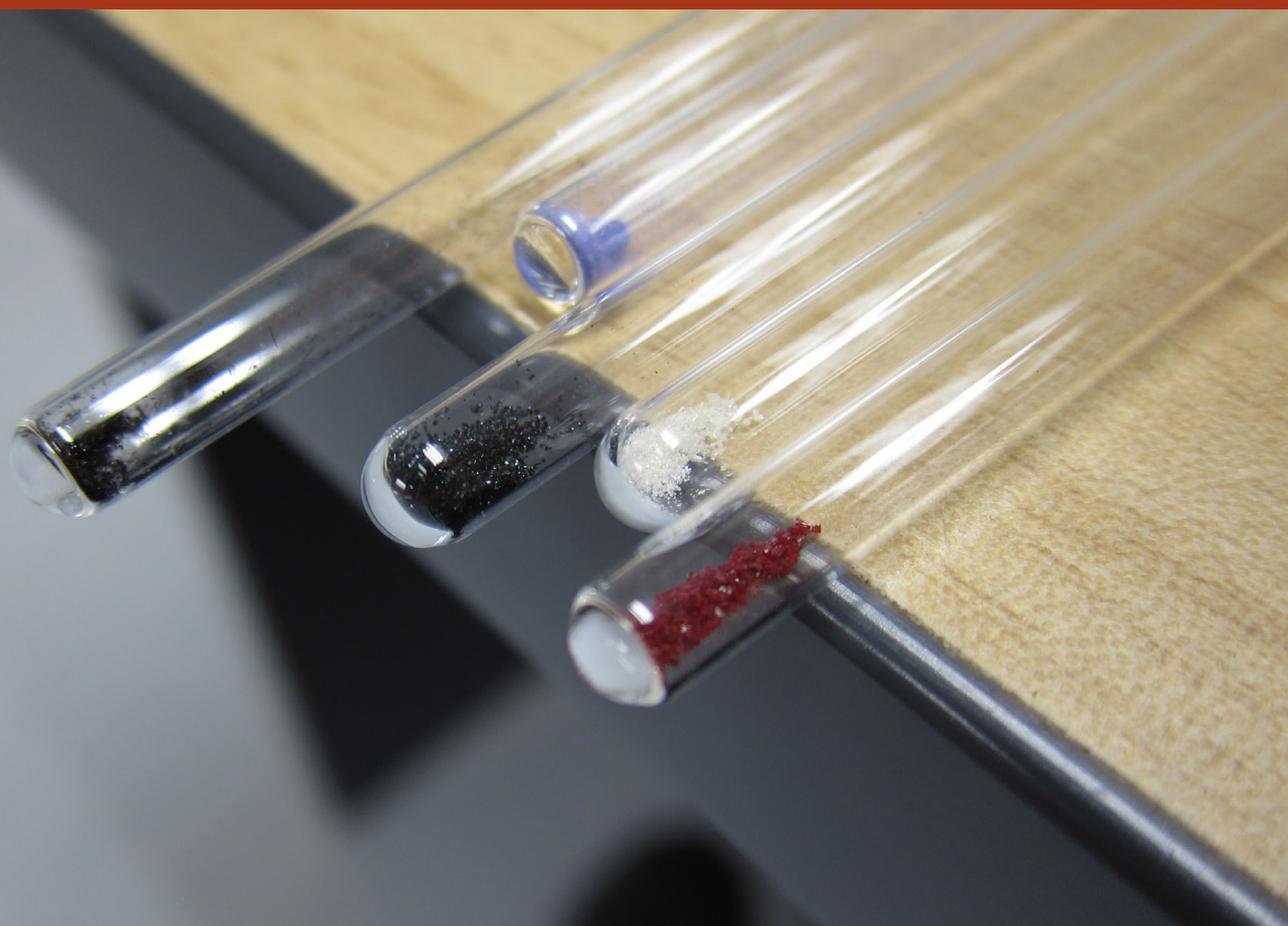


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Preparation and Characterisation of Metal Acetylacetonate Complexes



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Objectives

The aim of this experiment is for students to synthesise a metal acetylacetonate complex, understand the keto-enol tautomerisation of acetylacetonone and determine the electronic structure of their compound using Evans method and the Spinsolve NMR spectrometer.

Introduction

Acetylacetonone (2,4-pentanedione) is an organic compound, a β -diketone, that exists as an equilibrium mixture of tautomeric keto and enol forms. In basic solution, it is deprotonated to form the acetylacetonate anion (acac) (Figure 1).

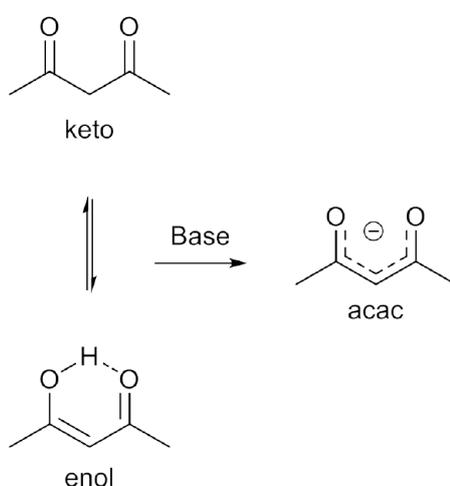


Figure 1: Keto-enol equilibrium of acetylacetonone and formation of acetylacetonate anion.

The acac anion can act as a ligand towards metal ions, typically forming a bidentate complex where the metal is bound to the two oxygen atoms, thus forming a 6-membered ring. Metal acac compounds are typically isolated as crystalline solids that are neutral, hence an M^{n+} metal forms a complex with n acac ligands (Figure 2).

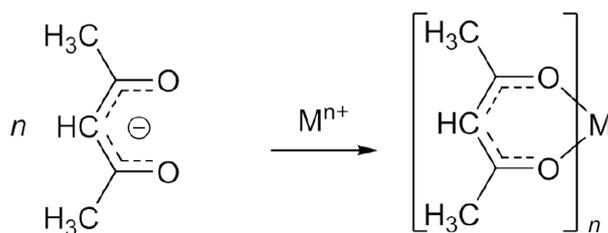


Figure 2: Acetylacetonate forms neutral complexes with most metals.

In organometallic chemistry, many of the complexes encountered contain paramagnetic metal ions. Unpaired electrons in a paramagnetic complex have significant effects on both the chemical shift and the relaxation of proton resonances in the ^1H -NMR spectrum of the complex, causing broadening of peaks and the chemical shifts of other compounds to move. NMR spectroscopy can therefore be used to determine whether a complex is paramagnetic or diamagnetic, and a technique called 'Evans Method' can be used to estimate the magnetic moment of the sample and thus the electronic structure of the complex.

The determination of magnetic susceptibility of substances in solution by NMR was published by Evans in 1959. Evans correlated and observed the difference in chemical shift of an inert reference compound (*t*-butanol) in the presence and absence of paramagnetic transition metal complexes with Equation 1.

$$\text{Equation 1: } \chi_g = \frac{3\Delta f}{2\pi f m} + \chi_o + \frac{\chi_o(d_o - d_s)}{m}$$

Where χ_g is the mass susceptibility of the solute, Δf is the observed shift in the frequency of the reference compound, f is the frequency of the spectrometer, m is the mass of the substance per cm^3 of solvent, χ_o is the mass susceptibility of the solvent, d_o is the density of the solvent and d_s is the density of the solution. The second and third terms may be dropped as they tend to cancel each other out or add more error. If the equation is also converted to a form appropriate for SI units, the equation becomes:

$$\text{Equation 2: } \chi_m = \frac{6}{1000} \frac{1}{c} \frac{\Delta f}{f}$$

Where χ_m is the molar susceptibility of the solute ($\text{m}^3 \text{mol}^{-1}$), c is the concentration of the substance (mol dm^{-3}), Δf is the observed shift in the frequency of the reference compound (Hz) and f is the frequency of the spectrometer (Hz).

In order to determine the difference in chemical shift of the reference compound caused by paramagnetic compounds, Evans used an NMR tube with a coaxial insert. The NMR tube contains a solution of the paramagnetic substance with 2% reference substance (*t*-butanol) and the coaxial insert contains only the solvent and reference substance. Due to the direction of the magnetic field, this approach does not work on the Spinsolve. However, due to the stability of the magnet, it is possible to run a separate reference sample (containing solvent and reference substance) and then run the sample which contains the paramagnetic substance in solution with reference substance added using the Spinsolve.

