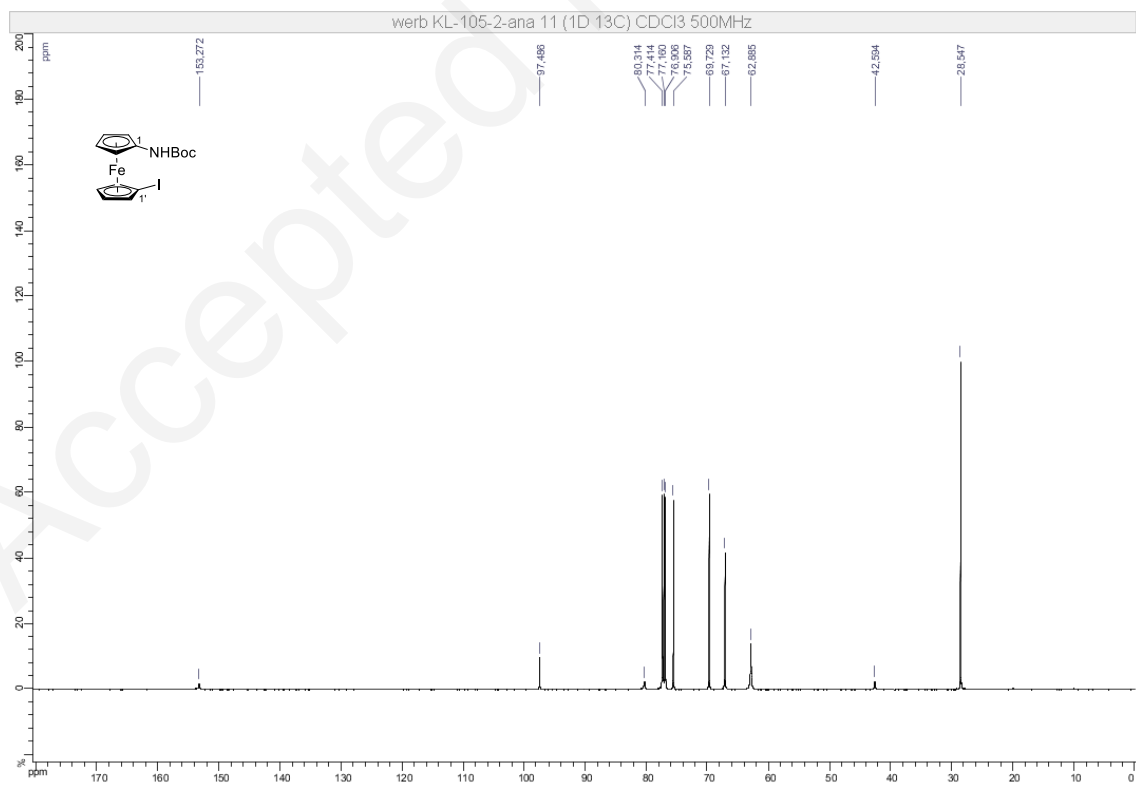
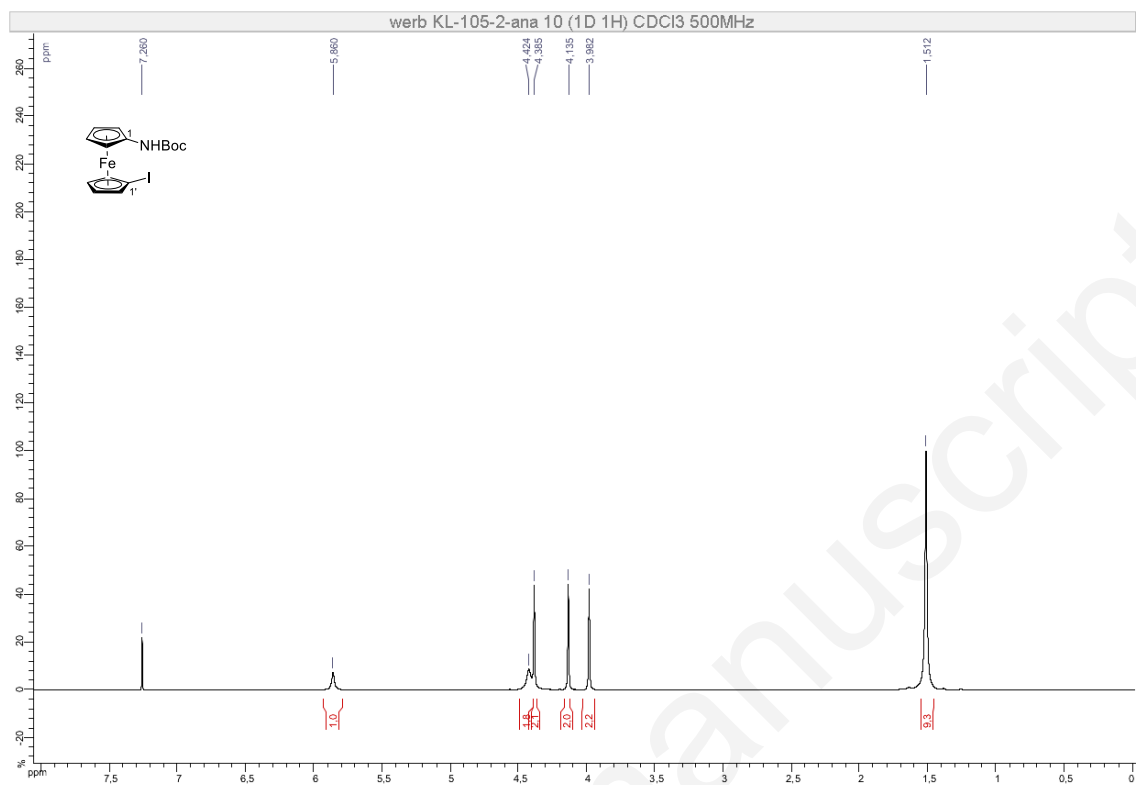
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