## ADSORPTION STUDIES OF Fe[III] ON BENTONITE IN THE PRESENCE OF D-VALINE AS CHELATING AGENT

Dissertation submitted in Partial fulfillment for the requirements for the award of degree of

# Bachelor of Science ín CHEMISTRY

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Tara Government College, Sangareddy(A) 2021-22

### <u>CERTIFICATE</u>

This is to certify that the project work entitled "ADSORPTION STUDIES OF Fe[III] ON BENTONITE IN THE PRESENCE OF D-VALINE AS CHELATING AGENT " is presented by B.Sc (CHEMISTRY) students in partial fulfillment of the requirements for the degree of Bachelor of Science in Chemistry by the Tara Govt. College, Sangareddy(A) (Affiliated to Osmania University, Hyderabad) during the academic year 2021-2022.

The results embodied in this report have not been to any other University or Institution for the award of any degree.

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*Finally, we take this opportunity to thank one and all that has directly or indirectly helped me in completing the task.* 

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## <u>DECLARATION</u>

We hereby declare that the project report entitled "ADSORPTION STUDIES OF Fe[III] ON BENTONITE IN THE PRESENCE OF D-VALINE AS CHELATING AGENT" is the work done by us in the campus at Department of Chemistry, Tara Government College, Sangareddy(A)during the academic year 2021-2022 and is submitted in partial fulfillment of the requirements for the degree of Bachelor of Science by Tara Govt. College, Sangareddy(A) (Affiliated to Osmania University, Hyderabad) during the academic year 2021-2022.

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## **Chapter-I**

## **INTRODUCTION**

#### **ADSORPTION**

Adsorption is a process in which solids come into contact with liquids or gases, and the mass transfer occurs from liquids to solids. Desorption is the reversal of this action. Adsorption operations take advantage of a solid's capacity to concentrate certain chemicals from a fluid on to its surface. Adsorbate refers to the adsorbed substance, while adsorbent refers to the solid substance. The following are some examples of solid-liquid and solid-gas applications:

- Removing dissolved moisture from gasoline.
- Decolorization of sugar solutions and petroleum products.
- Removing noxious odors and tastes from water. Dehumidification of air and gases is one of the solid-gas activities.
- To fractionate mixtures of hydrocarbon gases such as methane, ethane, and propane.
- To remove undesirable odors and contaminants from gases.
- To recover valuable solvent vapors from dilute gas mixtures.

#### NATURE OF ADSORBENTS

Adsorbents are typically granular in nature, ranging in size from 0.5 mm to 12 mm. They can't have a lot of pressure decrease or get swept away by a fast-moving stream. During handling, they must maintain their shape and size. They'll need a lot of pores and a lot of surface area per unit mass.

Some of the commonly used adsorbents, their sources and applications are given below:

Sl. No.	Adsorbent	Source	Application
1.	Fuller's earth	Naturally occurring clay is heated and dried to get a porous structure.	De-colorizing, drying of lubricating oils, kerosene and engine oils.
2.	Activated charcoal	Bentonite or other activated clay which are activated by treatment with sulfuric acid and further washing, drying and crushing.	Used for de-colorizing petroleum products.
3.	Bauxite	A naturally occurring hydrated alumina, activated by heating at 230-815	Used for de-colorizing petroleum products and for drying gases.

4.	Alumina	A hard hydrated aluminum oxide, which is activated by heating to drive off the moisture and then crushed to desired size.	Used as desiccant.
5.	Bone-char	Obtained by destructive distillation of crushed bones at 600-900	Used for refining sugar and can be reused after washing and burning.
6.	Silica gel	A hard granular and porous product obtained from sodium silicate solution after treatment with acid. Normally has 4 to 7% water in the product.	Dehydration of gases and liquids, and separation of gas-liquid hydrocarbon mixture.

7.	Activated carbon	<ul> <li>(1) Vegetable matter is mixed with calcium chloride, carbonized and finally the inorganic compounds are leached away.</li> <li>(2) Organic matter is mixed with porous pumice stones and then heated and carbonized to deposit the carbonaceous matter throughout the porous particle.</li> <li>(3) Carbonizing substances like wood, sawdust, coconut shells, fruit pits, coal, lignite and subsequent activation with hot air steam. It is available in granular or pelleted form.</li> </ul>	De-colorizing of sugar solutions, chemicals, drugs, water purification, refining of vegetable and animal oils, recovery of gold and silver from cyanide ore-leach solution, recovery of solvent vapor from gas-mixtures, collection of gasoline hydro- carbons from natural gas, fractionation of hydrocarbon gases.
8.	Molecular sieves	These are porous synthetic zeolite crystals, metal alumino-silicates.	Dehydration of gases and liquids, and separation of gas- liquid hydrocarbon mixture.

#### SIGNIFICANCE OF RESEARCH PROBLEM

Adsorption of heavy metals is an important strategy to develop newer remediation technologies for the sustainable environmental protection. But the efficacy of adsorption of heavy metals under the normal conditions using suitable adsorbent depends on several factors which need to be finely tuned to get efficient adsorption process. In the presence of proper facilitating agents, the adsorption of heavy metals enhanced which will certainly improves the existing heavy metal techniques.

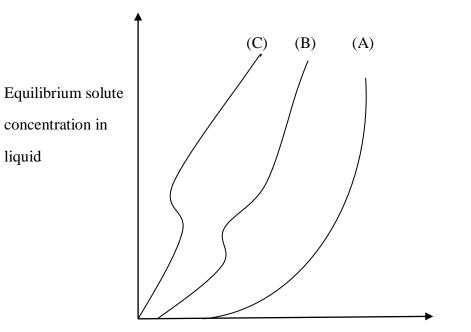
#### **OBJECTIVE OF THE PROJECT**

To evaluate the impact of D-Valine as a chelating agent in the adsorption of Fe (III) from aqueous solution by Bentonite as an adsorbent to develop efficient remediation technology using concept of Coordination chemistry.

