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Detection of (zinc, lead and Iron) in the Iraqi rocks.

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ABSTRACT

Reliable methods for the determination of lead, Iron and zinc in silicate rocks are given. Possible interference effects are investigated and precision and accuracy evaluated. All elements are determined directly after decomposing the sample with hydrofluoric and perchloric acids using the Atomic spectrophotometer Perkin—Elmer Model 303. The results are reliable for the contents higher than 0.0004% for Iron and zinc and 0.001% for Lead in silicate samples, respectively. The purpose of this study was to determine an optimal dissolution method for silicate rock samples for further analytical purposes(2,5).

Keywords: silicate rock dissolution, trace elements, iraqi rocks.

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INTRODUCTION

We studied two sections of rocks obtained through northern Iraq areas of the province (Erbil and Mosul) rocks. In the northern areas affected by movements alpine spread of metallic minerals notes where and are out Ahramaia Areas adjacent to Turkey, containing different concentrations of metals lead, Iron and zinc Carbonatih rocks associated with the iron sediment from Alsediraat and Almataat, while mineralization north-eastern areas bordering Iran objects Alsarrah and nowhere mineralization containing copper, nickel, chromium and iron are associated (Almagnetayt) in addition to Carbonatih rocks and clays. Lead and zinc (the provinces of Dohuk and Alsellmanah) there are these ores in the north of Zakho like Saragozh Toavan and Marciss and Birzanak and positioned in Ckorcarboonatih deposited in the marine environment to form Kara Jenny In Saragozh and configure Akre Bekhme in the rest areas. Mineralization appears in veins and nests in the rocks Babysitter or much like the parallel layers of the layers of the incubator, there is a prime location and a solo in Alj h. North East in the near Qaladiza the mineralization of lead and zinc are no Marrapasta area Qandil within the rocks are exposed to a series of transformations. Base metals are Algalina and Ambassaleraat And pyrite and Alparat (north of Zakho) is added except Alparat Asamthsonayat and Alsirusaat and in Albrootaat(7,8,9).

Iron ore of the kind associated with igneous rocks basal and above basal as well as the type of the variable in the Qandil series as well as its existence associated with manganese oxides in Alklklh series and all these Ahawaad exist within the volcanic concave. The first type (iron) are induced differentiation Alsaara complexes of firearms and unstable type (iron) produces thermal switch Carbonayatah sedimentary rocks rich in iron grew while Type III (manganese and iron) of volcanic processes under freely. All of these materials date back to the Cretaceous. The metal core of iron is Almagnetayt(10,11,12).

PROCEDURE

Determination of **Iron** in these Rocks : Convert ferrous to ferric oxidation by air during the preparation of the rock sample process and stages of succession determinate caution must be taken in the event of oxidative stress. Oxidation process for ferrous occur in the rock through the crush sample for this reason, the samples of the container must be crushed completely crushed until they reach the youngest particles. Crushing process must be carried out fully the existence of absolute alcohol until it reaches a high speed in the mechanical. It has been added to 10gm of the form in Beaker capacity of 400ml and melted sample in 30ml of de-ionized water and then add 5ml of diluted hydrochloric acid HCL. Preheat the sample for 5 minutes until boiling and then add 1ml of nitric acid HNO₃ conc in the form of droplets and became a light yellow color. The solution was reduced to 100ml of distilled water and then slowly add ammonia solution diluted 1: 1 with about 20ml constant stirring and there was a slight increase from the smell of ammonia emitted and the color is light yellow. The heating gently (without boiling) and then left to stagnate sludge observed that serene light yellow indication of the presence of ferric ions in solution. It have been burning rock deposits temperature (1300 m) depending on the presence of the element iron.

