

Techniques for
preparation of solid
samples with a desired
concentration of analyte

Aim

- **Learn to prepare solid samples with desired concentration of a solute and its uncertainty**

Importance

- Preparation of calibration samples
- Preparation of certified reference materials (CRM)
- Production of commercial solid products
- Conducting research experiments

Concentration

- general measurement unit stating the amount of solute present in a known amount of solution

$$\text{Concentration} = \frac{\text{amount of solute}}{\text{amount of solution}}$$

- Amount – mass, volume or amount of substance

Units of concentrations of solids

Liquid samples:

- volume %;
- mol/L;
- g/L;
- ppm (w/v); ppb (w/v); ppt (w/v)

Solid samples:

- weight %;
- g/kg;
- ppm (mg/kg or $\mu\text{g/g}$); ppb ($\mu\text{g/kg}$); ppt (ng/kg)

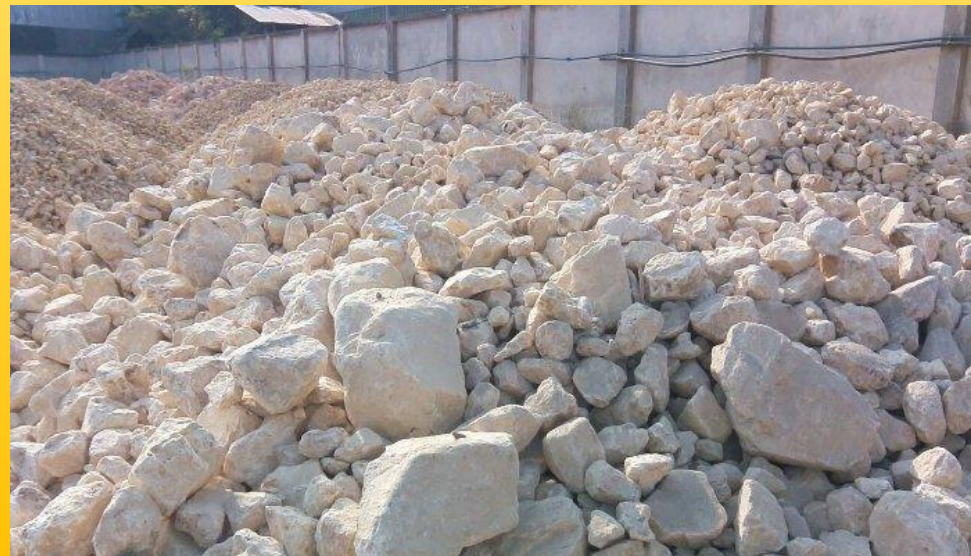
Gaseous samples:

- volume %;
- ppm (v/v) – milliliters of gaseous compound in 1 m³ of gas mixture;
- ppm (w/v) – milligrams of gaseous compound in 1 m³ of gas mixture
- mg/m³, $\mu\text{g/m}^3$, ng/m³

Solid samples

- Soil and ground
- Ore, minerals
- Metals
- Polymers
- Biological samples (wood, tissues, bones, hair, etc.)

Geological samples

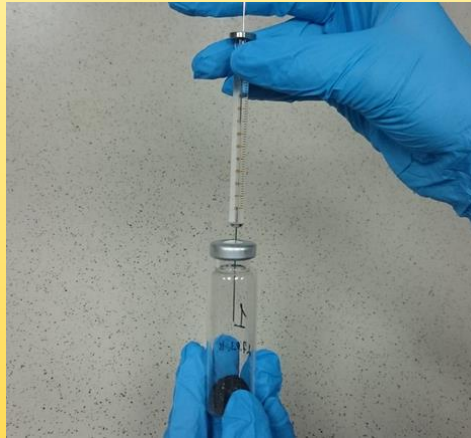


Approaches for preparing solid samples

- Spiking liquid standard into a ground sample + mixing
- Mixing ground solid standard and sample
- Melting or alloying

Solid samples for calibration of the method based on solid-phase microextraction (SPME)

1 – spiking with IS



2 - heating



3 - equilibration



4 - extraction



Question

- How much naphthalene must be introduced into the 20-mL vial with 1.00 g of pure soil to prepare soil with naphthalene concentration 5 ng/g?

$$m_{C_{10}H_8} = m_{soil} \times C_{C_{10}H_8} = 1 \text{ g} \times 5 \frac{\text{ng}}{\text{g}} = 5 \text{ ng}$$

- Q: how could you introduce 5 ng of naphthalene to the vial with soil?

Possible methods

- Add 1 μL of C_{10}H_8 solution ($C = 5 \text{ ng}/\mu\text{L}$) in CH_3OH
- Add 2 μL of C_{10}H_8 solution ($C = 2.5 \text{ ng}/\mu\text{L}$) in CH_3OH
- Add 5 μL of C_{10}H_8 solution ($C = 1 \text{ ng}/\mu\text{L}$) in CH_3OH
- Add 10 μL of C_{10}H_8 solution ($C = 0.5 \text{ ng}/\mu\text{L}$) in CH_3OH
- Add 10 mg of soil containing C_{10}H_8 at $C = 0.5 \text{ ng}/\text{mg}$

Task

- Calculate uncertainties of all prepared soil samples if uncertainty of a concentration of a spiked solution is 1%. Spiking is done using a 10- μL microsyringe (scale 0.1 μL).

Task

- Propose the method to prepare soil sample with a concentration of toluene 200 $\mu\text{g/kg}$.

Task

- Propose the method to prepare a sample of soil ($m = 100 \text{ g}$) with a concentration of crude oil from Tengiz oil extraction site 500 mg/kg .