

Edition 6



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John Barron, MD, giving a tour of the plant during our annual seminar.



Vicky Howard, HR Manager and Michelle Hynes, VMI Manager accepting the OHSAS 18001 Certificate on behalf of the team at Reagecon.



Leo Sun, Sales Executive Asia Pacific pictured at our booth at the BCEIA held in China.

Welcome to the latest edition of the Metrologist.

From a New Product Development (NPD) perspective, 2017 was a very successful year for Reagecon. At our annual conference, in Shannon, in October, we launched three major new product ranges, all of which are covered in this edition of The Metrologist.

The first of these families, X Ray Fluorescence (XRF) Standards, represent the start of a significant R&D programme on XRF, within the company. The range launched – XRF Environmental Standards in Water, will be followed by several non-aqueous and petrochemical ranges shortly, including sulphur (S) and lead (Pb) in various matrices. These will be followed by a range of metal standards in lubricants. The theory and practice of XRF was presented at the conference in detail and a brief synopsis is presented in the following pages.

Reagecon has been producing reagents and solvent mixtures for DNA synthesis and sequencing for many years on a private label basis. We are now in a position to produce these products under the Reagecon label and also produce other similar products on a customised basis. Our facilities for manufacturing these products are extensive as can be seen in the photographs on pages 15 - 19. We are very excited about the prospects of utilising these valuable tools on an even more extensive scale, so we would very much welcome your feedback on possible opportunities to utilise this plant. Standards and reagents for Process Analysers utilise several technologies. These include the use of:

- Colourimetic/spectrophotometric analysis
- Electrochemical measurement
- Titration
- Ion chromatography
- TOC/TIC analysers

Irrespective of the technology used, the measurement is done in-situ, rather than on-line and an extensive range of reagents and standards are used. All of the standards must comply with basic metrological principles, including traceability, accuracy, precision, fitness for purpose and the use of the standards for calibration, quality control, method validation and instrument qualification. An extensive range of these new products from Reagecon, launched at the annual conference, is presented on pages 21 and 22.

The scientific and technical literature output by Reagecon, continues to grow. Next year will see the launch of our new textbook: Spectroscopic and Electrochemical Measurement of Cations and Anions; Metrology, Calibration and Control. The content of this publication, the rationale for selecting the content, the benefits of the content to you, the reader and the metrological relevance of the content, are all covered on pages 4 - 9, of this publication. We look forward to the publication date and believe the contents will bring significant value, to your analytical measurement.

Over the years, Reagecon has presented many scientific papers, two of them, The Effect of Temperature on pH Measurement and The Effect of Temperature on Conductivity Measurement are frequently requested and several of our readers have requested, that we reproduce them here. Although both of these papers were published over a decade ago, their relevance to modern measurement is as strong as ever.

Overall, it has been an excellent year for the company. The success we continue to enjoy could not be achieved without two key elements – our excellent and dedicated staff and you, our customers. On behalf of all our staff, I would like to thank you for your help and support, in 2017. Of course, 2018 is a big exhibition year, with Analytica and Achema occurring in Germany, Arablab in Dubai and Analytica China in Shanghai. We will have a substantial booth at all of these shows and look forward with excitement to 2018 and in particular to seeing you, at one of these shows.

Once again, thanks for all your help and support.

John J Barron Editor Spectroscopic and Electrochemical Measurement of Cations and Anions; Metrology, Calibration and Control.

1.0 Introduction

The necessity to measure cations and anions continues to grow in importance. Environmental considerations are growth drivers in all industries and regulatory changes are of growing importance in several industries, with recent changes in the pharmaceutical industry mandating that such measurement is an imperative, as an example. The sensitivity and versatility of the test methologies continues to expand, therefore the accuracy levels of detection and chemical purity of the standard and calibration materials continues to be more demanding.

However, it must be remembered that as the use of newer technologies grows so does the use of existing or older technologies continue to grow. In parellel, standards, reagents and calibration materials for older technologies are also growing in importance. Ultimately, all analytical techniques are about fitness for purpose. Therefore, less demanding, sporadic or occasional measurements often require a less sophisticated or less expensive piece of scientific apparatus.



We are in the final stages of producing a new textbook that covers in a simple and basic manner most of the electrochemical and spectroscopic techniques forcation and anion analysis, with detailed information on the Metrology, Calibration and Control of the various methods. The methods being covered are:

- ICP/ICP-MS
- Atomic Absorption (AA)
- Flame Photometry (FP)
- Ion Chromatography (IC)
- X Ray Fluorescence (XRF)
- Process Analyser Standards and Reagents (ISE, Colourimetric Methods)
- Ion Selective Electrodes (ISE's)

2.0 Objectives

The textbook has several technical and commercial objectives:

- 1. Demonstrates our competence and commitment to Standards and Reference Materials
- 2. Gathers into one publication all of the New Product Development (NPD) that is either already produced or will be available soon, published material, accreditations and other technical activities from within Reagecon.
- 3. Forms a companion for the Physical and Chemical Standards Compendium
- 4. Identifies applications of technologies, methods and standards that you may not know about.
- 5. Demonstrates and identifies the technical options available to you.
- 6. Shows the full span of all applications of standards not just calibration
- 7. Shows the very rigorous and detailed regime that goes into the production of standards and the price justification
- 8. Gives you a knowledge platform to answer more of your technical queries yourself, as they arise.



