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**A RAPID METHOD FOR ARSENIC ANALYSIS
OF
SOIL AND WATER BY ATOMIC ABSORPTION**

**ASSESSMENTS BRANCH
NONEXPLOSIVE MUNITIONS DIVISION**

TECHNICAL REPORT AFATL-TR-70-107

OCTOBER 1970

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AIR FORCE ARMAMENT LABORATORY

AIR FORCE SYSTEMS COMMAND • UNITED STATES AIR FORCE

EGLIN AIR FORCE BASE, FLORIDA

**A Rapid Method for Arsenic Analysis
of
Soil and Water by Atomic Absorption**

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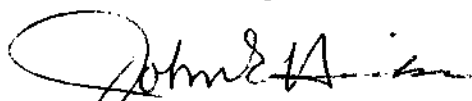
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FOREWORD

The active Air Force project directly related to the information discussed in this report is Exploratory Development Project 5066. Further information or comments on any portion of this report may be referred to (DLNA), Eglin Air Force Base, Florida 32542.

The statistical analyses performed by Booz-Allen Applied Research are gratefully acknowledged.

This report has been reviewed and is approved.

A handwritten signature in dark ink, appearing to read "John E. Hicks", is written over the printed name.

JOHN E. HICKS, Colonel, USAF
Chief, Nonexplosive Munitions Division

ABSTRACT

One of the major herbicides used in military programs is dimethylarsinic acid (cacodylic acid). Its use in conjunction with an Air Force program of testing aerial spray equipment necessitated a rapid and accurate technique for the determination of arsenic in soil and water at concentrations of approximately one part per million. The atomic absorption spectrophotometer appeared to fulfill the instrumentation requirement. Ten-gram soil samples were placed in 250-milliliter Erlenmeyer flasks and extracted with 20 milliliters of extracting solution (0.10N hydrochloric acid (HCl) and 0.05N sulfuric acid (H₂SO₄), and 0.10 grams carbon black). The samples were mechanically shaken for five minutes and filtered. It was found that up to 93.4 percent of the arsenic could be recovered from soils to which known amounts of organic arsenic were added. Water samples were analyzed. By concentrating the samples by a factor of 10 or more, arsenic could be determined down to 0.05 part per million. Comparison of standards made from inorganic arsenic **dissolved** in dilute nitric acid with standards prepared from cacodylic acid and with sodium cacodylate-cacodylic acid showed excellent correlation in the range from 0.5 to 40 parts per million. The atomic absorption technique did not differentiate between organic and inorganic arsenic.

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SECTION I
INTRODUCTION

For many years, compounds containing arsenic have been used extensively as insecticides, herbicides, soil sterilants, and silvicides. Initially, the inorganic forms of arsenic predominated, with such compounds as lead arsenate, calcium arsenate, and Paris green being used effectively against insects, while arsenic trioxide, sodium arsenite, and sodium arsenate were effective in weed control programs. More recently, organic forms of arsenic have been accepted because of their effectiveness as desiccants and also as herbicides in cotton, orchards, and turf. One of the major defoliants used by the military is agent Blue, an organic arsenical containing dimethylarsinic acid (cacodylic acid). Despite the widespread use of the different arsenicals, the fate of arsenic in soil is not well known because it is a difficult element to qualitatively and quantitatively identify. A technique for the rapid extraction and quantitation of arsenic in soil would be of value.

In this research, arsenic levels were quantitatively determined in soil and water after spraying with agent Blue (Table I); and the reliability of the extraction procedures was evaluated. A fast, relatively

TABLE I. FORMULATION DESCRIPTION OF THE MILITARY HERBICIDE BLUE (PHYTAR 560 G)	
Constituent	Percent
Cacodylic Acid	4.7
Sodium Cacodylate	26.4
Surfactant	3.4
Sodium Chloride	5.5
Water	59.5
Antifoam	0.5
Total Organic Arsenic	15.4

accurate method for determining arsenic levels as low as one ppm from agent Blue was required. The atomic absorption spectrophotometer, which is capable of reading arsenic at a wavelength of 1937 angstroms, appeared to fulfill the instrumentation requirement.

