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Determination of chromium by atomic absorption spectrometry (AAS) and comparison with inductively coupled plasma optical emission spectrometry (ICP OES)

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Abstract

This study introduced an analytical approach for directly measuring chromium in baby formula using high-resolution continuum source electrothermal atomic absorption spectrometry along with solid sample analysis (AAS). The method for analyzing heavy metals involved Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES), which allows for quick and precise detection of trace elements. It's also important to note that many labs are now using inductively coupled plasma–mass spectrometry for even more sensitive metal analysis. The results from stability tests showed that using appropriate masking agents improved the reagent's selectivity and reduced interference from other metal ions such as copper (II) and iron (III). Therefore, this method is suggested for concentrating and measuring chromium (VI) at low levels using AAS, and it can also be used for analyzing real-world samples like industrial waste.

Keywords: Chromium (VI), Cu (II) and Fe (III), inductively coupled plasma optical emission spectroscopy (ICP-OES), potassium isobutyl xanthate (KIBX)

Introduction

Chromite was first used to create chromium chemicals between the years 1800 and 1816. It was first used as a refractory material in France around 1879. The use of chromium metal in steel production increased significantly between 1908 and 1919. Today, the main uses of chromium are in chemical, refractory, and metallurgical applications. Chromium, especially in its hexavalent form, can be toxic to animals and humans at high levels, but the difference between its essential role and its harmful effects is quite large. In small amounts, chromium is an essential part of the diet for both humans and animals. It plays a key role in the glucose tolerance factor, which helps bring high levels of blood glucose back to normal. Because of this, chromium is very important in preventing diabetes, as its function is closely connected to insulin.

When workers are exposed to too much chromium dust or mist, it can lead to lung cancer, nose ulcers, nasal septum perforation, and several other breathing problems. It can also affect the skin. If someone ingests too much chromium, they may experience stomach pain, nausea, vomiting, severe diarrhea, and bleeding. Long-term exposure can cause the cornea to become less sensitive, disrupt the blinking reflex that protects the eyes, and lead to tearing, swelling of the endothelial layer in blood vessels of the lungs, liver, kidneys, adrenal glands, brain, spleen, stomach, and intestines.

Chromium is used in many manufacturing processes across various industries. In the textile industry, it functions as an oxidizing agent. It is also widely used in the tanning industry to process all types of skins and hides. Hexavalent chromium compounds are well known for their unique properties among corrosion inhibitors used for metals such as iron, steel, zinc, aluminium, copper, brass, lead, and related alloys. Chromium chemicals play a vital role in the oil and gas industries, where they are used for controlling corrosion and preparing catalysts. Additionally, chromium is an important environmental pollutant, often released through industrial waste in the form of particles or dissolved substances.

A comprehensive review of existing literature showed that various reagents have been used for preconcentrating and determining chromium (VI) using atomic absorption spectroscopy. Different reagents such as ammonium tetramethylene, diethylamine dithiocarbamate (3), diethyl amine diethyloxycarbamate (1), a diethyo carbomate (2), mmonium pyrrolidine dithiocarbamate (3), thiosemicarbazide (4-5), sodium ethyl dithiocarbamate (6), 1,5-diphenylcarbazide (7), pentamethyl dithiocarbamate (8), and 1,8-dihydroxyanthroquinone (9) have been employed for estimating Cr (VI) through atomic absorption spectrometry. Along with these, several other atomic absorption and extractive atomic absorption methods have been developed for chromium analysis (10-35). However, studies on the use of potassium isobutyl xanthate (KIBX) as an analytical reagent are limited. Therefore, this research was conducted to evaluate the potential of KIBX as a complexing agent for the extraction of chromium (VI) and its subsequent determination by atomic absorption spectrometry.

A first look at how chromium (VI) reacts with potassium isobutyl xanthate (KIBX) in a neutral environment shows that the solution becomes cloudy and the precipitate forms more strongly when the pH is lowered to 7.0. To understand what kind of precipitate is formed, attempts were made to dissolve it into an organic layer using several solvents such as n-amyl alcohol, isoamyl alcohol, n-butanol, benzene, carbon tetrachloride, chloroform, chlorobenzene, methyl isobutyl ketone, nitrobenzene, and n-propyl acetate. It was found that the precipitate dissolves easily when shaken with a small amount of methyl isobutyl ketone (MIBK). The organic layer, which contains the dissolved precipitate in

MIBK, is separated from the water layer and then analyzed for chromium (VI) using atomic absorption spectrometry to determine if it forms a complex or just a simple associate. Further experiments were conducted to find out the nature of the chemical species involved.

