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Composition of Some Common Household Products

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Contents

Manuscript prepared by Sarah Moore.

This experiment using the Spinsolve is carried out with final-year high school chemistry students, who do an analytical instrumentation laboratory course, at RMIT in Melbourne, Australia. This lab manual has been adapted from the material kindly provided by Dr Jeff Hughes such that it is also suitable for undergraduates being introduced to NMR spectroscopy.

Objectives

In this experiment, students use ^{1}H-NMR spectroscopy to determine the main organic compounds found in common household products such as vinegar, nail polish remover and spirits. This is done by comparing the spectra of household products with spectra of common laboratory solvents.

Experiment

You will be allocated one household product and one reference solvent from the list below. Add approximately 0.5 mL of each liquid to a 5 mm NMR tube. Measure the ^{1}H-NMR spectrum of each of your samples (Quick Scan). Phase your spectra and integrate the peaks. Record the information obtained from the NMR spectrum of the reference solvent in a table. In groups of six (one of each household product and reference solvent), identify the organic compounds present in each household product (there may be more than one). Determine the volume percentage composition of each component.

Household product:	**Reference solvent:**
Nail polish remover (pink)	Acetone
Nail polish remover (blue)	Ethyl Acetate
Rubbing alcohol	Ethanol
Vinegar	Acetic Acid
Vodka	Water
Methylated Spirits	Isopropanol

Safety:

All of the liquids are highly flammable and should be kept away from heat and open flames. Acetic acid is corrosive and may cause burns to skin and eye damage.

Questions

1. By comparing the spectrum of your household product with the reference spectra what compounds can you identify?

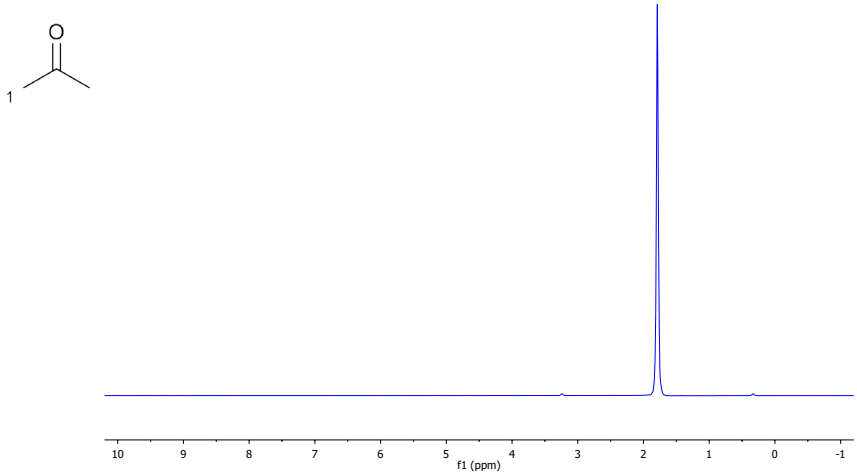
2. Does the composition you calculated agree with the label on the bottle? (For products with one compound, does the bottle specify only one major ingredient?).

