Determination of Carbon and Manganese in Steel Using Optical Emission Spectrometer

By
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A thesis submitted to the Avinashilingam Institute for Home Science and Higher Education for Women (Deemed University) Coimbatore-641 043
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DETERMINATION OF CARBON AND MANGANESE IN STEEL USING OPTICAL EMISSION SPECTROMETER

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Certified as Bonafide reasearch work

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Introduction

INTRODUCTION

Steel is essentially iron, Containing 0.15% to 1-5% carbon. The essential difference between cast iron and steel is in the amount of their carbon contents. As the percentage of carbon is increased from 0.15% to 1.5%, the steel becomes harder and tougher, because up to a content of about 1.5% all the carbon gets into chemical combination with iron as Fe3C and none of it exists in the free graphite form. Such a ferrous material is called steel. If the carbon content increases beyond 1.5% then it does not combine chemically with iron and is present as free graphite. It falls in the category of cast iron[Jain et al;1990].

Steel is broadly classified as alloy or special steels and plain carbon steels. Plain carbon steels do not contain any alloying element in more than 2% or 3% Based on the carbon content it is further classified as

(i) Very mild or dead mild steel - up to 0.25% carbon

(ii) Low or medium carbon steel - up to 0.6% carbon

(iii) High carbon steels between 0.6% and 1.5% carbon

[Balasubramanian et al;1992].

Composition of steel :

In addition to carbon all plain carbon steels contain the following elements

Manganese - up to 1%

Sulphur - up to 0.03%

Silicon - up to 0.3%

Phosphorus - up to 0.05% [Jain et al;1990].

Function of elements present in steel :-

Carbon:

(i) The strength of steel increases with increase in the proportion of carbon, till it reaches 0.83% after which the effect is in reverse direction.

(ii) The ductility, decreases with increase in carbon content. This correspondingly effects the workability of the steel.

(iii) The hardness increases with increase in carbon content.

(iv) The susceptibility to heat treatment also increases with the increase in carbon content.

Whatever may be its effects, the extent of carbon content in steel is never more than 1.5% even in the highest carbon steels.[Jain et al;1990].

Manganese :

Manganese is an important element in steel. It deoxidises ferrous oxide, which is harmful. If sulphur is present above 0.06%, it makes the steel brittle. Manganese acts as a desulphuriser. It combines with sulphur to from manganese sulphide, which is relatively harmless when present in small amounts. It imparts strength and responsiveness to heat treatment.Manganese is usually present in quantities from 0.5% to 2%[Jain et al;1990].But certain special steels are made in the range of 10% to 15%.Large percentages of Manganese makes steel tough and impact resistant.[Abramov 1986].

Silicon :

Silicon in ordinary amounts is used in steel manufacture as a scavenger. It eliminates gases, thus making the metal sound and free from blow holes. It improves tensile strength and deepens hardening properties.

Sulphur :

Sulphur is valueless to the metal and so is kept as low as possible in all steels. Sulphur above 0.06% should not be present in steels; otherwise the steel becomes very brittle and red short. [(i.e.) the condition of Iron and steel in which it cannot be worked by hammering or rolling at or above a dull-red heat] When Sulphur is present along with Manganese, it improves the machinability of steels.

Phosphorus :

Phosphorus should not be present above 0.06%, otherwise the steel becomes cold short [i.e., the condition of iron and steel in which it cannot be worked by hammering or rolling at or below a dull-red heat] This property is developed in steels containing more than 0.1% Phosphorus and is marked when the amount of carbon and Phosphorus together is more than 0.3%. Therefore, this effect is more pronounced in medium and high-carbon steels. In low carbon steels, Phosphorus improves yield strength and tensile strength [Jain et al; 1990].

Carbon and Manganese are the two important elements present in steel. By varying the concentration of carbon and Manganese in steel, Physical properties like hardness, tensile strength, ductility and malleability etc., can be changed as per the requirement of the product to be manufactured. Hence it is necessary to determine the concentration of elements present in steel and grade them before they are made into a product.

Salem Steel Plant :

Salem Steel Plant - an unit of Steel Authority of India is the finest producer of stainless steel in the land.

In Salem steel plant , steel in the form of ingots is rolled into final finished product with the help of Hot rolling mill and cold rolling mill[Fig.1&2]. The technology employed here is based on the know - how provided by M/s Usinor Chatillion, now, ugine ACG of France, one of the reputed manufactures of cold rolled stainless steel flat products. Salem steel plant is equipped with modern steel production lines supplied by leading manufactures of steel all over the world. Hence steel coils and sheets from the cold rolling mill complex are characterised by their superior quality.

In Salem steel plant both instrumental methods and conventional methods are available for analysis. The author in this project has determined the concentration of carbon and Manganese in plain carbon steel instrumentally and compared with the values obtained by conventional method.

The author has selected Optical Emission Spectrometer for analysis due to the following reasons.

(i) It requires no chemical separations or concentration.

Samples can be analysed as in received condition.

(ii) It is an excellent method for elemental analysis, as low a concentration level as 0.001% can be determined

(iii) It can be used for all ferrous metals.

(iv) International Standards are available for comparison Viz. BCS (British Chemical Standards).

(v) This method can be used for routine quality control works, and if automated, can provide analytical results on previously selected elements in 30 seconds to one minute[Gurdeep chatwal, 1994].

This project is done with an intention to find out a more suitable and economical method of analysis of steel which involves the application of the theoretical principles of chemistry to practical situations, the very purpose of Applied Chemistry.

Review of Literature

REVIEW OF LITERATURE

The Steel Industry in India:

The production of steel in India is around to million tons per year. It is estimated that the required level of production would be around 22.5 million tons by the year 2000 as per the report of the national mission for Iron and steel.

Development of the Iron and steel Industry in India owes its origin to the vision and perseverance of Jamsetji Nusserwanji Tata. The transformation of his dream in to reality resulted in the installation of the first integrated Iron and steel plant in India, The Tata Iron and Steel Co. Ltd., Which went in to operation in 1911-12[Amit-chaterjee , 1996]'.

At present, there are eight integrated iron and steel plants, in India [Parakh, 1991].

(i) Tata Steel

(ii) Vizag Steel

(iii) Essar Steel

(iv) Bhilai Steel Plant (BSP)

(V) Bokaro Steel Plant (BSL)

(Vi) Durgapur Steel Plant (DSP)

The last five are under steel Authority of India LTD (SAIL). In addition to this SAIL has three special steel plants namely

(1) Alloy steels Plants

(2) Salem Steel Plant

(3) VISL

(The geographical location of these steel plants are given in fig.3)

From the available information on past trends world trade in finished steel, it is observed that, whereas between 1980-1990 global trade amounted to about 25% of global production, by 1993 the figure was almost 33% a review of ratio of exports to steel production for the major steel exporting countries reveals that the ratio has been on the decline in these countries in the recent part, indicating that other steel producing countries have been steadily entering the global steel market[Amit chaterjee 1996]'.

Currently India, ranks tenth among the steel producing countries of the world contributes about 2.5% of the global out-put [Steel News 1996]'.

Improvements in uses of steel

Merely looking at the production of crude steel to determine the maturity of the steel industry has the short coming that it does not indicate the benefit derived by the user. If the same application is considered overtime, then a given quantity of crude steel in 1994 is more useful than was the case 10 years ago.

(1) Manufacturing process yields have increased, which means that more product (Sheet, plate, etc.,) is obtained from the same quantity of crude steel

(2) Less steel is required is perform the same junction

(3) Steel now needs to be replaced less often because of increased use of corrosion resistant and wear resistant-steels[J.C.Vandenberg , 1996]

Problems facing steel industry

Cost:

In the past couple of years, steel plants have faced a stiff challenge to their existence on account of uneconomic operations. Increase in the cost of essential in puts, especially scrap: the international price of shredded scrap has soared;

this uncertainty in price coupled with inadequate availability and deterioration in quality are important factors throttling the steel industry [Amit Chaterjee 1996].

Labour:

The number of personnel required to make one ton of steel can be reduced in the following ways

(i) Larger equipment or higher specific throughput; this has a simultaneous capital and labour cost advantage.

(ii) Equipment that requires fewer people to operate, such as equipment that contains multiple process functions.