Results and Discussion

Various parameters such as pH, reagent concentration, shaking time, sample volume, metal ion concentration and the effect of foreign ions which / influence quantitative recovery of chromium (VI) were studied.

Effect of pH on the extraction of Chromium (VI) with KIBX

A preliminary study showed that the formation of Cr (VI) – KIBX complexes was affected by the hydrogen ion concentration. The pH studies were carried out varying the pH from 2 to 10. In each case, a mixture containing 130 ug each of chromium (VI) and double distilled water, 3.5 ml of 2.0 per cent KIBX solution was maintained constant, varying the pH values of the aqueous phase from 2.0 using suitable buffer solution. Each of the above-mentioned solutions was shaken for 2.5 minutes with 10.0 ml of methyl isobutyl ketone. The organic extract was, collected and the absorbencies of the organic extracts were measured. The data pertaining to the influence of pH on the extraction of Cr (VI) - KIBX are presented in Table 1 and graph 1. It was observed from the results, above pH 5.0 the extraction is found to be less and the stability of the complex is also decreasing, below pH 5.0 the development of the full intensity of the complex is inhibited. Hence the optimum pH of 5.0 is recommended.

Table 1: Influence of pH on the extraction of Cr (VI) with KIBX

S. No	pH	Absorbance
		Cr
1	2	0.386
2	3	0.462
3	4	0.582.
4	5	0.634
5	6	0.543
6	7	0.448
7	8	0.328
8	9	0.252
9	10	0.128

Volume of water: 100 ml; [KIBX]: 3.5 ml of 0.5%; Shaking time: 2.5 minutes; Amount of metal ion: 130 µg

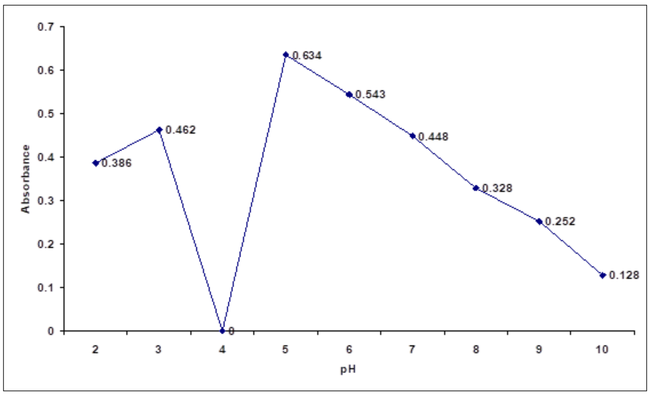

Fig 1: Influence of pH on the extraction of Cr (VI) with KIBX

Effect of sample volume on the extraction of chromium (VI) with KIBX

The extraction of chromium (VI) was studied at different volumes of the water sample in the range 100 - 300 ml. The study reveals that extraction was quantitative from 100 - 200 ml of water sample. Hence, 100 ml of water was employed throughout in the course of study.

Effect of shaking time on the extraction of Chromium (VI) with KIBX

The effect of shaking time on the extraction of chromium (VI) was studied. Chromium (VI) was readily extractable into MIBK. No change was observed in the extent of extraction, when the mixture was shaken from 1-5 min. Hence, 2.5 min of shaking time was enough for the complete extraction of the complex into MIBK.

Effect of reagent concentration on the absorbance of chromium (VI) with KIBX

The effect of reagent concentration was studied by measuring the absorbance of solutions containing 130 µg each of chromium (VI) in 100 ml of double distilled water and pH was adjusted to 5.0 with acetic acid-sodium acetate solution. To each of these solutions 0.5 to 5.0 ml of 2.0 percent KIBX was added. The metal complexes were extracted individually into 100ml portion of MIBK. The absorbance of these extracts was measured. The values are given Table 2 and Fig. 2.

Table 2: Effect of Reagent Concentration on the Extraction of Cr (VI)

Sl.No.	Volume of reagent (2.0%) (ml)	Absorbance
		Cr
1	0.5	0.242
2	1.0	0.293
3	1.5	0.356
4	2.0	0.458
5	2.5	0.528
6	3.0	0.592
7	3.5	0.628
8	4.0	0.622
9	4.5	0.618
10	5.0	0.614

Volume of water: 100 ml; pH: 5.0;
Shaking Time: 2.5 minutes;