3. What properties of the compounds you identified make them useful in the product they are incorporated in?

Example Spectra

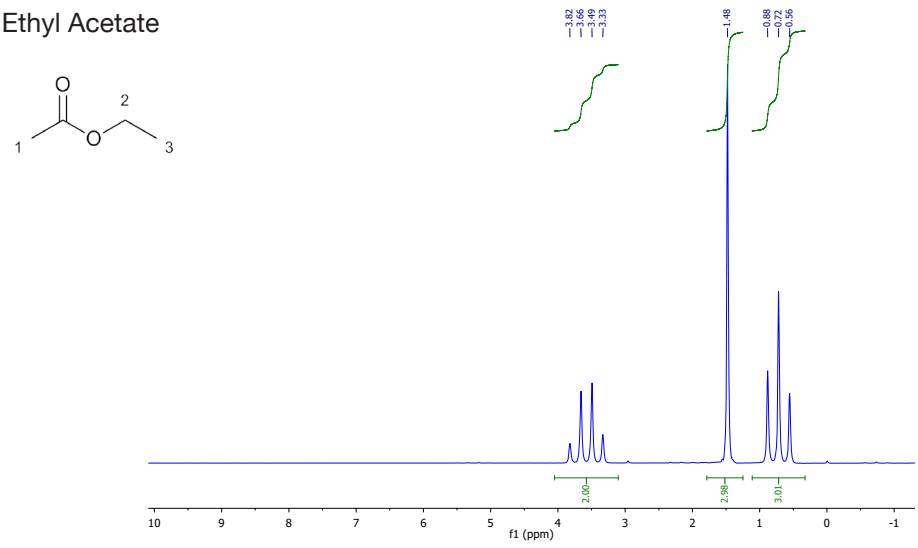
Reference solvents

Acetone



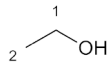
Chemical Shift (ppm)	Integration	Multiplicity	Assignment
1.8	-	singlet	1

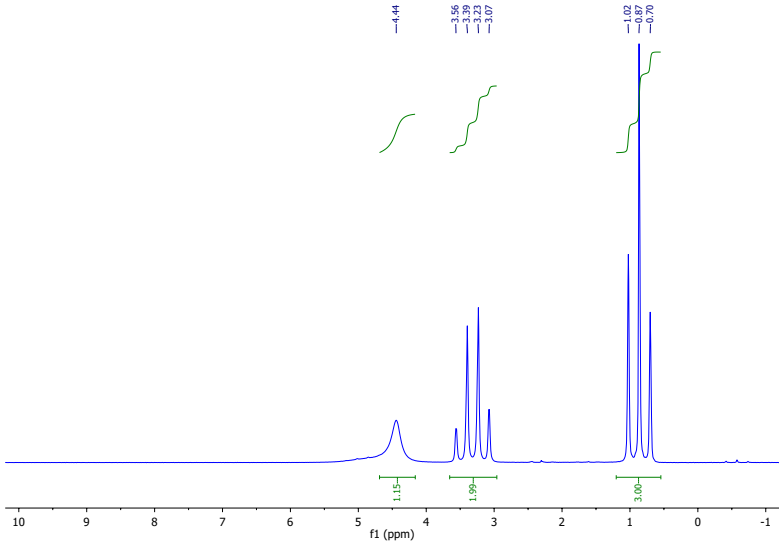
Ethyl Acetate



Chemical Shift (ppm)	Integration	Multiplicity	Assignment
3.6	2	quartet	2
1.5	3	singlet	1
0.7	3	triplet	3

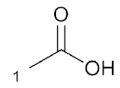
Ethanol

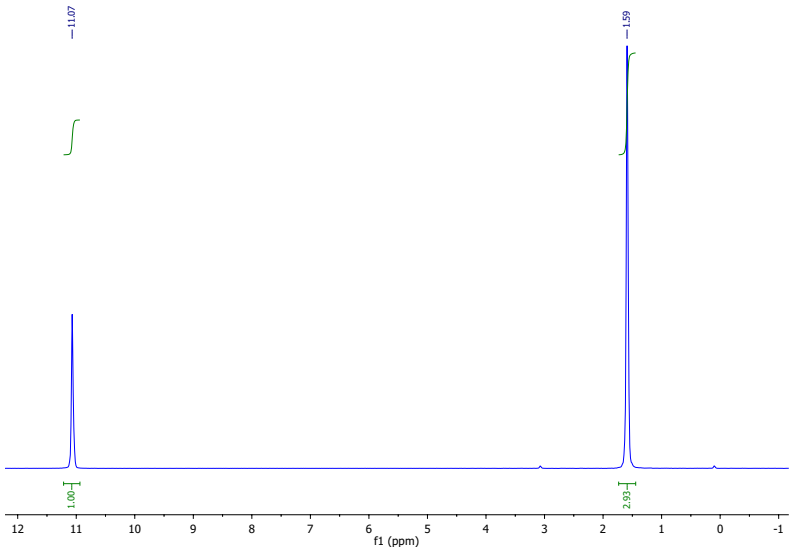




Chemical Shift (ppm)	Integration	Multiplicity	Assignment
4.5	1	singlet	OH
3.3	2	quartet	1
0.9	3	triplet	2

