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# Development and Testing of a Double-Beam Absorption Spectrograph for the Simultaneous Determination of Different Cations in Water

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DEVELOPMENT AND TESTING OF A DOUBLE-BEAM ABSORPTION  
SPECTROGRAPH FOR THE SIMULTANEOUS DETERMINATION  
OF DIFFERENT CATIONS IN WATER

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## ABSTRACT

Construction and testing of a double-beam absorption spectrographic analysis system using a d. c. arc multielement source has been conducted. An optical system design which brings analytical and reference beams together to illuminate the upper and lower portions of the spectrograph slit has been shown to be functional. However, a d. c. arc will not serve as a multielement source for the intended purposes because of excessive thermal broadening of emission lines.

A direct excitation d. c. arc method was reviewed which can be used for the rapid determination of some cations in water.

## ACKNOWLEDGMENTS

The assistance of Mr. Philip Davis during the preliminary phases and Mrs. Kristin Dennen as the project technician are recognized as critical to the conduct of this work. I wish also to thank Prof. Gary Christian for the loan of various burners and Prof. W. H. Blackburn for the use of instruments in the Cabot Spectrographic Laboratory.

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## CHAPTER I

### INTRODUCTION

The objectives of this project were to develop and test a simple double-beam absorption spectrographic system capable of providing simultaneous determination of different cations in samples of water.

A functional double-beam external optical system was constructed on a Bausch and Lomb 3m Littrow-mounted prism spectrograph. The beams arise in the same source, provide a reference and analytical path, and are brought together at the spectrographic slit where they comprise the upper and lower portions of spectral lines.

The analytical scheme originally attempted was to measure cation concentration in water by passing light from a simple multielement source (d. c. arc) through a water sample aspirated into a flame. Neither this source nor simple variations of it proved satisfactory for reasons later given. Direct excitation techniques were also reviewed and could well be applied to some water problems.

## CHAPTER II

### RESEARCH PROCEDURES AND RESULTS

The double-beam optical path constructed and used is shown diagrammatically in Figure 1 (photographs are also available from the author). It should be noted that this system does not require a chopper, multiple sources, monochromators, or other devices; the reference and analytical beams arise in the same source, and are arranged to illuminate different portions of the slit. The relative intensity of light in the analytical and reference paths is found by comparing the upper and lower portion of a spectral line.

Following construction of the double-beam path, numerous tests were carried on in an attempt to obtain a working analytical system to measure cation concentration in water. These were unsuccessful. The tests were conducted using a flame into which water samples were aspirated as the absorber, and several different multielement sources.

Flames were generated with various standard burners as used in atomic absorption analysis or flame photometry, and different fuel-oxidizer and aspiration rates were tried. Absorption was readily obtained in the flame from sodium and mercury vapor lamps.

Initially, an ordinary d. c. arc charged with multielement powder was tried as the line source. An arc was chosen because of its low cost, simplicity, and flexibility. Negligible absorption was obtained because of thermal (Doppler) line-broadening in this source. Unsuccessful attempts were then made to reduce the line temperature by modifying the source powder, excitation conditions, source atmosphere (vacuum) and selecting lower temperature portions of the arc column. Figure 2 illustrates the relative intensity (temperature) variations measured for representative

