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ATOMIC ABSORPTION AND FLAME EMISSION SPECTROSCOPY
ANALYSES OF BASALT CORE SAMPLES AND WATER SAMPLES
FROM A DEEP WELL

By

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ABSTRACT

Atomic absorption was found to be a fast and accurate method for determining the major metal oxides and several trace elements in basaltic rock flows. Rock samples were fused with lithium metaborate, then dissolved with dilute hydrochloric acid. Trace elements as well as the major constituents were easily analyzed from the fusion solution. Experimentation also showed that a treatment of this solution with ion exchange followed by a dithizone-ethyl propionate extraction enabled a 15-fold improvement in the detection limits of Pb, Co, Cu, and Ni.

Water samples were successfully analyzed by atomic absorption and flame emission for 19 elements. Determinations were made directly or from dilutions of the samples. Several trace elements of very low concentrations were chelated with ammonium pyrrolidine dithiocarbamate and extracted into methyl isobutyl ketone. This extraction effectively improved detection limits of the elements up to 30-fold.

ATOMIC ABSORPTION AND FLAME EMISSION SPECTROSCOPY
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FROM A DEEP WELL

INTRODUCTION

In April, 1969, Atlantic Richfield Hanford Company (ARHCO) began drilling an exploratory deep well near the 200-East Area at the U.S. Atomic Energy Commission's Hanford site. The purpose of the well was to become familiar with the composition patterns of the various basaltic flows which are present in this area. This knowledge would, in turn, enable comparison of the basaltic flows with earlier exploratory wells.

A method which would provide fast, reliable results for the major and minor elements was needed for analysis of both well water and basalt core samples. Atomic absorption and flame emission have recently proven capable of similar tasks. In the past five years, atomic absorption in particular has proved to be highly sensitive and relatively free from interferences even in complex solutions. In addition, both techniques are rapid--and at the same time produce high-precision results. For these reasons, atomic absorption and flame emission spectroscopy were chosen as tools for the task.

SUMMARY AND CONCLUSIONS

An analytical scheme was developed which enabled dissolution and analysis of 18 elements at both major and trace concentrations in the basaltic core samples. In addition, a scheme of analysis for water samples was prepared which allowed determination of 19 elements--several of which were in the parts per billion range.

The results showed that atomic absorption and flame emission may be used exclusively to provide reliable results on both water and basalt core samples for the major constituent elements and many trace elements. Sample preparation is minimal; interferences are rare, especially with the atomic absorption mode. Analytical range is very broad and precision is generally good.

EXPERIMENTAL

APPARATUS AND REAGENTS

A Varian Techtron AA5 Atomic Absorption Unit was used for all of the analyses. Results were recorded from a digital indicator either in absorbance or directly in concentration.

Reagent grades of lithium metaborate (LiBO_2), methyl isobutyl ketone (MIBK), ammonium pyrrolidine dithiocarbamate (APDC), dithizone, ethyl propionate, and lanthanum nitrate (99.997 percent) were used.

WATER SAMPLES

After their removal from the ground, water samples were immediately acidified with 10 milliliters of concentrated hydrochloric acid per gallon of water. This prevented precipitation of ferric hydroxide.

For several elements the sample was analyzed directly. When dilutions were required, they were made into small, plastic, 15-ml screw-cap vials containing the proper diluent. Sodium, magnesium, and calcium were found to be leached from glass vials into the diluent on long periods of standing.

The analytical scheme used for water samples is described in Table I, page 3. This table summarizes dilutions and instrumental parameters.

