

# The Metrologist



Edition 4 February 2017

**Reagecon**

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Reagecon's Leo Sun pictured at our stand in Analytica Shanghai.



Reagecon's Josie Murphy pictured at our stand in WWEM (Water, Wastewater and Environmental Monitoring) Exhibition held in Telford, England.



John Barron from Reagecon pictured at an ExportMatters event held recently in Shannon Airport supported by Ulster Bank. This event was held to highlight the importance of Shannon Airport to the Irish Economy.



One of a suite of temperature controlled distillation and reflux apparatus photographed in our purification, synthesis and raw material characterisation laboratory.



# Dear Customers, Readers and Business Partners

Welcome to the first edition for 2017 of The Metrologist. Thanks for all your support, custom, feedback and encouragement during 2016, which was a milestone year in the history of Reagecon.

One of the major events of the year was the launch of our Global Metrology Development Centre here in Shannon.

A Metrology Centre would normally be the preserve of a government funded body, but now due to your support, we have the financial and technical competence and confidence to open this new centre, which was launched during our annual distributor conference in October. The rationale, features and benefits of this centre are presented in this edition of the Metrologist on pages 4,5 and 6. We believe, that from a customer's perspective, the features and benefits are compelling and we believe it will bring major value to you over the coming years.

Another major event in 2016 was the launch of our new Physical and Chemical Standards Compendium. This publication contains the largest and most complete range of Physical and Chemical Standards ever produced and brings our total offering of products, to over 8000. Significant information on the compendium was published in the August 2016 edition of the Metrologist, but in order to reflect the importance of this document to you, our customers, and all our channel partners, we have published an overview on pages 7 and 8.

Ultimately Reagecon is a high-technology company that specialises in the field of Metrology. It is incumbent on us, as part of our offering to provide you with as much scientific, technical and product information as possible. We have taken the liberty to include several papers, that cover these objectives, in this Metrologist.

We hope you find this information of value. We also hope that we can bring you the best possible value to help you with your analysis during 2017 and beyond. On behalf of all of our colleagues here in Reagecon, we wish you well in your daily efforts to achieve the correct results, in all your tests and that we as a standards manufacturer can enhance and offer proof of that correctness.

Regards,  
**John J Barron**  
Managing Director



Reagecon's Managing Director John Barron and his wife Brigid pictured at the launch of the Global Metrology Development Centre.



The official handshake celebrating the launch of the new Global Metrology Development Centre.

**Pictured Left to Right:** Bernard Gleeson (Reagecon's Export Sales Manager), John Barron (Reagecon's Managing Director), Maurice Buckley (CEO NSAI) and Don Foynes ( Reagecon's Commercial Director)

# Reagecon's Global Metrology Development Centre



Our new Global Metrology Development Centre was officially launched as part of our 2 day Annual International Distributor Conference. The event took place on 20th and 21st October 2016 and was attended by over 70 delegates from 40 companies, located in 28 countries.

## 1.0 Introduction and Context-Global Metrology Development Centre

As a world leader in the manufacture of Physical Standards, Chemical Standards and Certified Reference Materials (CRM's), we have an offering of over 8,000 different part numbers, all of which have been developed in-house. Our four main competitive advantages as stated in all of our marketing and business planning material are:

- New product development
- Accreditations
- Business development
- Efficiency and effectiveness

The core intellectual property (IP) and expertise underlying the development, manufacture, testing and certification of such products is the science of Metrology.

The science of Metrology also underpins all four components of our suite of competitive advantages to a greater, or lesser extent, in particular, new product development and accreditations.

The main applications of Standards and CRM's are as follows:

- 1) Instrument Calibration
- 2) Quality Control Materials
- 3) Test Method Validation
- 4) Instrument Qualification (pharmaceutical industry)
- 5) Proficiency Testing
- 6) Analyst Qualification

The key elements of the science of metrology from a technical perspective include but are not limited to the following:

- Measurement uncertainty
- Accuracy
- Precision
- Specificity
- Sensitivity
- Comparability
- Certification

There are several drivers currently causing the market for Standards and CRM's to grow exponentially. These include:

- Globalisation
- Population expansion
- Pharmaceutical development
- Advances in food technology
- Greater regulation
- More advanced and sensitive scientific instruments

We believe this Metrology Centre will impact our competitive advantages, enhance our application support, develop our Metrology capability and enable us to respond more rapidly and comprehensively to the market drivers.





## 2.0 Collateral Value

However, it is from a marketing, image and perception value that the Global Metrology Centre will have its greatest customer value. The tangible benefits in terms of outputs include, but are not limited to the following:

- A training facility for 300 international distributors on Metrology
- A training facility for 1,000 Irish customers on new products
- A training facility for our 25 Business Development staff on new products.
- Area for upskilling existing staff
- Provide an area for collaboration and research with National Metrology and National Reference Centres worldwide
- Establish Reagecon as a global Metrology Centre of excellence in the Science of Metrology
- Facilitate the rapid development of Certified Reference Materials in all four sciences of pH, Conductivity, Refractive Index and Density
- Form a platform for adding other Primary Reference Methods in areas such as Viscosity, Colour and Osmolality

The Graphic below and photos shows an outline of some of the equipment required for this project. This includes a goniometer-spectrometer, a number of photos of hydrostatic weighing equipment and density pycnometry equipment and one photo of a signal processing meter.



# Reagecon's Physical and Chemical Standards Compendium.

In the past few years substantial changes have occurred in the field of Analytical Chemistry. Stringent regulatory demands, combined with major economic implications and increased competitiveness, places necessity for validation on every analytical test performed, either in the laboratory or in the field. Not only must the correct result be obtained, but proof must also be provided of its fitness for purpose, validity and accuracy. Such proof must then be accessible, retrievable and presented in an easily understood format. Reagecon continues to respond to these challenges by presenting to its customers, an ever increasing range of highly specified, stable, traceable and certified standards.

The use of standards such as calibrators or control materials can greatly increase the possibility for the analyst to obtain the correct result and can provide definitive proof of the correctness of such a result from a technical perspective. Such materials can also be used for method validation, instrument qualification, verification and analyst qualification.

Since the beginning of 2011, we have developed a major pipeline of new products and in our 2016 Compendium, we now have a broader and more comprehensive range of physical and chemical standards than any other producer worldwide. We are privileged to be able to present these new ranges to you, (in excess of 8,000 product numbers). This is the largest range of physical and chemical standards ever presented in one publication.

We hope you find this compendium beneficial; that the products on offer match your technical specifications; represent value for money and that they will greatly enhance your ability to achieve valid and correct analytical results now and in the future. Many of the chapters and families of products contained within the compendium are introduced with detailed technical notes. The compendium can be viewed in full on our website [www.reagecon.com](http://www.reagecon.com) and the following summarises the contents.

## Organic Standards

- Volatile Organic Compound Standards (VOCs)
- Phenol Standards
- Polycyclic Aromatic Hydrocarbon Standards (PAHs)
- Pesticide Standards
- Azo Dye Metabolite Standards
- Fatty Acid Methyl Ester & Fatty Acid Ethyl Ester Standards (FAME & FAEEs)
- Nitrosamine Standards
- Polybrominated Biphenyl Standards (PBBs)
- Polybrominated Diphenyl Ethers (PBDE) & Other Flame Retardant Standards
- Polychlorinated Biphenyl Standards (PCBs)
- Phthalate Standards
- Semi Volatile Organic Compound Standards (SVOCs)
- PIANO, PONA & PNA Standards
- Petrochemical Standards



## Total Organic Carbon/Total Inorganic Carbon Standards

- Premium Range
- Quality Range
- Instrument Specific Range

## Electrochemistry Standards

- Conductivity Standards
- pH Buffer Solutions
- Electrode Care & Maintenance Solutions
- Redox Standards
- Turbidity Standards
- Chemical Oxygen Demand
- Ion Selective Electrode Standards & Ionic Strength Adjustors

## Standards for Anion & Cation Analysis

- ICP-MS/ICP-OES Standards
- Ion Chromatography Standards
- Atomic Absorption Standards
- Flame Photometry Standards



## Titration

- Analytical Volumetric Solutions & Indicator Solutions
- Total Acid Number/ Total Base Number Standards & Reagents

## Physiochemical Standards

- Colour Standards
- Spectrophotometry Standards
- Melting Point Standards
- Density Standards - Premium Range
- Density Standards - Quality Range
- Viscosity Standards
- ISO Guide 34 Certified Reference Materials: Sucrose in Water Standards
- Brix Standards (Stabilised)
- Refractive Index Standards
- Osmolality Standards
- Cryoscope Standards



## Standards & Solutions in Compliance to Pharmacopoeias

- United States Pharmacopoeia Solutions
- European Pharmacopoeia Solutions
- Buffered Eluents
- Dissolution Media - Concentrates
- Dissolution Media - Ready to use



## Industry Specific Standards & Reagents

- Dairy Standards & Reagents
- Standards & Reagents for APHA, AWWA & WEF Test Methods
- Wine Standards & Reagents
- Soil Testing Standards & Reagents
- Pulp & Paper Standards & Reagents

## General Laboratory Standards & Reagents

- Laboratory Water
- Cleaning Solutions
- Analyst Qualification Sets



# pH Buffer Solutions

## 1.0 Guaranteed Traceability

Reagecon's pH buffer standards are directly traceable to the IUPAC pH scale by an unbroken chain of traceability. Reagecon achieve this traceability through a series of comparisons, with the key reference materials being Standard Reference Materials (SRMs) manufactured by NIST.

