

1CHEM 202 Laboratory report

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Experiment 2

Lab Day:

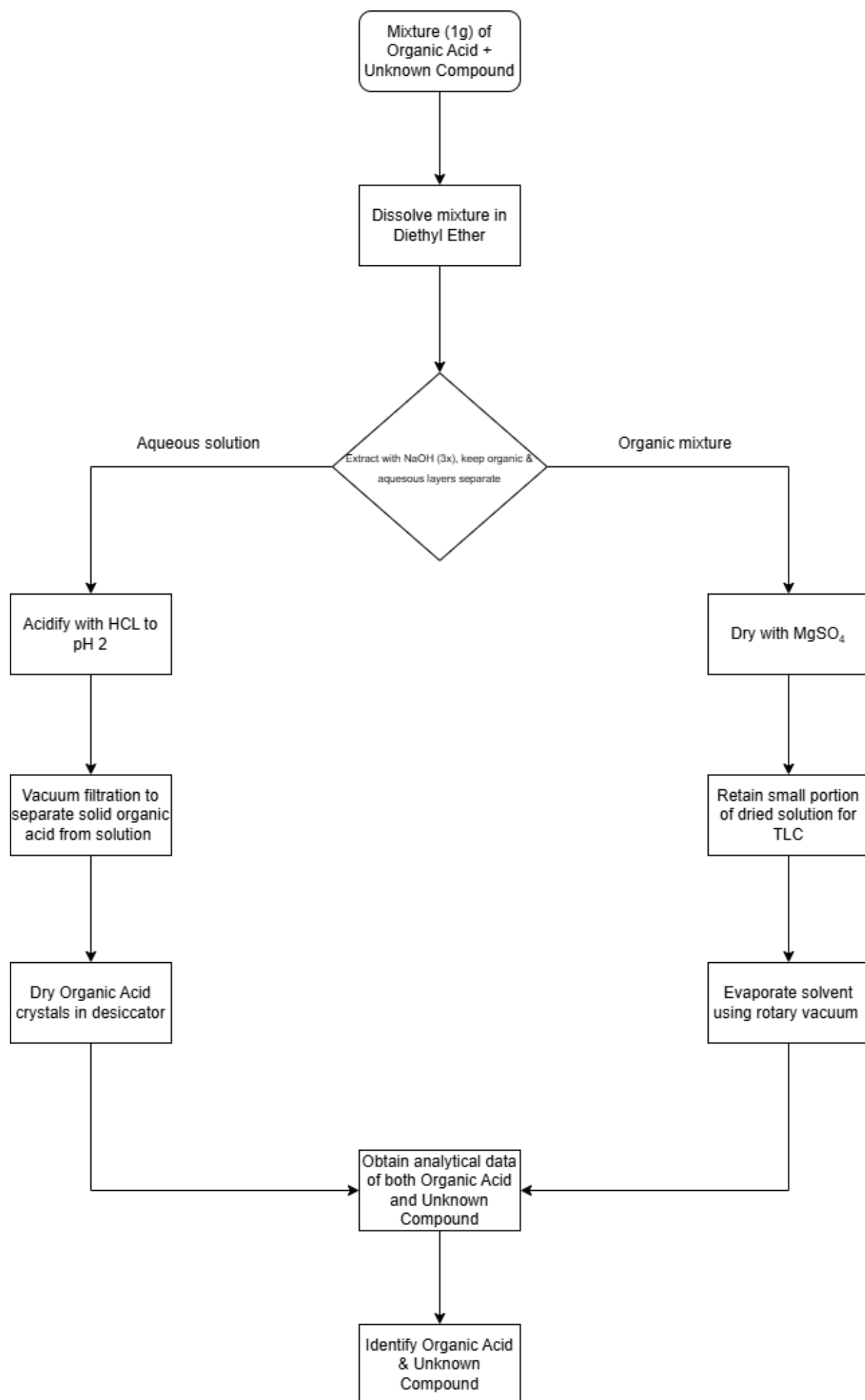
Thu

Date: 13-03-25

Separation of an organic acid and an unknown compound by solvent extraction

Abstract

In this experiment, a mixture of a solid organic acid and an unknown compound was separated into the two components via solvent extraction. The organic acid was then recrystallised and analysed by melting point, IR spectroscopy, mass spectroscopy, ^{13}C NMR to determine its identity, while the unknown compound was identified using thin layer chromatography and IR spectroscopy.



Experimental

The experiment was performed as detailed in the CHEM 202 laboratory manual (p21), without modification.