3.0 Core Contents

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The major contents of the textbook will focus on the following subject areas, all of which are vital to the successful use of the techniques, technologies and methodologies covered. An outline of this content is as follows:

- The hierarchy of standards
- Theory and fundamentals of each technique
- Comparative sensitivities, accuracy, cost, throughput and fitness for purpose
- Direct/indirect measurement
 - o Standard addition
 - o Standard subtraction
 - o Sample addition
 - o Sample subtraction
 - Classical applications of the various methods
- General principles of sampling (non method specific)
- Matrix effects in spectroscopic measurement
- Problem elements in ICP/ICP-MS analysis
- Internal standardisation, isotope dilutions in spectroscopy
- Multi-element chemical standards element compatibility
- The periodic table as applied to anion and cation analysis (Group 1 Group 16)
- Application of GLP in the selection and use of standards for accurate, traceable ionic and elemental concentration measurements
- Care, maintenance and fault diagnosis of ion selective electrodes (ISE's)
- Effects of temperature on ISE measurement
- The role of accreditations in the production and use of standards and reference materials
- Comparison between the various accreditations and certifications and the accreditation landscape within Reagecon.



4.0 The Principle Uses of Standards and Reference Materials

Substantial detail will be covered on the application and benefits of standards:

- Instrument calibration almost all analytical chemistry is comparative
- Reference Material or Standard as a calibrant also establishes metrological traceability, comparability comparable in time and place
- Control Material the use of a Reference Material or Standard is a very effective and an almost universally mandatory means of Quality Controlling an unknown test result, provided matrix, homogeneity and stability are taken into account.
- The Reference Material or Standard enables metrological data pertaining to accuracy, precision, reproducibility, bias and uncertainty of measurement.
- Instrument Qualification (IQ, OQ, PQ, MQ)
 - The preserve of the Pharmaceutical Industry
 - o This is seen in several industries as good laboratory practice
- Proficiency Testing
 - Here the test results of the Reference Materials or Standards are compared with the results of a statistically significant number of other laboratories
 - o A mandatory component of method validation
 - o A mandatory component of the requirements of accreditation bodies for a laboratory
- Analyst Qualification
 - In this instance the laboratory results are not compared with those of other laboratories
 - Compared with the analytical value obtained by the producer of the reference materials or standard
 - o Values withheld from the analyst until after the test is performed
 - Objective view of the level of training and competence of the analyst to perform a particular test



5.0 Mature Technologies

Because certain techniques, have been to some extent superseded by modern technologies, it does not mean such technologies are no longer relevant. In fact, the converse is the case, older technologies that are fit for purpose and fit for a particular requirement or price bracket are more relevant than ever.



For example, the use of ISE's has to some extent been superseded by the emergence and popularity of IC. However, ISE's have a new and exciting role in on-line measurement, but the use of ISE's in laboratories also continues to grow, judging by the increasing popularity of Reagecon's ISE Standards and Ionic Strength Adjustor's (ISA's).



In that context we believe it is prudent to revisit this old technology and therefore the following areas relating to ISE's in the laboratory will be covered in detail:

- Care and maintenance of ISE's
- Electrode faults
- Fault diagnosis and solutions
- Applications
- Specifications

In a similar vein the use of Flame Photometry has been superseded by several other techniques such as AA, ICP and IC to name but a few. In the textbook, we will cover Applications, Standards and Calibrators available and the features and benefits of such standards for Flame Photometry.

6.0 The Accreditation Landscape in Reagecon

The Accreditation of standards and reference materials is now a major component of Metrology. We will cover the benefits of Accreditation to the user, what to expect from a standards producer that is accredited, the scope of ISO 17025 and ISO Guide 34 Accreditations. The relationship between the hierarchy of standards and such accreditations is presented in Diagram 1 below.



Diagram 1 – Accreditation Landscape Within Reagecon and Its Relationship to the Hierarchy of Standards

7.0 Conclusion

Detailed information on many aspects of ICP/ICP-MS, IC, AA and XRF will be provided, with particular emphasis in all cases on the features, benefits, specifications, selection and use of standards and the metrology applicable to these materials. Although the measurement of cations and anions by colourimetric measurement is too broad a subject area, and outside the scope of this text, colourimetric measurement in the narrow context of on-line analysers will be covered, with special emphasis on the metrology of the standards.

It is our view that there is significant need for a textbook such as described above and such a book is long overdue. We look forward to its publication and we hope once published, it will bring significant value to the quality of your analytical results for cations and anions.



1.0 Introduction

Reagecon announced the launch of three new families of products at our recent International Conference in Shannon. In addition to XRF Standards, which will be covered in these pages, we also launched extensive ranges of on-line analyser Standards and Reagents and a range of DNA synthesis and sequencing reagents (both of these families are covered in detail elsewhere in this edition of The Metrologist)

2.0 Theory Of How XRF Works

This is a very valuable and versatile technology, whereby a spectrometer generates a primary x ray beam (energy source). This beam excites cations within the sample, by the absorption of energy. Then, the cations emit secondary fluorescent x rays which are element specific and quantitative in that the intensity is dependent on the quantity of element in the sample, which is then measured by a detector. A simplified diagram detailing the process is presented in Diagram 1.





There are two main types of XRF

- Energy Dispersive XRF
- Wavelength Dispersive XRF

Energy Dispersive XRF (EDXRF): works on the basis that all fluorescence from the sample is captured simultaneously and the detector "sorts" the response from different elements.

Wavelength Dispersive (WDXRF): fluorescence from the sample is diffracted by an analysing crystal and individual elements are measured sequentially.

