

Determination of Arsenic, Chromium and Lead in titanium dioxide pigments by ICP- OES with Concomitant Metals Analyser

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ABSTRACT

A simple method has been developed for the determination of trace toxic elements like arsenic, lead and chromium in titanium dioxide pigment samples by ICP OES attached with a Concomitant Metals Analyser. Open mineral acid decomposition was used for sample dissolution employing a mixture of nitric and hydrofluoric acids. The continuous online generation of hydrides into the plasma was achieved through a concomitant metals analyser. The recovery of arsenic, lead and chromium and the matrix effects of titanium on these elements have been studied with spiking experiments. The proposed method has been successfully applied to the determination of arsenic and other elements in titanium pigment samples. The continuous hydride generating system, Concomitant Metals Analyser (CMA) improved the sensitivity of analysis nearly five times in pigment samples. The precision of the measurements was found to be less than 10% RSD.

Keywords : Titanium pigment, CMA-ICP OES, Arsenic, Chromium, Lead

I. INTRODUCTION

Titanium dioxide plays an important role in many fields of modern industry. It is mainly used as a white pigment in paint industry. The pigments finds use in paint industry owing to its colour, finish, protect the surface from corrosion and weathering as well as helping to hold the paint together [1]. Besides the use in paint industry, titanium dioxide is also used in food industry, medicine, pharmacy, cosmetics, textile industry, in ceramics, tooth pastes and other various fields in modern technology due to its brilliant whiteness and resistance to colour change. The metal impurities in TiO₂ like As, Cr, Pb, Fe, V, Zr and Cu affect its properties by changing the colour, electrical properties, increasing the toxicity etc. Due to the wide use of titanium dioxide pigment in different industries, it is very essential to determine the trace

toxic elements like lead, chromium, arsenic, antimony etc. in pigments.

Lead (Pb) is routinely determined toxic element in paints due to its critical environmental toxicity especially to children and all the countries have been taking steps to phase out the lead from paints [2]. There were many standard test methods reported in literature for the determination of Pb in paints and pigments by wet chemical methods [3]. Arsenic is also toxic and carcinogenic and the toxicity depends on the chemical speciation and oxidation state of As. Studies reported earlier revealed that the trivalent arsenic is about 60 times more toxic than the pentavalent state. The sulfhydryl groups present in human body react with arsenite, whereas arsenate does not react with it [4-5]. The toxicity of As increases in the order of arsenobetane, arsenosugar,

dimethyl arsinic acid, monomethyl arsonic acid, arsenate and arsinite [6]. The As and Pb content in the product should meet the purity requirements of typical maximum limits of 3 and 10 mg/Kg [Commission Directive 95/45/EC of specific purity criteria concerning colours for use in foodstuffs, 1995].

Lead (Pb), Chromium (Cr) and Arsenic (As) are natural components in Earth's crust and are widely distributed throughout the environment in the air, water and land. There are many methods available for the determination of As and other toxic elements in food, plant, soil, coal, biological, environmental and geological samples [7]. Many analytical methods like Gutzeit method, Hydride generation and determination by AAS, Spectrophotometry etc had been reported for the determination of Arsenic in paints [8]. Wet chemical methods are seldom available for the decomposition of this pigment for the determination of volatile As due to the highly refractive and hydrolysable nature of titanium pigment.

Various Direct analytical techniques were used to analyse the solid titanium dioxide samples for its detrimental impurities. Direct solid sampling Electrothermal AAS was used for analysis of As, Cd, Hg, Pb, Sb and Zn in titanium dioxide samples by B. Docekal et al [9]. Direct solid sampling for ICP- OES for TiO₂ and Al₂O₃ powders [10] and slurry nebulisation and fluorination assisted electrothermal vapourisation ICP OES for Cr, Cu, Fe and V [11] are also reported. D. Wildhagen et al used INAA and RNAA for direct determination of numerous metal impurities in titanium dioxide samples including As [12]. Microwave digestion methods were applied for the decomposition of titanium dioxide based sunscreen creams using combination of acids like HNO₃, HCl, HF and H₂SO₄ [13]. Microwave assisted digestion methods were also used to prepare the titanium dioxide pigment slurry sample using

