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A RADIOISOTOPE SOURCE – TARGET ASSEMBLY FOR X-RAY SPECTROMETRY

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The development of high-resolution semiconductor detectors has made them applicable for X-ray emission spectrometry when used in conjunction with radioisotope sources for exciting the X-rays(1). Semiconductor detectors with resolutions better than 0.5 k.e.V. (full width at half maximum) are now available commercially. L. Reiffel (2), J. S. Watt (3), and J. R. Rhodes (4), employing other detection systems, have described radioisotope source-target assemblies for nondispersive X-ray fluorescence analysis. A modification of these assemblies is described here which makes it compatible with semiconductor counters as detectors.

The source-target assemblies reported by Reiffel (2), Watt (3), and Rhodes (4), all employ a two stage system of excitation. A radioactive source is used to excite the K X-rays of a selected target material, and these in turn excite X-rays from the specimen. The geometrical arrangement is such that the specimen sees primarily the X-rays of the target while the detector sees primarily the X-rays excited from the specimen and the backscattered target X-rays. In these systems, point radioactive sources were used along with scintillation or proportional counters as detectors.

The assembly described here is a modification made necessary by the fact that the lithium-drifted silicon and germanium detectors of high resolution are quite small in area. As shown in Figure 1, the radioisotope source has annular geometry as does the primary target. Some of the radioisotopes that can be used as sources are ^{241}Am, ^{109}Cd, ^{125}I, and ^{57}Co. Table I lists the half-lives of these radioisotopes, their useful radiations, and the intensities of these radiations per disintegration. ^{241}Am is a particularly good source because its useful radiations cover a wide range of energies and its long half-life provides a constant intensity source. In order to obtain high sensitivity in analysis, primary targets are chosen whose characteristic K X-ray energies are not too far above the absorption edges of the elements to be determined. Similarly the radioisotope source should have photons not too much higher in energy than the absorption edge of the target.

^{241}Am annular sources have been made by absorbing a solution of $AmCl_3$ on Al_2O_3 powder and loading the powder in an aluminum tube which has been previously annealed. The tube has an od of 1/16 inch and a wall thickness of 0.0005 inch. Once the tube, bent into annular shape, has been placed in the groove of a lead block source holder, a thin layer of epoxy resin is cast over the top of the tube. The purposes of the Al_2O_3 powder and the aluminum tube are to distribute the activity evenly and to contain the alpha radiation. When exciting K X-rays of targets with an atomic number higher than 39 the NpLα and Np Lβ X-rays are useless and should be filtered out in order to eliminate the small peaks in the spectrum which appear from double scattering. The filter consists of a 0.005 inch thick copper disc covered in turn by a 0.005 inch aluminum disc which absorbs the CuK X-rays which are excited.

This filter reduces the intensity of the 59.6 k.e.V. γ-ray by only about 10% but the NpLα and NpLβ X-rays by 99.5%.

^{125}I sources have been made in the same manner as the ^{241}Am sources except that a tin holder is used instead of lead so that the source radiation will be free of PbL X-rays. Typical activity for these sources is 20 millicuries. The shield used to hold the target materials is made of lead. When determining the content of elements where the excitation of a small amount of Pb X-rays from the shield collimator would interfere in the analysis, either the collimator must be lined with an appropriate material or a shield may be made from another material such as tin.

The primary targets are either cut from pure metal sheets or made from castings. Metal powders, metal oxides, or appropriate compounds are mixed with a minimum of epoxy resin and plasticizer and cast in aluminum cups after pumping the air from the mixtures. These castings are machined and polished.

Rhodes (4) reports the use of a radioisotope source-target assembly to correct for several of the major problems of X-ray fluorescence analysis, matrix enhancement and adsorption effects. High analytical sensitivity is obtained by using a target which has its characteristic Kα X-ray energy just above the absorption edge of the element to be determined. Matrix enhancement is usually eliminated by this procedure and matrix absorption is compensated by normalizing to the backscatter peak of the target's Kα X-rays.