Aqueous waste generated from the separation and extraction of compounds was disposed of down the fumehood sink. Magnesium sulfate used to dry the organic extracts was also disposed of down the fumehood sink with the aid of cold water. Ether was removed from the product using a rotary evaporator, and any residual solvent or rinses were collected in a designated organic waste container for proper disposal.

Results for the Analysis of Sample 623

- **Identification of the organic acid**

Microanalysis shows the following percentage composition by mass:

C: 68.85 %; H: 4.95 %

The empirical formula of the product can be calculated as follows:

$$\% \text{ of Oxygen} = 100 - (68.85 + 4.95) = 26.2 \%$$

$$\text{Moles of Carbon} = \frac{68.85 \text{ g}}{12 \text{ g mol}} = 5.738 \text{ mol}$$

$$\text{Moles of Hydrogen} = \frac{4.95 \text{ g}}{1 \text{ g mol}} = 4.95 \text{ mol}$$

$$\text{Moles of Oxygen} = \frac{26.2 \text{ g}}{16 \text{ g mol}} = 1.638 \text{ mol}$$

$$\text{C ratio} = \frac{5.738 \text{ mol}}{1.638 \text{ mol}} \approx 3.5$$

$$\text{C: } 3.5 \times 2 = 7$$

$$\text{H ratio} = \frac{4.95 \text{ mol}}{1.638 \text{ mol}} \approx 3$$

$$\text{H: } 3 \times 2 = 6$$

$$\text{O ratio} = \frac{1.638 \text{ mol}}{1.638 \text{ mol}} \approx 1$$

$$\text{O: } 1 \times 2 = 2$$

Empirical formula: $\text{C}_{3.5}\text{H}_3\text{O}_1$

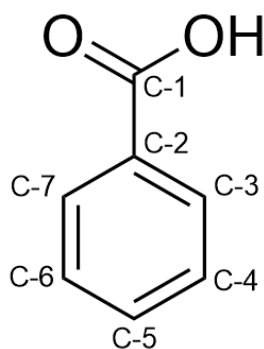
For this formula, $M_r = (3.5 \text{ mol C} \times 12 \text{ g/mol}) + (3 \text{ mol H} \times 1 \text{ g/mol}) + (1 \text{ mol O} \times 16 \text{ g/mol}) = 61 \text{ g}$

From the mass spectrum, m/z for M^+ (product) = 122 g

The product of the experiment is: (Formula: $\text{C}_7\text{H}_6\text{O}_2$)

Degree of unsaturation:

$$\frac{7C \times 2 + 2 - 6}{2} = 5$$



Benzoic acid

Mass of organic acid = 0.534 g

Melting point = 156 °C

Comparison to literature melting point 121-123 °C (PubChem, n.d.)

The initial reading from the automatic melting point instrument returned a melting point of 156 °C, whereas when tested with an electric hot bench at resulted in a melting point of 124 °C – very close to benzoic acid's melting point of 121-123 °C. This difference suggests the automatic melting point machine may not have been properly calibrated or possibly was not used correctly. Employing the manual method more reliably matched the literature range, indicating the sample is consistent with benzoic acid

IR data of organic acid

ν / cm^{-1}	intensity	appearance	assignment	inference
3071	w	sh	C-H stretch	Aromatic C-H stretch
1678	s	Slightly br	C=O stretch	Carboxylic acid conjugated with aromatic ring
1600	m	sh	C=C	Aromatic skeletal stretch
1582	m	sh	C=C	Aromatic skeletal stretch
703	s	sh	C-H deformation	Monosubstituted benzene derivative

The IR spectrum shows aromatic C-H stretches around 3071 cm^{-1} consistent with an aromatic ring. Strong and slightly broad absorption at 1678 cm^{-1} is a characteristic of the C=O stretch in a carbonyl suggesting the presence of a possible carboxylic acid or ketone and aldehyde. Peaks at 1600 cm^{-1} and 1580 cm^{-1} are consistent with the C=C skeletal stretch in aromatic rings. In the

fingerprint region we see absorption at 703 cm^{-1} consistent with the C-H deformations in a monosubstituted benzene derivative.