In previous experiments, arsenic in organic arsenates at the technical and formulation level has been determined by a fusion procedure⁽¹⁾ in which the arsonate was decomposed to pentavalent arsenic from a potassium bromate-nitric acid solution; further study was suggested using this method. Another method determined the arsenic residues in tomato plants and soil samples by activation analysis⁽²⁾ and is sensitive to 0.2 ppm arsenic in less than gram quantities of material; however, the equipment required is quite expensive and is not available in many laboratories. The Gutzeit method⁽³⁾ and the colorimetric method⁽⁴⁾ require special apparatus and involve hazardous extraction procedures; also, certain naturally occurring compounds interfere. A dry ashing method has been used in analyzing poultry tissue⁽⁵⁾, and the arsenic was determined colorimetrically. A nitrogen (entrained air) - hydrogen flame has been used for atomic absorption analysis⁽⁶⁾ of arsenic, and another method uses oxyacetylene flames⁽⁷⁾. Numerous other techniques and methods of analysis for arsenic have been reported (8 to 13); the majority make use of atomic absorption spectrometry. The basic principle of atomic absorption was established in 1860 by Kirchhoff⁽¹⁴⁾. It was not until 1955, however, that Walsh⁽¹⁴⁾ foresaw the analytical potentialities of atomic absorption, and he is credited with the theoretical background of most of the work in this field.

Atomic absorption, like other spectrophotometric methods, is a comparison method of analysis. Determinations are made by comparing samples with three or more standards having the same concentration range. The concentration of the element of interest in a sample is determined by measuring the absorption of radiation in atomic vapor produced from the sample at a wave-length that is specific and characteristic of the element. In operation, a hollow cathode light beam is passed through a flame. Samples are aspirated into the flame, where molecules are dissociated into atomic form. While in the flame, most atoms remain in the ground, or neutral, state and, therefore, are capable of absorbing the hollow cathode radiation, only atoms of the element of interest absorb, and the amount of radiation absorbed is proportional to the concentration of the element of interest in the sample. After passing through the flame, the hollow cathode beam passes into a spectrophotometer to be measured; a permanent record of the measurement can be provided by an attached recorder.

SECTION II

TEST PROCEDURES

1. PREPARATION OF SOIL SAMPLES

Soil samples were prepared with known concentrations of Blue (penta-valent arsenic as cacodylic acid and sodium cacodylate). The soil was Gulf Coast Flatland having a pH of 5.0 to 5.5 and consisting of 93.9-percent sand, 3.7-percent silt, 2.4-percent clay, and 0.3-percent organic matter. Blue was applied as a liquid in 50 milliliters of water (plus a 25-milliliter rinse) with a hand atomizer at rates of 0, 1, 2, 3, 5, 10, 20, 30, 35, and 40 ppm arsenic. Each of the treated soils was uniformly mixed, and after one week, two 10-gram samples were taken. The 10-gram soil samples were placed in 250-milliliter Erlenmeyer flasks with 20 milliliters of extracting solution consisting of 2.4 milliliters of concentrated H_2SO_4 (reagent grade); 14.6 milliliters of concentrated HCl (reagent grade); 18 grams of carbon (decolorizing neutral); and 1.790 milliliters of distilled water. The solutions were shaken for five minutes on a reciprocating shaker set at 175 excursions per minute, filtered through Whatman No. 1 filter paper (11.0 centimeters) and then aspirated in duplicate in the atomic absorption instrument.

2. PREPARATION OF WATER SAMPLES

To evaluate the accuracy of analyzing water samples for low amounts of arsenic, standard samples were prepared and concentrated from 100 milliliters to 5 milliliters. One drop of concentrated nitric acid (reagent grade) was added to 100 milliliters of each of ten samples (in duplicate) containing 0, 0.01, 0.02, 0.03, 0.04, 0.05, 0.10, 0.20, 0.50, and 1.00 ppm arsenic as cacodylic acid. The samples were then evaporated to slightly less than 5 milliliters, and one drop of an acid mixture (73 milliliters HCl , 12 milliliters H_2SO_4 and 15 milliliters distilled water) was added to make the solution approximately 0.05N HCl and 0.25N H_2SO_4 . All samples were diluted to 5 milliliters. Samples and standards were aspirated in the atomic absorption instrument.

