

# Comparison of Spectrophotometric Methods for Determination of Iron in Acid Cleaning, and Passivating Stainless Steel Solution by UV-VIS, ICP/MS, and FAAS

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**Abstract:** Generally, more sophisticated techniques such as, flame atomic absorption (AAS), inductively coupled plasma spectrometer (ICP-MS), and UV/Visible Spectrophotometer were used to determine trace iron metals in pickling (acid cleaning), and passivating stainless steel solutions. Simple and novel spectrophotometric methods are described for simultaneous determination of iron. While these techniques provide excellent sensitivity and selectivity. The results were found to be in satisfactory agreement (shows no significant difference) with those acquired by the flame atomic absorption spectrophotometer (FAAS), UV-Vis Spectrometer, and ICP/MS techniques. The results shows that the concentration of iron are very close each other (less than 5.0%) in the pickling and passivating solutions.

**Keywords:** Acid Cleaning (Pickling) Solution, Passivating Solution, Iron Determination, ICP-MS, UV-VIS Spectrophotometer, and FAAS

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## 1. Introduction

It is well known that iron is found in surface and drinking water and plays a vital role in biological systems and also it is the most abundant metal in the human body. Many proteins use iron for oxygen transport, electron transport, and as a catalyst in oxido-reductase reactions [1]. An excess of iron in a living cell can catalyze the production of reactive oxygen via the Fenton reaction, which damage lipids, nucleic acids, and proteins. Serious diseases such as Alzheimer's, Huntington's and Parkinson's result from the toxicity of iron ion [2, 3], so the concentration of iron becomes a parameter for the quality of drinking water [4].

Metal surface can contain impurities that may affect usage of the product or further processing like plating with metal or painting. Various chemical solutions are usually used to clean these impurities. Strong acids, such as hydrochloric acid and

sulfuric acid are common, but different applications use various other acids. Also *alkaline* solutions can be used for cleaning metal surfaces. Solutions usually contain additives such as wetting agents and corrosion inhibitors. Pickling is sometimes called "acid cleaning" if descaling is not needed [5, 6].

Several analytical methods have been used for the determination of iron in aqueous solutions.

In this present work, a chromatic reagent of 1,10 phenanthroline is used for the determination of iron Fe (III) based on pickling and passivating stainless steel solutions by UV-Vis spectrometry, A Perkin Elmer 5100 PC atomic absorption spectrophotometer with deuterium arc background correction, and Inductively Coupled Plasma Spectrometer (ICP-MS) were used for determining of iron (Fe) and comparing the results.

### 1.1. Acid Cleaning (Pickling)

Pickling is a metal surface treatment process that removes impurities, such as stains, inorganic contaminants, rust or scale from ferrous metals, copper, precious metals and aluminum alloys [7]. A solution called “*pickling*” because it involves the use of an acidic solution known as *pickle liquor* [8], which usually contains acid, is used to remove the surface impurities. Mixture of nitric acid and hydrofluoric acids are usually used for pickling stainless steels. Stainless steel is composed of iron (Fe), nickel (Ni), chromium (Cr), and several other minor components. Stainless steel is not resistant to chemical or physical attack. The corrosion resistance of stainless steel depends on the formation of a “passive surface film” composed of nickel and chromium oxides (Cr<sub>2</sub>O<sub>3</sub>&NiO). Pickling involves metal removal and a charge or dulling in the visual brightness of the metal [9, 10]. Pickling normally involves using an acid mixture containing 8-20 vol% nitric acid (HNO<sub>3</sub>) and 0.5-5 vol% hydrofluoric acid (HF). Chloride containing agents such as hydrochloric acid (HCl) should be avoided [11, 12].

### 1.2. Passivating Solution

According to ASTM A 380, passivation is “the removal of exogenous iron or iron compounds from the surface of stainless steel by means of a chemical dissolution, most typically by a treatment with an acidic solution that will remove the surface contamination but will not significant effect the stainless steel itself.” [12]. Passivating usually occurs naturally on the surfaces of stainless steels, but it may sometimes be necessary to assist the process with oxidizing acid treatments.