In order to determine the electronic structure of a complex, students must determine the effective magnetic moment, μ_{eff} , using the following:

$$\text{Equation 3: } \mu_{\text{eff}} = \sqrt{\frac{3k}{N_0 \mu_0 \mu_B^2}} \sqrt{\chi_m T}$$

Where k is the Boltzmann constant, N_0 is Avogadro's number, μ_0 is the permeability of a vacuum and μ_B is the Bohr magneton. Since the first term is a constant, the equation can be simplified as:

$$\text{Equation 4: } \mu_{\text{eff}} = 798 \sqrt{\chi_m T}$$

Where T is the temperature in Kelvin and χ_m is the molar susceptibility determined by Evans method. The theoretical magnetic moment can be determined using the number of unpaired electrons on the metal atom of a complex, n , using the following:

$$\text{Equation 5: } \mu_{\text{eff}} = \sqrt{n(n + 2)}$$

For example, a low spin Fe^{2+} complex is diamagnetic, thus $n = 0$ and the effective magnetic moment is $0 \mu_B$. Whereas a high spin Fe^{2+} complex is paramagnetic with four unpaired electrons, hence the effective magnetic moment would be expected to be $4.90 \mu_B$. Comparing the theoretical magnetic moment of their complex with their experimental value, students are able to determine whether their complex is high or low spin.

The Spinsolve is equipped with a 'Paramagnetic' protocol which allows paramagnetic compounds to be measured at a higher concentration than in the standard 1D proton protocol. Students first obtain spectra of their transition metal acac compound which will be informative about whether their complex is paramagnetic or diamagnetic, and if it is paramagnetic, some indication of the number of unpaired electrons.

The $^1\text{H-NMR}$ spectrum of acetylacetone is also measured as both tautomers exist in solution on the NMR time scale. Comparison of this with the $^1\text{H-NMR}$ spectrum of a diamagnetic acac complex (since the enol form can be regarded as a complex of H^+ , the $^1\text{H-NMR}$ spectrum is expected to show similarities to a metal complex) allows students to assign the peaks for each tautomer and determine the keto:enol ratio.

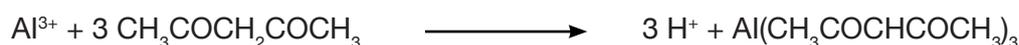
Experiment

Students will synthesise a diamagnetic and a paramagnetic metal acetylacetonate complex (or each student synthesises one, and do their analysis in pairs) and obtain a $^1\text{H-NMR}$ spectrum using the Paramagnetic protocol. Evans method is used to determine the number of unpaired electrons and thus the electronic structure of paramagnetic complexes. The resonances in the $^1\text{H-NMR}$ spectrum of the diamagnetic complex are assigned. Students also obtain a $^1\text{H-NMR}$ spectrum of acetylacetonate and determine the keto:enol ratio by comparison of the spectrum with that of the diamagnetic acetylacetonate complex.

Safety

Ammonium hydroxide is corrosive and may cause burns, avoid contact with skin. Petroleum ether, *t*-butanol and methanol are highly flammable, use in a fume hood and keep away from sources of ignition. Deuterated chloroform is toxic, do not ingest or inhale.

Al³⁺ Complex



Tris(acetylacetonato)aluminium



Aluminium Sulfate

To a conical flask, add 3 mL acetylacetonate and 40 mL distilled water followed by 8 mL of dilute (5 mol L⁻¹) ammonia solution. Dissolve aluminium sulfate [Al(SO₄)₃ · 16 H₂O] in 30 mL distilled water. To this solution, add the ammoniacal acetylacetonate solution gradually with stirring. After complete addition of acetylacetonate, check that the solution is neutral. If the solution is still acidic, add further small portions of ammonia solution. Place the flask in an ice bath until a cream coloured precipitate forms. Filter the product using Buchner filtration, wash with small amounts of cold distilled water and dry the product in a vacuum desiccator. Determine the percentage yield of this dry product.

Recrystallise a small sample from warm petroleum ether. The resulting needles should be filtered using Buchner filtration, rinsed with small portions of cold petroleum ether and dried in a vacuum desiccator.

Obtain a ^1H -NMR spectrum of the product in chloroform-d using the Paramagnetic protocol on the Spinsolve (Powerscan = 2 minutes, 40 scans).

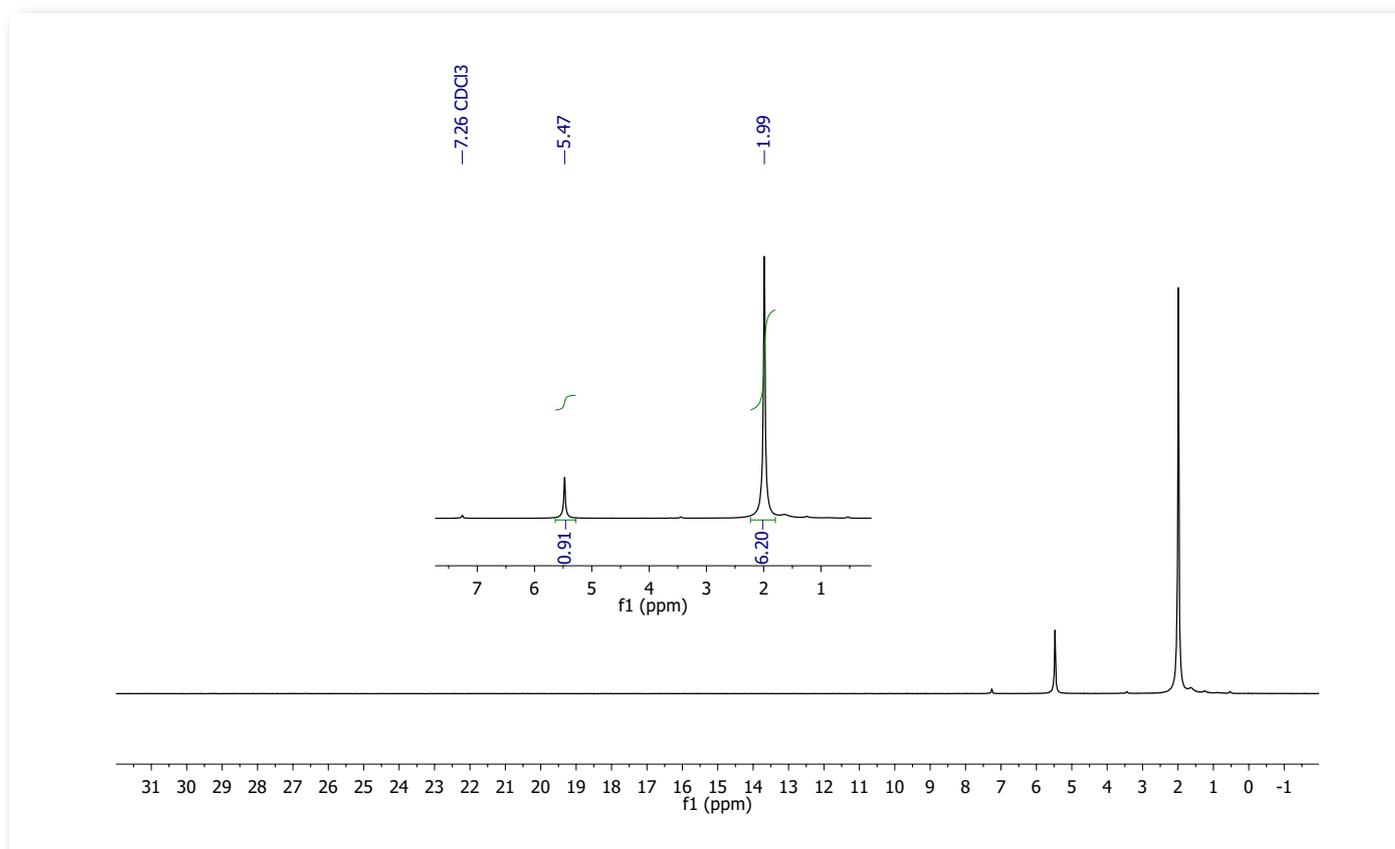
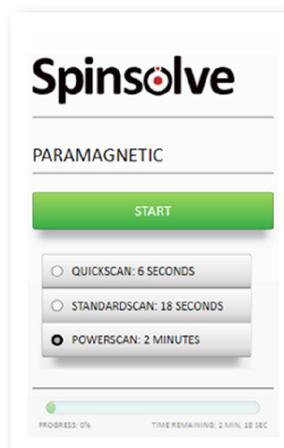
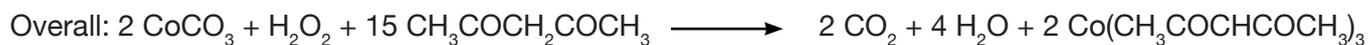
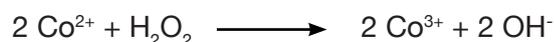
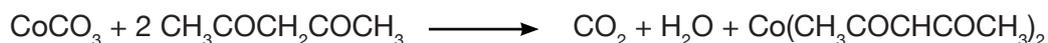


Figure 3: ^1H -NMR spectrum of $\text{Al}(\text{acac})_3$.

