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HIGH ENERGY ELECTRON IMPACT IONIZATION

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A method has been demonstrated in which the emission of characteristic X-rays following impact ionization by electrons of several MeV energy is used for elemental analysis by energy dispersive X-ray spectrometry. Because of the unique properties of MeV electrons with respect to their X-ray production yield and range in solids, the method has comparable sensitivity over a wide range of elements and does not require stringent sample preparation techniques. By using energy dispersive analysis to detect the X-rays, a simultaneous determination of the weight fraction of the elements in an unknown sample is possible from a procedure based entirely on empirically determined relative X-ray yields.

The relative X-ray yields were established from simultaneous measurements of the X-rays from targets containing a known ratio of two elements. The targets were made by dissolving solid samples and adsorbing the solutions onto identical thin adsorbent supporting matrices of either filter paper (8.15 mg/cm^2) or lens tissue (1.35 mg/cm^2). The thinner lens tissue targets were found to be more advantageous in several respects and were therefore used in the majority of the work. Typically, these latter targets contained ~1 mg/cm^2 of sample material. Various detection systems using high resolution liquid nitrogen cooled Ge(Li) and Si(Li) detectors were employed for the studies. For one of the detection systems utilized, the Si(Li) detector was directly vacuum coupled to the evacuated electron beam tube. In this case, it was possible to detect all elements down to aluminum ($Z = 13$). The method developed for measuring the relative yields and making the elemental assays was shown to be both simple and rapid. The entire procedure requires only relative measurements. All of the measured quantities are obtained from ratios of simultaneously recorded X-ray intensities and weight ratios. It, therefore, does not require any absolute measurements of the number of atoms or the number of X-ray produced.

The capability of the method for quantitative analysis was tested by analyzing three standard metal alloy samples obtained from the National Bureau of Standards. Elements ranging from iron ($Z = 26$) to lead ($Z = 82$) with concentrations from 0.06 % to 88 % were quantitatively determined. In general, good approximate agreement with the reported assays was found. For even the minor constituents, the accuracy of the results were of the order of 5 - 10 %.

Relative cross sections for K-shell ionization by 2.04 ± 0.01 MeV electrons for 31 elements were extracted from the relative Kα X-ray yield measurements. The measurements were made with the Si(Li) detector for 27 of the elements in the range $Z = 23 - 70$, and with the Ge(Li) detector for 14 of the elements in the range $Z = 35 - 83$. In the cases when comparisons were possible, the two sets of measurements were in good agreement. The results were compared to the Kolbenstvedt[*] theory for K-shell ionization by relativistic electrons. In general, the theoretical values were found to be in good approximate agreement (± 25 %) with the measurements. In the medium Z region ($Z \simeq 50$), the agreement is excellent, but systematic deviations were found in both low and high Z regions. The cross sections predicted by the Kolbenstvedt theory are systematically too small in the low Z region ($Z \lesssim 40$) and too large in the high Z region ($Z \gtrsim 60$). The deviation of the Kolbenstvedt theory in the heavy elements was observed in prior measurements for gold ($Z = 79$) at 2 MeV[†] and also at 2.5 and 7.1 MeV.[‡]

The relative cross sections were combined with the absolute cross section measurement of Rester and Dance[†] for tin at 2 MeV [σ_K (Sn) = 44 ± 4 b] to obtain absolute cross sections for all 31 elements. They are (in barns): V, 361 ± 53; Cr, 277 ± 41; Mn, 276 ± 43; Fe, 261 ± 38; Co, 246 ± 40; Ni, 240 ± 38; Cu, 203 ± 30; Zn, 188 ± 30; As, 138 ± 21; Se, 114 ± 19; Br, 111 ± 12; Rb, 105 ± 15; Sr, 91.2 ± 13.1; Ag, 57.8 ± 5.2; Cd, 46.8 ± 4.1; In, 44.2 ± 4.0; Sb, 42.1 ± 3.7; Te, 38.6 ± 3.4; Ba, 30.9 ± 2.8; La, 19.2 ± 2.5; Ce, 23.6 ± 3.1; Pr, 24.1 ± 3.2; Nd, 22.0 ± 2.0; Sm, 21.1 ± 2.0; Eu, 21.5 ± 3.1; Gd, 21.2 ± 2.0; Er, 16.9 ± 1.6; Yb, 15.6 ± 1.6; Pt, 11.9 ± 1.5; Pb, 10.0 ± 1.2; Bi, 9.5 ± 1.2.

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*H. Kolbenstvedt, J. Appl. Phys. 38, 4785 (1967).

†D. H. Rester and W. E. Dance, Phys. Rev. 152, 1 (1966).

‡K. H. Berkner, S. N. Kaplan and R. V. Pyle, Bull. Amer. Phys. Soc. 15, 786 (1970).

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RENSSELAER POLYTECHNIC INSTITUTE

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Doctor of Philosophy

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by

Ronald P. Collé

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ABSTRACT

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