TABLE I
SUMMARY OF ANALYTICAL AND INSTRUMENTAL
PARAMETERS FOR AA MEASUREMENTS ON WATER SAMPLES

Element	Wavelength Angstrom	Mode*	Flame**	Dilution
Iron	2483	AA	Air-Acet	None or APDC
Zinc	2139	AA	Air-Acet	None or APDC
Manganese	2795	AA	Air-Acet	None
Chromium	3579	AA	Air-Acet	None
Potassium	7655	FE	Air-Acet	None
Lithium	6708	FE	Air-Acet	None
Rubidium	7800	FE	Air-Acet	0.5% K solution
Sodium	5890	FE	Air-Acet	100-fold water
Calcium	4226	AA	Air-Acet	10-fold 1% La solution
Magnesium	2852	AA	Air-Acet	10-fold 1% La solution
Strontium	4607	AA	Air-Acet	1.25-fold 5% La solution
Aluminum	3093	AA	N ₂ O-Acet	0.15% K solution
Barium	5535	AA	N ₂ O-Acet	0.15% K solution
Silicon dioxide	2516	AA	N ₂ O-Acet	0.15% K solution
Cobalt	2407	AA	Air-Acet	APDC-MIBK 10:1 A/O
Copper	3247	AA	Air-Acet	APDC-MIBK 10:1 A/O
Cadmium	2288	AA	Air-Acet	APDC-MIBK 10:1 A/O
Nickel	2320	AA	Air-Acet	APDC-MIBK 10:1 A/O
Lead	2170	AA	Air-Acet	APDC-MIBK 10:1 A/O

* AA = atomic absorption; FE = flame emission.

** Air-Acet = air acetylene; N₂O-Acet = nitrous oxide acetylene.

Iron, Zinc, Manganese, and Chromium

Concentrations of Fe, Zn, Mn, and Cr were such that they could be directly determined. These samples were compared with simple aqueous standards by atomic absorption using an air-acetylene flame. Where iron or zinc concentrations were very low, analyses were made after chelation with APDC and extraction into MIBK. This procedure is described further on page 5. Manganese and chromium were usually found in very low concentrations, but could not be determined by the organic extraction as the presence of other metals (especially iron) tended to inhibit their extraction.

Lithium, Rubidium, and Potassium

Lithium, rubidium, and potassium were analyzed directly by using flame emission. In all cases an air-acetylene flame

was used with a 10-cm burner. For lithium analysis, sodium was added to the standards at a concentration equal to that present in the sample. Slight interference was observed from the presence of large amounts of sodium. Rubidium sensitivity was enhanced about 5-fold by the presence of excess potassium, which permits an increase in neutral Rb atoms in the flame.⁽¹⁾ Consequently, all samples to be analyzed for rubidium were spiked with enough potassium to provide an 0.5 percent potassium solution. Potassium-containing standards and a potassium blank were prepared to compare with the samples.

Magnesium, Calcium, and Strontium

Magnesium and calcium were determined by atomic absorption after diluting the sample 10-fold into a 1 percent lanthanum solution.

In all cases lanthanum is present to act as a releasing agent for Ca, Sr, and Mg from the inhibiting effects caused by interfering components.⁽²⁾

Sodium

Sodium concentration in the water samples was generally very high. In most cases a 100-fold dilution was made and flame emission was used for the determination.

Aluminum, Barium, and Silicon

These elements were determined by atomic absorption after addition of sufficient potassium to produce an 0.15 percent potassium solution. A nitrous oxide-acetylene flame was used. The potassium is present to prevent ionization interferences⁽³⁾ in the cases of aluminum and barium.

Cobalt, Copper, Cadmium, Lead, Iron, Zinc, and Nickel

These elements were chelated with ammonium pyrrolidine dithiocarbamate (APDC) and all were quantitatively extracted at one time into methyl isobutyl ketone (MIBK). The elements were determined using atomic absorption; the organic phase was aspirated into an air-acetylene flame. Using an aqueous-to-organic ratio of 10 to 1, the detection limit was increased about 30-fold with this method. The absorbances were compared against reference standards prepared by the same method as the samples. The reference standards consisted of aqueous solutions synthesized to contain 10 times the typical sample concentrations of the major metals plus known amounts of various trace elements. When it was necessary to run a sample, a portion of the standards were diluted 10-fold with demineralized water before being chelated and extracted into MIBK. Two reference standards were used; and, after 10-fold dilution, contained the following: 150 ppm sodium, 3 ppm calcium, 0.25 ppm magnesium, 0.1 and 0.2 ppm iron, 0.012 and 0.025 ppm zinc, 0.1 and 0.2 ppm nickel, 0.1 and 0.2 lead, 0.1 and 0.2 ppm cobalt, 0.05 and 0.1 ppm copper, and 0.012 and 0.025 ppm cadmium.