The source-target assembly in Figure 1 has been used to analyze glass and ceramic archaelogical artifacts where the destruction of these artifacts was not permitted. A detailed description of the equipment used along with a multichannel pulse-height analyzer has been given elsewhere (1). When present

in low cencentrations (1% or less), several elements with comparable X-ray energies often may be determined with the same target. Detection limits of 100 p.p.m. and less for many elements can usually be obtained. Typically, analysis of a specimen for more than a dozen elements (Mn and higher atomic number elements) could be accomplished in less than one hour. Figure 2 shows a Mosley plot of a glass specimen containing 1.0% Cu, 0.15% Co, 0.8% Fe, and 0.9% Mn excited with AsK X-rays excited from an As_2O_3 target by an ^{241}Am source and detected with a lithium-drifted silicon detector. It will be noted that the peaks from manganese, iron and cobalt are not resolved. With the data still in the memory of the pulse-height analyzer, the specimen is replaced by a piece of iron and the analyzer is run in the "subtract mode" until the iron X-ray peak, as seen in the oscilloscope screen, is reduced to the background level. The operation takes only a few seconds and the results are shown in Figure 3. The accuracy of many of these analyses were often good only to 10%. An accuracy of around 1% should be obtainable for many analyses where the composition of the samples do not vary over a wide range, when particular care is taken in the sample preparation, and where sufficient care is taken in standardization.

Literature Cited

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Acknowledgments

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Table I. Radioisotopes for Source - Target Assemblies

Radioisotope	Half-life	Useful radiations	Intensity per disintegration[5]
Americium - 241	458 years	13.9 k.e.V. NpLα X-rays	0.14
		17.8 k.e.V. NpLβ X-rays	0.18
		20.8 k.e.V. NpLγ X-rays	0.05
		26.4 k.e.V. γ-rays	0.03
		59.6 k.e.V. γ-rays	0.36
Cadmium - 109	453 days	22.1 k.e.V. AgKα X-rays	0.7
		25.0 k.e.V. AgKβ X-rays	0.15
		87.7 k.e.V. γ-ray	0.03
Iodine - 125	60 days	27.4 k.e.V. TeKα X-rays	1.1
		31.0 k.e.V. TeKβ X-rays	0.2
		35.5 k.e.V. γ-ray	0.07
Cobalt - 57	270 days	14.4 k.e.V. γ-ray	0.09
		122.0 k.e.V. γ-ray	0.87
		136.3 k.e.V. γ-ray	0.11

