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# AOIL NEWS LETTER MARCH 2018

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#### News Letter of AOIL March, 2018

#### INTRODUCTION

AOIL is a registered, non-profit making body and aims to serve as a common platform for laboratories. The Association of Indian Laboratories (AOIL) has been formed and registered, (Registration No: HR 018201401605).

These laboratories are being managed by all sectors of economy such as Public, Private and the Central and State Government Authorities.

These Laboratories are contributing in following ways by providing their services.

- Industry and Infra-structure sector
- Safeguarding interest of consumer
- In Protection and Monitoring of the health of Public through Medical Laboratories
- In generating relevant information on environmental safety, health & toxicity
- To assist the authority, by supporting criminal justice system related activities, as the forensic science laboratories, customs and excise laboratories etc.

#### **OBJECTIVES**

Promote Co-operative efforts to identify common problems faced by the
member laboratories and arrive at the possible solution.

Maintain liaison/ relationship with the technical and scientific associations, accreditation body of the country and laboratories

associations having common interest worldwide.

To facilitate Inter Laboratory Comparison / Proficiency Testing and
training activities amongst the member laboratories.

To maintain the highest standard of professional and ethical conduct in laboratories in general and in particular amongst its member.



President desk

#### Mr. N Kalyan

I am extremely honored to serve as President of "Association of Indian Laboratories" which is dedicated to serve as the voice of Indian Laboratories. I look forward to achieving new vistas for the association with your support & efforts.

Welcome you to the newsletter of the Association of Indian Laboratories (AOIL). We are glad to Inform AOIL has been accepted as a stakeholder member of ILAC (International Laboratory Accreditation Cooperation).

AOIL promotes cooperative efforts to identify common problems faced by the member laboratories and arrive at the possible solutions and maintains close relationship with NABL (National accreditation board for testing and calibration laboratories) in India for common interests and shall provide consolidated feedback to accreditation & regulatory bodies in India.

The Association of Indian laboratories (AOIL) will look after the interests of the large number of laboratories in India. This includes Testing, Medical, GLP & Calibration Laboratories.

Its Headquarter is at Gurgaon, while the regional offices are based at Mumbai and Bengaluru.

The AOIL publishes a quarterly newsletter besides, organizing International and National Seminars, workshops and training program etc. for the benefits of its members.

The objectives of AOIL is to become a truly representative association for accredited and non-accredited member laboratories.



Chairman desk

### Shri Devi Saran Tewari



#### Dear Friends

It is matter of satisfaction that after a gap AOIL Newsletter has resumed its publishing. Hope the break would not happen again. Being Editor is challenging and a tough job. Editor needs cooperation from all in AOIL and beyond. I request all AOIL members to contribute their best in making AOIL a magazine of repute and contribute articles that are of interest to laboratories.

AOIL being a platform of laboratories provides an opportunity to share the views and aims to resolve the problems being faced by laboratories. Each one of us can grade AOIL's functioning and the extent to which mutual co-operation has been effective!

#### But has AOIL done enough on its part?

1. Question has been hounding most of us. The analysis is as follows;

- I. Joining hands to-gather and forming the association (AOIL) has made NABL, induct AOIL in its Board, which its highest decision body.
- II. NABL Board considered the dissenting plea of AOIL, and did not increased NABL fee by 10%, that was as agenda item for 2016-17.
- III. NABL agreed for monthly meeting with AOIL, to resolve the issues that concern laboratories. The outcome of such meetings is not fruitful, as the methodology of NABL's working is committee based and NABL officers, who participate in monthly meetings are not impowered to agree or disagree. And this forbids them to commit for resolving the issues that concern laboratories.
- IV. AOIL has organized three workshops on CD 17025 from three different cities of the country, in April 2017, involving NABL. and again, in November 2017, three seminars from three cities by involving NABL. Director NABL was present in all the six events and NABL also provided financial support, both times. It may be noted that Mr. Peter Unger, Chair ILACand Jeff Gust were the speakers.
- V. AOIL could resist increase in Overhead charges, which was proposed in agenda for financial year 2018-19.



#### Shri Devi Saran Tewari

- VI. AOIL has not been able to communicate to the ministries of the government of India as well state governments about the formation of the Association of laboratories, as AOIL.
- VII. One can feel that formation of AOIL has bridged the gap between laboratories and NABL. Hopefully the attitude of NABL towards laboratories would change, by reciprocating to resolve their genuine problems. Laboratories do communicate their problems, but not in writing that forbids

AOIL to take up the matter with the concerned authorities, as in the absence of documentary evidence, it cannot go or contact the concerned authority.

AOIL needs documentary support to keep itself on solid ground.

AOIL had developed a questionnaire for laboratories. And if this information is volunteered, then this compiled data base, would help to take up the concerns of aboratories. I appeal to the management of laboratories to furnish the details of the problems they face, even by hiding their identity.

It would the ground to AOIL, for projecting the problems faces by laboratories. AOIL has been organizing training courses from time to time and different cities and regions, and it is the time to start training on new version of international standard ISO/IEC 17025 (2017).

It is planned to conduct two days training for those who have attended 4-days training on ISO/IEC 17025 (2005), and there after 4-days training on ISO/IEC 17025 (2017) for freshers.

