

Industrial and Environmental Applications of X-ray Fluorescence Spectrometry

The following is a summary of one of the papers presented at a Joint Meeting of the Atomic Spectroscopy Group and Midlands Region held on November 22nd, 1989, at Inco Alloys Ltd., Hereford.

Exploiting Energy Dispersive X-ray Fluorescence Spectrometry for the Determination of Trace Elements in Geological Samples

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Energy dispersive X-ray fluorescence (ED-XRF) spectrometry is widely accepted as a technique capable of rapid qualitative and semi-quantitative analysis. What is not as widely appreciated is its quantitative capabilities. Many major and trace elements, and in particular the heavier trace elements, can be determined in geological samples to a degree of accuracy, precision and limit of detection equivalent to that expected of routine wavelength dispersive (WD) XRF analysis.¹⁻² Several apparent deficiencies, including poor spectrometer resolution and a limited maximum count rate capacity, are often erroneously perceived to limit the performance of ED-XRF in quantitative applications. In this paper, we review the progress made in developing ED-XRF for the determination of trace elements in geochemical samples and discuss specific features of the technique, including spectrometer resolution, spectral analysis software and calibration methods, that relate to the quantitative capabilities of the ED-XRF method.

Instrumentation and Spectrum Analysis

Data discussed here were recorded on a first-generation (ca. 1977) Link Analytical MECA 10-44 ED-XRF spectrometer fitted with a 50 W air-cooled silver anode tube and an Si(Li) detector having a nominal resolution of 165 eV at 5.9 keV (40 μ s time constant equivalent). Particular features that have contributed to the quantitative capabilities of this instrumentation are as follows.

(a) *Direct excitation*, normally using a silver anode tube and primary beam filter, if appropriate. Multi-element major and trace analysis can be achieved using an optimised combination of tube kV and filter (where appropriate) from two recorded spectra. The more specific capabilities of ED-XRF using secondary target excitation will not be considered here.

(b) *Pulsed tube operation*. By electronic gating of the intensity output of the X-ray tube in sympathy with the pulse processor busy signal, the proportion of dead time induced by pulse pile-up is substantially reduced.³ Maximum output count rates of 10 000 counts s^{-1} can then be achieved compared with 2000 counts s^{-1} for conventional ED systems, so maximising the precision of measurements within a pre-set count time.

(c) *Spectrum analysis* using digital filtering and a least-squares fit of "library" element profiles. The so-called FLS deconvolution procedure⁴ is highly effective in analysing accurately complex ED-XRF spectra to a degree of sophistication unavailable in WD spectrometry. This performance is substantiated by the results of spectrum deconvolution experiments on the notorious Ti K-line and Ba L-line interference.⁵

Composite spectra were recorded by superimposing spectra of the mineral, baryte, and titanium metal, excited by an electron microprobe (Fig. 1). By varying the count times for which Ti K- and Ba L-line data contributed to the combined spectra, a range of samples was simulated having different Ti - Ba compositions. Results from the analysis of composite spectra using the FLS deconvolution software showed that accurate data could be obtained down to concentrations equivalent to 2.5% Ba in spectra dominated by 97.5% Ti. This remarkable performance is placed in perspective by noting that the presence of the Ba L-lines could not be recognised unambiguously by visual appraisal of composite spectra until the equivalent concentration of Ba had been increased to about 12.5%. It is well known that electron excitation is associated with substantially higher background count levels. The successful analysis of electron microprobe excited Ti/Ba spectra at the mass per cent. level, therefore, lends considerable confidence to the reliable analysis of trace element spectra excited by ED-XRF.

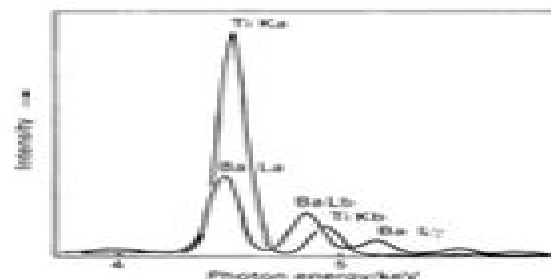


Fig. 1. Electron microprobe spectra of titanium metal and the mineral, baryte, showing the degree of spectral overlap between the Ti K-lines and the Ba L-lines.