The main difference between the two is sensitivity and detection limits. Wavelength dispersive XRF is more sensitive and has lower detection limits compared to Energy dispersive XRF (see Table 1).

Irrespective of the type of XRF used most systems are capable to a greater or lesser extent of measuring elements right through the periodic table from Sodium (Na) to Uranium (U). XRF is a particularly valuable analytical test in that it can measure levels of cations present in samples, in either solid or liquid form, with minimum preparation in all cases.

Technique	Typical S limit of reporting	Advantages/disadvantages
ED-XRF (direct)	5-50 ppm (depending on specification	Cheap, simple, can analyse wide range of other elements.
		Relatively high limits of detection.
ED-XRF	2 ppm	Can analyse wide range of other elements.
(polarised)		Operating close to performance limits of some materials.
WD-XRF	0.5-2 ppm (depending on system	Good limits of detection. Most systems can analyse a wide range of elements.
		Complex, potentially expensive.

Comparative Features and Benefits of the various types of XRF

Table 1

3.0 Sample Applications of XRF

A significant application of XRF is the measurement of sulphur (S) in the petrochemical industry. This element undergoes the following reaction, with the ultimate production of sulphuric acid (H_2SO_4) , which is toxic, corrosive and environmentally harmful.

 $S + O_2$ SO₂ (sulphur dioxide)

 $2SO_2 + H_2O + O_2$ $2H_2SO_4$ (sulphuric acid)

Rigaku WD-XRF in Reagecon



There is a substantial body of legislation that governs the permissible levels of sulphur, allowable in various fuels around the world. Some examples are presented in Table 2 below. Unless stated, the data applies to regulations within the EU.

Examples of current legislation for Sulphur (S) in fuels:

Fuel Type	*Max Permissible Sulphur
Road fuels (retail petrol and diesel)	10 ppm
Non Road fuels (e.g. gas oil for heavy plant vehicles)	10 ppm
Gas oil for static systems (e.g. domestic heating/small generators)	0.1%
Heavy fuel for static systems (e.g. large industrial generators/furnaces)	1.0%
Marine fuel	Global: 3.5%
	European "ECAs": 0.1%
Tabl	e 2

ECAs = emission control areas (ports)

*Data available from the European Commission website

There is also a requirement to measure wear and tear metals in various types of lubricating oils. XRF is an ideal technology for either the measurement of sulphur (S) or metals in oils (organometallic compounds) and typical limits are presented in Table 3.

Element	Mg/kg (3sigma)	Element	Mg/kg (3sigma)
Chlorine	1.0	Copper	0.4
Vanadium	0.5	Mercury	0.4
Chromium	0.2	Cobalt	0.7
Lead	0.3	Arsenic	0.2
Thallium	0.4	Zinc	0.4
Cadmium	0.5	Nickel	0.4
Antimony	0.9	Manganese	1.6

Typical Limits of Detection of Metals in Oil

Table 3

Reagecon is currently embarking on a significant R&D programme to develop standards and reference materials for all of these applications. Already we have launched a small range of cations in an aqueous matrix, which are presented in Table 4. Additional products will be developed and presented to the market throughout 2018.

Code	Description	Pack Size
REXAL101	Aluminium 1000ppm	100ml
REXSB101	Antimony 1000ppm	100ml
REXBA101	Barium 1000ppm	100ml
REXB101	Boron 1000ppm	100ml
REXCA101	Calcium 1000ppm	100ml
REXCS101	Cesium 1000ppm	100ml
REXCU101	Copper 1000ppm	100ml
REXFE101	Iron 1000ppm	100ml
REXPB101	Lead 1000ppm	100ml
REXLI101	Lithium 1000ppm	100ml
REXMG101	Magnesium 1000ppm	100ml
REXMN101	Manganese 1000ppm	100ml
REXMO101	Molybdenum 1000ppm	100ml
REXP101	Phosphorus 1000ppm	100ml
REXK101	Potassium 1000ppm	100ml
REXSI101	Silicon 1000ppm	100ml
REXNA101	Sodium 1000ppm	100ml
REXSR101	Strontium 1000ppm	100ml
REXS101	Sulphur 1000ppm	100ml
REXTI101	Titanium 1000ppm	100ml
REXW101	Tungsten 1000ppm	100ml

XRF Environmental Standards in Water

Table 4

Reagents and Solvents for DNA Synthesis and Oligonucleotide Manufacturers

The demand from companies involved with Pharmaceutical Research, Genomics and Oligonucleotide Production for Reagents and Solvents of the highest purity, lowest moisture content and batch to batch consistency remains undiminished.



Reagecon recognises the industry demands for increased yields and increased efficiency. To help meet this demand, Reagecon have invested in a dedicated production suite, specifically for the multiple distillation, processing and mixing of a comprehensive range of solvents and reagents.



All products are packaged in a variety of formats and presented, ready for use on the most popular laboratory and commercial scale synthesisers for DNA synthesis and Oligonucleotide manufacturing. The products are shipped to customers, worldwide in full compliance with all international regulations pertaining to the documentation required and different modes of transport.





