# Supporting information for: Structural Properties of *Trans* Hydrido-Hydroxo M(H)(OH)(NH$_2$CMe$_2$CMe$_2$NH$_2$)(PPh$_3$)$_2$ (M = Ru, Os) Complexes and their Proton Exchange Behaviour with Water in Solution

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Table 1: X-Ray crystal structure and refinement data for complexes **3Ru** and **3Os**.

| Compounds | 3Ru | 3Os |
|---|---|---|
| Empirical formula | $C_{42}H_{50}N_2O_2P_2Ru$ | $C_{42}H_{50}N_2O_2P_2Os$ |
| Formula mass | 777.85 | 866.98 |
| Temperature (K) | 150(2) | 150(1) |
| Wavelength (Å) | 0.71073 | 0.71073 |
| Crystal system | Monoclinic | Monoclinic |
| Space group | $P2_1/n$ | $P2_1/n$ |
| a (Å) | 13.9422(4) | 13.9210(3) |
| b (Å) | 15.7847(2) | 15.8226(4) |
| c (Å) | 16.9683(4) | 16.9605(6) |
| $\alpha$ (°) | 90 | 90 |
| $\beta$ (°) | 94.2440(9) | 94.033(2) |
| $\gamma$ (°) | 90 | 90 |
| Volume (Å$^3$) | 3724.0(2) | 3726.5(2) |
| Z | 4 | 4 |
| Density (calculated, g/cm$^3$) | 1.387 | 1.545 |
| Absorption Coefficient (mm$^{-1}$) | 0.545 | 3.546 |
| F(000) | 1624 | 1752 |