(iii) Automation [Vandenberg ,1996][4]

Environmental restrictions

Disposal of waste materials and dust poses health hazards, since the disposal sites are limited. Given these constrains, further unlimited growth, in steel, or the mere continuation of production at current levels, through current production processes is not sustainable - for this reason the concept of `global sustainability' is being introduced to evaluate and assess individual processes, materials and products [M.Tokuda, 1994][3].

Today's steel R & D focus is on

(i) Development of short, automated processes

(ii) Development of high quality products

(iii) Alignment of developments with environmental needs [J.C.Vandenberg 1996][4].

Analysis of steel

The carbon content of a steel has a pronounced effect on the physical and mechanical properties of the finished product. A carbon content in the liquid steel outside the specifications of the desired product can cause reclassification of heat to a lower quality alloy . This can influence inventories, delivery dates, and revenues[Cote et al;1991][2].

Steel makers are constantly required to produce alloys to within ever narrowing tolerances. More precise chemical analysis and better control of the final carbon content are being demanded. At the same time speed is of the essence with rapid and accurate analysis being essential in order to keep steel making installations competitive [Cote et al; 1991][2]

Special steels or alloy steels contains elements such as tungsten, titanium, molybdenum, manganese, tantalum, zirconium, vanaduim etc., which gives various properties to steel. Determination of microgram amounts of molybdenum (VI) and tungsten(VI) in carbon steel, mild steel, plain steel and Mn-Mo steel has been done using spectrometer [Barve et al ; 1994]

Different methods available for analysis of carbon in steel :-

(I) Gravimetric analysis [Cote et al;1991]

(II) Volumetric analysis [Cote et al;1991]

(III) Electrical conductivity [Cote et al;1991]

(IV) Coulometric analysis [Cote et al;1991]

(V) Calorinmeter method [Agarwal et al;1984]

(VI) Combusion method [Agarwal et al;1984]

(VII) Potentiometric method [Cote et al;1991]

(VIII) Infra red absorption [Cote et al;1991]

Manganese in steel :

Manganese is largely used in the form of ferro-manganese as a deoxidiser in the steel industry.It is an important constituent in many alloy steels [Agarwal et al;1984].Manganese increases mechanical and antifriction properties and corrosion resistance[Abramov, 1986].

Different methods available for analysis of manganese are

(i) Bismuthate method [Vogel-1978]

(ii) Persulphate Arsenite method[Vogel-1978]

(iii) Gravimetric method [Agarwal & Jain-1984]

(iv) Calorimetric method [Agarwal & Jain-1984]

Manganese and silion occur in solution, i.e. they are dissolved in the iron and are not visible when a steel sample is examined under microscope. Both elements confer strength and hardness through their influence on the transformation the steel undergoes when cooling from an elevated temperature.[Richard W.Heine et al;1982]

Emission spectrometry

Analytical emission spectrometric methods utilize the characteristic radiation produced when materials are introduced in to thermal or electrical sources. In theory analytical emission spectrometry is applicable to all elements, but using standard techniques, only the metals and metalloids can readily be detected. However, in several instances, specialised techniques have been devised for the determination of the non metals and the permanent gases [Richard N.Kniseley, 1961][c].

The applications of emission spectrography includes

(1) The examination of single metal or an alloy for impurities [Vogel, 1978].

(2) The analysis of an alloy for its general composition, including a search for minor components and traces of impurities [Vogel, 1978].

(3) The analysis of ash of organic substance and other chemicals and other materials[ex.natural waters] amenable to similar treatment[Vogel, 1978].

(4) Detection of contaminants in food.

Chief advantages of spectrographic method are [Vogel, 1978]

(1) The procedure is specific for the element being determined.

(2) This method is time saving, most metals and some non-metals [ex: Phosphorus, Silicon, Arsenic and boron] may be determined.

(3) It may be (and is usually) applied to the determination of small quantities of added constituents or of traces of impurities where conventional methods of analysis are difficult , fail or give less accurate results. Lengthy and difficult separations by chemical methods, example: of zirconium and hafnium and of niobium and tantalum, can be avoided [Vogel, 1978].

A number of analysis are done using emission spectrometer such as [standard methods of analysis of steel & Associated products]

(I) Air pollutants - Metals in air

(II) Determination of metals in Alloys : Iron, steel, ferro-alloys and related products

(III) Analysis of non-ferrous materials

(a) Analysis of aluminium and aluminium base alloys

(b) Trace elements in Non-ferrous metals and alloys

(c) Hydrogen, nitrogen, oxygen in nonferrous metals and alloys.

Experimental Methods

DETERMINATION OF CARBON AND MANGANESE IN STEEL

Conventional Methods

Determination of Carbon using Carbon- Sulphur analyser

Equipment used : Leco-CS-244, Michigan, USA[Fig.4]

Principle :

Total carbon as carbon-di-oxide is detected on a continuous and simultaneous basis. Carbon-di-oxide absorbs Infra red energy at a precise wavelength within the Infrared Spectrum. Energy is absorbed as the gas passes through the cell body in which the Infra red energy is being transmitted; thus at the detector less energy is received. All other infrared energy is eliminated from reaching the detector by a precise wavelength filter. Thus, the absorption of Infrared energy can be attributed to only carbon-di-oxide and the concentration of carbon-di-oxide is detected as changes in energy at the detector.

Instrumentation:

A single cell is used as both reference and a measure chamber. The Infrared cell consists of

(i) An infrared source - The infrared source consists of a nichrome wire which is resistance heated to approximately 850 C. The infrared source radiates visible energy as well as all wavelength in IR Spectrum.

(ii) Chopper Motor

(iii) One precise wavelength filter

(iv) one condensing cone.

(V) One IR energy detector

(vi) and the cell body.

Theory of working:

The radiated energy is chopped at the 85Hz before entering the cell body. The chopped energy enters the cell body through a sapphire window and a precise wavelength filter. The filter selectively passes only the carbon-di-oxide absorption wavelength into a condensing cone which concentrates the energy at the detector. The solid state detector responds to a change in energy and is not wavelength selective; The output of the detector is AC-coupled to the pre-amplifier. This Voltage approximates an exponential function as the gas concentration increases [an exponential decrease in energy received by the detector]

A Starting reference level for the detector is established through the 100% oxygen environment of the flo-thru cell design. The pure oxygen environment permits the maximum amount of energy level which is AC coupled to the Pre-amplifier, Where it is amplified, rectified and filtered. From this point, it is sent to the Analog to Digital card for conversion to a digital signal. The nominal energy which reaches at the cell output, via the Ambient Monitor routine, is 8.500 VDC.

DETERMINATION OF MANGANESE IN STEEL BY WET ANALYSIS

Manganese in steel is usually determined by volumetric method (i.e.) Persulphate-Arsenite method.

Persulphate-Arsenite method:

In this method, Manganese salts are oxidised to permanganic acid by ammonium persulphate in the presence of silvernitrate solution which acts as a catalyst. The permanganic acid is then reduced by a standard Sodium arsenite solution and form the amount of the arsenite solution used, the percentage of Manganese in steel sample is calculated [Agarwal et al;1984].

DETERMINATION OF CARBON AND MANGANESE IN STEEL BY OPTICAL EMMISSION SPECTROMETER

Equipment used :Thermojarrell Ash Atom comp-81 [Fig.5]

The Thermojarrell Ash Atom comp-81 is a direct reading emission spectrometer using high voltage spark as the emission source. The system consists of a spectrometer, source assembly and a personal computer which is used for preparing and sorting analytical programs as well as for data collection and analysis.

Principle :

The purpose of any emission spectrometer is to measure the concentration of elements of interest in a sample. The first step in this process is the measuring of emission intensity produced when a sample containing these elements is excited by a spark. These emission intensities are then converted in to concentrations in percentage.

Instrumentation :

The Thermo jarrell Ash Atom comp-81 is equipped with 0.75 meter focal length paschen range optical system capable of accepting upto 63 channels. The spectrometer is a rugged, heat treated cast iron.