While Determination of **lead** in these Rocks: Weigh accurately (o.15 gm) from one of the samples of lead and move it to the size Beaker ((200 ml then desolve it in (75 ml) of dionized water. Acid solution acetic acid diluted (1: 1) by ((1ml then heated until boiling Add by pipette Potassium dichromate solution (4%), a slight increase 5ml)) is usually enough then boil the mixture gently for (5-10 mint) or even stabilized sludge Raa'iq liquid must be a little colored in yellow to increase the presence of chromate ions CrO₄⁻² as evidence fully precipitation. Filter it and move nominated precipitate sludge quantitatively to the filter paper and wash it off with a dilute solution of hot sodium acetate (10 ml) and then distilled hot water, Dry the sludge temperature oven (120 C) until the weight lbot(13), Record the weight.

Analysis of **Zinc** In Iraqi rocks : Ion-exchange column, set up a small ion-exchange column 8-cm in length and 0.8-cm in diameter, of a strongly basic ion exchange resin, such as Dowex 1 x 8, and wash thoroughly with 1.2 M hydrochloric acid, followed by 0.01 M hydrochloric acid and then water. Leave the column 1.2 M in hydrochloric acid ready for use.

Ammonium citrate buffer solution, dissolve 25 g of citric acid in 300 ml of water, bring pH of the solution to 8.5 by adding dilute aqueous ammonia, and dilute to 500 ml with water. Sodium borate buffer solution, dissolve 14.4 g of sodium hydroxide, 2.7 g of boric acid and 29.8 g of potassium chloride in about 950

ml of water, cool to room temperature and dilute to 1 litre. The pH should be 10.2. Zincon solution, transfer 0.130 g of the solid zincon reagent to a 100-ml volumetric flask, add 2 ml of 1 M sodium hydroxide solution and dilute to volume with water after the zincon is in solution. The solution is stable for about 10 days. Standard zinc stock solution, ignite zinc sulphate heptahydrate to constant weight in an electric muffle furnace set at a temperature of $^{\circ}50$. Allow to cool, and weigh 0.2V7 g Qf the anhydrous salt into a 200-ml volumetric flask and dilute to volume with water. This solution contains 500 μg Zn per ml. Standard zinc working solution, dilute 5 ml of the stock solution to 250 ml with 1.2 M hydrochloric acid. This solution contains 10 μg Zn per ml. Diethyldithiocarbamate solution, dissolve 0.5 g of the sodium salt in 100 ml water.

Weigh approximately 0.5 g of the finely ground sample material into a small platinum dish or crucible, moisten with water and add 10 ml of concentrated nitric acid and 10 ml of hydrofluoric acid. Cover the vessel, and set it aside for several hours, preferably overnight. Add to the dish 5 ml of perchloric acid and 10 ml of 20 N sulphuric acid. Transfer the dish to a hot plate and evaporate to a volume of about 3 ml. Cool the dish, wash down the sides with a little water, add a further 5 ml of perchloric acid and again evaporate, this time just to dryness - but avoiding baking the residue. Add 10 ml of concentrated hydrochloric acid and 25 ml of water to the residue, rinse the solution into a beaker and digest on a steam bath for 30 minutes. Cool the solution and transfer to a 100-ml volumetric flask. Transfer the entire 100 ml of solution (or a suitable portion of it containing from 20 to 100 μg zinc and diluted to a volume of 100 ml with 1.2 M hydrochloric acid) to the resin column. Regulate the flow rate of the solution through the column to about 1 ml per minute by adjusting the stopcock at the bottom of the column. When the flow ceases, discard the solution that has passed through, wash the column with 50 ml of 1.2 M hydrochloric acid and discard the wash solution. Place a clean 150-ml beaker under the column and elute the zinc by passing $^{\circ}5$ ml of 0.01 M hydrochloric acid through the resin also at a flow rate of about 1 ml per minute. Add 3 drops of phenolphthalein indicator solution to the beaker containing the zinc solution, and adjust the pH to 8.5 - 0.5 by adding dilute ammonia solution dropwise until the pink indicator colour just but only just forms. Quantitatively transfer the solution to a 125 ml separating funnel, add 2 ml of the (azoxy benzene or α -phenylsulfonyl pinacolone or DMG) solution, stopper the funnel and shake the solution. Add 10 ml of chloroform to the funnel and shake to extract the zinc-carbamate complex, draining the chloroform solution into a clean separating funnel. Rinse the stem of the extraction funnel with about 2 ml of chloroform and add this chloroform to the organic extract. Repeat the extraction and rinsing operations once more using 5 and 2 ml volumes of chloroform respectively, and then discard the aqueous solution. Add 10 ml of water to the chloroform solution and wash by shaking the funnel for about 30 seconds. Drain the chloroform into a clean 125-ml separating funnel, rinse the stem with about 2 ml of chloroform and add the chloroform wash liquor to the main portion of chloroform. Add 10 ml of 0.16 M hydrochloric acid to the combined chloroform solution and strip the zinc from the organic layer by shaking for at least 1 minute. Remove and discard the lower chloroform layer. Wash the aqueous phase with 10 ml of chloroform by shaking for 30 seconds and again drain, remove and discard the organic layer. Filter the aqueous solution through a 5.5-cm filter paper previously washed with 0.16 M hydrochloric acid, and collect the filtrate in a 50-ml volumetric flask.