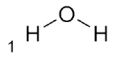
Acetic Acid

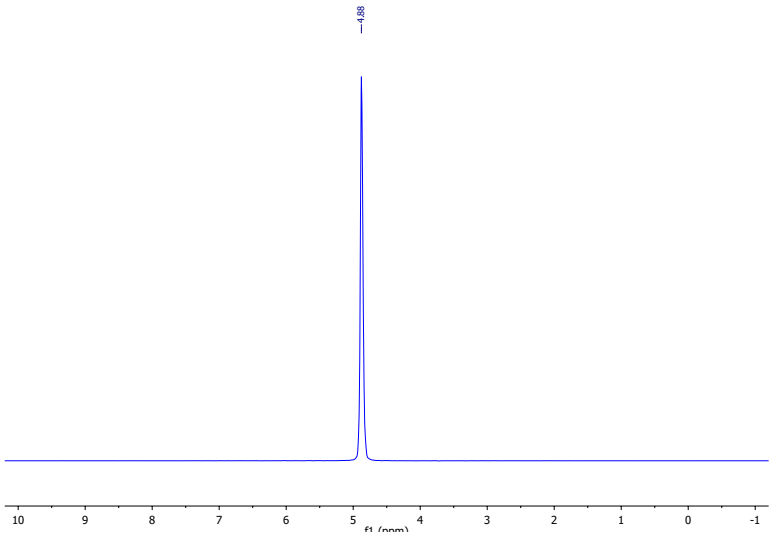




Chemical Shift (ppm)	Integration	Multiplicity	Assignment
11.1	1	singlet	OH
1.6	3	singlet	1