A frame base to which are mounted the entrance slit for each wave length which is to be measured. In addition, the entire spectrometer is carefully baffled to minimise stray light and 'cross talk'a condition where light from one slit reaches a photo multiplier which belongs to a different slit. The entire assembly may be mounted in a vacuum chamber for determination of elements with wavelengths below 190nm. Each exit slit is equipped with its own photo multiplier tube and integrator control, allowing the simultaneous measurement of response for all channels. The system is shown in Fig.6

During assembly, the position of each channel is very carefully fixed in relation to all other channels. Fine adjustments are made with refractor plate mounted immediately infront of each slit. This plate may also serve other purposes, such as order sorting and stray light reduction. The plate is a thin piece of material appropriate for its purpose at the particular wave length. Any slight residual misalignment of the optical system can be corrected by reference to a single channel using a mechanism which is present to allow manual adjustment of the entrance image position on the grating. One of the channels is chosen and the position of the entrance slit image is adjusted so that the maximum intensity reaches the photo multiplier tube. This procedure is called profiling the spectrometer. A mercury lamp is installed in the spectrometer extensively for this purpose.

Source :-

A high voltage spark is used as a source. It converts the sample into a vapor composed primarily of free atoms and ions and then exit this vapor to emit radiation characteristic of the type and the quantity of the atoms present in the vapor.

Theory of Operation :-

The sample is placed in the sparking chamber. A high voltage is applied between the sample and a tungsten electrode. Atoms of different elements presents in the sample get excited and when they come back to their normal state emit light at a wavelength characteristic of the element to which the atoms belong. The sparking is carried out in an atmosphere of Argon gas to avoid interference from oxygen and nitrogen. The number of atoms excited is proportional to the concentration of the elements present in the sample. After excitation emission intensities are measured. The quantum of light emitted by atoms of each element is proportional to its concentration in the sample. The emission intensities are then converted into concentration in percentage. This is done by allowing the combined light to fall on the diffraction grating via an entrance slit. The grating disperses the light into spectrum. Since the wavelengths of different elements are known, the position of these wavelengths in the spectrum are also known. Wavelengths of interest are allowed to pass through small openings called slits on the screen.

The light that emerges out of the slit falls in a photomultiplier which converts the light energy into electrical energy. The electrical energy is measured for each element and expressed in terms of concentration.

Experimental Procedure

EXPERIMENTAL PROCEDURES

Conventional Methods

Determination of concentration of carbon by LECO-CS-analyser:

Before analysis the 'starting Reference level' is read by the computer. Then this level is adjusted digitally until the nominal level is achieved. About one gram of steel is taken in a porcelein crusible and little of tungsten flux is added and introduced into the furnace. As the analysis begins the cell output decreases proportionally with the amount of carbon (as carbon-di-oxide) present in the cell. The computer reads the cell output four times per seconds [The results are given in table-1].

DETERMINATION OF MANGANESE

Persulphate -Arsenite method

Solutions required:

(a) Silver nitrate solution - Dissolved 1.3gm of the salt in one litre of distilled water.

(b) Ammonium per sulphate solution - Dissolved 25gm of the salt in 100ml distilled water.

(c) Sodium chloride solution - Dissolved 1gm of Nacl in 100ml of water.

(d) Standard Sodium Arsenite Solution - 0.1N.

Procedure : -

Weighed about 0.1gm of the sample. Dissolved the sample in 20cc of nitric acid (50cc of H2O and 30cc of HNO3) heated the solution and boiled to expel nitrous fumes. Added 1cc of Ammonium persulphate and boiled for 10 to 15 minutes to oxidise any reducing agents present in the solution and to destroy the excess of persulphate. Added 10cc of Silver nitrate and 15cc of Ammonium persulphate stirred well, boiled for half an minute. The solution would turn pink cooled to room temperature. Diluted the solution to 100ml taken 10ml of the solution and added 10ml of Sodium chloride Solution. Titrated immediately with 0.1N Sodium Arsenite solution. End point is the disappearance of pink colour [The results are given in table-2].

DETERMINATION OF CARBON AND MANGANESE IN STEEL BY OPTICAL EMMISSION SPECTROMETER

Experimental procedure :

Calibration :

The first step is the calibration of the equipment. Calibration refers to the process of analysing many standards, in order to acquire data necessary to establish the relationship between intensity ratio and concentration. This is done in a 3 part procedure.

1. A table is prepared containing the standard names and element concentrations.

2. The standards are run and the intensity ratios are stored in the file of calibration standards data.

3. Finally, the data is evaluated to determine the best fit for the analytical data of know concentrations. A graph is drawn between concentration and intensity of absorption [Fig.7&8].The results are given table 3&4

Standardization :

The next step is standardization. It is a procedure for relating the intensity ratio now to the intensity ratio at the time the instrument was calibrated. It relies on the use of two standards.

The output of the standardisation Algorithm is standardised Intensity ratio(SIR)

$$SIR = M\ IR + b$$

IR = Intensity ratio now. M & b are slope and intercepts of the line which relates the current intensity ratios to the originally measured.

The standards were sparked and the values are obtained. The standardisation is done before and after the completion of the experiment to make sure that equipment works properly during the period of analysis.

The final step is the determination of concentration of carbon and manganese in samples. The samples are first polished on a 60 grit Aloxite emery paper and placed on the plate holder with the polished surface face down. The sample is sparked and the concentration data is read out on the personal computer[The results are given in table-5&6]. The sample is sparked twice to get accurate results. The results obtained from emission spectrometer is compared with the results due to usual method of analysis.

Results and Discussion

RESULTS AND DISCUSSION

The results of the conventional method and the instrumental method are given in the tables 1 to 6

The results of the statistical analysis are given in appendices I

Comparison between the standard method and the new method is given in appendices II

Discussions:

Analytical measurements are subjected to t-test inorder to ascertain whether there is any significant difference between the new method and the standard method already accepted for these measurements.

Since the experimental value is less than the critical value of t, it is evident that there is no significant difference between the two methods. This is shown in bar diagram. [Fig 11 &12]

From the results obtained [Appendices II] it is clearly known that the time and cost for analysis by instrumental method is lower than that of the standard method.In conventional method it takes nearly 6 hours to analyse carbon and manganese in steel, whereas in instrumental method it takes 1hour and 30 minutes to analyse carbon & manganese in steel.Not only carbon and manganese all other elements present in steel, can also be determined simultaneously within this time scale and the cost for analysis will not increase with the increase in number of elements, since the cost is only due to electricity in the case of instrumental method of analysis. The non-use of chemicals, in this project using optical emission spectrometer, renders it pollution free which is an added advantage.

Conclusion

SUMMARY AND CONCLUSION

From the data obtained so far, it is obvious that by the use of spectrometer for the analysis saves valuable amount of time, man power and helps to make right decisions at right moment. The results obtained by using spectrometer when compared with that of the usual method of analysis are very close to each other the possibility of experimental errors due to human mistakes is eliminated in this method. Though it may seem the initial cost of installation of spectrometer with AC arrangements is high, in the long run it is economical. As a consequence of faster results obtained by instrumental analysis the production will increase resulting in huge profits off-set the initial installation cost of the instruments.

The major problem threatening the world is the problem of pollution. Since this method uses no chemicals at all the pollution due to waste chemicals can be prevented. This is another striking feature of this project work.

This project is mainly confined to the determination of carbon and manganese in steel. The concentrations of other elements such as sulphur, phosphorus, silicon and any other alloying element present in steel can also be determined successfully using optical emission spectrometer.

Since the concentrations of all the elements present in steel can be determined simultaneously within 30 seconds to one minute, this method is highly advantageous. Here the author has analysed only plain carbon steel and low carbon steel. In addition to this stainless steel and all types of ferrous materials can be analysed by this method. In this equipment about 55 elements can be determined as such, but provisions are given so that 30 more elements can be determined. Special attachments are provided for analysing liquid and gaseous samples. Therefore not only the products, the raw materials can also be analysed thereby saving considerable amount of time. This method can be used for routine quality control works, for pollution monitoring, for rapid analysis of gases like oxygen, nitrogen, for water testing and for any other purposes involving elemental analysis.

Thus we can conclude that the method of analysis by optical emission spectrometer is the most suitable and economical method for large scale industries.