Passivating is the process by which a stainless steel will spontaneously form a chemically inactive surface when exposed to air or other oxygen-containing environments.

Passivating is the chemical treatment of a stainless steel with a mild oxidant, such as a nitric acid solution, for the purpose of enhancing the spontaneous formation of the protective film [12].

#### Safety

Pickling (acid cleaning) and passivating solutions use strong acids, and normal precautions for safety should be followed. All iron solutions should be discarded into a “Heavy Metals” waste container.

## 2. Materials and Method

### 2.1. Determination of Iron by ICP/MS

#### 2.1.1. Instruments

- 1) NexIon 300X ICP-MS, Inductively Coupled Plasma computer-controlled sequential emission spectrometer with interelement and background correction capabilities, and provisions for interfacing to a printer and an auto sampler.
- 2) Ethos Plus Microwave
- 3) ETHOS One Closed Vessel Microwave Digestion

System, with temperature control and rotating turntable, well ventilated with corrosion-resistant cavity.

- 4) Microwave digestion vessels for water samples, Teflon, capable of holding ~75 milliliters (mL), designed " for temperatures up to 260 C with self-regulating pressure control
- 5) Digestion vessels for soil samples, capable of holding ~250 mL
- 6) Watch glasses or vapor recovery device
- 7) Glass dispensers, 2-liter (L), 1-L, or 1-gallon, checked quarterly for accuracy
- 8) Graduated Cylinder, Class A, 50 mL
- 9) Volumetric flasks, Class A, assorted volumes
- 10) Balance, top-loading, capable of reading to 0.01 grams (g), for weighing digestion vessels before and after digestion
- 11) Henke SASS plastic syringes or equivalent
- 12) Corning SCFA 0.45 microns (μm) filters or equivalent
- 13) Argon Plasma Support Gas in pressurized cylinders.

#### 2.1.2. Reagents and Solutions

- 1) Concentrated nitric acid, Seastar Chemicals. 67-70% (w/w HNO<sub>3</sub>), purified by re-distilled, ≥99.999% trace metals basis.
- 2) Nitric acid, 2 percent (%) volume to volume (v/v), for the preparation of working standards, also to be used for the initial calibration blank.
- 3) Hydrogen peroxide solution contains inhibitor, were used in all procedures. 30 wt.% in H<sub>2</sub>O, Sigma-Aldrich, ACS grade.
- 4) Pickling (Acid Cleaning) Stainless Steel Solution
- 5) Passivating Stainless Steel Solution
- 6) Deionized water, on the day of use. High-purity deionized water was obtained by Aries High Purity Water System, Aries Filter Works. Type I Deionized water, for the preparation of all reagents and calibration standards and as dilution water. Calibration curves were constructed using commercial Fe standards from analysis.

#### 2.1.3. Microwave Digestion

##### Sample Preparation

200 mg of sample weighed and placed in each digestion vial. 8 mL of HNO<sub>3</sub> nitric acid and 2 mL Hydrogen peroxide have been added to each vial including a blank. Tighten the vials in the vessels, twist the screw on top by hand and then use the teardrop ratchet to tighten further.

No sample was added in a vessel for the blank preparation. For the blank, line up the holes and ensure that the tube goes in. Place them in a microwave. After digestion; take out the vials from the microwave. The teardrop ratchet was used to loosen the vials from the vessels.

All the liquid from each digestion vial were transferred into 50 mL centrifuge tubes. About 10 mL Deionized (DI) water, Type I Water was added to wash and rinse the vials to make the total volume 20 mL using a 5 mL pipette. Digested sample were diluted with 1:10, 1:100, and 1: 1000 with 18.2 MΩ-cm H<sub>2</sub>O and iron standards [13].

Table 1. Intensity of Fe by ICP/MS.