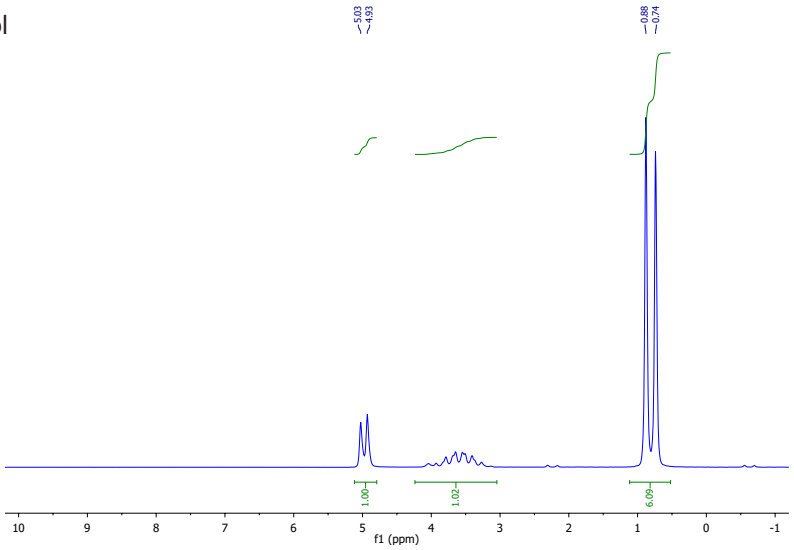
Water





Chemical Shift (ppm)	Integration	Multiplicity	Assignment
4.9	-	singlet	1

Isopropanol



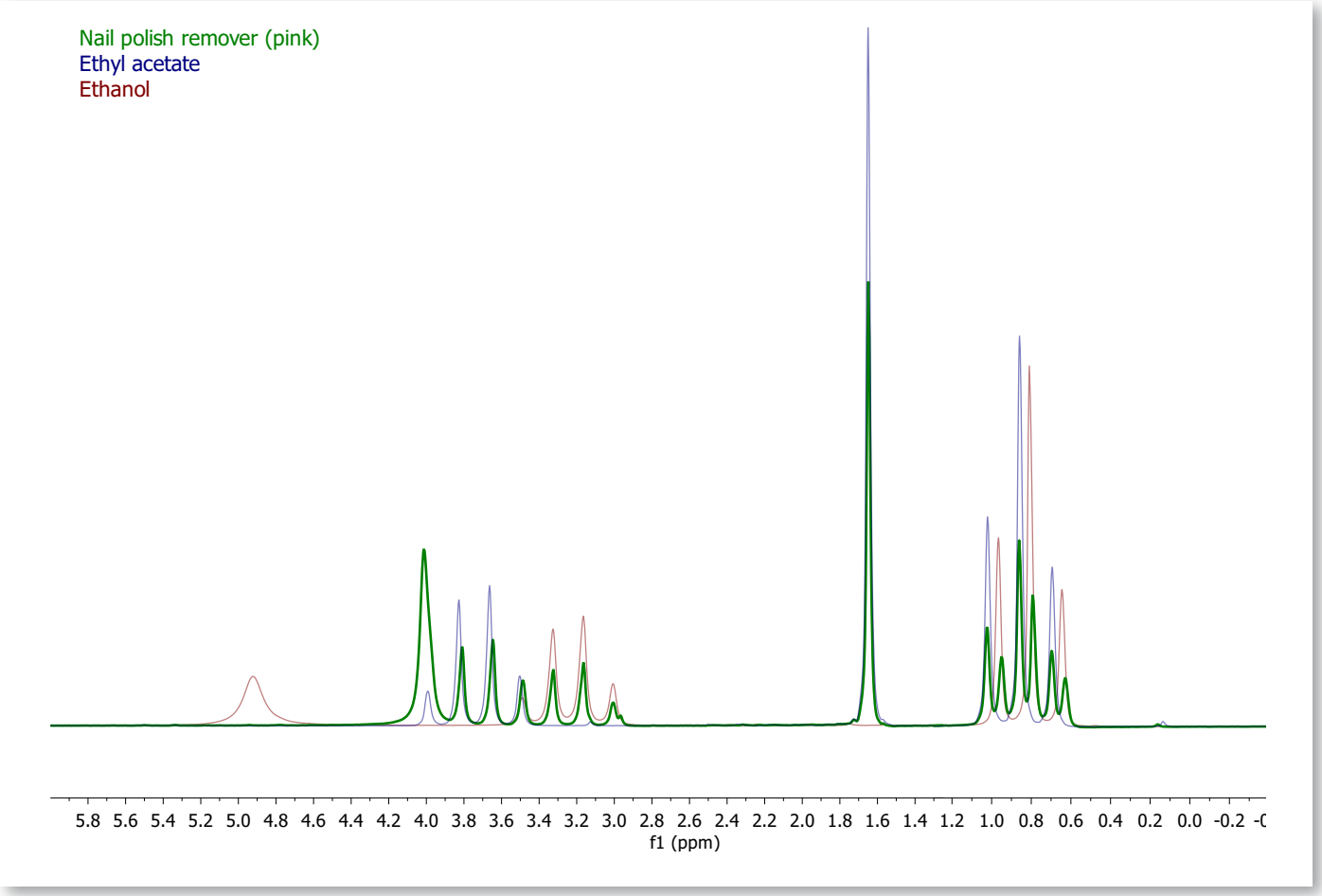
Chemical Shift (ppm)	Integration	Multiplicity	Assignment
5.0	1	doublet	OH
3.6	1	multiplet	1
0.8	6	doublet	2

Household Products

Pink Nail Polish Remover

From the integrals in the spectrum above it is clear that there is more than one compound in the pink nail polish remover sample. The spectrum has peaks in regions very similar to ethyl acetate, thus the integral of the peak at 1.6 ppm, which appears to be a single compound, was set to 3 (ethyl acetate methyl). If one compound is ethyl acetate, the chemical shifts for the other compound(s) is very similar to the ethyl chain of ethyl acetate. This, combined with the presence of an exchangeable proton at about 4 ppm, suggests that the other compound is likely ethanol. The exchangeable peak is too large to be a result of just ethanol, therefore there is some water in the sample too.