The ^1H -NMR spectrum of $\text{Al}(\text{acac})_3$ (Figure 3) has sharp resonances in the region between 0 – 8 ppm, typical for most molecules. From this it can be determined that the complex is diamagnetic, which is expected as Al^{3+} has the electron configuration of neon and thus no unpaired electrons.

The integrals of the two resonances (inset, Figure 3) show a 1:6 relationship, thus the peak at δ 5.47 that integrates for one proton corresponds to the CH while the peak at δ 1.99 that integrates for 6 protons corresponds to the two methyl groups (Figure 2).

Co³⁺ Complex



To a conical flask, add 2.5 g of cobalt carbonate and 20 mL of acetylacetone and heat to 90°C with stirring. While heating, add dropwise 30 mL of a 10% hydrogen peroxide solution, covering the flask with a watch glass between additions. The complete addition of hydrogen peroxide should take around 30 minutes. Continue to heat for a further 15 minutes, followed by cooling in an ice bath. The dark green precipitate is filtered using Buchner filtration, sucked dry at the water pump and then dried in the oven (100°C). Determine the percentage yield of the dry product.



Cobalt carbonate



Tris(acetylacetonato)cobalt

Recrystallise a small sample from hot petroleum ether. The resulting green needles should be filtered using Buchner filtration, rinsed with small portions of cold petroleum ether and dried in a vacuum desiccator.

Obtain a ¹H-NMR spectrum of the product in chloroform-d using the Paramagnetic protocol on the Spinsolve (Powerscan = 2 minutes, 40 scans).

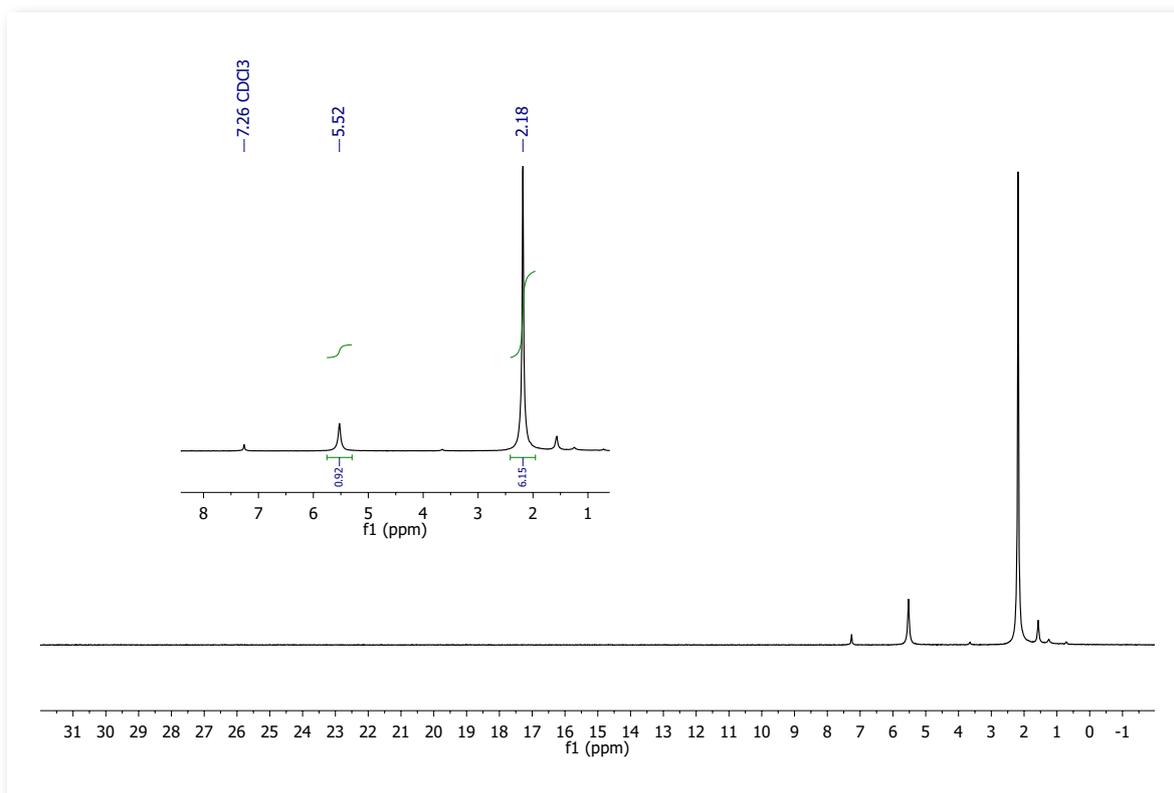


Figure 4: $^1\text{H-NMR}$ spectrum of $\text{Co}(\text{acac})_3$.

The $^1\text{H-NMR}$ spectrum of $\text{Co}(\text{acac})_3$ (Figure 4) has sharp resonances much like the aluminium complex. From this it can be determined that the complex is diamagnetic. Octahedral $\text{Co}(\text{III})$ centres have two possible electron configurations, high spin or low spin (Figure 5). The high spin complex has 4 unpaired electrons and would therefore be paramagnetic. The low spin complex has no unpaired electrons which is diamagnetic, thus the cobalt complex adopts a low spin configuration.

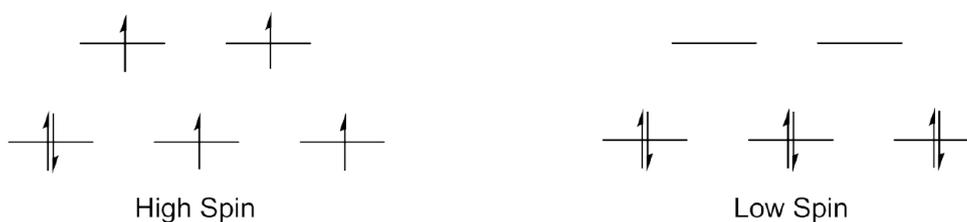
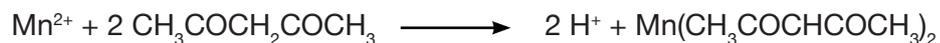


Figure 5: Two possible electronic structures of $\text{Co}(\text{III})$.

The integrals of the two resonances (inset, Figure 4) show a 1:6 relationship, thus the peak at δ 5.52 that integrates for one proton corresponds to the CH while the peak at δ 2.18 that integrates for 6 protons corresponds to the two methyl groups (Figure 2).

Mn³⁺ Complex



Dissolve 2.6 g of manganese chloride ($\text{MnCl}_2 \cdot 4 \text{H}_2\text{O}$) and 6.8 g of sodium acetate in 100 mL of distilled water and add 10 mL acetylacetone. Dissolve 0.52 g of potassium permanganate (KMnO_4) in 25 mL of distilled water and add to the acetylacetonate solution, with stirring, over a period of about 10 minutes. After stirring for 10 minutes, add a solution of sodium acetate (6.3 g) in 25 mL distilled water, with stirring, over a period of about 10 minutes. Continue stirring and heat the flask on a hot plate to 60-70°C for 15 minutes, followed by cooling to room temperature and finally placed in an ice bath. Using Buchner filtration, filter the dark precipitate using small portions of cold distilled water for washing, followed by drying in a vacuum desiccator. Determine the percentage yield of this dry product.