Standard Iron (Fe) Intensities	Standard Mode	
0.1	4805.5	
1.0	7343.6	
2.0	8103.0	Slope 514.7
10.0	10142.3	Intercept 5981.8
50.0	32131.1	$R^2=0.99789$
100.0	57318.8	

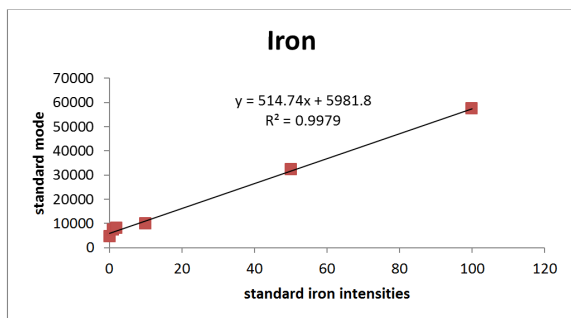


Figure 1. Calibration curve of Iron by ICP/MS.

Table 2. Intensity of samples.

Sample	Weight (g)	Volume (L)	Dilution	Iron Intensity
Acid Cleaning	0.2022	0.020	10	213792.9
	0.2022	0.020	10	219107.2
	0.2022	0.020	100	20653.3
	0.2022	0.020	100	20213.0
	0.2022	0.020	1000	2085.2
	0.2022	0.020	1000	2122.8
Passivating	0.2057	0.020	10	39.0
	0.2057	0.020	10	35.7
	0.2057	0.020	100	48.0
	0.2057	0.020	100	41.3
	0.2057	0.020	1000	34.3
	0.2057	0.020	1000	42
	0.2057	0.020	1000	6000

Table 3. Intensity of Iron (cont.).

Conc (diluted), ug/L	Conc (undiluted), ug/L	Amount (ug)	Conc (ug/g)	Mean
403.7	4037.2	80.7	399.3	
414.0	4140.5	82.8	409.5	
28.5	2850.3	57.0	281.9	
27.6	2764.8	55.3	273.5	277.7
-7.6	-7570.1	-151.4	-748.8	
-7.5	-7497.0	-149.9	-741.5	
-11.5	-115.5	-2.3	-11.2	
-11.6	-115.5	-2.3	-11.2	
-11.5	-1152.8	-23.1	-112.1	
-11.5	-1154.1	-23.1	-112.2	
-11.6	-11554.5	-231.1	-1123.4	
-11.5	-11539.5	-230.8	-1122.0	
0.0	35.4	0.7	3.4	<5

## 2.2. Iron with 1,10-Phenanthroline by UV-Vis Spectrophotometer

1,10-Phenanthroline (phen), is organic bases chemical (Figure 2) react rapidly with  $Fe^{2+}$  ions in weakly acid media to give orange-red which are a basis for determining Fe(II). Iron (II) and total iron can be determined with phenanthroline

after reduction of Fe to Fe(II). Hydroxylamine reduces Fe (III) within a few minutes in a weakly acidic medium (pH 3–4). The complex of 1,10-phenanthroline with Fe(II) is called ferroin and has been widely used in titrimetric analysis as a redox indicator. The compound 1,10-phenanthroline, P, is a bidentate ligand with the following structure.

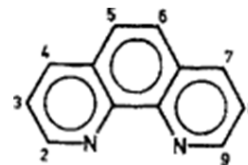


Figure 2. 1,10-Phenanthroline.

### 2.2.1. Instrument

UV/Visible spectrophotometers are widely used by many laboratories including those in academia and research as well as industrial quality assurance. The technique is mainly used quantitatively. The absorbance spectra for all measurements were carried out using a Shimadzu 1601 PC double beam UV-VIS Spectrophotometer, with 1 cm quartz cells and 2.0 nm fixed slit width. The spectrophotometer was connected to a computer, loaded with Shimadzu UVPC software, and equipped with an Epson LQ-850 printer [9, 14, 15].