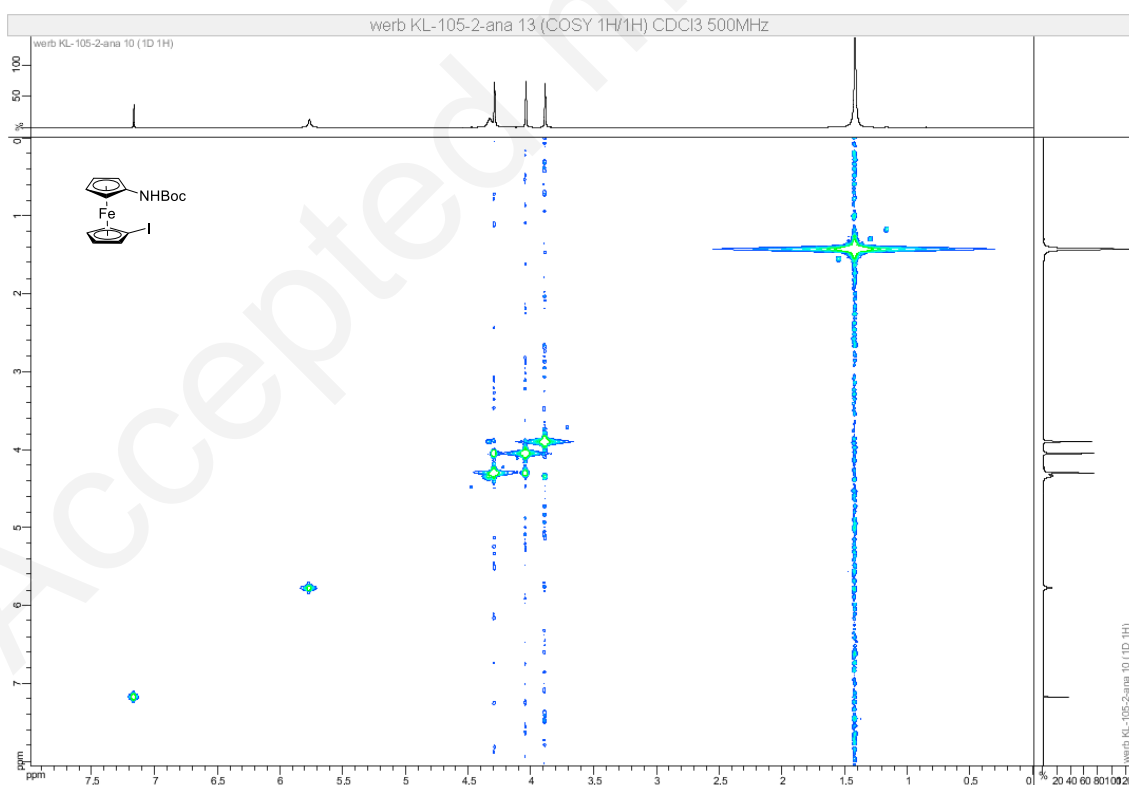
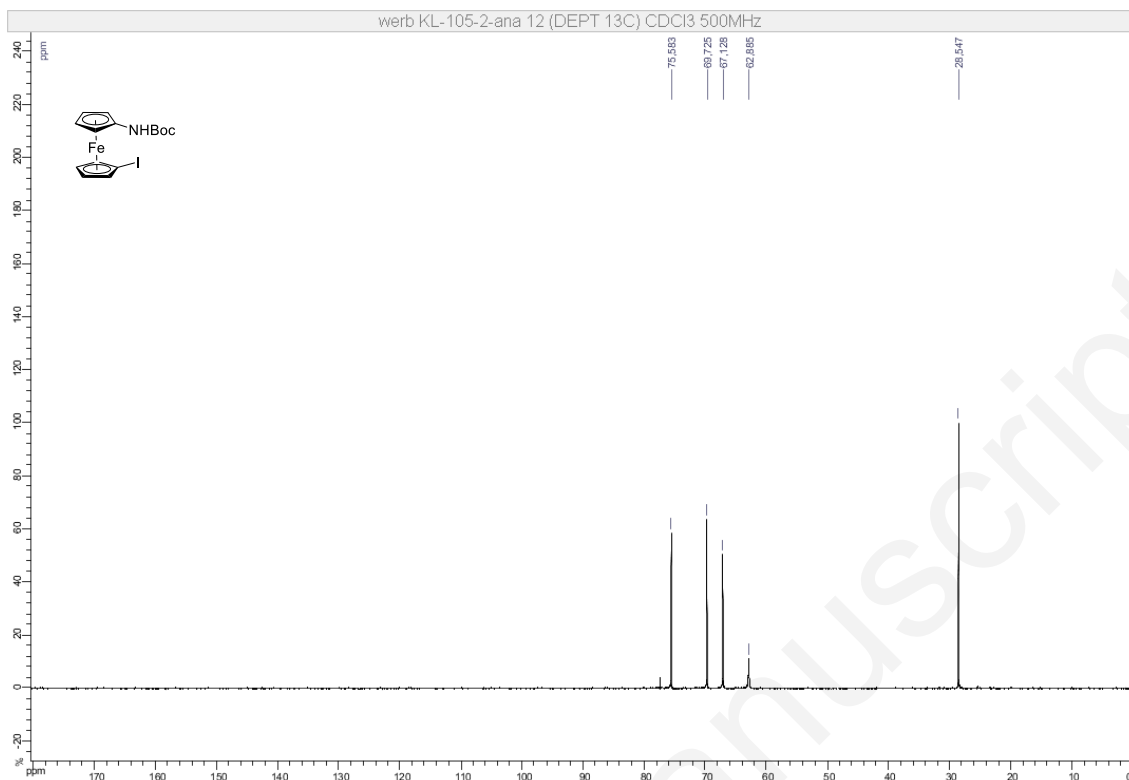


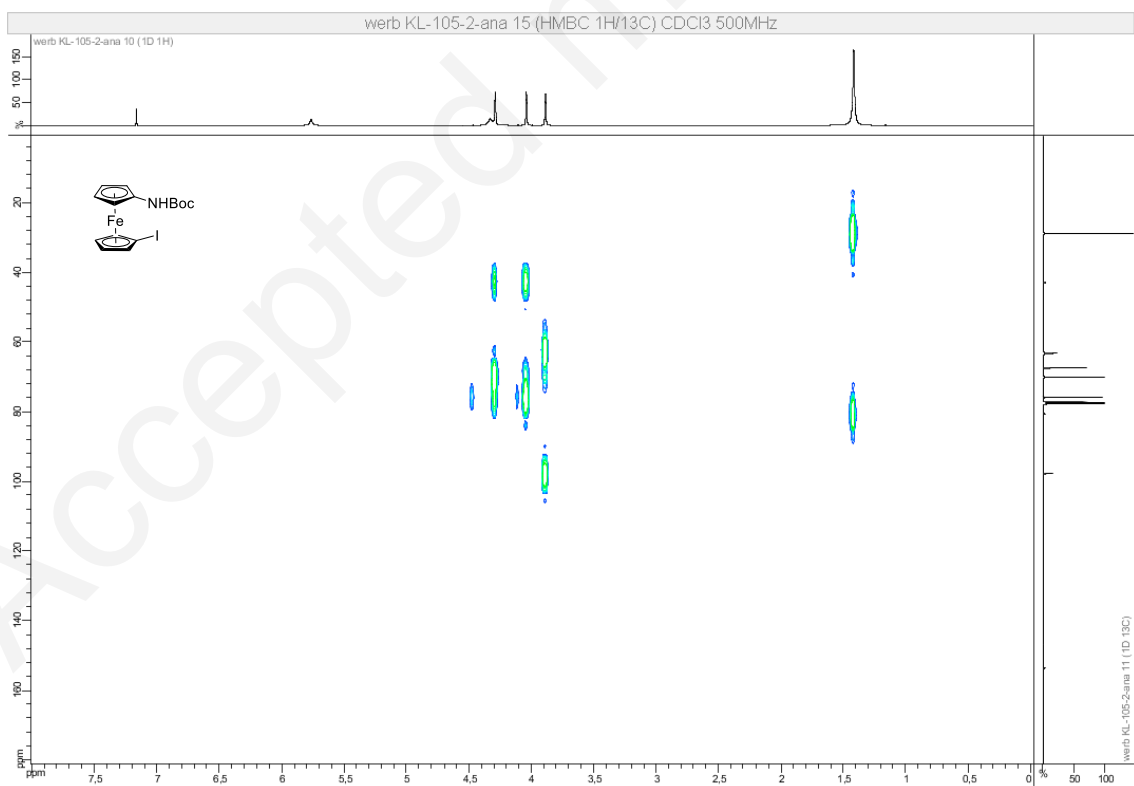
