

Progress Notes contain *interim* data and conclusions and are presented as a service to other wildlife biologists and agencies.

No. 49, October 1975

Experimental use of a helium atmosphere in X-ray spectroscopy

by John P. Kelsall¹ and Roland Burton²

Abstract

An X-ray fluorescing spectrometer, using 25 mCi of Americium 241 as a radioactive source has been used in a CWS study dealing with the chemical elements in waterfowl flight feathers. The spectrometer was run in air. In an effort to enhance results some experimental spectra were accumulated in a helium atmosphere. We found enhancement of a few low energy elements between sulphur and iron inclusive on the energy scales. Our equipment is not suitable for measuring elements below sulphur, but is suitable for measuring elements up to approximately 40 keV (iron lies at approximately 7 keV). The conclusion was that enhancement of the spectra was over too narrow an energy range and involved too few elements to be of real value. In addition, control of the helium atmosphere would present real difficulties if large numbers of samples were to be run on a routine basis.

Résumé

Au cours d'une étude du SCF sur les éléments chimiques des plumes rectrices d'oiseaux aquatiques, nous avons utilisé un spectromètre à fluorescence par rayons X et 25mCi d'américium 241 comme source radioactive. Les mesures ont été faites en atmosphère normale. Afin d'améliorer les résultats, nous avons établi certains spectres expérimentaux dans une atmosphère d'hélium. Nous avons constaté une augmentation d'intensité pour quelques éléments de faible énergie situés entre le soufre et le fer inclusivement, dans les échelles d'énergie. Nos appareils ne nous permettent pas de mesurer les éléments en-dessous du soufre, mais ils conviennent à la mesure des éléments jusqu'à 40 keV environ (le fer se situe à 7 keV environ). Nous en avons conclu que l'augmentation d'intensité du spectre touchait une zone énergétique trop étroite et impliquait trop peu d'éléments pour être réellement valable. De plus, le contrôle de l'atmosphère d'hélium comporterait des difficultés importantes s'il fallait analyser, de façon régulière, un grand nombre d'échantillons.

Introduction

Canadian Wildlife Service Project 6252 used an X-ray fluorescing spectrometer, and 25 mCi of Americium 241 as a radioactive source, in a study dealing with the chemical elements to be found in the primary flight feathers of lesser snow geese (*Chen caerulescens*). The equipment provides greatest efficiency when chemical spectra are accumulated in a vacuum. However, vacuum-based systems are expensive; they often hold only one small sample at a time and, even when set up for automatic sample changing, will not deal with the sorts of biological materials which we use. As a result of these considerations, we purchased a system which

runs in air and which incorporates a sample changer, of our own fabrication, approximately 1 m in diameter.

Low energy X-rays are absorbed to some extent by air, and thus our equipment has a reduced sensitivity to low energy elements. In the hope of improving on performance in air, we decided to find out if accumulating spectra in helium would enhance sensitivity.

Materials and methods

An enclosure was made of 4 mil plastic sheeting fitted around the radioactive source, with a detector, sample holder, and one standard sample of three clean snow goose feathers superimposed one on top of the other. The enclosure was held together with masking tape and was constructed so that it could be collapsed to replace the air in it with helium. A preliminary look at the M emission lines for lead, at 2.3 to 2.6 keV, suggested a 2x enhancement factor when accumulating in helium.

Using the same standard set of three feathers, several spectra were accumulated in both helium and air. Each sample was run until 1,000,000 emission rays had been counted, and each such spectrum took about 3 hours to accumulate.

Results

Figure 1 shows a typical spectrum resulting from running a standard feather sample in air. Figure 2 shows the spectrum resulting from the same sample run in a helium atmosphere. The other spectra developed show no significant deviations from Figures 1 and 2, and are not reproduced here.

For all practical purposes the two figures are identical above the iron K beta peak which occurs at 7.057 keV. The large peaks which occur to the right of that point are all due to such factors as electronic drift, backscatter from the tungsten shielding about our radioactive source and backscatter from the source itself. This last factor, incidentally, provides a clear illustration of why Americium 241 must be considered a relatively "dirty" source of radiation. Backscatter peaks are so frequent and large between 8 and 27 keV, that they frequently mask peaks within that range that are due to chemical elements.

When Figures 1 and 2 are compared, the peaks for iron and manganese are seen to be only marginally larger when developed in a helium atmosphere. Calcium sensitivity is about doubled, and sulphur about quadrupled. No new elements were seen by running the spectrum in helium — a considerable disappointment.