#### **Chapter-II**

### ADSORPTION OF SOLUTES FROM DILUTE SOLUTIONS

Both the solvent and the solute are adsorbed whenever a mixture of solute and solvent is adsorbed using an adsorbent. As a result, only relative or apparent solute adsorption can be determined. As a result, treating a known volume of solution of original concentration Cwith a known weight of adsorbent is standard procedure. Let C\* be the solution's final equilibrium solute concentration. If v is the volume of solution per unit mass of adsorbent (cc/g), and C and C\* are the starting and equilibrium concentrations (g/cc) of the solute, then the apparent adsorption of the solute per unit mass of adsorbent is  $v(C - C^*)$ , (g/g), neglecting any volume change. This statement is most useful in the case of dilute solutions. The C\* value is determined by the temperature, nature, and properties of the adsorbent when the proportion of the original solvent that can be adsorbed is tiny. The Freundlich adsorption isotherm,  $C^*=K[v(C - C^*)]n$ , represents the adsorption phenomena in dilute fluids across a small concentration range. The Freundlich adsorption equation is especially useful in situations where the identification of the solute is unknown, such as the removal of coloring substances from sugar solutions, oils, and other liquids. A spectrophotometer or colorimeter can quickly determine the color composition of the solute. In worked example 2, the interpretation of this data is demonstrated. Adsorption is good if the value of n is high, say 2 to 10. If it's between 1 and 2, it's relatively challenging, and if it's less than 1, it's easy and it indicates poor adsorption characteristics. Freundlich adsorption equation is also useful in such a case where the actual identification of solute is not known, e.g., removal of coloring substance from sugar solutions, oils etc. A typical adsorption isothermal for the adsorption of various adsorbents A, B and C in dilute solution at the same temperature for the same adsorbent is represented in a graph.



Kg solute apparently adsorbed /kg adsorbent

Adsorption isotherms for various adsorbents

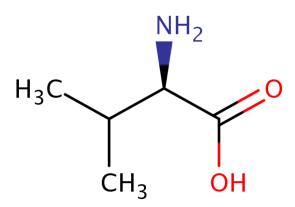
#### METAL COMPLEXES OF AMINO ACIDS

Transition metal amino acid complexes are a large family of coordination complexes containing the conjugate bases of the amino acids, the 2-aminocarboxylates. Amino acids are prevalent in nature, and all of them function as ligands toward the transition metals. Not included in this article are complexes of the amides (including peptide) and ester derivatives of amino acids. Also excluded are the polyamino acids including the chelating agents EDTA and NTA.

Most commonly, amino acids coordinate to metal ions as N,O bidentate ligands, utilizing the amino group and the carboxylate. They are "L-X" ligands. A five-membered chelate ring is formed. The chelate ring is only slightly ruffled at the sp<sup>3</sup>-hybridized carbon and nitrogen centers. For those amino acids containing coordinating substituents, the resulting complexes are more structurally diverse since these substituents can coordinate. Histidine, aspartic acid, methionine, and cysteine sometimes form tridentate N,N,O, N,O,O, S,N,O, and S,N,O complexes, respectively. Using kinetically inert metal ions,

complexes containing monodentate amino acids have been characterized. These complexes exist in either the N or the O linkage isomers. It can be assumed that such monodentate complexes exist transiently for many kinetically labile metal ions (e.g.  $Zn^{2+}$ ). Mixing simple metal salts with solutions of amino acids near neutral or elevated pH often affords bis- or tris complexes. For metal ions that prefer octahedral coordination, these complexes often adopt the stoichiometry  $M(aa)_3$  (aa = amino carboxylate, such as glycinate,  $H_2NCH_2CO_2^{-}$ ). Complexes of the 3:1 stoichiometry have the formula is  $[M(O_2CC(R)HNH_2)_3]^2$ . Such complexes adopt octahedral coordination geometry. These complexes can exist in facial and meridional isomers, both of which are chiral. The stereochemical possibilities increase when the amino acid ligands are not homochiral. Complexes with the 2:1 stoichiometry are illustrated by copper(II) glycinate  $[Cu(O_2CC(R)HNH_2)_2]$ , which exists both in anhydrous and pentacoordinate geometries. When the metal is square planar, these complexes can exist as cis and trans isomers. The stereochemical possibilities increase when the amino acid ligands are not homochiral. Homoleptic complexes are also known where the amino carboxylate is tridentate amino acids. One such complex is  $Ni(\kappa^3-histidinate)_2$ .

D-Valine can easily involved in M-L complex formation due to its functional flexibility. Moreover its water solubility encourages the metal-ligand complex formation under aqueous conditions.



**D-Valine** 

#### **BENTONITE AS ADSORBENT**

Because of heavy metals toxicity and non-biodegradable nature, the advent of heavy metals in water is turning into a critical environmental and public fitness concern. A variety of technology were evolved to do away with poisonous heavy metals from wastewater. The maximum crucial technology for the heavy steel ions elimination from wastewater consist of perception, ion exchange, adsorption, coagulation, evaporation and opposite osmosis. Adsorption on strong matrices has been proven to be an economically viable opportunity method (Abollino et al., 2003<sup>1</sup>; Hoda et al., 2009<sup>2</sup> Kapoor and Vira Raghavan, 1998<sup>3</sup>). Cheaper Na-bentonite has been observed to be so beneficial for elimination of heavy steel ions from aqueous answers that it has attracted geologists and environmental engineers (Al-Quadbit et al., 2005<sup>4</sup>; Guo et al.,(2009).

The Na-bentonite from Gaomiaozi has been used as boundaries to save you infection of wastewater containing heavy metals. For this cause it's far crucial to take a look at the adsorption of metals of metals via way of means of Na-bentonite so one can offer crucial parameters and essential principle for the knowledge of adsorptive elimination of heavy metals via way of means of Na-bentonite from an aqueous environment. The bivalence ions of copper and nickel are not unusual place observed withinside the commercial wastewater. The bivalence ions of copper and nickel are commonly found in the industrial wastewater. On a Na-montmorillonite turned into studied as feature of answer pH, dosage of Na-bentonite, temperature and make contact with and make contact with time. The aggressive isothermal Adsorption conduct of copper and nickel on Na-bentonite in single-aspect structures and binary-aspect structures has been investigated.



### **Chapter-III**

#### ATOMIC ABSORPTION SPECTROMETRY

Atomic absorption spectrometry (AAS) is an analytical technique that measures the concentrations of elements. Atomic absorption is so sensitive that it can measure down parts per billion of a gram (µg dm<sup>-3</sup>) in a sample. The technique makes use of the wavelengths of light specifically absorbed by an element. They correspond to the energies needed to promote electrons from one energy level to another, higher, energy level.

Atomic absorption spectrometry has many uses in different areas of chemistry.

Clinical analysis: Analysing metals in biological fluids such as blood and urine.

**Environmental analysis:** Monitoring our environment- eg finding out the levels of various elements in rivers, seawater, drinking water, air, petrol and drinks such as wine, beer and fruit drinks.

**Pharmaceuticals:** In some pharmaceutical manufacturing processes, minute quantities of a catalyst used in the process (usually a metal) are sometimes present in the final product. By using AAS the amount of catalyst present can be determined.

**Industry:** Many raw materials are examined and AAS is widely used to check that the major elements are present and that toxic impurities are lower than specified- eg in concrete, where calcium is a major constituent, the lead level should be low because it is toxic.

**Mining:** By using AAS the amount of metals such as gold in rocks can be determined to see whether it is worth mining the rocks to extract the gold.

