

## Practical work 2

### Determination of copper (Cu) in surface water using an atomic-absorption spectrometer.

#### Sample Preparation

**Determination of dissolved metals.** For the determination the dissolved metals, sample must be filtered on the vacuum filtration apparatus through a filter with diameter of 0.45  $\mu\text{m}$  immediately after sampling. The first portion of the filtrate (about 50 ml) should be discarded. Then, the filtrate was acidified to  $\text{HNO}_3$  (1:1)  $\text{pH} \leq 2$  and stored in a plastic container.

**Determination of the total metal content.** Samples should be acidified with  $\text{HNO}_3$ (1:1)  $\text{pH} \leq 2$ . Then sample must be filtered and ready for analysis.

If the initial concentration of the metal is lower than the method detection limit, should apply:

Methods of samples concentration.

- Evaporation of sample (pic.5)
- Extraction concentration of metals
- Ion exchange concentration

(Choice of method depends on the type of sample)

#### Equipment and Support Materials

1. Vacuum filtration apparatus, „Millipore“ (pic. 2)
2. Membrane filter with a pore size 0.45  $\mu\text{m}$  (pic. 3)
3. Atomic absorption spectrometer AAnalyst 800, “PerkinElmer”(pic.1)
4. Automatic pipette with nozzle of different sizes (pic.4)
5. Measured flasks (50ml and 100 ml)
6. For collection of sample, storage and transportation, will used vessel made of polymeric material (Polyethylene, polypropylene, or Teflon).

#### Reagents

1. Standard solutions P/N S4400-1000141 Single-Element Copper Standart Cu in 2% $\text{HNO}_3$
2. Working solution of metal (mg/l or  $\mu\text{g/l}$ )-are manufactured with a dissolving of standard solution;
3. Calibration solutions of metal (mg/l or  $\mu\text{g/l}$ )-are manufactured with a dissolving of working solution. Mass concentration in the samples should be in the operating range of measurement;
4. An acid nitric of special cleanliness;
5. Gaseous Argon of high cleanliness;
6. Gaseous Acetylene of high cleanliness;
7. Deionized water.

#### Process of measurements:



Atom-absorption spectrometer AAnalyst 800 is prepared for work according to the management (instruction) by operation. Operating modes of the device are established according to recommendations of the firm - manufacturer of the device.

After inclusion of device it is possible to choose a desired method of atomization by means of the computer, with the use of software support WinLab32 (installation and specification of atomizer will automatically take place). In a case of choosing graphite atomizer it is necessary to check the adjustment (via WinLab32) and if necessary to adjust (manually) tip (end) of an autozator. It is important to put the necessary lamps.

Measurement results will be obtained in mg/l or  $\mu\text{g/l}$  in the dialog window Results.



Pic.1 Atomic absorption spectrometer AAnalyst 800



Pic.2 Vacuum filtration apparatus Millipore



Pic.3. Membrane filter



Pic.4. Automatic pipette



Pic.5. Evaporation of sample

Standard Operation Procedures (SOP) was employed for researchers which had been developed by us on the basis of international methods of ISO.

Were used:

5667–1:1980 Water quality- Sampling-Part 1: Guidance on the design of sampling programmes

Including:

5667–5 Water quality- Sampling-Part 5: Guidance on sampling of drinking water.

5667-6 Water quality- Sampling-Part 6: Guidance on sampling of rivers and streams.

5667–4 Water quality- Sampling-Part 4: Guidance on sampling from lakes, natural and manmade.

5667–11 Water quality- Sampling-Part 11: Guidance on sampling of groundwaters.

5667–9 Water quality- Sampling-Part 9: Guidance on sampling from marine waters.

5667–10 Water quality- Sampling-Part 10: Guidance on sampling of waste waters.

5667–2 Water quality- Sampling-Part 2: Guidance on sampling techniques.