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THE INTERNATIONAL JOURNAL OF METROLOGY

METROLOGY 101: PREPARATION & USE OF AN ICE-POINT BATH

2011
OCTOBER
NOVEMBER
DECEMBER

An Introduction to Mass
Metrology in Vacuum

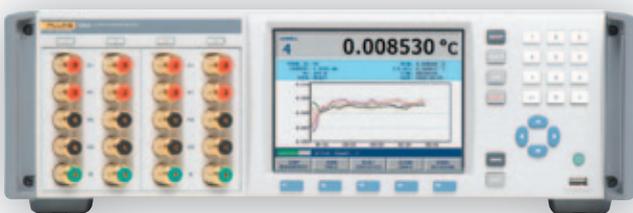
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CALENDAR

CONFERENCES & MEETINGS 2011 - 2012

Nov 13-18 26th Annual Meeting of the American Society for Precision Engineering. Denver CO. Website: http://www.aspenet/meetings/2011_Annual/ASPE_Annual_2011.html.

Nov 14-17 IEST Fall Conference 2011. Durham, NC. <http://www.iest.org/fallconference>.

Nov 15-16 LVMC Large Volume Metrology Conference & Exhibition 2011. Manchester, UK. The LVMC is the only European event solely dedicated to portable and large volume 3D measurement technology. <http://www.lvmc.eu/>.

Dec 8-10 India Lab Expo. New Delhi, India. The 3rd International Exhibition and Conference on Scientific & Lab Instruments. <http://www.indialabexpo.com>.

Jan 26-27 NCSLI Technical Exchange. Charleston, SC. The 2-day event will include hands-on tutorials, mini-concurrent sessions and exhibits. Visit: <http://www.ncsli.org>

Mar 6-8 2012 South East Asia Flow Measurement Conference. Kuala Lumpur, Malaysia. www.tuvnel.com

Mar 8-9 METROMEET – 8th International Conference on

Industrial Dimensional Metrology. Bilbao, Spain. <http://www.metromet.org/>.

Mar 14-15 Quality Expo Texas. Fort Worth, TX. In 2012, the regional, biennial edition of Quality Expo will move on from Charlotte to join Canon's newest advanced design and manufacturing event planned to launch in Texas. Website: <http://www.canontradeshows.com/expo/qexpos10/>.

Mar 19-23 Measurement Science Conference. Anaheim, CA. Measurement Science: Challenges in the Future. Held in conjunction with the International Temperature Symposium (ITS9). Website: <http://www.msc-conf.com/>.

Mar 19-23 9th International Temperature Symposium. Anaheim, CA. The NIST Temperature and Humidity Group has organized the 9th International Temperature Symposium, in conjunction with the Measurement Science Conference. Website: <http://www.its9.org/>.

Mar 21-23 ICSM2012. Annecy, France. The 3rd International Conference on Surface Metrology. Website: <http://www.icsm3.org>.

Apr 23-27 CAFMET 2012. Marrakech, Morocco. The African Committee on Metrology (CAFMET) is organizing the 4th International Metrology Conference. Website: <http://www.acmetrology.com/CAFMET2012>.

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**Conference Spirit
& Social Media**

NCSLI pulled off another great conference this year, despite the natural disasters. Staff commitment in making the 50th Anniversary event exceptional did not go unnoticed.

One of my jobs as editor, is to keep CallabMag.com up to date with metrology related conferences around the world, and I'm impressed by the flurry of metrology activity and conferences *outside* of the States. I recently read "The Role of Measurement and Calibration" from the United Nations Industrial Development Organization; it helped me understand the importance of metrology, and how France, Switzerland, the United Kingdom and Sweden are trying to harmonize measurement related activities. It really highlights the collaborative efforts taking place around the globe.

As the need for a stronger metrology community continues to grow, social media has helped to bridge a gap. There are a handful of metrology blogs that have been around awhile. The amount of people participating online has continued to increase as individuals from all over the globe chime in, on manufacturer blogs and newer forums such as LinkedIn. This is where the discussion on ice-point baths came about, when our publisher started a thread on the Cal Lab Magazine group on LinkedIn. Jerry Eldred, with EAG, came to the fore and was good enough to take the time to write this issue's Metrology 101 article.