Manganese chloride



Potassium permanganate



Tris(acetylacetonato)manganese

Recrystallise a small sample from warm petroleum ether. The resulting black needles should be filtered using Buchner filtration, rinsed with small portions of cold petroleum ether and dried in a vacuum desiccator.

Obtain a ¹H-NMR spectrum of the product in chloroform-d using the Paramagnetic protocol on the Spinsolve (Powerscan = 2 minutes, 40 scans).

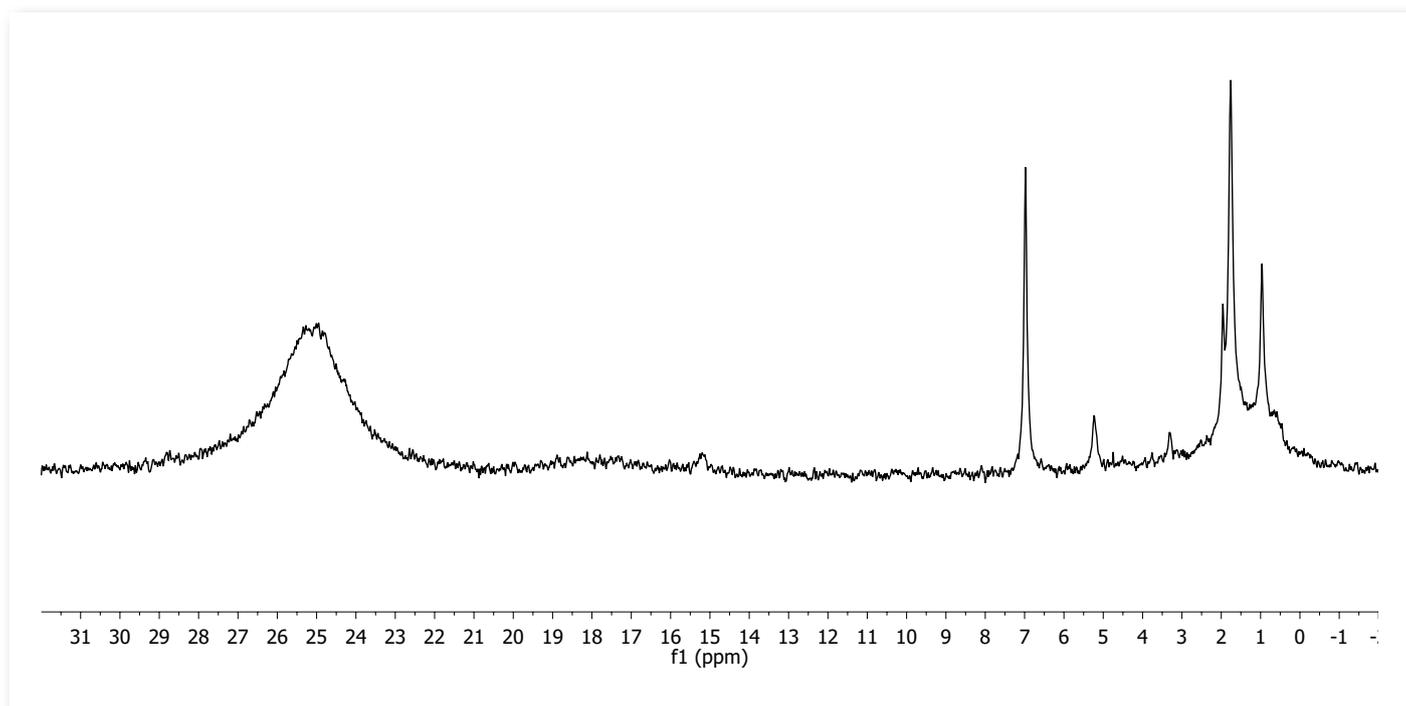


Figure 6: $^1\text{H-NMR}$ spectrum of $\text{Mn}(\text{acac})_3$.

The $^1\text{H-NMR}$ spectrum of $\text{Mn}(\text{acac})_3$ (Figure 6) contains broad and downfield resonances which suggests that the complex is paramagnetic. Octahedral $\text{Mn}(\text{III})$ centres have two possible electron configurations, high spin or low spin (Figure 7). $\text{Mn}(\text{III})$ is d^4 , thus both configurations have unpaired electrons and would therefore be paramagnetic.

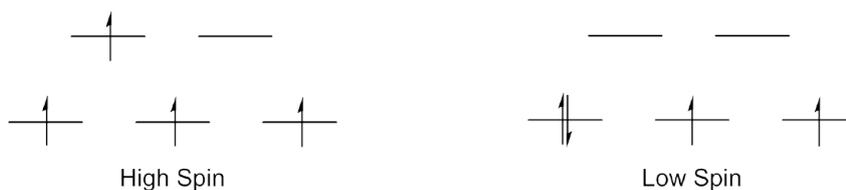


Figure 7: Two possible electronic structures for $\text{Mn}(\text{III})$.

Evans method is used to determine the magnetic moment and therefore electronic structure of $\text{Mn}(\text{acac})_3$.

Accurately weigh approximately 5 mg of complex into an NMR tube and add 0.5 mL of solvent that has been made up with 10% *t*-butanol in CDCl_3 . Prepare a reference sample containing 0.5 mL of the 10% *t*-butanol in CDCl_3 solution. Shim the Spinsolve using Quick Shim and measure the reference sample using Quick Scan (1 scan, 10 seconds) in the 1D Proton protocol.

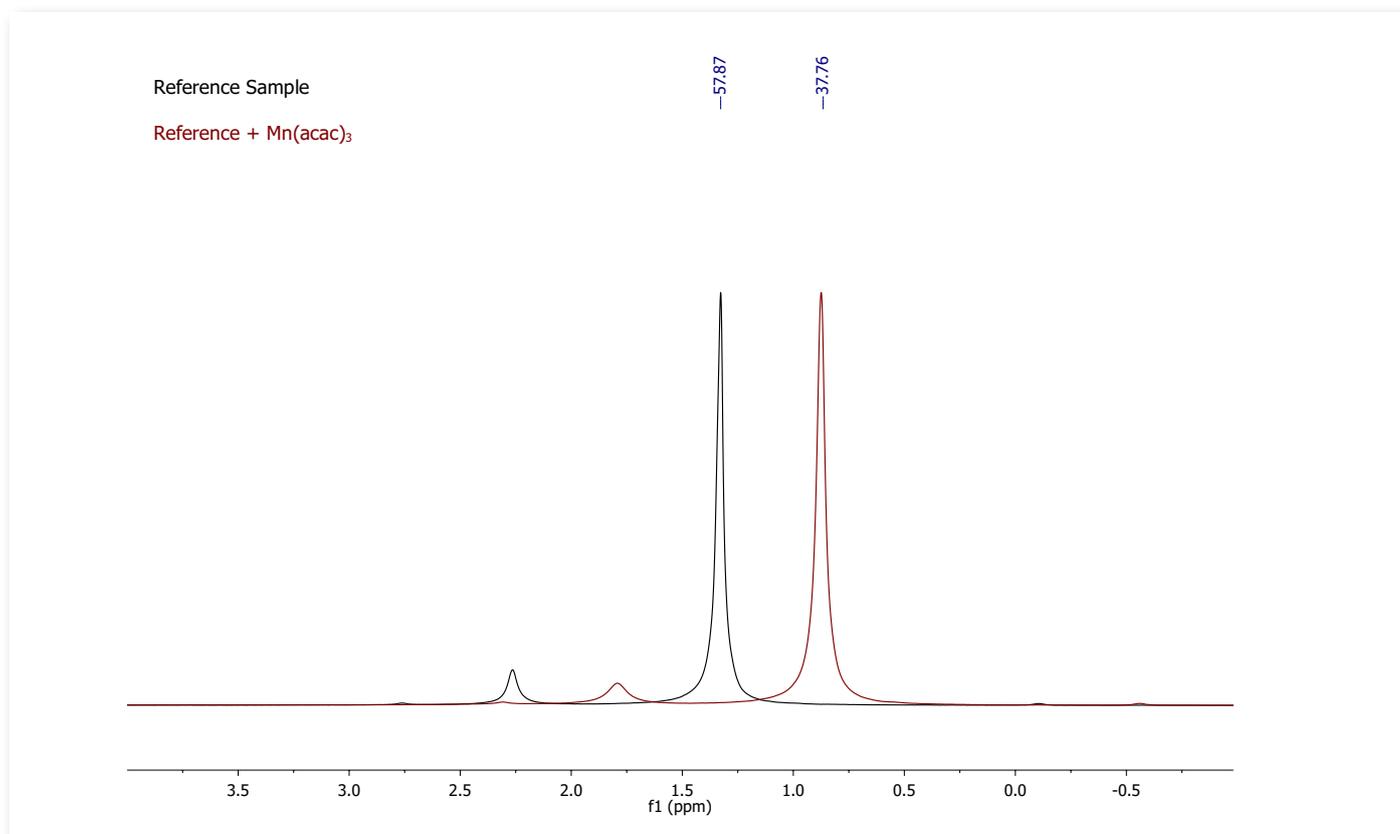
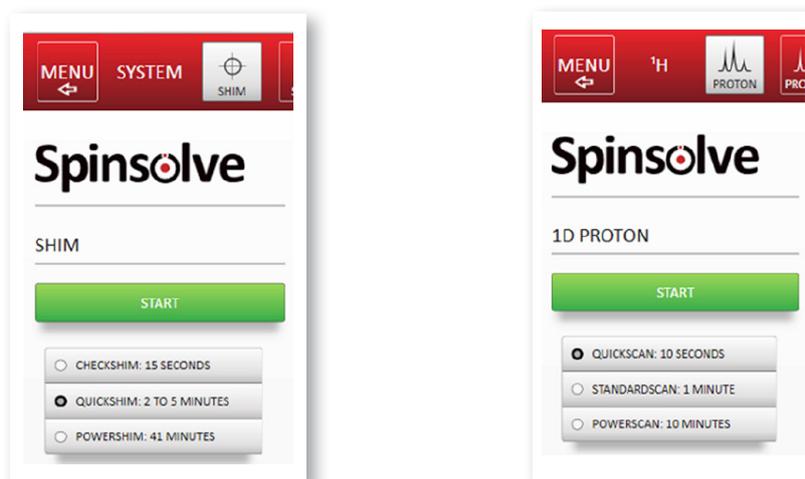