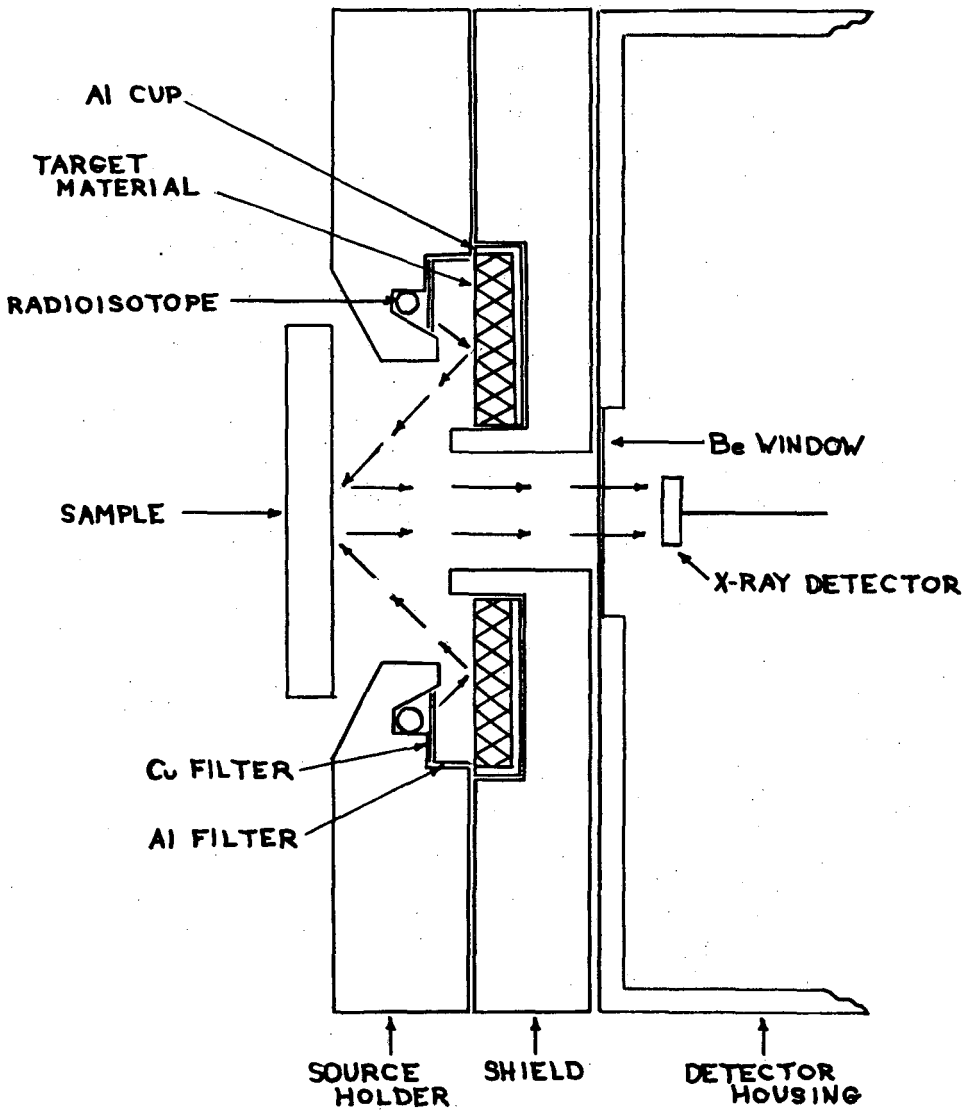
Figure Captions

Fig. 1 Source-target assembly.

Fig. 2. Spectrum from a glass specimen containing 1.0% Cu, 0.15% Co, 0.8% Fe, and 0.9% Mn excited with AsK X-rays and detected with a lithium-drifted silicon detector.

Fig. 3 Spectrum from Figure 2 with the iron peak subtracted out by exciting a pure piece of iron metal with the analyzer in the "subtract mode."

