Methods for Determination of Microamounts of Impurities in Pure Iron*

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Synopsis

Applicability of the solvent-extraction spectrophotometry for the determination of impurities contained in microamounts in pure iron has been studied by using the filter-photometer with 50 mm pass at 0.5 ml solution. As the results, the methods, which were able to determine the content up to 0.0001% by using the sample of 0.1 g, were developed for the following 31 elements, Ag, Al, As, Au, B, Bi, Ca, Co, Cr, Cu, Ga, Ge, Mo, N, Nb, Ni, P, Pb, Pd, Pt, S, Sb, Se, Si, Sn, Ta, Ti, V, W, Y, and Zr. In addition, it was found that the other elements could also be determined up to 0.0001% in accuracy by the use of such methods as carbon by combustion-electroconductometry with 1 g sample, oxygen by vacuum-fusion mass spectrometry with 1 g sample, magnesium and zinc by atomic absorptiometry with 0.1 g each sample, and manganese by flame photometry with 0.1 g sample.

These methods were also applied to determine the impurities in pure iron on the market and the interim samples on the pure iron making process.

I. Introduction

The techniques for the purification of metals have been recently developed along with the rapid improvement of the production process of semiconductors.¹⁾ On the other hand, with the development of fundamental research on iron and steel, it became necessary to explain the essential character of iron by using high purity iron. From these reasons, the author intended to establish the methods for determining 36 elements present in microamounts in pure iron and the interim products of pure iron making process by using 0.1 g sample for each element with the accuracy of 0.0001%. As the results, the following methods were developed.

II. Analytical Methods for Determination of Impurities in Pure Iron

1. Silver²⁾

The sample ($\text{Ag} < 5 \mu\text{g}$) is dissolved in 10 ml of 1 : 1 HNO_3 or aqua regia, add 10 ml of 1 : 8 H_2SO_4 and evaporate the solution to white fumes. Dissolve the salts with a small quantity of water, transfer the solution to a separatory funnel, dilute to 20 ml with water, add 10 ml of dithizone benzene solution, shake for a minute, reject the lower layer after letting the solution stand to settle, and wash the remained solution twice with 10 ml of 1 : 35 H_2SO_4 . Add 10 ml of the back extraction solution, shake for a minute, transfer the lower layer to another separatory funnel, and repeat this extraction. Dilute the mixture to 50 ml with water, and add 5 ml of 20% tartaric acid solution, 0.5 ml of 5% EDTA solution and 10.0 ml of dithizone benzene solution (the same as above). After shaking it for a minute, reject the lower layer, wash the solution three times with 10 ml of the washing solution, filter the

benzene layer with dry filter paper, pour the filtrate into a cell of 50 mm, and measure the absorbance at 566 m μ against benzene to determine the content of silver.

Dithizone benzene solution : 0.1 g of dithizone is dissolved in 50 ml of benzene, shake it for about 2 min with 200 ml of the washing solution, add dropwise 1 : 8 H_2SO_4 to separate the aqueous layer and to slightly acidify it, again shake the mixture with 100 ml of benzene, and dilute the separated benzene layer to 500 ml with benzene.

Washing solution : 50 ml of saturated sodium sulphite solution is mixed with 1 000 ml of 1 : 100 ammonia water.

Back extraction solution : Dissolve 100 g of NaCl in water, add 1.7 ml of HCl, and dilute to 1 000 ml with water.

2. Aluminum

Dissolve 0.1 g of the sample in 10 ml of 1 : 9 H_2SO_4 and 5 ml of 15% H_2O_2 in a quartz beaker, filter off the residue through a close texture paper, and wash the residue thoroughly with water. Place the residue with the paper in a platinum crucible, reduce to ashes, ignite, fuse it with 1.00 g of KHSO_4 , extract the melts with 5 ml of water and 10 ml of 1 : 9 H_2SO_4 , and mix it with the main solution. Electrolyze the solution using a magnetic mercury cathode cell in order to remove the majority of iron and evaporate the solution down to 25 ml. Add 0.5 ml of 10% thioglycollic acid and 1.00 ml of 0.2% chromazurol S solution, add dropwise 1 : 1 ammonia water until the solution shows the yellow color with the last drop. Transfer the solution to a 50 ml measuring flask, add 5 ml of 20% ammonium acetate solution, let the solution stand 10 min, measure the absorbance with a 20 mm cell at 550 m μ against water to determine the content of aluminum.

3. Arsenic

Dissolve 0.5 g of the sample in 10 ml of 15% H_2O_2 and 20 ml of 1 : 9 H_2SO_4 at room temperature, heat the solution to expel the excess H_2O_2 , add 4 to 5 drops of 3% KMnO_4 solution, add dropwise 15% H_2O_2 , to extinguish the red color, and heat the solution to expel the excess H_2O_2 by boiling. Dilute to 100 ml exactly with water, pour 20.0 ml of the solution, which is the aliquot of the total volume of the solution, into the distillation flask shown in Fig. 1, add 6 ml of 1 : 1 H_2SO_4 and 2 ml of 15% KI solution, mix well, add dropwise 40% $\text{SnCl}_2\text{-HCl}$ solution to decolorize and dilute to 80 ml with water. Add 3 g of zinc metal

* Received June 18, 1968.

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Fig. 1. Distillation apparatus

not containing arsenic, distill AsH_3 at 20°C for 70 min and absorb AsH_3 in 3 ml of 0.5% silver diethyldithiocarbamate-pyridine solution. Measure the absorbance of pyridine solution with a 25 mm cell at $530\text{ m}\mu$ against pyridine to determine the content of arsenic.

4. Gold⁽³⁾

Dissolve 0.1 g of the sample in 5 ml of aqua regia, dry on a water bath and dissolve the salts in 4 ml of 1 : 1 HCl. Pour the solution into a separatory funnel with 10 ml of water, add 15 ml of ethylether, shake for a minute, reject the lower layer, and wash the remained 5 ml of solution twice with 1 : 6 HCl. Draw off the ether layer into a beaker. Add 5 ml of water, and evaporate the ether on a water bath. Dissolve the salts in 2.5 ml of 1 : 1 HCl, and pour the solution into a separatory funnel together with 2.5 ml of water. Add 5 ml of saturated NH_4Cl solution, 5 ml of 0.04% Rhodamine B solution and 5.00 ml of iso-propylalcohol, shake for a minute, and reject the lower layer. Filter the ether layer with a dry filter paper and measure the absorbance of the filtrate with a 50 mm cell at $555\text{ m}\mu$ against isopropylether to determine the content of gold.

