



Technische Universität München

General principles of extraction

Organic Chemistry: Basic Operations

Jakub Niklas

Technische Universität München

December 6, 2023

Overview

1. General principles

2. Liquid-liquid extraction

- 2.1 The theory of liquid-liquid extractions
- 2.2 Practical application and equipment
- 2.3 Example extraction

3. Citations and references

General principles

Definition

Extraction is a collective term for processes that utilize a solvent to extract one or multiple analytes from a given mixture. The driving force behind the extraction is the better solubility of the analytes in the used solvent than in the mixed compound.

Examples

Brewing tea or coffee uses water (solvent) to extract flavor compounds (analyte) from tea leaves or coffee beans (mixture).

Types of extractions

Different types of extractions are classified based on the phase of the analyte and the solvent.

Examples

- Solid-liquid-extraction
 - Brewing tea or coffee
- Liquid-liquid-extraction
 - Main method used in the lab
 - Aqueous two-phase systems for the extraction of biomolecules
- Liquid-gas-extraction
 - Used in gas chromatography
- and any other possible combination...

Liquid-liquid extraction

The theory of liquid-liquid extractions

In liquid-liquid extractions (LLEs), two immiscible liquids are used. Often, an inorganic solvent (water) is used in conjunction with an organic solvent, such as diethyl ether or DCM.

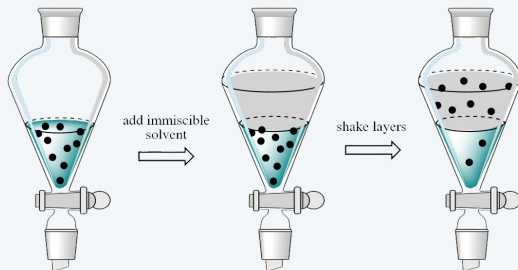


Figure 1: Overview of an extraction [3]

Nernst's distribution law

This equilibrium is described by Nernst's distribution law:

$$K(A) = \frac{c(\text{Analyte in Phase 1})}{c(\text{Analyte in Phase 2})} = \frac{c_i^\beta}{c_i^\alpha} \quad (1)$$

with $K(A)$ being the equilibrium coefficient.

- If $K(A) < 1$: The analyte's solubility in α is higher, it is the solvent, for $c_i^\alpha > c_i^\beta$.
- If $K(A) > 1$: β is our solvent, for $c_i^\beta > c_i^\alpha$.

Calculating the analyte's concentration

After transforming the equation and assuming β to be our solvent, the following term can be used to calculate the analyte's concentration after n extractions.

$$c_n^\alpha = \frac{c_0^\alpha}{(1 + K(A) \cdot \frac{V^\beta}{V^\alpha})^n} \quad (2)$$

c_0^α being the starting analyte concentration in the initial phase.

Practical application and equipment

The main piece of equipment used in an LLE is the separatory funnel (cf. Figure 2). In preparation for an extraction, the following steps are to be made:

1. The separatory funnel is secured using a ring clamp
2. A beaker or an Erlenmeyer flask is placed below the funnel
3. The stopcock is closed, and the stopper is removed

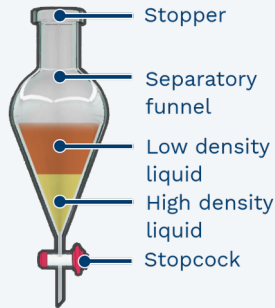


Figure 2: Separatory Funnel [2]

Starting the LLE

4. The liquid phases are added to the separatory funnel, and the stopper reinserted.
5. The funnel is taken out of the ring clamp and shaken. The developing pressure has to be relieved by opening the stopcock every once in a while.
6. This is to be repeated until there is no audible hiss while venting the funnel.
7. Subsequently, the mixing is complete, and the funnel can be replaced into the ring clamp. The two layers will separate.
8. Thereafter, the bottom phase is to be transferred to a clean beaker via the stopcock.
9. The top layer is to be removed out of the top of the funnel, into a separate vessel.

Additional steps

The initial extraction is finished, but there are additional steps that can result in a better yield and/or purity.

- Additional extractions can be done for both of the phases. The phase originally containing the analyte can be washed again, up to 3 times.
- The organic phase can be washed with a concentrated sodium chloride solution to dry it.
- To further dry the organic phase, sodium sulfate can be added and filtered off afterwards.

Example extraction I

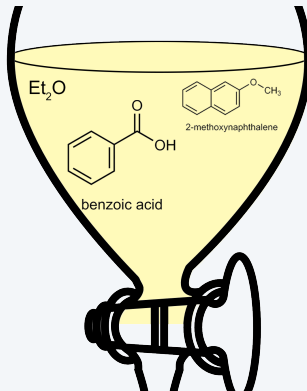


Figure 3: Example extraction: organic phase

Example extraction II

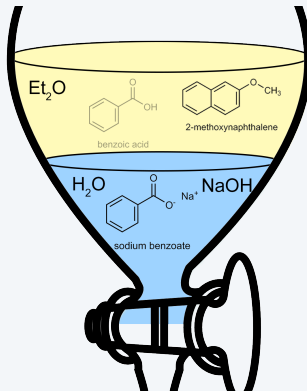


Figure 4: Example extraction: Extraction of the benzoate ion into the aqueous phase

Citations and references

References I

All illustrations without direct source reference were created by myself.

- [1] Mujahid Iqbal, Yanfei Tao, and Shuyu Xie. “Aqueous two-phase system (ATPS): an overview and advances in its applications”. In: *Biological Procedures Online* (2016). DOI: 10.1186/s12575-016-0048-8.
- [2] Labster. *The separatory funnel*. [Online; Stand 2. Dezember 2023]. URL: https://theory.labster.com/separating_funnel/.
- [3] Lisa Nichols. *Overview of Extraction*. [Online; Stand 2. Dezember 2023]. URL: https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Organic_Chemistry_Lab_Techniques_%28Nichols%29/04%3A_Extraction/4.02%3A_Overview_of_Extraction.

References II

- [4] Wikipedia. *Flüssig-Flüssig-Extraktion* – Wikipedia, die freie Enzyklopädie. [Online; Stand 2. Dezember 2023]. 2022. URL: <https://de.wikipedia.org/w/index.php?title=Fl%C3%BCssig-Fl%C3%BCssig-Extraktion&oldid=223898658>.
- [5] Wikipedia. *Nernstsches Verteilungsgesetz* – Wikipedia, die freie Enzyklopädie. [Online; Stand 2. Dezember 2023]. 2023. URL: https://de.wikipedia.org/w/index.php?title=Nernstsches_Verteilungsgesetz&oldid=234121934.
- [6] Wikipedia contributors. *Extraction (chemistry)* – Wikipedia, The Free Encyclopedia. [Online; accessed 2-December-2023]. 2023. URL: [https://en.wikipedia.org/w/index.php?title=Extraction_\(chemistry\)&oldid=1187460317](https://en.wikipedia.org/w/index.php?title=Extraction_(chemistry)&oldid=1187460317).



Technische Universität München

Thank You for Your attention!

Jakub Niklas

Technische Universität München

December 6, 2023