In order to confirm the assignments the spectra were superimposed in Mnova below. The splitting of the two methylene quartets and methyl triplets of the ethyl chain of ethanol and ethyl acetate are clearly explained in this exercise.



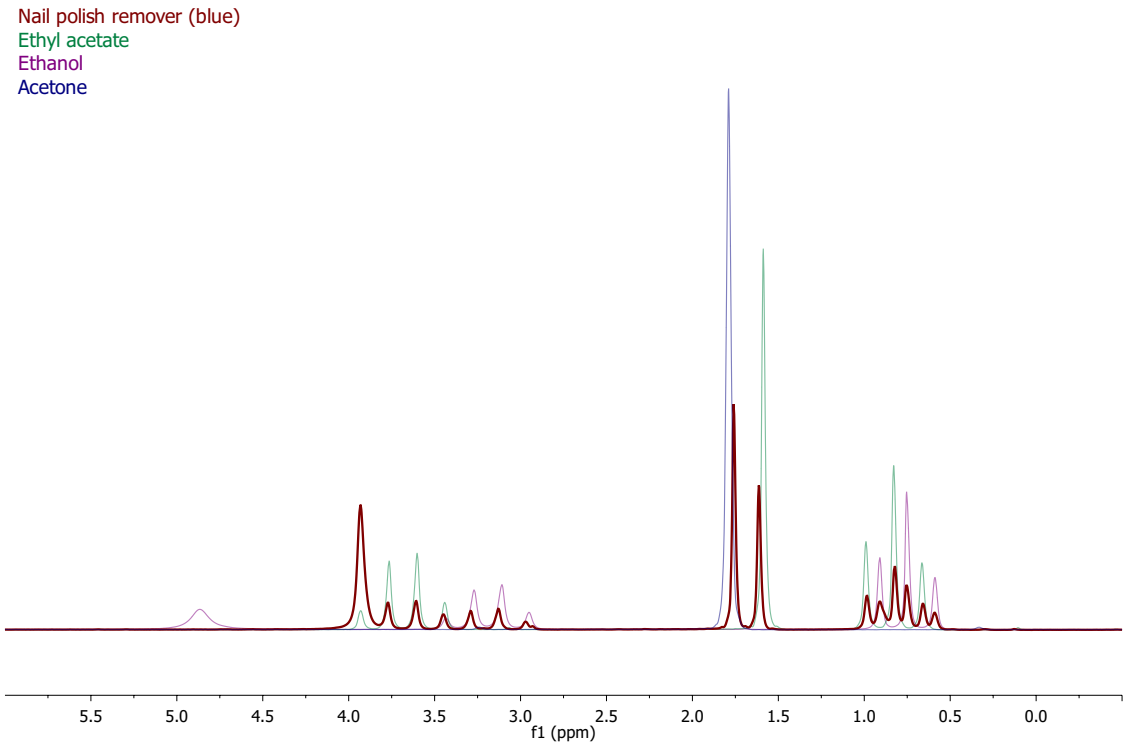
Percentage composition:

The singlet at 1.7 ppm corresponds to the methyl adjacent to the carbonyl of ethyl acetate, thus accounts for three protons. The remaining integrals were normalised using this fact. The other methyl of ethyl acetate is more up-field and coincides with the methyl group of ethanol. Because the integrals were normalised to the singlet at 1.7 ppm, 3 of the 5.25 protons correspond to the methyl of ethyl acetate. Therefore the remaining 2.25 protons are the methyl of ethanol. The methylene peaks and exchangeable protons all coincide downfield between 4.3 and 2.9 ppm. This includes the methylene group of ethyl acetate which accounts for two of the 6.61 protons. In this region there are three protons resulting from ethanol – the methylene and OH, thus ethanol corresponds to 2.25 protons in the integral. The remaining 2.36 protons can therefore be attributed to water.