Figure 1

DOUBLE - BEAM OPTICAL PATH

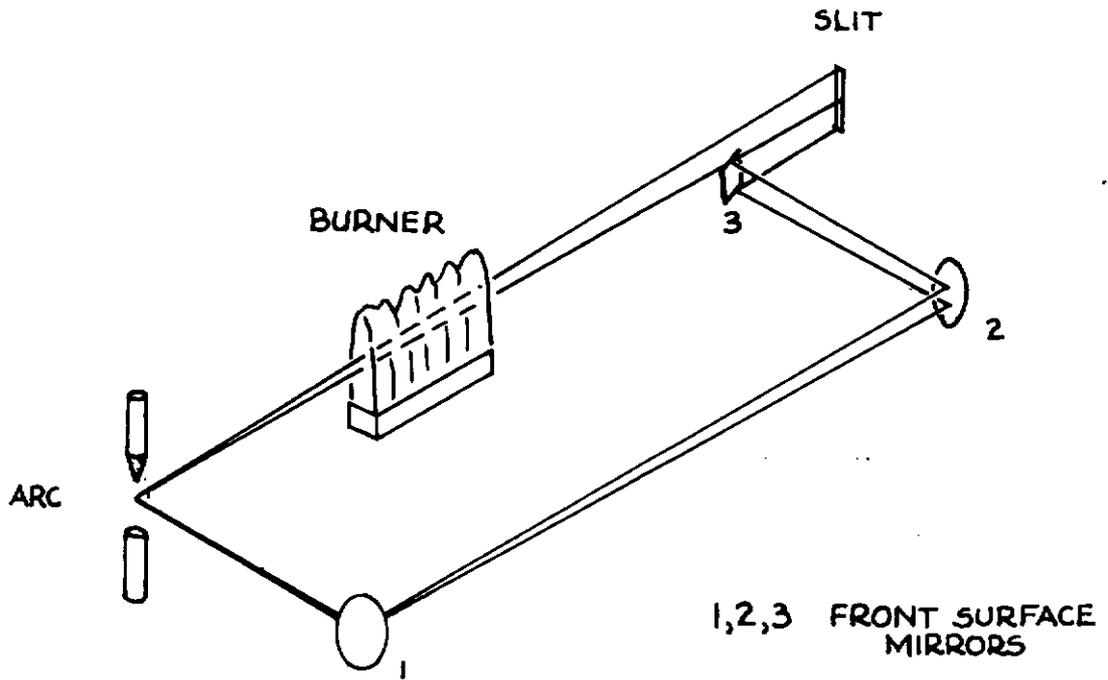
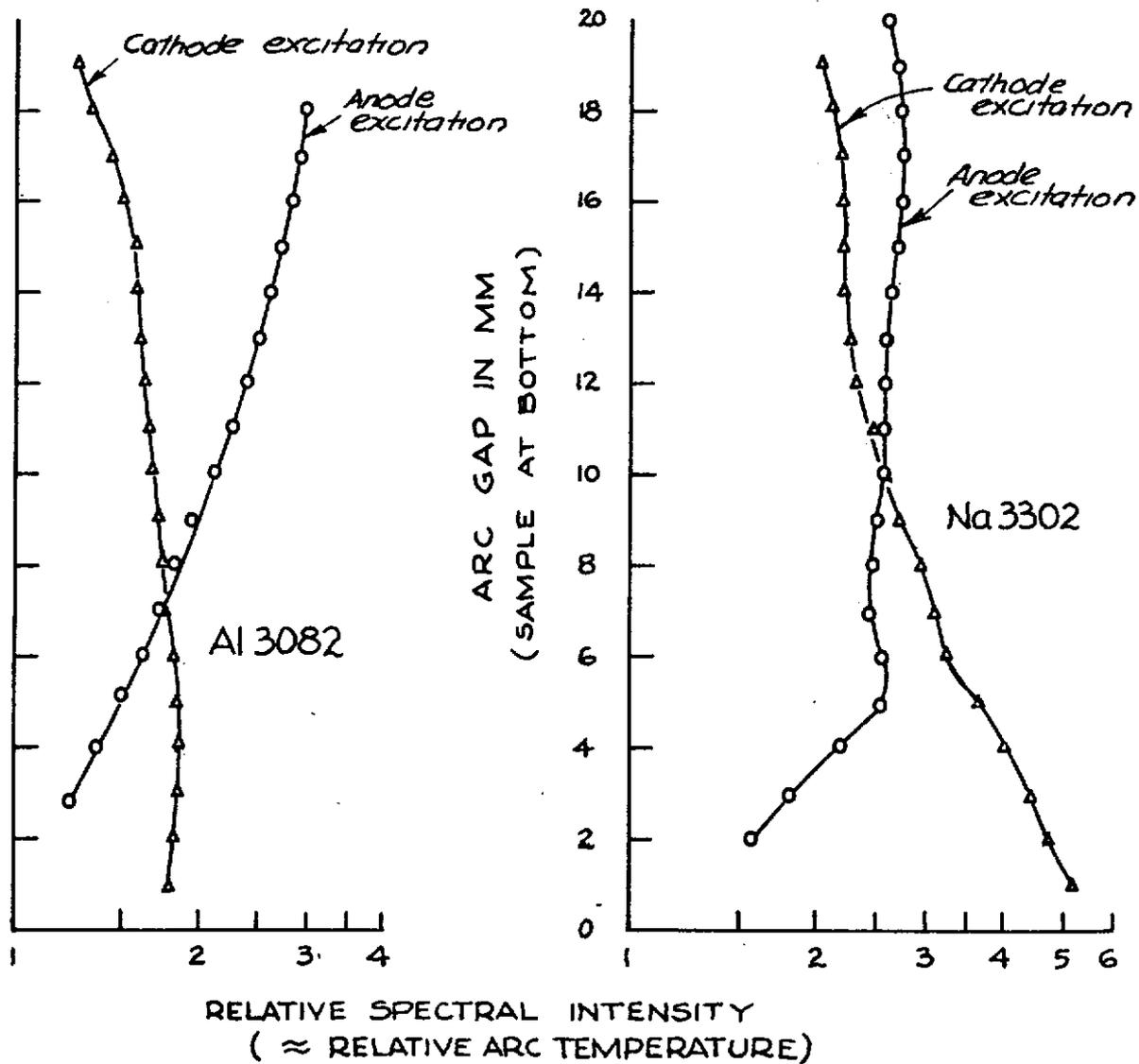