In a typical analysis of well water using organic extraction, 500 ml of well water sample were poured into a clean, one-liter separatory funnel and 10 ml of fresh 2 percent APDC solution were added. The solution was shaken for 10 seconds, after which 50 ml of MIBK were added. The solution was then shaken for 10 minutes to allow extraction of the chelates into the MIBK layer. The reference standards were diluted 10-fold from 50 milliliters with demineralized water, and treated as above. The organic MIBK layer was analyzed for the various metals using as a blank pure MIBK which was previously pre-equilibrated with water.

In those samples where iron was quite high (0.5 ppm or

more), it was necessary to perform a second extraction on the aqueous. The large amount of iron interfered with the chelation of some of the other trace elements. If the first extraction was visually found to contain large amounts of black-colored iron chelate, 10 ml more of 2 percent APDC were added to the aqueous and the solution was extracted with 25 ml of MIBK. The first and second extracts were then combined for analysis.

ROCK SAMPLES

Dissolution

The crushed core samples were prepared for analysis by fusion with lithium metaborate (LiBO_2). This technique, described by Yule and Swanson,⁽⁴⁾ proved to be short, provided high precision, and allowed little chance for introduction of contamination.

In this dissolution procedure, 400 milligrams of 100 mesh crushed sample were heated at 800 C for 10 minutes to drive off the volatiles. After cooling and reweighing, the percent volatiles was recorded. The sample was then mixed in a platinum crucible with 2 grams of LiBO_2 . This mixture was fused for 15 minutes in a furnace at 1000 C. The crucible was removed from the furnace and placed into a 400-ml Pyrex beaker. Thirty seconds after removal from the furnace, approximately 100 ml of hot, 10 percent hydrochloric acid solution were poured into the crucible and the solution was stirred and heated until the fusion melt was dissolved. Additional 10 percent HCl was added if crystallization occurred. A watch glass was kept over the beaker at all times to prevent evaporation. After cooling, the solution was brought to 200 ml total volume with 10 percent HCl. All dilutions were made from the fused solution into 15-ml glass or plastic vials.

The United States Geological Survey (USGS) BCR-1 and

USGS W-1 crushed rock samples were used as control references for basalt analyses. These samples were dissolved and analyzed by the same procedure as the well core samples.

All basalt analyses were made by reading five basic dilutions of the fusion solutions against standards. For several elements it was necessary to read the fusion solution directly. The analytical scheme is presented below. Table II summarizes dilutions and instrumental parameters.

TABLE II
ANALYTICAL SUMMARY FOR FUSION SOLUTION
OF BASALT ROCK SAMPLES

Element	Wavelength Angstrom	Mode*	Flame**	Dilution
Aluminum	3093	AA	N ₂ O-Acet	1) 1-ml sample and 500 λ 10% KCl + 10 ml H ₂ O.
Silicon	2516	AA	N ₂ O-Acet	
Calcium	4226	AA	Air-Acet	2) 50 λ sample + 10 ml 1% La.
Magnesium	2852	AA	Air-Acet	
Sodium	5890	AA	Air-Acet	3) 250 λ sample + 10 ml H ₂ O.
Potassium	7665	AA	Air-Acet	
Iron	2483	AA	Air-Acet	
Strontium	4607	AA	N ₂ O-Acet	4) 4-ml sample + 1 ml 5% La.
Manganese	2795	AA	Air-Acet	5) Sample read direct using blank and standards with matrix similar to samples.
Nickel	2320	AA	Air-Acet	
Cobalt	2407	AA	Air-Acet	
Lead	2170	AA	Air-Acet	
Copper	3247	AA	Air-Acet	
Chromium	3579	AA	Air-Acet	
Zinc	2139	AA	Air-Acet	
Barium	5535	AA	N ₂ O-Acet	6) 1 ml 10% KCl + 10-ml sample. Potassium-containing blank necessary for Rb.
Rubidium	7800	AA	Air-Acet	
Titanium	3643	AA	N ₂ O-Acet	

* AA = atomic absorption.