Additionally the Reagecon range of specialist Solvents and Reagents offer:

- Low acidic and Ultra Violet (UV) adsorbing impurities
- High compatibility with Fmoc and similar protection chemistries
- Reduction of secondary aggregates
- Comprehensive Certificate of Analysis with each batch of product





Solvents and Reagents for DNA Synthesis and Oligonucleodtide Manufacturing From Reagecon

Code	Description	Pack Size
REI2002ONS01	Oxidiser solution 0.02M for Oligo Synthesis	100ml
REI2002ONSW	Oxidiser solution 0.02M for Oligo Synthesis	2.5L
RE12002ONS05	Oxidiser solution 0.02M for Oligo Synthesis	500ml
REAANPTNS018	Acetic Anhydride Pryidine THF Capping Solution A	100ml
REAANPTNS05	Acetic Anhydride Pryidine THF Capping Solution A	500ml
REACNONS004	Acetonitrile, Anhydrous for Oligo Synthesis	100ml
REACNONS01	Acetonitrile, Anhydrous for Oligo Synthesis	100ml
REAMTDIL01	Amitide Diluent Anhydrous Acetonitrile	100ml
REDCMONSW	Dichloromethane for Oligo Synthesis	2.5L
REDCMONS4	Dichloromethane for Oligo Synthesis	4L
REKCNPYPSM2	Potassium Cyanide Pyridine Solution M2 for Peptide Synthesis	20ml
REPHETPSM1	Phenol in Ethanol Solution M1 for Peptide Synthesis	100ml
REDIENMPS02	Diisopropyl N-methylpyrrolidone 2M for Peptide Synthesis	250ml
REMETPS045	Methanol for Peptide Synthesis	500ml
RENINETPSM3	Ninhydrin in Ethanol Solution M3 for Peptide Synthesis	20ml
RENMTFONS2	1-Methylimidazole THF Capping Solution B	2.5L
RENMTFONS045	1-Methylimidazole THF Capping Solution B	500ml
RETFAONS05	Trifluoroacetic acid for Oligo Synthesis	500ml
RETFAONSW	Trifluoroacetic acid for Oligo Synthesis	2.5L
RETCDCMONS2	Trichloroacetic acid Dichloromethane Solution	2.5L
RETCDCMONS045	Trichloroacetic acid Dichloromethane Solution	500ml
RETCDCMONS4	TCA DCM Deblock Solution	4L
RETTDACONS018	TETD Acetonitrile Sulphurising Solution	500ml
RETETACONS2	Tetrazole Acetonitrile Phosphoramidite Diluent	2.5L
RETETACONS045	Tetrazole Acetonitrile Solution for Oligo Synthesis	500ml
RETETACONS018	Tetrazole Acetonitrile Oligo Activator Solution	100ml
RETFTEA201	Trifluoroacetic acid Triethylamine 2M solution	100ml
RETEAA205	Triethylamine Acetate 2M	500ml
REDIMAPS1	Dimethyl Acetamide for Peptide Synthesis	1L
REDMFPS2	N, N Dimethylformamide for Peptide Synthesis	2.5L
REACNTHFPS	Acetonitrile Tetrahydrofurn for Peptide Synthesis	500ml
REFACN0105	Formic acid 0.1% in Acetonitrile	500ml
NH1P22001	Ammonia solution 2M in 1-Propanol	1L
NH1P22005	Ammonia solution 2M in 1-Propanol	5L
REAMETH2J	Ammonia solution 2M in Ethanol	100ml
REAIPA2F	Ammonia solution 2M in Isopropanol	1L
REAIPA2J	Ammonia solution 2M in Isopropanol	100ml
REAM2F	Ammonia solution 2M in Methanol	1L
REAM2J	Ammonia solution 2M in Methanol	100ml
REAMETH2F	Ammonia solution 2M in Ethanol	1L
REAMM7J	Ammonia solution 7N in Methanol	100ml

During 2017, Reagecon has upgraded its facilities significantly, so that in addition to reagents and solvents for oligonucleotide synthesis and sequencing, we have developed capability for solvent distillation, purification, drying and mixing. This capability operates from laboratory scale (1 litre reactors) up to 400 litre glass or glass lined vessels.

The laboratory is:

- Ex-rated
- Fully bunded and fire proofed
- Has 25 air exchanges/hour
- Contains glass vessels and reactors from 1 litre to 400 litres
- Several of the vessels are temperature controlled (-40°C 250°C)
- Is positioned beside an ex-rated, fully bunded solvent warehouse

The extent of the capability can be seen in the suite of recently taken photographs printed in the following pages.



As a result of this expansion, Reagecon expanded its private label manufacturing capability and we will be very happy to discuss any customised requirements that you may have for such solvents in either pure form or presented as solvent mixtures.











Standards and Reagents for Process Analysers

Standards and Reagents for Process Analysers

As analysis increasingly transfers from being laboratory-based to being process-based then the demands on the quality of process analytical measurements is increasing. All Process Analyser sample measurements are stated as a numerical analyte concentration and a concentration unit. It is only valid to quote the relevant measurement unit if there is a direct link to its definition, which can only be established through the use of Standards with metrological traceability. Reagecon ensures that this requirement is satisfied by rigorous analysis of all of its Process Analyser Calibration and Control Standards to provide metrological traceability to the SI units and to assign their certified values.

Reagecon's Quality Control for Process Analysers Reagents is performed using appropriate techniques from our extensive analytical capabilities to ensure that every batch of each individual reagent is within the customer agreed specification. Reagecon also perform Quality Control on the entire set of reagents and standards as a kit for a process analyser by:

- Testing the reagent-set's performance on the target analyser.
- Testing the reagent-set's performance by simulating its use on the target analyser using appropriate laboratory instrumentation.