| Crystal Size (mm$^3$) | $0.14 \times 0.12 \times 0.06$ | $0.10 \times 0.10 \times 0.10$ |
| Theta range for data collection (°) | 2.73–27.56 | 2.57–27.55 |
| Reflections collected | 20367 | 34052 |
| Independent reflections | 8073 $[R(int) = 0.0473]$ | 8502 $[R(int) = 0.0843]$ |
| Completeness to $\theta = 25.00°$ (%) | 98.0 | 98.9 |
| Absorption correction | Multi-scan | Multi-scan |
| Max. and min. transmission | 0.969, 0.882 | 0.712, 0.577 |
| Largest diff. peak and hole (e Å$^3$) | 1.355, -1.163 | 3.258, -1.037 |
| Refinement method | Full-matrix least-squares on $F^2$ | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 8073/0/449 | 8502/0/449 |
| Goodness-of-fit on F$^2$ | 1.040 | 1.054 |
| Final $R$ indices $[I > 2\sigma(I)]$ [a] | $R_1 = 0.0461$ | $R_1 = 0.0428$ |
| $R$ indices (all data) [b] | $wR_2 = 0.1268$ | $wR_2 = 0.0974$ |

[a] $R_1 = \frac{\Sigma(F_O - F_C)}{\Sigma(F_O)}$

[b] $wR_2 = \sqrt{\frac{\Sigma(w(F_O^2 - F_C^2)^2)}{\Sigma(w(F_O^2)^2)}}$
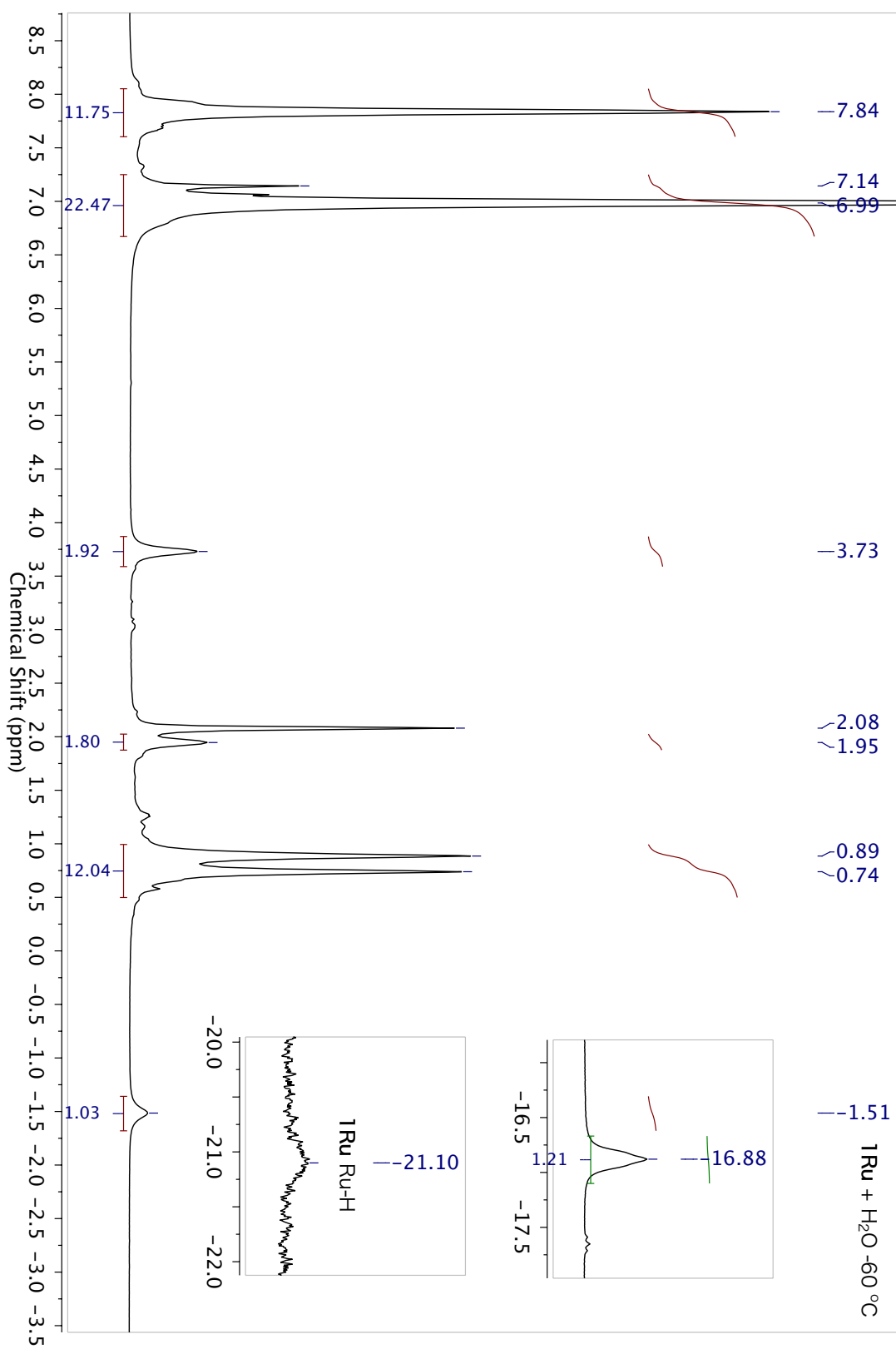
Figure 1: $^1$H NMR spectrum (400 MHz) of **1Ru** + $H_2O$ at -60 °C in toluene-$d_8$.