5. Boron⁽⁴⁾

Dissolve 0.2 g of the sample in 5 ml of 5N H_2SO_4 in a platinum crucible, add 2 ml of H_2O_2 (30%), boil the solution to expel the excess H_2O_2 , after cooling, filter through a close texture paper and wash the paper with a small quantity of hot water. Collect the filtrate and washings in a polyethylene beaker, add 5 ml of HF (5%), let the mixture stand an hour, transfer the solution to polyethylene separatory funnel and dilute to about 50 ml with water. Add 3.00 ml of 0.001M methylene blue solution and 25.0 ml dichlorethane, shake the mixture for a minute, and then pour the lower layer into another polyethylene separatory funnel. Add 10.0 ml of water, shake for 2 min, allow the two liquids to settle by standing it for 15 min, filter the lower layer through a dry filter paper, and measure the absorbance of the filtrate with a 10 mm cell at $660\text{ m}\mu$ against dichlorethane to determine the content of acid soluble boron.

Place the residue on the filter in a platinum crucible, dry, reduce to ashes, fuse with 1.00 g of anhydrous Na_2CO_3 , extract the melt with 5 ml of 5N H_2SO_4 and succeed to the same manner as mentioned above to determine the content of acid insoluble boron.

6. Bismuth

Dissolve 0.1 g of the sample in 4.5 ml of HClO_4 (60%), allow to cool, transfer the solution to a separatory funnel, dilute to 20 ml with water, add 0.8 g of Na_2SO_3 , 0.4 g of ascorbic acid, and 1.0 ml of NaI solution (2%), mix well, add 5.00 ml of isoamyl acetate and shake the mixture for 3 min. Reject the lower layer, add 10 ml of the wash solution, shake for a minute, and reject the lower layer. Add 15.0 ml of dithizone solution, shake for a minute, let the mixture stand to settle, reject the lower layer, filter the organic layer through a dry filter paper and measure the absorbance of the filtrate with a 50 mm cell at $490\text{ m}\mu$ against the blank solution to determine the content of bismuth.

Wash solution: Dissolve 0.4 g of Na_2SO_3 and 0.2 g of ascorbic acid in water, add 1.3 ml of HClO_4 (60%) and dilute to 10 ml with water. Prepare the solution when it is necessary.

Dithizone solution: 1.0 mg dithizone is dissolved in 200 ml of the buffer solution.

Buffer solution: 10 g of ammonium citrate and 30 g of potassium cyanide are dissolved in 800 ml of water, add dropwise ammonia water to adjust the pH to 9, extract the impurities twice with 10 ml of 0.02% dithizone carbon tetrachloride solution, add dropwise ammonia water to adjust the pH to 10.5, and dilute to 1 000 ml with water. Dissolve 10 g of ammonium citrate and 30 g of KCN in 800 ml of water, add ammonia water drop by drop to the pH 9, extract the impurities twice with 10 ml of dithizone CCl_4 solution (0.02%), add ammonia water drop by drop to the pH 10.5 and dilute to 1 l with water.

7. Carbon⁽⁵⁾

Dissolve 11.2 to 50.2 mg of sodium oxalate (standard reagent) in water and dilute to 1 l (2 to 9 $\mu\text{g c/ml}$) with water, pour 1.00 ml of the solution into a quartz crucible and heat to dryness at about 280°C . Decompose the residues in a combustion tube at about 900°C under a current of oxygen, absorb the produced CO_2 gas in $\text{Ba}(\text{OH})_2$ solution ($1.2 \times 10^{-3}\text{ g/ml}$) containing 2 g of BaCO_3 , and measure the difference of electric conductivity of the solution to make a calibration curve. Treat 1 g of the sample with the same manner as previously stated to determine the content of carbon.

8. Calcium⁽⁶⁾

Dissolve the sample ($\text{Ca} < 6\text{ }\mu\text{g}$) in 20 ml of 1 : 1 HCl, add 5 ml of 1 : 1 H_2O_2 , and boil the solution to expel the excess H_2O_2 . After cooling, pour the solution into a separatory funnel together with 2 : 1 HCl, add 20 ml of MIBK (methylisobutylketone), and shake it for a minute. Let the solution stand to settle, transfer the lower layer to another separatory funnel, and repeat these operations until the MIBK layer no longer shows the characteristic color of ferric ion. Boil the separated lower layer to expel the organic solvent. After cooling, dilute with 10 ml of water, add 20% NaOH solution drop by drop to the pH 12.5, and pour the mixture into a separatory funnel. Add 1.5 ml of 0.5% GHA (glyoxalbis-(2-hydroxyl-anil)) solution, mix well, add 10.0 ml of butylalcohol at a time, shake for a minute, let the mixture stand to settle, filter the lower

layer through a dry filter paper, and measure the absorbance of the filtrate with a 50 mm cell at 516 $m\mu$ against butylalcohol to determine the content of calcium.

9. Cobalt⁷⁾

Dissolve 0.1 g of the sample in 5 ml of HCl and 1 ml of HNO_3 , boil the solution to expel the oxides of nitrogen, allow to cool, transfer the solution to a separatory funnel together with 20 ml of 10 : 6 HCl, add 20 ml of methylisobutylketone, and shake it for a minute. After boiling the separated lower layer for a few minutes, add 5 ml of HClO_4 (60%), and heat the solution to white fumes. After cooling, pour it into a separatory funnel together with 20 ml of water, add 2 ml of 50% citric acid solution, neutralize with ammonia water, add 5 ml of 0.04% β -nitroso- α -naphthol solution, mix well, let the solution stand for 2 min, add 20.0 ml of 1 : 1 butyl acetate-ethyl acetate mixture, and shake it for a minute. Let the solution stand to settle, reject the lower layer, wash with 1 : 10 HCl and 4% NaOH solution, filter the organic layer through a dry filter paper, and measure the absorbance of the filtrate with a 50 mm cell at 430 $m\mu$ against the solvent mixture to determine the content of cobalt.