Reagecon manufacturing capabilities are aligned with the complete set of chemicals required for operation of Process Analysers, including the following:

•Colourimetric / Spectrophotometric Analysers Reagents Calibrations Standards Cleaning Solutions Analyser Qualification Sets

•Ion Selective Electrode & pH Analysers

Calibration Standards Ionic Strength Adjuster (ISA) Reagents Electrolytes Electrode Cleaning, Care & Maintenance Solutions Analyser Qualification Sets

•Ion Chromatography Analysers

Eluents (Concentrates & Ready to Use) Calibration Standards Analyser Qualification Sets •Direct Measurement Analysers (Conductivity, ORP (Redox), Suspended Solids, Turbidity) Calibration Standards Cleaning Solutions Analyser Qualification Sets

•Titrimetric Analysers

Titrants Titration Solvents Electrode Calibration Standards Electrolytes & Electrode Cleaning Solutions Analyser Qualification Sets

•TOC / TIC Analysers

Acidification Reagents Oxidation Reagents Calibration Standards Analyser Qualification Sets

Reagents and Standards for Applitek Process Analysers From Reagecon

Code	Description	Pack Size
REA3MPSILST025	Envirolyser	2.5L
REA3MPSILOA	Oxalic Acid Solution for 3MP Silica Envirolyser	2.51
REA3MPSILRR	Reducing Reagent Solution for 3MP Silica Envirolyser	2.51
REA3MPSILSTO	Envirolyser	500ml
REA3MPSILST01	Envirolyser	500ml
REA3MPSILST0125	Envirolyser	2.5L
REA3MPSILCLS	Cleaning Solution for 3MP Silica Envirolyser	10L
REA3MPSILCLS5	Cleaning Solution for 3MP Silica Envirolyser	5L
REA3MPSILCS	Colour Solution for 3MP Silica Envirolyser	2.51
REA3MPSULB5	Sulphate Buffer Solution for Envirolyser	51
REA3MPSULSTO	Sulphate Calibration Standard Omg/I for Envirolyser	500ml
REA3MPSULST4	Sulphate Calibration Standard 4000mg/l for Envirolyser	500ml
REAP2MPHLOH	Phenol Calibration Standard Omg/l ref 1 for Envirolyser	500ml
REAP2MPHL1H	Phenol Calibration Standard 1000mg/l for Envirolyser	500ml
REAP2MPHL2H	Phenol Calibration Standard 2mg/l ref 2 for Envirolyser	500ml
REAPAM2MPRAW	Reagent A for Envirolyser 2MP Ammonia-Berthelot	2.5L
REAPAM2MPRBW	Reagent B for Envirolyser 2MP Ammonia-Berthelot	2.5L
REAPCOLPO4U	Phosphate colour reagent for Anaylser	2.5L
REAPE2MPB325	TISABIV Buffer3 for Envirolyser 2MP Fluoride	2.5L
REA3MPBACL5	0.1M Barium Chloride Solution for Envirolyser	51
REAP2MPBS5	Buffer Solution for Envirolyser	51
REAP2MPCLS	Cleaning Solution for Envirolyser	10
REAP2MPCSW	Colour Solution for Envirolyser	2.51
REAPE3MPCREF1	3MP	1L
REAPE3MPCREF2	3MP	1L
REAPE3MPFEB1	Total Iron Buffer Solution for Envirolyser Colorimetric 3MP	1L
REAPE3MPFECOL	Total Iron Colour Solution for Envirolyser Colorimetric 3MP	1L
REAPE3MPFERD1	Total Iron Reductor Solution for Envirolyser Colorimetric 3MP	1L

