

TRACE-ELEMENT ANALYSIS USING X-RAY EMISSION SPECTROSCOPY

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Advances in the manufacturing of semiconductor detectors for X-rays, especially the remarkable improvement in resolution, have made X-ray emission spectroscopy a valuable tool for trace-element analysis¹⁾. The principle of X-ray spectroscopy is the detection of X-rays produced in the sample by an incoming beam of photons or charged particles. The X-ray spectrum thus obtained shows characteristic X-ray lines from all elements present in the sample with concentrations above the detectability limit of the system. The energies of X-ray lines identify the elements, while their intensities are proportional to the concentrations. Three sources of excitation are generally used: Charged-particle accelerators, X-ray tubes and radioactive sources.

X-ray emission spectroscopy may be used in a variety of analytical applications involving materials ranging from ores to semi-finished products including chemicals and metals. This method helps to isolate and understand the role of many elements present in very small quantities in the environment. Hence, it can be successfully used for the study of water and air pollution. Its application in biology and medicine, in human and animal nutrition, offers an extensive field of research owing to the capability of the method to detect several elements simultaneously. The virtues of X-ray emission spectroscopy may be quoted as follows:

- simultaneous determination of all medium-heavy and heavy metals using their characteristic K and L-ray lines; the detectability is fairly uniform across the periodic table;

- simple sample preparation in most of the cases and small quantities needed for the sample. The X-ray technique is nondestructive, i.e., the majority of samples does not change any of their properties during the analysis and therefore can be retained for further use;

- high sensitivity and accuracy; concentrations of 1 in 10^6 (1 ppm) or lower can be detected within a relatively short collection time.

A system for trace-element analysis by proton-induced X-ray emission spectroscopy was developed at T.W. Bonner Nuclear Laboratories at Rice University²⁾. The computer facilities of this nuclear laboratory allowed fast collection, processing, and analysis of data. A special computer program was written for the automatic analysis of X-ray spectra and extraction of absolute elemental concentrations. Fig. 1 shows a typical spectrum obtained by proton bombardment of a human hair sample. Elements present in the hair are indicated by arrows. The system is widely used for the trace-element analysis of different samples of biological origin (hair³⁾, blood, animal organs, etc.), as well as for the study of environmental pollution (Air, water, etc.).