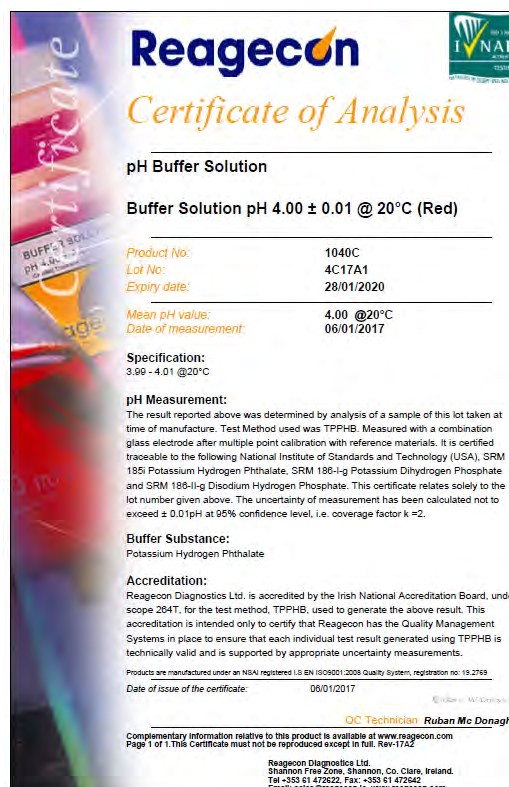
For proof of traceability, all of these comparisons must be made in a technically - valid manner and the accuracy of each step must be quantified by calculating the associated Uncertainty of Measurement. Reagecon's pH buffer standards meet the ISO definition of traceability: "The ability to relate measurements back to a stated reference (usually an international standard) through an unbroken chain of comparisons, each having stated uncertainties of measurement." Reagecon's traceability claims are guaranteed by our accreditation to ISO/IEC 17025.

## 2.0 Why use traceable pH buffers?

Your pH measurements can only be as good as the pH buffers that you use. If your pH calibration is made using traceable pH buffers then you have a direct link to the International pH scale for your measurements. Without this link, you are not entitled to report your measurements in pH units so the number displayed on your pH meter is just that - a number and is not a pH value. The common link that is achieved by traceability allows comparability of results regardless of:

- When the measurements were made
- Where the measurements were made
- What instrumentation was used to make the measurements

Traceable analysis is necessary for consistency and universal acceptance of your pH results - including acceptance by regulatory bodies.



### 3.0 Fully Accredited

Reagecon's pH analysis is accredited to ISO/IEC 17025 (INAB Ref:264T) "General requirements for the competence of testing and calibration laboratories". Reagecon's accreditation to ISO/IEC 17025 gives independent proof of three key areas:

- Our pH analysis is technically valid and is carried out in a thoroughly controlled manner by highly qualified staff
- Our claims over the accuracy of our pH analysis are valid and we have properly quantified our accuracy in our Uncertainty of Measurement calculations.
- Our pH analysis is traceable to NIST SRMs. It is important to note that NIST do not police claims of traceability to their SRMs. Any manufacturer of pH buffers can claim that their buffers are traceable to NIST, but only manufacturers that are accredited to ISO/IEC 17025 have independent proof of their traceability.

Reagecon's accreditation is indicated by the Irish National Accreditation Board (INAB) logo on our Certificates of Analysis for pH Buffers. Accreditation by INAB and all other accreditation boards validated to accredit ISO/IEC 17025 are mutually recognised as being directly equivalent.

Why take chances with your pH buffer supplier's traceability? By using buffers from a manufacturer that holds ISO/IEC 17025 accreditation you have a guarantee of traceability.

### 4.0 Stability

Reagecon's pH buffers have been specially formulated to ensure their stability. The packaging bottles that we use have also been selected and tested to provide maximum stability. We have conducted stability trials on both freshly-opened and part-full bottles of our pH buffers to validate their shelf-life - an example is given in Figure 2. This demonstrates that Reagecon's pH buffers will stay within their specification limits up to the stated expiry date regardless of when the bottle was first opened (provided that the pH buffer is stored in accordance with good laboratory practice). Most of Reagecon's pH buffers have an expiry date of either 2 years or 3 years from the date of manufacture.

This means that our pH buffers' expiry dates are an absolute value and they have a long "Active Life". We do not quote a short usage period after opening the bottle and there is no need to record by hand an "Opened on date" and a "Use by date". With Reagecon's pH buffers you just open the bottle and use the contents - with other manufacturers' pH buffers you need to record these extra dates and may need to dispose of most of the contents of the bottle at the end of its short "Active Life".

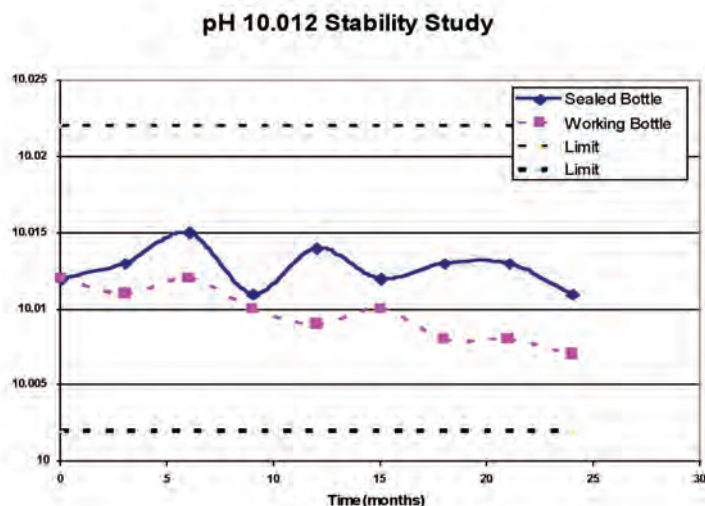


Figure 2: Stability Data for Reagecon pH 10.012 @ 25°C



## 5.0 Extensive Range of pH values

Reagecon manufacture the most comprehensive range of pH reagents in the world; these are designed to suit all end user requirements. These include laboratory grade buffers, the Professional Range (buffer standards as per N.I.S.T/DIN and high resolution buffers), low ionic strength buffers and pH buffer capsules. We are delighted to add several new offerings that include buffers to calibrate Antimony electrodes, Sterile Buffers and colour coded pH buffers with a three decimal place specification.

All are manufactured to exacting specifications with an extended shelf life and cover the pH range of pH 1.00 to pH 13.00 inclusive. All are supplied with a detailed Certificate of Analysis which outlines traceability to N.I.S.T (the N.I.S.T SRM(s) Lot No. is stated on the certificate). Temperature dependence data is printed on the label as are lot numbers and expiry dates.

In summary Reagecon Manufacture the most comprehensive range of pH reagents in which are designed to suit all end user requirements. These include;

- pH Buffers @ 25°C, range : pH 1-13
- pH Buffers @ 20°C, range : pH 1-13
- pH Buffers @ 38°C, range : pH 4,6,7 & 8
- Colour Coded Buffers at both 20 and 25°C, range : pH 1-13
- Single use Calibration Buffers, range : pH 4,7,9& 10
- Buffer Capsules, range : pH 4,7,9& 10
- DIN 19266 values @ 25°C
- DIN 19267 values @25°C
- NIST Values @ 20°C
- High Resolution Buffers, range
- Antimony Buffers Technical
- pH Buffer Solutions
- Low Ionic Strength Buffers
- Sterile buffers

**Reagecon have been world leaders in the manufacture of pH buffers for over 30 years and have published many technical papers on pH including the following paper; "The Effects of Temperature on pH Measurement"**



# The Effects of Temperature on pH Measurement

Authors: John J. Barron, Colin Ashton & Leo Geary – Technical Services Department, Reagecon Diagnostics Ltd, Shannon Free Zone, County Clare, Ireland

## Abstract

*Some of the effects of temperature on pH measurement have been described or alluded to in both instrument manufacturers instructions and independent scientific literature. Some of these have been presented in a very theoretical manner and thus have been of limited practical value, others have been described adequately from a practical perspective. However a comprehensive classification of the principal effects of temperature on pH with practical remedial actions to correct, reduce or eliminate these errors has never previously been reported. This paper presents for the first time a clear classification of the practical effects of temperature on pH. It provides a definite set of guidelines on how the different errors can be reduced or eliminated thus giving the user greater confidence in the result of their pH measurements.*

## 1 Introduction

Accurate measurement and reporting of pH data has been a long-standing problem due to the effects of temperature. An increase in any solutions' temperature will cause a decrease in its viscosity and an increase in the mobility of its ions in solution. An increase in temperature may also lead to an increase in the number of ions in solution due to the dissociation of molecules (this is particularly true for weak acids and bases). As pH is a measure of the hydrogen ion concentration, a change in the temperature of a solution will be reflected by a subsequent change in pH<sup>(1)</sup>.