10. Chromium⁸⁾

Dissolve 0.1 g of the sample in 10 ml of HCl and 3 ml of HNO_3 , heat the solution nearly to dryness, pour the solution into separatory funnel together with 25 ml of 10 : 6 HCl, add 25 ml of 2 : 1 methylisobutylketone-amyl acetate mixture, and shake it for a minute. After letting the solution settle, heat the separated lower layer just to dryness, add 5 ml of HNO_3 , and again heat nearly to dryness. Dissolve the salts in 10 ml of 1.4 V/V% H_2SO_4 , add 1 ml of 0.01% KMnO_4 solution while heating, and boil it for 3 min. After cooling, add 10 ml of 20% urea solution, mix thoroughly, and add 10% NaNO_2 solution drop by drop until the red color of permanganate disappears. Transfer the solution to a separatory funnel, dilute to 25 ml with water, add 3 ml of 0.2% diphenylcarbazide ethylalcohol solution, mix well, allow the mixture to stand a minute, add 8 ml of 1 : 1 H_2SO_4 , 5 g of anhydrous Na_2SO_4 and 10.0 ml of iso-amylalcohol, and shake the mixture for a minute. After letting the solution settle, reject the lower layer, filter the organic layer through a dry filter paper, and measure the absorbance of the filtrate with a 50 mm cell at 543 $m\mu$ against iso-amylalcohol to determine the content of chromium.

11. Copper⁹⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HCl and a few ml of H_2O_2 (30%), boil the solution to expel the excess H_2O_2 , allow to cool, add 15 ml of 20% ammonium citrate solution, and add dropwise ammonia water to the pH 6. Transfer the solution to a separatory funnel, dilute to about 40 ml with water, add 2 ml of 10% hydrochloric hydroxylamine solution and 2.0 ml of 0.1% bathocuproine ethylalcohol solution, mix well, and allow the mixture to stand 5 min. Add 5.00 ml of chloroform, shake for 2 min, allow the solution to settle, filter the organic layer through a dry filter paper, and measure the absorbance of the filtrate with a 50 mm cell at 477 $m\mu$ against chloroform to

determine the content of copper.

12. Gallium¹⁰⁾

Dissolve 0.1 g of the sample in 20 ml of 2 : 1 HCl, allow to cool, transfer the solution to a separatory funnel, add 17% TiCl_3 solution drop by drop until the red titanous color does not fade away, add 10 ml of methylisobutylketone, and shake it for a minute. After letting the solution settle, separate the upper layer, add 2 ml of 2 : 1 HCl, boil the solution to expel the organic solvent, allow to cool, pour it into a separatory funnel, and dilute to 15 ml with 7 : 5 HCl. Add 0.5 ml of 1 : 2 butylcellosolve, 0.40 ml of 0.5% Rhodamine B solution and 10.0 ml of benzene, and shake it for a minute. After letting the solution settle, filter the upper organic layer through a dry filter paper and measure the absorbance of the filtrate with a 50 mm cell at 562 $m\mu$ against benzene to determine the content of gallium.

13. Germanium

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HNO_3 , add 1 : 1 ammonia water to excess 10 ml, and boil the solution for a few minutes. After cooling, filter through a rapid filter paper, wash the precipitates several times with water, and dissolve the precipitates with 30 to 50 ml of HCl. Pour the solution into a separatory funnel, add 20 ml of CCl_4 , shake for a minute, allow the solution to settle, transfer the lower layer to another separatory funnel, shake it for a minute with 5 ml of water, and pour the upper aqueous layer into a 10 ml measuring flask. Add 1.2 ml of HCl, 0.8 ml of 5% arabic gum solution and 1.3 ml of 0.04% phenylfluorone solution, and dilute to the mark of the flask with water. After letting the solution stand 15 min, measure the absorbance with a 50 mm cell at 517 $m\mu$ against the blank solution to determine the content of germanium.

Phenylfluorone solution : 40 mg of phenylfluorone is dissolved in 80 ml of ethylalcohol and 5 ml of 1 : 6 H_2SO_4 , and dilute to 100 ml with ethylalcohol.

14. Magnesium¹¹⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HCl in a quartz beaker, add dropwise HNO_3 to oxidize iron, and then evaporate the solution to 2 ml. After cooling, add 10 ml of water and 10 ml of 10% ammonium citrate solution to adjust the pH to 8.5, pour the mixture into a separatory funnel, add 10.0 ml of 0.01M TTA (thenoyltrifluoroacetone) -MIBK solution, and shake it for 2 min. After letting the solution stand to settle, reject the lower layer, filter the organic layer through a dry filter paper, transfer the filtrate to a cell, and measure the atomic absorbance at 285.2 $m\mu$ to determine the content of magnesium.

15. Manganese¹²⁾

Dissolve 0.1 g of the sample in 5 ml of 1 : 1 HCl, 1 ml of HNO_3 , and 3 ml of HClO_4 (60%), and heat the mixture to white fumes. After cooling, transfer the solution to a separatory funnel, together with 20 ml of 10 : 6 HCl, add 20 ml of methylisobutylketone, and shake it for a minute. After letting the solution settle, separate the lower layer, boil it for a few minutes, add 1 ml of HNO_3 , and heat the solution nearly to dryness. After cooling, pour the solution into a separatory

funnel together with 10 ml of water, add 5 ml of 10% ammonium citrate solution and 8 ml of the buffer solution, and dilute to 25 ml with water. Shake the solution for a minute with 10 ml of TTA solution, allow the mixture to settle, filter the upper organic layer through a dry filter paper, and measure flame-photometrically the luminescence of the filtrate at 403 m μ to determine the content of manganese.

Buffer solution: Mix 660 ml of 2N HN_4Cl , 165 ml of 0.1M KH_2PO_4 , and 165 ml of 0.1N NaOH , and add ammonia water to adjust the pH to 8.5.

TTA solution: Dissolve 0.56 g of thenoyltrifluoroacetone in 166 ml of methylisobutylketone and dilute to 500 ml with 2-octane.

16. Molybdenum

Dissolve 0.1 g of the sample in 20 ml of 1 : 1 HCl and 2 ml of 1 : 1 H_2O_2 , boil the solution to expel the excess H_2O_2 . After cooling, pour the solution into a separatory funnel, add 4 ml of 50% tartaric acid solution and 1 ml of 0.02% $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solution, and dilute to about 36 ml with water. Add 4 ml of 25% KCNS solution and 4 ml of 25% $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ HCl solution, mix well, add 10.0 ml of MIBK, and shake the mixture for 2 min. After letting the solution stand to settle, reject the lower layer, add 30 ml of washing solution, shake for a minute, allow to settle, reject the lower layer, add 30 ml of 1 : 4 HCl , and shake the mixture for a minute. After letting the solution stand to settle, reject the lower layer, filter the organic layer through a dry filter paper, transfer the filtrate to a 50 mm cell, and measure the absorbance at 460 m μ against MIBK to determine the content of molybdenum.