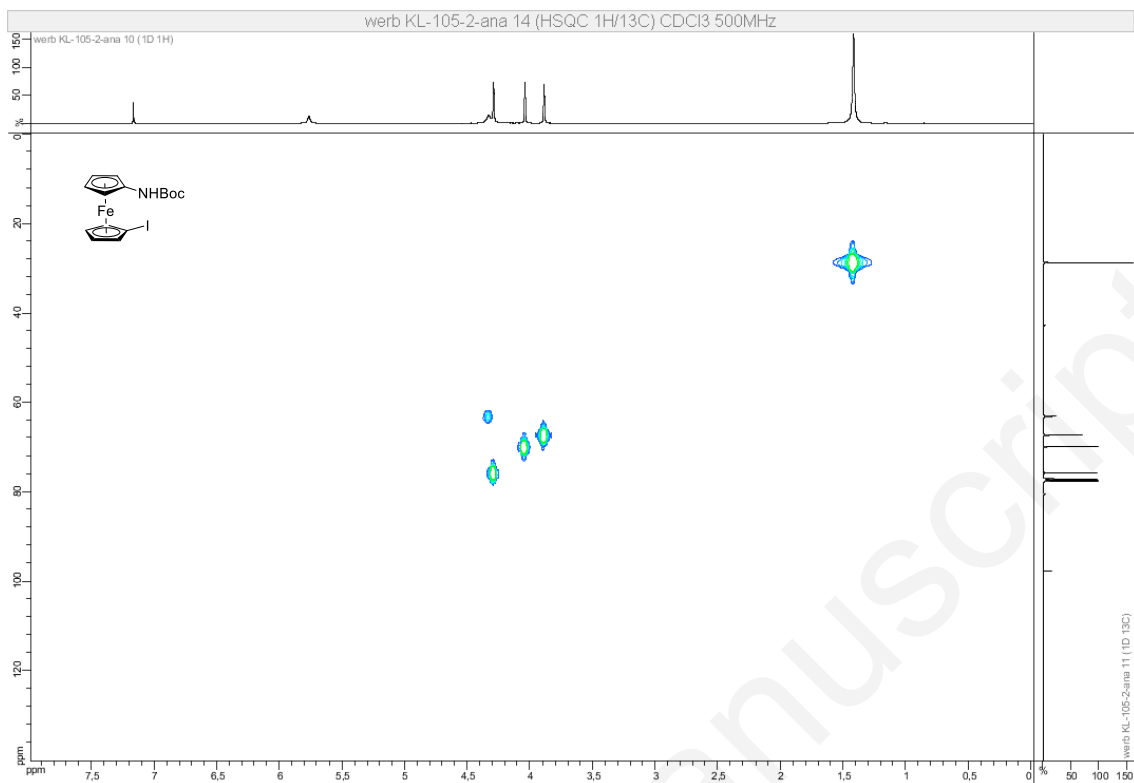




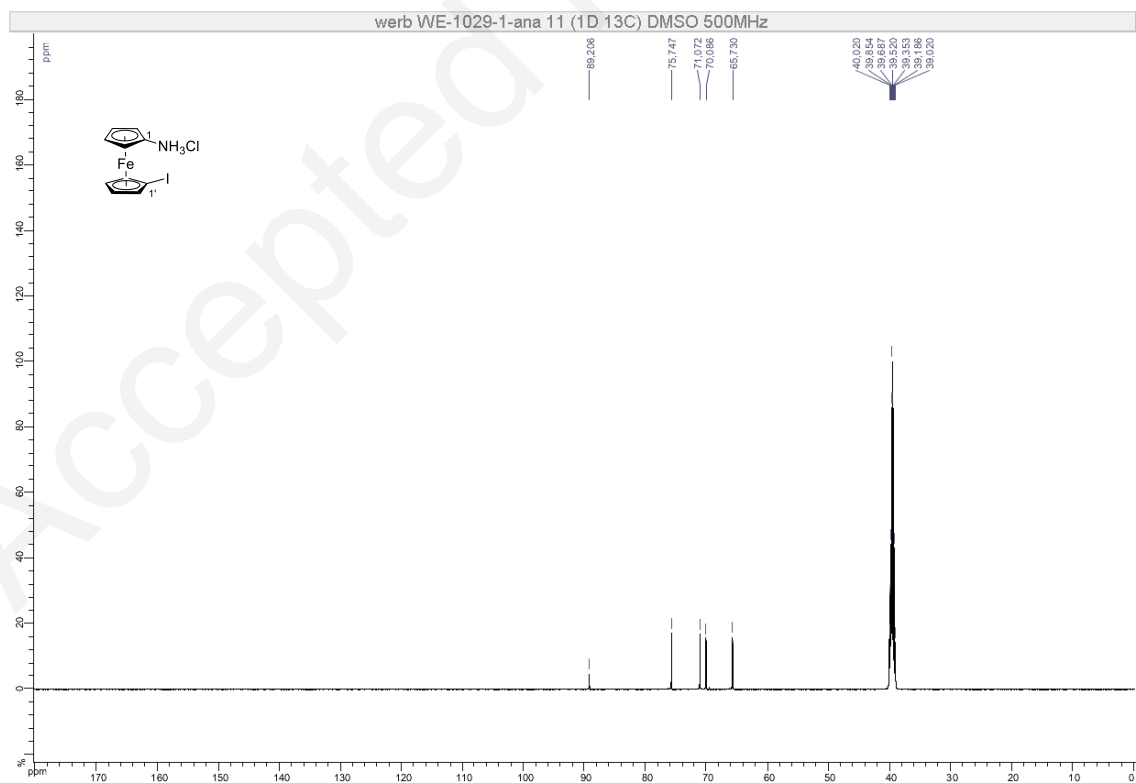
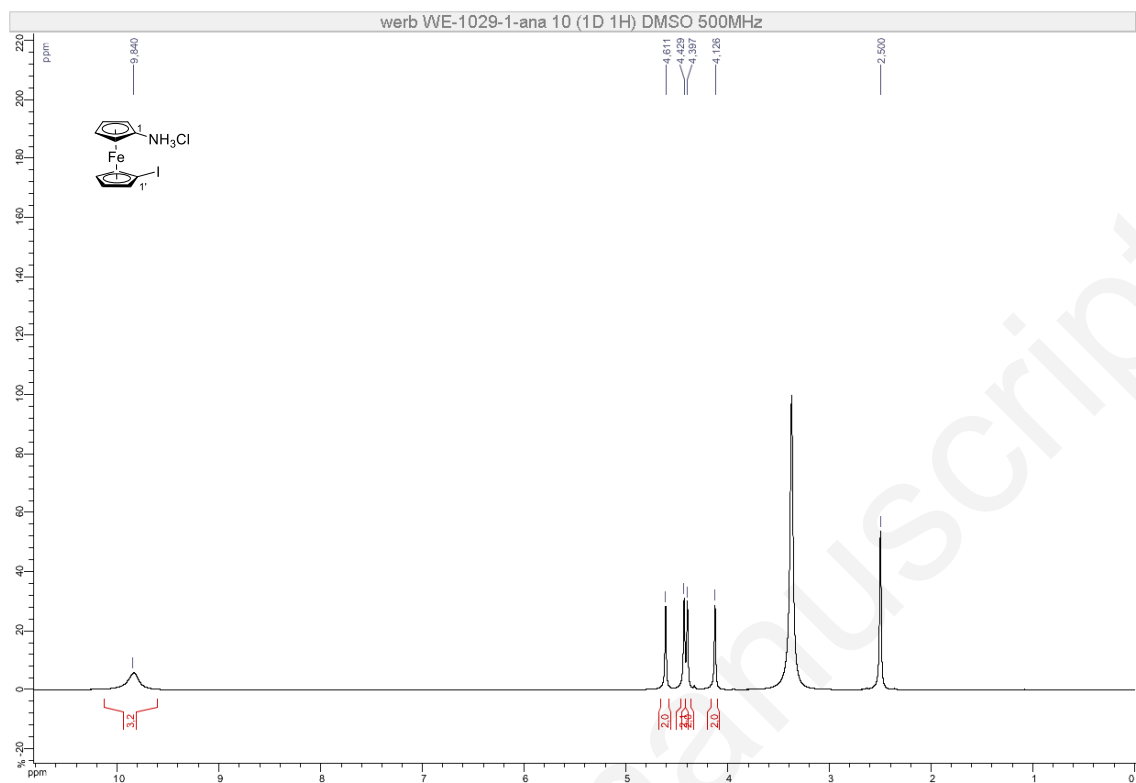
