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THE SPECTROGRAPHIC ANALYSIS OF RADIOACTIVE MATERIALS

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ABSTRACT

A convenient and flexible arc-spark chamber has been built and mounted in gloved boxes to be used in the spectrochemical analysis of alpha-active elements. Tests indicate that the radioactivity released during use of the equipment is efficiently removed by the accompanying filter system.

INTRODUCTION

The spectrochemical analysis of the transuranium elements, which are mainly alpha emitters, presents a problem not usually encountered in spectroscopy. The primary problem is the health hazard to operating personnel and the secondary problem, the contamination of valuable spectrographic equipment which could render it unusable for normal operation.

Since the Copper Spark Method\(^1\) can use samples up to about 50 micrograms of the transuranium elements, the danger to personnel may be seen when you consider that one microgram continuously present in the body is a maximum lifetime tolerance after which a person should be retired from work on radioactivity.\(^2\) These elements have specific activities ranging from \(10^4\) to \(10^9\) counts per minute per microgram. If air contamination level is kept below \(5 \times 10^{-10}\) micrograms per cc of air, the body cannot accumulate
the above amount. It has been the aim of this laboratory to build equipment which would work at levels many fold below tolerances. To accomplish this it was found that the chamber, filter system and all connections had to be vacuum tight.

SPARK CHAMBER

An arc-spark chamber has been built which confines the contaminated atmosphere to the vicinity of the discharge. (See Fig. 1). Each electrode is held by means of a length of spring steel against a \( \frac{1}{4} \)\" grooved block of stainless steel which is fastened to a Lavite insulator by machine screws. Each insulator, which is mounted on the end of a 1/2" steel shaft, may be loosened and the whole assembly pivoted to align the electrode horizontally. This adjustment is allowed so that the electrodes are positioned one above the other. The steel shafts are connected on the under side of the base plate to metal bellows which allow up and down motion under vacuum tight conditions. The vertical motion for each shaft is provided through a set of bevel gears and a screw which is connected to the shaft. The electrical leads into the chamber are mica insulated rods. (3) The lid of the arc-spark chamber is of brass 5 inch in diameter and 5 1/2 inches high and built with three windows. (Fig. 2). The left hand one, that is the one facing the spectrograph is of quartz, the other two are of Pyrex. The lid is counter-balanced to aid in handling. The collar, which has the 4 handles, is threaded with a quarter turn breech type thread. This engages the thread on the base plate and gives a quick vacuum tight lock with a minimum of motion which is a great advantage when working in a confined space such
as a gloved box. The gasket for the windows and between the base and lid are of round rubber. (4)

The entire assembly is mounted on rails to allow cross alignment on the optical axis.

An air inlet is provided near the roof of the chamber and an outlet at the base.

ARG-SPARK CHAMBER FILTER SYSTEM

Before, during and after the excitation of the sample, the chamber is flushed with air at atmospheric pressure. The flow rate is adjusted to draw 1 cubic ft. per minute through the system. This air is then passed into the first monitor filter which consists of two rectilinear brass shells between which is clamped a piece of 9" x 4 1/2" filter paper. (5) The air then goes to a special vacuum tight unit made to hold two 9" x 9" x 3 1/16" CWS filters (6) in series. (Fig. 3). The air passes to a second monitor unit which is a duplicate of the first and then to the air mover. The air mover is a mechanical vacuum pump (7) which has its exhaust connect to the box blower system. The second monitor filter permits monitoring of the air from the CWS filter without opening it to the room.

The filter is located in a small gloved box on the shelf and has filtered air continually circulating through it. Should this second monitor filter show activity it would indicate a breakdown of the CWS unit.

GLOVED BOX FILTER SYSTEM

The incoming box air passes through a pad of PF 105 (8) filter material and is introduced into the boxes through horizontal slotted tubes
located in the lower corner of the boxes. The outlets near the top of the boxes have sealed damper units to control the flow rate. The air is drawn from the box into a CWS Filter then to the air mover and then exhausted into the atmosphere on the roof of the building.

The air mover is a ILG\(^{(9)}\) blower, and it is mounted on the roof to eliminate noise and to keep the pressure side of the blower system outside of the room. There is actually a duplicate blower arrangement with one blower acting as a standby which will be automatically energized upon the electrical or mechanical failure of the first. Emergency power connections are also incorporated into the electrical system for it is essential to maintain the boxes under negative pressure at all times.

**EFFECTIVENESS OF THE EQUIPMENT**

Several samples of americium totaling 100 micrograms (approx $10^8$ counts per minute) were sparked according to the copper spark method. The room air was monitored and showed no activity. The second monitor filter showed no activity. Chemical recovery of the activity on the first filter paper accounted for approximately 60% of the material sparked. Stainless steel counting plates placed in the arc-spark chamber indicated that approx. 7% of the initial material sparked was deposited on the inside surfaces.

Tests on the inside surface of the gloved boxes showed that if any activity from the arc-spark chamber got into the box it was swept into the air flow and did not settle on the box walls.

Additional samples have been analyzed and as yet no activity has been detected in the room or on the second monitor filter. By the monitoring methods used, 5 counts per minute can be detected and measured during a
a 4-hour air sampling time. If 5 counts per minute were detected after radon decay, this would be 17 times lower than the tolerance set at the Chalk River Conference (10) and approximately 112 times below that of the California State Industrial Tolerance (11).

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6. Chemical Warfare Service No. 6 Filter.


8. Fiberglass Aerocar PF 105 .6#/cu. ft. manufactured by Owens-Corning Co.

9. ILG #7 1/2P. Manufactured by ILG Electric Ventilating Co., Chicago, Ill.


FIGURE CAPTIONS

Fig. 1 A close-up view of the arc-spark chamber in the open position.

Fig. 2 A general view of the arc-spark chamber mounted in a gloved box. On the right is the first monitor filter in the open position.

Fig. 3 A general view of the arc-spark chamber box and the sample preparation box as it is positioned at the end of the optical bench. On the low shelf is the chambers CWS unit and the second monitor filter; on the shelf above is the box CWS unit; on the floor is the chambers air mover.