

How to Prepare Samples for IR Spectroscopy – NaCl plates

Reading: Mohrig, et al., *Laboratory Techniques in Organic Chemistry*, 4th edition, Chapter 21, Section 21.5.

Salt (NaCl) plates (for liquids and solids)

- Rule 1: **ONLY** clean NaCl plates with dry dichloromethane (aka, methylene chloride, CH₂Cl₂), acetone, or cyclohexane (C₆H₁₂). **NEVER** use water (H₂O) to clean a salt plate as it will ruin the plate, permanently!
- Rule 2: **NEVER** hold the shiny side of a plate between your fingers (to avoid fingerprints). **ONLY** hold them by the edges.
- Rule 3: Never scratch a plate with an item such as a spatula or other sharp object (expensive!)
- Rule 4: Do not drop the salt plates, as they are quite fragile and will break easily (expensive!).

1. For *liquid* compounds, apply 1 drops of liquid compound in the center of one plate then place a second plate on top, then press together using a soft tissue and rotate slightly to give a thin film of compound.

For *solid* compounds, prepare a Nujol (mineral oil) mull. Add ~10-15 mg of solid sample, then mineral oil (3-4 drops) to a mortar and pestle and grind the mixture to a thick paste (like that of honey). Dab a small amount (without scratching the NaCl plates!) of the mull on a salt plate and cover with a second plate. (Note that the IR absorptions from the mineral oil will always be present in the resulting spectrum). Alternatively, the “dry film” method can be used.

2. Place the salt plates in the holder that is in the sample chamber of the IR spectrophotometer and obtain the spectrum (see IR instructions for use.)
3. Immediately after use, clean the NaCl plates with dichloromethane (aka, methylene chloride, CH₂Cl₂) and/or cyclohexane (C₆H₁₂) and return them to the oven or the desiccator (ask instructor which one). Hold them only with forceps when washing with CH₂Cl₂ then wipe each plate dry with a clean soft tissue (e.g., a Kimwipe).

Thin Film Method using salt (NaCl) plates

1. Dissolve ~15 mg of sample in ~1-2 drops of dichloromethane (CH₂Cl₂, aka methylene chloride)
2. Place 1 drop of this solution of one salt plate (only), let the solution evaporate, then proceed as above starting with step 2. (Don't forget about Rules 1-4 above)