#### HOW IT WORKS

Atoms of different elements absorb characteristic wavelengths of light. Analysing a sample to see if it contains a particular element means using light from that element. For example with lead, a lamp containing lead emits light from excited lead atoms that produce the right mix of wavelengths to be absorbed by any lead atoms from the sample. In AAS, the sample is atomized- i.e. converted into ground state free atoms in the vapour state- and a beam of electromagnetic radiation emitted from excited lead atoms is passed through the vaporized sample. Some of the radiation is absorbed by the lead atoms in the sample. The greater the number of atoms there is in the vapour, the more radiation is absorbed. The amount of light absorbed is proportional to the number of lead atoms. A calibration curve is constructed by running several samples of known lead concentration under the same conditions as the unknown. The amount the standard absorbs is compared with the calibration curve and this enables the calculation of the lead concentration in the unknown sample.

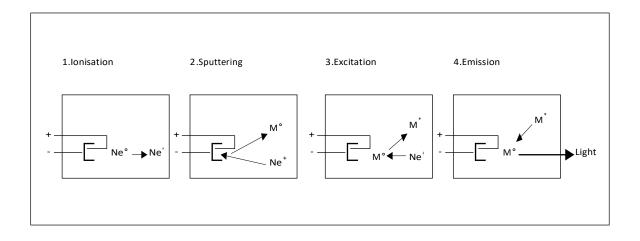
Consequently an atomic absorption spectrometer needs the following three components: a light source; a sample cell to produce gaseous atoms; and a means of measuring the specific light absorbed.

#### THE LIGHT SOURCE

The common source of light is a 'hollow cathode lamp'. This contains a tungsten anode and a cylindrical hollow cathode made of the element to be determined. These are sealed in a glass tube filled with an inert gas- e.g neon or argon- at a pressure of between 1 Nm<sup>-2</sup> and 5 Nm<sup>-2</sup>.

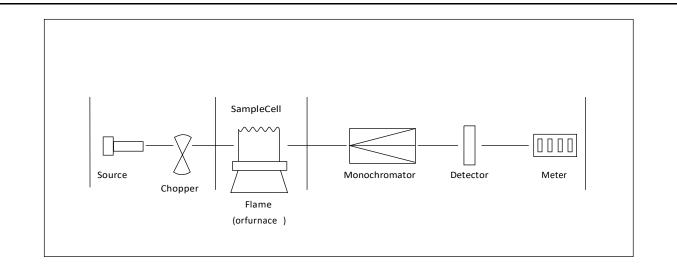


The ionization of some gas atoms occurs by applying a potential difference of about 300-400V between the anode and the cathode. These gaseous ions bombard the cathode and eject metal atoms from the cathode in a process called sputtering. Some sputtered atoms are in excited states and emit radiation characteristic of the metal as they fall back to the ground state  $-egPb^* \rightarrow Pb + h v$ . The shape of the cathode concentrates the radiation into a beam which passes through a quartz window, and the shape of the lamp is such that most of the sputtered atoms are redeposited on the cathode. A typical atomic absorption instrument holds several lamps each for a different element. The lamps are housed in a rotating turret so that the correct lamp can be quickly selected.



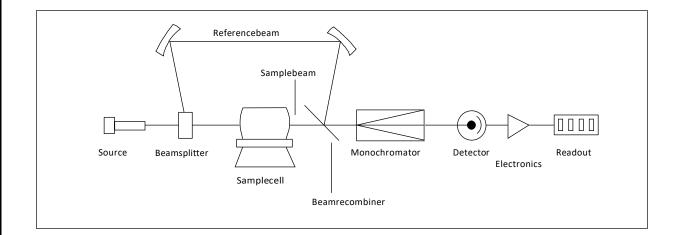
#### THE OPTICAL SYSTEM AND DETECTOR

A monochromator is used to select the specific wavelength of light –ie spectral line – which is absorbed by the sample, and to exclude other wavelengths. The selection of the specific light allows the determination of the selected element in the presence of others. The light selected by the monochromator is directed onto a detector that is typically a photomultiplier tube. This produces an electrical signal proportional to the light intensity



#### **DOUBLE BEAM SPECTROMETERS**

Modern spectrometers incorporate a beam splitter so that one part of the beam passes through the sample cell and the other is the reference. The intensity of the light source may not stay constant during an analysis. If only a single beam is used to pass through the atom cell, a blank reading containing no analyte (substance to be analysed) would have to be taken first, setting the absorbance at zero. If the intensity of the source changes by the time the sample is put in place, the measurement will be inaccurate. In the double beam instrument, there is a constant monitoring between the reference beam and the light source. To ensure that the spectrum does not suffer from loss of sensitivity, the beam splitter is designed so that as high a proportion as possible of the energy of the lamp beam passes through the sample.



#### ATOMIZATION OF THE SAMPLE

Two systems are commonly used to produce atoms from the sample. Aspiration involves sucking a solution of the sample into a flame; and electrothermal atomization is where a drop of sample is placed into a graphite tube that is then heated electrically.

Some instruments have both atomization systems but share one set of lamps. Once the appropriate lamp has been selected, it is pointed towards one or other atomization system.

#### FLAME ASPIRATION

Ethyne/air (giving a flame with a temperature of 2200–2400°C) or ethyne/dinitrogen oxide (2600–2800°C) are often used. A flexible capillary tube connects the solution to the nebulizer. At the tip of the capillary, the solution is 'nebulized' –ie broken into small drops. The larger drops fall out and drain off while smaller ones vaporize in the flame. Only ca 1% of the sample is nebulized.

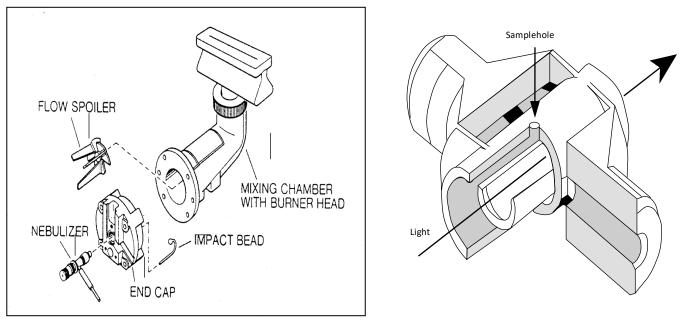


Figure 1

Figure 2

#### **ELECTROTHERMAL ATOMIZATION**

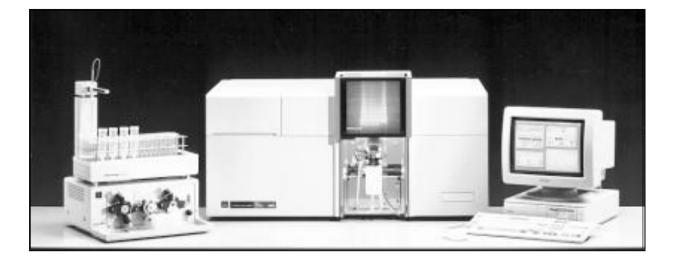
 $25 \ \mu$ l of sample (ca 1/100th of a raindrop) is placed through the sample hole and onto the platform from an automated micropipette and sample changer. The tube is heated electrically by passing a current through it in a pre-programmed series of steps. The details will vary with the sample but typically they

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might be 30–40 seconds at 150°C to evaporate the solvent, 30 seconds at 600°C to drive off any volatile organic material and char the sample to ash, and with a very fast heating rate (ca 1500 °C s<sup>-1</sup>) to 2000-2500°C for 5–10 seconds to vaporise and atomize elements (including the element being analysed). Finally heating the tube to a still higher temperature –ca 2700°C – cleans it ready for the next sample. During this heating cycle the graphite tube is flushed with argon gas to prevent the tube burning away. In electrothermal atomization almost 100% of the sample is atomised. This makes the technique much more sensitive than flame AAS.

