

# CHEMICAL ANALYSIS AND STANDARD REFERENCE MATERIALS

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Chemical characterisation and indepth knowledge of any sample require establishments of types and amount of species present in that material. Analytical chemistry is directed to these answers. Qualitative analysis comprises the tests that enable the chemist to determine the types of species as well as their states of combinations. Quantitative analysis provides the means to determine the exact amount of the species in a unit quantity of sample.

Both qualitative and quantitative analysis resemble one another in some respects and differ in others. Both require (i) the measurements of some chemical or physical property and (ii) preliminary treatment steps to ensure the measurement of only the component of interest. In contrast to these similarities, many problems are unique only to quantitative measurements. For example, analyst must avoid the loss of the component during analysis. Secondly the base reaction must proceed to completion. Quantitative information is not a prerequisite for a successful qualitative analysis whereas qualitative description of the sample is normally essential for selection of a suitable quantitative method.

In this industrially developed world, the role and importance of chemical analysis cannot be underestimated. Virtually every item of commerce has been subjected to analytical testing at one or more stages of its manufacture. The clothes we wear, the food we eat, the drugs we need, the automobile we drive - all these and many more require the aid of chemical analysis to assure the production of goods having required quality, characteristics and specification.

In addition to these considerations the study of quantitative chemical analysis requires development of systematic and orderly work habits as well as intellectually honest observations. These traits are worthy of cultivation regardless of one's ultimate field of endeavour.

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Ultimately in every quantitative analysis, there is a final measurement whose magnitude can be related to amount of substance to be determined. Accuracy is the prime consideration in any quantitative chemical analysis. There are various factors & considerations to achieve accuracy. Standard Reference Materials are one such means. These materials are commonly known as standard samples.

The standard samples are of vital importance to metallurgical and mineral industries for rapid and accurate analyses of the raw-materials and finished products. Research and other institutions use them for checking their results, instruments and adopted methods of analysis require large varieties of standards. Non-availability of standard samples in India caused drainage of foreign exchange and it is often observed that many of the imported standards are quite different in composition from what the users in our country would prefer.

A standard sample is a material closely resembling in chemical and physical nature the materials with which the analyst expects to deal and one that has been analysed and tested by a sufficient number of methods and analysts to establish its composition and reliability for the intended use.

In order to make our country self sufficient and self reliant and to save foreign exchange, National Metallurgical Laboratory (NML) started a project for preparation of standard samples as early as 1964 and now we are able to cater the need of the users of our country as indicated in the pi-chart (Fig-1). A collaborative project with National Bureau of Standard of U.S.A was also started to prepare standard samples of high carbon ferro-manganese, low carbon ferro-manganese and ferro-titanium and the task was successfully completed.

## **METHODS OF PREPARATION**

Preparation of Standard Samples involves the following eight stages:

- (1) Selection and procurement of material
- (2) Examination of procured material
- (3) Reduction to a suitable size
- (4) Testing of homogeneity
- (5) Mixing
- (6) Chemical Analysis
- (7) Certification
- (8) Storage

### ***Selection and procurement of material :***

The substance to be taken up for preparation of standard sample is decided by market survey. The other important factor which is taken into consideration while selecting the material is its homogeneity.

In procuring materials, considerable difficulties and delays are experienced as most of the required materials are not available in the open market. Quotations are called giving full specifications. The sample is first examined for specifications prior to placing order. The suppliers are told the importance of specifications and homogeneity. Moreover, due to inherent inhomogeneity in ferro-alloys and alloy-steels, as well as variation in composition from melt to melt, it is necessary to procure the material from the same melt. In many cases for inspection and collection of representative samples from the bulk, project scientists have to visit the suppliers. Due to these difficulties, we, in NML, have carried out melting, casting and forging for many of the samples. It will not be improper to mention here that a great expertise is required in this work, otherwise all efforts will be in vain. We are lucky in this respect to have many such experts in our institution. Sometimes even local factories also help us in these treatments.

### ***Examination of procured material :***

Before machining the material for preparing standard sample, it is checked again for its homogeneity. This is carried out by sulphur prints and the analysis of samples taken from different area of the cross-section. The chemical analysis is carried out for the different constituents of the material. Generally outer layers and ends of bar-samples are rejected because these areas are more prone to oxidation and segregation.