Washing solution: Dissolve 1 g of oxalic acid in 50 ml of 1 : 10 HCl and add 4 ml of 25% $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ HCl solution.

17. Nitrogen¹³⁾

Dissolve 1 g of the sample in 20 ml of 1 : 1 HCl , pour the solution into the distillation apparatus containing 15 ml of 50% NaOH solution in it, pass a current of nitrogen to the system at the rate of 100 ml per minute, and continue the distillation until 5 ml of the distillate is obtained in a 10 ml measuring cylinder. Pour the solution into a separatory funnel, add 1 ml of 25% phenol alcohol solution and 2 ml of 25% NaOH solution, mix well, add 2 ml of NaOCl solution (available chlorine 4 to 5%), and again mix the solution. After letting the solution stand 10 min, add 3 g of NaCl and 5.00 ml of isobutyl alcohol, and shake the mixture for a minute. Allow the solution to settle, reject the lower layer, and filter the organic layer through a dry filter paper. Pour the filtrate into a 50 mm cell, and measure the absorbance at 660 m μ against isobutyl alcohol to determine the content of nitrogen.

18. Niobium¹⁴⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HNO_3 , 4 ml of 1 : 1 H_2SO_4 and 5 ml of 46% HF in a platinum dish and evaporate the solution to white fumes by heating. After cooling, dissolve the salt in a small quantity of 1 : 1 H_2O_2 and 3% ammonium oxalate solution, and electrolyze by the magnetic mercury

cathode method to remove iron. Transfer the solution to a beaker, evaporate to 20 ml by heating, allow to cool, pour the solution into a separatory funnel, add 2 ml of 0.01% Lumogallion solution and 3 ml of n-butyl alcohol, and shake it for 3 min. After letting the solution stand to settle, transfer the lower layer to another separatory funnel, add 10 ml of n-butyl alcohol, and shake it for 3 min. Allow the solution to settle, reject the lower layer, mix the organic layer together with the original organic layer, add 10 ml of water, and shake the mixture for a minute. After letting the solution stand to settle, pour the lower layer, shake for a minute, and mix the lower layer with the solution. Add 5 ml of 1 : 1 H_2SO_4 , 2 ml of 15% thioglycolic acid and 10 ml of 50% NH_4CSN solution, allow to cool the solution after gently heating, pour into a separatory funnel, add 15 g of MgCl_2 , and dilute to about 30 ml with water. Add 5.00 ml of 0.01M TOPO (trioctylphosphine oxide)-cyclohexane solution, and shake it for 15 min. Allow the solution to settle, reject the lower layer, filter the organic layer through a dry filter paper, pour the filtrate into a 50 mm cell, and measure the absorbance at 372 m μ against cyclohexane to determine the content of niobium.

19. Nickel

Dissolve 0.1 g of the sample in 3 ml of HNO_3 and 5 ml of HClO_4 , evaporate the solution to white fumes by heating, allow to cool, dissolve the salts in a small quantity of water, pour the solution into a separatory funnel, and dilute to about 20 ml with water. Add 5 ml of 20% citric acid solution, neutralize with ammonia water using bromthimolblue as the indicator until the color of the solution changes to green, and then add three more drops in excess. Add 2 ml of 1% dimethylglyoxime alcohol solution and 5 ml of chloroform, and shake it for 2 min. After letting the solution stand to settle, pour the lower chloroform layer into the second separatory funnel, add 5 ml of chloroform to the original funnel, again shake for a minute, and transfer the lower chloroform layer to the second funnel. Add 10 ml of 1 : 30 ammonia water, shake the mixture for 2 min, pour the lower chloroform layer into the second funnel, again shake for 2 min, and add the chloroform layers to the third funnel.

Add 5 ml of 1 : 20 HCl , shake for 2 min, allow the mixture to settle, transfer the lower chloroform layer to the fourth separatory funnel, add 5 ml of 1 : 20 HCl , again shake for 2 min, transfer 2 HCl layers, and evaporate the solution to about 2 ml by heating. Pour the solution into a 10 ml measuring flask, dilute to about 5 ml with water, add 1 ml of saturated bromine water and 2 ml of ammonia water, allow to cool, add 1 ml of 1% dimethylglyoxime alcohol solution, and dilute to the mark of the flask with water. Let the solution stand 5 min, transfer the solution to a 50 mm cell and measure the absorbance at 440 m μ against water to determine the content of nickel.

20. Oxygen¹⁵⁾

Apparatus: Vacuum fusion-mass spectrometric method has been developed for the analysis of oxygen. A schematic diagram of the apparatus used and the detail of the furnace are shown in Figs. 2 and 3, res-

Fig. 2. A schematic diagram of the gas analyzer for iron and steel

- ① Glass hook ⑤ Graphite fiber felt
 ② Mo-wire ⑥ Graphite fiber powder
 ③ Quartz furnace tube ⑦ Induction coil
 ④ Transparent quartz cup ⑧ Graphite crucible

Fig. 3. Furnace section

pectively.

Procedure: Give the electrolytic finishing for the sample of 6 mm ϕ $\times$ 6 mm in the electrolyte consisted of 1 : 10 acetic acid-HClO₄ for 5 to 10 min, wash the sample with in the order of water, absolute alcohol, and ether, and dry in a desiccator. After weighing, set the sample in loading tube, and evacuate the system to 10⁻² mmHg by mechanical vacuum pump and to 10⁻⁶ mmHg mercury diffusion pump. Heat the graphite crucible by high frequency current at about 2 300°C for 3 hr, and make a blank test for oxygen at 1 700°C for 5 min. Put the sample into the crucible to determine the content of oxygen, if the result of the

Table 1. The pattern factor and sensitivity

M/e	CO	N ₂	H
2			100
12	8.67	14.89	
14	0.60		
16	4.03		
28	100	100	
Sensitivity	30.61	28.32	9.86

blank test is less than 0.002 ml.