Reagents and Standards for Aztec and Navigator Process Analysers From Reagecon

Code	Description	Pack Size
REZ620NHS	Aztec AAM631 ISE Ammonia Monitor Reagent Set	kit
REZ600FS	Aztec 600 Fluoride ISE Reagent Set	kit
REZ600ALS	Aztec 600 Aluminium Reagent Set	kit
REZ721R15	Aztec 600 Aluminium Acid Reagent R1	5L
REZ722R25	Aztec 600 Aluminium Buffer Reagent R2	5L
REZ723R35	Aztec 600 Aluminium Colour Reagent R3	5L
REZ724CS25	Aztec 600 Aluminium Calibration Standard 0.2ppm	2.5L
REZ001R15	Aztec 600 Ammonia Salicylate Reagent R1	5L
REZ002R25	Aztec 600 Ammonia Nitroprusside Reagent R2	5L
REZ003R35	Aztec 600 Ammonia D.I.C. Reagent R3	5L
REZ004CS25	Aztec 600 Ammonia Calibration Standard 0.288 mg/l NH3- N	2.5L
REZ600AMS	Aztec 600 Ammonia Colorimetric Reagent Set	kit
REZ600FES	Aztec 600 Iron Reagent Set	kit
REZ620R15	Aztec 600 Iron Acid Reagent R1	5L
REZ621R25	Aztec 600 Iron Buffer Reagent R2	5L
REZ622R35	Aztec 600 Iron Colour Reagent R3	5L
REZ623CS25	Aztec 600 Iron Standard 0.2ppm as Fe	2.5L
REZ001LR15	Aztec 600 Low Range Manganese Reagent 1	5L
REZ002LR25	Aztec 600 Low Range Manganese Reagent 2	5L
REZ003LR35	Aztec 600 Low Range Manganese Reagent 3	5L
REZ600LMNS	Aztec 600 Manganese Reagent Set - Low Range	kit
REZ100LCS25	Aztec 600 Low Range Manganese Reagent 0.1 mg/l	2.5L
REZ001HR15	Aztec 600 Manganese High Range Reagent 1	5L
REZ002HR25	Aztec 600 Manganese High Range Reagent 2	5L
REZ003HR35	Aztec 600 Manganese High Range Reagent 3	5L
REZ0500HCS25	Aztec 600 High Range Manganese Standard–0.50 mg/l/l	2.5L
REZ600HMNS	Aztec 600 Manganese Reagent Set – High Range	kit
REZ001PR15	Aztec 600 Phosphate Reagent 1	5L
REZ002PR25	Aztec 600 Phosphate Reagent 2	5L
REZ600PS	Aztec 600 Phosphate Reagent Set	kit
REZ1000PCS25	Aztec 600 Phosphate Standard Solution - 1.0ppm as P	2.5L
REN600SI200S	Navigator 600 Silica Reagent Set	kit
REN600PSIS	Navigator 600 Silica with Phosphate Reagent Set	kit
REN600SI50S	Navigator 600 Silica Reagent Set	kit
REN60050PSIS	Navigator 600 Silica Reagent Set – phosphate present in sample	kit
REN600PS	Navigator 600 Phosphate Reagent Set	kit
REN600LPS	Navigator 600 Phosphate Reagent Set 10mg/L	kit
REN0006SICS	Navigator 600 Silica Cleaning Solution Pack of 6	6 x 500ml
REN600SIS	Navigator 600 Silica Reagent Set	kit
REN000BP05	Navigator 600 Phosphate Zero Solution	500ml
REN004A25	A: Navigator 600 Reduction Reagent (Brown Channel)	2.5L
REN004B	B: Navigator 600 Reduction Reagent	50g
REN1196XCS05	Navigator 600 Monitor Extra Strength Cleaning Solution	500ml
REN1195RCS05	Navigator 600 Monitor Regular Cleaning Solution	500ml
REN001PMO25	Navigator 600 Phosphate Molybdate Reagent (Violet Channel)	2.5L
REN010CS05	Navigator 600 Phosphate Standard 10ppm as PO4	500ml

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⁽¹⁾.

In addition, changes in temperature will also have an influence on the measuring senosr 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⁽²⁾:

$$E = E^0 - 2.3 (RT/nF) \log a_H^+$$

where:

- E = total potential (in mV) developed between the sensing and reference electrode
- Eo = standard potential of the electrode at $a_{H}^{+} = 1 \text{ mol/l}$
- R = Gas constant
- T = temperature
- n = valency of ion

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

- F = Faraday constant
- $a_{\rm H}^{+}$ = 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⁽³⁾. 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.

2.2 Calibration Isothermal Point

The isothermal point of an electrode is defined as the intersection point of calibration lines plotted for different temperatures⁽⁴⁾ 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⁽²⁾. For real electrodes the individual potentials which contribute to the potential overall 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 temperate 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⁽⁶⁾. 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.



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

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⁽⁷⁾. 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 (m Ω) at 25°C would increase to over 1000 m Ω at 10°C⁽⁸⁾.

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 $100 - 200 \text{ m}\Omega$ 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⁽⁹⁾.

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)⁽¹¹⁾. 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 \pm 0.01 @ 25°C ⁽¹⁰⁾	pH 7.00 \pm 0.01 @ 25°C° ⁽¹⁰⁾	pH 10.00 \pm 0.01 @ 25°C° ⁽¹⁰⁾
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.

		Temperature	
pH Range	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

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.

5 References

- Steven S. Zumdahl, Chemistry 3rd Edition. D.C. Heath & Co., 1993, p645.
- Helmuth Galster, "pH Measurement: Fundamentals, Methods, Applications, Instrumentation". VCH Publishers Inc., 1991, p21.
- 3. Ursula Tinner. Electrodes in Potentiometry Metrohm Monographs, p2.
- 4. Principles and problems of pH measurement. . Ingold Messtechnik AG, 1980, p22.
- Helmuth Galster, "pH Measurement: Fundamentals, Methods, Applications, Instrumentation". VCH Publishers Inc., 1991, p159
- 6. Guide to pH measurement. Mettler Toledo, 1997, p10.
- 7. Orion US pH Electrode Catalog and Guide to pH Measurement.1986, p3.
- Stephen Russell. pH A guide to measurement in water applications. WRc Instrument Handbook, 1994, p13.
- 9. Laboratory Electrodes Catalogue. Schott Instruments GmbH, 2002, p6.
- 10. Reagecon Product labels, Buffer Solution pH4.00 \pm 0.01 @ 25°C (Product No. 1040), Buffer Solution pH 7.00 \pm 0.01 @ 25°C (Product No. 1070), Buffer Solution pH 10.00 \pm 0.01 @ 25°C (Product No. 110025)
- 11. Operating manual for CG843P pH meter. Schott Instruments GmbH, 1999, ba12244e_2, p85.

Biographical Notes:

John J Barron is Managing and Technical Director of Reagecon Diagnostics Limited. The company, which was founded in 1986, is the largest producer worldwide of Conductivity Standards and is also a major producer of other chemical standards. Mr. Barron is an expert in several areas of analytical chemistry, including electrochemical analysis, good laboratory practice (GLP) and chemical metrology. He has written and lectured extensively and is credited with several scientific discoveries including stable low level conductivity standards.