In addition, changes in temperature will also have an influence on the measuring sensor used.

Overall, the effects of temperature can be divided into two main categories:

- Temperature effects that diminish the accuracy and speed of response of the Electrode.
- Temperature Coefficient of Variation effects on the material being measured by the sensor, whether it be calibration buffer or sample.

Further sub-classification is necessary for each of these categories in order to understand the cause of the problem and determine the optimal remedial action.

## 2 Temperature Effects on the Electrode

Temperature has a variety of effects on pH electrodes of both a physical and chemical nature. These will be discussed in detail under the following headings and include recommendations on how to reduce or account for these sources of error.

- Temperature effects on electrode slope
- Calibration Isothermal point
- Thermal equilibrium
- Chemical equilibrium
- Membrane resistance

## 2.1 Temperature effects on electrode slope

The response of an ideal pH electrode is defined by the Nernst Equation<sup>(2)</sup>:

$$E = E^0 - 2.3 (RT/nF) \log a_{H^+}$$

where:

- E = total potential (in mV) developed between the sensing and reference electrode
- E<sub>0</sub> = standard potential of the electrode at a<sub>H<sup>+</sup></sub> = 1 mol/l
- R = Gas constant
- T = temperature
- F = Faraday constant
- n = valency of ion
- a<sub>H<sup>+</sup></sub> = activity of the hydrogen ion in solution

The term 2.3RT/nF is referred to as the Nernst slope. Table 1 shows the changes in Nernstian slope at increasing temperatures for the ideal electrode<sup>(3)</sup>. This change in slope with temperature can be compensated for by the meter using Automatic Temperature Compensation (ATC). All modern pH meters allow for the measurement of the sample temperature using a temperature probe placed in the sample. The meter then adjusts the slope to be correct in accordance with the Nernst equation at the measured temperature during calibration. For manual compensated instruments, a potentiostat is built into the instrument, which can be adjusted by the operator to facilitate the correction.

ATC relies on the accurate measurement of the temperature of the solutions. This can be achieved by using an electrode with a built in temperature sensor or a separate temperature sensor. Combined pH/temperature electrodes offer the advantage that only one probe is required to perform measurements. Such sensors can suffer from a sluggish response since the glass, which surrounds such sensors, has a low thermal conductivity. Alternatively, a separate temperature sensor can be used with the pH meter. However, this can be cumbersome for measurement in small vessels.

Temperature °C	Nernst slope (mV/pH)
0	54.20
10	56.18
20	58.17
25	59.16
30	60.15
40	62.14
50	64.12
60	66.10
70	68.09
80	70.07
90	72.06

**Table 1: The variation in Nernst slope with temperature for an ideal electrode**

## 2.2 Calibration Isothermal Point

The isothermal point of an electrode is defined as the intersection point of calibration lines plotted for different temperatures<sup>(4)</sup> and is characterised by both a pH value and a potential. For an ideal electrode this would represent an isothermal intersection at the zero point (0mV at pH 7) for different temperatures<sup>(2)</sup>. For real electrodes the individual potentials which contribute to the overall potential output have separate temperature coefficients of variation. Thus the isothermal intersection point hardly ever coincides with the zero point of the electrode (see Figure 1). Substantial work has been done on bringing the isothermal intersection and the zero point as close as possible so that the resultant error due to temperature effects can be reduced. This error increases as the temperature difference between the calibration buffer and the sample solution widens. The error can be as high as 0.1 of a pH unit<sup>(6)</sup>. It can only be eliminated when the temperature of the calibration and the sample solutions are identical under controlled conditions, for example by incubating the calibration buffers and samples in a water bath at a constant temperature. It is important to point out that this error cannot be eliminated by ATC.

## 2.3 Thermal Equilibrium

Thermal equilibrium effects will be seen when the electrode is transferred between solutions of different temperatures or the temperature of a particular measuring solution changes rapidly during measurement. The resulting slow response of the electrode can manifest itself in drift, which continues until the sample and electrode have reached thermal equilibrium.

A number of researchers have devised solutions to this problem. These solutions include electrode designs with symmetrical warming up or cooling down of the lead-off elements on both the sensing and reference electrodes. An alternative solution developed has been the manufacture of electrodes of different internal chemistry, which are independent of temperature effects<sup>(7)</sup>. Such dramatic temperature differences are rarely encountered in a laboratory situation and where accurate work is being performed, the remedial action is to perform the calibration and sample measurement at a uniform controlled temperature using a water bath.

## 2.4 Chemical Equilibrium

Reference electrodes function on the basis of a chemical equilibrium between a metal and a solution of its ions, which generates a potential. This is affected by temperature because the solubility of the metal salt in the reference electrolyte solution varies with temperature. Changes in this equilibrium will cause the electrode to respond slowly and leads to drift. This error is minimal with modern good quality electrodes and can be overcome if necessary in the same way as thermal equilibrium by incubation of controls and samples in a temperature controlled water bath.

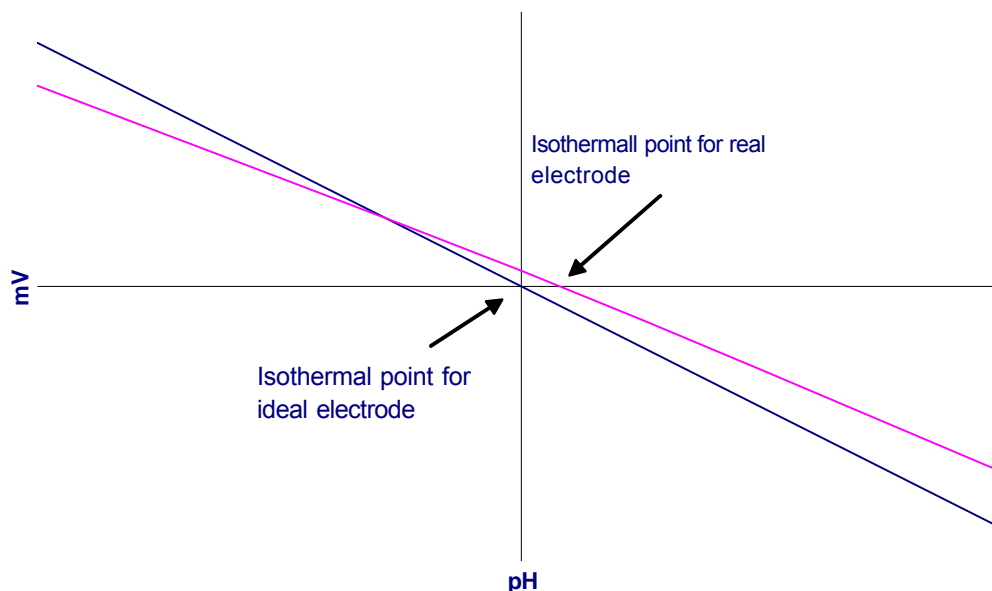
## 2.5 Membrane Resistance

The resistance of pH membrane glass increases with decreasing temperature. The resulting increase in membrane resistance leads to the electrode having a sluggish response. The degree of change in resistance varies depending on the membrane glass formulation and the membrane shape. This change in resistance may be five-fold for as little as a 15°C drop in temperature. Therefore, a membrane with a resistance of 200 megaohms (mΩ) at 25°C would increase to over 1000 mΩ at 10°C<sup>(8)</sup>.

The lowest possible operating temperature of a pH electrode is often determined by the resistance increase of the glass membrane at lower temperatures. The effects of membrane resistance are as follows:

- The electrode may not be useable at all at a particular temperature.
- Even if the electrode is useable, there may be electronic noise from the power cable
- Low resistance at connectors or plugs due to corrosion or moisture may manifest themselves at greatest effect when resistance is very high.

In order to avoid the errors associated with this high resistance, it is important to only use an electrode at low temperatures which has resistance of the order of 100 – 200 mΩ at 25°C.