** Air-Acet = air acetylene; N₂O-Acet = nitrous oxide-acetylene.

Aluminum and Silicon

One ml of sample solution and 500 μ l of 10 percent KCl were added to 10 ml of deionized water and compared with

aqueous silicon and aluminum standards using a nitrous oxide-acetylene flame.

Calcium and Magnesium

Fifty μ l of sample were added to 10 ml of 1 percent lanthanum solution. The sample was compared with appropriate standards using an air-acetylene flame. Occasionally with this dilution the magnesium concentration was so high that it necessitated turning the burner several degrees to reduce the absorbance. Larger-concentration Mg standards were also used in this case so that the standard concentration would bracket the samples. In all cases a lanthanum blank was used.

Sodium, Potassium, and Iron

Two hundred fifty μ l of sample were added to 10 ml of deionized water in a plastic vial. To prevent cross-contamination, the dilution was first analyzed for sodium--then for potassium and iron. Either flame emission or absorption may be used for sodium; only absorption is used for potassium and iron. For sodium and potassium analyses, a blank and standards were prepared containing the same amount of LiBO_2 as would be present in the samples. This enables correction for the slight sodium and potassium contamination found in reagent grade LiBO_2 . Sodium, like magnesium, was often found to be excessively high in concentration for this dilution size--thus the optical path through the burner was shortened by turning the burner several degrees to reduce the absorbance.

Strontium

Four ml of sample were mixed with 1 ml of 5 percent lanthanum solution and compared with appropriate standards using an N_2O -acetylene flame.

Manganese, Nickel, Cobalt, Lead, Copper, Chromium, and Zinc

These elements were determined by directly aspirating the sample solution into an air-acetylene flame. They were compared with a specially-prepared trace element standard. This standard contained all of the trace elements in question in a solution matrix similar to an actual sample solution. A blank solution was prepared which was also similar to a typical sample, but contained no trace elements. The composition of the blank was 500 ppm silicon, 250 ppm iron, 130 ppm aluminum, 50 ppm sodium, 120 ppm calcium, 30 ppm titanium, 10 g/liter lithium metaborate, and 1.2 molar HCl (10 percent). The trace element standard was of the same composition; but in addition contained 0.5 ppm Zn, 2.5 ppm Mn, 0.5 ppm Cu, 0.5 ppm Ni, 0.5 ppm Cr, 0.5 ppm Pb, and 0.5 ppm Co. It was found that the special blank matrix was necessary because of a slight absorbance that occurred even in the absence of the element in question. This slight absorbance is thought to be due to flame interference from the complex, high-salt sample matrix.

Ultra-Low Concentrations of Nickel, Cobalt, Copper, and Lead

Nickel, cobalt, copper, and lead concentrations were often near the detection limit of the instrument. It was found that these elements could be concentrated about 15-fold with limited success, utilizing a method similar to that described by Sachdev and West.⁽⁵⁾ They describe the determination of seven trace metals by extraction of the dithizone chelate into ethyl propionate from aqueous solutions. The use of basaltic rock solution, however, necessitated a preliminary separation of the large amount of iron present from the solution since iron formed a heavy chelate precipitate with dithizone. This tended to interfere with chelation and extraction of the trace elements. Making a solution of 5.5M HCl and passing it through an anion-form ion exchange column solved the problem by

adsorbing the iron and allowing the trace elements to pass.

A column of chloride-form anion exchange resin 3/4 in. by 4 in. was pretreated with 5.5M HCl. Thirty ml of concentrated HCl were mixed with 50 ml of fused sample solution and passed through the column. The effluent was collected and the column was washed with 75 ml of 4M HCl. The effluent was again collected and the combined effluents were added to a beaker. The sample pH was adjusted to 7.5 with concentrated NH_4OH . Ten ml of 0.1 percent dithizone in ethyl propionate solution were shaken with the sample for 5 minutes in a separatory funnel. After allowing the phases to separate, the organic layer was centrifuged. An 0.1 percent ethyl propionate blank was equilibrated by shaking with water at the same aqueous/organic ratio that was used with the sample. The reference standard was prepared by adding 0.2 ppm spikes of Co, Cu, Ni, and Pb to the blank solution described on page 9; with subsequent treatment being the same as the samples. The absorbances of Co, Cu, Ni, and Pb of the sample were compared with the reference standard by aspiration of the organic layers directly into the flame.