As far as NABL is concern it would continue accepting applications based on ISI/IEC 17025 (2005) till August 2018, and thereafter based on ISO/IEC 17025(2017). There would be tremendous pressure on laboratories and on NABL to cope up with the transition period given by ILAC. And in this situation AOIL would have to play its role.

## UPCOMING EVENTS PLANNED FOR 2018 (March - July 2018)

- National Vendor Development Programme Cum Industrial Exhibition 2018 Jointly Organized by AOIL and MSME for Testing, Calibration, Instrumentation and Allied Products :- 7th & 8th March 2018 at HSIIDC Complex, Manesar Gurugram.
- 2nd Congress of Association of Indian Laboratories (AOIL) :-19th April 2018 at The Ashok, New Delhi (Inauguration by Dr Harsh Vardhan, Hon'ble Minister at Ministry of Science & Technology / Ministry of Environment, Forest & Climate Change / Ministrry of Earth Sciences, Govt. of India.

#### Brief of Activities done in AOIL done so far;

- 1. AOIL has joined ILAC as its stakeholder member, and starting from 2015 till March-April 2015, it has participated in two annual meetings of ILAC and in two midterm meetings.
- 2. In the month of Nov. 2016 three seminars were organized from Delhi, Chennai and Mumbai, by inviting three international experts on imminent topics that are of\ interest to laboratories. The speakers were Peter Unger the then ILAC CHAIR, Jeff Gust member CASCO WG 44, responsible to draft ISO/IEC 17025 and Mr Raj Nathan Senior Vice President International Accreditation Services, (IAS) USA. In these seminars more than 800 laboratories participated from all regions.
- 3. A PT program was organized for calibration laboratories, in which more than 30 laboratories participated.
- 4. AOIL is frequently organising various Interaction Meets & Training Program to Improve interaction & Co-operation within Member Labs

## **Brief on AOIL Events in 2017**

21st April, 2017	AOIL Governing Council Members Election.
21st May, 2017	16th Meeting of the Governing Council of AOIL in Gurgaon.
25th June, 2017	17th Meeting of the Governing Council of AOIL in New Delhi.
25th June, 2017	First Interaction Meet at New Delhi
30th July, 2017	18th Meeting of the Governing Council of AOIL in Chennai.
27th August, 2017	Second Interaction Meet at Lucknow.
03rd October, 2017	Third Interaction Meet at Lucknow.
26th November, 2017	Fourth Interaction Meet at Udaipur.

# **AOIL Governing Council Members**

Sr. No.	Posts	Name of Members	Laboratory Name
1	Chairman	Mr. D.S. Tewari	DVG LABORATORIES & CONSULTANTS PVT LTD
2	President	MR. N. KALYAN	ELCA LABORATORY
3	General Secretary	MR. RAJESH DESWAL	EMMTECH CALIBRATION
4	Joint Secretary	MR. LALIT PANERI	RAHUL ENGINEERING LABORATORY
5	Vice President (Calibration)	MR. PRAFUL G. YELVE	GODREJ & BOYCE MFG. CO. LTD
6	Vice President (Testing)	DR. R.B. SINGH	ANULAB INDUSTRIAL TESTING & ANALYTICAL LABORATORIES
7	Vice President (Medical)	MR. NEERAJ JAIN	JAIN DIAGNOSTICS
8	Vice President (GLP)	MR. ARUN R. FREDRICK	INTERNATIONAL INSTITUTE OF BIOTECHNOLOGY AND TOXICOLOGY
9	Treasurer	MR. RAHUL GUPTA	SIGMA TEST & RESEARCH CENTER
10	Executive Member - GC	MR. ROHIT KUMAR PATRA	NENO TECHNICAL SERVICES
11	Executive Member - GC	MR. GAURAV TEWARI	DVG LABORATORIES & CONSULTANTS PVT LTD
12	Executive Member - GC	MR. A.K. NEHRA	BELZ CALIBRATION LAB
13	Executive Member - GC	MR. ARUN KUMAR SINGH	ARUN SOIL LAB PVT. LTD.
14	Executive Member - GC	MR. D. MATHUR	FARE LABS
15	Regional Chairman -North	MR. PRAVEEN BHARGAVA	PERFACT RESEARCHERS P.LTD.
16	Regional Chairman -South	Mr. A SASHI KUMAR	PRECISE TESTING AND CALIBRATION CENTRE
17	Regional Chairman -West	DR. NILESH S. AMRITKAR	ENVIROCARE LABS PVT. LTD.

#### **# FIRST AOIL INTERACTION MEET IN DELHI**

• First AOIL Interaction meet was organized on 25<sup>th</sup> June 2017 by Mr. Praveen Bhargava, Regional Chairman-North, AOIL at Holiday Inn, New Delhi. All the members of the association were assembled to share their experience & interact on various points associated with the laboratories and what to be done for the benefit of the laboratories.



#### SECOND & THIRD INTERACTION MEET IN LUCKNOW

Second AOIL Interaction meet was organized on 27<sup>th</sup> August 2017 in Lucknow discussed about the monthly review meetings. AOIL Head offices were assigned the responsibility of reviewing the same.