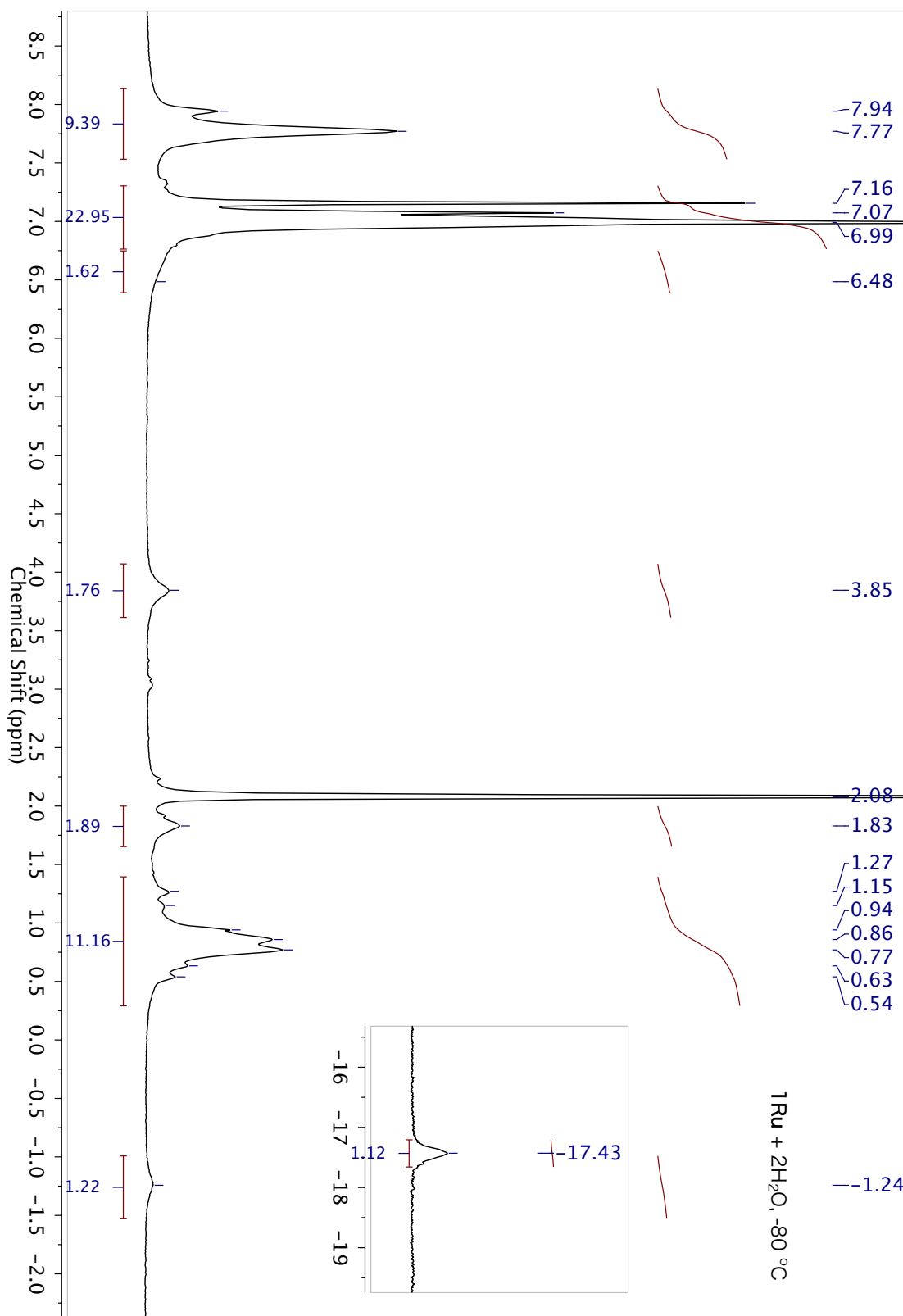
Figure 2: $^1$H NMR spectrum (400 MHz) of **1Ru** + 2 H$_2$O at -80 °C in toluene-$d_8$.