Wash the separating funnel twice with about 3 ml of water, passing the washings through the paper into the flask. Add 10 ml of the sodium borate buffer solution to the flask and mix with the solution. A pH of 9 - 0.5 should be obtained. Add 3 ml of the zinc on reagent solution to the flask to give a brownish mixed colour, with a transition to blue as the zinc concentration increases. Dilute the solution to 50 ml with water. The intensity of the colour complex reaches a maximum very quickly and is stable for a few hours. Measure the optical density of this solution in 1-cm cells, against the reagent blank as the reference solution, using the spectrophotometer set at a wavelength of 620 nm(1,3,4,6).

Calibration: Transfer aliquots of 0-10 ml of the standard solution containing 0-100 μg Zn to separate 50-ml volumetric flasks, add 10 ml of 0.16 M hydrochloric acid, and 10 ml of the sodium borate buffer solution and mix well. Add 3 ml of the zinc on reagent solution, dilute to volume with water and mix well. Measure the optical densities in 1-cm cells at a wavelength of 620 nm and plot these values against zinc concentrations to obtain a standard working curve.

Determination of Zinc by Atomic Absorption Spectroscopy

The method below is based upon that described by Sanzalone and Chao. The determination of zinc by this method can be combined with the determination of a number of other elements of adequate sensitivity

by atomic absorption spectroscopy. The calibration curve for zinc is slightly convex towards the concentration axis. (11)A procedure for determining zinc and copper in the same solution has been given by(12) (13) Belt and the technique has been used by Burrell for determining zinc in amphibolites. Procedure. Accurately weigh approximately 0.5 g of the finely powdered silicate rock material into a small platinum dish or crucible, and evaporate to dryness with 1 ml of concentrated perchloric acid and 5 ml of concentrated hydrofluoric acid. Moisten the dry residue with a further 1 ml of perchloric acid and again evaporated to dryness. Allow to cool, add 0.3 ml of perchloric acid and rinse the residue into a small beaker. Warm until dissolution is complete, then cool, and dilute to 25 ml with water in a volumetric flask. Using an atomic absorption spectrophotometer fitted with a zinc lamp and operating according to the manufacturer's instructions, measure the absorption at a wavelength of 213.9 nm. Use a 10 ppm zinc standard solution to prepare a series of working standards covering the concentration range 0 to 10 ppm Zn. The calibration line is appreciably curved(14). Explained the Erbil rock behaviour contains Zinc metal after experiments , shows in Fig (1 and 2) , while Data experiment of Erbil rock contain Zn shows in Data (1).