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**\* FOURTH INTERACTION MEET IN UDAIPUR** 





#### Article 1 :-The Role of Laboratories in testing, Inspection and certification

R.B. Singh Vice President (Testing), AOIL, India Chief Coordinator, ANULAB, Agra Email: research@anulab.org

#### Introduction

Measurement and testing are of fundamental importance to science, technology and the economy. "There is no science without measurements, no quality without testing and no global market without standards."

Measurement1, testing2 and inspection3 play an important role in healthcare, food safety, undisputed drinking after quality, consumer protection, improving safety at work, monitoring and minimizing environmental hazards. It is easily seen that almost all aspects in consumers' daily life is affected, but the economy in large is also dependent on the providers of measurement, testing and inspection services.

Every sector has its different measurement, testing and inspection needs, chemical analysis differs from electromagnetic compatibility testing, just as regular vehicle inspection differs from inspection of financial services. The measurement, testing and inspection sector is therefore a highly diversified and specialized sector that depends on trust in the results that the service providers deliver to consumers, customers, authorities or the economy at large.

#### The testing, inspection and certification (TIC) sector

Testing is a specialised service, where the ability of the laboratory performing the tests to achieve accurate test results is the crucial factor. This ability is mainly determined by the competence and experience of the personnel performing the tests, the quality and suitability of the test equipment, including accurate calibration, and the quality assurance system of the laboratory. With respect to the methodology of testing, the following aspects are crucial in order to achieve the right level of quality:

- The laboratory must be able to choose and apply the test method that provide a technically correct and reproducible answer to the problem.
- The testing must be performed efficiently, and in such a way that delivery times can be met and costs and charges are economically optimized viable & affordable.
- The laboratory must be able to provide reliable results with accuracy appropriate to the needs.
- The testing laboratory must have the necessary expertise available to be able to assist in the evaluation of the test results and to provide other relevant technical services of an advisory nature. The expertise may be in another organisational unit.
- The reports and presentations of the results must be clear and complete, and the recipient must be able to understand and apply them correctly.
   Activities related to testing, inspection and certification4 often are performed by third

parties, i.e. entities that are fully independent of the manufacturer of a tested product or system, the service provider of an inspected service, the organisation that is audited etc. and where the entity has no user interest on the object or service.

- 1 Measurement : process of experimentally obtaining one or more values that can reasonably be attributed to a quantity ISO/IEC GUIDE 99 (2.1)
- 2 Testing : determination of one or more characteristics of an object of conformity assessment, according to a procedure ISO/IEC 17000 (4.2)

3 Inspection : examination of a product design, product , process or installation and determination of its conformity with specific requirements or, on the basis of professional judgment, with general requirements ISO/IEC 17000 (4.3)

Some of these activities may also be performed by first parties themselves (e.g. manufacturer's laboratories or internal audit departments) and second parties (e.g. manufacturer's laboratories for testing bought-in components, retailers testing laboratories etc.). If these activities are performed by accredited laboratories, their impartiality has been proven by their accreditation5 and is monitored during accreditation audits. The results of accredited first and second party TIC entities are equivalent to those of third parties. The role of first, second and third party laboratories is further described in the EUROLAB Position Paper "First, second and third party testing – how and when".

Testing and inspection services support the quality and safety of products through product performance evaluations. For placing a product falling in the scope of a regulation on the market in nearly all countries a proof of conformity with the regulation is required. This proof may be drawn up by the manufacturer himself, based on verification of conformity in the form of test reports or by certification. Certification is a special form of these services, where an independent third party attests that a product confirms with given specifications or a service or a management system is performed or operated according to the specification laid down. Certification is based on test reports, inspection reports and audit reports. The certification body may run its own laboratories, employ its own inspectors or auditors or may rely on reports from external institutions of proven quality.

Manufacturers and second parties have the choice of using the services offered by third party companies, individual or bundled services, or to establish their own service departments or testing laboratories. For companies it is for every requested service a case by case decision whether to establish their own expertise or to buy services provided on the market. Results of these services needed for other than internal purposes normally require that the service provider gives proof of his competence and the quality of the service provided. Normally this is done by accreditation through the national accreditation body (NAB) which is equally applicable to first, second and third party service providers.

#### Market segments and trends

The TIC sector is characterized by a variety of segments, industries and technical niches which have different characteristics, drivers and growth stimuli.

The Industrial sector is mainly dominated by oil & gas, energy and transportation. The building and construction sector is a cyclical market with growing limitations in the western economies. Nevertheless, this sector is constantly developing thanks to the ongoing regulations focused on green and sustainable buildings. The Consumer sector includes testing, product inspection, process assessment and technical assistance for all types of products (e.g. electronics, toys and textiles). This sector is dominated by tighter

regulations and the need to assure that all important criteria are properly and thoroughly evaluated. Thus there is an increased demand coming from the manufacturers' side for product testing services in order to reinforce the reputation

and consumers' trust in their products. Both mid-market and larger players have been targeting consumer focused TIC companies.

