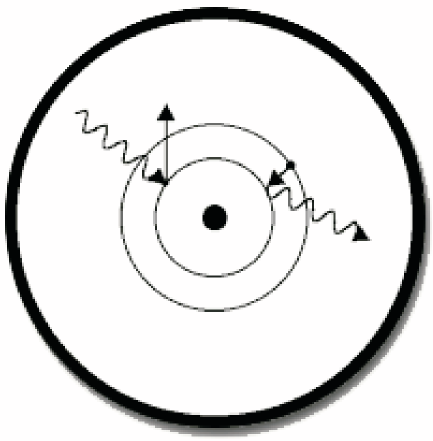
X-ray Fluorescence in the IAEA and its Member States

XRF



NEWSLETTER

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XRF NEWSLETTER

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Introduction

The major objective for publishing this periodical XRF Newsletter is to inform the XRF laboratories in IAEA Member States on recent developments in the field of XRF spectrometry and to exchange views on fundamental and applied aspects of sampling, sample preparation, instrumentation, quantitation, quality control, etc. The XRF Newsletter intends also to present the XRF activities and the main XRF results obtained at the IAEA's model XRF Laboratory in Seibersdorf and in XRF laboratories in the Member States. This Newsletter will include input from the Member States and will help the XRF laboratories to improve their analytical performance, extend the applicability range of XRF techniques and initiate co-operation. It will be distributed to current and potential end users of the analytical services of XRF laboratories and will further promote the utilisation of this technique in environmental pollution monitoring, mineral exploration, archaeometry and industry.

The first issue of the Newsletter provides the XRF laboratories with a description of the facilities, the activities and selected results obtained at the IAEA XRF Laboratory in Seibersdorf, Austria.

The IAEA XRF Laboratory

The XRF Laboratory was established some years ago as a model XRF laboratory and is now equipped with energy-dispersive XRF spectrometers based on X-ray tube and radioisotope excitation, including a total-reflection XRF (TXRF) unit with various modes of total-reflection such as direct excitation, cut-off reflector, multilayer (Fig. 1), and a capillary-based X-ray microfluorescence system (Fig. 2); in total five EDXRF spectrometers are currently in operation. In addition, basic facilities for sample preparation prior to conventional (bulk) XRF, TXRF and micro-XRF are available. Secondary target XRF and TXRF systems are integrated into a dual measurement EDXRF spectrometer using one single X-ray tube only (for more details please visit the IAEA Laboratories homepage http://www.iaea.org/programmes/naal).

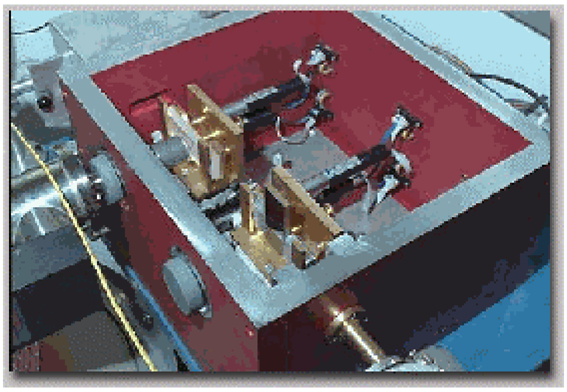

Fig. 1. Total reflection XRF system

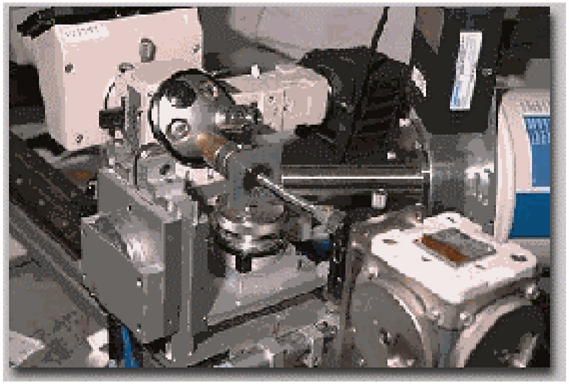

Fig. 2. Capillary based X-ray microfluorescence set-up

The methodological work carried out at the IAEA Laboratories include, i.a., the following: (i) modification of the emission-transmission method to take into account the absorption edges for minor and/or major elements present in the sample, (ii) assessment of the influence of the uncertainties of the incident and emerging angles and of the mass absorption coefficients on the accuracy of the calibration factors and selection of the optimised calibration procedure, (iii) development of a quantitative procedure for the XRF analysis of inhomogeneous thin samples, (iv) assessment of the accuracy of the emission-transmission method with identification of the individual contribution from various sources of uncertainty (which can be used also in selection of an optimum analytical strategy), (v) development of optimised sample preparation procedures for the analysis of solid materials (including biological, geological and environmental) by total reflection XRF, taking into account speed and completeness of sample digestion and the overall precision and accuracy, (vi) optimisation of sample preparation methods for X-ray microfluorescence analysis, and (vii) development of a procedure for homogeneity testing of candidate reference materials including also materials intended for microanalytical techniques. The XRF techniques available at Seibersdorf have been applied, i.a., for the analysis of bulk and individual airborne particles, rock phosphates, coals, sediments, soils, water samples, fiber optic conductors and hot particles as well as for the homogeneity test of a number of candidate reference materials.