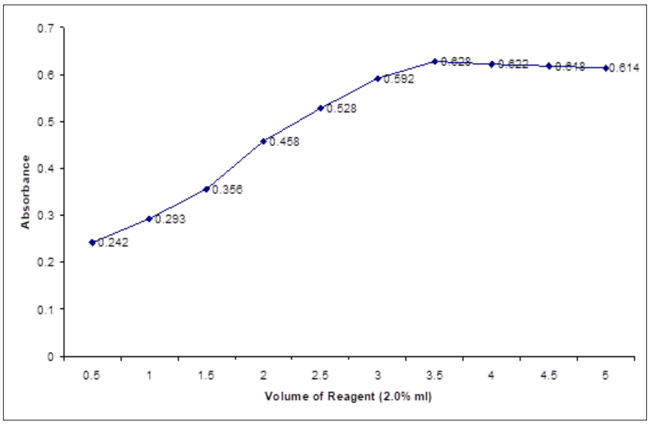

Fig 2: Effect of Reagent Concentration on the Extraction of Cr (VI)

From the data, it was noted that 3.5 ml of 2.0 per cent KIBX was sufficient for maximum extraction of chromium (VI) Hence this concentration was used for further studies.

Effect of foreign ions on the extraction of chromium (VI) with KIBX

In order to assess possible analytical applications of the method, the effect of diverse ions was examined by carrying out determination of 130µg each of chromium (VI) with a known amount of foreign ion under consideration, using the recommended procedure. The tolerance limit was set at the amount required to prevent an error not exceeding ± 2 per cent in the change of absorbance of chromium VI alone. The results are represented in Table 3.

Table 3: Effect of Foreign ions on the Extraction of Cr (Vl) with KIBX

Ion	Surface of Ion	Cr
Cd (II)	$(CH_3\ COO)_2\ Cd.H_2O$	2000
Se (IV)	SeO_2	2000
Mn (II)	$MnS0_4.H_20$	2500
Zn (II)	$Zn\ (SO_4).7\ H_2O$	1000
Ni (II)	$(NH_4)_2Ni\ (SO_4)_26H_2O$	2250
Bi (III)	$Bi\ (NO_3)_3.\ 5\ H_2O$	2500
Te	Pure metal	2500
V(V)	$NH_4\ VO_3$	2500
Cu (II)*	$CuCl_22\ H_2O$	-
Fe (III)*	$FeCl_3$	I
Acetate	$CH3COONH_4$	5000
Phthalate	$2(COOH)C_6H_4COOK$	4000
Bicarbonate	$NaHCO_3$	4500
Oxalate	$Na_2C_30_4$	3500
Phosphate	KH_3PO_4	5000
Citrate	$(NH_4)_3C_6H_60_7$	4000
Thiocyanate	$NH_4\ SCN$	3500
EDTA	$[CH_2N(CH_2COOH)\ CH_2COONa]\ 2\ H_2O$	5500

*Masked by using 2.0 ml of 0.5% EDTA solution
Amount of each metal ion: 130 µg;
[KIBX]: 3.5 ml of 2%; pH: 5.0;
Shaking time: 2.5 minutes

During the determination of chromium (VI) with potassium isobutyl xanthate by atomic absorption spectrometry, cations like Mn (II), Bi (III), Te and V (V) do not interfere up to 2500 µg, Cd (II), Se (II) can be tolerated up to 2000µg, Zn (II) does not have any effect up to 1000µg, Cu (II) and Fe (III) interfered severely in the present investigation. The interference due to Cu (II) and Fe (III) can be suppressed using 2.0 ml of 0.50 per cent EDTA solution. Anions like acetate up to 5000µg, phthalate up to 4000µg, bicarbonate up to 45000µg. carbonate up to 3500µg, phosphate up to 5000 µg, citrate up to 4000µg, thiocyanate up to 3500µg and EDTA up to 5500µg do not interfere in the determination.

Effect of metal ion concentrations on the extraction of chromium (VI) with KIBX

From the optimum conditions described above, a calibration curve was constructed. To the 100 ml of water sample, metal ion solution containing. 10-170µg each of chromium (VI) was added and the analysis was carried out using the recommended procedure. The data is presented in Table and the corresponding graph is shown in Table 4 and Fig 3. The sensitivity was found to be10.7841 mg /ml for chromium (VI) The calibration plot discloses a linearity in the concentration range of 1. 0 to 13.0 ppm of chromium (VI) Aliquots containing 8.0 ug/ml of Chromium (VI) gave a standard deviation of 0.9042 x 10"^{3} while the coefficient of variation is 0.3248 per cent.