According to Bureau Veritas the estimated size of the TIC in 2015 was €200 billion, based on external macroeconomic data such as investment volume per market, operational spending

- 4 Certification: third-party attestation related to products, processes, systems or persons ISO/IEC 17000 (5.5)
- 5 Accreditation: third-party attestation related to a conformity assessment body conveying formal demonstration of its competence to carry out specific conformity assessment tasks ISO/IEC 17000 (5.6)
- 6 EUROLAB Position Paper 'FIRST-, SECOND- AND THIRD-PARTY TESTING HOW AND WHEN?'; No. 1/2000, May 2000, EL/01- 01/00/380
- 7 Mergers Alliance, Global testing, Inspection and Certification, M&A updates, Summer 2012

per market, the production value of goods and services, and the level of imports and exports. In the image below TIC was divided by sector, showing that the biggest markets are those relating to consumption, followed by oil & gas, construction, chemicals and mining.8

Challenges for the laboratories in a changing world

There are several major trends which have a considerable impact on overall technical and technological development and consequently on development in the field of measurement and testing:

- The globalisation of world trade leads to a harmonisation of requirements and standards as well as to the development of global markets for products and services. This globalisation process also has repercussions on industrial and private testing laboratories.
- Small laboratories are being taken over by larger ones who are able to offer economies of scale. Larger organisations often merge and the past ten years has seen the rapid growth of global companies, which set up and operate laboratories in many countries.
- International agreements, such as GATT, WTO and mutual recognition agreements, dominated by the preoccupation of facilitating international trade, are aimed at avoiding unnecessary duplication of testing.
- Local and regional networking of industry and service providers is taking place, the aim being

to conquer international markets. As a result, the challenge for the laboratories is to get recognition/acceptance of their reports outside the national borders and thus increased visibility and impact.

• The quest of customers and society for improving the quality and safety of products is resulting in a greater awareness of the importance of market surveillance. In measurement and testing, there is a need to get comparable results in the determination of the characteristics irrespective of the organisation performing the market surveillance.

#### Testing, inspection and certification support standardization

Measurement, testing, and inspection all depend on and rely on standardisation. For comparability and therefore mutual acceptance of test results, a test must be performed against the same requirements using the same test method, specified in international standards. On the other hand, the competence of laboratories must be of the same level to achieve test results of equal quality and at comparable prices. These requirements are laid down in international standards.

As a service provider, conformity assessment bodies themselves must meet differentiated specification standards that describe the state of the art with regard to the organisation and the competence of conformity assessment bodies. The ISO/IEC 17000 series of standards brings uniformity to conformity assessment and therefore makes a key contribution to the comparability and recognition of conformity assessment results.

8 Bureau Veritas Performance Perspective, 2015 Registration Document incorporating the Annual Financial Report, Chapter 1.4. The TIC industry, pages 12-13

# Accreditation improves the robustness and the comparability of testing, inspection and certification activities

A harmonised and credible accreditation system is of great importance for the laboratory sector Accreditation is the method of ensuring competence in delivering conformity assessment activities. Such a system must be based on harmonised requirements and their application, be transparent, impartial efficient and authoritative. The accrediting process is based on a reliable and systematic approach to determine the competence of a laboratory, inspection or certification body.

Laboratory accreditation attests the technical competency of a laboratory to perform specific tests, types of tests or calibrations. When it comes to testing, accreditation attests that test results are obtained according to valid methods and procedures that comply with precise standards. When it comes to calibration, accreditation guarantees the laboratory capacity to carry out calibrations and metrological verifications in a certain domain and with specified uncertainties.

Using accredited laboratories also facilitates trade and economic growth. The accrediting process is based on a reliable and systematic approach to determine the competence of a laboratory, an approach that has been accepted and implemented across borders. But at the same time, accreditation needs to enable a fair competition among accredited laboratories on a global market. This will be compromised if the application and interpretation of the necessary requirements and competences of assessors varies between national accreditation bodies.

#### Conclusions

Measurement and testing underpins the welfare of a modern society and touches almost every part of daily life from ensuring the safety and effectiveness of healthcare diagnostics and treatments, ensuring consistency of international standards and air quality monitoring, security and sustainability of our food supply to products safety. Measurement also plays a fundamental part in the innovation process. To develop new and improved products and processes, companies are looking for: improvements in quality or performance, reductions in waste, use of new materials or techniques.

The goal of AOIL and its members is to be recognised as the voice of safety, ompliance and quality in India and world-wide. But in order to achieve this goal continuous laboratory selfassessment is required. The laboratory community must be valued for its contribution to the improvement of quality and reliability of testing, and its role for making sure that the liberalisation of trade is not detrimental to health, safety and environmental protection.

AOIL considers it fundamental to convince Indian Govt. officials and the business community that the measurement and testing industry adds value, protects consumer interests, guarantees the compliance of products placed on the market with relevant legislation and standards. AOIL endevours to focus on finding solutions to the wide range of challenges and will continue to be the effective voice of the Indian laboratory community.

9 Measurements in daily life, The National Measurement System, http://www.npl.co.uk/upload/pdf/ measurementmatters.pdf

#### Article 2 : An Alternate method for the calibration of Density Hydrometers -Cuckow's Method

Mr. A. Murugappu & Mr. L. Muthukuwarau, State President, AOIL North Lab India Pvt. Ltd., Chennai E-mail : sales@northlab.in

#### Introduction

Calibration is a set of operation under specified conditions where the relationship between values of realized by standards and values of quantities indicated by a measuring instrument are compared. It is crucial for determining and validating the accuracy of measuring instruments. Almost all measuring instruments such as hydrometer, viscometer, Liquid in glass thermometer, pressure meter and standard weight need calibration to validate their accuracy.