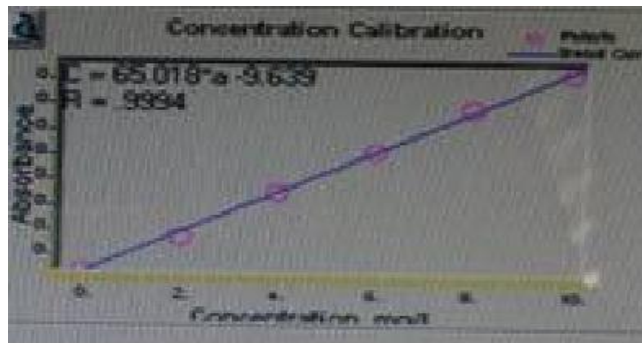


Fig (1)

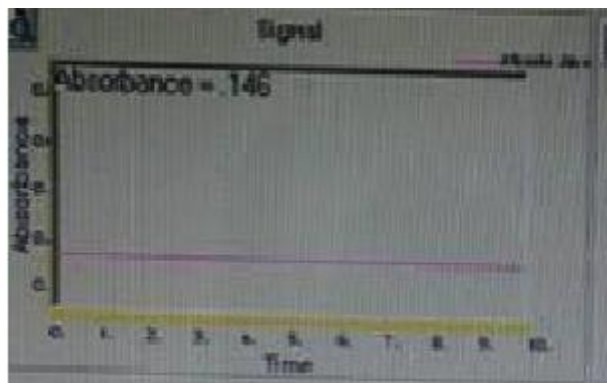


Fig (2)

NH	Type	Rep	Name	Weight	Volume	Dilution	Abs.	Conc	Cal. Conc	Unit	Sample Conc.
1	CSTD	1	Blank				0.1499	0		mg/L	
2	CSTD	1	Standard -1			1.00	0.1760	2		mg/L	
3	CSTD	1	Standard -2			1.00	0.2099	4		mg/L	
4	CSTD	1	Standard -3			1.00	0.2416	6		mg/L	
5	CSTD	1	Standard -4			1.00	0.2731	8		mg/L	
6	CSTD	1	Standard -5			1.00	0.3005	10		mg/L	
7	S	1	Sample -1				0.1530		0.3092	mg/L	
8	S	1	Sample -2				0.1477		-0.0358	mg/L	
9	S	1	Sample -3				0.1454		-0.1870	mg/L	
10	S	1	Sample -4				0.1486		0.0200	mg/L	
11	S	1	Sample -5				0.1484		0.0067	mg/L	
12	S	1	Sample -6				0.1463		-0.1272	mg/L	

Data experiments (1)

RESULT AND DISCUSSION

After making the calculations on the analytical work of the rock sample found Mosulazoxy with benzene reagent give :Weight of Zn about (0.03 gm) , Percentage ratio of zinc element in this sample of rock about (6 %) . While Rock Arbil after the addition of reagent DMG give : Weight of Zn about (0.089 gm) and Percentage ratio of zinc element in this sample of rock about (17.5%) . Rock Arbil treated with α -phenylsulfonyl pinacolone reagent and after addition ,Zinc has been detected and getting by weight 0.01 gm ,The percentage equal (2%) for Zn .It took weights obtained using gravimetric analysis and quantitative manner precipitation normal existence digestion to conduct tests through automated analysis using spectroscopic device , the rock sample found Mosul with azoxy benzene reagent give absorption (A = 99.4) , Solubility various Iraqi rocks models in various types of organic and inorganic solvents test0.2 gm weight was six kinds of rocks and went in 2 ml of inorganic and organic solvents were obtained results outlined shows in table(1,2,3,4)

Table (1) shows the solubility test rock Basra in various solvents, a hot or cold

Solvent	hot	Gold
H ₂ O	Complete	Partially
CCl ₄	Complete	Partially
CH ₃ OH	Complete	Insoluble
CH ₃ CH ₂ OH	Complete	Insoluble
CHCl ₃	Complete	Insoluble
NH ₃	Insoluble	Insoluble

Table (2) shows the solubility test Rock Kirkuk in various solvents, a hot or cold