A working calibration curve (or standard curve) was prepared in which peak height was plotted against arsenic concentration. The concentration of arsenic in the soil and water samples was determined by comparing the peak heights to the standard curve.

3. CHEMICAL STANDARDS AND OPERATING CONDITIONS

Inorganic arsenic standards were prepared in 100-milliliter volumetric flasks from a 1000-ppm arsenic atomic absorption standard by making a 100-ppm arsenic standard and then taking various aliquots from this and

diluting to 100 milliliters with distilled water. Ten standards were prepared, ranging from 0.5 to 100 ppm inorganic arsenic.

Other standards were prepared by diluting 6.4935 grams of Blue to 1000 milliliters with distilled water, and then, ten different concentrations were made by taking aliquots and diluting to 100 milliliters in volumetric flasks. Standards containing only cacodylic acid were prepared from a solution containing 1.8422 grams of cacodylic acid (purified grade) in 1000 milliliters of distilled water.

The atomic absorption instrument was operated under the following conditions: wavelength setting, 1937 angstroms; lamp (JA45315) current; 15 milliamperes; burner, tri-flame type; fuel, hydrogen at 13 SCFH; oxidant, compressed air at 19 SCFH; monochromator with entrance slit of 100 microns and exit slit of 150 microns. The unit was operated in conjunction with a chart recorder at a range of 0 to 10 millivolts.

SECTION III

TEST RESULTS AND DISCUSSION

1. ASSESSMENT OF METHOD

The dilute-acid, cold-extraction technique and subsequent analysis by atomic absorption spectrophotometry has been found to be a rapid, reliable method for determining arsenic in a Gulf Coast Flatland soil. Extractable arsenic from the organic arsenicals (cacodylic acid and sodium cacodylate) and from inorganic arsenicals can be determined. However, this technique does not differentiate between an organic arsenic (e.g., cacodylic acid) and an inorganic form, or arsenic (e.g., arsenic trioxide).

A typical standard curve for the determination of arsenic is shown in Figure 1. This curve was obtained by analysis of inorganic arsenic standard with the range shown for four determinations. The peak height (in centimeters) for each concentration of arsenic was obtained from the chart recorder attached to the instrument. From a previous study (unpublished data) on the linear calibration of the atomic absorption spectrophotometer, it was found that peak height readings were preferable to peak area readings because: (1) the necessary relationship between height and level of arsenic was present without quadratic influences, and (2) the confidence intervals computed for peak height versus each amount of arsenic were one-third the width of the intervals computed for the peak area readings. In describing their relationship, an equation for predicting the concentration of arsenic can be obtained, given peak height. The equation for the data in Figure 1 is:

$$Y = -0.045 + 0.481 X$$

Where Y is the peak height in centimeters, and

X is the concentration of arsenic in parts per million

For this equation, a correlation coefficient of 0.999 was found. Because of the variability inherent in the instrument, in fuel-oxidant parameters, and in the extraction technique, it is necessary that standard curves be prepared for each new series of analysis and for each soil type.

From soil samples with known amounts of arsenic (as Blue) added in the laboratory, it was possible to recover an average of 93.4 percent of the arsenic. Figure 2 shows the comparison of arsenic recovered versus original arsenic added to the Gulf Coast Flatland soil. The data represents four determinations. The 95-percent confidence intervals from these determinations illustrated the importance of limiting the effective