hydrofluoric acid, sulphuric acid and hydrogen peroxide at 250°C and pressure of 2.6MPa for 20minutes [9]. There are different methods of separation for total As and its speciation studies. Chromatographic and solvent extraction techniques like As-APDC (Arsenic-Ammonium Pyrrolidine Dithiocarbamate) were used for the separation and preconcentration of arsenic from the rest of the matrix [14].

The present work deals with a simple decomposition technique for titanium dioxide pigment with HF, HNO₃ and a drop of 1:1 sulphuric acid and the determination of total As, Cr and Pb were carried out with a modified spray chamber called concomitant metal Analyser (CMA) equipped with ICP OES for the online generation of hydrides which gives a better sensitivity than the normal ICP OES [15].

II. METHODS AND MATERIAL

A. Instrumentation

HORIBA JOBIN YVON Model Ultima -2 with 40.68 MHz sequential inductively coupled plasma optical emission spectrometer (ICP OES) with Concomitant Metal Analyser (CMA) with nitrogen purging for spectrometer was used for the experiments. The instrumental parameters were given in Table -I.

B. Reagents and Standards

Certified plasma calibration standard solution traceable to NIST procured from Alfa Aesar was used for calibration of the instrument. 1% NaBH₄ (W/V) in 0.5M NaOH and 6M HCl (37% EMSURE) were used for online hydride generation process. All the reagents used were analytical grade and acids used were either Merck EMSURE grade or purified by sub-boiling distillation.

C. Procedure

1) Sample decomposition using mineral acid mixture of HF and HNO₃: 0.2-0.5g pigment sample was weighed into a clean and dry platinum dish (75 mL capacity); 5mL HF (40% EMSURE) and 2 mL HNO₃ (sub boiled acid of ExcelsaR 69-70%) were added and kept on a boiling waterbath. The content in the dish was allowed to evaporate till it becomes a moist mass. The process was repeated twice with 3mL HF, 1mL HNO₃ and a drop of 50% (v/v) sulphuric acid (EMSURE 95-97%). The treatment was continued with only 3mL HNO₃ for three times. The sample should be allowed to become insipient dryness like a syrupy liquid. The syrupy nature of the sample was shown in (Figure-I). 1.5mL HNO₃(15N) was added to the sample and 15-20 mL deionised water, kept on waterbath to digest (Figure-II) and made upto 50mL in Tarsons make polypropylene volumetric flask with deionised water.

2) Sample decomposition using Sulphuric acid fuming: 0.2 g sample was weighed into a clean dry 100mL beaker, sulphuric acid fuming was carried out with 10mL 50% H₂SO₄ on a hot plate at 150-180°C. The sample was allowed to fume off till insipient dryness. After cooling, it was made upto 50mL in a borosil make volumetric flask using deionised water.

D. CMA-ICP OES measurement

The concomitant Metals Analyser (CMA) is a hydride generator used with ICP OES to improve the sensitivity of hydride forming elements (Figure-III). It is a modified spray chamber attached with a separate peristaltic pump to transport the sodium borohydride and HCl solutions. These reagents get mixed with the sample in the chamber, where the reaction takes place. The analysis can be performed simultaneously for both normal and hydride forming elements. The readings were taken in all solutions by

ICP OES equipped with CMA using 0.1, 0.5, 1 and 5 ppm calibration standards. The spectrometer was continuously purged with nitrogen for taking care of the wavelengths below 200nm and operating conditions are given in Table-I.