Colin Ashton has worked in the Reagecon group since 1994 and is currently Head of the Chemical Metrology Department. A graduate of the University of Southampton, he has developed particular expertise in the development, stabilisation, manufacture and validation of cation, anion and electrochemical standards. He has particular scientific interest in all aspects of on line chemical analysis and has lectured and published on several areas of this field.

Leo Geary has worked for Reagecon Diagnostics Ltd. since 1998 and is currently the Senior Chemist in the Technical Services Department. In this role, he is involved in the provision of technical support for the complete Reagecon product range to customers to enable them to achieve high quality analytical results. This includes the provision of a traceable calibration and requalification service for all electrochemistry instruments.

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. It forms part of a comprehensive series of papers that the authors have written covering all of the practical requirements for accurate electrochemical measurement. These papers are available via Reagecon's website at <u>www.reagecon.com</u>.

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The authors wish to extend their gratitude to Ms V. Byrne for her assistance in compiling this paper and Ms R. Cooney for proofing this paper. The authors wish to thank all of their colleagues who have provided technical assistance in compiling Reagecon's series of pH measurement papers.

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The Effect of Temperature on Conductivity Measurement

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Abstract

The conductivity of all solutions changes as the solutions' temperature changes. It is a fundamental, practical requirement in the field of conductivity measurement that test measurements taken at different temperatures can be compared. This comparison can be facilitated by the use of Temperature Compensation, which provides an estimate of the samples' conductivity at a common reference temperature. This paper describes the principle of Temperature Compensation and provides details of the different types of Temperature Compensation that are available to the analyst so that the most appropriate type can be selected for the analyst's measurement application.

1 Introduction

An increase in a solution's temperature will cause a decrease in its viscosity and an increase in the mobility of the ions in solution. An increase in temperature may also cause an increase in the number of ions in solution due to dissociation of molecules. As the conductivity of a solution is dependent on these factors then an increase in the solution's temperature will lead to an increase in its conductivity.

The Temperature Coefficient of Variation is the rate at which a solution's conductivity increases with an increase of temperature and is expressed as the percentage increase in conductivity for a temperature change of 1°C. The Temperature Coefficient of Variation will be different for each solvent / solute mixture - examples are outlined in Table 1.

As can be seen from Table 1, temperature has a significant effect on the measured conductivity. For ultrapure water, a variation in temperature of 0.1° C will cause a change in conductivity of

0.55%. This example shows that accurate temperature measurement and control is of paramount importance for accurate conductivity measurement.

2 Temperature Compensation

In many applications, including field or process measurement, temperature control may prove impractical; but comparison of results is still required. This is achieved by using temperature compensation to calculate the samples' conductivity values at a common reference temperature.

To allow the analyst to compare conductivity measurements, results should be referenced to a specified temperature. Most conductivity measurements are referenced to 25°C, but some guidelines reference results to other temperatures – examples are given in Table 2.

Solution	Temperature Coefficient of Variation %/ °C at 25 °C
Ultrapure Water	5.5
NaOH 5%	2.01
NaOH 30%	4.50
HCl 5%	1.58
HC1 30%	1.52
KCl 5%	2.01
KCl 20%	1.68
Fresh water	~ 2.0

Table 1: Temperature Coefficient of Variation for Common Solutions (1,2)

Standard Procedure	Reference Temperature
United States Pharmacopoeia ⁽³⁾	25°C
ISO 7888-1985 ⁽⁴⁾	25°C
European Pharmacopoeia ⁽⁵⁾	25°C
ASTM D 1125 – 95 ⁽⁶⁾	25°C
U.K. Water Quality Supply Regulations ⁽⁷⁾	20°C

Table 2: Reference Temperature Cited in Commonly used Procedures



Graph 1: Temperature Compensation

Temperature compensation requires an algorithm to be applied to the measured conductivity reading and measured temperature to produce a compensated reading at a reference temperature. Graph 1 shows how a reading taken at 10°C is used to generate a temperature compensated conductivity value at 25°C by applying the algorithm represented by the curve.

2.1 Linear Temperature Compensation

For linear temperature compensation, it is assumed that the temperature coefficient of variation has the same value for all measurement temperatures. This assumption is not true; but for many measurements this does not result in a substantial contribution to the combined uncertainty of measurement of the reported result. The temperature coefficient of variation at 25°C, $\alpha_{0,25}$, is calculated as shown in Equation 1, with its value being expressed as a percentage change in conductivity value per 1°C change in temperature.

$$\alpha_{\theta,25} = \frac{\kappa_{\theta} - \kappa_{25}}{\kappa_{25}} \times 100$$

Equation 1

Where θ and 25°C are temperatures at which conductivities κ_{θ} and κ_{25} respectively are measured.

When the measurement is not carried out at 25° C, $\alpha_{0,25}$ can be used to calculate the solution's compensated conductivity at 25° C, as shown by Equation 2.

$$\kappa_{25} = \frac{\kappa_{\theta}}{1 + (\alpha_{\theta, 25} / 100) (\theta - 25)}$$

Equation 2

2.2 Non-Linear Temperature Compensation

As previously mentioned the relationship between temperature and conductivity does not follow a linear relationship. When the assumption of a linear relationship is not adequate, a polynomial may be applied to improve the accuracy of the calculated result.

Non-linear temperature compensation data for a variety of solutions is widely available, e.g. ISO 7888-1985⁽⁴⁾ provides data for natural waters having κ_{25} of 60 to 1000 μ S/cm over the temperature range 0 to 35°C. The standard

warns that the data is not applicable to potassium chloride calibration solutions - a different temperature compensation algorithm must be applied during calibration.