Social media also gets credit for getting us in touch with the good people at Spark Measurement Technologies, who contributed a nice laboratory shot for this issue's cover art. So if you haven't joined in on some juicy discussion online about ice-point baths or something else metrology related, now is always a good time to start! Look for a new page under Metrology Links, for metrology related social media links, on our web site at www.callabmag.com.

Regards,

Sita



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Apr 25-27 The Americas Flow Measurement Conference. Houston, TX. Email events@tuvnel.com for further details. <http://www.tuvnel.com>.

Apr 26-29 APMAS 2012. Antalya, Turkey. APMAS 2012 intends to be a global forum for researchers and engineers to present and discuss recent innovations and new techniques in Applied Physics and Material Science. Website: <http://www.apmas2012.org/>.

May 13-16 2012 IEEE International Instrumentation and Measurement Technology Conference. Graz, Austria. "Smart Measurements for a Sustainable Environment." <http://imtc.ieee-ims.org/>.

May 23-25 MetroExpo2012. Moscow, Russia. The 8th Moscow International Forum "Precise Measurements - The Basis of Quality and Safety" will be held with specialized exhibition of measuring instruments and metrological equipment (MetroExpo), to ensure uninterrupted operation of production facilities (PromSafety), commercial energy accounting (ResMetering), means of verification and testing of medical devices (MedTest), and the 4th Moscow International Symposium "Accuracy. Quality. Security." For more information in English visit: <http://www.metro.exprom.ru/en/>.

Jun 20-22 International Symposium on Fluid Flow Measurement. Colorado Springs, CO. <http://www.isffm.org/>.

SEMINARS: Online & Independent Study

AC-DC Metrology- Self-Paced Online Training. Fluke Training. <http://us.flukecal.com/training/courses>.

Basic Measuring Tools – Self Directed Learning. The QC Group, <http://www.qcgroup.com/calendar/>.

Introduction to CMMs – Self Directed Learning. The QC Group, <http://www.qcgroup.com/calendar/>.

Introduction to Measurement and Calibration – Online Training. The QC Group, <http://www.qcgroup.com/calendar/>.

Intro to Measurement and Calibration – Self-Paced Online Training. Fluke Training. <http://us.flukecal.com/training/courses>.

ISO/IEC 17025 Compliance. Workplace Training, tel (612) 308-2202, info@wptraining.com, <http://www.wptraining.com/>.

Measurement Uncertainty – Self-Paced Online Training. Fluke Training. <http://us.flukecal.com/training/courses>.

Measurement Uncertainty Analysis – Online Training. The QC Group, <http://www.qcgroup.com/calendar/>.

Metrology for Cal Lab Personnel- Self-Paced Online Training. Fluke Training. <http://us.flukecal.com/training/courses>.

Precision Dimensional Measurement – Online Training. The QC Group, <http://www.qcgroup.com/calendar/>.

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SEMINARS: Accreditation

Nov 7-11 ISO 17025 Compliance and Auditing Techniques Including ANSI Z540.3 Requirement. Los Angeles, CA. Workplace Training, <http://wptraining.com/workshops.htm>.

Nov 16-17 ISO/IEC 17025 and Accreditation. Charleston, SC. The American Association for Laboratory Accreditation, <http://www.a2la.org>.

SEMINARS: Dimensional

Nov 15-17 Hands-On Gage Calibration. Elk Grove Village IL. Mitutoyo Institute of Metrology, tel 888-MITUTOYO, mim@mitutoyo.com

www.mitutoyo.com

Dec 6-7 MSA 4: Going Beyond Gage R&R. Chicago, IL. Mitutoyo Institute of Metrology, tel 888-MITUTOYO, mim@mitutoyo.com, www.mitutoyo.com

Dec 8-9 Gage Calibration and Repair. Clearwater Beach FL (Tampa Area). ICT Enterprises, tel 952-881-1637, fax 952-881-4419, info@consultinginstitute.net, www.consultinginstitute.net.

Dec 12-13 Gage Calibration and Repair. Atlanta GA. ICT Enterprises, tel 952-881-1637, fax 952-881-4419, info@consultinginstitute.net, www.consultinginstitute.net.

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Jan 10-11 Gage Calibration and Repair. Blaine MN. ICT Enterprises, tel 952-881-1637, fax 952-881-4419, info@consultinginstitute.net, www.consultinginstitute.net.