Integrals			**Assignment**	**Integral of 1 proton**	**Mass (g)**	**Volume (mL)**	**% Composition (v/v)**
6.61	3.00	5.25	Total	-	-	Total = 162.71	100
2.00	3.00	3.00	Ethyl acetate	1.00	88.11	97.68	60.0
2.25	0	2.25	Ethanol	0.75	34.55	43.79	26.9
2.36	0	0	Water	1.18	21.24	21.24	13.1

Blue Nail Polish Remover

The spectrum of the blue nail polish remover is very similar to that of the pink, but it has an additional peak at 1.8 ppm. This is the only additional peak visible in the spectrum. Acetone is the only refrernce solvent that contains just one peak, threfore the blue nail polish remover contains ethyl acetate, ethanol, water and acetone. This is confirmed by superimposing the spectra below.

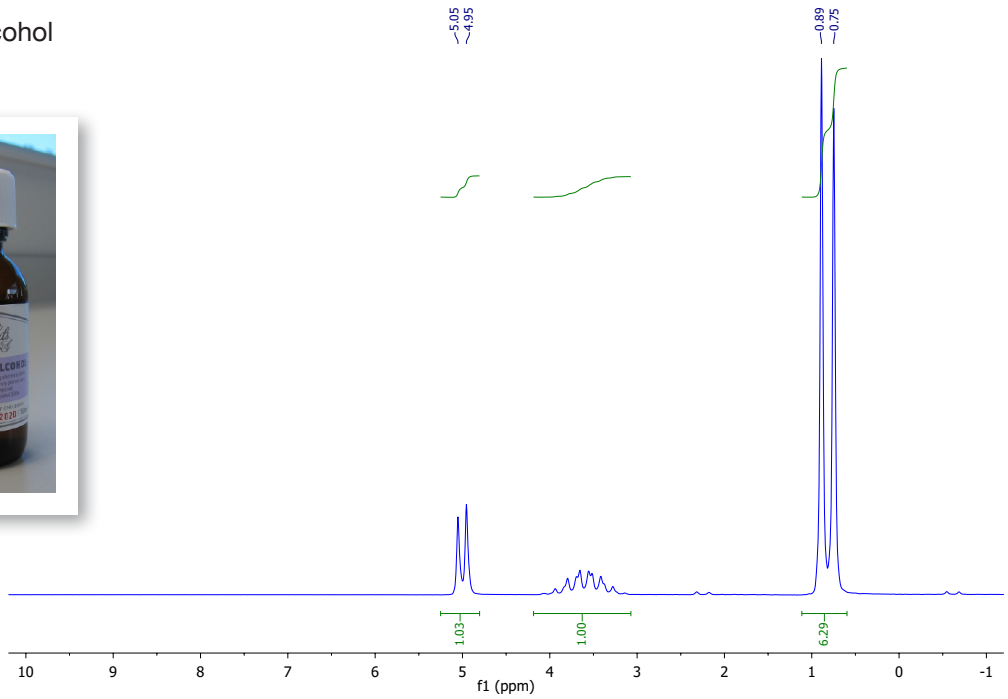


Percentage composition:

Use similar logic to pink nail polish remover example.

Integrals				Assignment	Integral of 1 proton	Mass (g)	Volume (mL)	% Composition (v/v)
2.91	1.48	1	1.94	Total	-	-	Total = 76.95	100
0	1.48	0	0	Acetone	0.25	14.52	18.36	23.9
0.67	0	1.00	1.00	Ethyl acetate	0.33	23.37	25.91	33.7
0.63	0	0	0.94	Ethanol	0.31	14.28	18.10	23.5
1.61	0	0	0	Water	0.81	14.58	14.58	18.9