Tables

Determination of Carbon by Leco Analyser

SNo.	Coil Number	Concentration %
1	A14946	0.05776
2	A14959	0.0695
3	A12738	0.0806
4	A12922	0.1509
5	A12427	0.06645
6	A13258	0.06928
7	A15031	0.03508
8	A11593	0.1507
9	A12722	0.06617
10	A12907	0.07735
11	A15035	0.06
12	A15026	0.06
13	A14705	0.045
14	A14709	0.05
15	A14718	0.058
16	A15206	0.07
17	A15230	0.063
18	A15231	0.07
19	A15266	0.087
20	A15268	0.064
21	A14974	0.072

Table-1

Determination of Manganese by Per Sulphate-Arsenite Method

S.No.	Coil Number	Concentration %
1	A14946	0.32
2	A14959	0.45
3	A12738	0.52
4	A12922	0.87
5	A12427	0.55
6	A13258	0.92
7	A15031	0.47
8	A11593	0.92
9	A12722	0.47
10	A12907	0.55
11	A15035	0.4
12	A15026	0.4
13	A14705	0.55
14	A14709	0.39
15	A14718	0.4
16	A15206	0.47
17	A15230	0.37
18	A15231	0.4
19	A15266	0.47
20	A15268	0.4
21	A14974	0.52

Table-2

Calibration Data

Carbon

Std.No	Concentration %	Intensity Ratio
1	0.038	0.0319
2	0.095	0.077
3	0.102	0.0821
4	0.14	0.113

Table-3

Manganese

Std.No.	Concentration %	Intensity Ratio
1	0.41	0.0692
2	0.6	0.1028
3	0.86	0.1463
4	1.34	0.2284

Table-4

Determination of Carbon by Optical Emission Spectrometer

S.No.	Coil Number	Concentration %	Intensity Ratio
1	A14946	0.055	0.451
2	A14959	0.065	0.054
3	A12738	0.075	0.061
4	A12922	0.145	0.1278
5	A12427	0.056	0.0455
6	A13258	0.07	0.0566
7	A15031	0.037	0.0299
8	A11593	0.15	0.121
9	A12722	0.065	0.0531
10	A12907	0.077	0.0622
11	A15035	0.06	0.0506
12	A15028	0.06	0.05
13	A14705	0.04	0.0325
14	A14709	0.046	0.0372
15	A14718	0.055	0.0448
16	A15206	0.075	0.0607
17	A15230	0.065	0.0528
18	A15231	0.07	0.0572
19	A15266	0.08	0.0652
20	A15268	0.065	0.0529
21	A14974	0.07	0.0585

Table-5

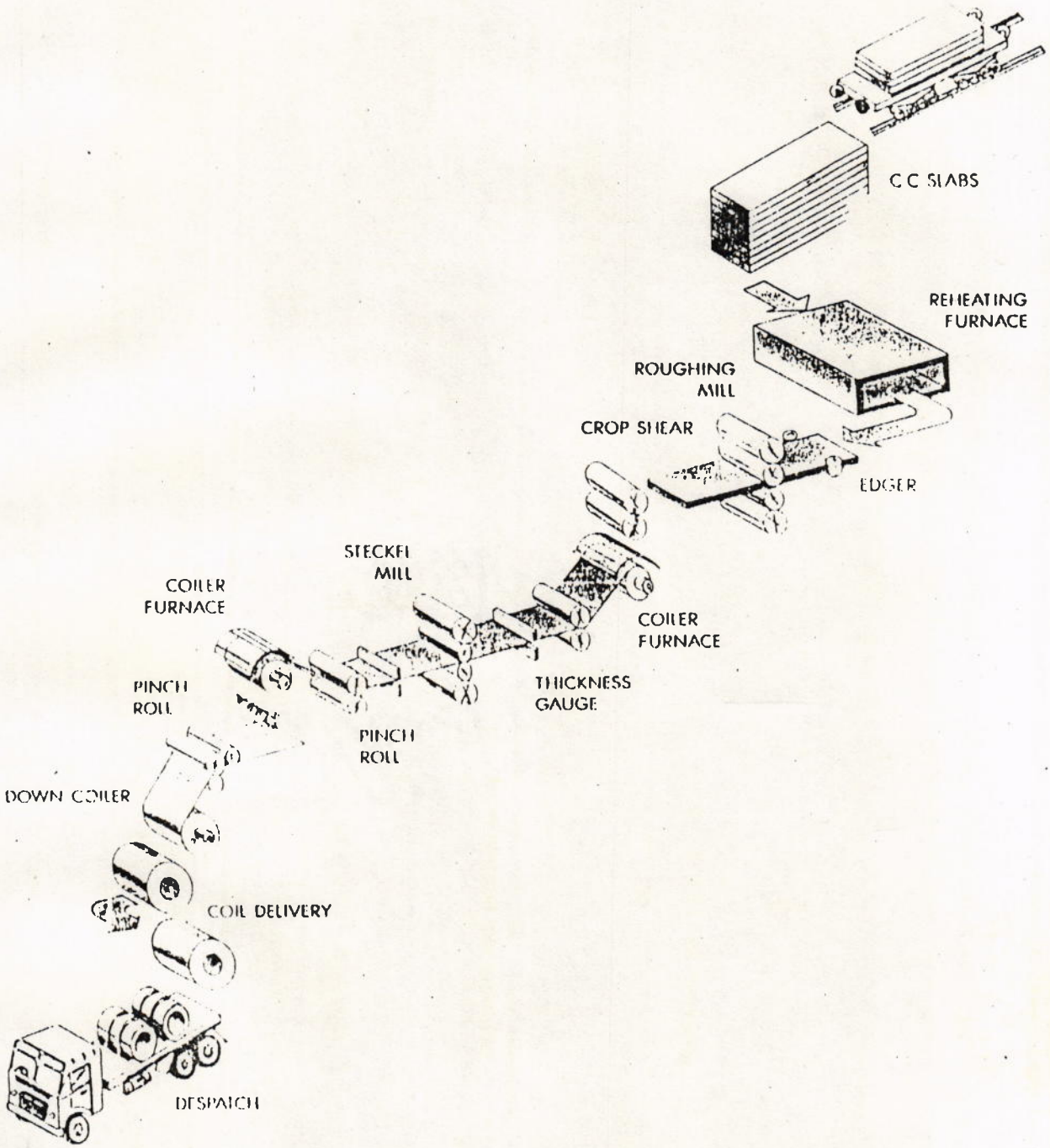
Determination of Manganese by Optical Emission Spectrometer

S.No.	Coil Number	Concentration %	Intensity Ratio
1	A14946	0.32	0.0519
2	A14959	0.43	0.0731
3	A12738	0.51	0.08662
4	A12922	0.86	0.1459
5	A12427	0.53	0.0908
6	A13258	0.9	0.1521
7	A15031	0.44	0.0741
8	A11593	0.9	0.1534
9	A12722	0.45	0.0767
10	A12907	0.53	0.0901
11	A15035	0.38	0.063
12	A15026	0.38	0.0632
13	A14705	0.53	0.0898
14	A14709	0.38	0.0638
15	A14718	0.36	0.06
16	A15206	0.45	0.076
17	A15230	0.35	0.06
18	A15231	0.38	0.0634
19	A15266	0.45	0.0759
20	A15268	0.37	0.0608
21	A14974	0.48	0.0817