Graph 2 demonstrates how linear and non-linear temperature compensation are used to provide temperature compensated readings at 25°C from a measured value at 10°C. The graph demonstrates that the two forms of temperature compensation will result in different reported conductivity values at the reference temperature of 25°C. It should not be assumed that non-linear temperature compensation will lead to measurement errors than lower linear compensation - this will only be the case if the non-linear temperature compensation is a good fit for the sample's conductivity variation with temperature.



Graph 2: Representation of Linear Compensation & Non-Linear Compensation

Whatever form of compensation is used, the reading will not be as accurate as taking a reading of the sample's conductivity at the reference temperature.

2.3 Manual Temperature Compensation

Manual Temperature Compensation involves taking a series of conductivity measurements on a sample at various temperatures. A linear or polynomial best fit is then applied to the data to produce an equation for the temperature compensation of results. The temperature compensated conductivity result is manually calculated using the derived equation and the measured conductivity and temperature.

2.4 Automatic Temperature Compensation (ATC)

ATC utilises a microprocessor within the instrument to determine a temperature compensated conductivity result. The ATC options available will depend upon the specification of the conductivity instrument:

- Basic conductivity instruments may only offer linear compensation with the temperature coefficient fixed at 2%/°C. This value is chosen because it is a reasonable approximation of the temperature coefficient of commonly encountered dilute salts. However, many solutions have a markedly different temperature coefficient and measurements of these solutions with basic instruments will result in significant errors in the reported conductivity value.
- Intermediate conductivity instruments may offer linear compensation with a user-definable temperature coefficient. These instruments may only allow the user to vary the temperature coefficient in relatively large increments, e.g. 0.5%/°C increments.

 Advanced instruments may offer both linear and non-linear temperature compensation functions as well as an uncompensated mode. These instruments will allow the analyst to input a temperature coefficient (normally 0.00 to 10.00 %/°C) and a reference temperature (normally 20 or 25°C). It is important to note that some procedures, such as USP 26 <645>⁽³⁾ require that no temperature compensation can be applied to the measured value.

The non-linear temperature compensation will carry out temperature compensation using preprogrammed data in the instrument. Non-linear ATC data is available for a variety of solutions including

- Natural Waters according to ISO 7888⁽⁴⁾
- Ultrapure water with traces of ammonia
- Ultrapure water with traces of HCl
- Ultrapure water with traces of NaCl
- Ultrapure water with traces of NaOH

In addition to temperature compensation for sample measurements, conductivity instruments may also be pre-programmed to allow temperature compensation to be utilised during calibration. In this instance, the appropriate compensation will be applied during the calibration process for common conductivity calibration standards such as potassium chloride based 147, 1413 & 12,880 µS/cm at 25°C. Many conductivity meters also allow calibration with a user-defined conductivity standard. Standards selected for this purpose should be provided with full characterization of their conductivity's variation with temperature so that this can be accounted for during the calibration process.

3 Conclusion

Measurement temperature has a significant influence on conductivity readings; but appropriate temperature compensation is a powerful tool to allow meaningful comparison of readings taken at different temperatures. The analyst must ensure that the type of temperature compensation utilized is appropriate for both the type of sample being analyzed and the required test accuracy. This is an essential factor for determining the suitability of a conductivity instrument for measurement applications.

A conductivity measurement taken with the sample at the reference temperature will always be more accurate than a temperature compensated reading taken away from the reference temperature – this point is essential for critical applications requiring high accuracy of measurements.

Further information on the implications of temperature effects on conductivity measurements is given in further Reagecon practical conductivity measurement publications^(8,9).

4 References

- 1. T.S.Light, "Temperature Dependance and Measurement of Resistivity of Pure Water", Analytical Chemistry, 1984, 56, 1138
- 2. Instruction Manual for CyberScan CON 500 Conductivity Meter, Eutech Instruments
- 3. United States Pharmacopoeia 26-NF 21, Monograph <645>
- "Water Quality Determination of Electrical Conductivity", ISO 7888:1985
- 5. European Pharmacopoeia, Edition 5.3, Section 2.2.38: "Conductivity"
- "Standard Test Methods for Electrical Conductivity and Resistivity of Water", American Society for Testing and Materials, D1125-95
- "The Water Supply (Water Quality) Regulations 2000" – Water, England and Wales, Statutory Instrument 2000 No. 3184, The Stationery Office Ltd.
- 8. J.J. Barron & C. Ashton, "A Practical Guide to Accurate Conductivity Measurements"*
- J.J. Barron & C. Ashton, "Conductivity Measurement in the Pharmaceutical Industry"*

* These papers form part of a comprehensive series of papers that the authors have written covering all of the practical requirements for accurate conductivity measurement. These papers and the authors' book, "A Practical Guide to Accurate Conductivity Measurement" are available via Reagecon's website www.reagecon.com.

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Reagecon Staff Events and Initiatives

As part of Reagecon's commitment to employee wellbeing, we are participating in an ongoing workplace wellbeing program.

We welcomed Dearbhla Byrne and Kieran Blake from the Bank of Ireland who launched the Workplace Direct programme, which brings branch services directly to our employee's. Pictured here with our HR Manager, Vicky Howard.





Reagecon staff pictured taking part in Trick or Treat for Temple Street to raise funds for Temple Street Children's Hospital.

Staff were provided with healthy snacks and a recipe booklet filled with healthy breakfast, lunch and dinner recipes.



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