Solvent	hot	Gold
H ₂ O	Complete	Partially
CCl ₄	Insoluble	Insoluble
CH ₃ OH	Complete	Insoluble
CH ₃ CH ₂ OH	Complete	Insoluble
CHCl ₃	Complete	Insoluble
NH ₃	Complete	Insoluble

Table (3) shows the solubility test Rock Nasiriyah in various solvents, a hot or cold

Solvent	Hot	Gold
H ₂ O	Insoluble	Insoluble
CCl ₄	Insoluble	Insoluble
CH ₃ OH	Insoluble	Insoluble
CH ₃ CH ₂ OH	Insoluble	Insoluble
CHCl ₃	Insoluble	Insoluble
NH ₃	Insoluble	Insoluble

Table (4) shows the solubility test Rock Arbil in various solvents, a hot or cold

Solvent	hot	Gold
H ₂ O	Insoluble	Insoluble

CCl ₄	Insoluble	Insoluble
CH ₃ OH	Insoluble	Insoluble
CH ₃ CH ₂ OH	Insoluble	Insoluble
CHCl ₃	Insoluble	Insoluble
NH ₃	Insoluble	Insoluble

Table (5) shows the solubility test ROCK Mosul in various solvents, a hot or cold

Solvent	Hot	Cold
H ₂ O	Insoluble	Insoluble
CCl ₄	Insoluble	Insoluble
CH ₃ OH	Insoluble	Insoluble
CH ₃ CH ₂ OH	Insoluble	Insoluble
CHCl ₃	Insoluble	Insoluble
NH ₃	Insoluble	Insoluble

Table (6) shows the results that obtained by add classical method to rocks Mosul and Erbil.

	Sample	Weight (gm)	%
1	Zn in mousl rock	0.04	7%
2	Zn in Erbil	0.09	18%
4	Fe in mousl rock	0.02	4.41%
5	Fe in Erbil rock	0.03	6%
6	Pb in mousl rock	0.01	2 %
7	Pb in Erbil rock	0.071	6.23%

Table (7) shows the results that obtained by add organic reagents to Erbil rock precipitates of Zn only.

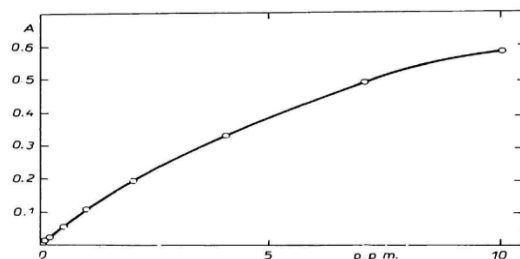
	Reagents	weight	%
1	azoxy benzene	0.03	6%
2	DMG	0.089	17.5%
3	-phenylsulfonyl pinacolone α	0.01	2%

Table (8) shows the results that obtained by using Spectrophotometry to Erbil rock precipitates of Zn with organic reagents only, if Cs= 0.005

	Zn precipitate in Erbil rock	C _s	As	A1
1	azoxy benzene	0.0057	86.5	99.4
2	DMG	0.005	82.6	95.3
3	α -phenylsulfonyl pinacolone	0.0059	82.6	98.6

Table (9) shows the results that obtained by using Atomic absorption to Erbil rock precipitates of Zn with organic reagents only, if Cs= 0.005

	Zn precipitate in Erbil rock	Abs	Conc mg/L
1	azoxy benzene	0.1667	1.7629
2	DMG	0.3181	3.2992
3	α -phenylsulfonyl pinacolone	0.0559	0.6772



(1) Analytical curve of Zinc

CONCLUSIONS

We conclude that the concentration in the rock Mosul atomic absorption measurement greater than rock Arbil either in measuring the absorbance gave rocks Mosul and Arbil concentrations too close together and the absorbance was in Mosul rock bigger than rock Arbil is for this rock Mosul contained zinc larger than rock Arbil .

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