Introduction to Analytical Separations

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Introduction

1.) Sample Purity
- Many chemical analysis are not specific for one compound
  - **Actually respond to many potential interferences in the sample**

- Often it is necessary to first purify the compound of interest
  - **Remove interfering substances before a selective analysis is possible**
  - **This requires a <u>separation step.</u>**

2.) Techniques available for Chemical Separations:
- Extraction
- Distillation
- Precipitation
- Chromatography
- Many others (centrifugation, filtration, etc)

Extractions and Chromatography are especially useful in analytical methods

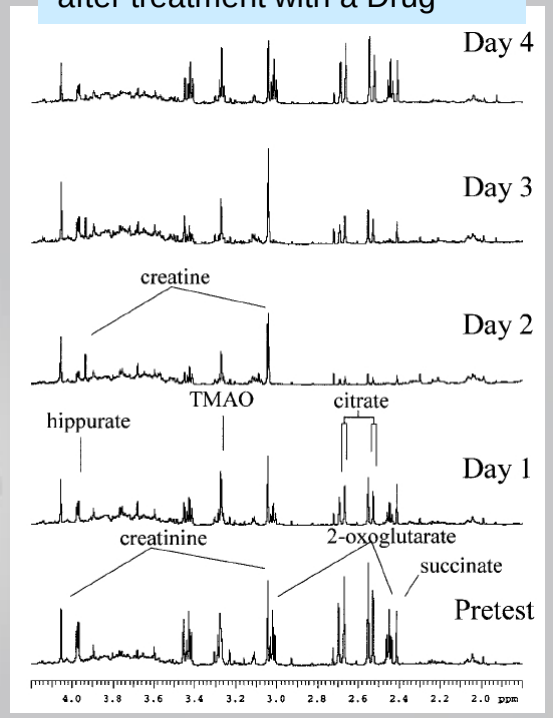
Introduction to Analytical Separations

Introduction

3.) Illustration

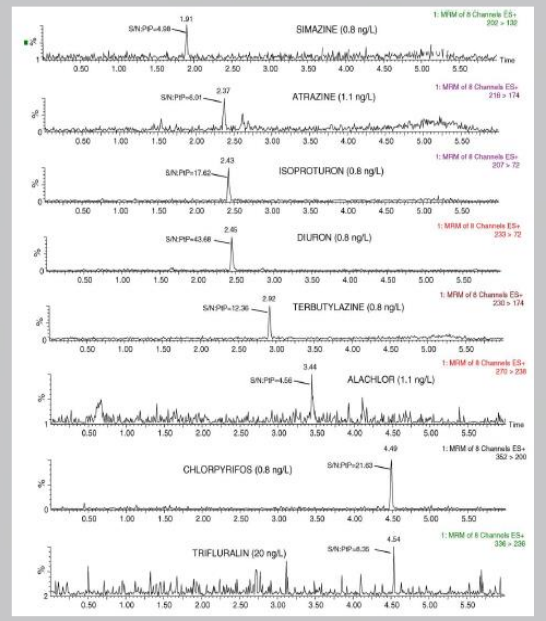
- Biological Samples are Composed of Complex Mixtures
  - **Analysis of composition and changes help in understanding disease and the development of treatments**

NMR Spectra of Mouse Urine after treatment with a Drug



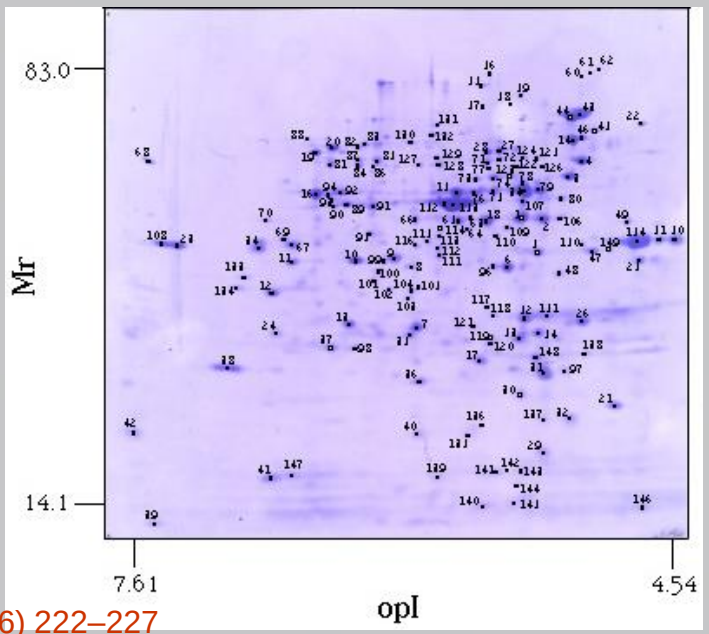
Toxicological Sciences (2000) 57:326-337

Analysis of Various Pesticides in Ground water using LC-MS



Journal of Chromatography A, 1109 (2006) 222–227

2D Gel Electrophoresis of total protein extract from *E. coli cells*



Electrophoresis (1997) 18:1259-1313

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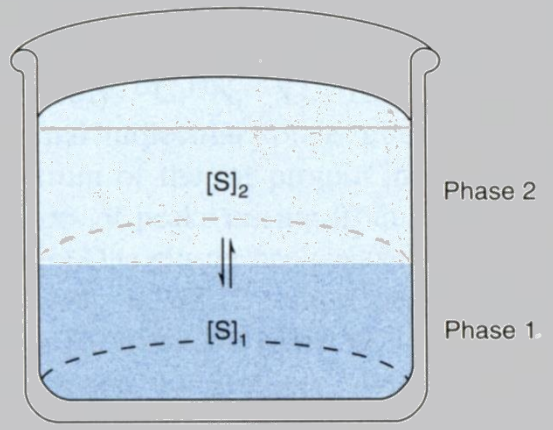
Extractions