Features of XRF Instrumentation

Many features of ED- and WD-XRF instrumentation are comparable, including excitation geometry (with direct tube excitation) and criteria for tube anode selection and optimising kV. Further, sample preparation requirements are equivalent (fused glass discs for the major elements and compressed

Fluorescent X Ray Spectrography Determination Of Trace Elements

Arturo Cuomo



Fluorescent X Ray Spectrography Determination Of Trace Elements:

Fluorescent X-ray Spectrography William Joseph Campbell, John W. Thatcher, 1962 **Quantitative X-Ray Spectrometry** Ron Jenkins, 1995-04-26 This work covers important aspects of X ray spectrometry from basic principles to the selection of instrument parameters and sample preparation This edition explicates the use of combined X ray fluorescence and X ray diffraction data and features new applications in environmental studies forensic science archeometry and the analysis of metals Developments in Applied Spectroscopy Volume 1 W. D. Ashby, 2013-04-17 On May 15 18 1961 the 12th Annual Symposium on Spectroscopy was held in Chicago Over the twelve year history of this meeting it has continually grown and now ranks as one of the major technical meetings in the field of spectroscopy The scope of the program itself represents a balance between research applications and control applications between applied and the more fundamental aspects of spectroscopy Papers are presented each year in the specialty areas of X ray ultraviolet visible infrared Raman EPR and NMR spectroscopy In many instances over the years excellent work has been reported at this symposium and no further publication was made These reports were then essentially lost for further reference It is the purpose of this publication to provide a source of reference for the papers presented at the 12th Annual Symposium This first attempt at publishing a proceedings does not include the entire program Several papers could not be given company clear ance for publication and several more were intended for verbal presentation only and were never written for publication I as Coordinator of the Symposium would like to express my gratitude to the Symposium Committee John Ferraro Elwin Davis Joseph Ziomek John Kapetan L S Gray Jr Russell J Hansen J A Sheinkop L V Azaroff and Carl Moore whose diligence and labor resulted in a truly fine symposium and subsequently made possible this publication *Report of Investigations* , 1962 Heats and Free Energies of Formation of Gallium Sesquioxide and Scandium Sesquioxide Adolph M. Poston, Alla D. Mah, Arthur R. Taylor, Charles Zahn, Daniel Bienstock, Donald F. Smith, F. L. Wideman, Harry Fagan Yancey, James M. Riley, Leonard Obert, Mary F. Koehler, Murray Jacobson, Ronald R. Lowery, Russell L. Lincoln, William Joseph Campbell, William M. Merritts, Willmar T. Benson, Andrew G. Sharkey, Harold J. Heinen, J. Vance Batty, John Nagy, John W. Thatcher, Joseph H. Field, L. B. Pankratz, Max Richard Geer, Robert H. Merrill, T. E. Caldwell, W. C. Miller, Abraham L. Engel, Austin R. Cooper, H. L. Gibbs, Irving Wender, J. G. Myers, Ronald Barany, Thomas A. Morgan, 1962 *Heats and Free Energies of Formation of Gallium Sesquioxide and Scandium Sesquioxide* Alla D. Mah, Clark F. Grain, Donald F. Smith, Eben L. Singleton, Edward Gerard King, Frank A. Peters, Gerald V. Gibbs, John K. Alley, Kenneth Keith Kelley, Mary F. Koehler, Miles E. Tyrrell, Ronald Barany, William A. Cole, William Joseph Campbell, George Fulford Hanson, Haskiel Roy Shell, Irving L. Turner, John W. Thatcher, L. B. Pankratz, L. Carpenter, Paul Watson Johnson, W. C. Miller, W. W. Weller, Arthur R. Taylor, F. Donald Bloss, Ralph C. Kirby, Raynard V. Lundquist, W. T. Westbrook, 1962 **Total-Reflection X-Ray Fluorescence Analysis and Related Methods** Reinhold Klockenkämper, Alex von Bohlen, 2015-01-27 Explores the uses of TXRF in micro and trace analysis and in

surface and near surface layer analysis Pinpoints new applications of TRXF in different fields of biology biomonitoring material and life sciences medicine toxicology forensics art history and archaeometry Updated and detailed sections on sample preparation taking into account nano and picoliter techniques Offers helpful tips on performing analyses including sample preparations and spectra recording and interpretation Includes some 700 references for further study *X-ray Emission Spectrography in Geology* Isidore Adler, 1966 **Symposium on X-Ray and Electron Probe Analysis**, 1964