Table 4: Effect of metal ion concentration on the extraction of Cr (VI) with KIBX

S. No	Amount of Chromium	Absorbance
1	10	0.052
2	30	0.149
3	50	0.284
4	70	0.378
5	90	0.469
6	110	0.538
7	130	0.629
8	150	0.638
9	170	0.682

Volume of water: 100 ml; [KIBX]: 4.7 ml of 2.0%; pH: 50
Shaking time: 2.5 minutes

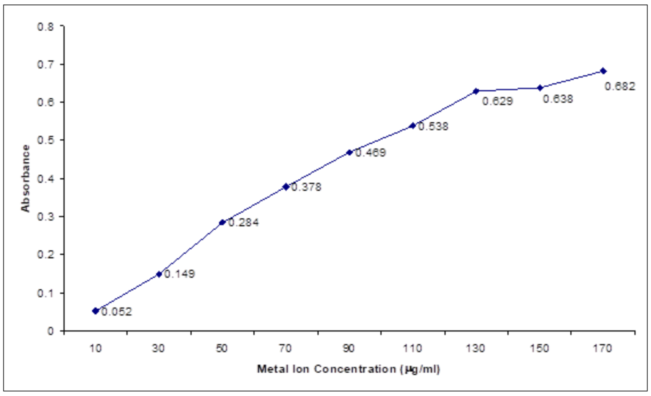

Fig 3: Effect of metal ion concentration on the extraction of Cr (VI) with KIBX

Applications of the developed method

The developed atomic absorption spectrometry method for chromium (VI) and was applied for the determination of these ions in industrial effluents.

Analysis of spiked water sample for the determination of chromium (VI) using the proposed new method

The recovery of chromium (VI) from spiked water sampled was examined using the general procedure. The results are given in Table 5 shows that the percentage developed method is applicable to analyse the spiked water samples with the percentage recovery for chromium (VI) being 99.6% This method was applicable for the analysis of real water samples.

Table 5: Determination of chromium (VI) in spiked water samples with Potassium Isobutyl Xathate

S. No.	Amount of Cr added(we)	Amount of Cr found(ne)	Percent recovery/ (Hg) /
1	50	49.1	98.2
2	70	69.2	99.3
3	90	89.2	99.1
4	110	109.4	99.4
5	130	129.5	99.6
6	150	149.2	99.46

Volume of water = 100 ml;
pH = 5.0
[KIBX] = 4.7 ml *of 2.0%;*
Shaking time = 2.5 minutes

Analysis of industrial effluents for the determination of chromium (VI) using the proposed new method

The proposed method was applied for the determination of chromium (VI) 'in industrial effluents, industrial effluents were collected from newly developed industrial areas in and around Kumool town. Analysis of chromium (VI) was done as described in the general procedure. The results are presented in Table 6. It was observed that of the 5 water samples analysed, chromium (VI) are present in all the samples.

The results obtained, by the present method and ICPOES method was in good agreement.

Table 6 Determination of chromium (VI) in industrial effluents with potassium isobutyl Xanthate

Name of the area Kurnool	Metal ion found (μg) (Average value of determinations)	
	Chromium	
	Present method A.A.S	ICP OES methods
Sample 1	8.3	8.34
Sample 2	8.9	8.91
Sample 3	7.8	7.82
Sample 4	4.7	4.70
Sample 5	3.9	3.92

Volume of water = 100 ml;
pH = 5.0
[KIBX] = 3.5ml2.0%;
Shaking time = 2.5 minutes

General and recommended procedure

To 100 ml of double distilled water taken in 250 ml beaker, an aliquot of standard metal ion solution containing 10-170μg of chromium (VI) was added, the pH was adjusted to 5.0 with acetic acid - sodium acetate buffer solution. The solution was transferred into a separating funnel, to which 4.7 ml of 2.0 per cent potassium isobutyl xanthate (KIBX) was added. The mixture was shaken vigorously for 2.5 minutes with 10 ml of methyl isobutyl ketone (MIBK). The two phases were allowed to separate the organic layer was collected and analysed for chromium (Vl) by atomic absorption spectrometry. The metal content was deduced from these results and the experimental conditions indicated in the Table 7.

Table 7: Condition of Employed for Flame Absorption Spectrometry

S.No.	Element	Lamp current (mA)	Wavelength (nm)	Slit (nm)	Flame gas
1	Cr	25	357.9	0.7	A-AC

A- Ac - Air acetylene

Conclusions

The method developed for concentrating specific heavy metals can be used for analyzing multiple elements in real samples using ICP-OES and AAS. The results show that this method has several advantages, including being simple, fast, and cost-effective, along with being sensitive. Some other elements present do not interfere with the analysis. The use of suitable masking agents improves the reagent's selectivity and helps reduce interference from certain metal ions like Cu (II) and Fe (III). Because of these benefits, the method is recommended for the preconcentration and simultaneous detection of chromium (VI) at low levels, as well as for analyzing real-world samples such as industrial waste.

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Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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