#### SAMPLE PREPARATION

Sample preparation is often simple, and the chemical form of the element is usually unimportant. This is because atomization converts the sample into free atoms irrespective of its initial state. The sample is weighed and made into a solution by suitable dilution. Elements in biological fluids such as urine and blood are often measured simply after a dilution of the original sample.



When making reference solutions of the element under analysis, for calibration, the chemical environment of the sample should be matched as closely as possible –i.e., the analyte should be in the same compound and the same solvent. Teflon containers may be used when analysing very dilute Solutions because elements such as lead are sometimes leached out of glass vessels and can affect the results

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#### **BACKGROUND ABSORPTION**

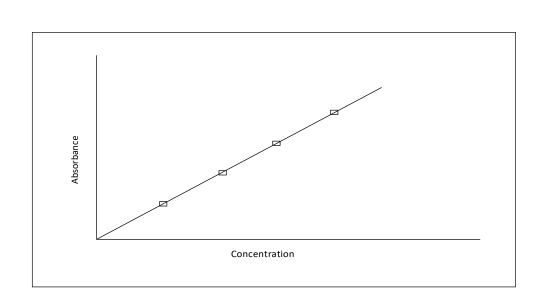
It is possible that other atoms or molecules apart from those of the element being determined will absorb or scatter some radiation from the light source. These species could include unvaporised solvent droplets, or compounds of the matrix (chemical species, such as anions, that tend to accompany the metals being analysed) that are not removed completely. This means that there is a background absorption as well as that of the sample.

One way of measuring and correcting this background absorption is to use two light sources, one of which is the hollow cathode lamp appropriate to the element being measured. The second light source is a deuterium lamp.

The deuterium lamp produces broad band radiation, not specific spectral lines as with a hollow cathode lamp. By alternating the measurements of the two light sources – generally at 50 - 100 Hz – the total absorption (absorption due to analyte atoms plus background) is measured with the specific light from the hollow cathode lamp and the background absorption is measured with the light from the deuterium lamp. Subtracting the background from the total absorption gives the absorption arising from only analyte atoms.

#### CALIBRATION

A calibration curve is used to determine the unknown concentration of an element -eg lead – in a solution. The instrument is calibrated using several solutions of known concentrations. A calibration curve is produced which is continually rescaled as more concentrated solutions are used – the more concentrated solutions absorb more radiation up to a certain absorbance. The calibration curve shows the concentration against the amount of radiation absorbed in the given figure. (a) The sample solution is fed into the instrument and the unknown concentration of the element-e.g., lead- is then displayed on the calibration curve given in the below figure. (b)





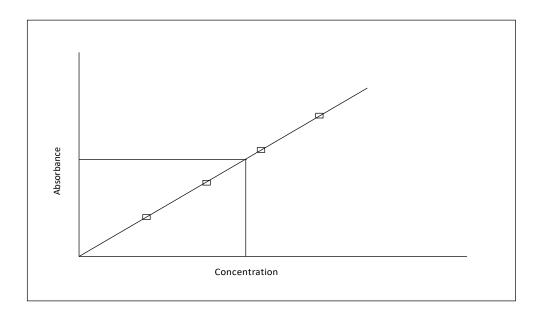


Figure (b)

## **Chapter-IV**

## METHODOLOGY

#### **MATERIALS REQUIRED**

- Ferric chloride Hexahydrate
- D-Valine
- Bentonite
- Volumetric flask
- Digital Weighing Machine
- Watch glasses
- Hot air oven
- Ultra-pure water (Demineralized)
- 2% of Nitric acid

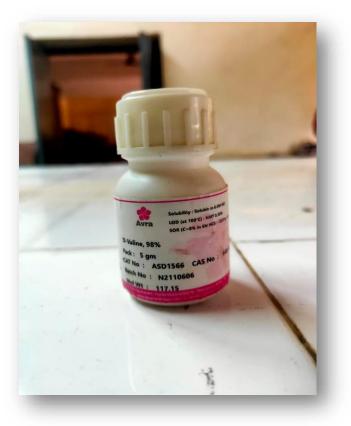
#### PROCEDURE

- Using a 250ml Volumetric flask prepare a metal ligand solution by adding 100ppm of Ferric chloride Hexahydrate (MOLYCHEM MCR-11580) and 200ppm of L-Valine (AVRAN2110606).
   Prepare 250ml solution by adding Ultra-pure water and then keep this system aside for a few hours.
- Weigh 5 grams of Bentonite (AVRA N2101070) using a Digital weighing machine (Citizen Scales(I) PVT LTD CTG302-300) and take this into a beaker.
- **3.** Now add 100ml of the above prepared metal ligand solution into the beaker and stir the mixture well for 10 minutes using a glass rod.
- **4.** Keep this mixture aside for 48 hours without disturbing it as at this step Ferric is going to be adsorbed on Bentonite in the presence of D-Valine which acts as a chelating agent.
- **5.** After completion of 48 hours take the mixture and filter it off using Whatman Grade 1 filter paper and a funnel.

- **6.** After filtration of the mixture again add Ultra-pure water for 3 times and then filtrate it to obtain pure concentration of Ferric solution which is get adsorbed on Bentonite.
- Collect the filtered Bentonite powder and place it on a watch glass and keep this in a Hot air Owen at 60 °C for 10 hours to get rid of moisture present in it.
- 8. Now weigh each 1 gm of Bentonite in glass vials.
- 9. Now take a beaker and rinse it with ultra-pure water then followed by Nitric acid.
- **10.** Take 0.5 grams of Bentonite sample in the beaker and add 2% of Nitric acid and stir the mixture well for 10-15 minutes.
- **11.** Filter the mixture using Whatman Grade 1 filter paper and again 3 times by using Ultra-pure water to obtain pure concentration of Ferric present in the mixture prepared using the sample.
- **12.** Take this collected sample solution and keep this system under AAS (Thermos Scientific iCE 3300)
- **13.** Calculate the concentration of Ferric adsorbed on Bentonite at different ppm levels. Observe the graph obtained and note down the readings of the result we obtained.
- 14. Same Experiment carried out without the interference/addition of Ligand i.e. D-Valine for Control Experiment.



