Paper-II

Quality control and Traditional methods of Analysis - 1

Unit-1 Quality control in Analytical chemistry

1) Characteristics of an analysis :-

*Quality of an analytical procedure :-

The stages or steps in all overall analytical procedure can be summarised as follows.

Definition of the problem :-

Analytical information and level of accuracy required cost, timing, availability of laboratory instruments and facilities.

Choice of technique and method :-

selection of the best technique for the required analyses, such as chromatography, Infrared spectroscopy, titrimetry, Thermogravimetry.

Sampling :-

select a small sample of the material to be analysed. Where this is heterogeneous special procedures need to be used to ensure that a genuinely representative sample is obtained.

Sample pre-treatment (or) conditioning :-

Conversion of the sample into a form

Suitable for detecting or measuring the level of the analytes by the selected technique and method. This may involve dissolving it, converting the analyte into a specific chemical form (or) separating the analyte from other components of the sample that could interfere with detection or quantitative measurements.

Qualitative Analysis :-

Tests on the sample under specified and controlled conditions. Tests on reference materials for comparison. Interpretation of the tests.

Quantitative Analysis :-

Preparation of standards containing known amount of the analyte(s) or of pure reagents to be reacted with the analyte (s). Calibration of instruments to determine the response to the standard under controlled conditions.

Measurement of the instrumental response for each sample under the same conditions as for the standards. All measurements may be replicated to improve the reliability of the data, but this has cost and time interpretations.

Preparation of report or certificate of analyses :-

This should include a summary of the analytical procedure. The results & their statistical assessment and details of any problems encounted at any stage during the analyses.

Review of the original problem :-

The results need to be discussed with regard to their significance and their relevance in solving the original problem.

Some times repeat analyses (or) new analyses may be under taken.

Limit of detection (LOD) :-

For analytical procedure it is important to establish the smallest amount of analyte that can be detected and measured quantitatively. In statistical terms and for instrumental data. This is defined as the smallest amount of an analyte giving a detector response significantly different from a blank or back ground response.

It is the lowest concentration level that can be determined by statistically difference from analyte blank.

Replicate blanks of one sample matrix are analysed to determine the mean blank value & its standard deviation.

Then a matrix is spiked with analyte near the detection limit.

The LOD is the concentration calculated to give a response equal to the blank signal plus '3' standard deviations.

LOD = Blank signal + 3 SD.

The concentration that give the signal equal to '3' times the S.D of the back ground is generally taken as detection limit.

*Sensitivity :-

The ability to discriminate among similar concentrations of the analyte.

The ability to detect (qualitative) & determine (quantitative) small amount of analyte in a sample.

It can be expressed via, Various Parameter.

$$S = \frac{x}{c}$$

Where

x = Variation of analytical signal

C = Analyte con

$$S = \frac{\delta x}{\delta c} = \frac{\Delta x}{\Delta c}$$

It is ability to distinguish two different concentrations and is determined by slope of the calibration curve. So measure the slope of sample of closely related high concentrations intermediate low concentration.

The sensitivity & precision will

(3) govern how many figures should be reported in a measurement.

*Safety :-

Safety in academic chemistry laboratories published by the American chemical society.

To guide discusses personal protections are

(a) Laboratory protocal
(b) Recommended laboratory tech
(c) chemical Hazard's
(d) Instructions on reading.
(e) Understanding material safety data sheet.
(f) Safety equipment.
(g) Emergency procedure

*Cost measurability :-

Analytical costs are expressed as a sum of money per result or sample are and less often in many hour's.

The lower analytical costs are the higher will be cost effectiveties which can be considered as an analytical property

Lowering cost usually sacrificing on some other analytical property whether capital basic (or). accessary.

Overhead costs :-

It arises from laboratory set up and maintance the purchase and maintance of equipment and the salaries of staff (managers, secretaries)

As a rule they are considered fixed costs. Even though they can certainly vary to some extent.

* Selectivity :-

It denotes basic analytical methods or procedure.

1. It defined as ability to proce produce exclusively depends on the analyte for its identification or quantification in sample.
2. It provides direct support to accuracy
3. It is extent method can measure the analyte of interest in the materials of sample being analysed with out interference from the matrix.
4. In this method two different parameters.