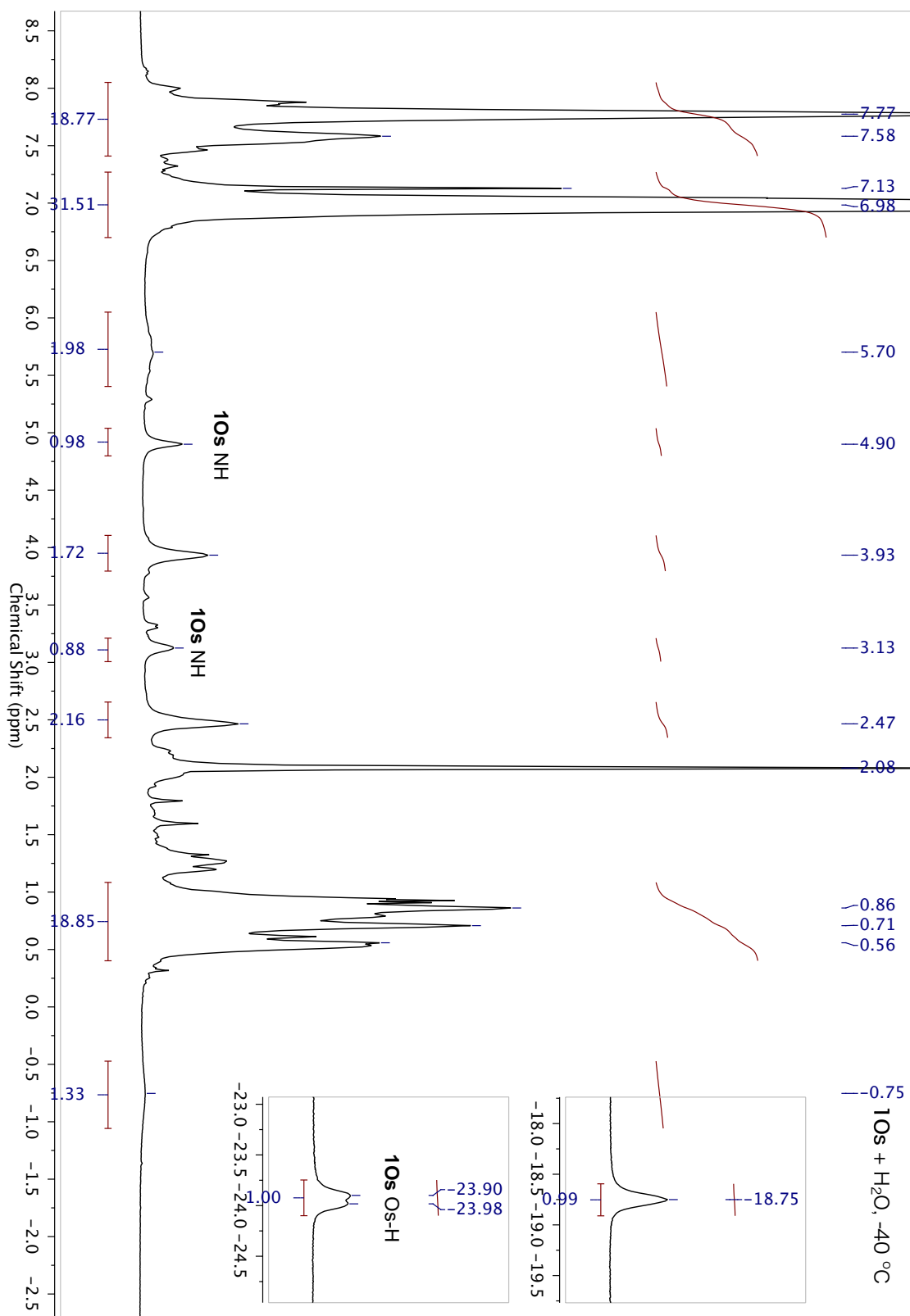
Figure 3: $^1$H NMR spectrum (400 MHz) of **1Os** + $H_2O$ at -40 °C in toluene-$d_8$.