**Figure 1: Isopotential points for the ideal and real electrodes at 25°C**

In practice there is a trade-off between membrane resistance and fitness for purpose. The shape of the spear membrane makes it ideal for use as a stick-in electrode for the measurement of pH in meat or cheese at 4°C and is supplied in this format combined with a low impedance glass membrane<sup>(9)</sup>.

### 3 Effects of Temperature coefficient variation on the calibration buffer or sample

The importance of temperature measurement when performing pH measurements has already been mentioned in reference to slope correction. Temperature also has an effect of both pH buffers and solutions, as the hydrogen ion activity will increase with increasing pH.

#### 3.1 Temperature coefficient of variation of pH buffers

Although the temperature coefficient variation of pH buffers may vary only minimally over a wide span of temperatures in the acid region, it may be quite dramatic in the alkaline region. The consequential error can be substantial in the alkaline region (see Table 2). There are three mechanisms to overcome this temperature coefficient variation:

- The correct pH at a particular temperature may be checked on “lookup” tables on the pH buffer label and the correct value manually inserted into the pH meter at calibration. This approach suffers from the drawback of requiring knowledge of the correct ambient temperature, which can vary quite substantially in a field situation.
- Most modern pH meters have an auto buffer recognition facility, whereby values of pH buffers at various temperatures are stored in memory. Meter standardisation and temperature coefficient of variation correction is therefore automatically done if the correct buffer is used. Meter manufacturers generally specify specific pH buffer types to be used for calibration - for example some pH meters can be calibrated using technical pH buffers or DIN/NIST buffers (values specified at 25°C)<sup>(11)</sup>. Use of the incorrect buffer type can lead to inaccurate calibration and hence incorrect pH measurements.
- The measurement of the sample and the buffers can be done under controlled temperature conditions in a water bath, for example at 20°C or 25°C. Such an option is generally only available or feasible for very accurate laboratory work.

Temperature °C	pH 4. ±00 0.01 @ 25°C <sup>(10)</sup>	pH 7. ±00 0.01 @ 25°C <sup>(10)</sup>	pH 10 ±00 0.01 @ 25°C <sup>(10)</sup>
10	4.00	7.07	10.18
15	4.00	7.04	10.14
20	4.00	7.02	10.06
25	4.00	7.00	10.00
30	4.01	6.99	9.95
35	4.02	6.98	9.91
40	4.03	6.97	9.85
50	4.05	6.96	9.78
60	4.08	6.96	9.75

**Table 2: Temperature effects on the value of pH buffers**



### 3.2 Temperature coefficient of variation of the sample

Under normal operating conditions this variation may be minimal in the acid region but can be quite dramatic in the alkaline region with significant variation in the neutral area. Table 3 gives an example of pH variation of different solutions with temperature.

The effects of temperature on the pH of the sample can be accommodated as follows:

- It is absolutely imperative for all samples when a pH value is reported that the temperature at which the measurement is done is also reported. This facilitates comparability and reduces the potential for error or misunderstanding.
- The sample may be placed in a water bath at 20°C or 25°C and measurements taken and reported only at these temperatures.
- In some instances where there is no variation in matrix and the sample is always homogenous, the temperature coefficient variation can be characterised and a “lookup” table established. However, in practice this is rarely done as such a study would be quite laborious. It is also entirely dependent on the make up chemistry of the sample always remaining the same.

pH Range	Temperature		
	0°C	25°C	60°C
Acid	pH 0.99	pH 1.00	pH 1.01
Neutral	pH 7.47	pH 7.00	pH 6.51
Basic	pH 14.94	pH 14.00	pH 13.02

**Table 3: Typical changes in pH for solutions due to temperature coefficient of variation effects**

### 5 References

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11. Operating manual for CG843P pH meter. Schott Instruments GmbH, 1999, ba12244e\_2, p85.

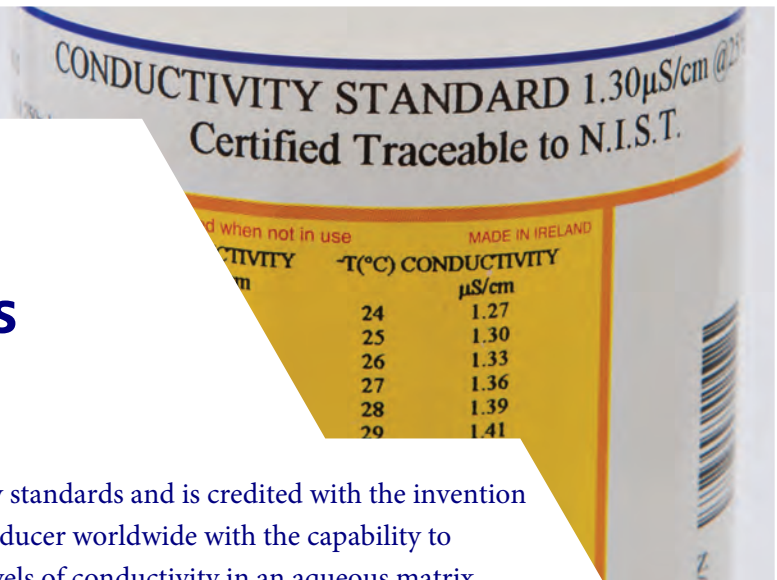
This paper was presented as a poster at the 57th Annual Meeting of the International Society of Electrochemistry, which was held in Edinburgh in September, 2006.

### 4 Conclusion

Temperature has a number of significant effects on pH measurement. Analysts must have an appreciation of these effects so that they can improve and maximise the accuracy of their pH measurements. Automatic Temperature Compensation (ATC) should always be utilised during calibration to correct for the non-Nernstian slope of electrodes. For pH meters that feature automatic buffer recognition, the correct calibration buffers, as specified by the instrument manufacturer, must always be used as the meter has the temperature profile for these buffers stored in memory. Additional sources of error include the calibration isopotential point, thermal or chemical equilibrium effects on the electrode and the temperature coefficient of variation on buffers and samples. To reduce these potential errors, instrument calibration and sample measurement should be performed at the same temperature ideally using a water bath. Also, as the pH of solutions is temperature dependent, the measurement temperature should always be recorded.

Implementation of these simple guidelines will lead to either a reduction or elimination of the problems associated with temperature. This will result in improved accuracy in pH results thus giving the user greater confidence in their pH measurements. Depending on the decisions made based on the pH readings the consequences of inaccurate pH measurement can be more than just a waste of the analysts’ time.

# Conductivity Standards



## 1.0 Introduction

Reagecon is the world's largest producer of conductivity standards and is credited with the invention of low level aqueous standards. We are still the only producer worldwide with the capability to manufacture and stabilise these products at such low levels of conductivity in an aqueous matrix. This low range of standards includes  $1.3\mu\text{S} \pm 0.5\mu\text{S}$  - the lowest aqueous conductivity standard available worldwide. The following summary details the principle features and benefits of this exciting range of products.

## 2.0 Extensive Range of Values

Reagecon offer over 45 different values of Conductivity and Total Dissolved Solids (TDS) standards, ranging from as low as  $1.3\mu\text{S}/\text{cm}$  to as high as  $500,000\mu\text{S}/\text{cm}$ . Customised or bespoke values can be manufactured on demand.

## 3.0 Matrix Matched

The matrix of a solution is defined as "the components of the sample other than the analyte". In all analytical measurements, it is of utmost importance that the matrix of the standard and the sample are the same.

As conductivity measurement is, in the main, a water quality measurement, the standard used should also have an aqueous matrix. Reagecon's conductivity standards are all aqueous based, thereby eliminating any errors attributable to matrix mismatch.

## 4.0 Non-Hazardous

As Reagecon's conductivity standards are aqueous, they are non-hazardous. They offer the following benefits over solvent-based conductivity standards

- Ease and cost of shipping, without the need to provide hazardous goods' paperwork
- Reduced Health & Safety requirements for storage and use
- Ease and cost of disposal - solvent-based conductivity standards require expensive, specialised disposal to comply with environmental regulations.



## 5.0 Guaranteed Stability

As a result of the extensive R&D that led to our innovative manufacturing process, Reagecon can guarantee the stability of their complete range of conductivity standards over their entire shelf life. The stability offered by Reagecon's conductivity standards varies from that of their competitors in one vital area.

We can guarantee that our conductivity standards will remain within specification, (up to their expiry date), right through their working life, regardless of when the bottle was first opened provided Good Laboratory Practice is adhered to. This eliminates the need to open a fresh bottle of standard every time the product is used. (The 1.30µS/cm conductivity standard is packaged in single-dose bottles and each bottle when opened can only be used once.)

The shelf life of the products from their date of manufacture are given below.