### 2.2.2. Chemicals and Equipment

- 1) BioSpec-1601 UV-VIS Spectrophotometer
- 2) Several 100 mL and 1-500 mL volumetric flasks with corks or lids
- 3) 10.00 and 5.00 volumetric pipets
- 4) 1% Hydroxylamine HCl solution buffered with sodium acetate to pH = 4.0
- 5) Sodium acetate - acetic acid buffer at pH = 4.0
- 6) 0.2% 1,10-phenanthroline solution
- 7) 3M  $H_2SO_4$
- 8) 10 mg/L (10.0 ppm) standard iron solution
- 9) Polythene bottle, about 25-ml capacity (about 1 oz.)
- 10) 1,10-Phenanthroline, powdered solid reagent.

### 2.2.3. Procedure

Determination of the Absorption Spectrum,  $\lambda_{max}$  and the Molar Absorptivity Constant, for the Fe-orthophenanthroline complex.

1. Prepare a 0.2% solution of 1,10-phenanthroline in a 100.0 mL volumetric flask. Weigh out 0.2 grams of 1,10-phenanthroline and transfer it to a volumetric flask. Add 75.0 mL of distilled water to the flask, and then use a stir bar and magnetic stirrer to dissolve the compound. Remove the stir bar, and then dilute the solution up to the mark on the volumetric flask. You will need to stir this solution.
2. Prepare serial Fe Standard Solutions; 0.05, 0.10, 0.15, 0.20, 0.25, Blank in 50 mL VF
3. Add 10 mL pH=4 Buffer, 5.0 mL of hydroxylamine HCl, and 10 mL of 0.2% phenanthroline solution. Allow 15 minutes after adding all the reagents so that the color of the complex can fully develop before making any absorption measurements, absorbance values at  $\lambda_{max}$  510 nm wavelength.

### 2.2.4. Experimental

A Shimadzu 1601 PC UV/Vis spectrophotometer with matching 10-mm quartz cells was used for reading all spectra. All chemicals were of analytical grade and were used without further purification. Distilled, deionized water was used for making dilutions and for preparing all aqueous solutions [16].

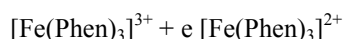
In this experiment, the amount of iron present in a sample is determined by first reacting the iron with 1,10-Phenanthroline to form a colored complex and measuring the amount of light, absorbed by this complex. Beer's law can then be used to determine the concentration relative to absorption:

$$A = \alpha bc$$

To form a complex, the iron must be first reduced to its ferrous state. This reduction is done by reaction the iron with hydroxylamine Hydrochloride by the following reaction:



Then the reaction with 1,10-Phenanthroline is:



Once a colored complex is formed, the wavelength of light which is most strongly absorbed is found by measuring the absorbance at various Wavelengths between 400-600 nm [17].

### 2.2.5. Spectrophotometric Curves

The spectrophotometric curves obtained for the standards and for the blank are shown in Table 4, and Table 5.

Deionized water was used as the blank. Since a faint red-brown color is produced by the reaction of iron and 1,10-phenanthroline in slightly acid solutions, and requires up to 15 minutes for full color development.

The color was stable for one hour.

Table 4. Calibration Curve Data.

[Fe] (mg/L)	Abs	Slope	0.2191
0.00	0.0000	Intercept	0.0013
0.05	0.0133	R <sup>2</sup>	0.9978
0.10	0.0234		
0.15	0.0353		
0.20	0.0450		
0.25	0.0553		

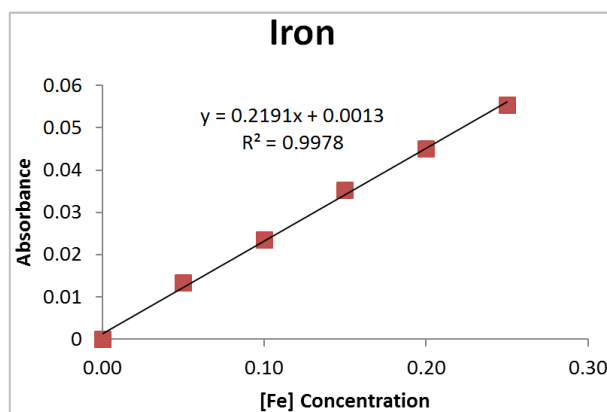


Figure 3. Calibration curve of Fe by UV-Vis Spectrophotometer.