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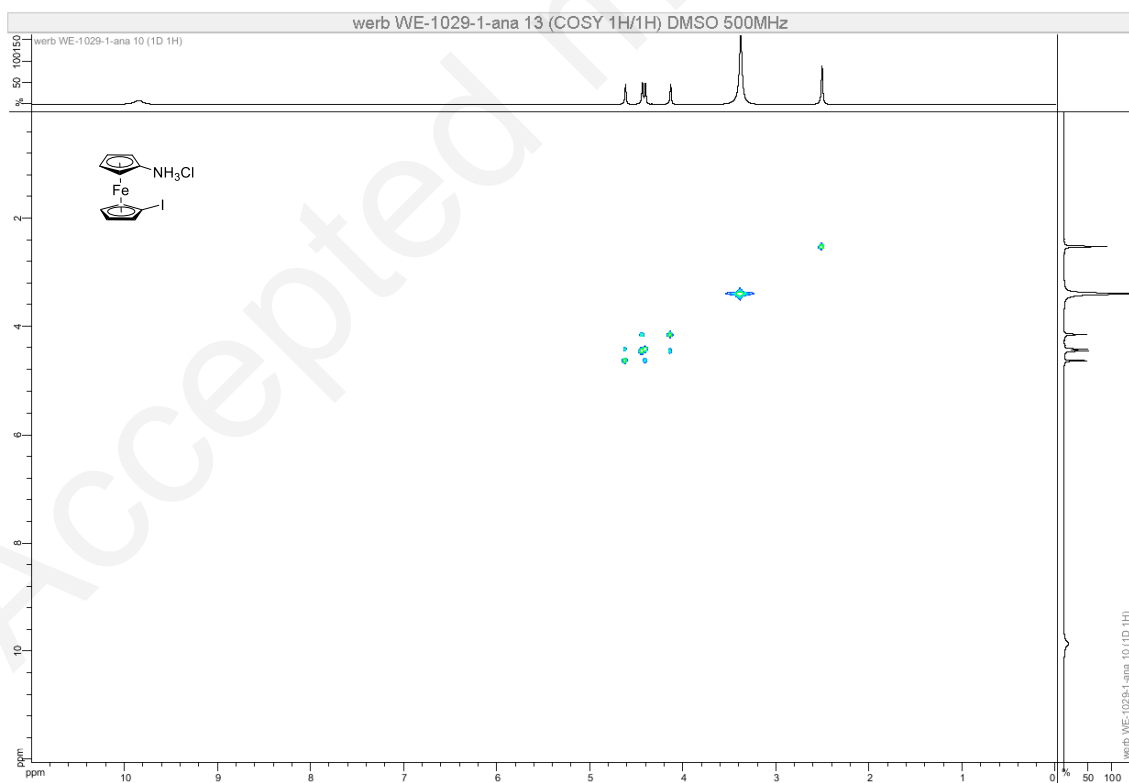