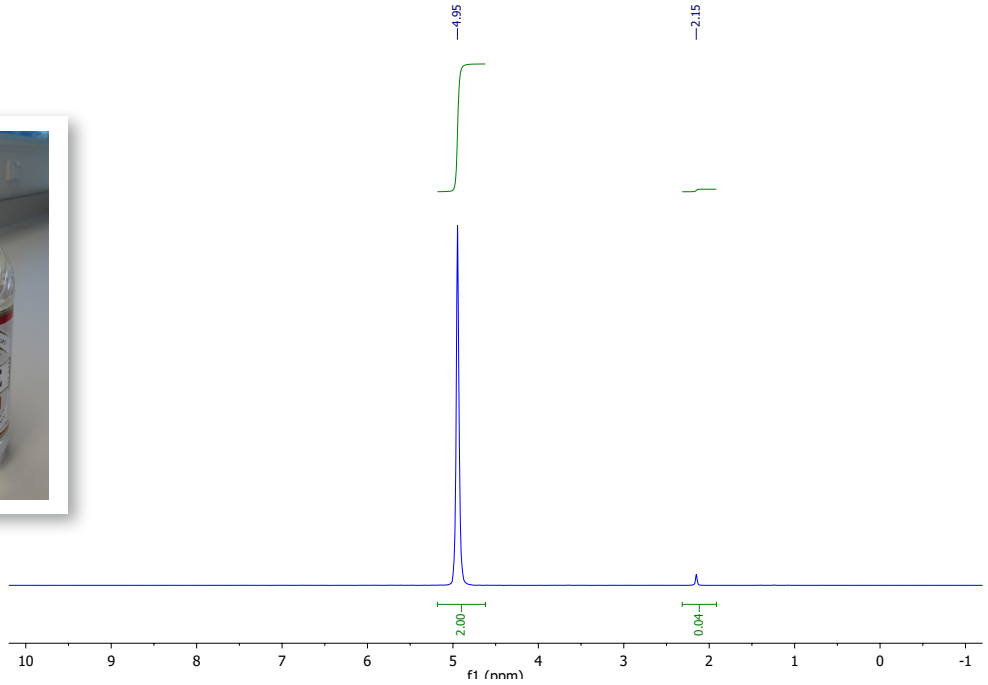
Rubbing Alcohol



The rubbing alcohol appears to contain only one component. The spectrum is identical to that of isopropanol.

Integrals			Assignment	Integral of 1 proton	Mass (g)	Volume (mL)	% Composition (v/v)
1.03	1.00	6.29	Total	-	-	Total = 76.46	100
1.03	1.00	6.29	Isopropanol	1	60.1	76.46	100