Hydrometer has gained its popularity as versatile instrument for determining liquid density and therefore, the degree of its accuracy will depend much on its calibration procedures.

There are two well-known hydrometer calibration methods: hydrostatic weighing method and comparison method.

Secondary calibration laboratories all over the world mostly use comparison method since it is much simpler, easier and cheaper compared to the hydrostatic weighing method.

#### **Cuckow's Method - Measurement Principle**

This method is based on the well known Archimedes' principle, which measures the value of buoyancy that the reference liquid has on the suspended hydrometer at the calibration mark. This buoyancy push is measured by using a weighing instrument, through the relationship between the value of the hydrometer mass in air and the (apparent) mass of the hydrometer partially immersed.



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Setup for hydrometer calibration Courtesy : NPL India

#### Measurement method for the calibration of hydrometers

The hydrometer calibration by Cuckow's method requires the measurement of the hydrometer mass in air, and the measurement of the hydrometer mass partially immersed in the reference liquid up to the mark to calibrate.

The level of the liquid should be aligned with the center of the mark.



There are different techniques for aligning the mark of the hydrometer to the level of reference liquid as well as different devices to aid the metrologist in this alignment, e.g. camera, zoom lens, etc. The method and device to be used by the metrologist depends on the level of required uncertainty in calibration.

#### Measurement Model

The measurement model for the calibration of the hydrometers by Cuckow's method is\ as follows:

The indication error E(I), of the hydrometer is calculated by:

 $E(I) = I(\tilde{n}) - \tilde{n}_x - a_d$ 

with

I( $\tilde{n}$ )-Indication of the hydrometer at the calibrated mark in kgm<sup>-3</sup>  $\tilde{n}_x$  Actual Value of liquid density at the calibrated mark in kgm<sup>-3</sup>  $a_d$  - Error due to the finite resolution of the hydrometer, it has zero mean but contributes to the uncertainty ~ 0 kgm<sup>-3</sup>

#### General Procedure of Calibration

The calibration procedure of hydrometers has the following stages:

- a. Identification of the characteristics of the instrument, (range, resolution, Series, maximum permissible error and required uncertainty).
- b. Cleaning and acclimatization of instrument to calibrate
- c. Preparation and stabilization of the measurement system (e.g. reference liquid, weighing instrument, thermostatic bath, thermometer for measuring the temperature of the liquid, instruments for the measurement of environmental conditions, standard weights, among others).
- d. Measurement of the hydrometer mass in the air
- e. Measurement of the hydrometer mass immersed at the mark to calibrate (calibrated in at least three different nominal values).

The setup consists of a balance of 200 g capacity with resolution of 10 g, a precision ambient pressure measuring instrument, a precision PRT, a thermometer with relative humidity sensor and a motorized system for maintaining the temperature of liquid (distilled water) and a liquid bath for pre set temperature. The PRT is used for continuous monitoring of the liquid temperature (depending upon the nominal range of the hydrometer) in the glass jar.

#### For detailed calibration procedure see reference [3]

#### Liquid Density at the Mark Level

The value of the liquid density at the calibrated mark  $\tilde{n}_{v}$ , is calculated as follows,

$$\rho_x = \left(\rho_L f_{t,L} - \rho_a f_{t,a}\right) \cdot \left[\frac{m_a + \frac{\pi D \gamma_x}{g}}{m_a - m_L + \frac{\pi D \gamma_L}{g}}\right] + \rho_a f_{t,a}$$

Where:

- $\tilde{n}_{_L}$  Density of the reference liquid at the measurement conditions; kg m  $^3$
- $\tilde{n}_{a}$  Density of the air during the measurement; kg m  $^{\cdot3}$
- $f_{\!\scriptscriptstyle tL}$  Correction factor of volume of the hydrometer material due to a change

in temperature Ät; the second sub index indicates whether this change relates to the air temperature or to the liquid temperature. (Please note that the correction factor is dimensionless.)

- m<sub>a</sub> Apparent mass of the hydrometer in the air in kg
- $m_{\!\scriptscriptstyle L}$  Apparent mass of the hydrometer immersed until the calibration mark in kg
- D Diameter of the stem to the level of the mark to calibrate in m
- g Acceleration due to local gravity in ms<sup>-2</sup>
- $\tilde{a}_x$  Surface tension of the reference liquid in Nm<sup>-1</sup>
- $\tilde{a}_{\scriptscriptstyle L}$  Surface tension of the liquid where the hydrometer will be used in Nm  $^{\cdot 1}$

#### Major factors contributing to measurement uncertainty in Cuckow's Method

- a) uncertainty due to weighing of hydrometer in air
- b) determination of air density
- c) uncertainty due to weighing of hydrometer in Liquid
- d) determination of Liquid density
- e) uncertainty due to temperature of buoyant liquid

Cuckow's method has many advantages. It eliminates the inconvenience of storing a collection of liquids, such as aqueous solutions of acids and mixtures of volatile hydrocarbons. It permits the calibration of a hydrometer at specified scale marks without the inconvenience of preparing liquid mixtures to specified densities. It doesn't need a set of fragile, breakable, master hydrometers.