The total rated (T_R) ratio of an interference is concentration ratio of interferent (C_{int}) and concentration ratio of analyte ($C_{analyte}$)

$$T_R = \frac{C_{interference}}{C_{analyte}}$$

The selectivity of method can be expressed as

$$T_R = \frac{S_{analyte}}{S_{interference}}$$

The selectivity factor may be expressed as

$$S_F = \frac{(T_R)_1}{(T_R)_2}$$

Specific costs :-

These are to be paid by the laboratorys clients, ie. by these who demand the information delivered. The difference bet what the client actually pays & specific costs can be used as a criterion to estab blish the quality of an analytical procedure

Specific costs can also be fixed (or) variable automation raises fixed costs (These of equipment) but decreases in variable costs (if reduce many hours).

Quality control :-

It is a process of ensuring that the operation tech and activities used in analytical laboratory provided results suitable for the intervien purpose.

It comprises a system of planned activities in an analytical laboratory where by analytical methods are monitored at every stage to verify complain with validated Procedure and to take steps to eliminate course of unsatisfactory performance.

Results are considered to be of sufficiently high quality if,

a) They meet specific requirements of required analytical work with in the context of a defined problem.

(b) There is a confidence in their validates.

(c) The work is cost effective.

* Ruggedness test:-

1, Ruggedness refers to the precision of one lab over multiple days, which may includes multiple analyte, multiple instruments different source of reagents, different chromatographic columns

2, A Ruggedness study will identify those factors that will contributes to variability of results, and which should not change.

3, This is related to Robustness or unintended small change in parameters. such as size of sample, temp, pH of solution, reagent concentrations, time of reaction etc.

* Control charts:-

The principle of a control chart is "A visual representation of quality using as a basis the desired quality and the limits".

1, For the latter are distinguishes between warning limits. which exceeded once still give tolerable results. and control (or action) limits.

More control samples are analysed in every analytical series.

2, A precision and Accuracy is performed on the analytical results. for every control sample and compared with established values.

3, For Acceptable analytical quality warning and control limits are established and control charts are constructed,

4, In the following control period, the advice to obtained. Limits is monitored and additional control samples are investigated.
5, The control chart can be visually evaluated after each additional energy.
6. The control chart these by allows the rapid recognisation of arising errors and establish control criteria indicated on a out of control situation for analytical process being monitored.

(Types control charts

(a) conventional chart or shew hart chart
(b) x - chart or Blank value chart.
(c) Recovery rate control chart.
(d) Range control chart (R - charts)
(e) x - R combination charts
(f) Difference charts
(g) Standard deviation chart
(h) Cusum charts

In thes, oldest & simplest type of control chart is the shewhart chart/

Youden Plot :

1, Youden method for the execution, evaluation of inter laboratory programmes is stated to laboratory monitering.
2. Since it is a statistical method for obtaining information about a precision & systematic errors.
3. Only two additional samples must be analysed once.
4. The inter laboratory test is elvated graphically by plotting to result as a Youden plot.

5, However application of Youden plots are not only limited laboratory monitoring by means of inter laboratory test.

Ranking test :-

The test used to determine several samples differs on the basis of a single characteristic a control need it be identified.

1, Analyses are produce sample. Simultaneous including [a standard or control] code no's are asked to rank all samples, according to the intensity of the specified characteristics

2, The analyses are asked to rank coded sample according to their preference.

3, The rank of sample numerical order. according to the preference characteristics of properties.

4. When more than 3 samples are required to be compound ranking method is suitable

5, The method can be used for the comparing one treatment with others.

6. for a sample to be considered atleast one of the rank either test sample or any other marked sample must be lower than the higher limit in the upper plane.

Hadonic Rating :-

It results. pleasure or unpleasure experience. The Hadonic rating measures a consumer acceptability of consumer product.

1, To results the numerical values are design

2, Hadonic rating evaluated by Rank test. In this each sample arranged in ↑ & ↓ numerical order ranking assign.

⑥ 3. The purpose of rating obtained knowledge on how well produce prefer for the uses.

Eg:

Specific sample (A) = Monthly sample size

$$A = a\sqrt{2u}$$

Where

u = Monthly Production

a = constant range depend on a nature of Product.

Units for measure rating :

Overall rating is the no. of demerits for the detectors is divided by no. of units of Products in the sample.

ie, $R = \frac{\text{No. of demerits for the detect. scored}}{\text{No. of units after products in the sample}}$