Figure 8: ^1H -NMR spectrum of the reference sample (black) and the reference with $\text{Mn}(\text{acac})_3$ (red) overlaid. The peaks are labelled in Hz.

Calculations

Molar susceptibility:

$$[\text{Mn}(\text{acac})_3] = 0.028 \text{ mol dm}^{-3}$$

$$\Delta f = 57.87 - 37.76 = 20.11 \text{ Hz}$$

$$f = 42,500,000 \text{ Hz}$$

Using Equation 2, $\chi_m = 1.00 \times 10^{-7} \text{ m}^3 \text{ mol}^{-1}$.

Effective magnetic moment:

The molar susceptibility calculated above is used to determine the effective magnetic moment according to Equation 4.

$$T = 294.16 \text{ K}$$

Using Equation 4, $\mu_{\text{eff}} = 4.33 \mu_B$.

The high spin complex has four unpaired electrons and the low spin complex has two. Use Equation 5 to estimate the expected effective magnetic moment for each electronic structure.

High spin, $n = 4$

$$\mu_{\text{eff}} = 4.90 \mu_B$$

Low spin, $n = 2$

$$\mu_{\text{eff}} = 2.83 \mu_B$$

The magnetic moment of the high spin complex is much closer to the experimental value, therefore it can be determined that the complex is high spin.

Fe³⁺ Complex



Dissolve 3.3 g of iron(II) chloride ($\text{FeCl}_2 \cdot 6 \text{H}_2\text{O}$) in 25 mL distilled water. Over a period of 15 minutes, with stirring, add a solution of 4 mL of acetylacetone in 10 mL methanol. To the resulting red mixture, add a solution of 5.1 g sodium acetate in 15 mL distilled water which should result in a red precipitate. Heat the reaction to 80°C for 15 minutes. Remove from the heat and when cool, place in an ice bath. Filter the product using Buchner filtration, wash with cold distilled water and dry in a vacuum desiccator. Determine the percentage yield of the dry product.



Iron(II) chloride



Tris(acetylacetonato)iron

Recrystallise a small sample from warm methanol. The resulting red crystals should be filtered using Buchner filtration, rinsed with small portions of cold methanol and dried in a vacuum desiccator.

Obtain a $^1\text{H-NMR}$ spectrum of the product in chloroform-d using the Paramagnetic protocol on the Spinsolve (Powerscan = 2 minutes, 40 scans).

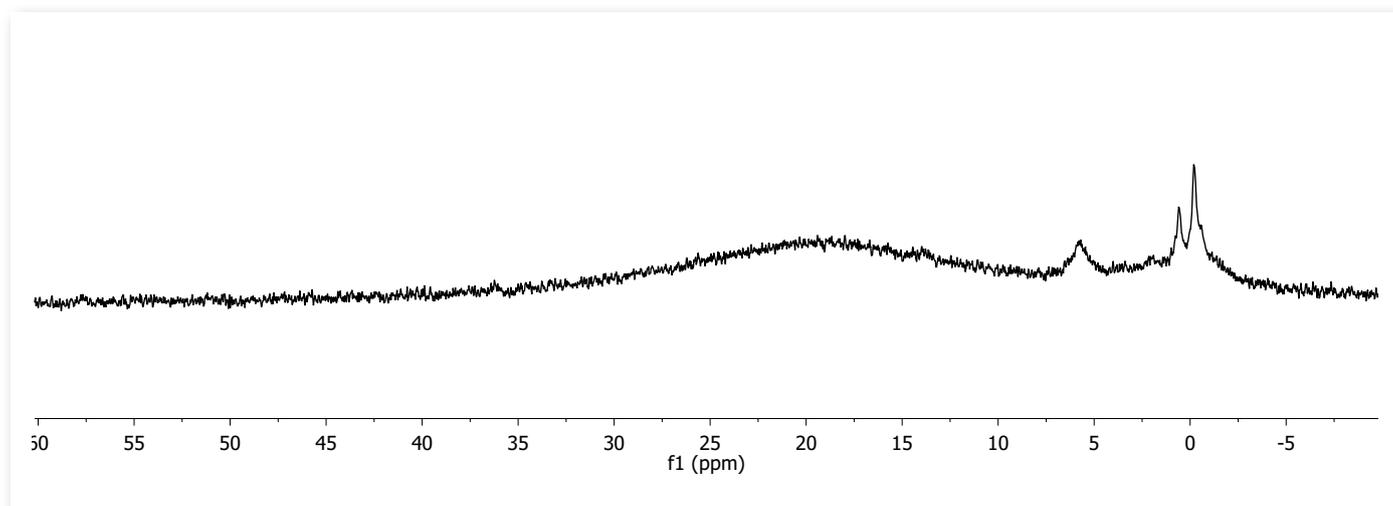


Figure 9: $^1\text{H-NMR}$ spectrum of $\text{Fe}(\text{acac})_3$.

Like the manganese complex, the $^1\text{H-NMR}$ spectrum of $\text{Fe}(\text{acac})_3$ (Figure 9) contains broad, downfield resonances, suggesting that the complex is paramagnetic. Octahedral $\text{Fe}(\text{III})$ centres have two possible electron configurations, high spin or low spin (Figure 10). $\text{Fe}(\text{III})$ is d^5 , thus both configurations have unpaired electrons and would therefore be paramagnetic.

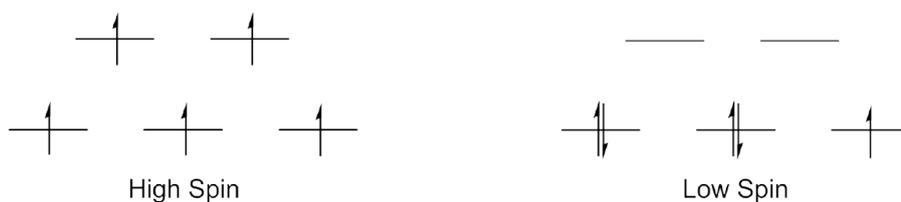


Figure 10: Two possible electronic structures of $\text{Fe}(\text{III})$.

Evans method is used to determine the magnetic moment and therefore electronic structure of $\text{Fe}(\text{acac})_3$ using the same method as described for the manganese complex.