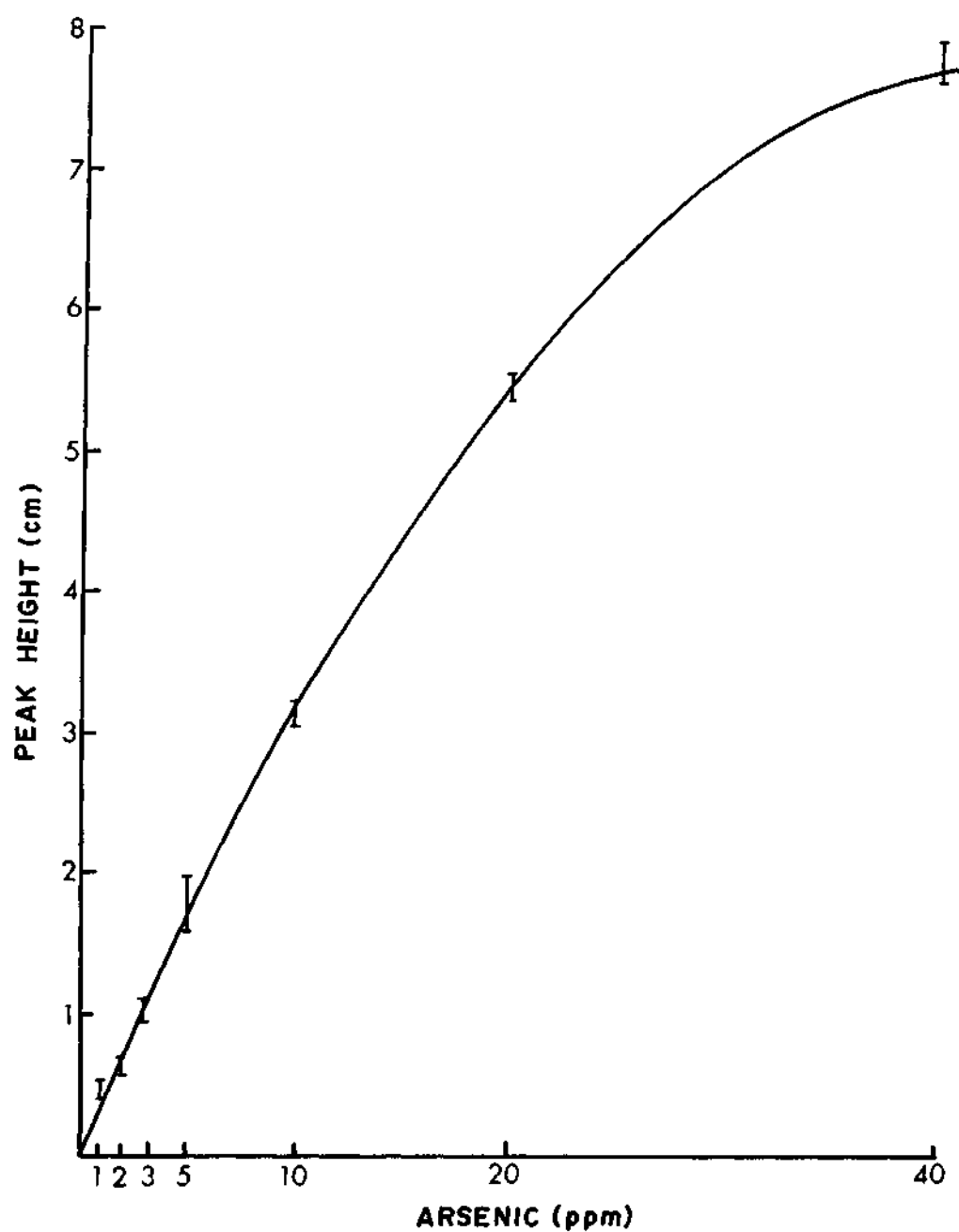


Figure 1. A typical standard curve for determining arsenic

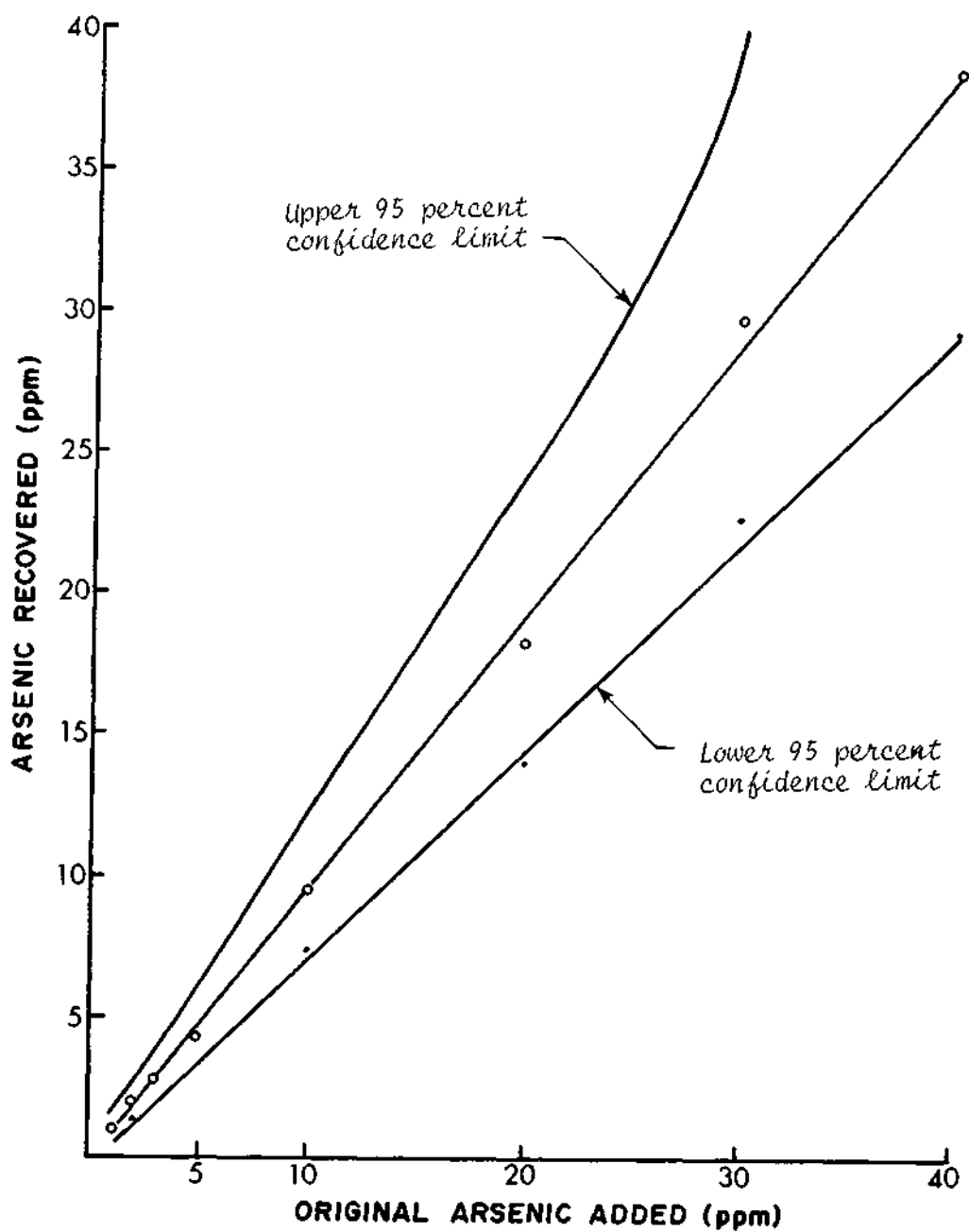


Figure 2. Comparison of arsenic recovered versus original arsenic added as Blue

operating range to less than 20 ppm arsenic. The rapid expansion of the confidence limits resulted from the method by which the limits were computed and in the increasing size of the standard deviation as concentration of arsenic increased; i.e., as the concentration increases, the sensitivity of the instrument to differentiate concentrations greater than 20 ppm decreases. The lower detection limit for soil samples was 0.5 ppm arsenic.

Soil samples which contain larger amounts of organic matter and/or clay particles would tend to bind the arsenic more tightly than would the Gulf Coast Flatland soil and low-percentage results would be obtained. Acid digestion may be required to free tightly bound arsenic.

