

Separation and Analysis of Three Unknown Solids

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Lab Section 1A05

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Figure

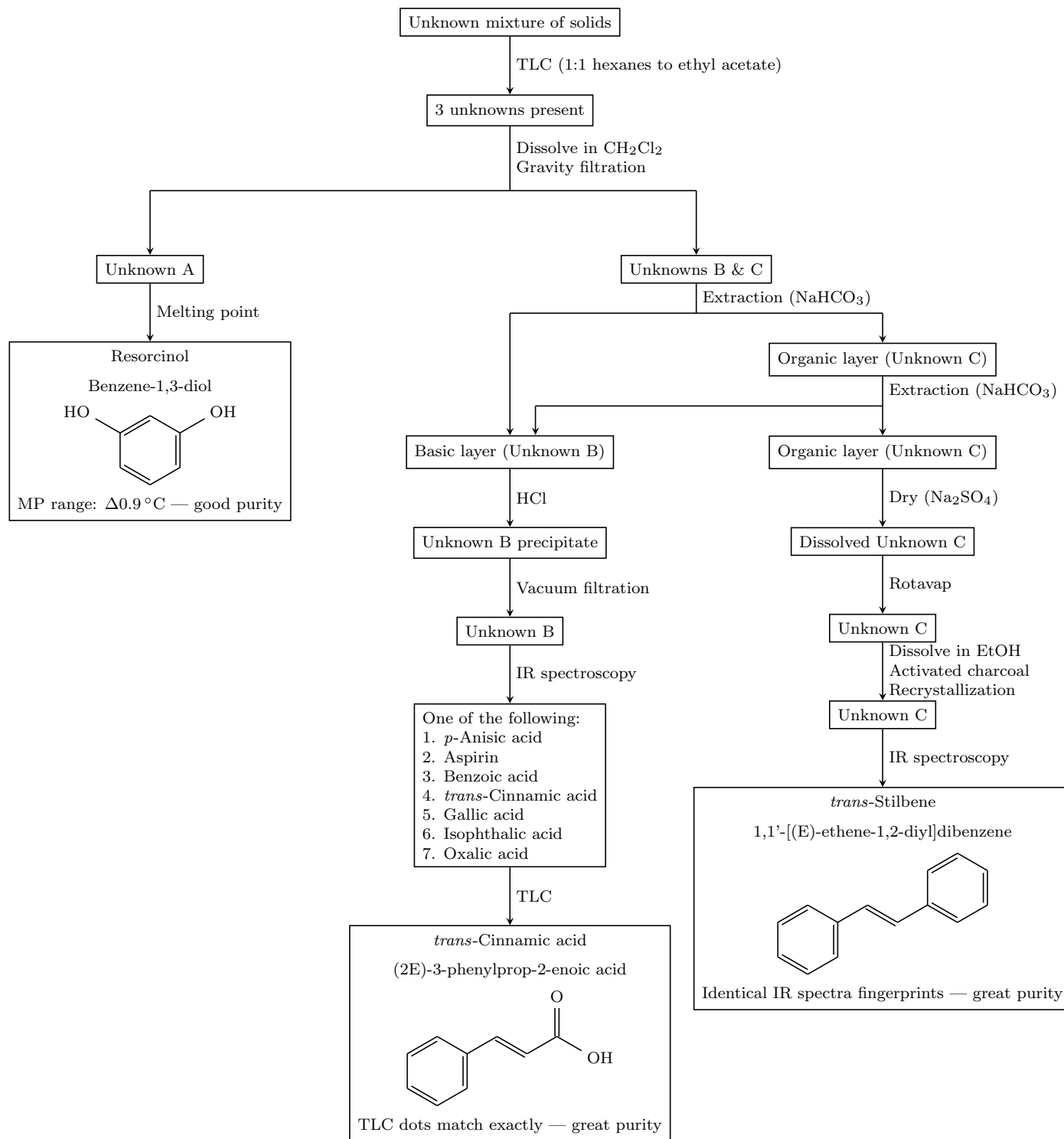
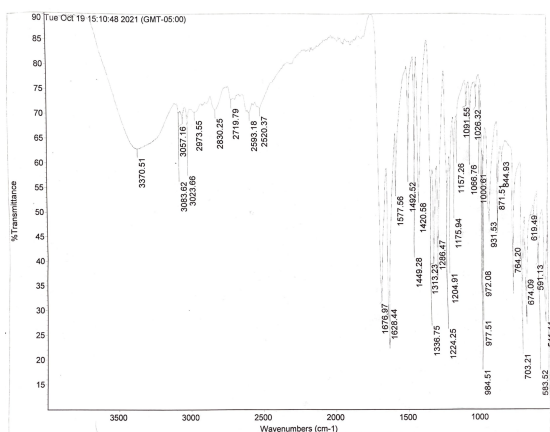