### ***Reduction to a suitable size :***

After selecting the required material and rejecting the unwanted portions, the next step is to reduce it to a particle size which requires no further preparation for chemical analysis. Steels, brass or other malleable materials, are machined to fine drillings on lathe machine. The drillings are then broken down to smaller sizes. Vary fine particles



are rejected by sieving. In case of ferro-alloys the materials are first broken into small pieces so that it can conveniently be fed into jaw crusher. After then it is passed through roll crusher and disintegrator to get fine particles. Materials of suitable size are separated by a multi panelled sieve shaker, coarse particles are recirculated in the operations while the finest are rejected.

### ***Testing of homogeneity :***

It is practically impossible to crush or machine all materials in one batch. Different fractions of the sample are collected separately and analysed. If the analyses of the constituents do not vary appreciably, it indicates the homogeneity of the sample and then only these fractions are mixed. In case some major variations are observed in some fractions, it is rejected. Generally such situation seldom arises when precautions are taken in the beginning.

### ***Mixing :***

After testing the homogeneity, all the suitable fractions are mixed in a special cuboid mixer made of stainless steel. Magnetic separation is done for ferrous material in case of refractories and non-ferrous samples.

### ***Chemical Analysis :***

Most accurate analysis for the samples are required. For this, very experienced and good analysts are selected so that most accurate analysis of the sample may be arrived at. First of all, the sample is analysed in our Analytical Section adopting most standard techniques of analysis of the constituents. When the composition is established by our analysts, the sample is sent to various outside independent parties. They are told in advance about the importance of the sample. In selecting the analysts we see their experience in analysing such samples and their past performance. For example, in sending aluminium and aluminium alloys we select parties who have sufficient experience in analysing aluminium and aluminium alloy samples. The list of analysts include Government organisations, manufacturers, users etc.

A bulk representative sample is divided into a number of portions corresponding to the number of analysts. Some portion of the representative sample is kept in reserve, in case some analysts requires more sample. Normally sufficient amount of samples are sent to the parties so that they can do checking and rechecking of their analytical results. Normally they are not advised about the methods to be followed but they are told that it should be standard and referee status. If the same results are obtained by various methods, it helps us in finalising the most accurate values. During our analytical campaign sometimes it so happens that we ask some of the participating analysts to check their results in case variation is more than the permissible limit.

### **Certification :**

On receiving the analytical results of all the participating analysts, the results are statistically evaluated and standard deviation calculated. Only those values are incorporated in our certificates which are within the limit, average value  $\pm 2S$ . We issue a certificate for each standard sample which gives the analytical results of each constituents received by all the participating analysts. This also gives the gist of methods followed by them. Every bottle of the sample, which is sold, is provided with such a certificate.

### **Storage :**

The materials are to be preserved for long periods and hence proper care should be taken from moisture and contamination. Fused calcium chloride or silica gel are used to prevent entry of moisture. The samples are protected from being contaminated with dehydrating agent.

## **SPECTROGRAPHIC STANDARDS**

Most of the procedures followed in chemical standards are also applicable to spectrographic standards. Homogeneity plays a more important role in spectrographic standards than chemical standards as these samples remain in disc form and thus they cannot be mixed in mixer like chemical standards. Processing like melting, casting, forging etc. should be done very carefully by foundry experts. The final material is very carefully examined both spectrographically and chemically

to confirm the homogeneity of the bulk sample. For better confirmation about the homogeneity, the sample may be further tested by instruments like electron microprobe analyser at various points. In case, concentrations are plotted against distance and a straight line obtained, it proves perfect homogeneity of the sample. On confirming the homogeneity of the bulk sample, it is cut into pieces of required shape and polished. Like chemical standards these should also be stored in a dry atmosphere to avoid corrosion.

The samples are used by laboratories using direct reading spectrographs <sup>or</sup> X-ray fluorescence spectrometers for their day today requirements.

## **PRECAUTIONS**

All the standard samples should be prepared under rigorous laboratory conditions to exclude the possibility of any error in sampling and analysis. All the samples supplied should be in a form requiring no further preparation for analysis. Machining, crushing, grinding operations should be carried out in a atmosphere free of contamination and hence work should be taken up in a special section which is free from pollution. All staff of standard sample section should be well trained and well informed about the importance of the work and they should take all the required precautions to prevent contamination. Smoking should be strictly prohibited in the machining room to avoid any contamination. After preparing one sample, the machines should be properly cleaned and properly rinsed with the sample to be taken up. Lathe machines, crushers, grinders, mixers should be well separated from each other.

