

# SPECTRA OF SOLIDS

## Introduction:

Mulling is a technique used to prepare solids for infrared analysis. Good results are obtained by this method only if the average particle size of the solid sample is 1 or 2 microns. Therefore, it is necessary to grind the sample.

The most common mulling agent is mineral oil. Mineral oil is used because it does not have a lot of absorbance in the infrared range. It may be advantageous to take a spectrum of plain mineral oil so the peaks that are caused by the oil can be differentiated when running a sample.

## Purpose:

The purpose of this experiment is to obtain the infrared spectrum of a solid prepared as a mull in mineral oil.

## Equipment / Materials:

mullite or agate mortar and pestle  
mineral oil  
NaCl windows  
Pasteur pipet and bulb  
safety gloves

*Samples:*  
benzoic acid  
citric acid  
talc (baby powder)  
acetylsalicylic acid (aspirin)  
salicylic acid  
sodium bicarbonate (baking soda)  
solid unknowns

## Safety:

- Always wear safety glasses and an apron in the lab.
- Always wear gloves when handling the samples and sodium chloride plates.

**Procedure:**

1. Place 10 to 15 mg of one of the samples in a mortar.
2. Break up the solid and distribute it over the surface of the mortar by lightly grinding with the pestle.
3. Grind the sample with a vigorous, hard, back and forth motion with the pestle until it becomes firmly caked on the sides of the mortar and quite glossy in appearance.
4. Add a small drop of mineral oil to the mortar with a pipet.
5. Grind the sample until all the material is suspended in the mineral oil. It may be necessary to add another drop of mineral oil.

*A note on sodium chloride cells:*

Sodium chloride dissolves in water. Consequently, great care must be taken to ensure that no water or moist materials come in contact with the NaCl cells. For this reason all cells are to be stored in a desiccator at all times. Always clean the cells before and after use. Wipe off the sample with a tissue or paper towel and clean thoroughly with acetone and allow to dry. A spectra of the clean sodium chloride cells can be run to ensure their cleanliness.

6. Transfer the mull from the mortar to the surface of a sodium chloride cell.
7. Place the other sodium chloride cell on top of the sample and smear the two together, making sure the whole surface area between the both cells is covered and no bubbles are present.
8. Place the cells into the cell holder.
9. Place the cell holder into the instrument and run a scan of the sample.
10. Clean the sodium chloride cells with acetone and store in a desiccator.
11. If time permits, repeat procedure using an unknown sample.

Name \_\_\_\_\_

Name \_\_\_\_\_

Period \_\_\_\_\_

Date \_\_\_\_\_

## SPECTRA OF SOLIDS

### Data Table:

List and Identify the major peaks.

Sample: \_\_\_\_\_

Unknown # \_\_\_\_\_

Major Peaks ( $\text{cm}^{-1}$ )

Functional groups

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Identity of Unknown: \_\_\_\_\_

Attach Spectrum

### Questions:

1. Why was it necessary to obtain a spectrum of mineral oil?
2. Why was the mineral oil used?

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## TEACHER NOTES

**Lab Time:** 45 minutes

### Answers to Questions:

1. Why was it necessary to obtain a spectrum of mineral oil?

*The mineral oil spectrum was taken to use as a background to compare the sample spectrums to.*

2. Why was the mineral oil used?

*The mineral oil was used because it dissolved the sample into a medium in which it could be analyzed, but would not dissolve the salt plates.*

### Considerations:

It takes approximately 10 minutes for each group to prepare and run their sample. If two sets of NaCl cells are available, one group can be preparing their sample while another is using the IR. The remainder of the lab time is used in analyzing the collected spectrum.

It is recommended to run a spectrum of mineral oil ahead of time or have the students run the spectrum, so that the peaks due to mineral oil can be disregarded in the analysis.

Substances can be identified either by comparing spectra to a library of pre-run spectra or by determining the functional groups present and matching to possible structures of the substance.

The IR Practice Activity could be used prior to or in combination with this lab. In addition, this lab could be used to verify the synthesis of aspirin.

### Reference:

R. Hannah and J. Swinehart, Experiments in Techniques of Infrared Spectroscopy. Perkin-Elmer Corporation, Infrared Applications Laboratory, September 1974.