Table 5. Linear Calibration - Calculated in Excel.

Sample	DF	Absorbance	Blank-corr Abs.	Fe [mg/L]
Blank		0.0032		
0.05	1	0.0165	0.0133	0.05
0.10	1	0.0266	0.0234	0.10
0.15	1	0.0385	0.0353	0.15
0.20	1	0.0482	0.0450	0.20
0.25	1	0.0585	0.0553	0.24
Acid Cleaning	1000	0.0672	0.0640	281.25
Acid Cleaning-D	1000	0.0666	0.0636	279.46
Acid Cleaning-S	1000	0.0693	0.0661	290.63
Acid Cleaning-SD	1000	0.0692	0.0660	290.18
Passivating	500	0.0058	0.0026	3.57
Passivating- D	500	0.0057	0.0025	3.35
Passivating-S	500	0.0062	0.0030	4.46
Passivating- SD	500	0.0061	0.0029	4.24
Check Std. 0.25 mg/L	1	0.0584	0.0552	0.24
LFB	1	0.058	0.0548	0.24

### 2.3. Determination of Iron (Fe) by Atomic Absorption Spectrometry

#### 2.3.1. Equipment/Instrument.

- 1) Perkin Elmer A Analyst 200 model Flame Atomic Absorption spectrophotometer with Deuterium arc background correction was used for comparing the results.
- 2) Magnetic stirrer, with TFE-Coated Stirring.
- 3) Timer.
- 4) Lamps: Fe element hollow cathode lamp.
- 5) Glassware: All glassware is washed in the following

sequence: Alconox detergent solution, tap water, 1:1 nitric acid, tap water, 1:1; hydrochloric acid, tap water, and final rinse with deionized water.

- 6) Pipettes: 5, 10, 20, 50, 100, 200, 500, and 1000  $\mu\text{L}$  pipettes with disposable tips are used.

#### 2.3.2. Reagents and Solutions

All chemicals were used of analytical grade. Doubly distilled deionized water was used in the preparations of solutions and used throughout. Stock solutions were kept in polypropylene bottles containing 1 mL concentrated nitric acid.

- 1) Pickling (acid cleaning) Stainless Steel Solution, obtained from ARDE Inc. New Jersey-USA.
- 2) Passivating Stainless Steel Solution, obtained from ARDE Inc. New Jersey-USA
- 3) Deionized water, on the day of use. Water was purified using a Millipore Mill-Q system via a pure water device marked Pure lab Option- Q7BP.
- 4) Iron standard solution (1000  $\mu\text{g/mL}$ ): Stock iron solution containing  $\mu\text{g/mL}$  Fe (III) was prepared by dissolving appropriate amounts of pure salt ( $\text{Fe}(\text{NO}_3)_3 \times 10 \text{ H}_2\text{O}$ ) in 100 mL of doubled distilled deionized water. The working solutions were prepared just before use by dilution of the standard solution with redistilled deionized water. Standards should be prepared fresh daily. Prepare three to five standards covering the range. Pipet 25 mL of 1000 ppm Certified Iron Standard Solution into a 250 mL volumetric flask. Add 10 mL of concentrated HCl. Dilute to volume with deionized water and mix well [18].

Prepare AA standards for iron of 1.0, 3.0, 5.0, 7.0, and 10.0 mg/L with double distilled deionized water.