It is environment friendly as we can deal with water as our buoyant liquid and its CMC is much better than the Comparison method CMC achieved by Cuckow's method is typically 0.47 mg/ml when compared to 0.8 mg/ml using the comparison method.

This method has been accepted by NABL. We are proud to state that one of our senior AOIL member from Chennai, namely Macro Calibration Services has already been accredited by NABL for calibration of hudrometers by this method.

#### **References and Acknowledgments**

- 1. <u>SIM Guidelines</u> on the calibration of hydrometers Cuckow method First Edition, May 2016 ISBN: 978-607-97187-5-6
- 2. Claude JACQUES, MWG7 Workshop on Hydrometers Queretaro (Mexico), Nov 2006.
- 3. **Anil Kumar** et al, Establishment of traceability of reference grade Hydrometers at NPL India International Journal of Modern Physics: Conference Series Vol. 24 (2013)

#### Article 3 :-

Statistical evaluati

Sampling

Sample preparation

#### Introduction to Solid Phase Microextraction for Trace Organic Analysis

#### Introduction

Solid Phase Micro Extraction (SPME) is a solventless, quick, reliable sample preparation technique used widely in the field of chromatography (Gas Chromatography, High Pressure Liquid Chromatography, Liquid Chromatography Mass Spectrometry). The technique [SPME] was invented by Prof Janus Pawlizyn, University of Waterloo, Waterloo, Ontario, Canada in the 1990s. He is one of the most important researchers in this field with more than 1000 papers on application on SPME using GC for environmental samples.He is currently the Canada Research Chair, Professor of Chemistry, Editor of AnalyticaChimicaActa and has won many awards for this invention and his application of the technique.SPME has been named as the invention of the year in field of analytical chemistry in more than 10 countries including UK, France and Canada.

The key places apart from Pawlizyn's Group in the world, where this technique is being used extensively is China, USA, UK, Korea, Italy and Spain. The Technique has been applied to environmental research, Food and flavour analysis, Medicinal analysis. It is used mainly to save time, money and reach lowest possible limits of detection. This means it is most useful in Air, Water, Food and other matrices where there is a possibility of human health impact.

#### **Requirement of Sample Preparation:-**

- Essential in isolating Analytes of interest from Sample matrix
- Clean up for dirty samples
- Enrichment/ Pre-concentration
- Eliminate Possible Interferences
- Selection of Target Groups
- Speed
- Time
- Conditioning of the Apparatus for next sample

SPME is able to tackle all the challenges without compromise on Cost, Time, Limit of Detection, Sample integrity and most importantly it has easy interface on GC/HPLC. It is selective, fast and is the best pre-concentration technique available in market.

		Extr	action technic	ques			
Flow-through equilibrium and pre-equilibrium			Batch eq and pre-e	uilibriur quilibriu	m Im	Stead	ly-state exhaustive
Exhaustive	Non-exhaustive	Exh	austive	Non	-exhaustive		Membrane
Purge and trap	In-tube SPME		LLE	ъЮ	Headspac	e	
Sorbent trap	]	H	Soxhlet	ЭЮ	LLME		
SPE	]	H	Sorbents	SPME			
SFE	]		MAE				
PFE	]						



## Types of Fiber(s):-

Core of a fiber is the solid support over which coating is done. It gives the fiber its shape, strength, length and majority of the weight. Three of the most important cores are :-

- Fused Silica: Most common, similar to highly inert glass, stable; but it can break!! Coating isn't very well bound to the core due to limitation of coating techniques that may be applied for this core. Almost all coatings are available in this core.

-Stableflex: This is a special core made for the purpose of providing a more elastic and inter core which can be used for high temperature desorption upto 320 °C. Only adsorbent coatings can be applied to this core. Modern process of coating such as electro-dipping and coating towers can be used with this core. This is extremely elastic and is made of 80µm Fused silica with 20µm thermally stable polymer.

-Metal Fiber Core: This core is developed to prolong life of fiber and provide an unbreakable, durable and thermally stable material. This type is used mainly in commercial autosamplers. It is stable upto 450 °C and lasts more than 300 cycles for some coatings. This highly inert core is the best available in market but is priced about 5 times than the fused silica core fibers. All fiber coatings are available for this core.

Type of Coating	Extraction Mechanism	Polarity
7 μm PDMS	Absorbent	Non-polar
30 µm PDMS	Absorbent	Non-polar
100 µm PDMS	Absorbent	Non-polar
85 µm PA	Absorbent	Polar
60 µm PEG (Carbowax)	Absorbent	Polar
15 µm Carbopack Z-PDMS	Adsorbent	Bipolar
65 µm PDMS–DVB,	Adsorbent	Bipolar
55 µm/30 µm DVB/Carboxen-PDMS	Adsorbent	Bipolar
85 μm Carboxen-PDMS	Adsorbent	Bipolar

### **Commercial Coatings Available**

As above, the types of Phase describes polarity which influences the selectivity of the fiber to extract analytes of interest. Essentially all SPME fibres are bi-polar to an extent but they are usually more selective to one polarity.