Table II illustrates the detection capability of atomic absorption for arsenic in water. By concentrating the water samples, the error attributable to procedure is increased. The more dilute the sample, the greater the need for concentrating the solution, and hence, the greater the error in detection. The acidic nature of the final solution influences the background reading of the instrument; thus, sensitivity of arsenic is limited to a concentration of 0.03 ppm as indicated by the 95-percent confidence limits in Table II.

TABLE II. COMPARISON OF ARSENIC ADDED (AS CACODYLIC ACID) TO WATER SAMPLES VERSUS ARSENIC DETECTED BY ATOMIC ABSORPTION ANALYSIS

Original Arsenic Concentration ^a , ppm	Arsenic Detected ^b , ppm	95-Percent Confidence Limits	
		Lower	Upper
0.00	0.027	0.023	0.032
0.01	0.029	0.024	0.034
0.02	0.030	0.025	0.034
0.03	0.036	0.031	0.042
0.04	0.046	0.040	0.052
0.05	0.055	0.048	0.062
0.10	0.100	0.090	0.110
0.20	0.189	0.172	0.208
0.50	0.496	0.451	0.558
1.00	0.972	0.867	1.095
^a Samples were concentrated to 1/20th of the original volume.			
^b Four determinations per concentration.			

Table III is a comparison by atomic absorption analysis of three different arsenic formulations in water. No differences could be detected in the response of the instrument (and/or methodology) to arsenic trioxide, cacodylic acid, or Blue. The organic arsenic solutions gave peak heights which correlated with those of the inorganic arsenic standards.

TABLE III. A COMPARISON OF PEAK HEIGHTS OF STANDARD ARSENIC SOLUTIONS PREPARED FROM ARSENIC TRIOXIDE, CACODYLIC ACID, AND BLUE

Arsenic Concentration ppm	Average Peak Heights ^a , centimeters		
	Arsenic Trioxide	Cacodylic Acid	Blue
0.5	0.40	0.35	0.39
1.0	0.56	0.53	0.62
2.0	1.05	1.07	1.06
3.0	1.28	1.36	1.37
5.0	1.75	1.87	1.80
10.0	3.12	3.18	3.06
20.0	5.60	5.67	5.40
40.0	9.13	9.38	9.28
60.0	12.11	11.99	11.86
100.0	15.24	14.89	14.70
^a Average of two determinations.			

2. INTERFERENCES WITH ANALYTICAL TECHNIQUE

Various concentrations of hydrochloric acid and sulfuric acid were added to soil and water samples containing known amounts of arsenic to determine what effect the variations would have on the results. Up to three times the concentration of acid used in the analyses had no significant effect; however, above that amount (for low concentrations of arsenic), the results indicated a greater amount of arsenic than was actually present. For arsenic concentrations above 10 ppm, slightly larger amounts of acid could be added without serious errors.

Concentrations of salt (sodium chloride) from 0.10 to 10.0 percent were prepared to determine their effect on arsenic analysis. The solutions were aspirated in the instrument, and the parameters used were the same as those used for analyzing arsenic. Results indicated that 0.5 percent, or more, salt had a significant effect on low-concentration arsenic samples (Table IV). It was not possible to concentrate salt water samples and obtain accurate analyses as the increased salt concentration would cause high results.

TABLE IV. COMPARISON OF PEAK HEIGHTS OF SALT SOLUTIONS WITH ARSENIC CONCENTRATIONS		
Salt Concentration, percent	Peak Height, centimeters	Arsenic, ppm
0.10	No noticeable effect	---
0.25	No noticeable effect	---
0.50	0.38	0.5
1.00	0.45	0.7
2.50	0.72	1.3
5.00	0.93	1.8
10.00	1.33	2.5

When Blue is used in testing and calibrating aerial spray equipment, a dye (methylene blue) is added to the solution. This dye interfered with the determination of arsenic. In order to remove the dye in samples analyzed, six drops of concentrated nitric acid were added to a 5 to 10 milliliter aliquot of Blue in a beaker. The samples were evaporated to 1 milliliter or less to insure disappearance by oxidation of the dye, then diluted to 5 milliliters or more depending on the amount of arsenic present. There was no longer any interference by the dye in samples treated as indicated.

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