#### 2.3.3. Instrument/ Operation Parameters

- 1) *Blank*: 100 mL of D. I. water is taken through the digestion process
- 2) *Duplicate*: A separate aliquot of random sample is digested or analysis (approximately 10%).
- 3) *Spike*: Using the formula  $C_1V_1=C_2V_2$  an appropriate volume and concentration of standards is brought through spiked into a sample and digestion procedure.
- 4) *Instruments*: Perkin-Elmer 5100 PC Atomic Absorption Spectrophotometer
- 5) *Start Program*: Perkin-Elmer 5100 PC Atomic Absorption Spectrum
- 6) Wavelength: 248.3 nm - UV
- 7) Slit width: 0.2 nm
- 8) Relative sensitivity: 1.0  $\mu\text{g/mL}$
- 9) Lamp energy: 67.0
- 10) Lamp Current: 5 mA
- 11) Light sources: Hollow Cathode Lamp
- 12) Burner Head: Air-acetylene
- 13) Flame stoichiometry: Air-acetylene, oxidizing (lean, blue)
- 14) Integration Time: 1.5 seconds (for optimizing only)
- 15) Average Readings: 3

#### 2.3.4. Sample Preparation/ Acid Digestion

Pipette 1.0 mL of acid cleaning (Pickling) stainless steel solution and, 20 mL of passivation stainless steel solution transfer into a 100 mL of volumetric flask. Add approximately 25 L of D. I. water and mix well. Then add 10 mL of concentration hydrochloric acid and swirl to mix. Dilute to volume with D. I. water and mix well.

### 3. Procedure

- 1) Prepare AA standards for iron 1.0, 3.0, 5.0, 7.0, and 10.0 mg/L standard solutions.

- 2) Run AA for iron as follows. Blank the instrument with deionized water. Run water and then a standard. Run water then a sample. Continue until all standards and sample have been measured.
- 3) Subtract average water signal from each standard reading. Construct a calibration curve and report slope, intercept, and correlation coefficient.
- 4) Subtract average water signal from each sample reading. Calculate concentration using calibration slope and intercept.
- 5) Calculate mg/L using the concentration in the volume taken, and the volume of solution.

## 4. Experimental

#### 4.1. Contamination Control

All reagent were of a high purity grade. Eppendorf pipettes with plastic tips were used for all pipetting. All sample handling operations were conduct under a laminar flow hood to reduce atmospheric contamination.

#### 4.2. Calibration Standards

The aqueous calibration standards were prepared in pre-cleaned 100 mL volumetric flasks using pipettes and fixed-volume Eppendorf pipette for volume transfers (Table 6 and Table 7). These standards represent the stock calibration standard were prepared.

#### 4.3. Backgrounds

The technique of atomic absorption spectrometry (AAS) depends on generating a cloud of free atoms in the ground or unexcited state. The first AA spectrometers used a flame to generate atoms of the element to be determined [19]. The most common method of background correction in atomic absorption (AA) spectrophotometer uses a continuum source such as a deuterium lamp to measure the background. Background is electronically subtracted from total absorbance to give background corrected atomic absorbance [20].

#### 4.4. Calibration and Operation

Before operation the calibration of the instrument is necessary. Rinse aspirator tube with D. I. water. Aspirate sample, ensure within range (between blank and highest standard). Dilute sample if necessary, and repeat for each sample.

##### Protocol:

Blank (purpose of this second blank measurement is to "zero" the reading)

Water  
Standard  
Water  
Sample  
Water  
Sample dup  
Water

Matrix Spiked  
Water  
Matrix Spiked Duplicate  
Water

Table 6. Calibration Curve Data.