SOURCE-TARGET ASSEMBLY



XBL 686-1016

Fig. 1

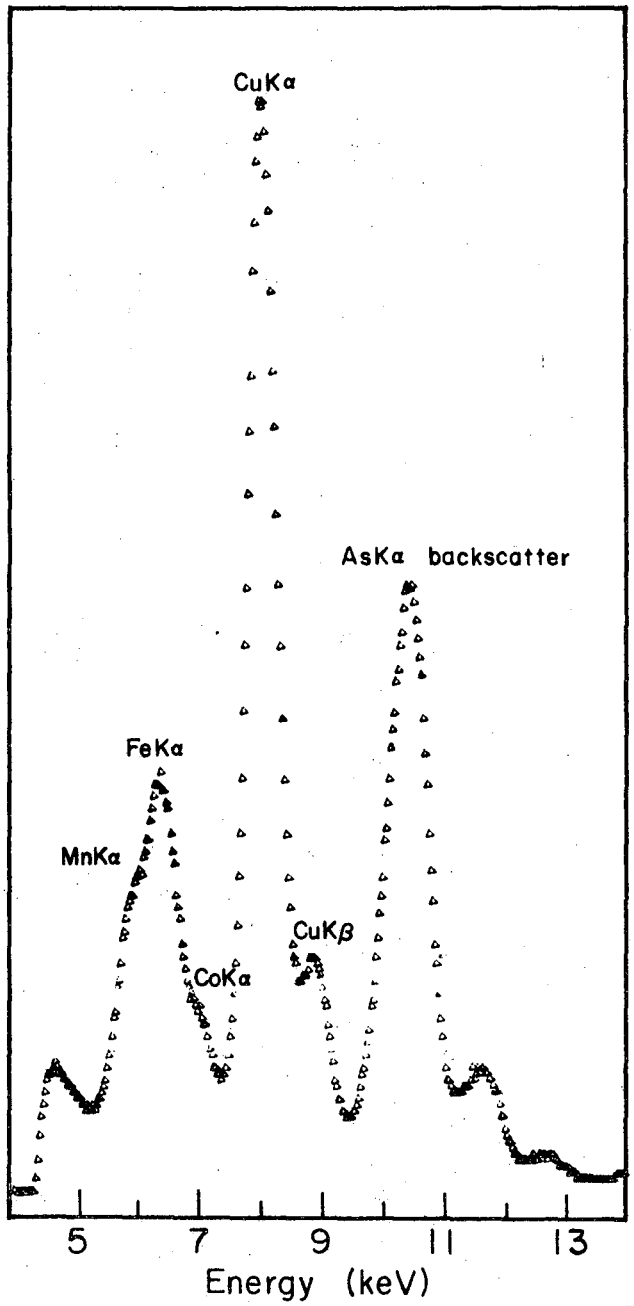

CuKα
AsKα backscatter
FeKα
MnKα
CuKβ
CoKα
5
7
9
11
13
Energy (keV)
XBL686-2943

Fig. 2

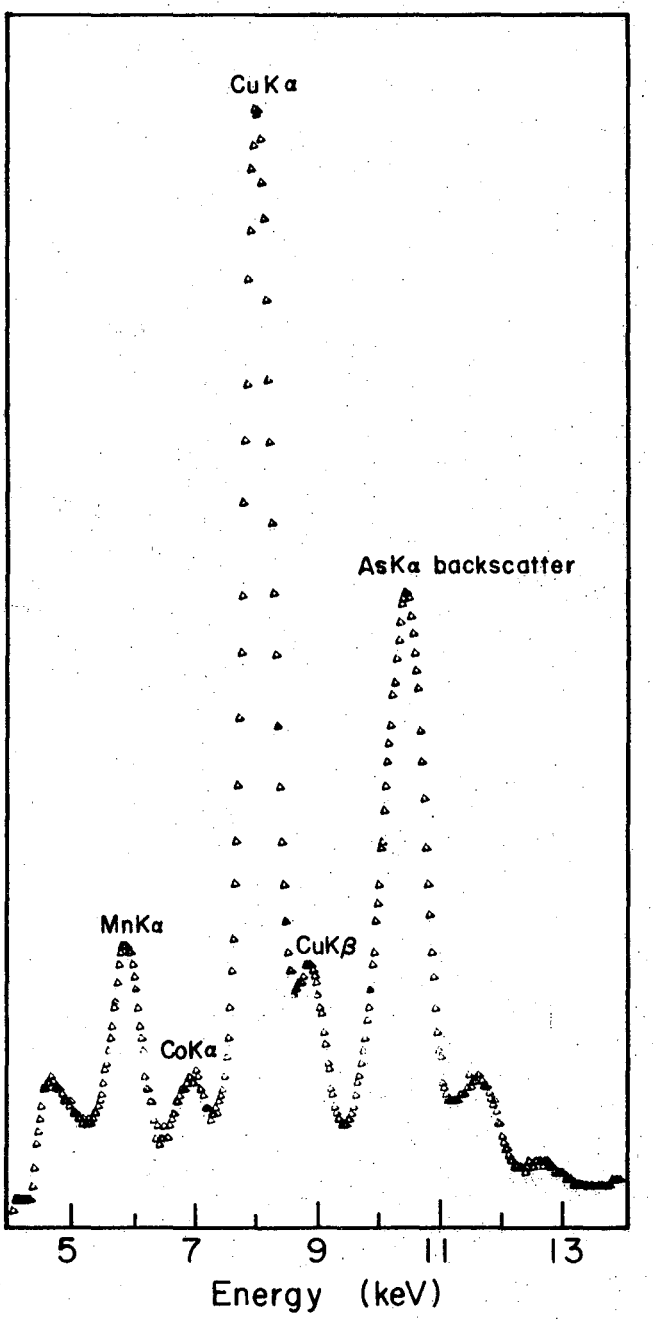

CuKα
AsKα backscatter
MnKα
CuKβ
CoKα
5
7
9
11
13
Energy (keV)
XBL686-2942

Fig. 3