## **Bentonite Powder**



## L-VALINE



## FERRIC CHLORIDE (HEXAHYDRATE) FeCL<sub>3</sub>6H<sub>2</sub>O

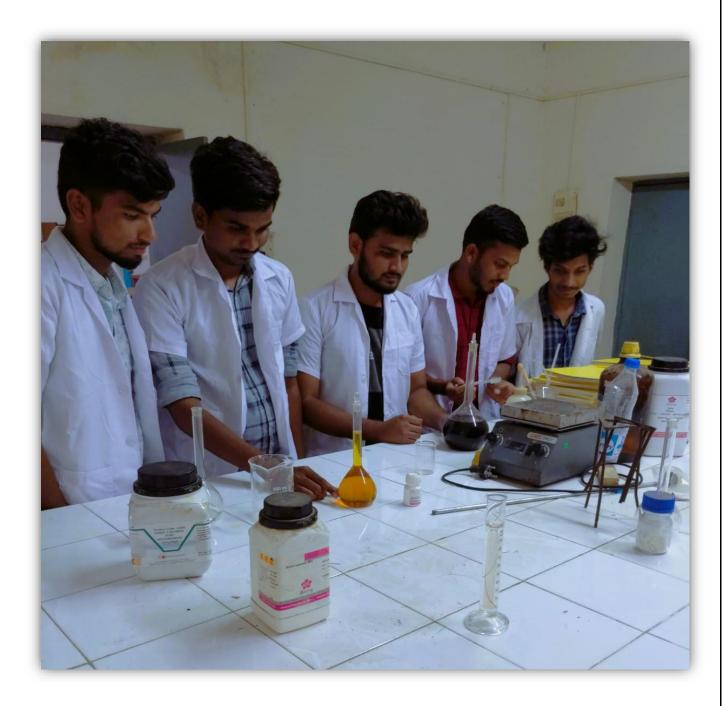


**Measuring Flask** 





Preparing metal ligand solution





## 5 grams of bentonite added to 100ml of metal ligand solution



Filtering The Mixture using Whatman grade 1 filter paper and Again, three times by using Ultra-Pure water







Hot air oven



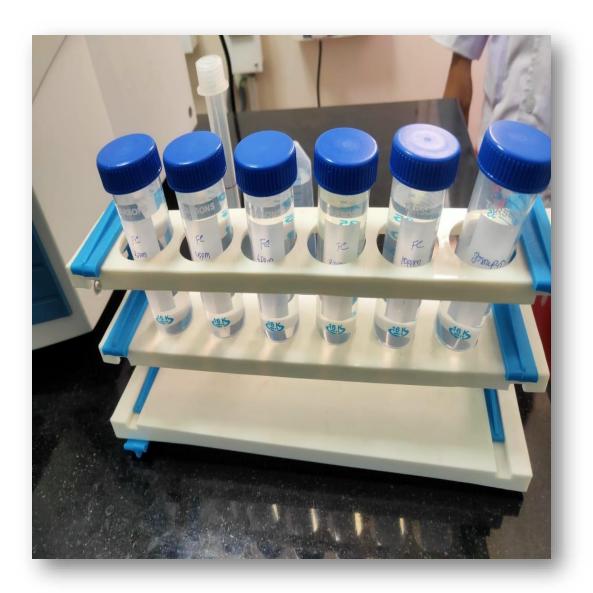
# **Glass vials**



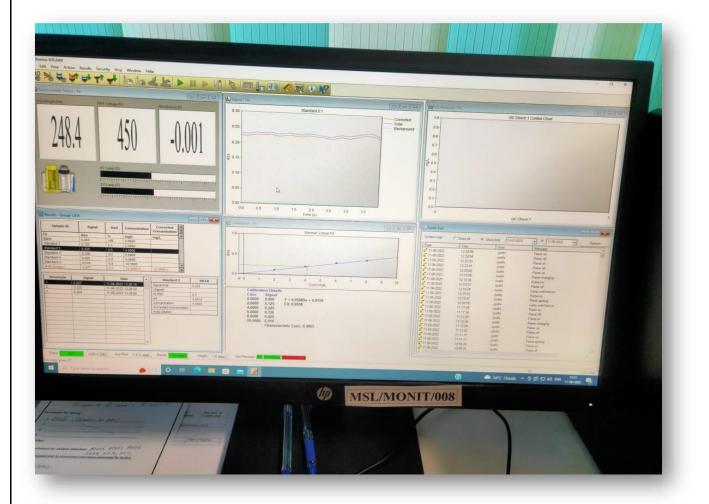
**Collected samples** 



AAS (Atomic Absorption Spectrometry)



Sample solution added to 2% of nitric acid

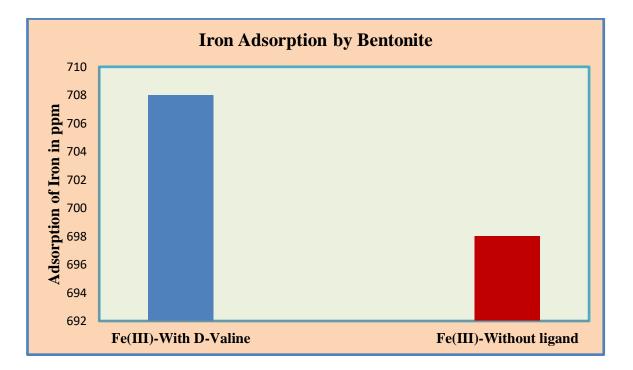


## **Results obtained on the monitor under AAS Method**

### **Chapter-V**

### **RESULTS AND DISCUSSIONS**

Bentonite adsorbs **708.0ppm** of Iron metal from aqueous solution of Fe(III)-D-Valine metal ligand solution. Whereas, Bentonite adsorbs only **698.0ppm** when D-Valine is absent. It is evident from the AAS results, ligand involvement enhanced the metal adsorption by initiating potential chemical interactions between adsorbate and adsorbent. D-Valine firmly coordinates with Fe(III) to form a stable complex in aqueous condition. The complex coordination sphere in the resulted complex facilitates strong interactions with the polar points of Bentonite. From the AAS results, it is conclusive that **1.432%** of adsorbtion increased in the presence of D-Valine as chelating agent.



Impact of D-Valine on Adsorption of Fe (III) ions from aqueous solution by Bentonite.