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Jan 26-27 Gage Calibration and Repair. Detroit MI. IICT Enterprises, tel 952-881-1637, fax 952-881-4419, info @consultinginstitute.net, www.consultinginstitute.net.

SEMINARS: Flow & Pressure

Jan 23-27 Principles of Pressure Calibration. Phoenix, AZ. Fluke Calibration, <http://us.flukecal.com/training>.

Feb 20-23 Comprehensive Hydrocarbon Measurement Training Course. Kuala Lumpur. Colorado Engineering Experiment Station Inc., www.ceesi.com.

Feb 27 Fundamentals of Ultrasonic Flowmeters Training Course. Kuala

Lumpur. Colorado Engineering Experiment Station Inc., www.ceesi.com.

SEMINARS: Mass & Weight

Dec 5-9 Intermediate Mass and Gravimetric Volume. Gaithersburg, MD. NIST, <http://www.nist.gov/pml/wmd/labmetrology/schedule.cfm>.

SEMINARS: Measurement Uncertainty

Nov 14 Introduction to Measurement Uncertainty. Web Event (1PM-3PM EDT). E=mc3 Solutions, <http://www.wptraining.com/>.

Nov 14-15 Introduction to Measurement Uncertainty. Charleston, SC. The American Association for Laboratory Accreditation, <http://www.a2la.org>.

Nov 15 Measurement Uncertainty for Testing Labs. Web Event (1PM-3PM EDT).

E=mc3 Solutions, <http://www.wptraining.com/>.

Nov 16 Calibration Interval Analysis. Web Event (1PM-4PM EDT). E=mc3 Solutions, <http://www.wptraining.com/>.

Nov 15-17 Measurement Uncertainty Analyst Class. Fenton, MI. Quametek Institute of Measurement Technology, <http://www.qimtonline.com/>.

Feb 6-7 SPC and Excel for Metrology Applications. Boulder CO. WorkPlace Training, Inc., <http://www.wptraining.com/>.

Feb 8-10 Measurement Uncertainty. Boulder CO. WorkPlace Training, Inc., <http://www.wptraining.com/>.



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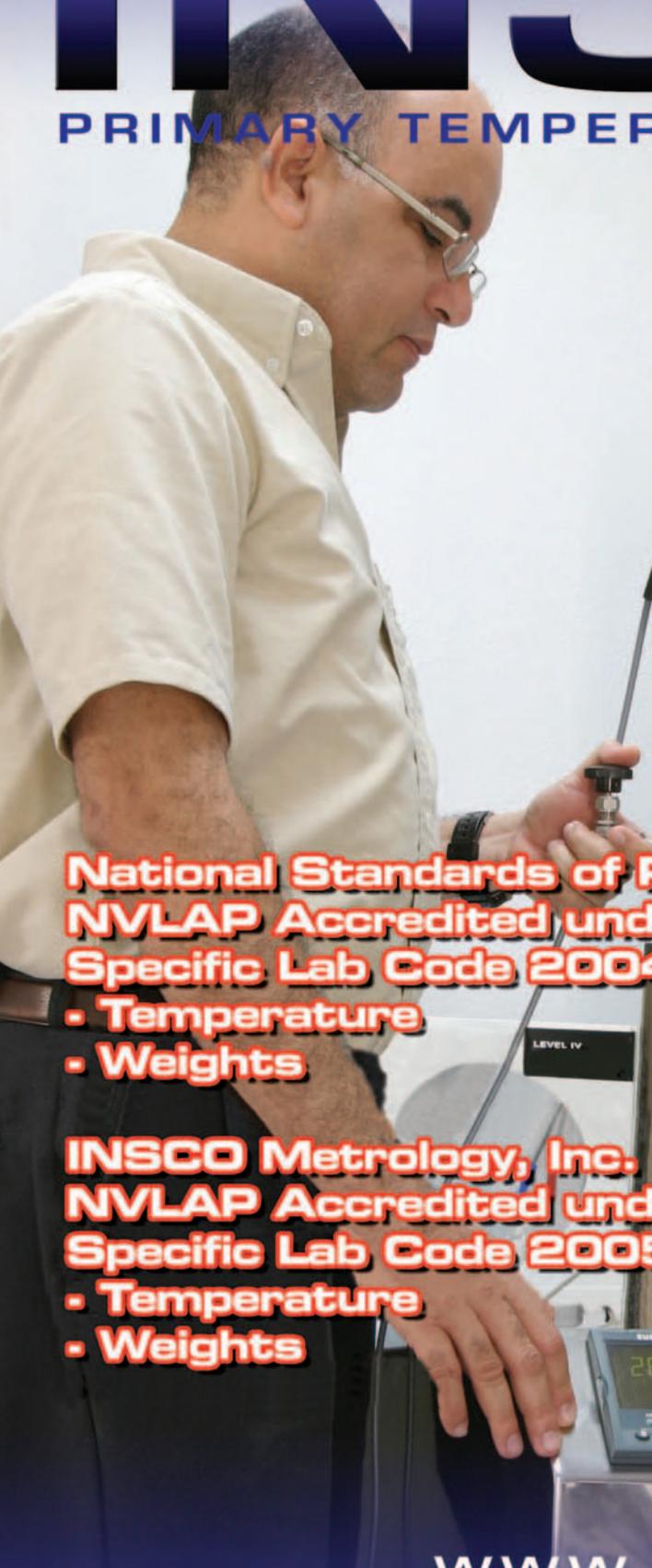
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MetricTest Acquired by Microlease