Barium, Rubidium, and Titanium

One ml of 10 percent KCl was added to 10 ml of sample. The sample was analyzed for barium and titanium with a nitrous oxide-acetylene flame. Atomic absorption rather than flame emission was used for rubidium analysis because of high spectral interferences encountered on basalt fusion solutions. For all determinations, a blank and standards of typical sample matrix were used similar to that described on page 9. Rubidium sensitivity, as previously noted with flame emission, was found to be highly enhanced by the presence of 0.5 percent potassium. Because a slight absorbance was noted due to the potassium, it was necessary to have an equal amount of potassium in the blank solution.

RESULTS AND DISCUSSION

The concentration range of the various elements analyzed in the Atlantic Richfield Hanford Company water samples is shown in Table III. Also shown is the coefficient of variation for recovery of metals using the APDC-MIBK method. In all cases, the recovery of the metals from the aqueous with 2 percent APDC and MIBK was greater than 90 percent. Accuracy and precision were determined by spike additions to samples.

TABLE III
CONCENTRATION RANGE AND
COEFFICIENTS OF VARIATION OF WATER SAMPLES

<u>Element</u>	<u>Concentration Range</u> <u>ppm</u>	<u>Coefficient</u> <u>of Variation</u>	<u>Level</u> <u>ppm</u>
Magnesium	0.179 - 11.75		
Calcium	1.1 - 20.0		
Sodium	47. - 234.		
Manganese	<0.01 - 0.09		
Chromium	<0.01 - 0.03		
Potassium	3.30 - 7.85		
Silicon dioxide	21. - 109.		
Lithium	0.009 - 0.023		
Rubidium	<0.01 - 0.03		
Aluminum	<0.1		
Strontium	<0.05		
Barium	<0.08		
Iron	0.02 - 2.10	+ 4%	0.075
Zinc	0.003 - 0.35	+ 8%	0.01
Nickel	0.006 - 0.040	+10%	0.075
Lead	<0.01 - 0.05	+ 9%	0.075
Cobalt	<0.002 - 0.030	+ 5%	0.075
Copper	<0.0005 - 0.006	+ 8%	0.05
Cadmium	<0.0005 - 0.009	+ 5%	0.02

Besides the seven elements in water samples that were extracted into the organic (see page 5), it was shown that Sb, Hg, Pd, Ru, and Rh could also be extracted with greater than 90 percent recovery. Although these elements were never

found to be present in the water samples, it is interesting to note that the APDC extraction is effective in quantitatively isolating twelve trace elements with only one extraction.

The W-1 and BCR-1 rock sample major elements results are shown in Table IV, page 13; and are in close accordance with the reported USGS values.^(6, 7) The trace element results compared favorably with the USGS samples when the trace element blank and standards were used as described on page 9. The only serious discrepancy is the unexplainable high results obtained for zinc on the W-1 samples.

In most cases the trace elements in the basaltic rocks were of high enough concentrations to preclude the use of the ethyl propionate extraction method. These elements were also only of limited interest to the geologists. Although the results obtained on the BCR-1 standard were quite consistent, higher than reported values were obtained in the case of copper and nickel. Cobalt and lead values checked well with the BCR-1 reported results. Spiked samples showed >95 percent recovery for 0.1 ppm spikes. The results obtained for the BCR-1 standard using this method are shown in Table V, below.

TABLE V
TRACE ELEMENT RECOVERIES FOR BCR-1 STANDARD
USING DITHIZONE-ETHYL PROPIONATE EXTRACTION

<u>Element</u>	<u>Found Range</u>	<u>Average - Range</u>
Cobalt	35 - 50	36. (29 - 60)
Copper	60 - 75	22.4 (7 - 33)
Nickel	30 - 40	15. (8 - 30)
Lead	17 - 29	18. (5 - 35)

A typical report of results on basaltic flows is shown in Table VI, page 14. Note that Sample 205 was run in duplicate as a check on analytical precision and the results compare well. Note also the interesting variations in some of the major metals reported as oxides.