III. RESULTS AND DISCUSSION

A. Studies on sample decomposition methods

The TiO₂ content in the pigment sample was found to be above 99%. There are not many methods available for the complete decomposition of titanium dioxide pigment by wet analytical methods for the analysis of trace and toxic volatile element like arsenic due to the complex nature of the matrix. These samples are not readily decomposed by mineral acids and the high titanium content in the sample readily undergoes hydrolysis in the presence of mineral acids. The development of suitable, sensitive analytical methods for determination of these detrimental impurities is an important task, because common vigorous methods to decompose titanium dioxide pigment suffer loss due to volatilization of arsenic, hydrolysable nature of titanium hydroxide leads to incomplete decomposition and desired clear solution may not be resulted. The main aim of the proposed decomposition method is to determine the highly volatile trace As, and toxic Pb and Cr in complex titanium dioxide pigment. Any vigorous treatments like fusion methods with fluxes like mixed phosphate; potassium bisulphate, sodium peroxide etc leads to loss of As by volatilization and precipitation due to hydrolysis. In the case of As, the hydride of As (III) having a low boiling point of -63°C is the most volatile of the arsenic gases, unstable under atmospheric conditions and is readily oxidisable in air. Arsenic (III) chloride compounds with a boiling point of 130°C are also

readily volatile [14]. The recovery of arsenic is satisfactory when the decomposition is carried out in the presence of oxidizing environment. HNO_3 and H_2O_2 are good oxidizing agents, which oxidize As (III) present in the sample to As (V) that gets retained in the solution. A very careful method of decomposition of the pigment is necessary to avoid loss of arsenic in solution. Much work has not been reported in the field of wet analytical chemistry for the determination of trace amounts of arsenic in titanium dioxide pigment samples.

In the present work, attempts were made to decompose the samples by three methods using mineral acids on water bath, sulphuric acid fuming on hot plate and microwave digestion with mineral acids in the presence of hydrogen peroxide. Spiked samples were used for the recovery studies. The presence of HF or H_2SO_4 is required to bring the matrix into solution and these acids are necessary to keep TiO_2 in solution. The hydrolysable TiO_2 present in the sample leads loss of analyte, due to the adsorption of arsenic into the hydrolysable precipitate. It is very essential to completely solubilise the pigment; otherwise there is a chance of loss of As due to sorption of As on TiO_2 . Granular or powdered TiO_2 pigment is found as a good adsorbent for As in the pH range of 4-11 and the oxidation of As(III) and As(V) has been studied in the presence of TiO_2 [17-19].

Microwave digestion method was attempted for the complete decomposition of pigment sample using hydrofluoric acid, nitric acid and hydrogen peroxide using CEM MARS microwave oven (CEM Corp., Matthews, NC, USA. The decomposition was carried out at a temperature of 150-180°C with a hold time of 20 minutes) using 12-position HP-500 vessel and rotor system. TiO_2 spec pure standard doped with As, Cr and Pb was processed along with the samples. The recovery studies with spiking samples showed that As, Pb and Cr were

not completely recovered by microwave digestion (MDS) decomposition method. The recovery of these elements was found to be very low, due to the incomplete dissolution of the sample and the undissolved titanium dioxide acted as a sorbent for As and other metals.

Titanium dioxide forms soluble complexes with sulphuric acid fuming. The sample was decomposed with sulphuric acid fuming at $\sim 200^\circ\text{C}$ using a temperature controlled hot plate. The resultant solution contained traces of unattacked matter with 0.2g sample weight. The decomposition was difficult with more than 0.2g sample, and the higher amount of sulphuric acid was posing challenges to the determination stages using CMA in the case of As with higher process blank and less recovery.

The decomposition was also carried out in platinum dishes on a water bath and a clear solution obtained with careful digestion, even in the case of more sample weight of 0.5g. The sample should not be completely dry and the evaporation of acids should leave a residue of syrupy nature of insipient dryness, which is shown in Figure-I. The sample should not be allowed to completely dry. The complete drying of the sample leaves a white residue, which is not soluble in nitric acid. Nitric acid acts as an oxidizing agent for As to remain in solution, otherwise As (III) fluorides are volatile. The traces of fluoride and sulphate present in the solution prevented the hydrolysis of titanium.