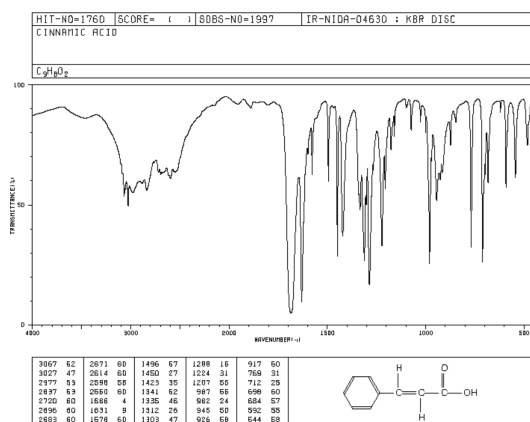
Figure 1: Isolation and purification process for a selection of unknown solids.

Lab Report

- What is the identity of unknown A and how did you determine this?
 - Unknown A is resorcinol. The experimental melting point range of 108.7°C to 109.6°C falls within the known melting point range of resorcinol, and is disjoint from all other known melting point ranges.
- Comment on the relative purity of unknown A and how you determined this.
 - Unknown A is pretty pure. With a ramp rate of 1°C/min, the change in temperature from when the sample started to melt to when it finished melting was only 0.9°C.
- What is the identity of unknown B and how did you determine this? Discuss both the IR and TLC data. Include a published IR spectrum in your report and comment on similarities between it and the spectrum you obtained.



(a) Experimental.

(b) Known¹.Figure 2: *trans*-cinnamic acid IR spectra.

- Unknown B is *trans*-cinnamic acid. The experimentally obtained IR spectrum contains a distinct carboxylic acid peak (see Figure 2a), leading to the conclusion that B must be one of the seven possible compounds containing carboxylic acid functional groups. Comparison of these seven compounds with unknown B on the same TLC plates yielded identical and indistinguishable motion for unknown B and *trans*-cinnamic acid. Lastly, Figure 2 shows that the IR spectrum of unknown B and that of *trans*-cinnamic acid match nicely, especially in the fingerprint region.
- Was the identity of unknown B definitive? In other words, were there any other possibilities?
 - Yes, it was definitive. Based on the exact match of the TLC dots (see notebook pages for sketch), unknown B is certainly *trans*-cinnamic acid.
 - What is the identity of unknown C and how did you determine this?
 - Unknown C is *trans*-stilbene. The IR spectrum obtained for the compound has a fingerprint region that matches nearly perfectly with the fingerprint region of the known IR spectrum of *trans*-stilbene.
 - Comment on the relative purity of unknown C and how you determined this.
 - Unknown C is quite pure. As per the above, the experimental and known IR spectra match nearly exactly.

References

- (1) SDBSWeb National Institute of Advanced Industrial Science and Technology <https://sdb.sdb.aist.go.jp/sdb/cgi-bin/landingpage?sdbno=1997> (accessed 10/20/2021).