## **Selection of Fibre Coating**

Selection of fibres is one the most important decisions to be made for the accuracy and sensitivity in the analysis. This is normally done based on thumb rules with respect to thickness of coating, analyte properties such as molecular weight, polarity and based on sample interferences. Coating Thickness plays an important role in mass transfer rates for high molecular weight compounds, it means that high molecular



weight compounds would have better mass transfer efficiency in the thin coatings.

Increasing the temperature usually decreases the distribution coefficient but in some cases usually HS mode it may actually increase. This is not due to the increase in distribution coefficient but due to increased amount of analyte into headspace causing more concentration in the extraction phase.

Addition of stabilisers such as NaCl/ Na<sub>2</sub>SO<sub>4</sub> may increase or decrease the amount of extraction depending on compound, coating on fiber, matrix. It has to date only been observed by experiment and cannot be correlated by equations.

pH usually makes no difference in the amount of extraction except that the fiber efficiencies are higher at neutral pH and fiber life is also best at pH of about 6-8 mainly due to stability of core and of the coating material of the fiber. It does however make a difference when the analyte of interest has a pH sensitive formation/ dissociation equilibrium in the solution

Polarity of Sample Matrix and Coating Material: Compounds have an affinity for a phase of similar polarity. Selection also depends on whether it is being operated in HS or DI mode. The Distribution constant strongly depend on molecular weight of compound due to pore size and difference in coating thickness of all fibres.

#### **Difference between SPME and SPME**

SPME is often mistaken for SPE(solid Phase Extraction) or micro-SPE. However, there are significant differences; In SPE Method of Sample Preparation it is passed through the sorbent bed and the analyte are transferred exhaustively from sample matrix to solid disks. The next step is to desorb the unwanted analytes from the disk using an appropriate solvent. Finally the analytes are washed using a solvent. The solvent is the pre-concentrated by evaporation to desired volume. The prepared sample can then be transferred to instrument for analysis.

SPME extraction takes advantage of equilibrium extraction and selective sorption from the matrix onto coating. The analytes adsorbed on the coating can then be desorbed on instrument for analysis and concentration can be estimated by suitable calibration curve.

#### **Calibration Techniques for Accurate Results using SPME**

- External Standardisation: Most Common, Peak Area Plotted vs Concentration of analyte in matrix, Requires high level of standard preparation as all standard points are to be made in the same procedure as that of sample.

-Internal Calibration: Peak Area Count Ratio plotted with ratio of Analyte concentration to IS concentration; thereby calculation of Response Factor. More suited to SPME but cannot be applied directly to Air analysis.

-Equilibrium Extraction: Fiber is exposed long enough to reach equilibrium and the mass on the fiber is calculated using GC; the Equilibrium equation can be used to estimate concentration of matrix. This is particularly useful in air as a long sampling time would allow an equilibrium and the concentration can be estimated by liquid calibration of mass in GC for each analyte. This requires known Partition coefficients, equilibration time, fiber geometry

-Exhaustive Extraction: This is quite similar to equilibrium extraction but here the sample volume is very small and therefore it is assumed that all of the mass of analyte in the matrix has been transferred to fiber. This is much like the analysis of VOCs/ Semi-VOCs using sorbent tubes and mass data. This has been applied to low volume systems in low concentration analytes using a cold fibre or internally cooled SPME system

-Fick's First Law of Diffusion: When the fibre-retracted devices are used and the fiber is moved inside the needle for a known distance then the only means of mass transfer for air/ water to fiber is by diffusion through the gap between opening and fiber coating. For this the Sampling Rate is proportional to molecular diffusion coefficient and ratio of area of opening to diffusion length.

#### Sh. Param Parkash, Vice President Polygamma Recycling LLP, Delhi Registered Ewaste Management & Plastic Waste Management Company

#### WASTE MANAGEMENT

What is waste: Waste is defined as material that is no longer serve a certain purpose and is discarded. Certain type of materials can be reused and certain types are particularly hazardous, if not disposed properly. Hazardous waste has a very negative impact on the environment and health.

Broadly waste is divided into following categories:

1. Municipal Solid waste (MSW): All type of solid wastes generated by households and commercial establishments is termed as municipal solid waste (MSW). It is composed of; vegetable waste 23%, paper/cardboard 30%, plastics 4%, tin, aluminum and other metals 9%, glass 10%, inert material including dust, cinders and miscellaneous 21% and textile 4%. 2. Construction and demolition waste: The construction and demolition (C&D) waste is generated in the construction, maintenance and demolition of buildings, roads, flyovers & bridges etc. It consists mostly of inert and non-biodegradable materials such as concrete, plaster, metal, wood, brick and rubble etc. This type of wastes is heavy and occupy considerable storage space.

**3. E-waste**: E-waste or WEEE (waste electrical and electronic equipment) such as IT and communication, technology equipment i.e. personal computers, laptops, notebooks, I- pads, calculators, telephones and mobile phones etc. Consumer electronics goods such as televisions, air conditioners, refrigerators and washing machines etc. Any equipment which works with electricity or electronic components and discarded is termed as E- waste. E-waste is growing is exponentially due to up gradation of technology (obsolescence of working electronic equipment) and globally rising demand.