TABLE IV
RESULTS OF REFERENCE STANDARD ANALYSES

	<u>SiO₂</u> <u>%</u>	<u>Al₂O₃</u> <u>%</u>	<u>Fe₂O₃</u> <u>%</u>	<u>CaO</u> <u>%</u>	<u>MgO</u> <u>%</u>	<u>Na₂O</u> <u>%</u>	<u>K₂O</u> <u>%</u>	<u>TiO₂</u> <u>%</u>	<u>MnO</u> <u>%</u>	<u>SrO</u> <u>%</u>
<u>USGS BCR-1</u>										
Obtained, avg	55.62	13.73	13.60	7.05	3.48	3.37	1.76	2.18	0.18	0.036
Std deviation	1.25	0.15	0.22	0.19	0.14	0.09	0.12	0.07	0.008	0.003
Reported value, avg	54.48	13.65	13.50	6.95	3.28	3.31	1.68	2.23	0.17	0.040

<u>USGS W-1</u>										
Obtained, avg	52.5	14.7	11.1	11.1	6.7	2.2	0.7	1.1	0.16	0.018
Reported, avg	52.64	14.85	11.09	10.96	6.62	2.15	0.64	1.07	0.17	0.021

	<u>Co</u> <u>ppm</u>	<u>Ni</u> <u>ppm</u>	<u>Pb</u> <u>ppm</u>	<u>Zn</u> <u>ppm</u>	<u>Cu</u> <u>ppm</u>	<u>Ba</u> <u>ppm</u>	<u>Cr</u> <u>ppm</u>
<u>USGS BCR-1</u>							
Obtained, avg	40	20	<15	145	20	850	14
Obtained, range	(35-45)	(14-25)		(135-150)	(18-30)	(740-890)	(<7-18)
Reported, avg	35.5	15	18	132	22.4	790	16
Reported, range	(29-60)	(8-30)	(5-35)	(94-278)	(7-33)	(480-1230)	(8-45)

<u>USGS W-1</u>							
Obtained, avg	48	80	20	108	112	198	125
Reported, avg	43	69	7	76	116	175	115

TABLE VI
TYPICAL BASALT RESULTS

	<u>200'</u>	<u>205'-1</u>	<u>205'-2</u>	<u>225'</u>	<u>250'</u>	<u>255'</u>	<u>265'</u>	<u>270'</u>	<u>280'</u>
<u>%</u>									
SiO ₂	52.6	68.0	69.6	71.2	58.9	54.1	53.5	53.6	52.9
Al ₂ O ₃	13.1	12.2	12.2	10.0	16.1	14.8	14.0	14.7	14.6
FeO	12.8	3.3	3.5	3.6	7.0	11.0	10.8	10.5	10.7
MgO	4.7	2.2	2.1	1.3	3.5	5.4	5.8	5.9	7.3
CaO	8.1	0.7	0.7	2.4	4.8	9.3	9.3	9.4	10.7
Na ₂ O	2.6	1.5	1.5	1.5	1.4	1.8	2.0	2.4	2.3
K ₂ O	1.3	2.6	2.6	1.6	1.5	0.5	0.4	0.6	0.5
MnO	0.20	0.06	0.06	0.11	0.11	0.17	0.19	0.17	0.19
TiO ₂	3.3	3.8	3.8	2.3	1.3	2.0	2.0	1.8	1.8
SrO	0.029	0.023	0.024	0.023	0.023	0.026	0.026	0.029	0.029
Volatility	2.53	7.57	-	2.20	5.03	1.80	2.40	2.41	0.91
<u>ppm</u>									
Zn	240	60	60	50	80	150	90	110	90
Ni	70	40	40	30	40	60	70	70	65
Co	90	50	55	40	50	65	80	80	70
Pb	90	90	105	60	70	90	95	120	90
Cu	30	40	30	20	30	50	70	80	60
Cr	60	40	20	40	40	120	80	100	120
Ba	1050	1500	1350	930	1000	750	750	750	750
Rb	72	85	88	88	120	75	73	55	55

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