## <u>Spectrometer Parameters</u> – <u>Fe</u>:

Element : Fe	Measurement mode : Absorbance	
Wavelength : 248.3nm	Band pass : 0.2nm	Lamp current : 75%
Background correction :	High Resolution : Off	Optimise Spectrometer
D2		Parameters : No
Signal type : continuous	Resamples : Fast	Number of resamples : 3
Measurement time :	Flier mode : No	
4.0secs		
Use RSD Test : No		

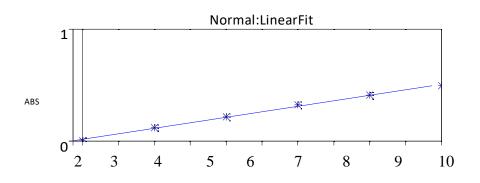
### <u>Flame parameters – Fe:</u>

Flame type : Air – C2H2	Fuel Flow : 0.9L/min	Auxiliary Oxidant : Off
Nebuliser Uptake : 4secs	Bunsen Stabilisation : Omins	Optimise Fuel Flow : No
Burner Height :7.0mm	Optimise Burner fuel : No	

## **<u>Calibration Parameters</u> – <u>Fe</u>:**

Calibration mode : Normal	Line fit : Linear	Use stored calibration : No
Concentration units : mg/L	Scales units : mg/L	Scaling factor : 1.0000
Acceptable fit : 0.990	Rescale Limits : 10.0%	Failure Action : Flag and Continue
Standard 1 - 2.0000	Standard 4 - 8.0000	
Standard 2 - 4.0000	Standard 5 - 10.0000	
Standard 3 - 6.0000		

**Solutions Results-Fe:** 



Y = 0.04924x + 0.0141

Fit: 0.9965

Characteristic Conc.: 0.0894

Sample ID	Signal	RSD	Conc.
	Abs	%	Mg/L
Fe Blank	0	<99	0
1	0	В	ackground: 0.000
2	0	В	ackground: 0.000
3	0	В	ackground: 0.000
Fe Standard 1	0.125	0.	3 2
1	0.125	В	ackground: 0.006
2	0.125	В	ackground: 0.006
3	0.126	В	ackground: 0.006
Fe Standard 2	0.225	0.	5 4
1	0.227	В	ackground: 0.007
2	0.225	В	ackground:0.007

2	0.702		_	ground: 0.005 ground: 0.005	
1	0.700		_	ground: 0.005	
Fe(III)-Bentonite (Without Ligand)	0.701	0.1		16.4138 C	
3	0.712	Background: 0.004			
2	0.713	Background: 0.005			
1	0.709		Back	ground: 0.005	
Fe(III)-D-Valine-Bentinite-	0.711	0.1	1	16.6490 C	
3	0.513	3 Background: 0.009			
2	0.509	9 Background: 0.009			
1	0.51		Backg	ground: 0.009	
Fe Standard 5	0.51		0.4		10
3	0.425		Backg	ground: 0.009	
2	0.425		Backg	ground: 0.008	
1	0.426		Backg	ground: 0.009	
Fe Standard 4	0.425		0.2		8
3	0.335		Backg	ground: 0.008	
2	0.336		Backg	ground: 0.008	
1	0.336		Backg	ground: 0.008	
Fe Standard 3	0.336		0.2		6
3	0.224		Backg	ground:0.007	

## TEST RESULTS:

S.No.	<b>Test Parameters</b>	Sample	Results
01.	Iron by AAS	Fe(III)- D-Valine-	708.0ppm
	Analysis: (ppm)	Bentonite Sample	
02.	Iron by AAS	Fe(III)-Bentonite	698.0ppm
	Analysis: (ppm)	(control)	

#### **CONCLUSION**

From the current project it is clear that D-Valine as a Chelating agent has played a vital role in adsorption of Fe(III) ions from aqueous solution and increases the adsorption up to **1.432** % using Bentonite. This aspect will be useful in designing the newer strategies of Heavy metal Remediation techniques using organic bifunctional Chelating Ligands as Facilitating agents in Metal Adsorption processes.

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## MART SPECIALITIES LAB LLP.

Operator Name: Jyothi Results File: E:\AAS System Data\2022\JUNE\11 06 2022\TARA 0064 2-6\Iron (Fe)1.SLR

#### **General Parameters**

Method : Iron (Fe) Autosampler : None Use SFI: No Operator : Jyothi

*Instrument Mode:* Flame *Dilution:* None

mA Hours

n/a

Report Date: 11-06-2022 17:40:20

#### Valid Method Signatures

11-06-2022 17:22:33 jyothi(M. Jyothi):DESKTOP-39TDEGC Signed with Reason : Analysed by:
11-06-2022 17:23:24 parjanya(Parjanya):DESKTOP-39TDEGC Signed with Reason : Approved by:

### **Method Audit Trail**

11-06-2022 17:22:18 Jyothi(M. Jyothi):DESKTOP-39TDEGC Record created
11-06-2022 17:22:33 jyothi(M. Jyothi):DESKTOP-39TDEGC Signed with Reason : Analysed by:
11-06-2022 17:23:24 parjanya(Parjanya):DESKTOP-39TDEGC

Signed with Reason : Approved by:

#### **Analysis Details**

Spectrometer: iCE 3000 AA01204906 v1.30

Analysis Name: Iron (Fe) 11-06-2022 Operator Name: Jyothi

n/a

Lamp Information Serial Number

Element(s) Fe

. . .

Deuterium Lamp Hours: 68.34

### Sequence Table

Shared Standards: Yes	
Action	Fe
Calibration	√
B.No.Group-II	√
B.No.Group-III	√
B.No.Group-IV	√
B.No.Group-V	√
B.No.Group-VI	✓

### **Sample Details**

		Nominal Mass: 1.0000				
No.	Sample Id	Sample Mass	Dilution Ratio			
1	B.No.Group-II	1.0000	1.0000			
2	B.No.Group-III	1.0000	1.0000			
3	B.No.Group-IV	1.0000	1.0000			
4	B.No.Group-V	1.0000	1.0000			
5	B.No.Group-VI	1.0000	1.0000			

## Valid Analysis Signatures

11-06-2022 17:38:41 jyothi(M. Jyothi):DESKTOP-39TDEGC Signed with Reason : Analysed by:
11-06-2022 17:39:33 parjanya(Parjanya):DESKTOP-39TDEGC Signed with Reason : Approved by:

### **Analysis Audit Trail**

- 11-06-2022 17:30:16 Jyothi(M. Jyothi):DESKTOP-39TDEGC Record created
- 11-06-2022 17:38:27 Jyothi(M. Jyothi):DESKTOP-39TDEGC Error MD147 - Activity manually aborted by user.
- 11-06-2022 17:38:41 jyothi(M. Jyothi):DESKTOP-39TDEGC Signed with Reason : Analysed by:
- 11-06-2022 17:39:33 parjanya(Parjanya):DESKTOP-39TDEGC Signed with Reason : Approved by:

## MART SPECIALITIES LAB LLP.

Operator Name: Jyothi Results File: E:\AAS System Data\2022\JUNE\11 06 2022\TARA 0064 2-6\Iron (Fe)1.SLR

#### **Spectrometer Parameters - Fe**

Element: Fe Wavelength: 248.3nm Background Correction: D2 Signal Type: Continuous Measurement Time: 4.0secs Use RSD Test: No

Flame Type: Air-C2H2 Nebuliser Uptake: 4secs Burner Height: 7.0mm

Sampling: None

Calibration Mode: Normal	
Concentration Units: mg/L	
Acceptable Fit: 0.990	
Standard 1	2.0000
Standard 2	4.0000
Standard 3	6.0000

No changes are recorded for this element

Measurement Mode: Absorbance Bandpass: 0.2nm High Resolution: Off Resamples: Fast Flier Mode: No

#### Lamp Current: 75% Optimise Spectrometer Parameters: No Number Of Resamples: 3

### Flame Parameters - Fe

Fuel Flow: 0.9L/min Burner Stabilisation: 0mins Optimise Burner Height: No Auxilary Oxidant: Off Optimise Fuel Flow: No

## Sampling Parameters - Fe

#### **Calibration Parameters - Fe**

Line Fit: Linear Scaled Units: mg/L Rescale Limit: 10.0% Standard 4 Standard 5 Use Stored Calibration: No Scaling Factor: 1.0000 Failure Action: Flag and Continue 8.0000 10.0000

### Element Audit Trail - Fe

Solution Results - Fe

Y = 0.04924x + 0.0141 Fit: 0.9965 Characteristic Conc: 0.0894

> 1 2 3

> 1 2 3

> 1 2 3

> 1 2 3

1 2

3

Sample ID

Fe Blank

Fe Standard 1

Fe Standard 2

Fe Standard 3

Fe Standard 4

	4				Norma	I: Linea	ar Fit				
	Abs		*		*		*		-*-		*
	0 <del>  *</del> -0-0	1	2	3	4	5	6	7	8	9	10
	-0-0		2	0		nc:mg/		,	0	0	10
Signal	Rsd	Conc				cted C					
Abs	%	mg/L			mg/L						
0.001	35.6	0.000	)		Ũ						
0.001	Backgro	ound: -0.0	03			11-06	-2022 1	7:31:26	i		
0.001	Backgro	ound: -0.0	03			11-06	-2022 1	7:31:30	)		
0.000	Backgro	ound: -0.0	03			11-06	-2022 1	7:31:35			
0.120	0.3	2.0000	)								
0.120	Backgro	ound: 0.00	)3			11-06	-2022 1	7:32:05			
0.119	-	ound: 0.00						7:32:09			
0.120	Backgro	ound: 0.00	)3			11-06	-2022 1	7:32:13			
0.215	0.3	4.0000	)								
0.215	Backgro	ound: 0.00	)4			11-06	-2022 1	7:32:40	)		
0.215	Backgro	ound: 0.00	)4			11-06	-2022 1	7:32:45			
0.216	Backgro	ound: 0.00	)4			11-06	-2022 1	7:32:49	)		
0.322	0.2	6.0000	)								
0.323	Backgro	ound: 0.00	)5			11-06	-2022 1	7:33:19	)		
0.322	Backgro	ound: 0.00	)5			11-06	-2022 1	7:33:23			
0.322	Backgro	ound: 0.00	)5			11-06	-2022 1	7:33:27			
0.411	0.3	8.0000	)								
0.410	Backgro	ound: 0.00	)5			11-06	-2022 1	7:33:59	)		
0.412	Backgro	ound: 0.00	)5			11-06	-2022 1	7:34:03			
0.412	Backgro	ound: 0.00	)5			11-06	-2022 1	7:34:07			

## MART SPECIALITIES LAB LLP.

Operator Name: Jyothi

Results File: E:\AAS System Data\2022\JUNE\11 06 2022\TARA 0064 2-6\Iron (Fe)1.SLR

#### **Solution Results - Fe**

Sample ID	Signal	Rsd	Conc	Corrected Conc
-	Abs	%	mg/L	mg/L
Fe Standard 5	0.493	0.1	10.0000	
1	0.494	Backg	ground: 0.005	11-06-2022 17:34:38
2	0.494	Backg	ground: 0.005	11-06-2022 17:34:42
3	0.493	Backg	ground: 0.006	11-06-2022 17:34:46
Fe B.No.Group-II	0.834	0.1	16.6490 C	16.6490 C
1	0.834	Backg	ground: 0.005	11-06-2022 17:35:14
2	0.833	Backg	ground: 0.005	11-06-2022 17:35:19
3	0.834	Backg	ground: 0.005	11-06-2022 17:35:23
Fe B.No.Group-III	0.759	0.1	15.1286 C	15.1286 C
1	0.758	Backg	ground: 0.003	11-06-2022 17:35:52
2	0.760	Backg	ground: 0.003	11-06-2022 17:35:56
3	0.758	Backg	ground: 0.004	11-06-2022 17:36:00
Fe B.No.Group-IV	0.645	0.3	12.8189 C	12.8189 C
1	0.647	Backg	ground: 0.003	11-06-2022 17:36:31
2	0.644	Backg	ground: 0.003	11-06-2022 17:36:35
3	0.645	Backg	ground: 0.003	11-06-2022 17:36:40
Fe B.No.Group-V	0.809	0.2	16.1488 C	16.1488 C
1	0.808	Backg	ground: 0.005	11-06-2022 17:37:11
2	0.809	Backg	ground: 0.005	11-06-2022 17:37:15
3	0.811	Backg	ground: 0.005	11-06-2022 17:37:20
Fe B.No.Group-VI	0.711	0.3	14.1597 C	14.1597 C
1	0.709	Backg	ground: 0.005	11-06-2022 17:37:55
2	0.713	Backg	ground: 0.005	11-06-2022 17:37:59
3	0.712	Backg	ground: 0.004	11-06-2022 17:38:03

Work : Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



DCA Approval No: 05/ML/TS/2020/G	CERTIFICATE OF ANALYSIS MSL/QA/017-03/F07-00
Name & Address of the Customer: Tara Government College Prashanth Nagar Colony, Balajinagar Sangareddy Telangana. 502000 Contact Person: Dr. Abhijeet Contact Number :9502344392	Reference / Report No.       :       MSL/2022/JUNE/TARA/0064-1         Sample Received Date       :       11/06/2022         Report Date       :       13/06/2022
I	DETAILS OF THE SAMPLE
Sample Name : NA	
Name of the Manufacturer : NA	
Storage condition To b	up-I Mfg. : NA Exp. : NA e stored at room date date
	Derature Batch : NA C±3°C size
	by AAS Analysis.
	6/2022
Analysis Completion Date : 11/0	6/2022
Mfg. License No. : Not	provided
A.R.NO : NA	The American Company