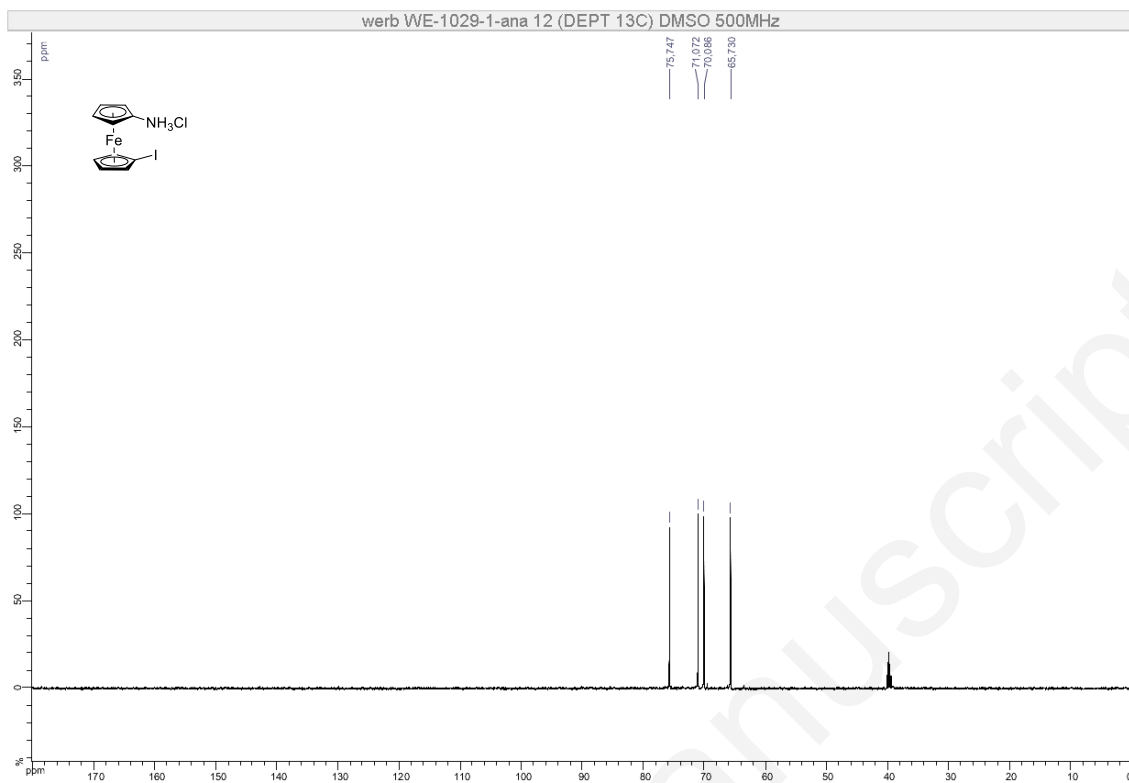


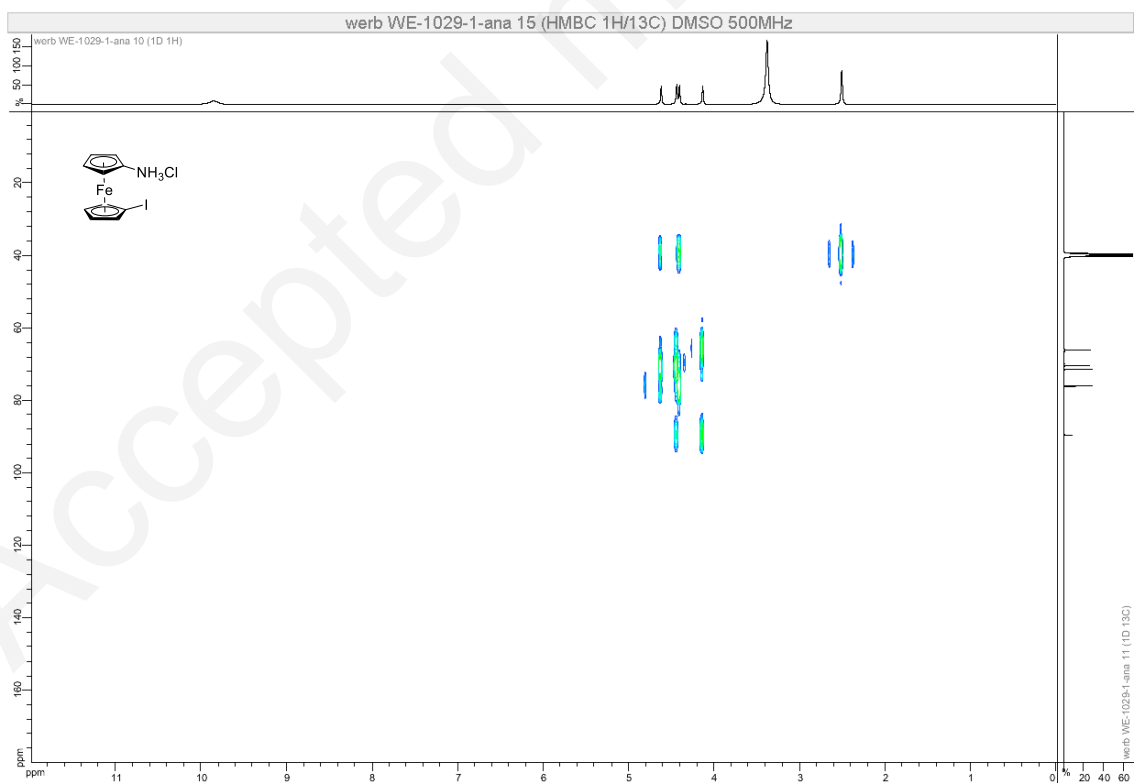
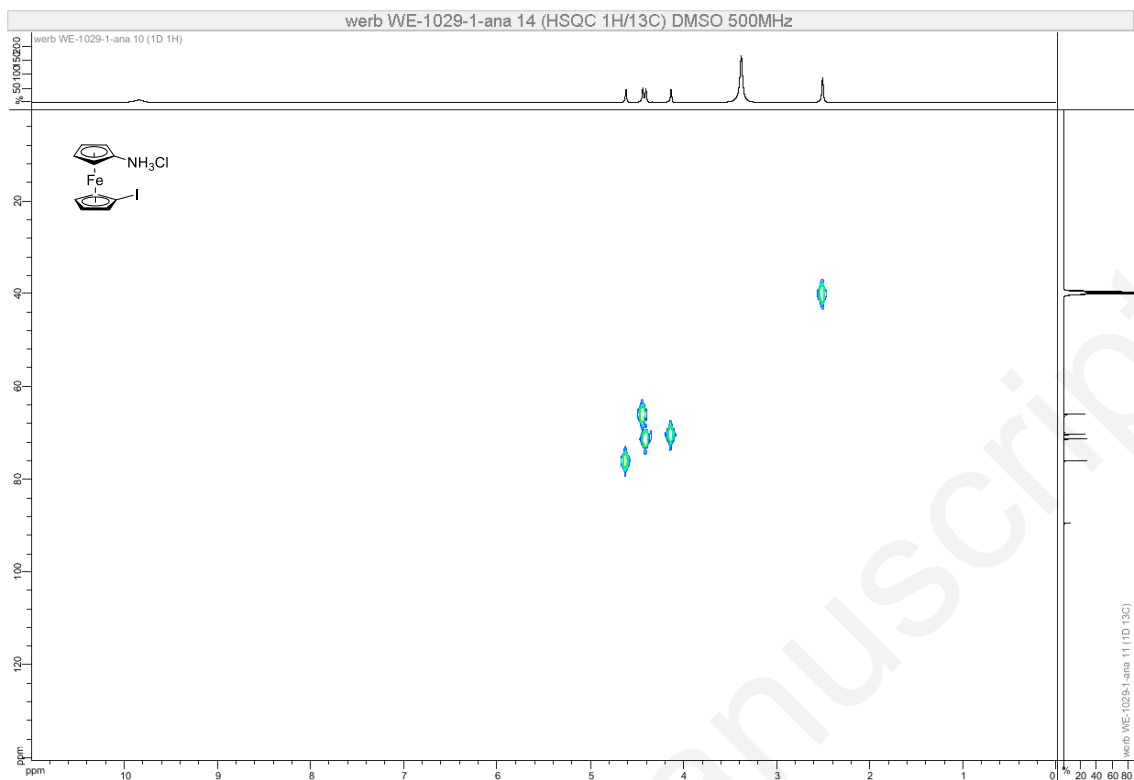