The decomposition methods involving HF and HNO_3 in a platinum dish on a water bath and sulphuric acid fuming on a hot plate were found to be very effective for the pigment samples. The total recovery of As, Pb and Cr is achieved with complete dissolution of the sample that was achieved due to the traces of sulphuric acid and fluoride resulted from the syrupy nature of the mixture. This method was found to be more

suitable for the determination of As, Cr and Pb in titanium dioxide pigment sample.

Due to the nonavailability of matrix matched reference materials, spiking technique with spec pure titanium dioxide was used for the recovery studies. The spiking experiments showed very good recovery of As and other toxic metals like Cr and Pb with the digestion on water bath due to the complete decomposition with a clear solution. The loss of As was minimized as the digestion temperature was maintained at 100°C. The sulphuric acid fuming on a hot plate was also effective in the case As, Cr and Pb recovery. The sulphuric acid fuming also resulted very minute unattacked matter, but the recovery of arsenic was more than 90% with spiking studies. The fuming method was not preferred due to the high amount of sulphates present causes problems in hydride generation, restrictions in sample intake and it is not considered as environmental friendly due to the evolution of thick sulphur trioxide fumes.

The proposed method of digesting sample on water bath can decompose higher sample amount with low process blank values, resulted improved determination limit. As, Cr and Pb are present in trace quantities in pigment sample, more signal obtained from more sample weight improved the precision and sensitivity. The process blank values were found to be lower in the proposed digestion method than the sulphuric acid fuming. The signal from the blank solution becomes significantly important in trace level determinations.

B. Selection of CMA-ICP-OES for determination of As, Cr and Pb

A Concomitant metals analyser was used along with ICP OES for online generation of hydrides, which improves the sensitivity of the element due to generation of volatile hydrides in the CMA. The

online generation of hydrides results separation and preconcentration of hydride forming elements from the matrix elements. Pb in water [20] and Arsenic in water and rock samples have been reported to be determined by CMA after prior reduction using KI and ascorbic acid [21]. The prior reduction of As with KI and ascorbic acid did not give satisfactory recovery results in the case of pigment samples spiked with known concentration of standards. It is found from the experiments that, the recovery was satisfactory without prior reduction. This may be due to the complex nature of sample due to the presence of high titanium. The analysis was carried out without prior reduction of As using CMA attached ICP OES with nitrogen purging and the operating conditions are given in Table-I. Other elements like Cr and Pb were determined by normal flame AAS. The advantage of CMA is that all the elements which forms hydrides or not can be analysed simultaneously without changing the CMA attachment, saving the analysis time.

The samples were analysed by an alternative technique of Hydride Generation- Atomic absorption spectrometry (HG-AAS). The hydrides are generated from the whole aliquot of the sample and the accumulated arsine is purged through the flame or electro-thermal furnace to get an enhanced transient signal in the case of HG-AAS. Only one element can be determined at a time, HG-AAS is commonly used for arsenic determination. But, in the case of CMA, the hydrides are continuously generated and separated from the matrix, and enters the modified cyclonic spray chamber and to the plasma. The sensitivity is improved by 5 times in CMA compared to the conventional ICP OES. Arsenic can be determined upto 2 µg/g, Pb by 10µg/g and Cr by 1µg/g on the basis of 0.5g/50mL by the proposed method in titanium dioxide pigment samples.

C. Selection of wavelengths of As, Cr and Pb in samples by CMA-ICP-OES

The wavelengths used for determination of As, Cr and Pb were selected judiciously by taking into consideration of parameters like sensitivity and interferences due to titanium dioxide matrix. As was determined at three wavelengths, 189.042nm, 193.695nm and 197.198nm and the values obtained at all the three wavelengths by CMA using purging the spectrometer with nitrogen were comparable. But the As values in sulphuric acid fuming solution were found to be lower at 193.695nm and 197.198nm. So the As I 189.042nm line was selected for the analysis and were given in the Table-II. The Cr determination was carried out 283.560 nm instead of 267.716 nm which has an overlapping peak at right side in this type of matrix. Pb was determined at 220.353 nm which was found to be free from interferences.