It may be apparent that the task of preparing standard samples is not simple, it needs a well established analytical laboratory and a band of expert analysts. The composition of the sample should be first established by manufacturer before sending to other outside parties. Rigorous statistical evaluation of the analytical results should be carried out before final certification.

## CONCLUSION

Until three decades ago, the quantitative analyses were carried out by conventional wet analytical methods. Now-a-days, the task has become easier due to adoption of new instrumental techniques. Most of the analytical loads in laboratories are being diverted to instrumental sections, though at the same time conventional analytical sections retain their firm position because the techniques are absolute in nature whilst the instrumental results are comparative. All the analytical instruments and newly developed analytical techniques are standardised by primary standards only.

Anywhere in the world, industrialization creates demands for more analytical instruments for quality control thus needing more number of standard samples. In addition to many chemical standards large varieties of spectrographic standards will be required to cater to the needs of industries in future. Considering these aspects, NML is planning to expand its programme of standard samples to meet the growing demand of the country.



# STANDARD REFERENCE MATERIALS DISTRIBUTION AMONG USERS

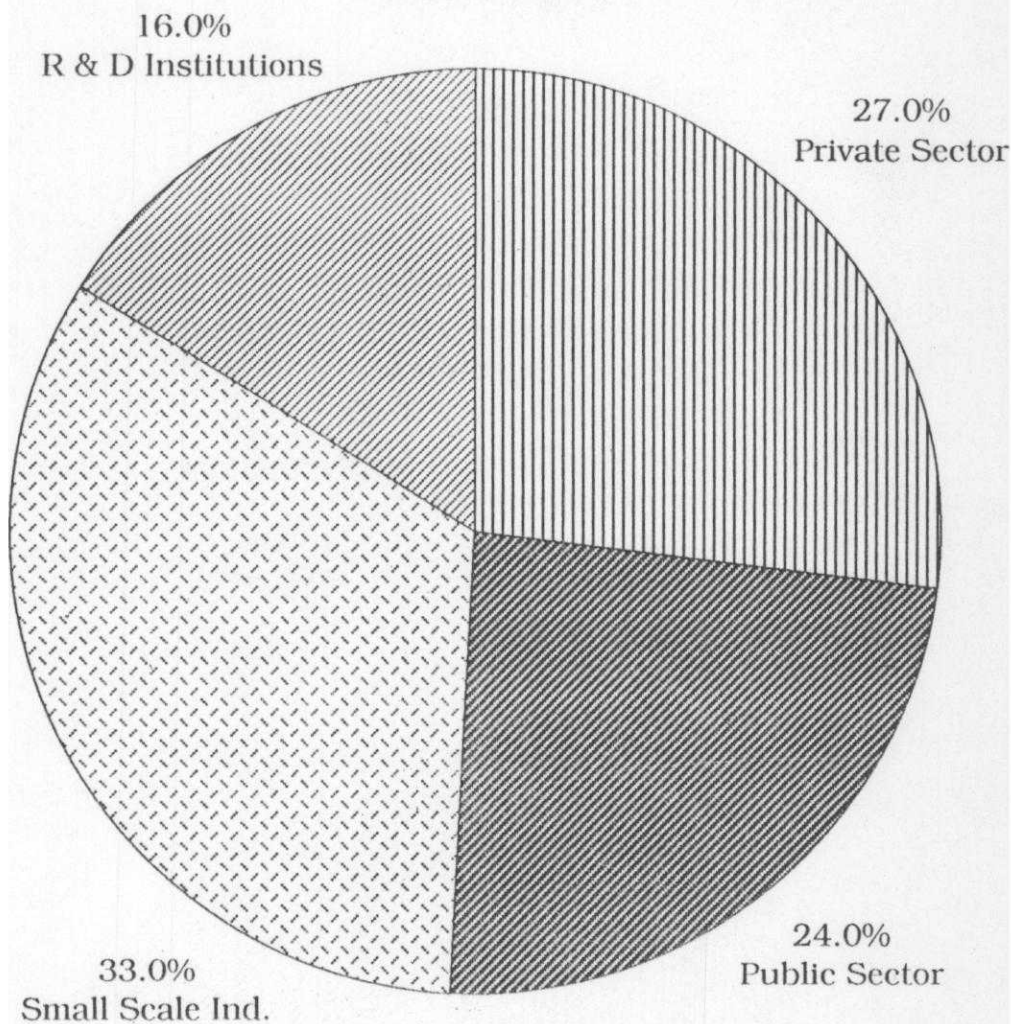


Fig. 1