

METHODS AND APPLICATIONS OF RADIOANALYTICAL CHEMISTRY - MARC VI

In the European Phebus project severe reactor accidents are studied in pilot scale. The products released during the melting of the fuel are collected in filters which are then leached with acid. The contents of the acid are then fractionated using solvent extraction and the nuclide concentrations determined by radiometrical methods (α - and γ -spectrometry, and liquid scintillation counting) and ICP-MS. The contribution will describe the used analysis scheme in detail. Experimental errors and uncertainties will also be discussed.

385 LIQUID-LIQUID EXTRACTION APPLIED TO ONE-ATOM-AT-A-TIME STUDIES OF TRANSACTINIDE ELEMENTS. G. Skarnemark*, C. Ekberg, Nuclear Chemistry, Department of Materials and Surface Chemistry, Chalmers University of Technology, S-412 96 Göteborg, SWEDEN

Liquid-liquid extraction is one of the methods that is used for one-atom-at-a-time separations of transactinide elements from heavy-ion reaction product mixtures. It is suitable for this purpose because it is fast, provided that a chemical system with negligible kinetics is used, and it can be used for continuous separations. It is, however, not quite easy to determine the uncertainties of the measured distribution coefficients or complex formation constants. In this paper methods for such estimates will be discussed.

388 IODINE DAILY DIETARY INTAKE IN A GROUP OF BRAZILIAN WORKERS. , V.A. Maihara¹, P.L.C. Moura¹, D.I.T.Fávaro¹, M.B.A.Vasconcellos¹ ¹Radiochemistry Division, Instituto de Pesquisas Energéticas e Nucleares, IPEN/CNEN-SP, SP, BRAZIL

Iodine is an essential constituent of the thyroid hormones thyroxine (T_4 and T_3). The main role of iodine in nutrition arises from the important part played by the thyroid hormones in the growth and development of humans and animals. The recommended dietary allowance (RDA) for adult men and women is 150 $\mu\text{g}/\text{day}$ and the tolerable upper intake level (UL) for adults is 1,100 $\mu\text{g}/\text{day}$. Low iodine levels in the diet lead to iodine deficiency disorders. Iodine deficiencies can be prevented or reduced by increasing of its dietary intake through fortification of food. There is an intensive international effort to fortify the cooking salt with iodine in several countries, including Brazil. In this study, iodine dietary intake was evaluated through determination of iodine in duplicate portion diet samples by epithermal neutron activation. The collection of samples was carried out in a group of twenty-six workers from a steel industry of São Paulo city. The 3 day-diet samples were prepared by freeze-drying process in an industrial freeze dryer and mixed and homogenized in a domestic blender. Epithermal neutron activation analysis (ENAA) was employed to reduce the main interferences present in the diet samples, such as Na and Cl. Three NIST reference materials: RM 8435 Whole Milk Powder, SRM 1549 Non Fat Milk Powder and SRM 1548a Typical Diet were analyzed for validation of the methodology. The average daily dietary intake found for worker's group was 840 $\mu\text{g}/\text{day}$, ranging from 400 to 1540 $\mu\text{g}/\text{day}$. Some of the daily iodine intakes were about 10 times higher than the RDA value.

389 INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS APPLIED TO THE CHEMICAL COMPOSITION OF METALLIC MATERIALS WITH STUDY OF INTERFERENCES. E. G. Moreira*¹, M. B. A. Vasconcellos¹, M. Saiki¹. ¹ Centro do Reator de Pesquisas, Instituto de Pesquisas Energéticas e Nucleares, São Paulo, SP 05508-000, BRAZIL.

Instrumental Neutron Activation Analysis was used to evaluate the chemical composition of iron, steel, silicon and ferrosilicon reference materials. Samples were irradiated at $10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ flux at IEA-R1 Research Nuclear Reactor at IPEN for 30 min for long lived radionuclides and 30 s for short lived ones. Induced radioactivity was measured in a CANBERRA gamma ray spectrometer for suitable periods of time after appropriate sample decay. The concentration of As, Co, Cr, Mn, Mo, Ni, V and W were analyzed in the iron and steel samples whereas As, Br, Co, Cr, K, Eu, Fe, La, Mn, Mo, Na, Nd, Sb, Sc, Sm, Tb Th, U, V, W and Yb were determined in silicon and ferrosilicon samples. Method validation was

assessed by comparison of the obtained results to reference material certified values. As relative element concentration may be large in metallic matrices, interferences in neutron activation analysis are possible to occur. Interference of Cr and Mn in V, Fe and Co in Mn, Co in Fe and Cr in Ti were quantified and only the last one was critical to the analysis of the materials employed in this work.