Table-6

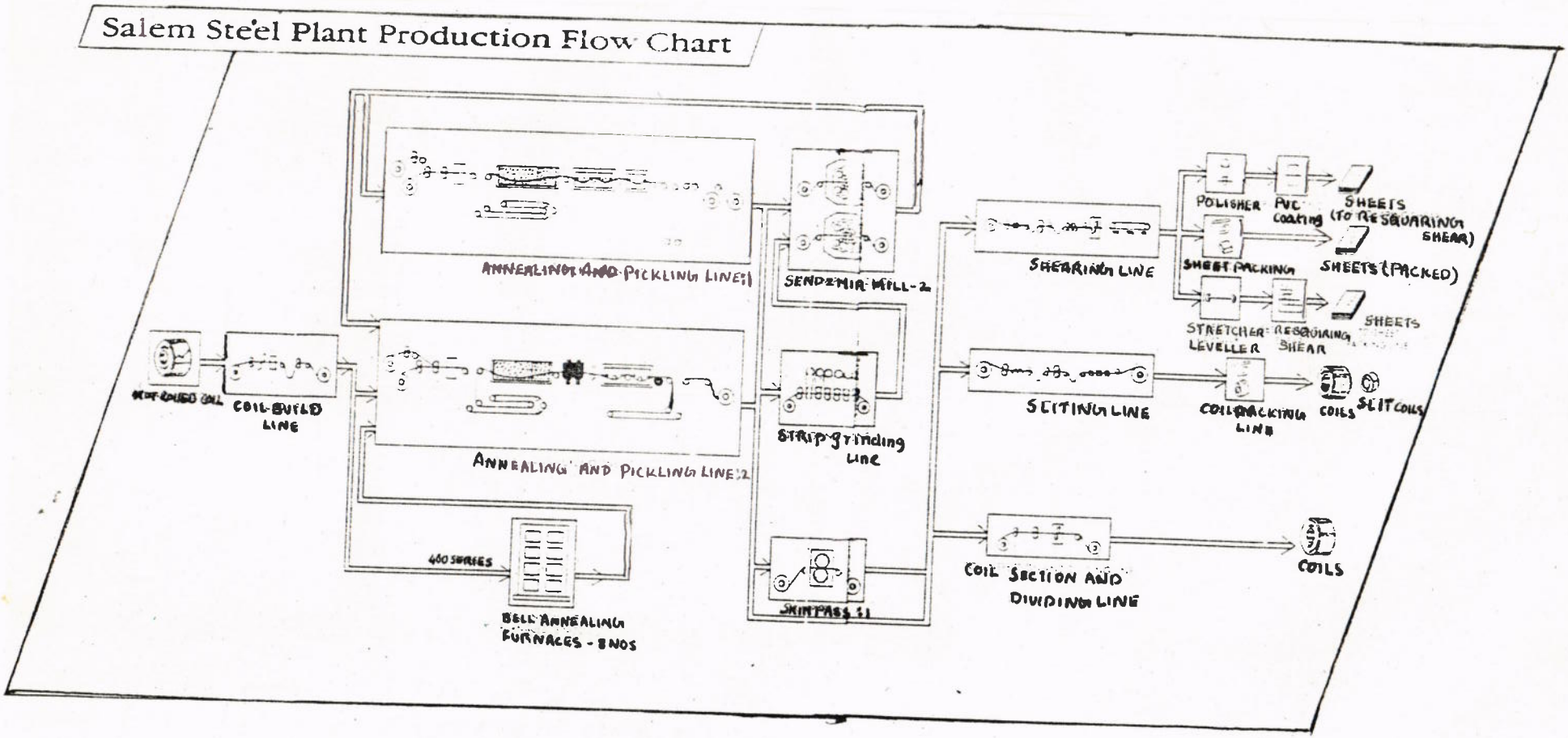
Figures

FIG (I)



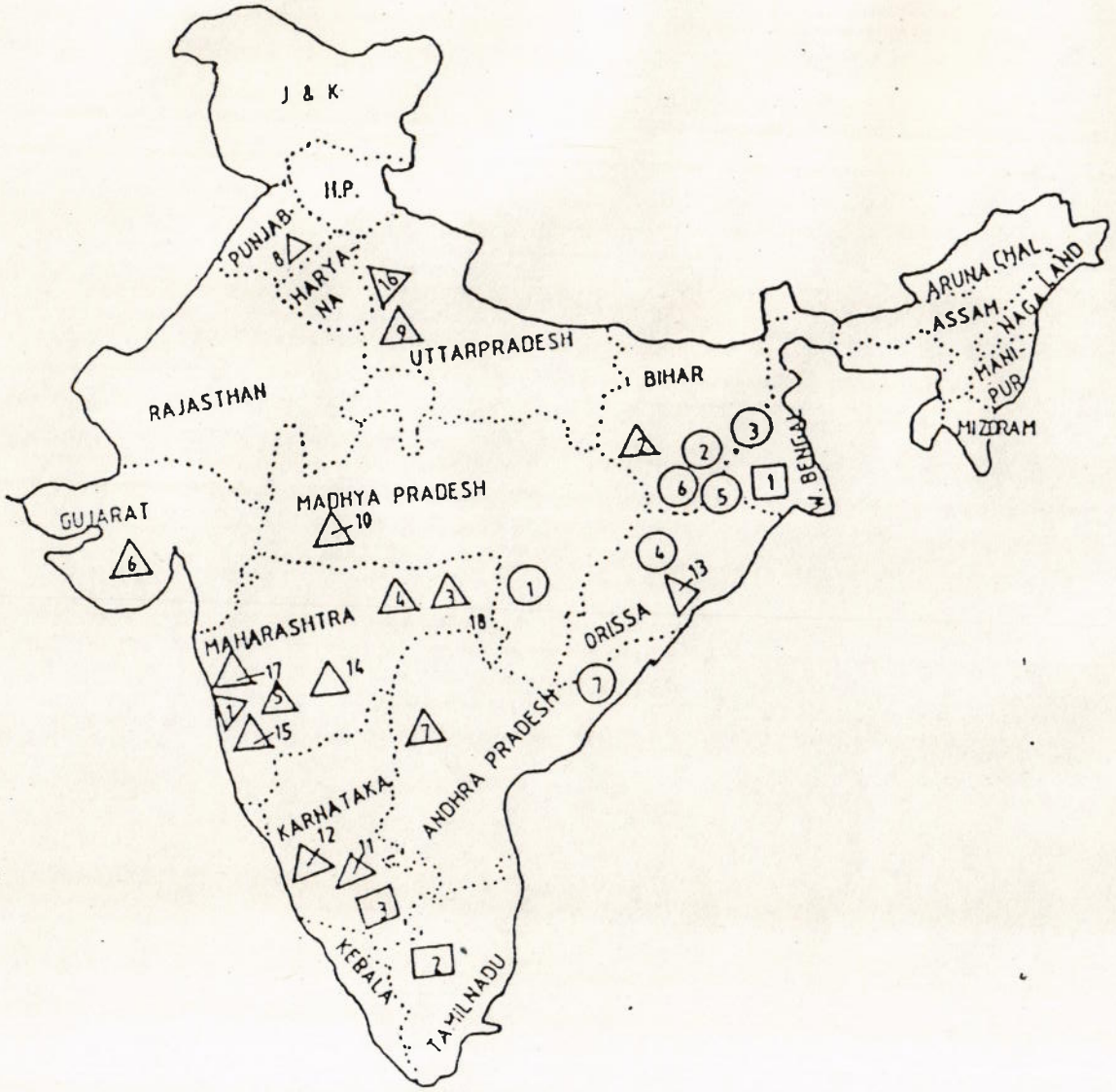
Hot Rolling - Flow Chart

FIG (II)



Cold Rolling - Flow Chart

FIG. (III)