FIGURE 2



LONGITUDINAL VARIATION OF SPECTRAL LINE INTENSITY AS A FUNCTION OF EXCITATION POLARITY FOR A REFRACTORY ELEMENT (ALUMINUM) AND A VOLATILE ELEMENT (SODIUM)

elements in the latter trial. Specific experiments performed together with pertinent remarks are given below:

1. Variations of exposure time, arc gap, slit width, amperage, sector: Low amperage (4-5 amps) and long exposure (30-90 secs), short gap (3-5 mm), and sectoring (passing  $1/8$ - $1/32$  of incident light) yielded best results. Note that all settings are in the direction of reduced spectral line intensities.

2. Use of decaying d. c. arc: Difficult to time, no noticeable improvement.

3. Arc in vacuum (9 amps, 20 secs): Reduced line intensity and generated a cooler line since reversal-sensitive lines were not reversed in reference beam. No apparent absorption in analytical path, however.

4. Arc in flame (direct excitation): Only "uniarc" will run in the flame of a total consumption burner and only cyanogen bands were excited. A second series of unsuccessful experiments involved the use of discharge (Geissler) tubes into which water vapor could be introduced as shown in Figure 3. This type of source holds some promise, but adequate sensitivity could not be obtained with the power supply used.

Finally, the adequacy of direct arc-excitation of water samples was reviewed to determine the utility of d. c. arc spectrography in water analysis applications. The results of measurements for Mg, Fe, Ni, Al, Cu, and Si confirm the known detection levels as shown in Table 1. This analytical method is very fast and cheap and could well be applied in reconnaissance studies. In operation, spectrographically pure electrodes are simply soaked in water, allowed to dry, and arced at 6 amps for 40 secs. Figure 4 shows the results of a field test.