MetricTest (www.metrictest.com) was acquired by Microlease (www.microlease.com), the leading test equipment rental and asset management organization in the UK. Recognized by test and measurement engineers as a North American leader in customer service, MetricTest is a complete solutions provider for rentals and new and refurbished equipment sales. Under Microlease's ownership, MetricTest customers will benefit from more inventory, additional client services and global account support.

Established 20 years ago by Marshall Hart, MetricTest has achieved strong business growth and, like Microlease,

has forged strong relationships with most of the major Original Equipment Manufacturers (OEM's) including Agilent Technologies, Tektronix, Advantest, Anritsu, Keithley and Rohde & Schwarz. The MetricTest executive board will now be led by Microlease CEO, Nigel Brown, as Chairman. MetricTest President and COO, Vern Stevenson, will become CEO.

As a result of the acquisition, Microlease anticipates its income in the US will exceed \$50 million in its current financial year and more than \$90 million in the following 12-month period.

Transcat Acquires Newark's Calibration Services Business

Transcat, Inc. (www.transcat.com), distributor of professional grade handheld test and measurement instruments and accredited provider of calibration, repair and other measurement services, announced that it has acquired the calibration services business from Newark, a subsidiary of Premier Farnell, PLC (LSE: PFL.L), and entered into a strategic alliance whereby Newark will market Transcat's calibration services to its broad customer base of electronic design engineers, maintenance, repair and operations engineers, and industrial buyers. Newark has also contracted for Transcat to provide its calibration services for its own test and measurement equipment distribution needs. The asset purchase includes the calibration services assets of Newark's calibration labs in Aurora, CO (Denver), Chandler, AZ (Phoenix) and Hendersonville, TN (Nashville).

Transcat paid \$3.0 million in cash for the acquisition.

UAE Converts to the Metric System

On November 11th, the United Arab Emirates will be switching from gallons to liters and from feet to meters, when it will require companies operating in the UAE to use the International System of Units (SI). The Emirates Authority for Standardization and Metrology (ESMA) is overseeing the changes to the SI, in conformance with World Trade Organization (WTO) mandates.

Another unit of measure to go by the wayside will be the traditional "wall" (yard) for measuring fabric and "tola," which is 11.62 grams measured as a liquid, such as perfume. The tola is also used by jewelers when weighing gold, but the switch to the SI will help standardize shops where the tola may be interpreted as 11 grams at one place and 10 grams at another. The SI is already utilized by many merchants and local manufacturers, while others are prepared for the change.

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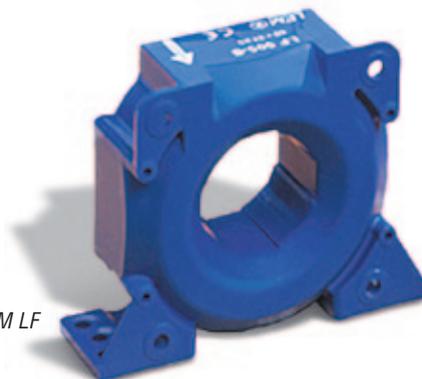
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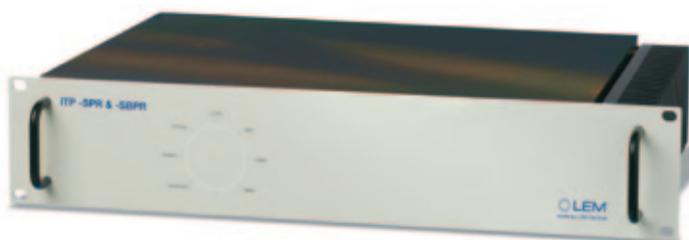