[Fe] (mg/L)	Abs	m	0.0186338
0.0	-0.0007	b	0.0009817
1.0	0.0202	R <sup>2</sup>	0.9992
3.0	0.0591		
5.0	0.0940		
7.0	0.1306		
10.0	0.1786		

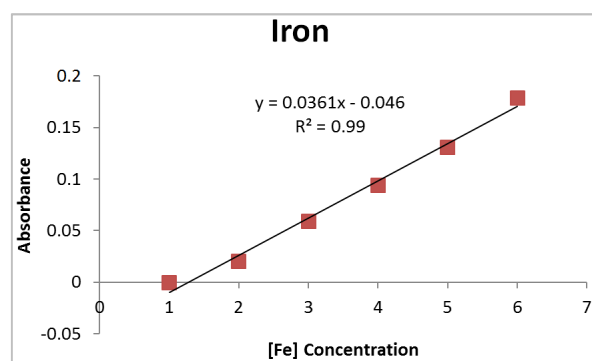


Figure 4. Calibration curve of Fe by AA.

Table 7. Linear Calibration –Calculated in Excel.

Sample	DF	Absorbance	Blank Correction	[Fe]mg/L
Air	1	0.0000		
Water	1	0.0036		
Blank	1	0.0000	-0.0007	-0.09
Water	1	0.0038		
1 mg/L	1	0.0249	0.0202	1.03
Water	1	0.0056		
3 mg/L	1	0.0648	0.0591	3.12
Water	1	0.0059		
5 mg/L	1	0.1006	0.0940	4.99
Water	1	0.0074		
7 mg/L	1	0.1390	0.1306	6.95
Water	1	0.0095		
10 mg/L	1	0.1884	0.1786	9.53
Water	1	0.0102		
Acid Cleaning sample	100	0.0632	0.0529	278.36
Water	1	0.0105		
Passivating Sample	5	0.0244	0.0136	3.39
Water	1	0.0111		
7 mg/L Check Std	1	0.1410	0.1295	25
Water	1	0.0119		

## 5. Result and Discussion

Simple and novel spectrophotometric methods are described for simultaneous determination of iron. The UV-Visible spectrometer is based on the metal ions-colored complexes (iron I, and iron II) formed by iron with colorimetric reagent 1, 10-phenanthroline that could be monitored

Spectrophotometrically[21]. In all instances, measurements were made at 510 nm against reagents blank (Figure3, Table 5).

Perkin-Elmer 5100 PC Atomic Absorption Spectrophotometer was used with a short part burner and air/acetylene flame optimized with hollow cathode lamp and a single element was measured. The background correction was selectable on an element by element basis. Water had been run between each reading; AA measurements were thus performed. The results of a single method for the detection of iron (Fe) in the Acid Cleaning and Passivating Stainless Solution were obtained and are shown in Figure 4 and Table 7. Calibration curve of iron linear plot of [Fe] concentration against blank-corrected absorbance at 248.3 nm.

Inductively Coupled Plasma Mass Spectrometry ICP/MS and Direct Spectrophotometric method was developed for estimation of Iron (Fe) and successfully used for quantitative extraction of Acid Cleaning (pickling) and Passivating Stainless Steel Solutions at acidic conditions. Since the equilibration time is very less; the method is quick and applicable for determination of Fe from different synthetic mixtures and catalysts. The results obtained are given in Table 8 and show that Fe can be successfully determined by three methods, and instruments.

## 6. Conclusion

The results are presented in Table 8, which reflects the equivalency of the three methods. When AAS, ICP/MS, compared with UV/Vis spectrophotometer is the preferred procedure. Spectrophotometry offers the analyst a relatively inexpensive, simple, fast, and readily available

alternative to more sophisticated techniques for the determination of iron (Fe) in acid cleaning (pickling) and passivating aqueous stainless steel solutions, as well as for quality-control applications.

**Table 8.** Results of Iron (Fe) by ICP/MS, UV-Vis, and FAAS.

Method	Specification	Average Results		Meet the specifications (Yes/No)
		Pickling (mg/L)	Passivating (mg/L)	
UV-Visible	20.000 mg/L max	280.25	3.91	Passes
ICP-MS	20.000 mg/L max	277.7	3.34	Passes
AAS	20.000 mg/L max	278.36	3.39	Passes

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