



Crystal City News



*A newsletter brought to you by
Starna... the cell people*

Issue 3

Welcome!

It is with great pleasure that I introduce you to our Issue 3 of Crystal City News.

Since our last issue we have been very busy producing new products, some of which are featured herein, e.g. our range of Low Headspace Cells, Ultra High Vacuum Cells and our extended range of Tablet Dissolution Cells etc.

In addition to the Starna Reference Materials catalogue we are proud to announce the release of the NEW Starna Cell catalogue (see picture) make sure you contact us today for your FREE copy.

We hope you find this newsletter of value and if we can be of any assistance, please do not hesitate to contact us.

Regards,

Sandra Greenwood, SWmbO



Calibration Science for UV-Visible Spectrophotometry.

John P. Hammond C.Chem MRSC - Technical & Marketing Manager, Optiglass Ltd., UK

Introduction

The value of the chemical measurement depends upon the degree of confidence that can be placed on the result and thereby it's 'fitness for purpose.' If you couple this statement with an appropriate section in any of the internationally recognized quality standards, one irrefutable observation is that both have a common requirement - effective equipment performance verification, often simply referred to as calibration.

The usual way to perform calibration is to subject known amounts of the parameter (e.g. using a reference material) to the measurement process and monitor the measurement response. In the case of UV-Visible Spectrophotometry, amongst the many parameters to keep in control, the three most usually measured are wavelength accuracy, linearity or absorbance accuracy, and stray light. Over the sixty years that UV-Visible Spectrophotometry has been available as an instrument technique, there is a range of materials that have been proposed, and then discarded for various reasons [1]. This article details the reference materials currently in general use for checking these fundamental indicators of performance.

Absorbance/Transmission

There are several methods for checking either the linearity or absorbance accuracy of an instrument. However, it is imperative that the certified reference material used for the calibration should be matched to the wavelength and absorbance ranges in use. Just because an instrument is compliant in the visible region using Neutral Density filters, do not assume that a similar degree of accuracy will be observed in the UV region! For the measurement of this fundamental parameter, Starna offers you the choice of either a sealed cell, or filter materials.

Potassium Dichromate - UV Absorbance and Linearity

The use of potassium dichromate solvated in dilute perchloric acid is an established and well-recognized method for the validation of the absorbance scale and linearity of a spectrophotometer in the UV region [2]. When prepared in 0.001 M perchloric acid, potassium dichromate gives a spectral scan containing characteristic peaks at 257 nm

and 350 nm, and troughs at 235 nm and 313 nm. Within the concentration range 20 - 100 mg/l, if the absorbance scale of a narrow SBW (< 2 nm) spectrophotometer is linear, the apparent absorbance of a series of concentrations will be a linear function of concentration.

Neutral Density Filters - Visible Absorbance and Linearity

Schott NG-type glasses have been used for over thirty years for the validation of the absorbance scale and linearity of a spectrophotometer in the visible region. When manufactured to a specific thickness, filters with a range of transmission and absorbance values can be produced. Whilst the spectral scan of these materials is essentially flat, NIST SRM 930e and SRM 1930 certify these glass filters at 440.0 nm, 465.0 nm, 546.1 nm, 590.0 nm, and 635.0 nm. These sets consist of three filters of varying transmissions together with an empty aluminium holder, to be used as a blank.

All Starna Neutral Density filters are manufactured and certified in accordance with the 'Technical Specification for Certification of Spectrophotometric NTRMs' (NIST Special Publication 260-140).

Wavelength

The spectra of the rare earth elements in various physical forms can be used as useful references. Holmium oxide in perchloric acid solution is a favorite with many regulatory bodies, and is available as a NIST traceable certified material from Starna. Also frequently used are holmium and didymium (a mixture of neodymium and praseodymium) glass filters. These have the advantage of being in solid form, but incorporation of the rare earth into the glass matrix does cause some shifting and broadening of the characteristic peaks.

Stray Light

Stray light is the most difficult of the three parameters to check. It is extremely wavelength and sample-dependent, so any checks must be qualified as to the procedure used.

However, most instrument manufacturers rely on the tests specified by the American Society for Testing and Materials (ASTM)[3], involving solutions with sharp cut-offs to measure stray light, and it is these solutions (at the ASTM concentrations), heat-sealed in far UV quartz cuvettes that are available from Starna. The most widely used are potassium chloride at 200 nm, and sodium iodide at 220 nm.

Conclusions

Calibration Science provides the foundation for generating quality data, and equally important, evidence to that fact. Related to this statement is the fundamental principle of Traceability.

Traceability means different things to different people. If we look at the ISO International Vocabulary of Basic and General Terms in Metrology, ISO 1993 definition:-

"Traceability is the property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties."

But this has to be broadened into what this requirement means in an international marketplace. An excellent summary document is the Co-operation on International Traceability in Analytical Chemistry (CITAC) paper on 'Traceability in Chemical measurement' which can be found on the CITAC website. This positioning paper clearly describes the role that traceability has to play in a global marketplace, where ISO/IEC 17025 accreditation has the anchor role to play as the Quality system provider.