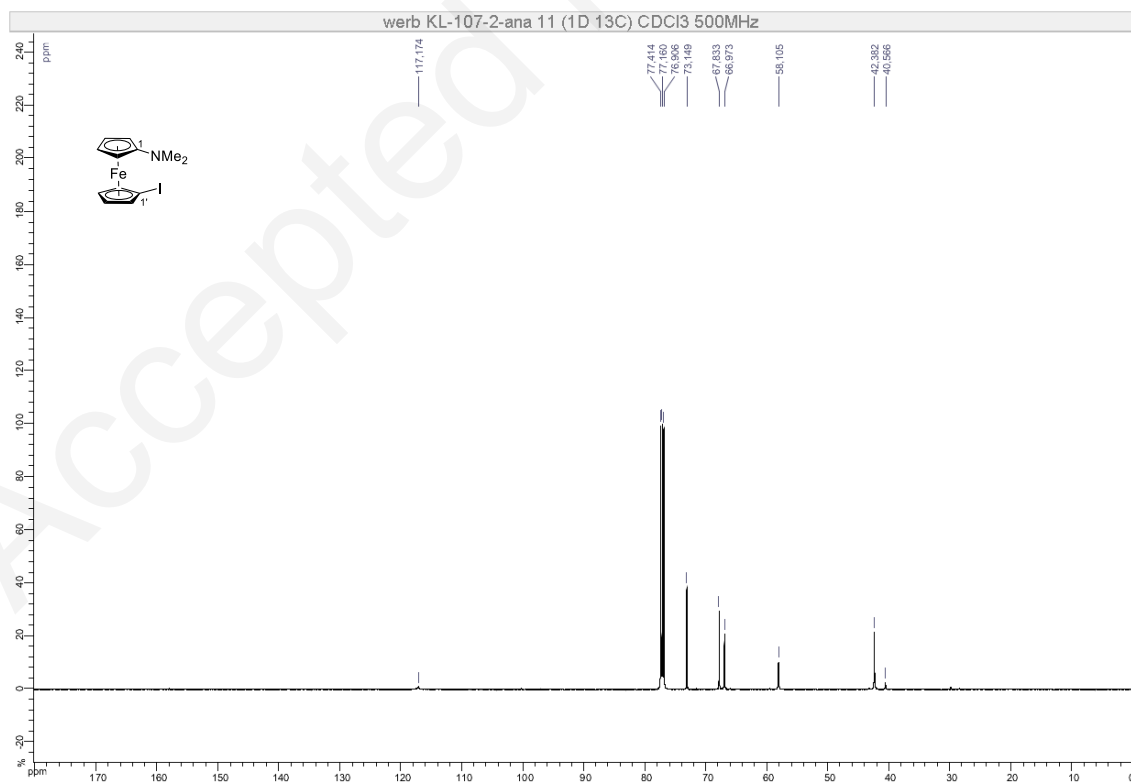
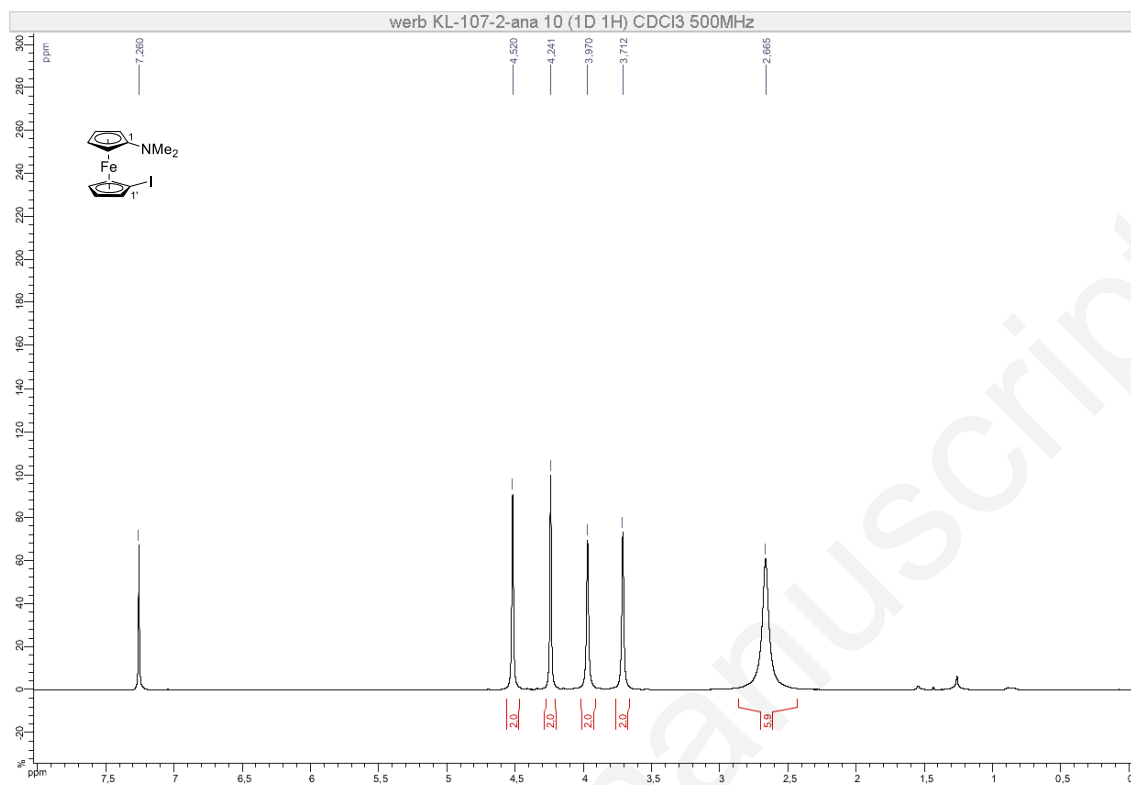
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