390 THE SUM-OF-SQUARES SPECTRUM WITH ZERO DEAD TIME COUNTING AND ITS RELATION TO STATISTICAL UNCERTAINTY. S. Pommé. EC-JRC-IRMM, Institute for Reference Materials and Measurements, Retieseweg, B-2440 Geel, BELGIUM

Important novelties implemented in the DSPEC^{plus}™ digital spectrometer are the 'Zero Dead Time' (ZDT) pulse loss correction system, in combination with the 'sum-of-squares' spectrum. The latter consists of the squares of the number of counts registered per event in the corresponding channels of the ZDT-spectrum. The constructor calls it the 'variance' spectrum, claiming that the statistical variance of the ZDT-counts corresponds to the number of counts in the 'sum-of-squares' spectrum. In this work, the validity of this statement is scrutinised on the basis of mathematical and experimental evidence.

391 PIXE ANALYSIS FOR THE BIOACCUMULATION OF SEVERAL TRACE METALS BY MARINE MICRO-ALGA. Y. IWATA*, A. SATOH, Y. SASAKI, R. ITO and K. KURAMACHI. Department of Chemistry, Faculty of Education and Human Studies, Akita University, Akita 010-850, JAPAN

The bioaccumulation by micro-alga in the ocean was simulated in the nutritive seawater containing known amount of trace metals and concentration factors for Fe, Zn, Cd and Pb were measured by PIXE. Trace metals in nearshore seawater obtained from Kamaishi Bay were removed by Celex-100[□]. Trace metals and nutritive salts were added to the purified seawater. Marine micro-algae (*Nannochloropsis sp.*, and *Phaeodactylum sp.*) were purely cultured in the nutritive seawater. An interested metal ion was added to the nutritive seawater (0.025 - 5.0 mg / l). The alga in 10 ml of the nutritive seawater was collected on a polycarbonate filter (pore size : 1.0 µm) by a suction filtration and subjected to the 2.9 MeV proton bombardment. Na, Mg, Si, P, S, Cl, K, Ca, Cr, Mn, Fe, Cu, Zn, Cd and Pb were simultaneously determined. PIXE could do multi-element analysis by less than 1 mg of analytical sample. The quantity of the metal in alga was increased in proportion to the concentration in the nutritive seawater. For example, the concentration factor for zinc was given as 10200 ± 300 ml / g (dry weight base) for *Phaeodactylum sp.*. It is shown that PIXE is a powerful tool for the measurement of the bioaccumulation of trace elements.

392 PREPARATION OF PROFICIENCY TEST SAMPLES CONTAINING 'FRESH' FISSION PRODUCTS. Arend V. Harms[†], Satwant K. Johal and Simon M. Jerome[‡], National Physical Laboratory, Teddington, UK

NPL was recently asked to provide sources for two proficiency test exercises. The aim of these exercises was to measure fission product radionuclides arising from the neutron irradiation of highly (>99%) enriched ²³⁵U, within 50 days (preferably less) of the irradiation taking place. Furthermore, the samples were to contain fission products, but no fissile material. The samples in the first exercise were to contain a mixture of fission products with (as far as possible) only the ²³⁵U removed, leaving the fission products as 'undisturbed' as possible. A number of radionuclides were reported in this sample and included; ⁹¹Y, ⁹⁵Zr, ^{95m}Nb, ^{95g}Nb, ⁹⁹Mo, ^{99m}Tc, ¹⁰³Ru, ¹⁰⁶Ru, ¹²⁷Sb, ^{129m}Te, ^{129g}Te, ¹³¹I, ¹³²Te, ¹³²I, ¹³⁷Cs, ¹⁴⁰Ba, ¹⁴⁰La, ¹⁴¹Ce, ¹⁴⁴Ce, and ¹⁴⁷Nd. Of these nuclides, NPL provided certified values for ⁹¹Y, ⁹⁵Zr, ^{95m}Nb, ^{95g}Nb, ⁹⁹Mo, ^{99m}Tc, ¹⁰³Ru, ¹⁰⁶Ru, ¹³²Te, ¹³²I, ¹³⁷Cs, ¹⁴⁰Ba, ¹⁴⁰La, ¹⁴¹Ce, ¹⁴⁴Ce, and ¹⁴⁷Nd. The second exercise focused on a smaller subset of radionuclides; ⁹⁵Zr, ⁹⁹Mo, ¹⁰³Ru, ¹⁰⁶Ru, ¹⁴⁰Ba, and ¹⁵⁵Eu, although ^{95g}Nb, ^{99m}Tc, ¹³²I and ¹⁴⁰La were present as daughter radionuclides and ⁹¹Y, ¹²⁷Sb, ^{129m}Te, ^{129g}Te, ¹³¹I, ¹³²Te, ¹³⁷Cs, ¹⁵²Eu, ¹⁵⁴Eu and ¹⁵⁵Eu were present as impurities. This paper describes the development of suitable separation techniques, based on a combination of extraction