Chromatography

Physical Separation Method

Separation of a mixture of cpds based on their interaction b/w a solid stationary phase & a liquid (or gas) mobile phase.

http://chemsite.lsrhs.net/Intro/images/Overheads_large/insect_chromatography.jpg
Elution: the movement of an *eluent* (*liquid, solvent*) *down a* or *up a* *[ ]* carrying *individual cpds* from a *mixture* at different rates, *depending on the* *[ ]* of each *cpd*. 
Thin Layer Chromatography (TLC)

TLC Plate Process:

- On the plate: Mark origin line w/
- Add analyte mixture:
  - create a saturated soln, add to plate w/
- What 2 items are needed in a developing jar?
- Put plate in jar: gently – avoid contact w/ filter paper. Why?
- When and what should be done as the plate is removed?
Thin Layer Chromatography (TLC)

Quantification:

- Measure $R_f = \frac{x_{\text{origintocpd}}}{x_{\text{origintofront}}}$

Uses:

- Determine eluent order for column
- Check chemical purity
- Follow a rxn over time
- Identify a compound

What are the $R_f$s for A, B, & C?

Does the size of plate matter?
Creating a Column:

- Insert a cotton plug, level w/ sand.
- Add the silica dry. (Careful, inhalation hazard.)
- Level w/ sand.
Running a Column:

- Load analyte. Begin adding eluent. Do NOT let eluent level go below top sand layer.

- Collect band solutions in Erlenmeyer flasks.

- Switch to next eluent once 1st cpd is completely off. Repeat until all cpds off column.
1. Mixture to be separated is dissolved in the mobile phase.

2. Mobile phase is added throughout the process.

3. Components separate.

4. Each component is collected as it reaches the bottom of the column.
### 3 Classes of “Chemicals”

<table>
<thead>
<tr>
<th>Silica SiO₂</th>
<th>Eluent</th>
<th>Analyte</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Phase?</strong></td>
<td><strong>Phase?</strong></td>
<td><strong>Definition?</strong></td>
</tr>
<tr>
<td><strong>Polarity?</strong></td>
<td><strong>Polarity?</strong></td>
<td><strong>Polarity?</strong></td>
</tr>
</tbody>
</table>

**Eluent use wrt polarity?**

*Pure Heptane, Acetone/Heptane mixtures, Pure Acetone*
Interaction b/w Analyte & ...

**Stationary Phase**

da\textit{sorption}:  

(absorption):

More polar \textit{cpds}, \textit{adsorb} on silica.

**Mobile Phase**

(dissolves) \textit{cpds away from} stationary phase

**First**: Least polar eluent moves

**Last**: Most polar eluents moves
**Mobile & Stationary Phases**

*compete (via intermolecular interactions)* for Analyte compounds.

If Mobile Phase wins,
### Example

**Polarity:**

<table>
<thead>
<tr>
<th>Stationary Phase</th>
<th>Mobile Phase</th>
<th>Analyte Mixture</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$... Si-OH</td>
<td>CH$_3$(CH$_2$)$_5$CH$_3$</td>
<td>R-CH$_3$</td>
</tr>
<tr>
<td></td>
<td>CH$_3$CH$_2$OCH$_2$CH$_3$</td>
<td>R-OCH$_3$</td>
</tr>
<tr>
<td></td>
<td>CH$_3$C(=O)CH$_3$</td>
<td>R-OH</td>
</tr>
</tbody>
</table>

*Rank Eluent Order and Analyte Elution Order...*
What happens if...

- the most polar eluent is used to develop a TLC plate?

- the most polar eluent is used first on the column?
More Questions

What happens if...

- the most nonpolar eluent is used to develop a TLC plate?
- the most nonpolar eluent is used first on the column?
Standard Solution: A solution of accurately known concentration.

Standard Addition: Adding known amounts of a standard solution to known amounts of a solution containing the same solute, but with an unknown concentration.

- Absorbances & concentrations with Beer’s Law to determine unknown concentration.
  - Known:
  - Measured:
  - Find:
Determined a Solution’s Concentration by adding a specific amount of a standard solution.

- According to Beer’s Law for a given cpd \( A = \varepsilon \ell C \),
  - \( A \) \( \uparrow \) linearly w/ \( C \).
  - \( \varepsilon \) and \( \ell \) are constants.

- **slope** of a BL Plot?
  - \( \Delta C = \) moles from a std soln added to an unknown

- Find the slope \( (\varepsilon \ell) \) by calculating \( \frac{\Delta A}{\Delta C} \)

- Find \( C_{\text{unk}} \) w/ BL:

- From \( V_{\text{unk}} \), find moles \( C_{\text{unk}} \), then w/ \( MW \), find mass \( C_{\text{unk}} \)

Find the mass % of each ferrocene w/ std addn!!!
I: 10 mL unknown, $A_I$, dilute to 25 mL

II: 10 mL unknown, 10 mL std, $A_{II}$, dilute to 25 mL

$A = \varepsilon l C$; \hspace{1cm} $m = \varepsilon l = \frac{\Delta y}{A_{II} - A_I}$

$\Delta x \hspace{1cm} C_{S-diluted}$

$C_I = \frac{A_I}{\varepsilon l}$

$C_I \times 0.02500L = \text{mol}_{unk}$

$\text{mol}_{unk} \times \text{FW} = g_{unk}$
A SERS Study on the Assembly Behavior of Gold Nanoparticles at the Oil/Water Interface

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Supporting Information

**ABSTRACT:** Herein, the assembly behavior of gold nanoparticles (AuNPs) at the oil/water interface is studied by surface-enhanced Raman scattering (SERS) spectroscopy. Two selected chemicals [1-dodecanethiol (DDT) and tetramethylammonium ion (TMA+)] are applied to tune the surface properties of AuNPs and the corresponding assembly behaviors at the oil/water interface are thoroughly investigated. Various AuNPs films, namely sparse 2D film, perfect monolayer, and multilayers are obtained. The SERS spectra analyses show that the surface composition of AuNPs is strongly dependent on the chemical environment around AuNPs and results in different morphologies of AuNPs film at the oil/water interface. Accordingly, we propose a rational relationship between AuNPs assembly behavior at the oil/water interface and their surrounding chemical environment, and thus reveals the physical mechanism underlying the nanoparticle assembly.
5-6 sentences that clearly communicate the basic nature of your report

Summary of
- objectives
- methods
- major results
- conclusion (interpretation)
- illustration (figure)

Must STAND ALONE. Someone can understand the basics of what you did without any other information.
Self-assembled monolayers were synthesized and characterized by contact angle measurement. Plasma cleaned silica wafers were heated at 35°C for 1 hour in ethanol solutions containing mercapto-functionalized chains. Solvent cleaned gold slides were treated with n-hexadecane solutions containing silane-functionalized chains for 30 minutes at room temperature. High resolution photographs of microliter water droplets were made on each SAM surface. Contact angle’s measured from these photographs correlated directly with the number of carbons in the functionalized chains and indirectly with the polarity of the group at the end of the chain. This synthesis and characterization of the surfaces was an effective way to create and show the hydrophobicity or hydrophilicity of a surface.

**Figure.** Formation of a SAM on a gold surface via sulfide bridges.