IV. VALIDATION OF METHOD

For ascertaining the accuracy, the values were compared with those obtained by alternate techniques and spiking studies. The accuracy of proposed method of decomposing the sample using mineral acids on waterbath was carried out by Sulphuric acid fuming decomposition method on a hot plate with controlled temperature of 150-180°C and the values are compared in Table-II. It was found that recovery is satisfactory for As, Cr and Pb with spiking studies and the recovery results are given in Table-III. The As results were compared with those obtained by Hydride generation attachment with Atomic Absorption Spectrometer (HGAAS). Pb and Cr were also determined and Atomic absorption Spectrometer (AAS). All the values are found to be agreeable with CMA – ICP OES within $\pm 10\%$. The values were reported with 95% confidence calculated from five replicate measurements. The data comparison was given in Table-IV.

Due to the unavailability of certified reference material of titanium pigment, spec pure solid 0.2 g TiO₂ standard was weighed in platinum dish and spiking was carried out for the recovery of As, Cr and Pb and matrix effect of TiO₂ on As, Cr and Pb. The process blank of TiO₂ was prepared with only 0.2g TiO₂ spec pure standard for evaluating the purity of TiO₂ standard with respect to the three elements and to ascertain the spectral interferences. It was established that, there are no spectral and matrix effect of titanium on As on the 189.042 nm wavelength selected for measurement using CMA. The Cr determination was carried out 283.560 nm instead of 267.716 nm which has an overlapping peak at right side, due to the major matrix of titanium present in the sample. The recovery was above 90% for As, Cr and Pb with the proposed method. The certified rock reference materials like GXR-1 and GXR-4 (Geochemical Exploration reference samples) were also analysed by the proposed decomposition method to verify the recovery of Cr, Pb and volatile As. These CRMs were mixed with spec pure titanium dioxide pigment in the platinum dish and were also analysed by the proposed decomposition method. The recoveries of Cr, Pb and volatile arsenic in all the cases were satisfactory and the data is given in Table-V.

V. CONCLUSION

The combination of HF, HNO₃ and H₂SO₄ effectively destroys the refractory nature of TiO₂ pigment, enabling the complete dissolution of the pigment into solution and effective measurement of As, Cr and Pb. The lower temperature of waterbath coupled with the oxidizing environment inhibits the loss of arsenic due to volatilization. The concomitant metals analyser coupled with ICP OES is an effective and sensitive method of determination of As in refractory samples like titanium pigment samples. Considering the refractory and hydrolysable nature of titanium

dioxide pigment, the proposed method of determination of As, Cr and Pb is fast, convenient and environmentally friendly for analyzing a large number of samples, which helps the manufactures to assess the extent of the trace toxic elements contamination in pigment samples, thus reducing the environmental pollution.

VI. ACKNOWLEDGEMENTS

The authors wish to acknowledge Shri. M. B. Verma, Director, AMD for his kind permission to publish the paper and Additional Director (R&D) for the support and encouragement in carrying out the research work. The authors gratefully acknowledge the Planning and Management Services Group for the samples.

VII. REFERENCES

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TABLE I. ICP-OES Instrumental Parameters and Operating Conditions

Mounting	Czerny-Turner
Focal length	1m
Grating	4320 grooves/mm & 2400 grooves /mm
Order of measurement	1 st
1 st order resolution	0.005
Type of generator	Solid state
Observation	Radial View
Frequency of generator	40.68 MHz
Power	1000W
Plasma gas flow rate	12 L/min
Sheath Gas flow rate	0.2 L/min
Nebulizer gas flow rate	0.8 L/min
Nebulizer type	Meinhard
Type of spray chamber	CMA type
CMA Peristaltic pump speed	10 rpm
Injector tube diameter	1.8 mm, Alumina
Purging gas used for below 200nm	Nitrogen