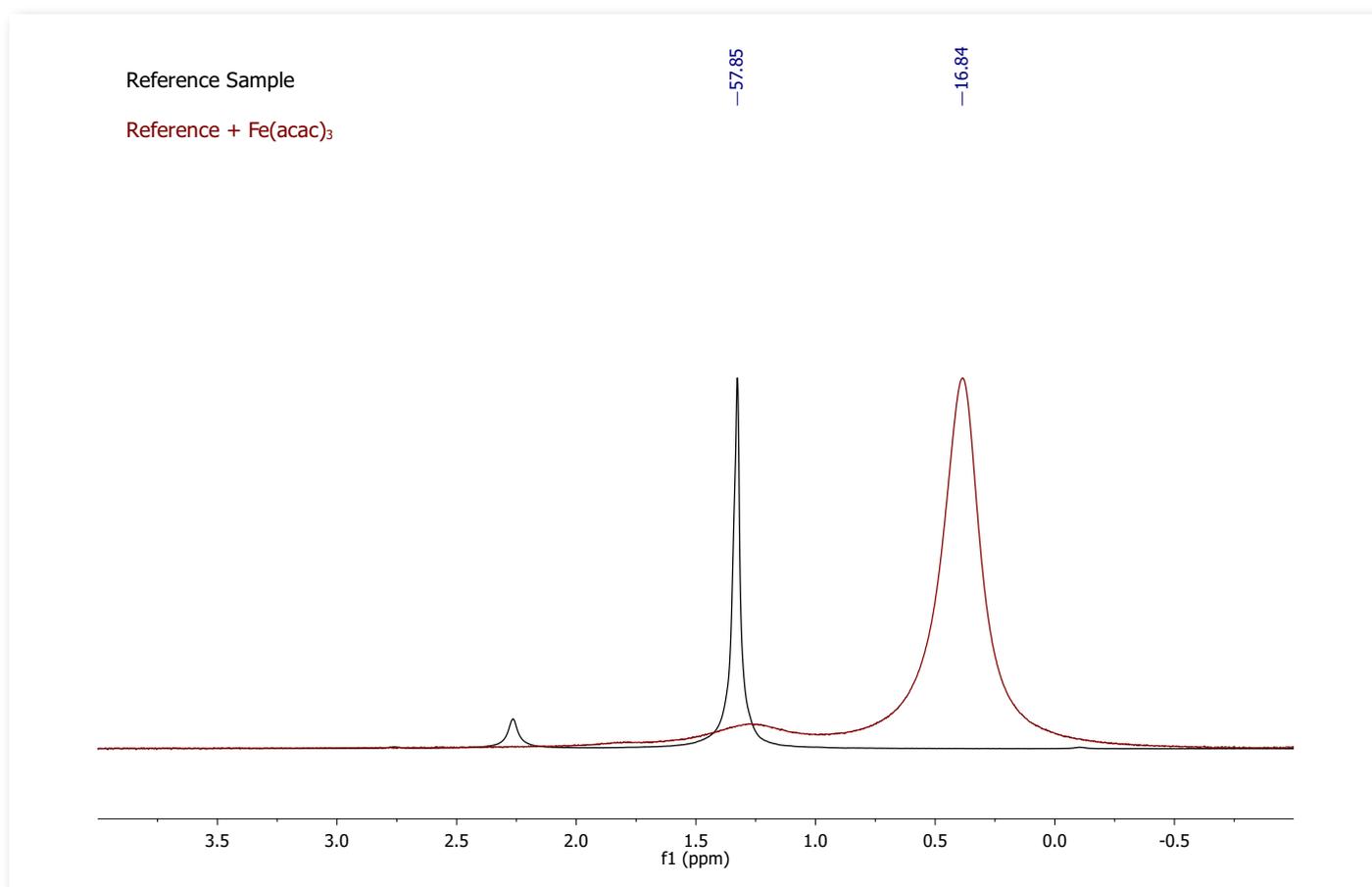


Figure 11: $^1\text{H-NMR}$ spectrum of the reference sample (black) and the reference with $\text{Fe}(\text{acac})_3$ (red) overlaid. The peaks are labelled in Hz.

Calculations

Molar susceptibility:

$$[\text{Fe}(\text{acac})_3] = 0.040 \text{ mol dm}^{-3}$$

$$\Delta f = 57.85 - 16.84 = 41.01 \text{ Hz}$$

$$f = 42,500,000 \text{ Hz}$$

Using Equation 2, $\chi_m = 1.46 \times 10^{-7} \text{ m}^3 \text{ mol}^{-1}$.

Effective magnetic moment:

$$T = 294.16 \text{ K}$$

Using Equation 4, $\mu_{\text{eff}} = 5.23 \mu_B$.

The high spin complex has five unpaired electrons and the low spin complex has one. Use Equation 5 to estimate the expected effective magnetic moment for each electronic structure.

High spin, $n = 5$

$$\mu_{\text{eff}} = 5.92 \mu_B$$

Low spin, $n = 1$

$$\mu_{\text{eff}} = 1.73 \mu_B$$

The magnetic moment of the high spin complex is much closer to the experimental value, therefore it can be determined that the complex is high spin.

Cu²⁺ Complex



In a conical flask, dissolve 4 g copper(II) chloride ($\text{CuCl}_2 \cdot 6 \text{H}_2\text{O}$) in 25 mL distilled water. Over a period of 10 minutes with stirring, add a solution of 5 mL acetylacetone in 10 mL of methanol. To the resulting mixture, add over 5 minutes 6.8 g of sodium acetate dissolved in 15 mL of water. Heat the reaction to 80°C for 15 minutes, followed by cooling in an ice bath. Filter the blue-grey precipitate using Buchner filtration, wash with cold distilled water and dry in an oven (100°C). Determine the percentage yield of the dry product.



Copper chloride



Bis(acetylacetonato)copper

Recrystallise a small sample from methanol. The resulting blue-grey needles should be filtered using Buchner filtration, rinsed with small portions of cold methanol and dried in a vacuum desiccator.

Obtain a $^1\text{H-NMR}$ spectrum of the product in chloroform-d using the Paramagnetic protocol on the Spinsolve (Powerscan = 2 minutes, 40 scans).

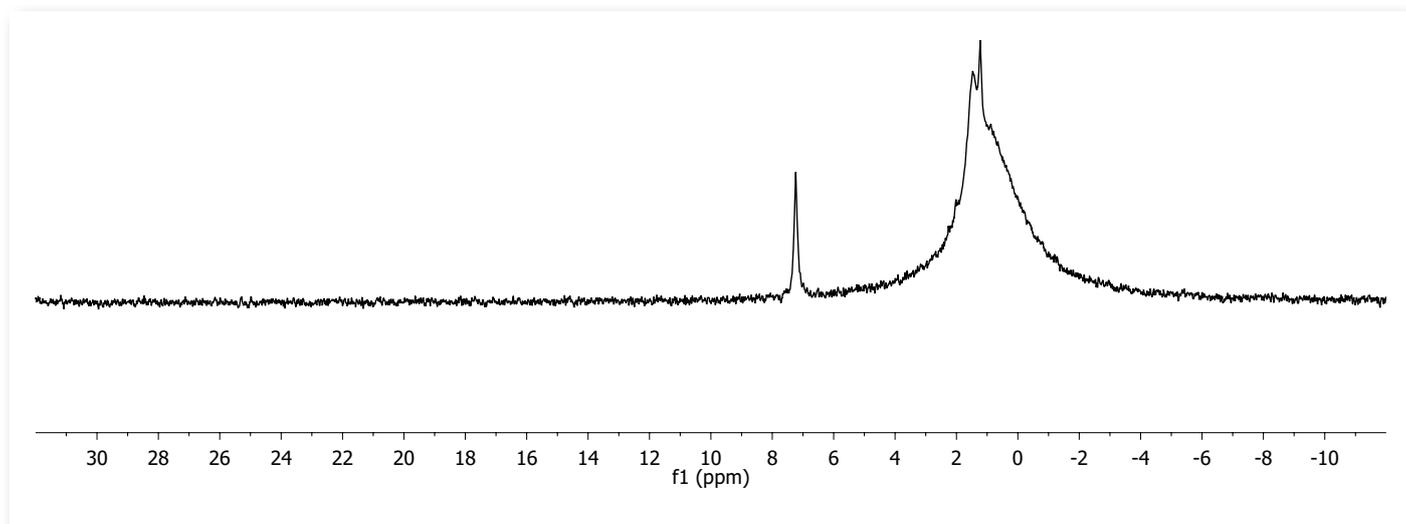


Figure 12: $^1\text{H-NMR}$ spectrum of $\text{Cu}(\text{acac})_2$.