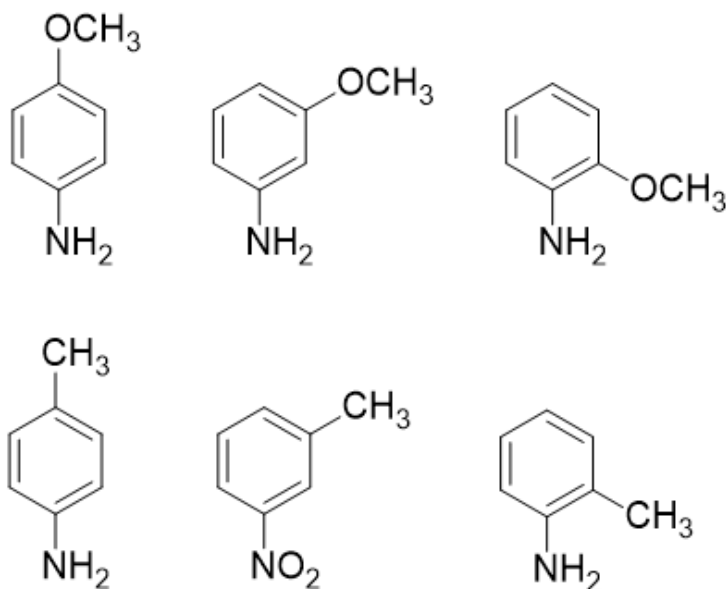
^{13}C NMR data of organic acid

^{13}C NMR (CDCl_3 , 100 MHz)

δ/ppm	Environment	Assignment
172.4	Carboxyl C	C-1
133.9	Aromatic quaternary C	C-2
130.3	Aromatic CH	C-3 or C-6
129.4	Aromatic CH	C-4
128.5	Aromatic CH	C-3 or C-5

The ^{13}C NMR spectrum reveals a carbonyl signal at 172.4, characteristic of a carboxylic acid instead of a ketone or aldehyde. The ring carbons then appear in the aromatic region (about 128-135 ppm), with the quaternary carbon identified as the downfield resonance signal that shows no direct coupling to a proton in the HECTOR spectrum.

- Identification of the other unknown**



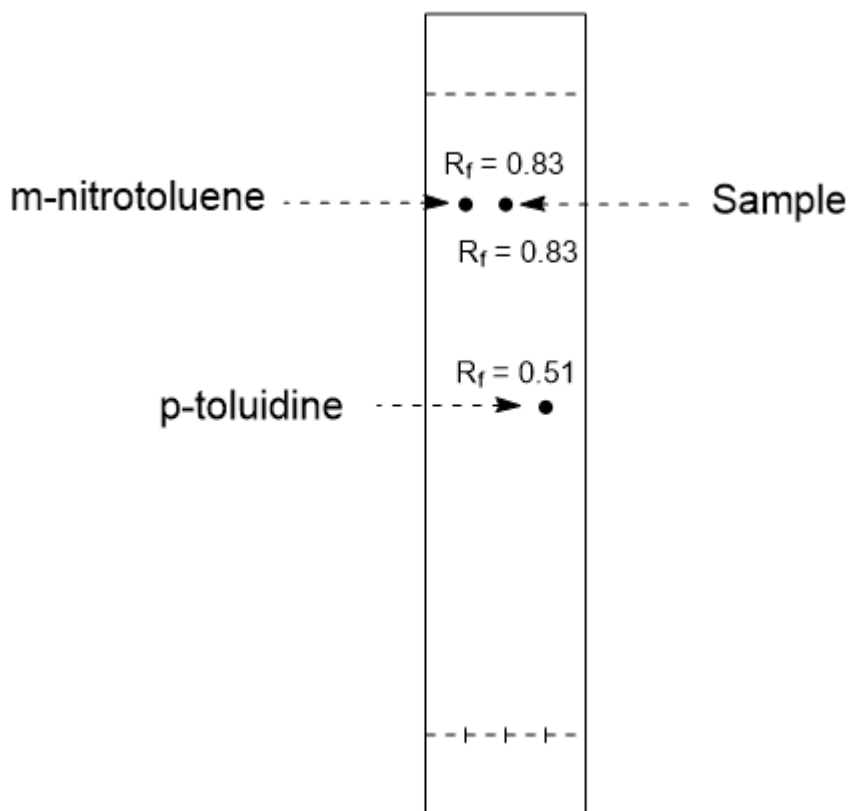
Mass of unknown = 0.096 g

Sample 623

$$\% \text{ Composition of organic acid} = \frac{0.534 \text{ g}}{1.00 \text{ g}} \times 100 = 53.4\%$$

$$\% \text{ Composition of second unknown} = \frac{0.096 \text{ g}}{1.00 \text{ g}} \times 100 = 9.6\%$$

TLC analysis of unknown



In the TLC analysis the unknown sample was spotted alongside of p-toluidine and m-nitrotoluene. The resulting R_f values for the unknown (0.83) exactly matched that of m-nitrotoluene (0.83) and differed substantially from the p-toluidine (0.53). Support the conclusion that the unknown compound is m-nitrotoluene.