The pattern factor and the sensitivity obtained for the standard sample by the apparatus used in the current investigation are shown in Table 1.

For the results mentioned above, the formulae used for the calculation are as follows.

$$O(\%) = V_O \times K \times 0.0714 / \text{wt of sample (g)}$$

$$N(\%) = V_N \times K \times 0.125 / \text{wt of sample (g)}$$

$$H(\%) = V_H \times K \times 0.009 / \text{wt of sample (g)}$$

where, V_O , V_N , V_H : volumes of CO, N₂, and H₂ in ml, respectively

$$K: 273 \times B / 760 (273 + t)$$

$$B: \text{pressure of gas at } t^\circ\text{C in mmHg}$$

$$t: \text{temperature of gas in } ^\circ\text{C.}$$

21. Phosphorus¹⁶⁾

Dissolve 0.1 g of the sample in 10 ml of the acid, boil the solution to expel the excess H₂O₂, allow to cool, add 5 ml 5N HNO₃, pour the mixture into a separatory funnel, and dilute to about 20 ml with water. Add 5 ml of 10% ammonium molybdate solution and 10 ml of 1 : 3 n-butyl alcohol-chloroform, shake for 2 min, allow the solution to settle, pour the organic layer into the second separatory funnel, add 5 ml of 1 : 3 n-butyl alcohol-chloroform to the aqueous layer,

shake for a minute, and transfer the organic layer to the second funnel. Add 10 ml of 1% $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ 1 : 10 HCl solution, shake for a minute, allow the solution to settle, reject the lower layer, pour the aqueous layer into a 20 mm cell, and measure the absorbance at 700 $m\mu$ against water to determine the content of phosphorus.

The acid: Add 50 ml of 30% H_2O_2 to 125 ml of HNO_3 and dilute to 500 ml with water.

22. Lead

Dissolve 0.1 g of the sample in 5 ml of HCl and 1 ml of HNO_3 , add 5 ml of HClO_4 , and evaporate the solution to dense white fumes by heating. After cooling, add 30 ml of 10 : 6 HCl, transfer the mixture to a separatory funnel, add 30 ml of MIBK, shake for a minute, allow the solution to settle, put the lower layer in a beaker, and evaporate it to dense white fumes by heating. After cooling, pour it into a separatory funnel, dilute to 20 ml with water, add 2 ml of 50% ammonium citrate solution, neutralize with 1 : 1 ammonia water, add 1 : 1 ammonia water 4 ml in excess and then 1 ml of 20% NaCN after the color of the solution changes to green with bromthymolblue as the indicator, and mix it well. Add 10 ml of dithizone benzene solution, shake for 2 min, allow the solution to settle, reject the lower layer, add 20 ml of the washing solution to the organic layer, shake for a minute, and reject the lower layer. Repeat the washing procedure until no color is observed in the lower layer.

Add 10 ml of 0.1N HCl to the benzene layer, shake for a minute, allow the solution to settle, and pour the lower layer into another separatory funnel. Again repeat this procedure and collect the acid solutions. Add 2 ml of 50% ammonium citrate solution and bromthymolblue as the indicator, neutralize with 1 : 1 ammonia water, and add 4 ml in excess. Add 1 ml of 20% NaCN solution and 5.00 ml of dithizone benzene solution, and shake it for 2 min. Let the solution stand to settle, reject the lower layer, add 10 ml of the washing solution to the benzene layer, shake for a minute, reject the lower layer, and repeat the washing procedure until the lower layer shows no color. Filter the benzene layer through a dry filter paper, put the filtrate in a 25 mm cell and measure the absorbance at 520 $m\mu$ against benzene to determine the content of lead.

Dithizone benzene solution: Dissolve 0.1 g of dithizone in 20 ml of benzene, wash with 20 ml of the washing solution, slightly acidify with 1 : 1 HCl, extract with 20 ml of benzene, and dilute to 100 ml with benzene.

Washing solution: Add 5 ml of saturated NaHSO_3 solution to 100 ml of 1 : 100 ammonia water.

23. Palladium¹⁷⁾

Dissolve 0.1 g of the sample in 10 ml of aqua regia, evaporate the solution to dryness, add a small quantity of HCl, evaporate it to dryness, and again repeat this procedure. Dissolve the salts in 20 ml of 10 : 6 HCl, transfer the solution to a separatory funnel, add 20 ml of MIBK, shake for a minute, allow the solution to settle, put the lower layer in a beaker, and evaporate the solution to 10 ml by heating. Add 5 ml of HNO_3

and 10 ml of HClO_4 , and evaporate the mixture to 4 ml by heating. After cooling, add 20 ml of 1 : 1 HCl, pour the mixture into a separatory funnel, add 5 ml of 0.5% thioxine HCl solution and 3.00 ml of chloroform, shake for 3 min, allow the solution to settle, pour the chloroform layer into another separatory funnel, add 5 ml of 1% thiourea HCl solution, and shake it for 2 min. After letting the solution stand to settle, filter the chloroform layer through a dry filter paper, transfer the filtrate to a 50 mm cell, and measure the absorbance at 505 $m\mu$ against chloroform to determine the content of palladium.

24. Platinum

Dissolve 0.1 g of the sample in 10 ml of aqua regia, evaporate it to dryness, add a small quantity of HCl, evaporate the mixture to dryness, and again repeat this procedure. Dissolve the salts in 5 ml of HCl, pour the solution into a separatory funnel, wash the beaker with 2 ml of water and 18 ml of SnCl_2 HCl solution, and add washings to the solution in the funnel. Add 3.00 ml of 1% resorcin iso-amyl acetate solution, shake for 1 min, let the solution stand 10 min, filter the organic layer through a dry filter paper, put the filtrate in a 50 mm cell, and measure the absorbance at 395 $m\mu$ against the resorcin iso-amyl acetate solution to determine the content of platinum.

SnCl_2 HCl solution: Dissolve 23 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 34 ml of HCl and dilute to 100 ml with water.