Like the manganese and iron complexes, the $^1\text{H-NMR}$ spectrum of $\text{Cu}(\text{acac})_2$ (Figure 12) contains broad resonances, suggesting that the complex is paramagnetic. $\text{Cu}(\text{II})$ is d^9 , therefore it must have 1 unpaired electron.

Evans method is used to determine the magnetic moment of $\text{Cu}(\text{acac})_2$ using the same method as described for the manganese and iron complexes.

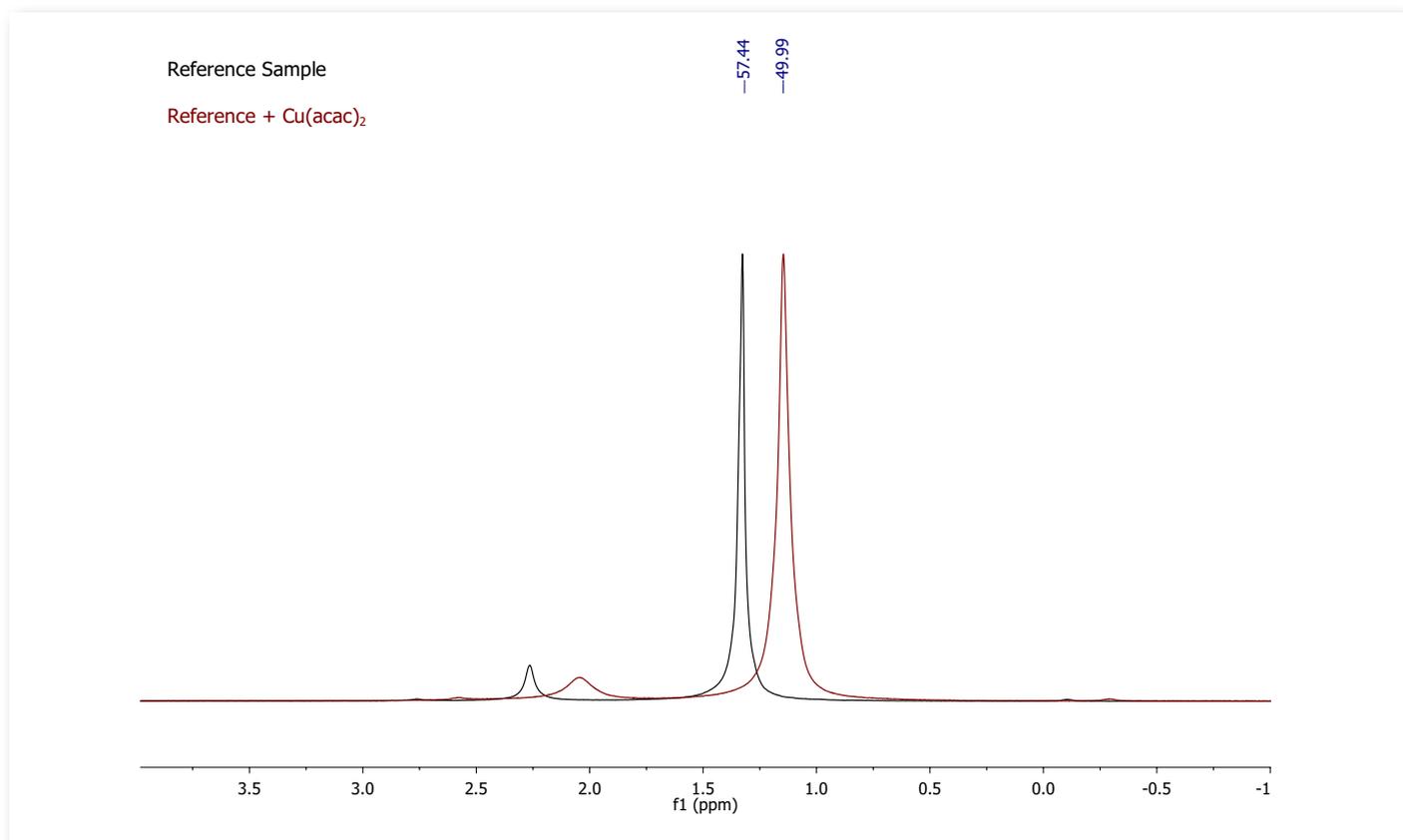


Figure 13: ^1H -NMR spectrum of the reference sample (black) and the reference with $\text{Cu}(\text{acac})_2$ (red) overlaid. The peaks are labelled in Hz.

Calculations

Molar susceptibility:

$$[\text{Cu}(\text{acac})_2] = 0.076 \text{ mol dm}^{-3}$$

$$\Delta f = 57.44 - 49.99 = 7.45 \text{ Hz}$$

$$f = 42,500,000 \text{ Hz}$$

$$\text{Using Equation 2, } \chi_m = 1.38 \times 10^{-8} \text{ m}^3 \text{ mol}^{-1}.$$

Effective magnetic moment:

$$T = 294.16$$

$$\text{Using Equation 4, } \mu_{\text{eff}} = 1.61 \mu_B.$$

Use Equation 5 to estimate the expected effective magnetic moment for $\text{Cu}(\text{II})$, which is a d^9 complex and therefore 1 unpaired electron.

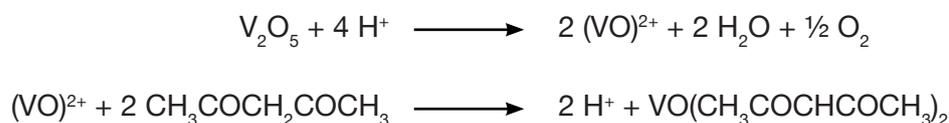
$$n = 1$$

$$\mu_{\text{eff}} = 1.73 \mu_B$$

The estimated magnetic moment agrees well with the experimentally determined value.

Additional complexes:

(VO)²⁺ Complex

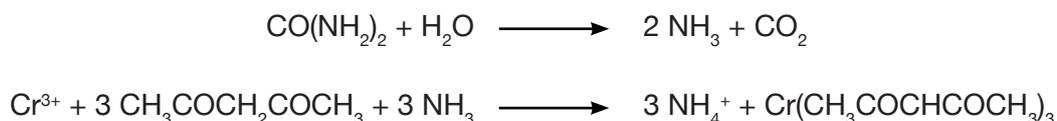


Caution: Vanadium pentoxide (V_2O_5) is toxic.

To a round-bottom flask, add 5 mL distilled water, followed by slow addition of concentrated H_2SO_4 . Then add 12 mL ethanol then 2.5 g vanadium pentoxide (V_2O_5). Fit the round bottom flask with a condenser and reflux for 90 minutes, which should result in a dark blue-green solution. Allow the mixture to cool and filter using cotton wool, discarding any solid residue. With stirring, add 6 mL of acetylacetone dropwise. To neutralise the solution, add it carefully with stirring to a large conical flask containing 20 g anhydrous sodium carbonate dissolved in 150 mL distilled water. The mixture should be cooled in an ice bath and the resulting precipitate filtered using Buchner filtration. Wash the solid with cold distilled water and dry the product in a vacuum desiccator. Determine the percentage yield of this dry product. Dissolve a small portion of the product (about 0.5 g) in 6 mL dichloromethane and carefully decant from any residue. Add 20 mL of petroleum ether, swirl the mixture and allow to stand for 10 minutes. Filter the product using Buchner filtration, rinse with cold petroleum ether and dry in a vacuum desiccator.

The resulting blue complex is a d^1 paramagnetic compound.

Cr³⁺ Complex



To a conical flask, add 1.4 g chromium(III) chloride ($\text{CrCl}_3 \cdot 6 \text{H}_2\text{O}$) and dissolve it in 50 mL distilled water. In 3 or 4 portions, add 10 g urea with stirring. Add 3 mL acetylacetone, cover with a watch glass and heat to 80-90°C. Initially the solution should be very dark, but as the reaction proceeds, maroon crystals start to form. After 90 minutes of heating cool the reaction mixture and filter the precipitate using Buchner filtration. Do not rinse the product. Dry the product in air and record the percent yield. Dissolve a small sample in boiling petroleum ether, decant the solution, and reduce the volume by half. Cool the solution in an ice bath, filter the crystals using Buchner filtration and suck dry at the water pump.