Figure 3

DISCHARGE TUBES

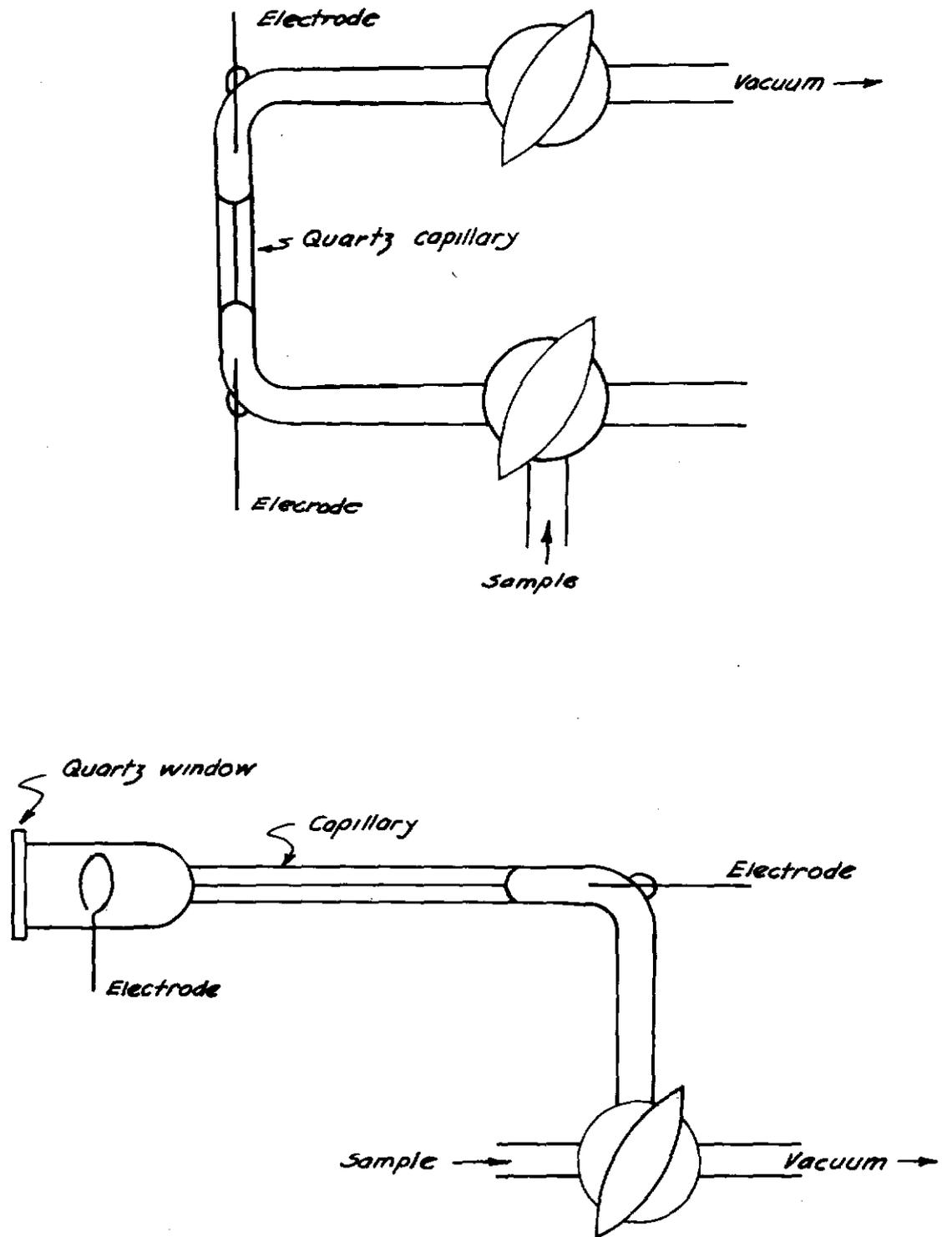


TABLE I

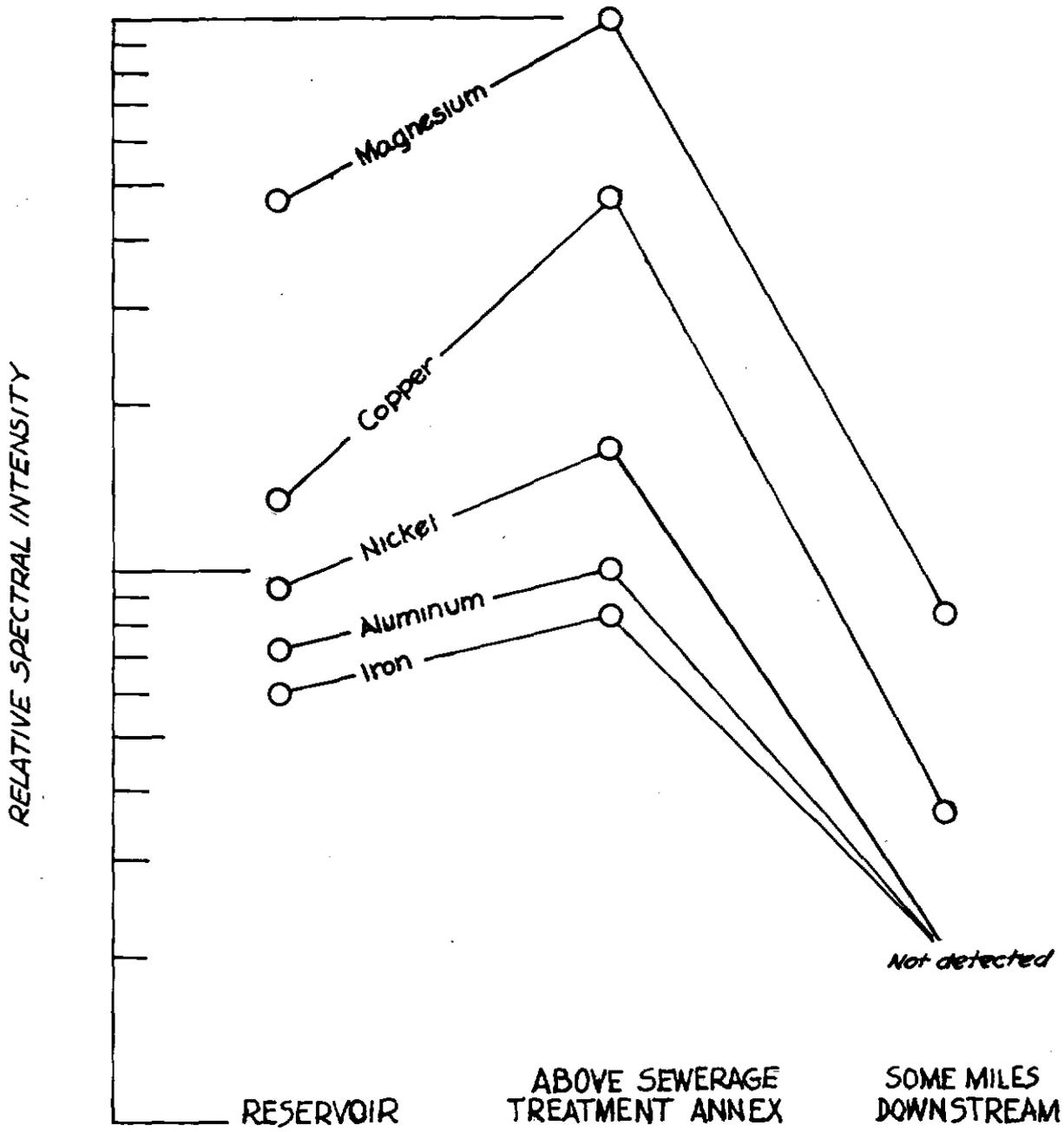
RANGE OF MINIMUM DETECTION, ppm

1 ppm		10 ppm		100 ppm	
	barium		aluminum		arsenic
	calcium		antimony		cerium
	chromium		beryllium		cesium
	cobalt		bismuth		gadolinium
	copper		boron		hafnium
	gallium		cadmium		iridium
	indium		germanium		mercury
	lithium		gold		osmium
	magnesium		iron		phosphorus
	manganese		lanthanum		potassium
	molybdenum		lead		rhenium
	scandium		neodymium		tellurium
	silver		nickel		thorium
	sodium		niobium		uranium
	strontium		palladium		zinc
	thallium		platinum		
	vanadium		rhodium		
			rubidium		
			ruthenium		
			silicon		
			tin		
			titanium		
			wolfram		
			yttrium		
			zirconium		

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Figure 4

FIELD TEST RESULTS



## CHAPTER III

### CONCLUSIONS

The investigations conducted have not contributed any new analytical procedures for the determination of cations in water. The double-beam, however, is demonstrated to be a simple and functional arrangement which may be incorporated into other analytical systems.