Geographical location of various steelplants in India

MAIN PRODUCERS

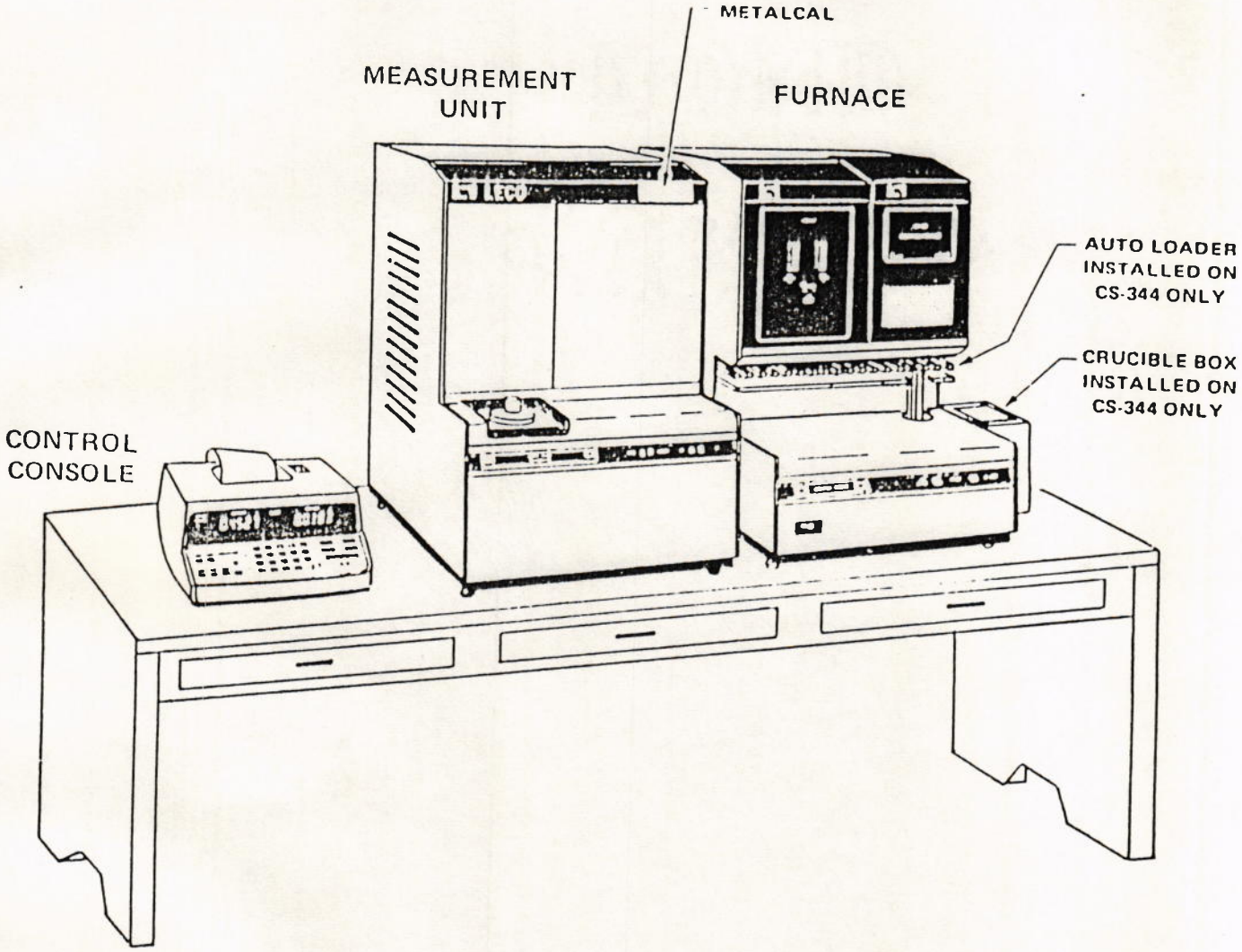
1. BHILAI STEEL PLANT
2. BOKARO STEEL PLANT
3. DURGAPUR STEEL PLANT
4. ROURKELA STEEL PLANT
5. IISCO
6. TISCO
7. VSP

MAJOR SECONDARY PRODUCERS

1. JINDAL I & S CO.LTD.
2. USHA ALLOYS & STEEL LTD.
3. NIPPON DENRO ISPAT LTD.
4. SUNFLAG I & S CO. LTD.
5. ISPAT PROFILS LTD.
6. ESSAR GUJARAT LTD.
7. NAGARJUNA STEELS LTD.
8. PUNJAB CON-CAST LTD.
9. RATHI ALLOYS & STEELS LTD.
10. SHRI ISHAR ALLOYS & STEELS
11. JINDAL VIJAYANAGAR STEEL
12. BHORUKA STEEL LTD.
13. MID-EAST I & S CO.
14. KALYANI STEELS LTD.
15. MUSCO LTD.
16. BHUSHAN STEEL & STRIPS
17. MUKAND LTD.
18. LLOYDS STEEL LTD.

SPECIAL STEEL PLANTS

1. ALLOY STEELS PLANT
2. SALEM STEEL PLANT
3. VISL



CS-244/CS-344 SYSTEM
FRONT VIEW
FIGURE 4

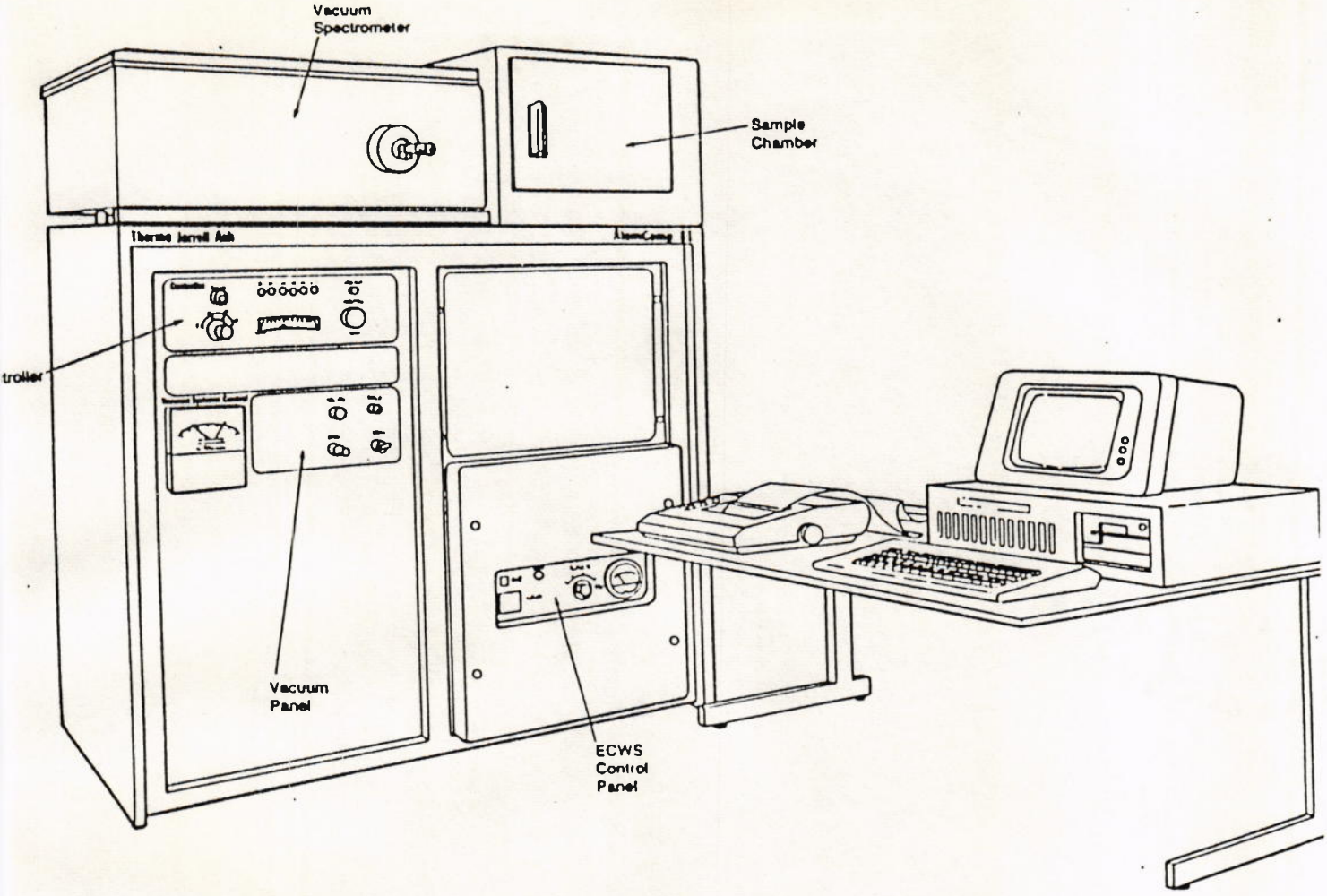


Figure 5 : Atomcomp 81 Spectrometer System.