25. Sulphur

Dissolve 1 g of the sample in 20 ml of 1 : 1 HNO_3 and 0.4 g of KClO_3 , evaporate it to dryness, add 10 ml of HCl to dissolve the salts, evaporate the solution to dryness, twice repeat this procedure, and finally evaporate the solution to 2 ml. Add 25 ml of 10 : 6 HCl, pour the mixture into a separatory funnel, add 25 ml of MIBK, shake for a minute, allow the solution to settle, transfer the lower layer to the second separatory funnel, wash the MIBK layer twice with 5 ml of 10 : 6 HCl, and collect the HCl layers to the second funnel. Add 15 ml of MIBK, shake for a minute, allow the solution to settle, transfer the lower layer to a distillation flask, wash the MIBK layer twice with 5 ml of 10 : 6 HCl, and collect the HCl layers to the flask. Evaporate the solution to dryness at 140° to 150°C, allow to cool, add 5 ml of the reducing agent, heat the flask connecting a cooler at 130°C, pass a current of nitrogen to the system with the rate of 100 ml per minute for an hour, and absorb the H_2S gas produced in the absorbing solution. Add 5 ml of 0.2% para-aminodimethyl-aniline 1 : 2 HCl solution and 1 ml of 10% ferric chloride solution, dilute to 50.0 ml with water, put it in a 20 mm cell, and measure the absorbance at 660 $m\mu$ against water to determine the content of sulphur.

Reducing agent: Add 50 ml of 48% HI to 25 ml of 50% Na_2PO_2 solution, allow the NaCl produced to settle, transfer the supernatant to a conical flask, pass a current of nitrogen to the system, heat it at 115°C for 15 hr, and allow to cool.

Absorbing solution: Add 1 ml of 1N NaOH solution to 34 ml of 0.1M CdCl_2 solution when it is necessary.

26. Antimony

Dissolve 0.1 g of the sample in 5 ml of 1 : 1 HNO_3 and 5 ml of HCl , add 5 ml of 1 : 1 H_2SO_4 , evaporate the solution to dense white fumes by heating. After cooling, add 5 ml of HCl , pour the mixture into a separatory funnel, dilute to 25 ml with water, allow to cool sufficiently, add 10.0 ml of iso-propyl ether, shake for a minute, allow the solution to settle, reject the lower layer, and wash the organic layer with 2 ml of 1% hydrochloric hydroxylamine 1 : 9 HCl solution and then 2 ml of 1 : 9 HCl . Add 5 ml of 0.02% Rhodamine B 1 : 9 HCl solution, shake for a minute, let the solution stand to settle, reject the lower layer, filter the ether layer through a dry filter paper, put the filtrate in a 50 mm cell, and measure the absorbance at 550 $\text{m}\mu$ against iso-propyl ether to determine the content of antimony.

27. Selenium¹⁸⁾

Dissolve 0.1 g of the sample in 3 ml of 1 : 1 HNO_3 , add 1 ml of HClO_4 , and evaporate the solution to white fumes. After cooling, add 20 ml of newly prepared acid mixture (HCl 3, HNO_3 2, H_2O 4), pour the solution into a separatory funnel, add 20 ml of MIBK, shake for a minute, allow the solution to settle, put the lower layer in a beaker, add 2 ml of HClO_4 , and evaporate the solution to white fumes. After cooling, add 30 ml of water, 3 ml of 2.5M formic acid and 5 ml of 5% EDTA 2Na solution, adjust the pH to 2.5 ± 0.2 with 1 : 1 ammonia water, add 2 ml of 0.5% 3,3'-diaminobenzidine solution, and boil it for 0.5 min. After cooling, adjust the pH to 6 to 7 with 1 : 1 ammonia water, pour the solution into a separatory funnel, add 5.00 ml of toluene, and shake it for a minute. After letting the solution stand to settle, filter the toluene layer through a dry filter paper, put the filtrate in a 50 mm cell, and measure the absorbance at 420 $\text{m}\mu$ against toluene to determine the content of selenium.

28. Silicon¹⁹⁾

Dissolve 0.1 g of the sample in 10.0 ml of 15 : 800 H_2SO_4 and 2 ml of 1 : 1 H_2O_2 in a platinum crucible, electrolyze by the magnetic mercury cathode method to remove iron, and dilute to 100 ml exactly with water. Transfer 25.0 ml of the solution, which is the aliquot for the total volume of the solution, to a polyethylene beaker, dilute to 100 ml with water, add 2 ml of 5% ammonium molybdate solution, mix well, let the solution stand 15 min, and add 5 ml of 5% oxalic acid solution. After letting the solution stand 5 min, add 5 ml of 1 : 3 H_2SO_4 and 5 ml of the reducing agent, mix well, and allow the solution to stand 10 min. Pour the solution into a polyethylene separatory funnel, add 2 to 5 ml of 5% polyoxyethylenelaurylamine 1 : 3 H_2SO_4 solution and 5.00 ml of chloroform, shake for 3 min, allow the solution to settle, filter the organic layer through a dry filter paper, transfer the filtrate to a 50 mm cell, and measure the absorbance at 800 $\text{m}\mu$ against chloroform to determine the content of silicon.

Reducing agent: 2.2 g of staneous oxalate, 4.2 g of oxalic acid, 4.0 g of sodium formate and 2.4 ml of formic acid are dissolved in 240 ml of water, and 10 ml of this solution is mixed with 15 ml of 1 : 3 H_2SO_4

when it is necessary.

29. Tin

Dissolve 0.05 g of the sample in 10 ml of 1 : 1 HCl , add dropwise HNO_3 to oxidize iron and then 5 ml of HClO_4 , and evaporate the solution to dense white fumes. After cooling, add 15 ml of 1 : 2 H_2SO_4 and 1.0 ml of 17% TiSO_4 solution, pour into a separatory funnel, add 10 ml of 1 : 2 H_2SO_4 , 3 ml of 75% NaI solution and 10.0 ml of benzene, shake for 2 min, allow the solution to settle, reject the lower layer, wash the organic layer with 10 ml of 1 : 2 H_2SO_4 , reject the lower layer, add 10 ml of 1 : 24 HCl to back extract tin, twice repeat the extraction procedure, and collect the acid layers. Evaporate the solution to 15 ml, allow to cool, add 1.0 ml of 0.05% pyrocatechol violet solution and 2.0 ml of 20% sodium acetate solution, adjust the pH to 3.5 to 4.0 with 1 : 10 ammonia water, and dilute to 25.0 ml exactly with water. Put the solution in a 50 mm cell and measure the absorbance at 560 $\text{m}\mu$ against the blank solution to determine the content of tin.