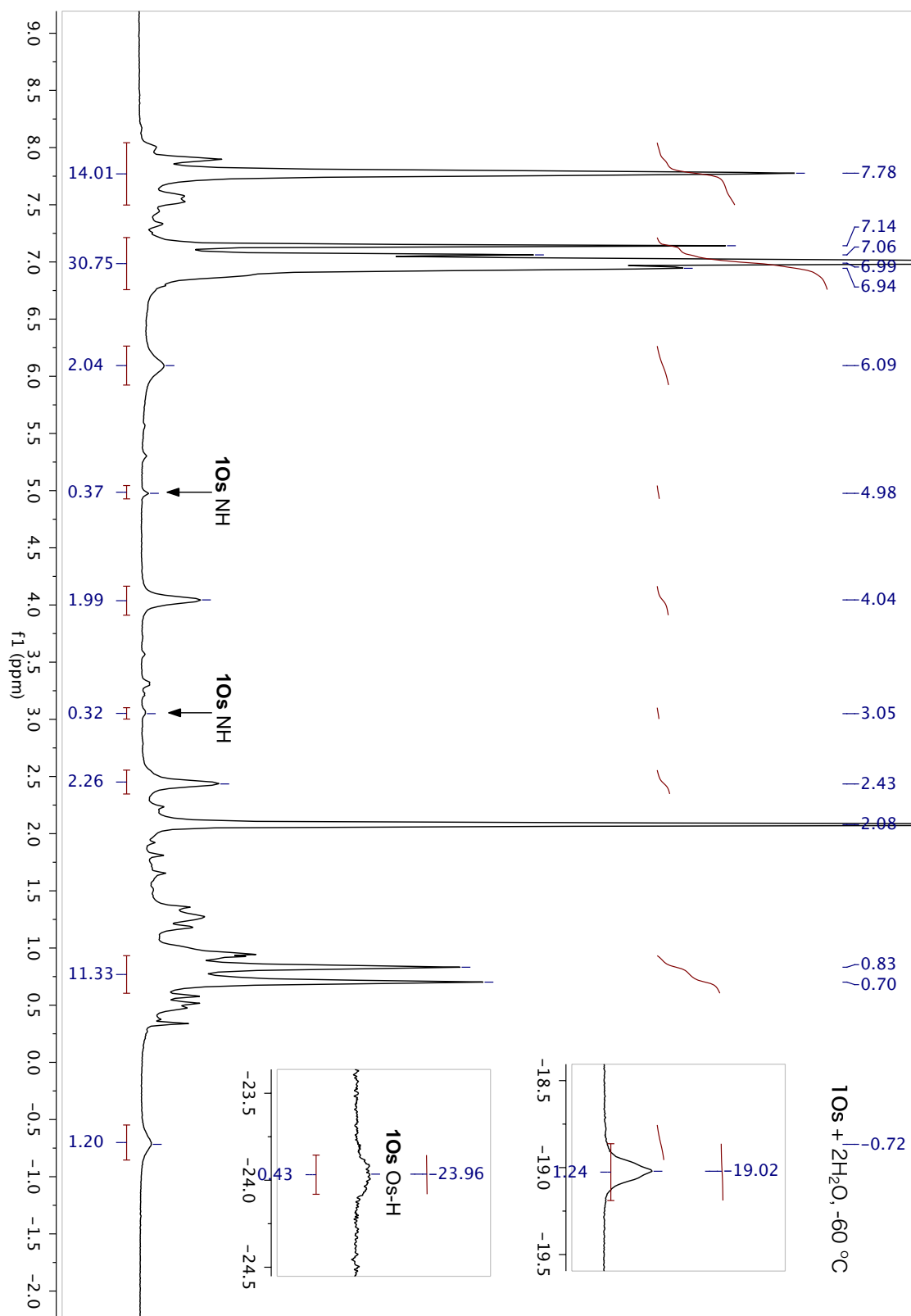
Figure 4: $^1$H NMR spectrum (400 MHz) of **1Os** + 2 H$_2$O at -60 °C in toluene-$d_8$.