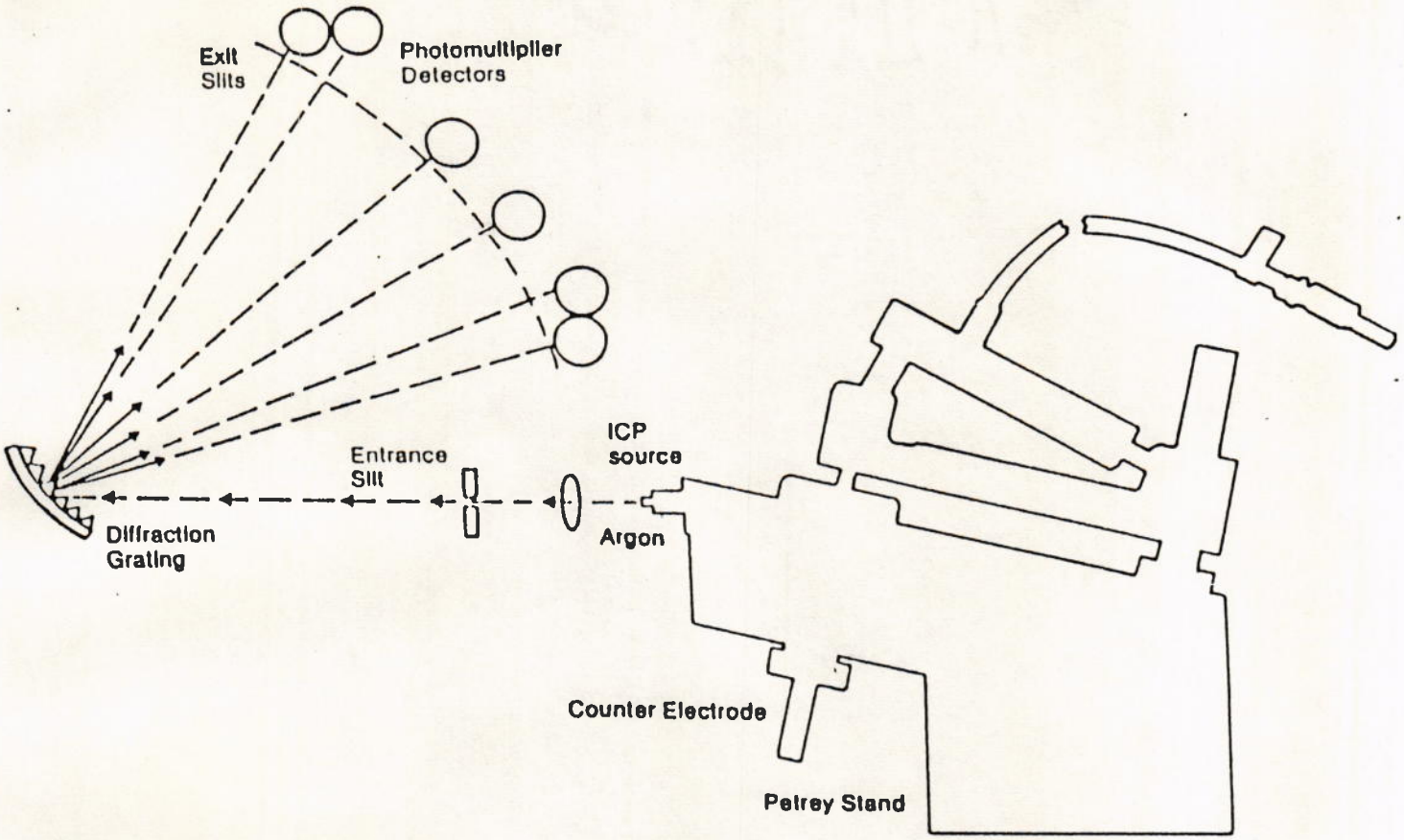
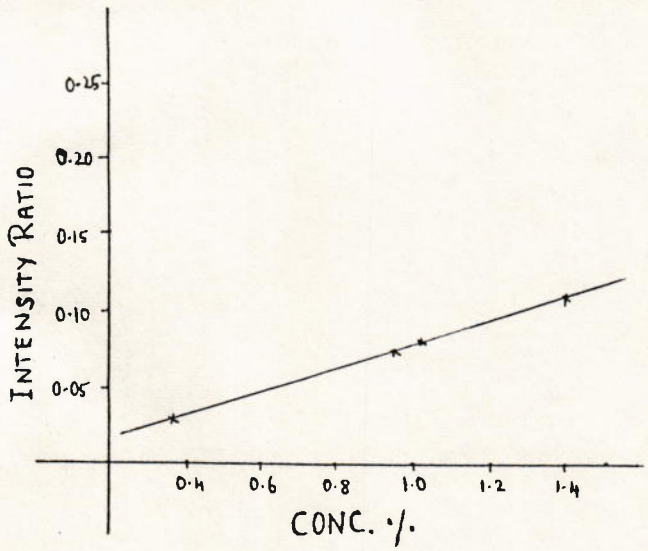


Figure.6 ; Atomcomp 81 Polychromator System.