Recently the interest of the Laboratory has been devoted to the development of portable XRF units based on radioisotope excitation and liquid nitrogen - or thermoelectrically-cooled semiconductor detectors (Si-PIN and CdZnTe) as well as in the development of quantitative procedures for in situ applications of XRF techniques. In order to assess the quality of the analytical performance of the XRF laboratories in the Member States and to assist them in further improvement a world wide intercomparison survey for quality control was performed. It included a performance test of the XRF setup(s), a test for the correct evaluation of XRF spectra and the analysis of several intercomparison samples (thin Al foil, water sample and geological and biological materials). The evaluation of the results of the intercomparison survey and the classification of the participating laboratories were done according to recognised international procedures based on z- and u-scores. The XRF laboratory in Seibersdorf is involved in the following activities: (i) analytical services for the Agency projects and research groups and for the Member States, (ii) support to Technical-Co-operation projects implementing nuclear analytical techniques in developing countries, (iii) comprehensive training in methodology and applications of XRF techniques, and (iv) research and development to extend the applicability range and to improve the accuracy of XRF techniques. More details on those activities are made available upon request.

Future activities of the IAEA XRF Laboratory

In 2000 a new Co-ordinated Research Programme(CRP) on "In situ applications of XRF techniques" was initiated. The CRP (foreseen for four years) in an activity of the Agency's project on Nuclear Instruments for Specific Applications whose major objective is to assist Member States in the development of nuclear instruments and special applications software. It is expected that 10-12 XRF laboratories from both industrialised and developing Member States will participate in the CRP in order to (i) develop and optimise sampling methodologies for in situ XRF measurements, (ii) improve the analytical performance of field portable XRF technique through the study of the interfering effects, (iii) develop quantitative and/or semi-quantitative procedures to be applied for in situ XRF analysis, and (iv) develop complete standard operating procedures for selected in situ applications. Other future activities include, i.a., (i) test of new IAEA reference materials to be used for quality control in nuclear spectrometry, (ii) establishing a metrological basis for XRF technique (including traceability and uncertainty components), (iii) optimisation and evaluation of sample preparation procedures for various X-ray emission techniques, (iv) evaluation of the suitability of new radiation detectors for X-ray emission techniques, (v) applications of synchrotron radiation for XRF and EXAFS, (vi) applications of XRF techniques to studies of cultural heritage (archaeometry), (vi) establishing and running a world-wide information network for XRF laboratories, and (vii) world-wide proficiency tests for XRF laboratories.

Comments and suggestions related to the planned activities of the IAEA XRF Laboratory are highly welcome as well as recommendations for future activities of interest to XRF laboratories in the Member States.

New developments in XRF - Detectors

A section presenting new development in some areas of XRF ("Detectors" in this first issue of the XRF Newsletter) is not intended as a comprehensive review of the current status and achievements. The purpose of the section is to summarise in a concise way the progress in the relevant field and to demonstrate some examples of new developments and applications. For more details on "Detectors" please refer to two recent review papers: (i) Sz.B. Toeroek et al., X-ray Spectrometry, *Anal. Chem.,* 1998, 70, 495R-517R,

(ii) P.J.Potts et al., Atomic Spectrometry Update - X-ray fluorescence spectrometry, *J.Anal.At.Spectrom.*, 1999, 14, 1773-1799. From recent papers related to energy-dispersive detection methods it is clearly seen that the thermoelectrically cooled semiconductor detectors and the cryogenic superconducting tunnel junctions (STJ) are the major areas of development. The thermoelectrically cooled detectors (based on Si, CdTe or CdZnTe) are still slightly inferior in resolution to the liquid nitrogen cooled Si(Li) or HpGe detectors. Because of their small size and low cost, however, they became very attractive for use in portable XRF systems for in situ applications. Potts et al. reported on a CdZnTe detector cooled to -40 ^{o}C with an energy resolution of 188 eV at 5.9 keV and 482 eV at 59.5 keV. High resolution cryogenic ED detectors based on STJ have an energy resolution of at least of one order of magnitude better than ionisation-based semiconductor detectors (e.g., below 10 eV at 277 eV at 10 kcps). Combined with a high operating count rate and a high detection efficiency makes them very attractive. Another type of high resolution detectors which are expected to gain importance in the future are the microcalorimeters based on superconducting technology and Si drift chambers. The microcalorimeters operate at an extremely low temperature (around 70 mK) and have an energy resolution of 3-5 eV in the energy range 0.1-2 keV; they are used both with analogue and digital electronics. According to an "optimistic forecast" the microcalorimeters might even replace the traditional Si(Li) detectors in the future.

Silicon charge coupled device (CCD) detectors which are ideal for X-ray imaging or very low X-ray energy region are currently receiving a lot of interest. One of the reason is that they are often applied in synchrotron radiation XRF. The devices based on CCD detectors comprise hundreds of pixels, each with an integrated, on-chip measurement channel. They are resistant to radiation damages (very good X-ray hardness). The typical dimension of a pixel is 0.15 x 0.15 mm^2 with an energy resolution of about 175 eV.

New books of potential interest to the XRF community

The following books might be of interest for the users of XRF technique:

1. R.Jenkins, X-ray Fluorescence Spectrometry, 2nd edition, Wiley-Interscience, New York, 1999
2. V.E.Buhrke, R.Jenkins, D.K.Smith, Eds., A Practical Guide for the Preparation of Specimens for X-ray Fluorescene and X-ray Diffraction Analysis, Wiley-VCH, New York, 1998
3. S.Landsberger and M.Creatchman, Eds., Elemental Analysis of Airborne Particles, Gordon and Breach Science Publishers, 1999
4. Pierre Gy, Sampling for Analytical Purposes, Wiley, 1998
5. J.W. Einax, H.W.Zwanziger and S.Geiss, Chemometrics in Environmental Analysis, John Wiley & Sons, New York, 1997
6. H.Saisho and Y.Gohshi, Eds., Applications of Synchrotron Radiation to Materials Analysis, Elsevier, 1996

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