All the Certified Reference Materials described above are available from Starna, and are manufactured and certified within our ISO/IEC 17025 accredited laboratory.

References **1.** Standards and Best Practice in Absorption Spectrometry - Edited by Chris Burgess and Tom Frost UVSG. ISBN 0-632-05313-5 Blackwell Science <http://www.blackwell-science.com> **2.** NIST Special Publication 260-54 Standard Reference Materials: Certification and use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard - SRM 935 **3.** ASTM standard E-387 (Annual Book of ASTM Standards) 1984.

Low Headspace Cells: Variants on Series 26 Low Volume Cells

Uniquely available from Starna, this extended range of cells takes the concept of micro volume design to the next logical step; i.e. reduction of air volume in the headspace above the sample liquid.

The requirement to perform DNA analysis on increasingly reduced sample volumes raises the problem of significant evaporation losses when performing tests on volumes as low as 8 – 10 μ l. The LHS range of cells uses a profiled PTFE stopper to reduce the headspace above the sample to an absolute minimum, and thereby minimizes the evaporation into this area.

Available as a variant of Starna's 26 Series low volume cells, transmission cells in the range 8 – 440 μ l are available; and for fluorescence measurements the 26-F series provides measurement capability using volumes of 8 – 80 μ l.

As with all Starna products, each cell is manufactured under stringently controlled conditions, to meet all aspects of ISO 9000 quality control.



Calibration Laboratory achieves NVLAP accreditation

The Calibration Laboratory at Optiglass Limited, UK (a subsidiary of the Starna Group of companies) has recently been granted NVLAP accreditation for the calibration of Neutral Density Glass Filters in the Transmission range of 1% to 50%.

This accreditation (currently based on ISO Guide 25) establishes – by an independent third party – the quality of the calibrations within the laboratory on these materials, and is one of the essential requirements for the Company to be able to supply the NIST Traceable Reference Materials. (NTRMS)

The following extracts are taken directly from the NIST Special Publication 260-140 titled 'Standard Reference Materials: Technical Specifications for Certification of Spectrophotometric NTRMS'.

'The NIST Traceable Reference Materials (NTRM) program was created to address the problem of increasing needs for reference materials with a well-defined traceability linkage to existing NIST chemical measurement standards. This traceability linkage is established via criteria and protocols defined by

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NIST and tailored to meet the needs of the metrological community to be served.

NIST traceability for these materials is assured through the National Voluntary Laboratory Accreditation Program (NVLAP) of NIST.'

Optiglass is one of only three companies currently accredited through the NVLAP scheme that is producing NTRM filter sets.

In addition to the NTRM filter sets, Starna Neutral Density Filter sets are also available under the Starna RM - 1N2N3N and RM - N1N35N part numbers.



Starna Tablet Dissolution Flow Cell Range Extended

Starna have extended their range of precision tablet dissolution flow cells.

Path lengths, in the standard aperture dimensions (583 series), now range from 0.1mm through to 100mm and in the 584 series from 0.01mm to 2mm.

Both series 583 and 584 are available to suit all tablet dissolution instruments. The most frequently requested material is Far UV Quartz, but cells can be supplied in other materials to special order.

All cells, regardless of path length, are housed in a precision-engineered encapsulating jacket ensuring precise accuracy while greatly reducing the possibility of damage during use. Individual testing - up to a pressure of 75 psi - ensures that all cells are leak proof.

Each cell is given a unique serial number for identification and traceability purposes. Cells can be supplied in single units or in matched sets that would normally comprise six to eight pieces. Certification is available for sets of the same path length for a nominal charge.

Available in Z dimensions of 8.5, 15 and 20mm, these cells are offered complete with single-ended connections as standard. Where instruments require special fittings the instrument of use should be specified at the time of ordering, together with any relevant fitting information.

As with all Starna products, each cell is manufactured under stringently controlled conditions, meeting all aspects of ISO 9000 quality control.



Ultra-High Vacuum (UHV) Cells



Specialist commissions have offered us the opportunity to produce a wide range of Ultra-High Vacuum cells for the advanced technology research sectors. A cell designed to meet these requirements is now in use at a leading UK university in the field of atomic physics.

This specific cell design included unique features while strict adherence to basic design requirements was essential. Incorporating large area optical flats, parallel low reflectance faces, and with dimensions quoted in cm rather than mm, these optical specifications require high precision manufacturing techniques. By necessity, the requirement for low reflection losses on these cells meant that the ability to multi-layer coat had to be handled as an integral part of the manufacturing process within a clean room environment.

Coupling the 'fine' optical requirements with the engineering essential in allowing the cell to be mechanically flange-coupled to an ultra-high vacuum system offered a range of conflicting manufacturing tasks that had to be achieved.

These Ultra-High Vacuum cells are now offered as part of the Starna range.

As with all Starna products, each cell is manufactured under stringently controlled conditions, meeting all aspects of ISO 9000 quality control.

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Prof/Dr/Mr/Mrs/Ms:
 Department:
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Please send me information on:

- Starna Reference Materials Low Headspace Cells Tablet Dissolution Cells NEW Cell Catalogue

If we can help with a problem, please state the question or requirements and we will do our best to find a solution:

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Starna... the cell people

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