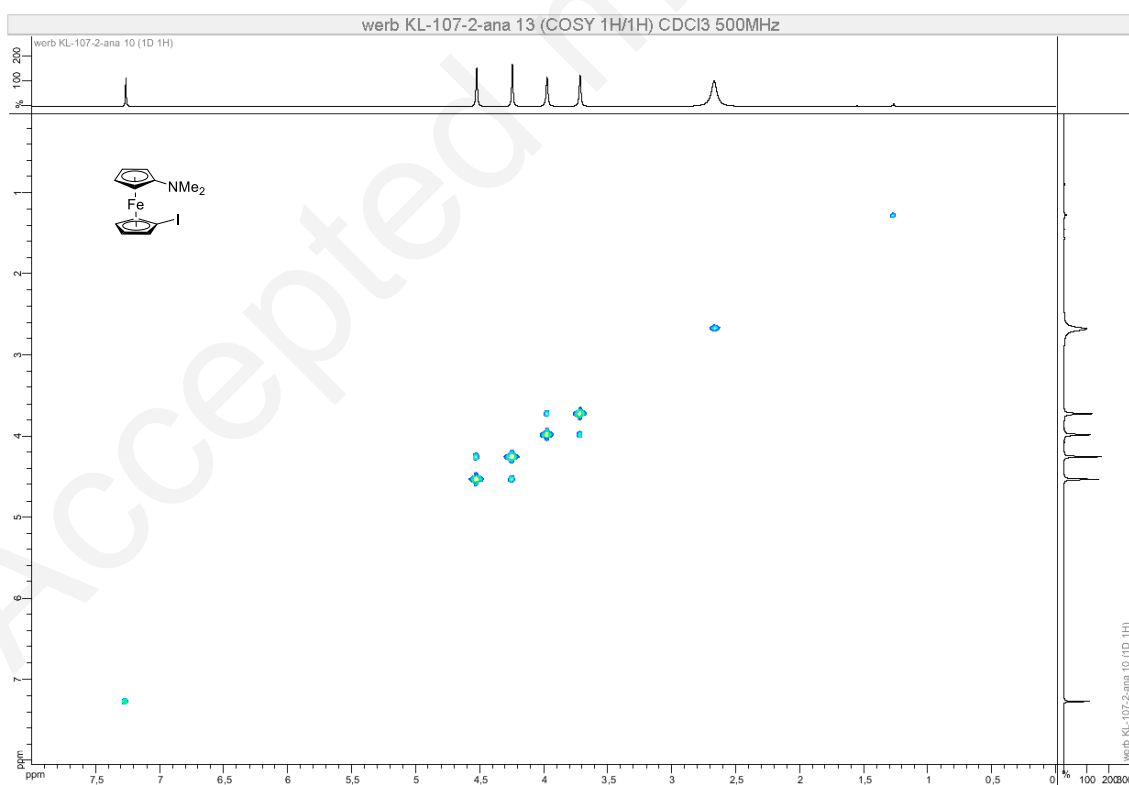
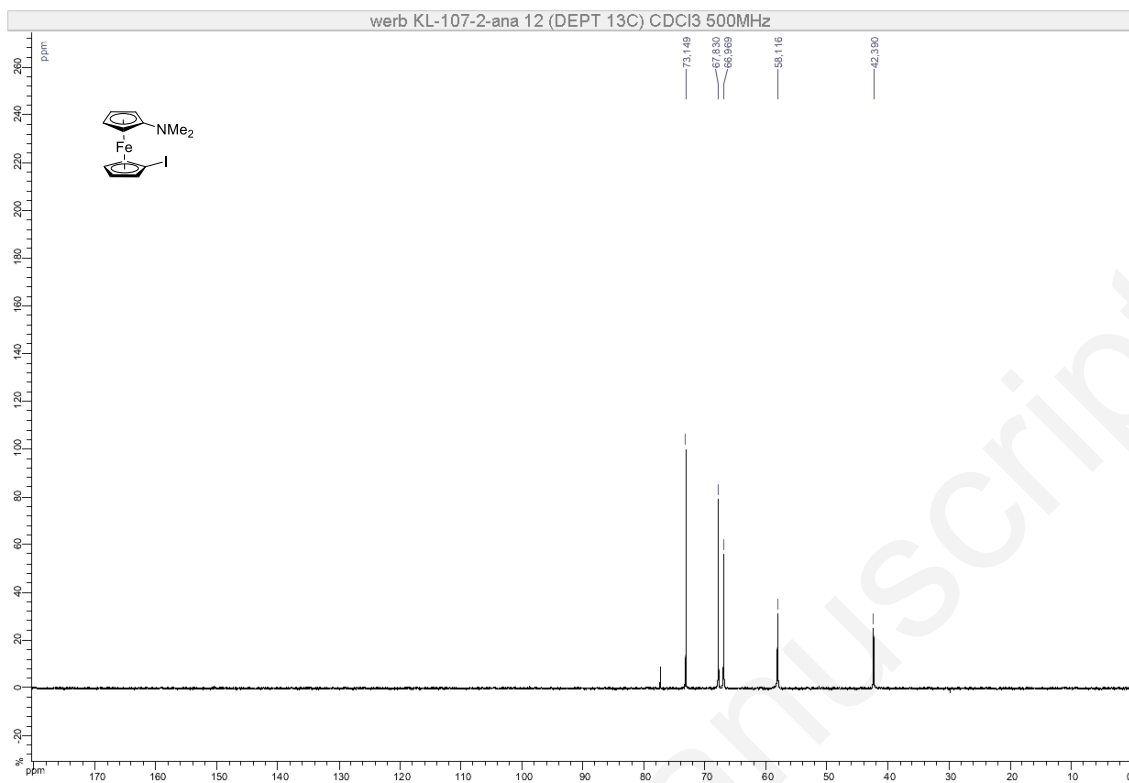


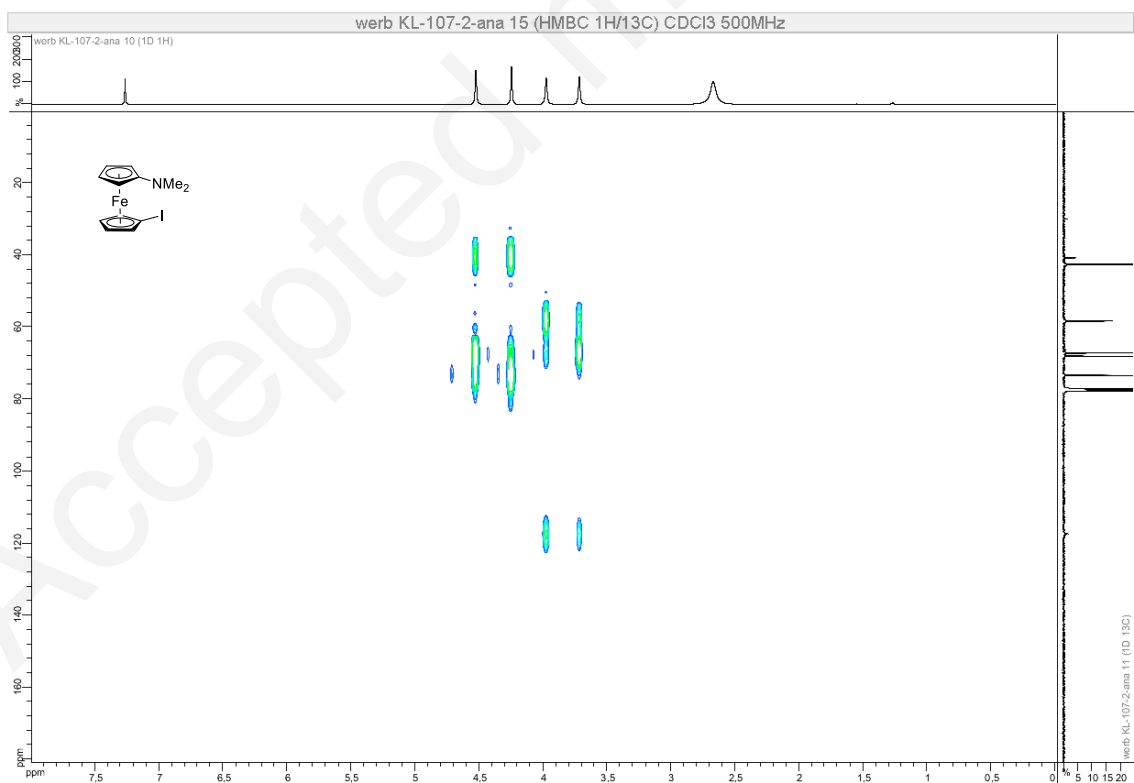