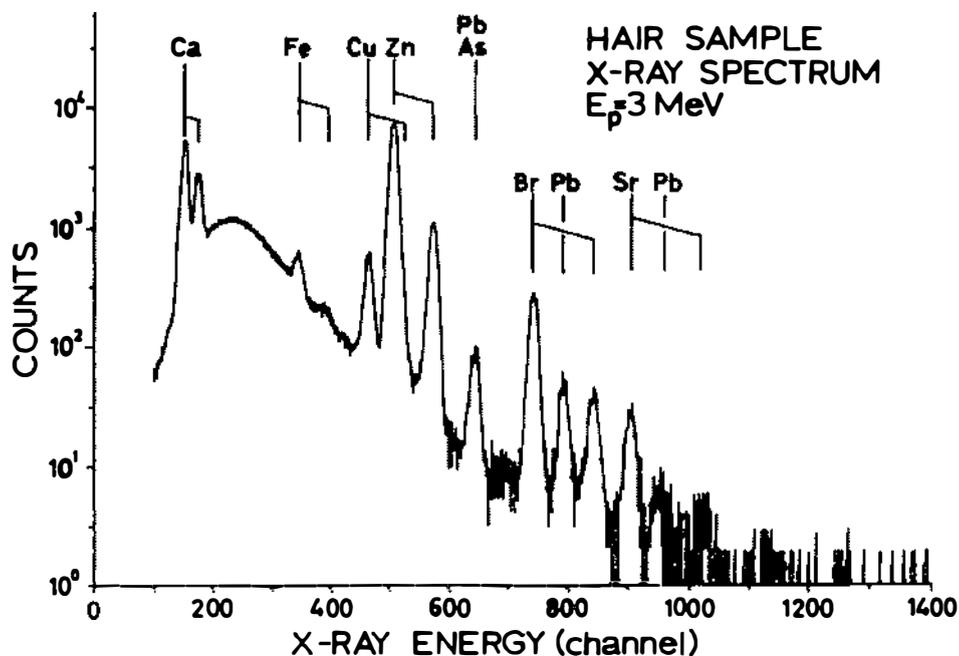


Fig. 1. X-ray spectrum obtained by the proton bombardment of a hair sample

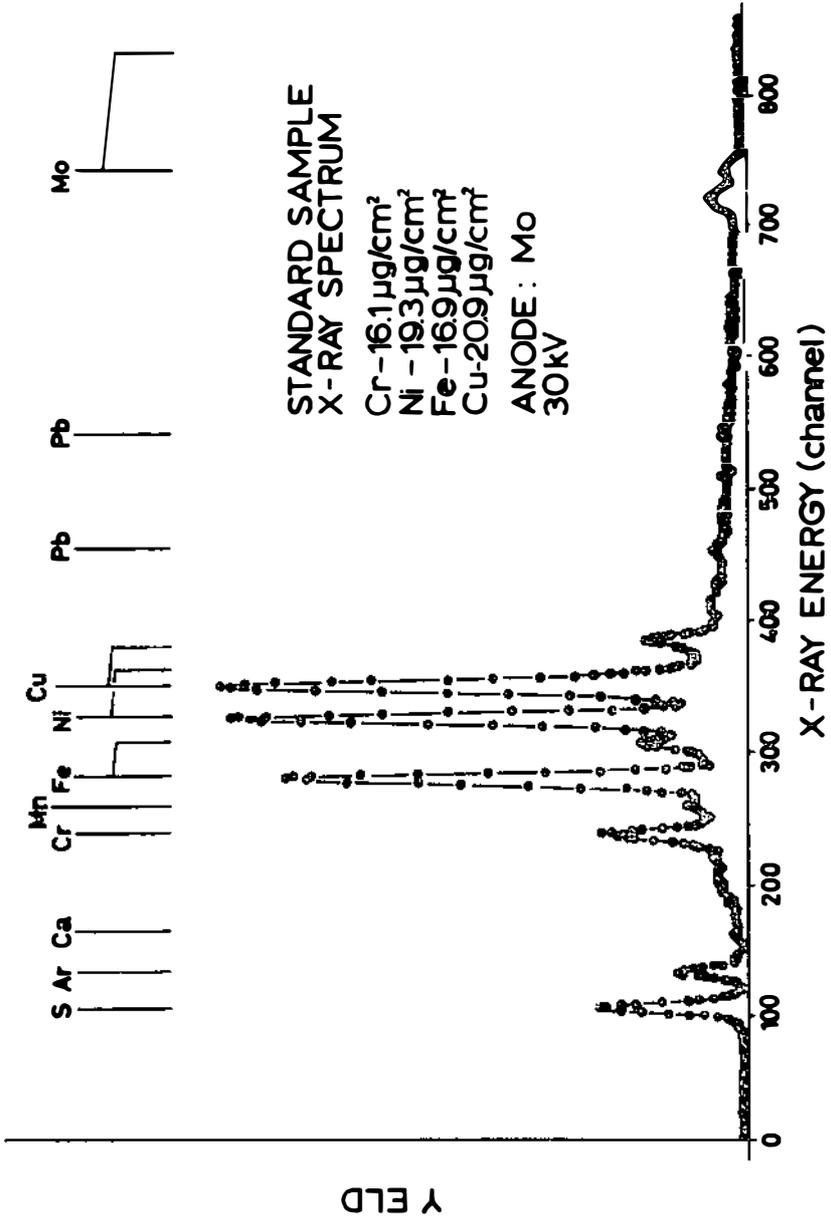


Fig. 2. X-ray spectrum of a standard sample obtained by the X-ray tube as a source

The experience acquired with this system is being applied to build a similar system with an X-ray tube, in the Department for Nuclear and Applied Physics at the Rudjer Bošković Institute. Similar programs for the analysis of X-ray spectra have been developed in the laboratory computer NOVA 1220 (8 k) attached to the Northern Scientific NS-660 data acquisition system. Preliminary spectra have been obtained and one of them is depicted in Fig. 2. The figure shows the spectrum of a standard sample made of the known concentrations of elements Cr, Ni, Fe and Cu. A few other elements, present as impurities in the standard (S, Ca, Mn and Pb), are also visible. The Ar line is present from the air (the sample was not in the vacuum), while the Mo lines come from the Mo anode used in the X-ray tube.

Preliminary results indicate all the advantages and capabilities of this method for the analysis of elements present in low concentrations. The use of an X-ray source of sufficient power (around 1000 W) would establish X-ray emission spectroscopy as a very powerful new technique in our Institute to be used in fundamental research in atomic physics and in applications to problems in medicine, environmental pollution, etc., as well as in routine analysis of various samples for different purposes.

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