1.) Definition

- The transfer of a compound from one chemical phase to another
  - **The two phases used can be liquid-liquid, liquid-solid, gas-solid, etc**
  - **Liquid-liquid is the most common type of extraction**

Immiscible liquids



$$S\ (\text{in phase 1}) \underset{}{\overset{K}{\rightleftharpoons}} S\ (\text{in phase 2})$$

$$K = \frac{[S]_2}{[S]_1}$$

- **The partitioning of solute s between two chemical phases (1 and 2) is described by the equilibrium constant *K***

K is called the <u>partition coefficient</u>

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Extractions

2.) Extraction Efficiency

- The fraction of moles of S *remaining* in phase 1 after <u>one</u> extraction can be determined
  - **The value of *K* and the volumes of phases 1 and 2 need to be known**

$$q = \frac{V_1}{(V_1 + KV_2)}$$

where: q = fraction of moles of S remaining in phase 1
V_1 = volume of phase 1
V_2 = volume of phase 2
K = partition coefficient

- The fraction of S *remaining* in phase 1 after <u>*n*</u> extractions is

$$q_n = \left[\frac{V_1}{(V_1 + KV_2)}\right]^n$$

Assumes V_2 is constant

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Extractions

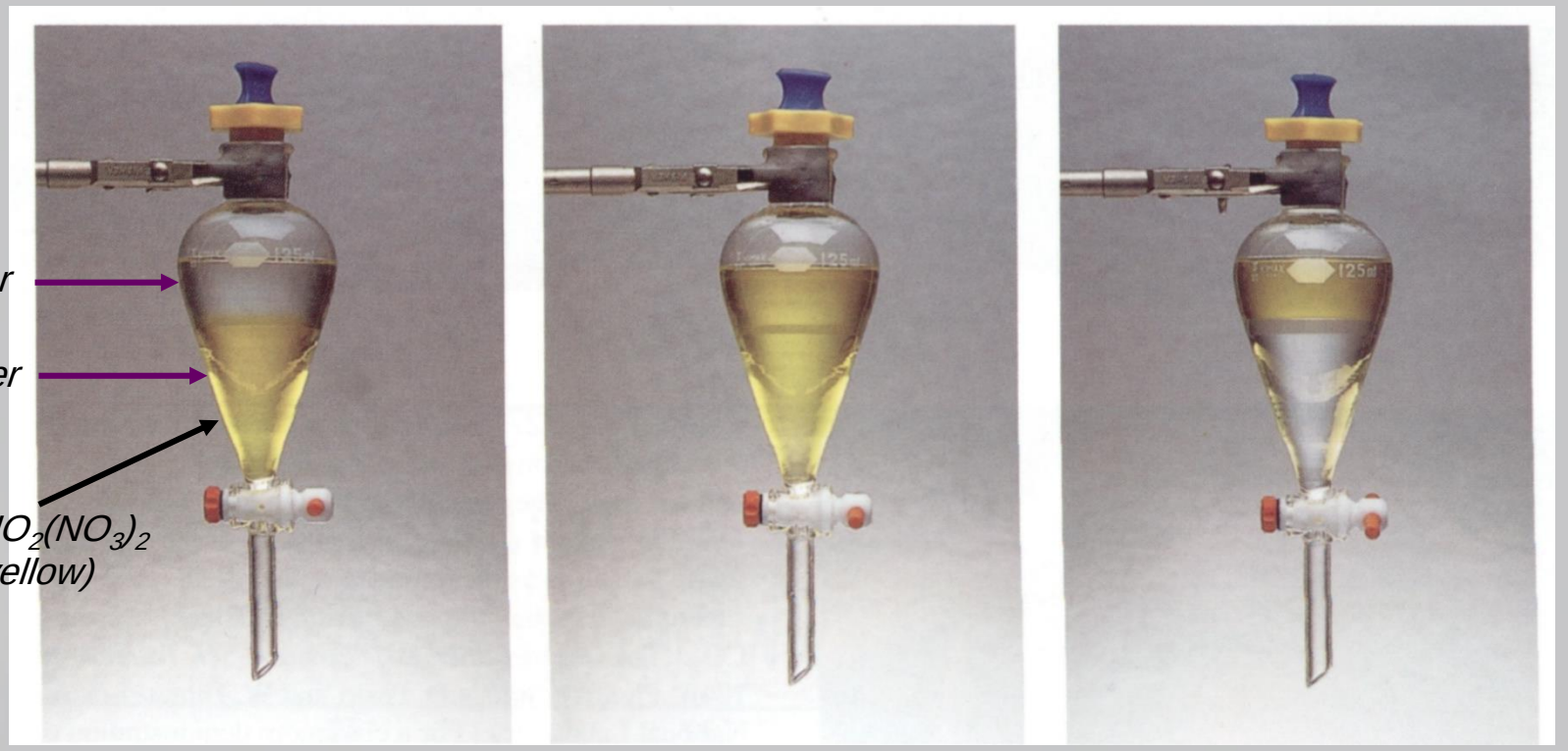
2.) Extraction Efficiency

- Illustration

Ether layer

Water layer

1M $UO_2(NO_3)_2$ (yellow)



After mixing, $UO_2(NO_3)_2$ Is distributed in both layers

After 8 extractions, $UO_2(NO_3)_2$ has been removed from water