Vinegar

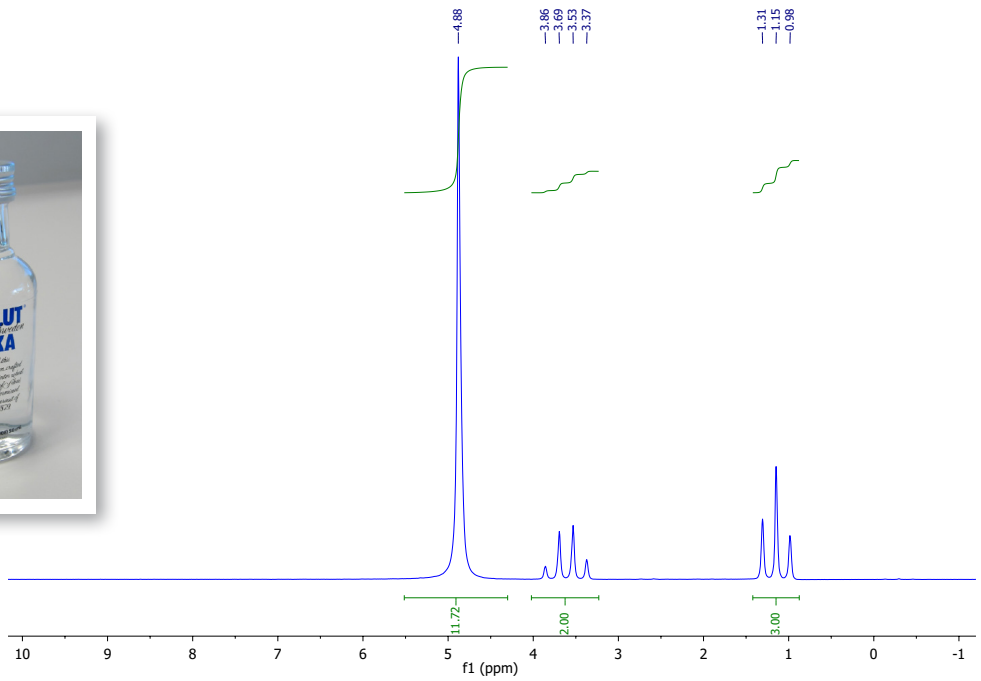


The NMR spectrum of vinegar contains two peaks. The large peak at 5 ppm must be water. The remaining peak is a singlet. There are two reference solvents that have an isolated, up-field singlet – acetone and acetic acid. The acetone peak would be expected to appear more more shielded, around 1.5 ppm. The peak in the reference solvent of acetic acid is 1.3 ppm, but because acetic acid is an organic acid, the chemical shift will vary with concentration and pH. The same behaviour is not expected for acetone, thus vinegar contains water and acetic acid.

Percentage composition:

Integrals		Assignment	Integral of 1 proton	Mass (g)	Volume (mL)	% Composition (v/v)
2	0.04	Total	-	-	Total = 18.63	100
0.013	0.04	Acetic acid	0.013	0.78	0.74	4.0
1.987	0	Water	0.994	17.89	17.89	96.0

Vodka

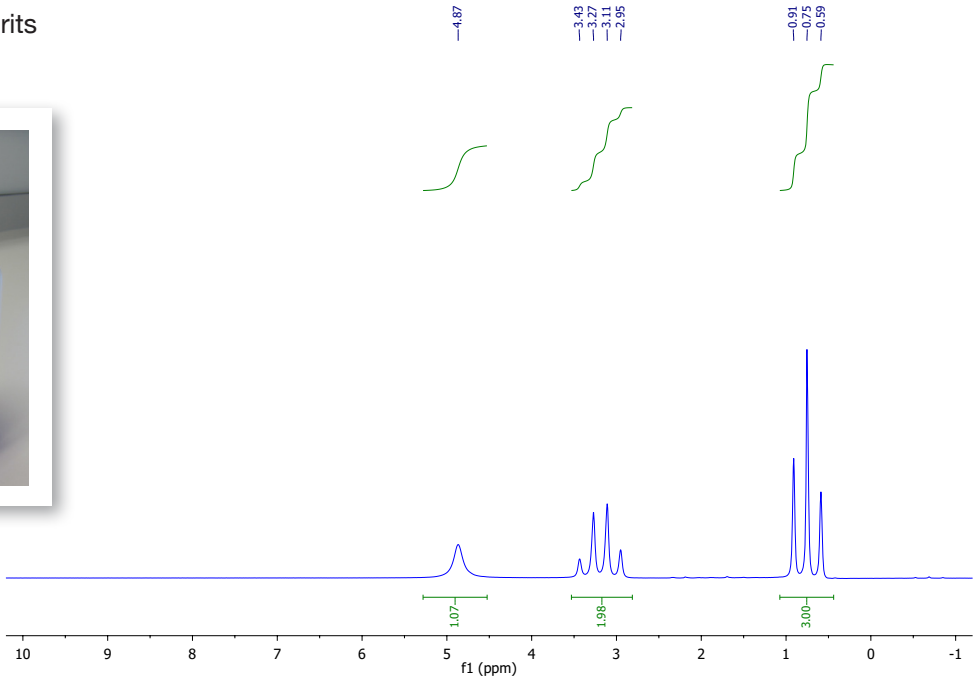


The spectrum of vodka appears to be identical to that of ethanol, but the exchangeable proton integrates for a lot more than 1 so must also contain water.

Percentage composition:

Integrals			Assignment	Integral of 1 proton	Mass (g)	Volume (mL)	% Composition (v/v)
11.72	2	3	Total	-	-	Total = 154.87	100
1	2	3	Ethanol	1	46.07	58.39	37.7
10.72	0	0	Water	5.36	96.48	96.48	62.3

Methylated Spirits



The spectrum of methylated spirits is identical to that of ethanol.

Integrals			Assignment	Integral of 1 proton	Mass (g)	Volume (mL)	% Composition (v/v)
1.07	1.98	3.00	Total	-	-	Total = 58.39	100
1.07	1.98	3.00	Ethanol	1	46.07	58.39	100

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