### **Test Results**

S.No.	Test Parameter	Result
01.	Iron by AAS Analysis: (ppm)	620ppm

Authorized Signatory

Work : Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



DCA Approval No: 05/ML/TS/2020/G	
	CERTIFICATE OF ANALYSIS MSL/QA/017-03/F07-00
Name & Address of the Customer: Tara Government College Prashanth Nagar Colony, Balajinagar Sangareddy Telangana. 502000 Contact Person: Dr. Abhijeet Contact Number :9502344392	Reference / Report No.       : MSL/2022/JUNE/TARA/0064-2         Sample Received Date       : 11/06/2022         Report Date       : 13/06/2022
Q	DETAILS OF THE SAMPLE
Sample Name : N	A
Name of the Manufacturer : N	A
Storage condition : To	roup-II Mfg. : NA Exp. : NA be stored at room date date
Room Temperature : 25	Batch : NA size
Tests Required:IreMethod:NAnalysis Starting Date:11	on by AAS Analysis.
	ot provided
A.R.NO : N	A

#### **Test Results**

S.No.	Test Parameter	Result
01.	Iron by AAS Analysis: (ppm)	832.5ppm

Authorized Signatory

**Work :** Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



OCA Approval No: 05/ML/TS/2020/G	CERTIFICATE OF ANALYSIS MSL/QA/017-03/F07-00
Name & Address of the Customer: Tara Government College Prashanth Nagar Colony, Balajinaga Sangareddy Telangana. 502000 Contact Person: Dr. Abhijeet Contact Number :9502344392	Sample Received Date : 11/06/2022
a.d.	DETAILS OF THE SAMPLE
Sample Name : N	JA
Name of the Manufacturer : N	JA
Storage condition	Group-III Mfg. : NA Exp. : NA To be stored at room date date Cemperature Batch : NA
	5°C±3°C size
	gm /
Method : I Analysis Startin <mark>g Date : I</mark>	ron by AAS Analysis. NA 1/06/2022 1/06/2022
and the second se	Not provided
A.R.NO	NA
Remark : S	Sample analyzed as received

#### **Test Results**

S.No.	Test Parameter	Result
01.	Iron by AAS Analysis: (ppm)	756.5ppm

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Authorized Signatory

(Dr.R.Marayya)

Office: 13-6-438/A/83, Sri Satyanarayana Nagar, Mehdipatnam, Hyderabad - 500 028, Telangana State, India Mobile: 98481 98350, 98481 25019 Mail: rmarayya@yahoo.com

**Work :** Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



OCA Approval No: 05/ML/TS/2020/G	
	CERTIFICATE OF ANALYSIS MSL/QA/017-03/F07-00
Name & Address of the Customer: Tara Government College	Reference / Report No. : MSL/2022/JUNE/TARA/0064-4
Prashanth Nagar Colony, Balajinagar Sangareddy Telangana. 502000	Sample Received Date: 11/06/2022Report Date: 13/06/2022
Contact Person: Dr. Abhijeet Contact Number :9502344392	
	DETAILS OF THE SAMPLE
Sample Name : N	A
Name of the Manufacturer : N	A
Storage condition : To	roup-IVMfg. : NAExp. : NAb be stored at roomdatedateemperatureBatch : NA
	s°C±3°C size
Method : N Analysis Starting Date : 11	on by AAS Analysis. A //06/2022 //06/2022
Mfg. License No: N	ot provided
A.R.NO : N	A
Remark : Sa	imple analyzed as received

#### **Test Results**

	A COLORED TO A COL	pristance of	
S.No.	Test Parameter	Result	
01.	Iron by AAS Analysis: (ppm)	641ppm	

Authorized Signatory

**Work :** Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



Name & Address of the Customer:Tara Government CollegePrashanth Nagar Colony, Balajinagar	ERTIFICATE OF ANALYSIS       MSL/QA/017-03/F07-00         Reference / Report No.       :       MSL/2022/JUNE/TARA/0064-5         Sample Received Date       :       11/06/2022         Report Date       :       13/06/2022
Tara Government CollegeSPrashanth Nagar Colony, BalajinagarS	Sample Received Date : 11/06/2022
Prashanth Nagar Colony, Balajinagar	
Prashanth Nagar Colony, Balajinagar	
Sangareddy Telangana. 502000	Report Date : 15/00/2022
Contact Person: Dr. Abhijeet Contact Number :9502344392	
DF	ETAILS OF THE SAMPLE
Sample Name : NA	
Name of the Manufacturer : NA	
Storage condition To be	o-V Mfg. : NA Exp. : NA stored at room date date erature Batch : NA
Room Temperature : 25°C±	elatare
Quantity Received : 4gm	
Tests Required:Iron bMethod:NAAnalysis Starting Date:11/06/	y AAS Analysis. /2022
Analysis Completion Date : 11/06/	
	rovided
A.R.NO : NA	And the second second
Remark : Samp	le analyzed as received

#### **Test Results**

S.No.	Test Parameter	Result
01. Ir	on by AAS Analysis: (ppm)	807.5ppm

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Authorized Signatory

(Dr.R.Marayya)

**Office :** 13-6-438/A/83, Sri Satyanarayana Nagar, Mehdipatnam, Hyderabad - 500 028, Telangana State, India Mobile : 98481 98350, 98481 25019 Mail : rmarayya@yahoo.com

**Work :** Road No. : 03, Plot No.: 31, Ground Floor & 1<sup>st</sup> Floor, Gajularamaram, ALEAP Industrial Area, Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India, **Tel.** : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com



DCA Approval No: 05/ML/TS/2020/G	
	CERTIFICATE OF ANALYSIS MSL/QA/017-03/F07-00
Name & Address of the Customer: Tara Government College Prashanth Nagar Colony, Balajinagar Sangareddy Telangana. 502000 Contact Person: Dr. Abhijeet Contact Number :9502344392	Reference / Report No.       : MSL/2022/JUNE/TARA/0064-6         Sample Received Date       : 11/06/2022         Report Date       : 13/06/2022
1	DETAILS OF THE SAMPLE
Storage condition To b	up-V1 Mfg. : NA Exp. : NA be stored at room date date perature Batch : NA C±3°C size
Tests Required:IronMethod:NAAnalysis Starting Date:11/0Analysis Completion Date:11/0Mfg. License No.:NotA.R.NO:NA	by AAS Analysis. 06/2022 06/2022 provided

#### **Test Results**

S.No.	Test Parameter	Result
01.	Iron by AAS Analysis: (ppm)	708ppm

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Authorized Signatory