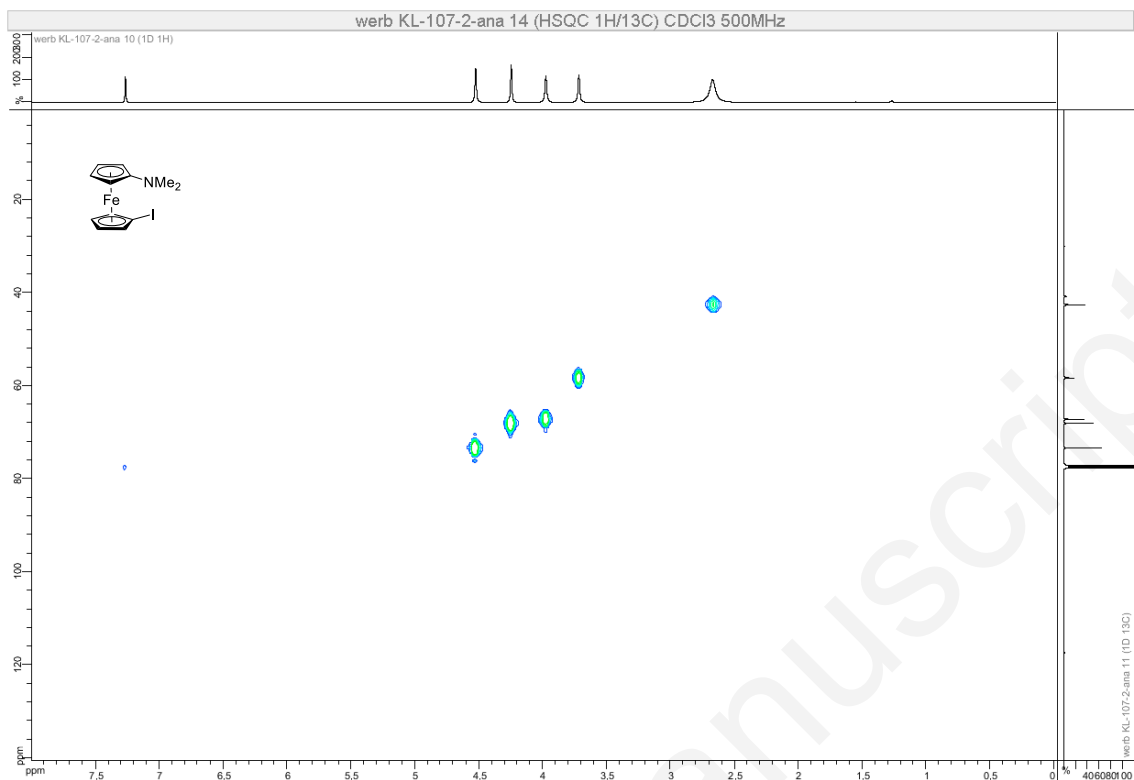




Compound 19







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