**4. Plastic Waste:** Plastic is a very important component in modern human life because of its physical and chemical properties and most of it is recyclable. The indiscriminate use of plastic has become a major threat to the environment.

**5. Biomedical Waste:** Biomedical waste comprises human and animal anatomical waste, treatment apparatus such as needles, syringes and other materials used in health care facilities in the process of treatment and research during diagnosis, treatment or immunization in hospitals, nursing homes, pathological laboratories and blood banks etc. 15% of generated biomedical waste is infectious/hazardous and rest of it is nonhazardous.

**6. Hazardous Waste:** Any waste which cause danger to health or environment is called hazardous waste. It can be extremely harmful and toxic. It is generated by burning or incineration leads to emission of toxic fumes comprising of dioxins and furans, mercury and heavy metals, causing air pollution and associated health problems. Radioactive waste generated by atomic plants and source of X-ray equipment and also comes under this type of waste.

**7. Waste Water:** Waste water is originated from the combination of domestic, industrial, commercial and agriculture activities, surface runoff or storm water and from sewer inflow. Municipal waste water (also known as sewage) is usually conveyed in combined sewer and treated at a waste water treatment plant. One more and important component of the liquid waste or waste water is effluent generated by chemical industries.

8. End of life vehicles Waste: This waste is new type of wastes and is generated by old end of life vehicles. This waste contains over and above metals, plastic wastes, E-waste and also oil waste.

**Waste reduction:** Waste can be reduced by alternative use and by recycling. In new terminology waste is also called wealth if it is processed systematically. This will not only lead to recovery of materials/metals/energy, but also will reduce land filling minimizing the leaching of chemicals thus protecting environment.

In order to handle and process wastes mentioned above, MOEF has framed rules for all types of wastes such as "Solid Waste Management Rules 2016", "Construction & amp; Demolition Waste Management Rules 2016", "E-waste (Management and Handling) Rules 2016" etc.

**Role of Laboratories:** Lab. Management plays very important role in analyzing critical parameters at various stages of Recycling of different types of wastes. Laboratories not only assists in recovery of precious metals and other rare materials, but also guide for environmentally friendly treatment of both air and water with aim of zero effluent discharge to the environment, thus **protecting the environment**.

To maintain the air emission quality and have 'SPM' at desirable level, systems are designed having suitable filtration medias as well as chemicals to neutralize the harmful gases mixed with air. Similarly, the liquid effluent is also designed to be treated at different stages in ETP plant, so that the final discharge of liquid is free from any harmful chemical which can damage the environment. Even the design take care to reuse the treated effluent after ensuring its quality. All the treatments are eco- friendly and conforms to various rules of MOEF and State pollution control boards.

## Dr D S Tewari got Achievement Award from International Accreditation Services, USA



#### **ISO 17025 New Delhi Training**



## **GC Meeting**, 15th September 2017, Delhi



## **GC Meeting**, 21st April 2017, Chennai



#### **# IAF-ILAC 2017, VANCOUVER**





## **OBITUARY**

#### Dr. T. Lal, Former Head Mass Metrology Division, NPL-CSIR, New Delhi

AOIL pays homage to Dr Tripurari Lal, an special invitee to AOIL GC meeting as an expert in mass metrology, Ex Scientist & Head, Mass Metrology Division of NPL-CSIR, New Delhi. His association with AOIL was further strengthened/ reinforced by his long & old friendship with AOIL Chairman Mr DS Tewari, since NCTCF days.

Dr T Lal remained active till his last in AOIL's Proficiency Testing Programs and organized the first PT program of AOIL for mass metrology. Few months before his sudden demise in a fatal car accident on the Yamuna Expressway, Dr Lal joined Farelabs Group, Gurugram as Vice President- Mechanical Calibrations.

Dr. Lal helped Indian weight manufacturers to establish weights calibration labs and subsequently their ISO 17025 accreditation. He his one of the recognized scientist from India for organizing many International Proficiency Testing by NPL in Mass Metrology. In his untimely death, AOIL has lost a senior expert scientist & anable technical activity organizer. His contribution to Indian Mass Metrology would be remembered for year to come.

#### Dr. S. Y. Pandey, Former Director, Global Chemical Research, Jai Research Foundation, NH-8, Valvada, Gujarat

AOIL is grieved to pay its homage, respect and regards to its Former Vice President- GLP, Dr Shiv Yagya Pandey, one of the key founder members of AOIL and close friend of Chairman Mr DS Tewari. He has contributed in development of the AOIL with his active involvement in scientific proceedings & technical symposiums.

Dr SY Pandey was an incredible human being with a great sense of humor and extraordinary intelligence. He touched many souls and minds in different ways, by making a personable connection with each person, he spent time with.

He was the Director, JRF Global, Business Development & Director JRF, Chemical Research for the last 20 years. He had worked with US-EPA, WHO, UNIDO, UNDP. He had closely worked with BIS, FAO, AOAC, CIPAC and AAPCO. Dr Pandey in lighter moments used to say " I am not an old man ageing seventy years, but I am a young man in twenties with an experience of just fifty years". This shows a great enthusiasm he used to spread amongst his fellow colleagues.

AOIL has lost an unique potential resource person, great motivator, global scientist and an able septuagenarian entrepreneur in him.



## **Association of Indian Laboratories**

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