30. Tantalum

Dissolve 0.1 g of the sample in 5 ml of aqua regia, evaporate the solution to 2 ml, allow to cool, add 10 ml of 10 : 6 HCl , transfer the mixture to a separatory funnel, add 10 ml of MIBK, shake for a minute, allow the solution to settle, put the lower layer in a beaker, add 3 ml of HNO_3 and 3 ml of 1 : 1 H_2SO_4 , and evaporate the solution just to dryness. Dissolve the salts in 5 ml of 1% ammonium oxalate solution and dilute to 10.0 ml with the oxalate solution. Pour 5.00 ml of the solution, which is the aliquot of the total volume of the solution, into a polyethylene separatory funnel, add 2 ml of 1 : 10 HF (46%) and the required quantity of 4% NaOH solution, allow to cool, add 1.5 ml of 0.2% Malachite green solution, and dilute to 10 ml with water. Add 5.00 ml of benzene, shake for a minute, allow the solution to settle, reject the lower layer, filter the organic layer through a dry filter paper, put the filtrate in a 50 mm cell, and measure the absorbance at 635 $\text{m}\mu$ against the blank solution to determine the content of tantalum.

Required quantity of 4% NaOH solution is determined as follows. Add 1 ml of 1N H_2SO_4 to 5 ml of 1% ammonium oxalate solution, dilute to 20 ml with water, and measure the pH(A) of this solution. Dilute 5.00 ml of remaining sample solution to 20 ml with water by the above stated procedure, and adjust the pH to A with 4% NaOH solution. The volume of 4% NaOH solution added to adjust the pH to A is called the required quantity.

31. Titanium²⁰⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 6 H_2SO_4 and 3 ml of 1 : 1 H_2O_2 , boil the solution to expel the excess H_2O_2 , allow to cool, electrolyze by the magnetic mercury cathode method to remove iron, and evaporate the solution to 10 ml. After cooling, add 2.5 ml of 10% thioglycolic acid and 10 ml of 50% NH_4CNS solution, pour the mixture into a separatory funnel, and dilute to 25 ml with water. Add 15 g of MgCl_2 and 10.0 ml of 4% TOPO cyclohexane solution, shake it for 15 min, allow the solution to settle, transfer

the organic layer to a 50 mm cell, and measure the absorbance at 420 $m\mu$ against cyclohexane to determine the content of titanium.

32. Vanadium²¹⁾

Dissolve 0.1 g of the sample in 10 ml of HClO_4 and 5 ml of HNO_3 , evaporate the solution to dense white fumes, allow to cool, electrolyze by the magnetic mercury cathode method to remove iron, and dilute to 100 ml exactly with water. Pour 10.0 ml of the solution, which is the aliquot of the solution prepared, into a separatory funnel, add 5 drops of 0.1% KMnO_4 solution, mix, allow the solution to stand 2 min, add 10 ml of HCl and 3.00 ml of 0.2% N-BPHA (N-benzoyl phenyl hydroxylamine) chloroform solution, and shake it for a minute. After letting the solution stand 5 min, filter the organic layer through a dry

filter paper, put the filtrate in a 50 mm cell, and measure the absorbance at 530 $m\mu$ against chloroform to determine the content of vanadium.

33. Tungsten²²⁾

Dissolve 0.1 g of the sample in 5 ml of HCl and 5 ml of 1 : 9 HF , add 4 ml of 25% hydroxylamine hydrochloride solution, and heat the solution to reduce iron and molybdenum. After cooling, pour the solution into a separatory funnel, add 1 ml of 10% tartaric acid solution, 4 ml of 1 : 9 HF , 4 ml of 25% KCNS solution and 7 ml of HCl , dilute to 30 ml with water, add 20 ml of n-butyl acetate, and shake it for a minute. After letting the solution stand to settle, put the lower layer in a beaker, wash the remaining solution with a small portion of 1 : 1 HCl (1 : 1) : HF (1 : 9), and add the washing to the main solution in the beaker. Add

Table 2. Determining condition, effect of co-existing elements, and time required

Element	Absorbance	Element added (μg)	Final vol. (ml)	Filter ($m\mu$)	Element disturbed	Time required (min)
Ag	0.70	5	10	460	None	60
Al	0.55	10	10	550	None	150
As	0.70	5	3	532	None	90
Au	0.70	5	5	550	None	90
B	0.85	5	20	660	None	120
Bi	0.85	3	10	495	None	90
C	Full scale	10	1 (g)	—	None	12
Ca	0.80	8	10	517	None	100
Co	0.90	2	5	430	None	60
Cr	0.85	5	10	532	None	60
Cu	0.85	5	5	460	None	60
Ga	0.65	5	10	562	None	60
Ge	0.65	5	10	517	None	70
Mg	0.70	8	10	285	None	30
Mn	Full scale	10	10	403	None	70
Mo	0.75	5	10	460	None	30
N	1.0	5	5	660	None	20
Nb	0.80	5	5	372	None	180
Ni	1.0	10	10	440	None	60
O	Full scale	10	1 (g)	—	None	20
P	0.80	10	10	700	None	40
Pb	0.75	5	5	517	None	120
Pd	0.80	10	3	495	None	90
Pt	0.60	10	3	390	None	120
S	0.70	5	100	560	None	30
Sb	0.75	5	10	550	None	60
Se	0.60	10	5	420	None	30
Si	0.95	1.5	5	800	None	90
Sn	0.65	10	25	562	None	40
Ta	0.85	5	5	610	None	80
Ti	0.85	2	10	420	Nb, Ta	80
V	0.90	5	3	530	None	60
W	0.40	10	5	420	None	120
Y	0.40	10	25	655	Ce	80
Zn	0.30	10	10	214	None	30
Zr	0.65	10	100	665	None	60

3 ml of 1 : 1 H₂SO₄, boil, add dropwise HNO₃ to decompose the organic matter, and evaporate the solution to dryness. Add 6 ml of 10% NaOH solution to dissolve the salts, then add 10 ml of HCl and 3 ml of 17% TiCl₃ solution, and heat the solution to reduce tungsten. After cooling, pour the solution into a separatory funnel, add 4 ml of 25% KCNS solution, adjust the acidity to 5N with HCl, add 5.00 ml of n-butyl acetate, and shake it for 3 min. Allow the solution to settle, reject the lower layer, wash the remaining solution with 30 ml of washing solution, and again repeat the washing procedure. Filter the organic layer through a dry filter paper, put the filtrate in a 50 mm cell, and

measure the absorbance at 420 mμ against n-butyl acetate to determine the content of tungsten.