Conclusions

The experiment demonstrated that a helium atmosphere is beneficial when examining elements between iron and sulphur on the energy scale. Our equipment is not suitable for measuring elements below sulphur, although some might be detected if present in large quantities in a vacuum. Sulphur generally exceeds 10,000 ppm in feathers and we sometimes

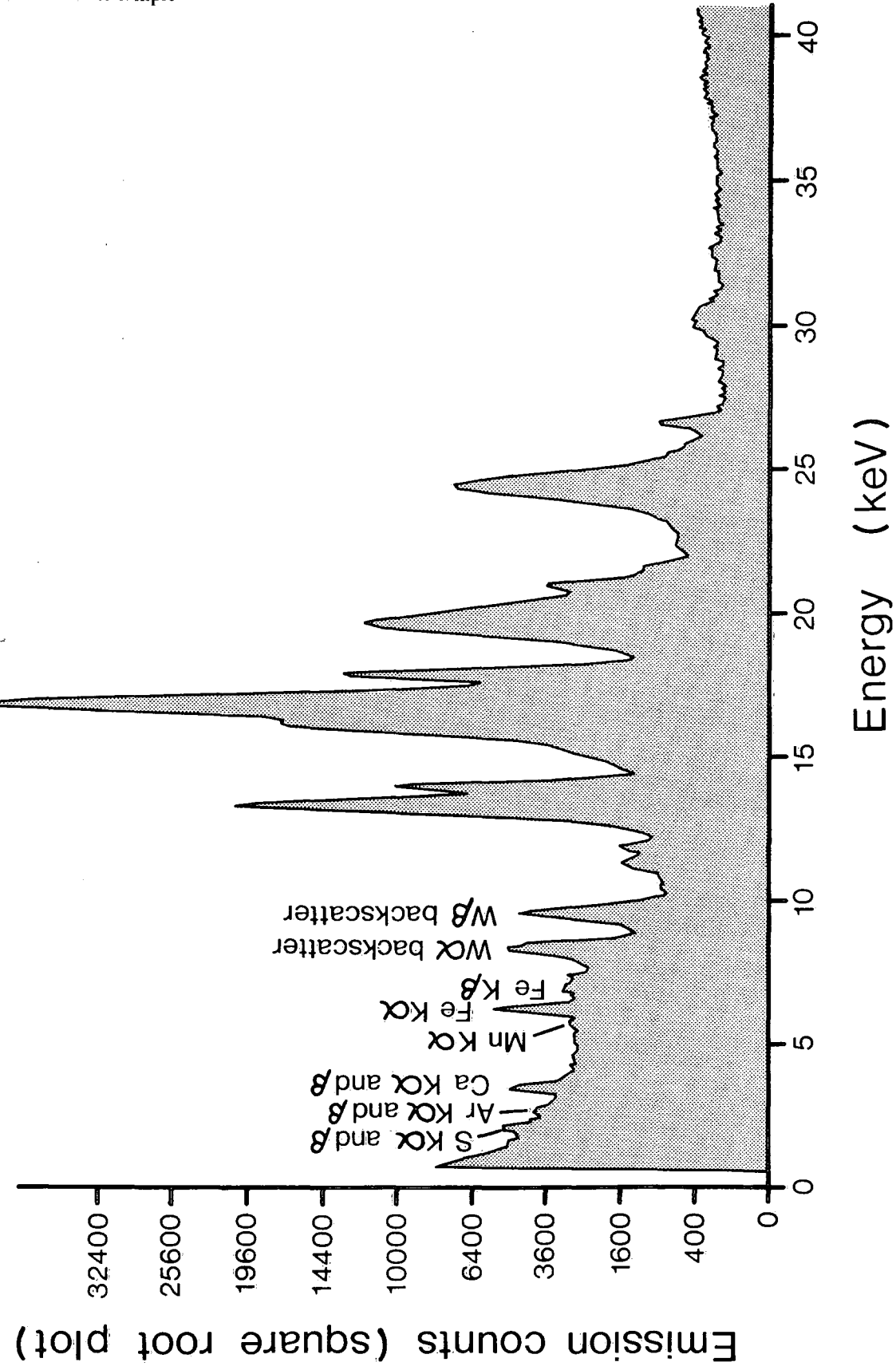
¹CWS, Western Region, Edmonton, Alta.
²4684 West 4th Avenue, Vancouver, B.C.