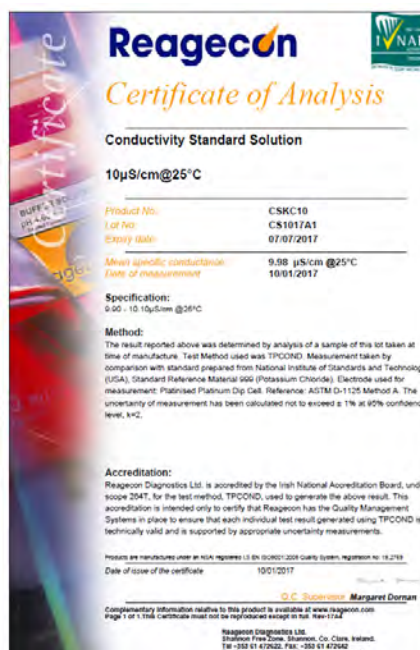
Conductivity Value (µS/cm)	Shelf Life
1.3 & 3	3 months
5 & 10	6 months
20 - 147	12 months
200 - 500,000	18 months

## 6.0 Accuracy

All standards have a specification of  $\pm 1\%$ , except 1.30µS/cm, which has a specification of 1.25 - 1.35µS/cm. This high level of accuracy enables the standards to be used as calibrators and/or controls in fulfilment of the most exacting industrial statutory requirements, for example the United States Pharmacopoeia monograph for Water for Injection.

## 7.0 Accreditation

Reagecon's conductivity measurement has been covered in the scope of our accreditation to ISO 17025 "General Requirements for the competence of Calibration and Testing Laboratories" and its predecessor, EN 45001, since 1990. ISO/IEC 17025 (INAB Ref. 264T). Achieving accreditation involves fulfilling many highly technical criteria, including fully validating our test methods and instrumentation systems and characterising our measurement uncertainty. Reagecon's accreditation proves the technical competence of our personnel, the technical validity of our test procedures and the traceability of our measurements. Therefore, in purchasing a conductivity standard from us, not only do you have transparent traceability to primary standards, but you also have confidence that our standards are of a well-defined and tightly controlled specification.





## 8.0 All values are Certified & Traceable

Comprehensive Certificates of Analysis are available for all of Reagecon's conductivity standards, including detailed information on:

- Product Number
- Lot Number
- Expiry Date
- Mean specific conductance
- Date of Measurement
- Assay Limits
- Test Method Used
- Uncertainty of Measurement and Traceability Data



The complete range is traceable to primary standards from the United States National Institute for Standards and Technology (NIST). The traceability of these standards is proven by the inclusion of conductivity testing in our ISO 17025 accreditation. It is a fundamental requirement of ISO 17025 that traceability is proven

## 9.0 Characterised Temperature Coefficient of Variation

Reagecon's standards are aqueous based and consequently have a very low temperature coefficient of variation. A table of conductivity variation with temperature is printed on the label of each bottle. This feature provides the user with all the information necessary to use the products across the full range of measurement temperatures encountered for their application.

Non-aqueous standards have a very high coefficient of variation which leads to measurement error and renders the products totally unsuitable for non-temperature controlled conditions, or field work.

## 10.0 Unparalleled Technical Support

We have been manufacturing conductivity standards for over 25 years. In that time, we have built up a vast resource of technical expertise on all aspects of conductivity measurement.

The members of Reagecon's Technical Services Department have written a comprehensive series of papers covering all of the practical requirements for accurate conductivity measurement.

These papers and the Reagecon book, "A Practical Guide to Accurate Conductivity Measurement" are available via our website - [www.reagecon.com](http://www.reagecon.com) Our Technical Services team is always on hand to answer any questions regarding the selection and use of conductivity instruments, sensors and standards.



# Titrimetry

Author: John J. Barron, Reagecon Diagnostics Ltd.

## 1.0 The Principle of Titrimetry

Titrimetry or measurement by titration includes a set of widely used analytical techniques, some of which have been in widespread use for almost 200 years.

Volumetric titration dates back at least to the work of French chemist Gay-Lussac, who devised a method in 1835 to determine the purity of Silver, using standardised Sodium Chloride as the titrant.

The principle of all titrimetry involves the determination of the quantity of the reagent of known concentration (titrant), that is required to react completely with an unknown analyte.

Volumetric titrimetry involves measuring the volume of the solution of known concentration (titrant) consumed, gravimetric titrimetry measures the mass of the reagent consumed and coulometric titration measures a direct electrical current of known magnitude that consumes the analyte. In coulometry, the time it takes to complete the electrochemical reaction, is the measurand.

An analytical volumetric solution (also called titrant, standard titrant or standard solution) is a reagent of known concentration that is added from a burette or other dispensing apparatus to a sample (analyte) until a reaction between the two liquids is judged to be complete.

This completeness (end point) is usually observed in a manual titration by the production of a physical change read visually as the titrant is added to the analyte. Such a change may include an appearance, disappearance or change of colour or appearance/disappearance of turbidity (cloudiness).

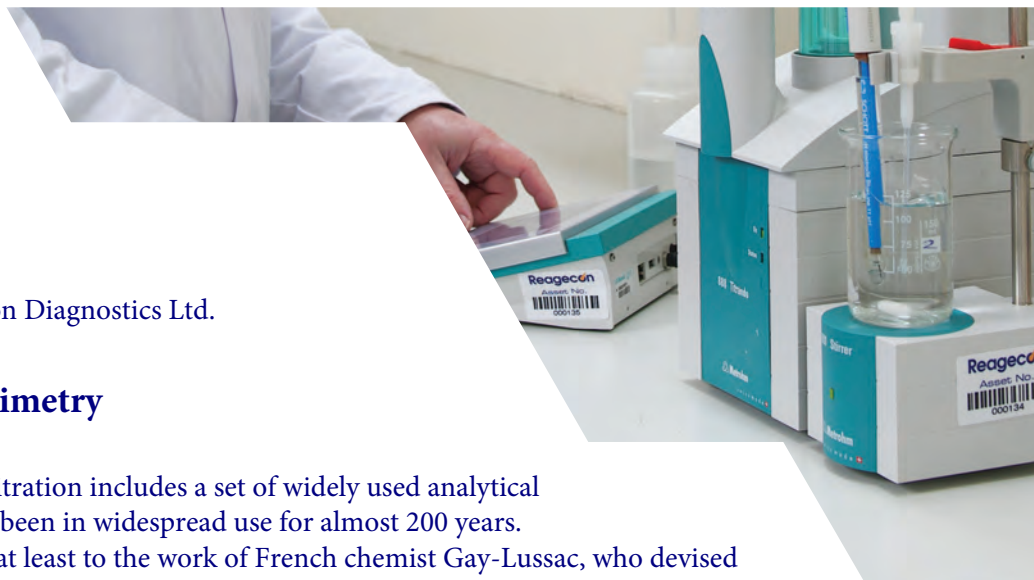
Nowadays, instruments are widely used to detect the end points by detection of any of several properties or characteristics of the analyte solution including colour, turbidity, temperature, refractive index, potential difference, current or conductivity.

In simple terms titrimetry is broadly divided into two main classifications - manual and instrumental - irrespective of how the end point is detected. In the case of manual titrations, indicator, titrant or analyte change of colour is by far the most important method of end point detection. Therefore, the availability of a wide selection of indicators is an integral part of any offering of Analytical Volumetric Solutions. Our compendium carries by far the most extensive offering of both indicators and titrants available in the market place. The end point in automatic titration is indicated most commonly by a change in potential of an electrode that responds to the concentration of the reagent or the analyte.

**Analysis by titration brings a large number of benefits to the analyst including the following:**

- Relatively easy to perform (although high accuracy manual titration requires practice, dexterity, experience and sound judgement)
- Rapid, cheap and versatile
- Accurate, reproducible, traceable and comparable

Furthermore, titration reactions should exhibit defined stoichiometry, be quantitative, establish equilibrium that is definite and fast, and provide unambiguous results.



## 2.0 Types of Titration Reactions

### 2.1 Acid/Base reactions (also called neutralisation titrations)

These are used to determine either the amount of acid/base in an analyte or substances that can be converted to an acid/base. They may also sometimes be used to track the progress of chemical reactions that produce or consume hydrogen ions. The titrants are always strong acids or bases and include hydrochloric acid, perchloric acid, sulphuric acid, sodium hydroxide, potassium hydroxide and sometimes barium hydroxide.

Weak acids or bases are not used because they react incompletely with the analyte. The colour indicator used in an acid base titration is a weak acid/base itself which in its undissociated form differs in colour from its conjugate acid or base form. Typical elements suitable to this type of titration method include carbon, nitrogen, chlorine, bromine and fluorine. Pretreatment of these elements converts the element to an inorganic acid or base that is then titrated. An example is nitrogen which occurs in a wide range of forms both organic, inorganic or as a constituent of biological materials. Therefore, a methodology for nitrogen measurement in amine groups such as the Kjeldahl method is extremely important in determining the protein content in grains, meats, and other human or animal foodstuffs. In addition to amines, others like esters and hydroxyl functional groups can also be determined. In addition, inorganic compounds such as carbonates, ammonium salts and several other NO<sub>x</sub> species can be determined.