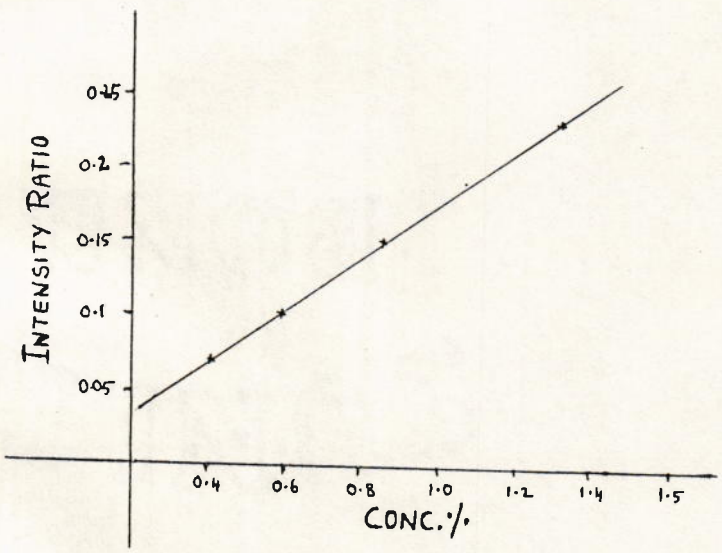
Calibration Curve

Carbon



Calibration Curve

Manganese



BAR DIAGRAM

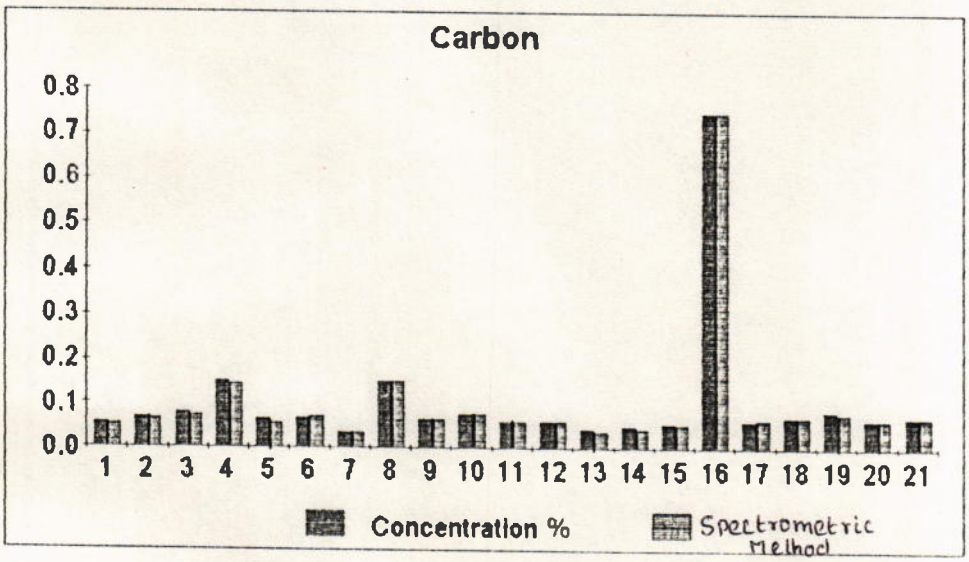


Fig. 11

BAR DIAGRAM

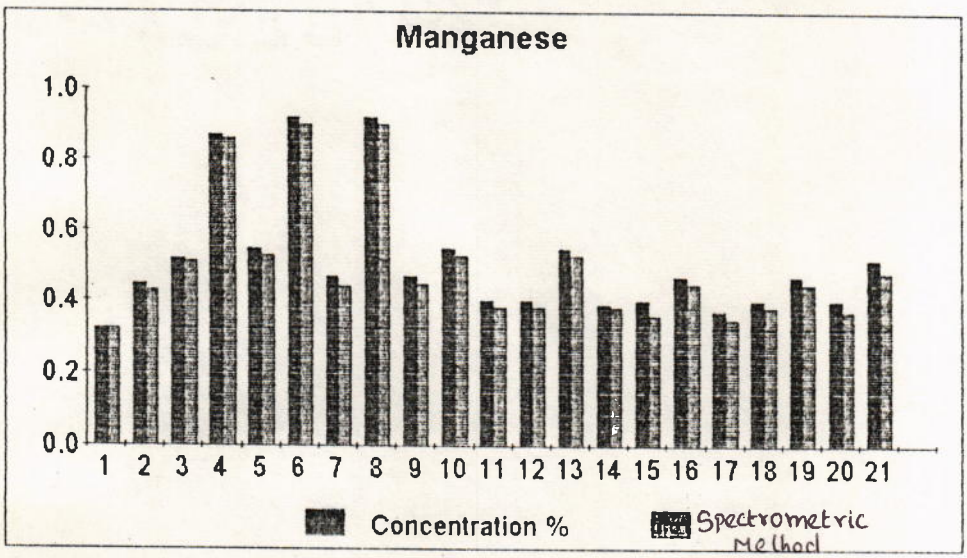


Fig. 12

Appendices

APPENDICES-I

Statistical Results

Determination of Carbon by Leco Analyser

Concentration %		
x_i	$x_i - \bar{x}_1$	$(x_i - \bar{x}_1)^2$
0.05776	0.1499	0.0002247
0.0695	0.00325	0.000010562
0.0806	0.00785	0.000061622
0.1509	0.07815	0.00611
0.06645	0.0063	0.00003969
0.06928	0.003472	0.000012087
0.03508	0.03767	0.00142
0.1507	0.07795	0.0060762
0.06617	0.00658	0.000043296
0.07735	0.004598	0.000021141
0.06	0.01275	0.000162562
0.06	0.01275	0.000162562
0.045	0.02775	0.00077
0.05	0.02275	0.0005176
0.058	0.01475	0.0002176
0.075	0.00225	0.0000051
0.063	0.00975	0.000095062
0.07	0.00275	0.000007562
0.087	0.0142	0.0002017
0.064	0.00875	0.0000765
0.072	0.000752	0.000000565

$\bar{x}_1 = 0.07275$ $\qquad$ $\sum (x_i - \bar{x}_1)^2 = 0.03247$

where $\bar{x}_1$ = average concentration of carbon
N = Number of observations
$S_1 = 0.04029$

Determination of Carbon by Optical Emission Spectrometer

Concentration % x_i	$x_i - \bar{x}_2$	$(x_i - \bar{x}_2)^2$
0.055	0.0155	0.0002402
0.065	0.0055	0.0000302
0.075	0.0045	0.0000202
0.145	0.0745	0.0055502
0.056	0.0145	0.0002102
0.07	0.0005	0.0000002
0.037	0.0335	0.0011222
0.15	0.0795	0.0063202
0.065	0.0055	0.0000302
0.077	0.0065	0.0000422
0.06	0.0105	0.0001102
0.06	0.0105	0.001102
0.04	0.0305	0.0009302
0.046	0.0245	0.000002
0.055	0.0155	0.0002402
0.075	0.0045	0.0000202
0.065	0.0055	0.0000302
0.07	0.0005	0.0000002
0.08	0.0095	0.0000902
0.065	0.0055	0.0000302
0.07	0.0005	0.0000002

$\bar{x}_2 = 0.0705$

$\sum (x_i - \bar{x}_2)^2 = 0.01573$

$$S_2 = \sqrt{\frac{\sum (x_i - \bar{x}_2)^2}{N - 1}}$$

$\bar{x}_2$ = Average concentration of carbon in %

N = Number of observations

S_2 = 0.02804

t-Test

$$S_2 = \frac{n_1 s_1^2 + n_2 s_2 2}{n_1 + n_2 - 2}$$

where n_1 = Number of Observations in Standard method
n_2 = Number of Observations in the new method

$$t_{obs} = \frac{\bar{x}_1 - \bar{x}_2}{s\sqrt{1/n_1 + 1/n_2}}$$

$t_{obs} = 0.25$
Critical Value of t is 1.96
Since $t_{obs} < t$ there is no significant difference between the two methods

Determination of Manganese by Per Sulphate - Arsenite Method

Concentration % x_i	$x_i - \bar{x}_1$	$(x_i - \bar{x}_1)^2$
0.32	0.195	0.03798
0.45	0.064	0.004144
0.52	0.0052	0.0000273
0.87	0.3552	0.1261805
0.55	0.0352	0.00124
0.92	0.4052	0.1642024
0.47	0.0447	0.0020008
0.55	0.0352	0.00124
0.4	0.1147	0.0131631
0.4	0.1147	0.0131631
0.55	0.0352	0.00124
0.39	0.1247	0.0155578
0.4	0.1147	0.0131631
0.47	0.0447	0.0020008
0.37	0.1447	0.020947
0.4	0.1147	0.0131631
0.47	0.0447	0.0020008
0.4	0.1147	0.0131631
0.52	0.0052	0.0000273

$\bar{x}_1 = 0.515$

$\Sigma(x_i - \bar{x}_1)^2 = 0.4479$

$$S_1 = \sqrt{\frac{\Sigma(x_i - \bar{x}_1)^2}{N - 1}}$$

where $\bar{x}_1$ = Average concentration of Manganese in percentage
N = Number o f observations
S_1 = 0.1496

Determination of Manganese by Optical Emission Spectrometer

Concentration % x_i	$(x_i - \bar{x}_2)$	$(x_i - \bar{x}_2)^2$
0.32	0.1742	0.0303605
0.43	0.0642	0.0041271
0.51	0.0157	0.0002467
0.86	0.365	0.1334857
0.53	0.0357	0.001275
0.9	0.4057	0.1645982
0.44	0.4378	0.1916798
0.9	0.4057	0.1645982
0.45	0.0442	0.0019536
0.53	0.0357	0.001275
0.38	0.1142	0.0130514
0.38	0.1142	0.0130514
0.53	0.0357	0.001275
0.38	0.1142	0.0130514
0.36	0.1342	0.0480211
0.45	0.0442	0.0019536
0.35	0.1442	0.0208059
0.38	0.1142	0.0130514
0.45	0.0042	0.0019536
0.37	0.1242	0.0154362
0.48	0.01428	0.0002039

$\bar{x}_2 = 0.4943$

$\Sigma (x_i - \bar{x}_2)^2 = 0.8055$

$$S_2 = \sqrt{\frac{\Sigma (x_i - \bar{x}_2)^2}{N - 1}}$$

where $\bar{x}_2$ = Average concentration of Manganese in percentage

N = Number of observations

$S_2 = 0.2007$

t - Test

$$S = \frac{n_1 s_1 + n_2 s_2^2}{n_1 + n_2 - 2}$$

n_1 = Number of Observations in the Standard method
n_2 = Number of Observations in the New method

$$t_{obs} = \frac{\bar{x}1 - \bar{x}2}{S\sqrt{1/n_1 + 1/n_2}}$$

$t_{obs} = 0.46$

The critical value of t is 1.96
Since $t_{obs} <$ t there is no significant difference between the two methods.

APPENDICES-II
COMPARISON BETWEEN CONVENTIONAL METHOD AND INSTRUMENTAL METHOD

Time for analysis

No.ofsamples	Conventional method			Instrumental method (Spectrometer)
	Carbon	Manganese	Total	both carbon and Manganese
21	50 mins	5 hours	5.50 hours	one hour and thirty minutes

Chemicals used for analysis

No. of samples	Conventional method		Instrumental method (Spectrometer)
	Carbon	Manganese	
21	Tungsten flux	Ammonium-per sulphate Nitric acid Silvernitrate Sodium arsenite Sodium chloride	No chemicals used

Cost for analysis

No. of samples	Conventional method					Instrumental method (Spectrometer)
	Manganese		Carbon			
	Name of the chemical	Cost Rs.	Name of the chemical	Cost Rs.	Total Rs.	
21	Ammonium per sulphate Nitric-acid Silver-Nitrate Sodium-Arsenite	25.20 8.12 3.95 3.85	Tungsten	3	43.10	cost is only due to electricity which is negligeble amount

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