

TLC notes:

Always co-spot!

1. Media for Separation

- Silica: Slightly acidic medium. Best for ordinary compounds, good separation is achieved.
- Florisil: Mild, neutral medium. 200 mesh can be effective for easy separations. Less than 200 mesh best for purification by filtration. Some compounds stick on florisil, test first.
- Alumina: Basic or neutral medium. Can be effective for easy separations, and purification of amines.
- Reverse phase silica: The most polar compounds elute fastest, the most nonpolar slowest.

2. Solvent systems

The *only* appropriate one-component solvent systems (listed from the least polar to the most polar):

- hydrocarbons: pentane, petroleum ether, hexanes
- ether and dichloromethane (very similar polarity)
- ethyl acetate

The most common two-component solvent systems (listed from the least polar to the most polar):

1. Ether/Petroleum Ether, Ether/Hexane, Ether/Pentane: Choice of hydrocarbon component depends upon availability and requirements for boiling range. Pentane is expensive and low-boiling, petroleum ether can be low-boiling, hexane is readily available.
2. Ethyl Acetate/Hexane: The standard, good for ordinary compounds and best for difficult separations.
3. Methanol/Dichloromethane: For polar compounds.
4. 10% Ammonia in Methanol Solution/Dichloromethane: Sometimes moves stubborn amines off the baseline.

Rules of Thumb:

- A compound with an R_f of 0.5 in 10% ethyl acetate/hexane will have an R_f of 0.5 in 20% ether/hexane. This conversion factor is general.
- Methanol can be used as polar solvent, but only up to 10 percent of the mixture. More than 10 percent methanol can dissolve the silica gel.

Tips:

- Dichloromethane can dissolve compounds better, but it will take longer to run through the silica.
- Benzene is sometimes useful as the non-polar component, but is usually avoided because of toxicity.
- If your compound is sensitive to acid, put 1-3 percent triethylamine in your solvent system to neutralize acid in the silica gel. The R_f of your compound may increase a little bit, so check first.

3. Visualization Methods (Including TLC Stains/Dips)

- Ultraviolet: Look at the plate under the light first when working with compounds with conjugated double bond systems.

- Iodine: Shake with powdered I₂. You can then heat the plate to remove the iodine stain, and use a liquid TLC stain as usual.
- Anisaldehyde (great for carbonyl groups):
 1. EtOH (200 mL); H₂SO₄ (10 mL); p-anisaldehyde (10 mL)
 2. EtOH (250 mL); H₂SO₄ (2.5 mL); p-anisaldehyde (15 mL)
 3. EtOH (425 mL); H₂SO₄ (16 mL); p-anisaldehyde (8-12 mL); HOAc (5 mL): mix everything but p-anisaldehyde, which you add when the mixture has cooled to rt. Store refrigerated.
- Ceric Ammonium Molybdate (great for hydroxy groups):
 1. Ce(SO₄)₂ (cerium sulfate: 5.0 g); (NH₄)₆Mo₇O₂₄·4H₂O (ammonium molybdate: 25.0 g); conc. H₂SO₄ (50 mL); H₂O (450 mL)
 2. Ce(NH₄)₄(SO₄)₄·2H₂O (ceric ammonium sulfate: 4.0 g); ammonium molybdate (10 g); conc. H₂SO₄ (40 mL); H₂O (360 mL)
 3. Ceric ammonium sulfate (0.5 g); ammonium molybdate (12 g); conc. H₂SO₄ (15 mL); H₂O (235 mL)
- Ninhydrin (great for amines): Dissolve 0.3g ninhydrin in 100 ml of n- butanol; add 3 ml AcOH.
- Phosphomolybdic Acid (generally useful): 10% in Ethanol
- Potassium Permanganate (generally useful):
 1. KMnO₄ (1g); Na₂CO₃ (2g); H₂O (100 mL)
 2. KMnO₄ (3g); K₂CO₂ (20g); 5 percent NaOH (5 mL); H₂O (300 mL)

Source:

<http://chem.chem.rochester.edu/~nvd/tlcnotes.html>

<http://chem.chem.rochester.edu/~nvd/chromatographynotes.html>