Washing solution : 2 g of oxalic acid is dissolved in 100 ml of 1 : 10 HCl and add 8 ml of 25% staneous chloride HCl solution.

34. Yttrium

Dissolve 0.1 g of the sample in 3 ml of 1 : 1 HNO₃ and 2.0 ml of HClO₄, evaporate the solution to white fumes, dissolve the salts in 30 ml of water, and electrolyze by the magnetic mercury cathode method to remove iron. Evaporate the solution to 10 ml, allow to cool, add 1.0 ml of 10% ammonium acetate solution and 3.0 ml of 0.01% Arsenazo III solution, adjust the

Table 3. Analytical results of impurities contained in pure iron on the market (unit : ppm)

Sample element	Puron (1962) (U.S.A.)	Puron (1965) (U.S.A.)	Johnson Matthey (England)	L. Light (England)	Fleisherman (U.S.A.)
Ag	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
Al	12, 13	12, 11	11, 12	13, 13	8, 10
As	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Au	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
B	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
Bi	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
C	20, 22	13, 13	7, 8	11, 10	9, 9
Ca	7, 5	25,	15,	24,	16,
Co	15, 15	10, 11	4, 2	14, 11	6, 5
Cr	5, 4	5, 3	3, 4	18, 20	1, <1
Cu	10, 7	8, 9	5, 2	3, 2	<1, <1
Ga	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
Ge	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
Mg	2, 4	2, 2	4, 3	6, 6	6, 6
Mn	5, 4	2, 3	5, 7	9, 10	6, 7
Mo	3, 3	7, 6	1, 2	1, 1	<1, <1
N	63, 64	67, 68	117,	261, 260	251,
Nb	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Ni	10, 11	10, 12	14, 14	26, 26	18, 19
O	540, 520	460, 480	45, 45	13, 18	<10, <10
P	8, 8	8, 8	<2, <2	<2, <2	<2, <2
Pb	30, 28	<1, <1	<1, <1	<1, <1	<1, 2
Pd	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Pt	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
S	19, 15	7, 8	10, 11	1, 3	<1, <1
Sb	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
Se	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Si	37, 34	12, 10	8, 9	9, 10	10, 9
Sn	2, 3	<1, <1	4, 2	<1, 1	1, 1
Ta	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Ti	1, 1	1, 1	<1, <1	<1, <1	<1, <1
V	<1, <1	<1, <1	<1, <1	<1, <1	<1, <1
W	21, 23	8, 7	9, 7	6, 5	2, 5
Y	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Zn	2, 2	1, <1	2, 1	<1, <1	<1, 2
Zr	<2, <2	<2, <2	<2, <2	<2, <2	<2, <2
Total* Iron (%)	803 99.920	672 99.933	222 99.978	406 99.959	331 99.967

* Average value of each two figures.

pH to 3.0 with ammonia water, and dilute to 25.0 ml exactly with water. Put the solution in a 50 mm cell and measure the absorbance at 665 m μ against water to determine the content of yttrium.

35. Zinc⁽¹⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HNO₃, add 10 ml of 10% ammonium tartarate solution, and neutralize with 1 : 1 ammonia water by using bromthymolblue as the indicator until the color of the solution changes to green, and then add two more drops in excess. Pour the solution into a separatory funnel, add 10.0 ml of 2.2% TTA-MIBK solution, shake for a minute, allow the solution to settle, filter the organic layer through a dry filter paper, put the filtrate in a cell, and measure the atomic absorbance at 213.8 m μ to determine the content of zinc.

36. Zirconium⁽²⁾

Dissolve 0.1 g of the sample in 10 ml of 1 : 1 HNO₃, add 5 ml of 46% HF to decompose the residue, add 5 ml of HClO₄, and evaporate the solution to dense white fumes. After cooling, add 30 ml of 10 : 6 HCl, transfer the mixture to a separatory funnel, add 30 ml of MIBK, shake for a minute, allow the solution to settle, put the lower layer in a beaker, add 5 ml of HNO₃, and evaporate the solution to 3 ml. After cooling, pour the solution into a 100 ml measuring flask, add 50 ml of HNO₃ and 5 ml of 10% urea solution, dilute to about 90 ml with water, allow to cool, add 5 ml of 0.1% Arsenazo III solution, and dilute to the mark of the flask with water. Put the solution in a 50 mm cell and measure the absorbance at 665 m μ against water to determine the content of zirconium.

III. Effect of Co-existing Elements

Effects of the co-existing elements on the methods developed for the determination of 36 elements such as Al, As, B, Ca, Ce, Co, Cr, Cu, Mg, Mn, Mo, Nb, Ni, P, Pb, Si, Sn, Ta, Ti, V, W, and Zn were also studied by adding each 20 μ g of co-existing elements, to the sample containing 5 μ g of the determining element. The results obtained are listed in Table 2. The sensitivities of the Hiram filter photometer mainly used in this experiment and the time required are also shown in Table 2.

IV. Analytical Results

Analytical results for the determination of the impurities in a high purity iron on the market obtained by the methods mentioned above are shown in Table 3. In this work, the determination was repeated twice for each element.

Purities indicated by the manufacturers are as follows.

Name	Purity (%)
Johnson Matthey	99.99 Cu<1, Mg 2, Mn 3, Ni 1, Si 3, Ag<1
L. Light	99.99 Cu<1, Mg 1, Mn 3, Ni 2, Si 1, Ag<1, Na 1
Fleishermann	99.995

V. Conclusion

In order to analyze the microamount impurities

contained in pure iron, wet chemical methods including organic reagent and solvent extractions have been developed by using a filter photometer in which 50 mm pass could be taken for 0.5 ml of sample solution. The methods developed were also applied to determine the impurities in pure iron on the market, and the analytical results were compared with those determined by the solid mass spectrometry. The results obtained are summarized as follows:

(1) The purity and the amounts of impurities indicated on the label of pure iron on the market may not be so reliable.

(2) From the results obtained by the solid mass spectrometry, it was found that the relative sensitivity of each element in pure iron was not uniform.

(3) Alkali metals and halogen were usually detected by the solid mass spectrometry even when the samples were carefully prepared. Therefore, it is possible to conclude that elements may be always contained in pure iron.

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