The resulting purple complex is a d^3 paramagnetic compound.

Acetylacetone Tautomerisation

Place 5 drops of acetylacetone in an NMR tube and add 0.5 mL CDCl_3 . Obtain a ^1H -NMR spectrum of the sample using Quick Scan (1 scan, 10 seconds) in the 1D Proton protocol.

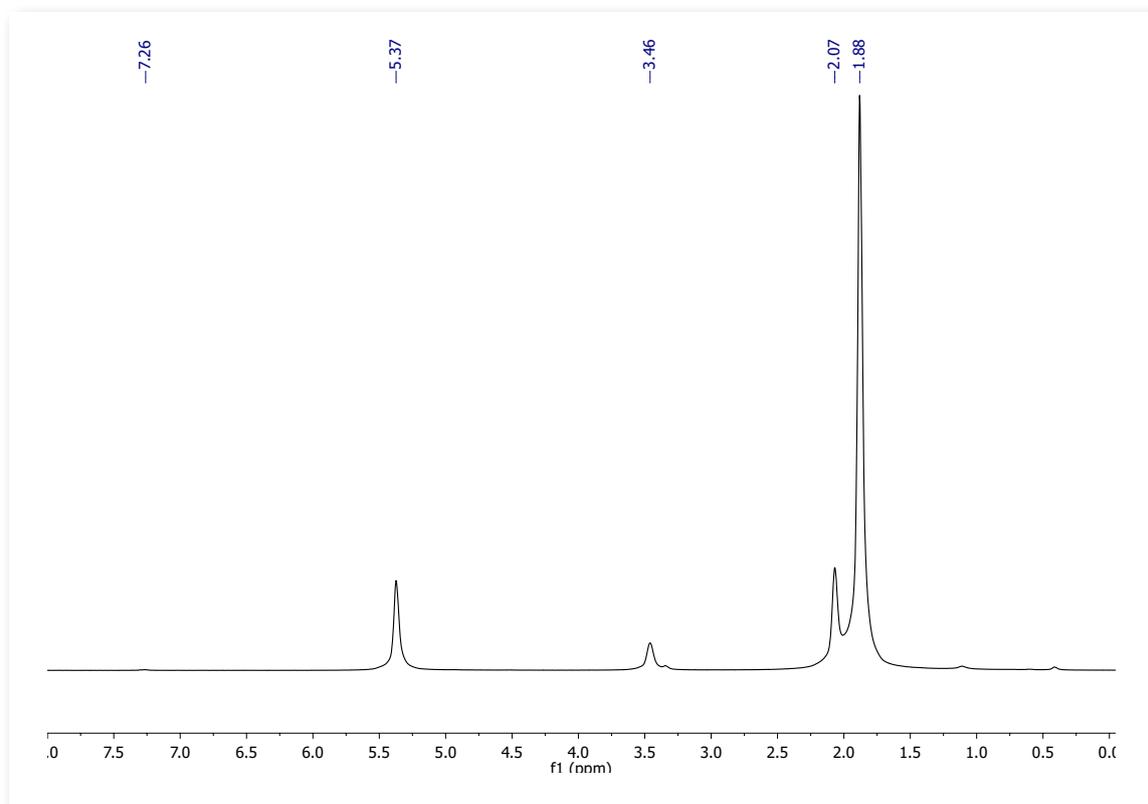


Figure 14: ^1H -NMR spectrum of acetylacetone.

The ^1H -NMR spectrum of acetylacetone (Figure 14) contains four resonances, δ 5.37, δ 3.46, δ 2.07 and δ 1.88. This is evidence that both the keto and enol tautomers are present in solution. Since the enol form of acetylacetone can be regarded as a complex of H^+ (in place of a metal), compare the spectrum above to that of $\text{Al}(\text{acac})_3$ or $\text{Co}(\text{acac})_3$.

The chemical shifts of the methyl groups (δ 1.88 and δ 2.07) in the ^1H -NMR spectrum of acetylacetone are similar, however the remaining resonances are very different (δ 5.37 and δ 3.46). The CH proton on the backbone of the ligand in the aluminium and cobalt complexes is around 5 ppm. This means the resonance at δ 5.37 is the CH proton of the enol tautomer. This tautomer, like the metal complexes, has a 1:6 peak relationship (one CH and two equivalent methyl groups) thus the resonances δ 5.37 and δ 1.88 correspond to the enol tautomer. This leaves the resonances δ 3.46 and δ 2.07 as the keto tautomer. In this tautomer, the backbone carbon has two protons attached, thus the ratio between the two peaks is 1:3, which is observed in the spectrum.

With the resonances in the spectrum assigned to each tautomer, the keto:enol ratio can be calculated. The CH of the enol tautomer and CH₂ of the keto tautomer are well separated, therefore their integrals can be used to determine the ratio.

Integrate the peaks at δ 5.37 and δ 3.46 and divide the keto integral by two, the number of protons it accounts for in the molecule (Figure 15).

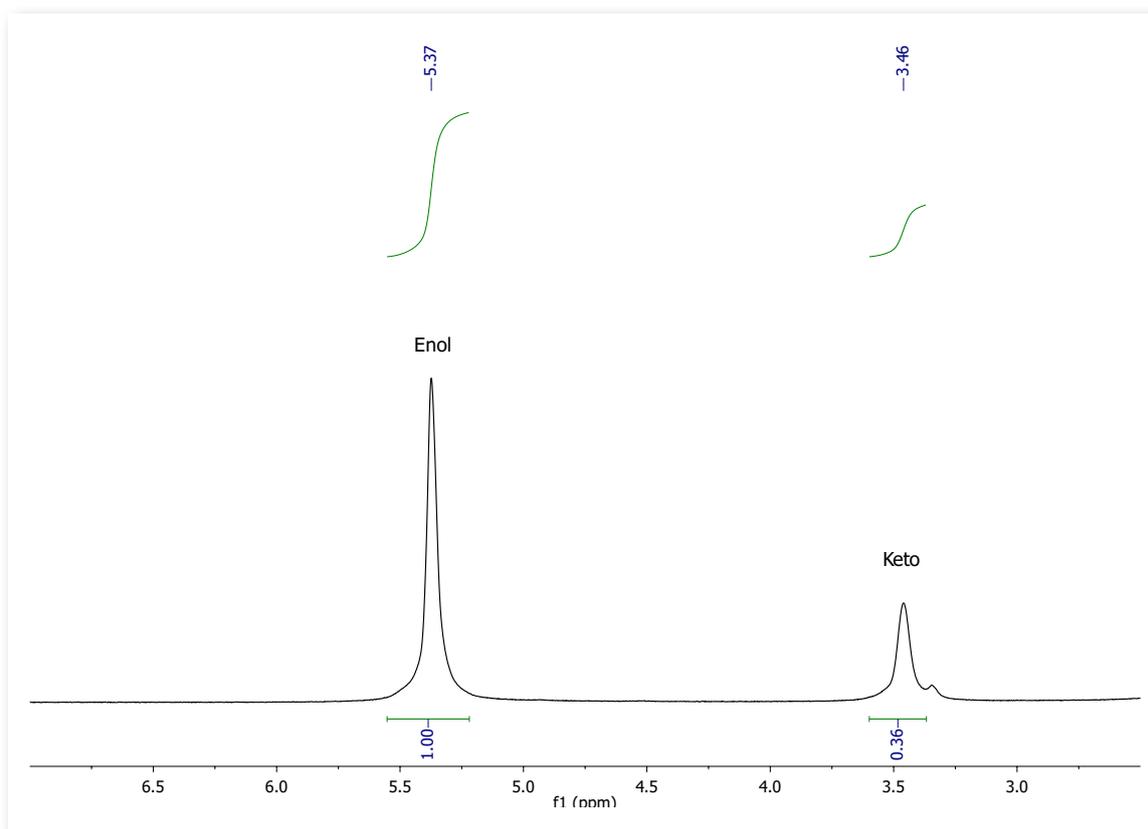


Figure 15: Determination of keto:enol ratio using the backbone protons of the tautomers of acetylacetone.

The keto resonance (δ 3.46) has an integral of 0.36, thus normalising to correspond this to concentration (dividing by two) gives 0.18. Therefore the keto:enol ratio is about 1:6 in chloroform.



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