IR data of unknown

ν / cm^{-1}	intensity	appearance	assignment	inference
3070	w	sh	C-H stretch	Aromatic C-H stretch
2925	w	sh	C-H stretch	Methyl C-H stretch
2866	w	sh	C-H stretch	Methyl C-H stretch
1521	s	slightly br	N-O symmetric stretch	Asymmetric N-O stretch
1381	w	sh	C-H Bend	Methyl bending
1344	s	slightly br	N-O symmetric stretch	Asymmetric N-O stretch

881	w	sh	C-H deformations	1,3-disubstituted benzene derivative
799	s	sh	C-H deformations	1,3-disubstituted benzene derivative

The IR spectrum shows aromatic C-H stretches around 3070 cm^{-1} consistent with an aromatic ring. Weak but sharp absorption at 2985 cm^{-1} and 2866 cm^{-1} suggest the presence of methyl C-H stretches, indicating that at least one methyl substituent on the ring. The two peaks at 1521 cm^{-1} and 1344 cm^{-1} are characteristics of the asymmetric N-O stretching in a nitro (NO_2) group. The peaks at 881 cm^{-1} and 799 cm^{-1} support a 1,3-disubstitution pattern on the benzene ring.

Discussion

When starting both compounds were dissolved in diethyl ether. However, adding aqueous sodium hydroxide selectively deprotonated the organic acid, converting it into a water-soluble carboxylate salt, allowing it to move into the aqueous layer. The neutral aromatic compound, lacking acidic functionality, remained in the organic layer. Subsequent re-acidification of the aqueous layer with hydrochloric acid regenerated the organic acid, which precipitated and was isolated through vacuum filtration and recrystallization.

Characterization of the organic acid was done using multiple analytical techniques. Microanalysis and mass spectrometry provided an empirical formula of $\text{C}_7\text{H}_6\text{O}_2$ and showed a molecular mass of 122 g/mol . The empirical formula allowed to calculate degree of unsaturation which came out to five, confirming the presence of an aromatic ring and an additional functional group, consistent with a carboxylic acid. IR spectroscopy showed key characteristic absorptions at 1678 cm^{-1} , suggesting a conjugated carboxylic acid carbonyl stretch. Aromatic C-H stretches appeared around 3071 cm^{-1} , and aromatic skeletal vibrations near 1600 and 1582 cm^{-1} indicated an aromatic ring structure. Additionally, a peak at 703 cm^{-1} was typical of a monosubstituted benzene ring. Analysis using ^{13}C NMR spectroscopy showed a distinct carbonyl resonance at 172.4 ppm , characteristic of carboxylic acid rather than aldehydic or ketonic groups. Multiple aromatic carbon signals observed between 128 and 135 ppm further supported the presence of an aromatic structure. All of this spectral data allowed for the deduction of the molecular structure to be benzoic acid ($\text{C}_6\text{H}_5\text{COOH}$), a monosubstituted benzene ring attached directly to a carboxylic acid functional group. The melting point of the recrystallized acid measured by the automatic instrument was 156°C , but the manual electric hot bench provided a melting point of 124°C , much closer to the literature value of $121\text{--}123^\circ\text{C}$, indicating improved accuracy with the manual method and further affirming its identity as benzoic acid.

The neutral aromatic unknown was analysed by IR spectroscopy and thin-layer chromatography (TLC). IR data provided evidence of an aromatic compound bearing methyl substituents, as indicated by peaks around 2925 cm^{-1} and 2866 cm^{-1} . The presence of a nitro group was supported by strong absorptions at 1521 cm^{-1} and 1344 cm^{-1} . The distinct out-of-plane deformation peaks at 881 cm^{-1} and 799 cm^{-1} confirmed a 1,3-disubstitution pattern on the benzene ring. These spectral features suggest that the neutral compound is m-nitrotoluene. TLC analysis showed that the unknown compound exhibited an R_f value of 0.83 , identical to the R_f value of m-nitrotoluene (0.83) and distinct from that of p-toluidine (0.51), supporting its identification as m-nitrotoluene.

References

PubChem. (n.d.). Benzoic acid. PubChem. <https://pubchem.ncbi.nlm.nih.gov/compound/Benzoic-Acid#section=Boiling-Point>