#### Fields of Application

- Acid content in wine, milk, ketchup, fruit juice (etc)
- Content of HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, NaOH, KOH
- Alkalinity determination in water
- TAN and TBN in petroleum products, edible or inedible oils and fats
- Determination of boric acid in cooling fluids of nuclear power stations
- Determination of free or total acidity in plating baths
- Determination of active ingredients in drugs or raw materials for the pharmaceutical industry
- Total nitrogen determination by Kjeldahl
- Wide range of inorganic, organic or biological species that possess inherent acidic or basic properties
- Use of chemical treatment that converts an analyte to an acid or base followed by titration with standardised strong acid or base



### 2.2 Oxidation/Reduction Titrations

These titrations may be performed manually or potentiometrically. In manual titrations, if indicators are used, they change colour upon being oxidized or reduced, independently of the chemical nature of the titrant or analyte. Instead, they depend on changes in the electropotential of the oxidation reduction system. Examples of such indicators include:

- Iron (III) complexes of orthophenothrolines
- Starch solutions
- Potassium thiocyanate

The principle of this type of titration involves a reaction between an oxidising and reducing pair, e.g. titration of iron

(II) with cerium (IV) sulphate

- Oxidising agents (examples)
  - Iodine (Iodometry), potassium dichromate, potassium permanganate, potassium bromate, cerium (IV) ammonium nitrate, cerium (IV) ammonium sulphate, cerium (IV) hydrogen sulphate, cerium hydroxide, chlorine
- Reducing agents (examples)
  - Sodium thiosulphate, oxalic acid, iron ammonium (II) sulphate (Mohr's salt), hydrogen peroxide, phenylarsine oxide (PAO), iron (II) ethylene diamine sulphate

## Fields of Application

- Environment
  - COD of water
  - Oxidation capacity of water by permanganate
- Food and beverage
  - Determination of free and total  $\text{SO}_2$  in water, wine, alcohol, dried fruit etc
- Pharmaceuticals
  - Vitamin C determination
  - Surface treatment
  - Titration of copper or tin using iodine
  - Titration of chromium (VI)
- Petrochemicals
  - Determination of water in hydrocarbons



## 2.3 Complexometric Titrations

Complexometric reactions have many applications in chemical analysis and in science in general. Their use in titrimetry is a very important one of these applications. The reaction end point is detected either potentiometrically or manually using an indicator, whereby, a metal ion reacts appropriately with a ligand to form a complex.

EDTA is the most widely used titrant in complexometric reactions although the use of other chemicals similar to EDTA are described in the literature; e.g. nitrilotriacetic acid. Generally, organic dyes that form complexes with metal ions to form chelates are used as indicators, a commonly used one being Eriochrome Black T.

Methods have been developed, validated and published for detection or quantification of almost every metal in the periodic table with the exception of the Alkali metals using EDTA complexation. This includes methods for at least 40 metals developed in our metals laboratory in Reagecon, with more at development or validation stage.

This methodology is regularly used to determine the concentration of divalent cations such as calcium, magnesium, copper, lead, zinc, cadmium, aluminum

## Fields of application

- Environment
  - Total hardness of water ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ).
- Surface treatment
  - Determination of  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Zn}^{2+}$  in plating baths





## 2.5 Precipitation Titrations

This analytical methodology is based on reactions that yield compounds of limited solubility. There is not a very wide range of precipitating agents that can be used gainfully in titrimetry and silver nitrate is by far the most important.

These titrations, (also called argentometric titration) is where silver nitrate is used as the titrant. Silver nitrate can be used for determination of halides (Cl<sup>-</sup>, I<sup>-</sup>, Br<sup>-</sup>) and anions that behave like halides (SCN<sup>-</sup>, CN<sup>-</sup>, CNO<sup>-</sup>). It can also be used for determination of Mercaptans and organic materials that include Fatty Acids.

Indicators typically used for precipitation titrations include sodium chromate, fluorescein and iron (III). A wide range of standardised silver nitrate titrants are available, some of which are standardised to specifically give a one to one equivalence with sodium chloride in various food stuffs.

### Fields of Application

- Environment
  - Determination of chloride in water
- Precious metals
  - Determination of silver
- Pharmaceuticals
  - Titration of halides
- Food and beverage
  - Determination of chloride in many finished products (cooked meats, dairy products, etc.)



**The choice of titrant and coloured indicator and the nature of the base (aqueous or non-aqueous) will be determined by the type of chemical reaction:**

Type of Chemical reaction	Typical Titrants	Typical Coloured Indicators
Acid/Base (Neutralisation)	Hydrochloric Acid, Sulfuric Acid Sodium or Potassium Hydroxide	Phenolphthalein Methyl Orange
Oxidation/Reduction (Redox)	Oxidiser such as Iodine, Potassium Dichromate Reducer such as Sodium Thiosulphate , Oxalic acid	Starch Solution Ferrouin
Complexometry	Complexing agent (EDTA)	Eriochrome Black T Ferrouin
Precipitation (Argentimetry)	Precipitation agent (Silver Nitrate)	Iron III Eosin

**Table 1**

## Applications within Industry

<b>Industries</b>	<b>Applications</b>
<b>Food and Beverages</b>	<ul style="list-style-type: none"> <li>- Salt content (NaCl) in ketchup, soya sauce, cheese, wine, milk.</li> <li>- Peroxide number, saponification number, iodine and acid numbers in fats and oils</li> <li>- Formol number in fruit juices</li> <li>- Calcium in milk products</li> <li>- Calcium in Cornflakes</li> <li>- Vitamin C (Ascorbic acid)</li> <li>- Nitrogen and protein by Kjeldahl method in milk, beer.</li> </ul>
<b>Environmental Testing</b>	<ul style="list-style-type: none"> <li>- COD in water</li> <li>- Chloride in tap water and sewage water</li> <li>- Calcium and magnesium hardness</li> <li>- pH of water</li> <li>- Water Alkalinity</li> <li>- Permanganate index</li> </ul>
<b>Plating-Metal</b>	<ul style="list-style-type: none"> <li>- Determination of copper, zinc, nickel and aluminium</li> <li>- Boric acid and chloride in nickel baths</li> <li>- Alkali in degreasing baths</li> </ul>
<b>Petrochemistry</b>	<ul style="list-style-type: none"> <li>- Acid and base number (TAN and TBN)</li> <li>- Bromine index</li> <li>- Water determination according to Karl Fisher</li> </ul>
<b>Pharmaceutical</b>	<ul style="list-style-type: none"> <li>- Determination of active ingredients in drugs</li> <li>- Content determination of pharmaceutical products with perchloric acid in pure acetic acid</li> <li>- Benzetonium Chloride in drugs</li> </ul>
<b>General chemistry and plastics</b>	<ul style="list-style-type: none"> <li>- Titration of strong acids and alkaline solutions</li> <li>- Epoxy number, isocyanates, acid number, hydroxyl number and saponification number</li> <li>- Amino end groups</li> </ul>

**Table 2**

## Features and Benefits of Reagecon's Titrants & Indicator

<b>Features</b>	<b>Benefits</b>
ISO17025 Accreditation	Guaranteeing Accuracy and traceability
NIST Traceability	Consistency of product and reference to CRM
Ready to Use	Convenience, versatility
Large choice of Titrants (Analytic Volumetric Solutions) and Coloured indicators, Aqueous and Non-aqueous based	All applications covered by one manufacturer Cost- effective (saving on preparation and testing time)
All containers fitted with GL45 neck	Compatible with all brand of auto-titrators
Innovative packaging (Bag in Box 5 and 10L)	Reduced H&S risks and waste disposal cost

**Table 3**

# Density

## 1.0 Theory

Density, also called Mass Density, is a fundamental physical property of a material. It is defined as the mass per unit volume at a specific temperature:

(Density = mass/volume where SI units is kg/m<sup>3</sup> but g/cm<sup>3</sup> or g/ml are widely used)

The density of a material varies with temperature and pressure. This variation is typically small for solids and liquids but much greater for gases. Density is traditionally determined by a manual method (pycnometry) but can also be measured by automated methods (vibrational density meter) or by hydrostatic weighing:

### 1.1 Density measurement by pycnometry, a fundamental measurement technique covered by ASTM D1480 and ASTM D1217:

- The liquid sample is introduced into a calibrated Bingham pycnometer, and allowed to equilibrate to the desired test temperature ( $\pm 0.01^\circ\text{C}$ ) in a circulating bath.
- The volume of liquid is adjusted to mark on pycnometer, then the pycnometer is removed from the bath and allowed to equilibrate to room temperature before weighing.
- The Density of the liquid sample is then calculated based on the weight of liquid in the pycnometer and the calibration factor for the pycnometer (correction is also made for air buoyancy).
- This is an extremely accurate method but also very time consuming

### 1.2 Density measurement by Vibrational density meter Covered by ASTM D4052:

- The liquid sample is injected into an oscillating U-tube through a port on the side of the instrument
- The density is calculated based on the change in frequency of oscillation of tube
- Fast, reliable and accurate method
- Temperature range from  $0^\circ\text{C}$  to  $95^\circ\text{C}$

### 1.3 Density measurement by Hydrostatic weighing:

- This is the method of measuring the density of liquids and solids, based on Archimedes' law
- The density of a sample is determined by weighing it twice—first in air and then in a liquid whose density is known (usually distilled water)
- First weighing determines the object's weight
- Second weighing determines the volume
- More commonly used for solids, but can also be used for liquids



To simplify the comparisons of density across the different systems of units (SI units is kg/m<sup>3</sup> but g/cm<sup>3</sup> or g/ml are widely used), it is sometimes replaced by the dimensionless quantity "specific gravity" or "relative density". Relative density or Specific gravity is the ratio of the density of a substance to the density (mass of the same unit volume) of a reference substance and the reference substance is nearly always water for liquids or air for gases. Therefore for liquids, Relative density or Specific gravity usually means relative density with respect to water and a specific gravity less than one means that the substance floats in water.

(Relative Density = Density of the substance measured / Density of the reference substance)

As Specific gravity is a ratio of densities it is a dimensionless quantity. Specific gravity is the density of a material relative to the density of water, whereas relative density is the density of a material relative to a specified reference material.

Finally, another term specific to American Petroleum Institute Gravity (API Gravity), is a measure of how heavy or light a petroleum liquid is compared to water. If its API gravity is greater than 10, it is lighter and floats on water.

## 2.0 Reagecon offers two ranges of Density Standards

### Reagecon Premium and Quality Ranges

Reagecon Density Standards	Premium Range Specifications	Quality Range Specifications
<b>Manufactured in accordance with</b>	ASTM D1480-12 for testing of Density or Relative Density (specific gravity) by Bingham Pycnometer.	ASTM D4052-09 for testing of Density, Relative Density and API Gravity of Liquids by Digital Density Meter
<b>Values and temperature</b>	<b>0.6960–3.1140g/ml @15°C-30values</b> <b>0.6619–3.1096g/ml @20°C-30values</b> <b>0.6878–3.1043g/ml @25°C-30values</b> <b>0.6752–3.0852g/ml @40°C-30values</b> <b>0.6668–3.0721g/ml @50°C-30values</b> <b>0.6582–1.0478g/ml @60°C-16values</b> <b>0.6407–1.0302g/ml @80°C-16values</b>	<b>0.6960–1.0337g/ml @15°C-15values</b> <b>0.6619–1.0301g/ml @20°C-15values</b> <b>0.6878–1.0265g/ml @25°C-15values</b> <b>0.6752–1.0152g/ml @40°C-15values</b> <b>0.6668–1.0073g/ml @50°C-15values</b> <b>0.6582–0.9990g/ml @60°C-15values</b> <b>0.6407–1.0302g/ml @80°C-16values</b>
<b>Direct applications</b>	Can be used as control and/or calibration standards for density measurement by pycnometric techniques, vibrational or hydrometer based techniques	Can be used as control and/or calibration standards for density measurement by vibrational or hydrometer based techniques
<b>Certification Test method</b>	These standards are tested in accordance with ASTM D1480 with a provisional uncertainty for this test method of ±0.00105 g/ml @ 95% confidence level, this is the value included on Reagecon's ISO9001 certificates for these products.	Reagecon's INAB ISO 17025 accreditation covers density standards from 0.65 – 1.0343 g/ml tested on the vibrational density meter in accordance with ASTM D4052. This test method has an uncertainty of ±0.001642 g/ml @ 95% confidence level.
<b>Packaging</b>	100ml amber glass bottle	100ml amber glass bottle

**Table 1**



### 3.0 Applications of Reagecon's Density Standards

Industries	Applications
<b>Beverages</b>	- Beer* - Beverages* - Spirits* - Wines *
<b>Cosmetics/Personal Care</b>	- Cosmetics - Fragrances - Hair/Skin
<b>Education/Research</b>	- Academic Research - Customs and Excise Authorities - Standardisation and Testing Institutes
<b>Paper/Textiles</b>	- Paper Coatings - Pulp - Textiles
<b>Petroleum Industry</b>	- Asphalt, - Bitumen Gases - Lubricants - Paraffin, Waxes - Crude Oils - Fuel Oils
<b>Pharmaceutical Industry</b>	- Biological Samples - Biotechnology - Forensics - Pharmaceuticals
<b>Power Generation</b>	- Energy Power Generation
<b>Environmental</b>	- Sediments - Sludge's - Soils - Water Potable/Waste
<b>Food Industry</b>	- Food - Sugar
<b>Materials Science/Nanotechnology</b>	- Catalysts - Liquid Crystals - Nano-materials
<b>Minerals/Mining/Raw Materials</b>	- Anti-freezing Agents - Building Materials - Cements - Ceramics - Coal mining - Glasses - Metals
<b>Electronics</b>	- Microelectronics - Semiconductors

**Table 2**

\*Specific Ethanol Density Standards for Calibration of alcoholmeters & Densitometers in oenology- Please check the Reagecon Wine Standards & Reagents catalogue or the Wine Testing section of the Reagecon Physical and Chemical standards Catalogue for specific information.

### 4.0 Features and Benefits of Reagecon's Density Standards

Features & Benefits	
Premium Range	Quality Range
Can be used with any brand or type of density measuring instrument	Can be used with any brand or type of vibrational density measuring instrument
ISO17025 Accreditation	Test results accredited to ISO17025. Uncertainty of measurement (assay procedure) $\pm 0.16\%$ .
No toxic heavy metals used in any formulation	No toxic heavy metals used in any formulation

**Table 3**

# Refractive Index

Author: John J. Barron, Reagecon Diagnostics Ltd.

## 1.0 Theory of Refractive Index

The speed of light travelling through a vacuum is always the same. When light moves through another medium, its speed slows down because the light is being constantly absorbed and re-emitted by atoms within the material through which the light is travelling. The ratio of the speed of light in a vacuum to the speed of light in another substance is defined as the Refractive Index ( $\eta$ ) for that substance. This relationship can be expressed as follows:

Fig. 1: 
$$\text{RI } (\eta) = \frac{\text{speed of light in a vacuum}}{\text{speed of light in substance measured}}$$



### 1.1 How a Refractometer works

When light changes speed as it crosses the boundary from one medium to another, as well as its speed changing, its direction also changes. In other words – it is refractive. The relationship between the speeds of light through the two media, the angles of both incident and refraction and refractive index of the two media is expressed as follows:

Fig. 2: 
$$\frac{V_A}{V_B} = \frac{\sin\theta_A}{\sin\theta_B} = \frac{\eta_B}{\eta_A}$$

Therefore, it is not necessary to measure the speed of light to determine its refractive index. Instead by measuring the angle of refraction and knowing the refractive index of the layer that is in contact with the sample – it is possible to measure the refractive index of the sample. Nearly every instrument on the market utilises this principle, although instruments may vary in optical design.

Samples with different refractive indices will produce different angles of refraction (see Fig. 2), bearing in mind that the angle of incidence and the refractive index of the prism are constant. The different angles of refraction in different samples will be reflected in a change in the position of the border line between the light and dark regions of the instruments. By calibrating the instrument appropriately, the position of the border line can be used to determine the refractive index of any sample.

### 1.2 Effect of Wavelength

In most sample types the speed of light and therefore the refractive index will vary with wavelength. This variation is called dispersion and it is this phenomenon that causes white light moving through a prism to be refracted into the component colours of the rainbow. (Red, Orange, Yellow, Green, Blue, Indigo, Violet). For accurate measurement of refractive index it is necessary to use monochromatic light and the commonest source of light used in refractometers is Sodium D Light at 589 nm (this does not apply to ABBE Refractometers).

### 1.3 Temperature Coefficient of Variation

The speed of light is slower in a sample, than in a vacuum, due to absorption and emission of light by the atoms, as has been already stated. Since the density of a sample normally decreases as temperature increases, the speed of light through the sample will increase. Consequently the refractive index will decrease. For water, this decrease in refractive index will be approximately 0.0001/°C.

