THE FINAL WORD

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CALIBRATION OF LABORATORY EQUIPMENT

Calibration is process in which equipment performance is compared against better accuracy standard; essentially which should be traceable to SI unit or to primary standard of country.

The formal definition of calibration by the International Bureau of Weights and Measures(BIPM) is the following:"Operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indication with associated measurement uncertainties (of the calibrated instrument or secondary standard) and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication."

For physical standards like Mass, Volume, length, temperature generally NPL (National physical laboratory) is consider as primary reference in India. NPL provide traceability to International system (SI unit).

The laboratory is maintaining at present six out of seven SI base units. These are meter, kilograms, second, kelvin, ampere candela. R&D work is under way for realizing and establishing the seventh SI base unit of mole (mol). The SI supplementary units are radian (rad) & steradian (sr).

At present seventh SI based unit mole is traceable to NIST (National institute of standard material, USA).

Results produce by laboratory may have problem due to equipment; which can be ascertain against assigned or known value by means of traceable reference standard. NABL (National accreditation board for testing and calibration laboratories) accredits various calibration based on their technical competence and ascertain unbroken chain of traceability to SI unit. Any calibration made by NABL accredited laboratory is traceable to SI unit.

Different types of calibrations are:

- + Mass: Weights, Weighing balance etc.
- + Thermal: Thermometers, Thermal sensors, Hot air ovens, Water baths, Muffle furnaces, and other temperature measuring equipment.
- + Volume: Volumetric cylinders, pipets, burettes, and othervolumetric equipment.
- + Electro technical : uv visible spectrophotometer, Flame photometer, Gas chromatography detectors, HPLC pumps, Atomic absorption spectrometers, pH meter, Electrical Conductivity meter etc.
- + Electrical: Ampere, volts etc.

Characteristics of Calibration

- + Accuracy: Is the closeness to true value Or ratio of error to the full scale output.
- + Tolerance: deviation from the value specified by standard; which is accepted. For example if test method of moisture mentions that temperature of oven should be 100 + 5 oC means the maximum error permissible in temperature is 5 oC from 100 oC and hence calibration of

oven should be done with reference thermometer having uncertainty of measurement less than $\frac{1}{4}$ of 5 oC i.e1.25 oC. Here tolerance is 5 oC at 100 oC.

- + Traceability: Traceability is defined by ANSI/NCSL Z540-1-1994 (which replaced MIL-STD-45662A) as "the property of a result of a measurement whereby it can be related to appropriate standards, generally national or international standards, through an unbroken chain of comparisons."
- + Uncertainty of measurement: It is the process of identification and evaluation of the factors that affects the results of calibration significantly.

Standards' accuracy requirements:

- Standard used for calibration must have 10:1 accuracy or must have at least 1/4th Measurement uncertainty than Device under calibration.
- ➔ It must be traceable to SI unit.
- It should have appropriate life or stability to produce same values.

Some typical example of Chemical laboratory equipment calibration:

UVVisible spectrophotometer:

- + Control of absorbance: it is performed using 0.006 % K2Cr2O7 Solution. Absorbance of this solution at 235, 257,313 and 350 nm wavelength is measured and from which value of A is calculated for which USP, IP and other pharmacopoeia has given limits. Healthy instrument should pass this test.
- + Wavelength accuracy: It is measured using holmium oxide glass filter or Holmium perchlorate solution, Instrument should produce specific peaks when scanned in UV and visible region using one of these with + 1 nm accuracy to comply the test.
- + Limit of stray light: It is done with traceable solution of 1.2 % KCl, and absorbance at 200 nm should be grater than 2.0 to comply the test.

- + Resolution power: It is performed using traceable hexane.
- + Carrier Gas flow: It is measured using calibrated bubble manometer
- + Detector response: It is deferent for different detector e.g for FID it is done using mixture of traceable hydrocarbon.
- + Oven temperature accuracy

pH meter ver Industrial Chem

+ pH 4, pH 7 and pH 9.2 comparison against NIST or BAM traceable buffer.

Electrical conductivity meter:

+ Comparison against Traceable conductivity reference standard of different conductivity KCl solutions

Atomic absorption spectrometer:

- + Absorbance of 5 ppm traceable copper solution should be more than 0.650
- + Using traceable reference standard of Individual metals to prepare calibration curve.

Any calibration report must contains following as they are very essential components in calibration reports.

- Statement of Uncertainty of Measurement with
 + MU value.
- Status of traceability of reference standard against which calibration is made.

Bibliography

- JCGM 200:2008 International vocabulary of metrology
- NPL web site.

Details of Summer Inplant Training of M.Sc Industrial Chemistry Second Semester Students (May 2018)

NO.	Name of Student	Name of Company
1	ABHISHEK JAYESHKUMAR RAVAL	Kusha Chemicals Pvt. Ltd., Godhra
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5	ASHUTOSH PANKAJBHAI PATEL	Centurion Laboratories Pvt. Ltd., Savli
6	BHAUMIK SURESH PATEL	Pharmanza Herbal Pvt., Ltd. Kaniya
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24	HIREN SOLANKI	Farmson Pharmaceutical Ltd., Nandesari
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39	KISHAN SUNILKUMAR RANA	Zydus Pharma Ltd., Ankleshwar
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43	MIHIRKUMAR SHAILESHKUMAR JOSHI	Mayur Chemicals Pvt. Ltd., Halol
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