X-RAY FLUORESCENCE: EXCITATION BY A RADIOACTIVE SOURCE

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The excitation by radiations from radioactive sources has been widely used in X-ray fluorescence spectroscope. Alpha, beta, gamma and X-ray emitting isotopes have been used to excite characteristic X-rays. The criteria of a good source are 1): a simple line spectrum at an appropriate energy, no high-energy β or γ radiation, long enough half-life, and high enough specific activity.

The radioisotope X-ray fluorescence system at the Laboratory for Trace Element Analysis of the University of Rijeka consists of:

1. A sealed radioactive ^{109}Cd source of 10 mCi emitting 99.0 keV and 22.2 keV X-rays. The source-target system can be placed in a vacuum chamber.
2. A semiconductor Si(Li) detector with an active area of 30 mm^2, 3 mm thick, cooled by liquid nitrogen. The energy resolution is 180 eV for 5.8 keV K X-rays from Fe. The Thickness of the beryllium window, placed 5 mm in front of the detector, is 5.8 µm.
3. The electronic system consists of a preamplifier, an amplifier and a 1024-channel analyzer (Canberra 8100).

The detector efficiency as a function of photon energies was determined using standard calibrated radioactive sources. The relative elemental efficiency of the system for ^{109}Cd excitation was determined using samples of known composition. Because of the low detector efficiency for X-ray energies above 30 keV, heavy elements in the sample can be detected only by their characteristic X-ray lines. The absolute efficiency was determined directly only for some elements. These measurements allowed the normalization of the relative elemental efficiency curve

Various targets prepared from environmental biological, geological and other samples were analyzed. The preparation of targets is a very critical stage in the analysis and a possible source of contamination and errors. Aqueous targets were prepared by evaporation of water, and by subsequent deposition of powder on milipore filter. For the analysis of dissolved trace metals in water, a method was used which involves the formation of insoluble metal chelates via coordination with dithiocarbamate (ammonium pyrolidine dithiocarbamate or diethyl dithiocarbamate), filtration through a membrane filter, and the analysis of the precipitate. Simultaneous analysis of most of the transition elements is possible, but alkali and alkaline-earth metals are excluded. The following elements can be separated by dithiocarbamate: V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Mo, Rh, Pd, Ag, Cd, In, Sn, Sb, Pt, Au, Hg, Tl, Pb and U2). The dithiocarbamate precipitation technique for target preparation offers several advantages over procedures based upon evaporation, among them the uniformity of distribution of elements on the target, the simplicity which reduces both handling time and contamination hazard, detection limits of 10^{-3} ppm and lower, and high efficiency within a wide pH range.

Targets from geological samples and other solid materials can be prepared as powders and deposited on filter membrane. For qualitative analysis, no target preparation is needed.

REFERENCES

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