Division of Soils Technical Paper CSIRO (Australia). Division of Soils, 1971 Energy Research Abstracts, 1977 Semiannual with semiannual and annual indexes References to all scientific and technical literature coming from DOE its laboratories energy centers and contractors Includes all works deriving from DOE other related government sponsored information and foreign nonnuclear information Arranged under 39 categories e g Biomedical sciences basic studies Biomedical sciences applied studies Health and safety and Fusion energy Entry gives bibliographical information and abstract Corporate author subject report number indexes **Physical Methods in Determinative Mineralogy** J. Zussman, 1967 Introduction to X-Ray Spectrometric Analysis Eugene P. Bertin, 1978-03 X ray fluorescence spectrometry has been an established widely practiced method of instrumental chemical analysis for about 30 years However although many colleges and universities offer full semester courses in optical spectrometric methods of instrumental analysis and in x ray dif fraction very few offer full courses in x ray spectrometric analysis Those courses that are given are at the graduate level Consequently proficiency in this method must still be acquired by self instruction on the job training and experience workshops held by the x ray instrument manu facturers the one or two week summer courses offered by a few uni versities and certain university courses in analytical and clinical chemistry metallurgy mineralogy geology ceramics etc that devote a small portion of their time to applications of x ray spectrometry to those respective disciplines Moreover with all due respect to the books on x ray spectrometric analysis now in print in my opinion none is really suitable as a text or manual for beginners in the discipline In 1968 when I undertook the writing of the first edition of my previous book *Principles and Practice of X Ray Spectrometric Analysis* my objective was to provide a student text However when all the material was compiled I decided to provide a more comprehensive book which was also lacking at that time Although that book explains principles instrumentation and methods at the begin ner s level this material is distributed throughout a mass of detail and more advanced material Methods of Soil Analysis: Chemical and microbiological properties Albert Lee Page, 1982 The latest installment in the well received *Methods of Soil Analysis* series *Methods of Soil Analysis Part 5 Mineralogical Methods* presents valuable techniques that will enable researchers to analyze mineralogy for a wide variety of applications An understanding of mineralogical composition provides crucial insight into the fundamental behavior of soils and their response to environmental conditions and management Highlights include extensive coverage of new techniques such as X ray absorption and diffuse reflectance spectroscopy and updated chapters on thermal analysis and selective dissolution

methodologies Each chapter provides the basic principles of the method guides the reader through the method itself and finally assists in the interpretation and analysis of results collected

Handbook of Practical X-Ray Fluorescence

Analysis Burkhard Beckhoff, Birgit Kanngießer, Norbert Langhoff, Reiner Wedell, Helmut Wolff, 2007-05-18 X Ray fluorescence analysis is an established technique for non destructive elemental materials analysis This book gives a user oriented practical guidance to the application of this method The book gives a survey of the theoretical fundamentals analytical instrumentation software for data processing various excitation regimes including grating incidents and microfocus measurements quantitative analysis applications in routine and micro analysis mineralogy biology medicine criminal investigations archeology metallurgy abrasion microelectronics environmental air and water analysis This book is the bible of X Ray fluorescence analysis It gives the basic knowledge on this technique information on analytical equipment and guides the reader to the various applications It appeals to researchers analytically active engineers and advanced students

Developments in Applied Spectroscopy ,1962 Vols 1 are the 12th proceedings of the Mid America Spectroscopy Symposium called 1961 Symposium on Spectroscopy

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Metallurgy Walter Westwood, B. S. Cooper, 1973 *Report - Nevada Bureau of Mines* Nevada Bureau of Mines, 1963

Methods of Soil Analysis: Chemical methods ,1996

X-Ray Fluorescence Spectrometry and Related Techniques

Eva Margui, Rene Van Grieken, 2013-01-25 X ray fluorescence spectrometry XRF is a well established analytical technique for qualitative and quantitative elemental analysis of a wide variety of routine quality control and research samples Among its many desirable features it delivers true multi element character analysis acceptable speed and economy easy of automation and the capacity to analyze solid samples This remarkable contribution to this field provides a comprehensive and up to date account of basic principles recent developments instrumentation sample preparation procedures and applications of XRF analysis If you are a professional in materials science analytic chemistry or physics you will benefit from not only the review of basics but also the newly developed technologies with XRF Those recent technological advances including the design of low power micro focus tubes and novel X ray optics and detectors have made it possible to extend XRF to the analysis of low Z elements and to obtain 2D or 3D information on a micrometer scale And the recent development and commercialization of bench top and portable instrumentation offering extreme simplicity of operation in a low cost design have extended the applications of XRF to many more analytical problems

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