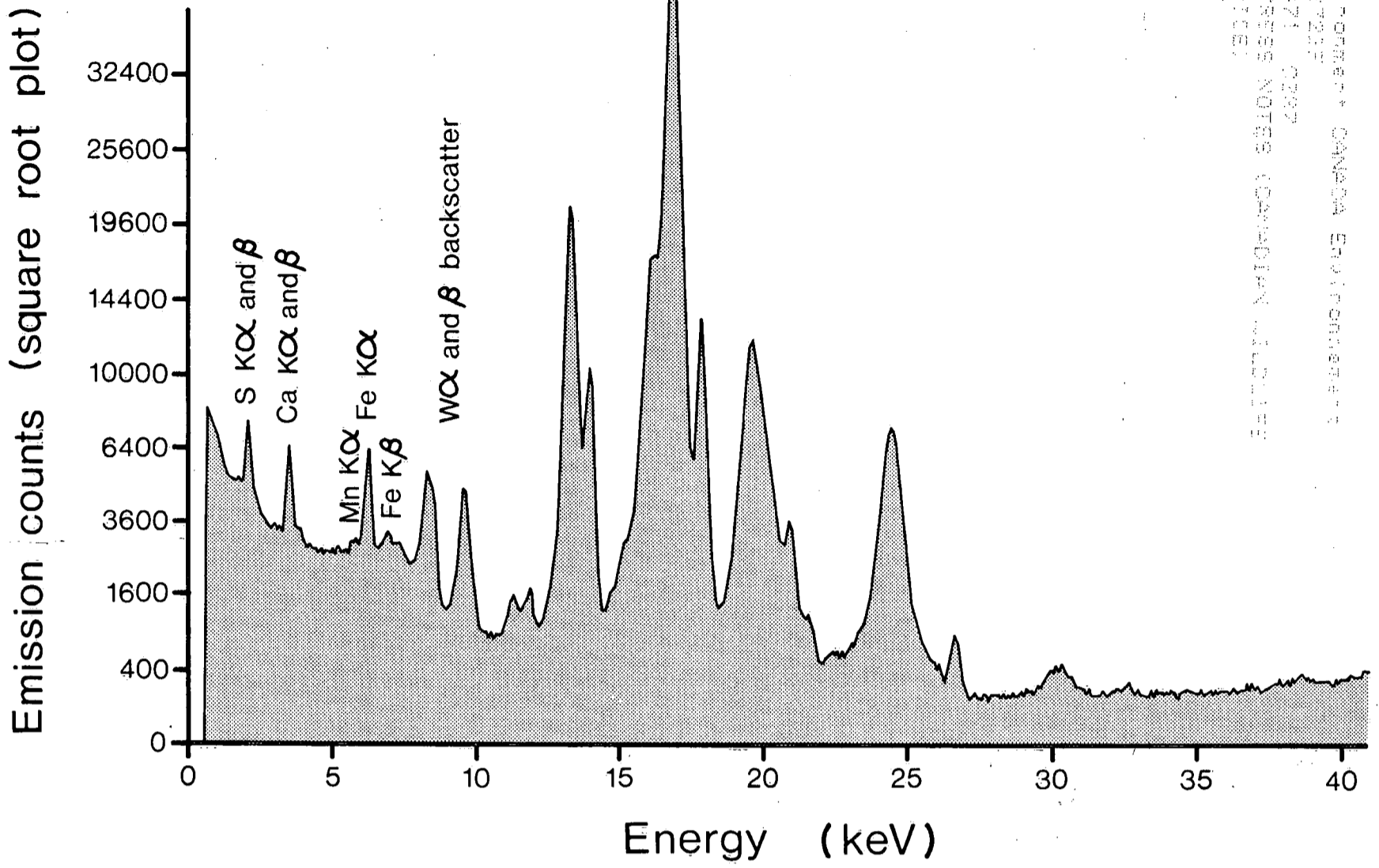
detect argon, which comprises about 1% of air, when dealing with samples (such as feathers) that have little density.

Although there is enhancement of some low energy elements, problems connected with running in a helium atmosphere make the procedure of doubtful value. This trial run, which involved amassing four spectra in helium over a 12-hour period, used about \$20 worth of that gas. A leak-proof enclosure, which would permit free revolution of the sample changer, would be difficult and costly to construct and would use much more helium per sample. A steady helium concentration would have to be reached in the enclosure. Any dilution with air would cause apparent and unpredictable changes in the concentrations of elements being measured.

Figure 2
Spectrum accumulated in a helium atmosphere from a standard feather sample



A74-118



5701008787+ CANADA Environmental
 02287222
 SK 071 0282
 PROCESSES NOTBS (CANADIAN MILITARY
 SERVICE)
 0000

Figure 1
 Spectrum accumulated in air from a standard feather sample