TABLE II. Analytical Data of toxic elements in titanium dioxide pigment by ICP AES using CMA system (n=5)

Elements	Wavelength nm	AMD-1		AMD-2	
		Proposed decomposition on water-bath	H ₂ SO ₄ fuming on hot plate	Proposed decomposition on water-bath	H ₂ SO ₄ fuming
As(μg/g)	189.042	19 ± 1.5	20 ± 2	10± 1.5	9± 2
Cr (μg/g)	283.560	11±2	11± 2	13±1.5	12± 1.5
Pb (μg/g)	220.353	<10	<10	<10	<10

TABLE III. Recovery of As, Cr and Pb from spiking experiments with 0.2 g spec pure titanium dioxide using CMA-ICP OES

Elements	μg added	μg recovered using Proposed method	μg recovered H ₂ SO ₄ fuming on hot plate
As(μg/g)	100	93	90
Cr (μg/g)	100	97	95
Pb(μg/g)	100	95	93

TABLE IV. Comparison of Analytical Data of As, Cr and Pb with HG-AAS and AAS

Elements	AMD-1			AMD-2		
	Proposed method	HG-AAS*	AAS*	Proposed method	HG-AAS	AAS
As(μg/g)	19 ± 1.5	20	-	10± 1.5	10	-
Cr (μg/g)	11±2	-	10±2	13±1.5	-	12±1.5
Pb(μg/g)	<10	-	<10	<10	-	<10

*As was determined by HG-AAS and Cr and Pb was determined by normal flame AAS.

TABLE V. Analytical Data of CRMs mixed with titanium dioxide by CMA-ICP OES

Elements	GXR-1		GXR-4	
	Proposed method	Reference value [22]	Proposed method	Reference value [22]
As(μg/g)	418±25	427	108±11	98
Cr (μg/g)	11±1.5	12	61±8	64
Pb(μg/g)	720±25	730	49±6	52



Figure I : The syrupy nature of digested sample using HF, HNO₃ and a drop of 1:1 H₂SO₄



Figure: II : The final solution in 3% HNO₃ in platinum dish on boiling waterbath

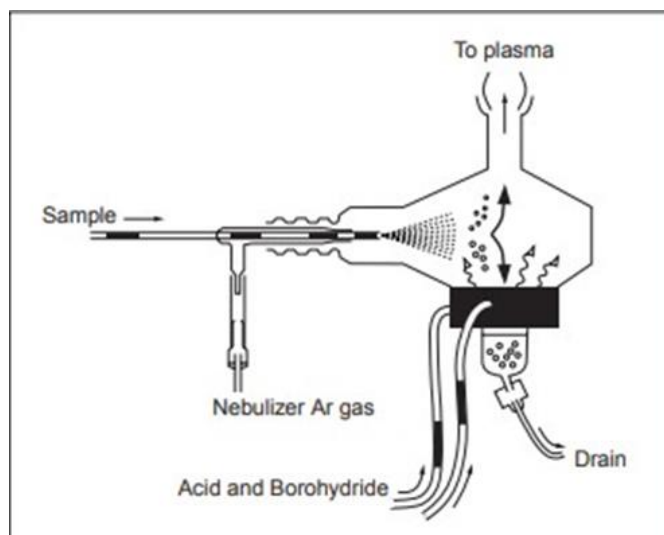


Figure-III : Concomitant Metals Analyser Chamber

Cite this article as :

Beena Sunilkumar, S. B. Singh, "Determination of Arsenic, Chromium and Lead in titanium dioxide pigments by ICP- OES with Concomitant Metals Analyser", International Journal of Scientific Research in Science and Technology (IJSRST), Online ISSN : 2395-602X, Print ISSN : 2395-6011, Volume 6 Issue 6, pp. 296-305, November-December 2019. Available at doi : <https://doi.org/10.32628/IJSRST196664>
Journal URL : <http://ijsrst.com/IJSRST196664>