For certain other types of sample including liquid organics, the refractive index decrease will be approximately 0.0005/°C. Therefore, the management of temperature is critical for accurate measurement of refractive index. All high quality bench, on line and handheld refractometers where accurate measurement is required are equipped with temperature management systems. Refractive index results should always be reported at the temperature of measurement, which is normally either 20 or 25°C and the temperature of measurement is often denoted with the wavelength of the light used.

## 1.4 Why Measure Refractive Index

Refractive index is measured as part of the characterisation of liquid samples and is analogous to the way the measurement of melting point is used to characterise solid samples. Each substance has its own refractive index and its measurement may be of value in the following situations:

1. To either identify or confirm the identity of a sample by comparing its refractive index to known values. Such known values can be derived experimentally, taken from the technical or scientific literature, or obtained from the manufacturer of the liquid.
2. Refractive index may be used to assess the purity of a sample by comparing the refractive index of the sample to the known value for the pure substance.
3. Refractive index can be expressed in BRIX values for the measurement of the sugar content of sugar rich samples such as jams, jellies, syrups and fruit juices. The BRIX value correlates to the sugar concentration in the sample measured and is expressed as the number of grams of sucrose in 100 grams of sucrose/water solution. The relationship between the refractive index and BRIX is determined by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA). Samples that have multiple ingredients such as salts and sugars (liquid sauces like soya sauce) can be measured for total percentage concentration of these substances using refractive index. The value can be expressed as BRIX or refractive index and the BRIX value can be read and interpreted as the total concentration of dissolved materials in the sample.
4. Refractometers can be used to determine the concentration of aqueous solutions such as oil-based solutions, pharmaceuticals and heavy chemicals (such as machining oil, cleaning solutions, glycols, Cesium Chloride, Sodium Hydroxide and others). Each solution has a correlation value between BRIX or refractive index and the concentration of solute can be established by comparing refractive index or BRIX to known concentrations of the solute using a standard curve. A conversion table can be established from the standard curve and several manufacturers of Refractometers input the conversion tables into the memory of their instruments and thus by measuring their refractive index can display concentration directly. In the market place, up to 80 different instruments are available with dedicated, fit for purpose concentration scales dedicated to specific end user needs and niches.

## 1.5 The ASTM Standards

There are a wide range of ASTM standards dedicated to different applications where Refractive Index is commonly measured. These include the following:

- D542 Index of Refraction of Transparent Organic Plastics
- D1045 Sampling and Testing Plasticizers Used in Plastics
- D1218 Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
- D1416 Rubber from Synthetic Sources – Chemical Analysis
- D1747 Refractive Index of Viscous Materials
- D1807 Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids Use of Refractometer for Field Test Determination of the Freezing Point of Aqueous Engine Coolants
- D4095 Use of the Refractometer for Determining Non-Volatile Matter (Total Solids) in Floor Polishes
- D5006 Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels Rubber from Synthetic Sources – Bound Styrene in SBR (styrene-butadiene Copolymers)
- D5775

## 1.6 Why Use Refractive Index Standards

1. Most workers have focussed on the use of RI standards for the calibration or re-calibration of the Refractometer. Few have focussed on an equally important, but much more widespread use of the standards as a control material for each test carried out in line with the principles of metrology and good laboratory practice. The advent of the 'science of chemical metrology' has given renewed emphasis to the use and necessity of control materials. The benefits of such controls cannot be overstated. As with all analytical standards or reference materials, RI standards should fulfil the following criteria:

- Provide traceability (needed to quote results in SI units)
- Demonstrate the accuracy of results
- Calibrate the equipment and methodology
- Monitor the user performance
- Validate the test
- Facilitate comparability, which is to ensure that when the correct procedures have been followed, the same analysis of the same materials will produce results that agree with each other whenever or wherever they are performed.

Such materials must also be able to fulfil the criteria required for quality control, accreditation and proficiency testing where appropriate (Barron, 1996). Standard and reference materials should be produced and characterised in a technically competent manner, should be homogenous, stable, certified and have available a known uncertainty of measurement as a function of time (Barron and Papacosta, 1997).

2. Production of primary standards rarely focuses on measurement uncertainty as a function of time that is realistic in the context of the production, transport and use of such standards under commercial or routine conditions. Such standards are rarely suitable for use as secondary or working standards.

3. Secondary or working standards need to be affordable, widely available and certified. The development, production, stability, assignation of uncertainty as a function of time and commercialisation of an extensive range of fit for purpose standards that includes refractive index standards has occupied the time and resources of the author's laboratory for several years.

4. In the context of normal commercial or routine use, RI standards (or any other standards) need to be rugged, have extensive shelf life, and be fit for purpose.

## 2.0 Technical Information:

### 2.1 Types of Brix Standards from Reagecon

Reagecon Brix Standards	Brix Standards ICUMSA* Compliant	Brix Standards Non ICUMSA Compliant*
<b>Manufactured in accordance with</b>	ICUMSA And ISO Guide 34 guidelines	ISO Guide 34 guidelines
<b>Values and temperature</b>	0, 5, 7, 10, 11.2, 11.5, 12, 12.5, 15, 20, 25, 30, 35, 40, 45, 50, 55 and 60% Brix @20°C * 14.9, 19.4 and 23.8% Brix @20°C also available	0, 5, 7, 10, 11.2, 11.5, 12, 12.5, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60 and 67.5% Brix @20°C
<b>Packaging</b>	Presented in 1x15ml dropper bottles Customised pack options available	Presented in 1x15ml dropper bottles Customised pack options available

\* ICUMSA- International Commission for Uniform Methods of Sugar Analysis. The guidelines issued by ICUMSA state no preservatives can be added to Brix standards so as a result such standards have a shorter shelf-life.



## 2.2 Types of Refractive Index Standards

Reagecon Refractive Index Standards	Refractive Index Sucrose based	Refractive Index Solvent based
Values and temperature	Range covers 1.34325 to 1.44193 <sub>nD</sub> @ 20°C -16 values	Range covers 1.38779 to 1.65808 <sub>nD</sub> @ 20°C -10 values
Certification Test method	The R.I. value of the standard is verified using a high performance calibrated, temperature controlled refractometer. The control of this instrument is completed using high purity ISO Guide 34 accredited Brix standards.	The R.I. value of the standard is verified using a high performance calibrated, temperature controlled refractometer. The control of this instrument is completed using high purity ISO Guide 34 accredited Brix standards.
Packaging	Presented in 1x15ml and 6x15ml dropper bottles Customised pack options available	Presented in 1x15ml and 6x15ml dropper bottles Customised pack options available

## 3.0 Applications and type of Industries of Reagecon's Refractive Index & Brix Standards

Industries	Applications
Food and Beverages	<ul style="list-style-type: none"> <li>- Coffee extract</li> <li>- Soy Bean Oils</li> <li>- Milk</li> <li>- Wine</li> <li>- Juices</li> <li>- Jam</li> <li>- Spirits</li> </ul>
Sugar Milling, Refining, Processing	<ul style="list-style-type: none"> <li>- Molasses</li> <li>- Liquid Sugar</li> <li>- Cane Sugar milling and refining</li> <li>- Beetroot sugar milling and refining</li> </ul>
Water Based Chemicals	<ul style="list-style-type: none"> <li>- Gels</li> <li>- Glycols</li> <li>- Resins</li> <li>- Polymers</li> </ul>
Petroleum Based Chemicals	<ul style="list-style-type: none"> <li>- Plastics</li> <li>- Solvents</li> <li>- Oil based paints</li> <li>- Resins</li> </ul>
Petroleum Industry	<ul style="list-style-type: none"> <li>- Additives</li> <li>- Lubricants</li> <li>- Fuel Oils</li> <li>- Paraffin, Waxes</li> </ul>
Pharmaceutical Industry	<ul style="list-style-type: none"> <li>- Toxicology testing</li> <li>- Pharmacy compounding</li> <li>- Drug Testing</li> <li>- EP and USP test methods</li> </ul>
Flavour, Fragrance and Cosmetic	<ul style="list-style-type: none"> <li>- Perfumes</li> <li>- Lemon Oil</li> <li>- Waxes</li> <li>- Creams</li> </ul>

## 4.0 Features and Benefits of Reagecon's Refractive Index & Brix Standards

Range	Features & Benefits			
	Brix ICUMSA	Brix Non ICUMSA	Refractive Index Sugar Based	Refractive Index Solvent Based
Accreditations	ISO 17025 for the whole range. ISO Guide 34 for Sucrose in water (% Brix)	ISO 17025 up to 60% Brix	ISO 17025 for the whole range	ISO17025 up to 1.44193 <sub>nD</sub>
Shelf-life	12 weeks (6 weeks from competitors)	12 months	12 months	24 months
Traceability	Traceable to International Standards			
Applications	Suitable for use on any brand and type of refractometer			

# Reagecon

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