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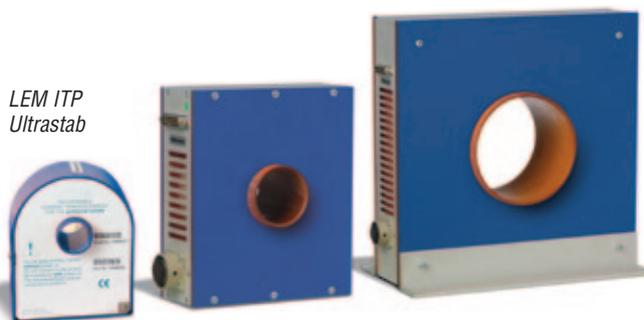
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Huber Petite Fleur – Temperature Control Systems

The Petite Fleur is the smallest dynamic temperature control system in the Unistat series and is ideally suited to controlling small investigation reactors. Since its launch the baby Tango has become a bestseller in the Huber product range and as a result of its success, the product line has been expanded to include the new Petite Fleur-eo. Designed specifically for externally open applications, the Petite Fleur-eo eliminates the risk of overflow or running dry, unlike conventional bath circulators.

Like all Unistats, the Petite Fleur models have outstanding thermodynamic properties and their ability to rapidly and accurately control and change temperature make them unique. Thermodynamically they cannot be matched by any other system within the price range. The range of case studies created by Huber document that Unistats are significantly faster with better control, than competitive models offering more cooling power. The Petite Fleur was tested with reactor volumes from 0.3 to 6 litres and despite its size there is no drop in performance. All the case studies are available to download at www.huber-online.com.

All Petite Fleur models are built as compact, space saving designs and are easy to handle and manoeuvre into place. The Petite Fleur is available with working temperatures from -40 to +200 °C and cooling capacities of 480 Watts at full pump speed (according to DIN 12876). Developments at Huber show that reducing the pump speed can make additional cooling power available – in some cases an extra 50 Watts can be achieved. The powerful circulation pump with flow rates up to 33l/min guarantees optimum heat transfer and the soft-start protects delicate glassware from breakage by compensating for changes in the fluid's viscosity and balancing the pressure in the fluid circuit.

The Petite Fleur is equipped with the CC-Pilot controller which is preinstalled with the "Professional" version of the controller software. The CC-Pilot offers a colour TFT screen, graphic display and, with a data cable, can be removed from the unit and used as a remote control. Further features such as: a programmer, calendar and clock, user menus, ramp function, calibration and many other functions are offered. The Com.G@te interface module is factory fitted offering digital and analogue interfaces complying with the NAMUR standard, enabling integration into a process control system.



All models are supplied with natural refrigerants making them an environmentally friendly temperature control option.

More information can be found in the new 2011/12 catalogue which can be downloaded at www.huber-online.com or requested free by calling +49 (0) 781 9603 123 or emailing info@huber-online.com.

New Agilent M9181A PXI DMM

Agilent Technologies Inc. (NYSE: A) introduced the M9181A digital multimeter to complement its growing family of PXI DMMs. This new 6½ digit PXI DMM offers basic measurement features, without compromising resolution and reliability, at a competitive price point. With the M9181A, test engineers in aerospace/defense, electronic manufacturing, and automotive industries now have an economical PXI DMM alternative.

The M9181A 6½ digit PXI DMM measures common parameters such as DCV, DCI, ACV, ACI, and two- and four-wire resistance. The DMM offers 90-parts-per-million basic DCV and 800-parts-per-million basic ACV one-year accuracies and inputs up to 200 volts.

The M9181A DMM is compatible with PXI, PXI Hybrid and CompactPCI® instrument mainframes, including Agilent's PXI chassis & accessories.

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For more information on Agilent's new M9181A PXI DMM, go to www.agilent.com/find/M9181A.



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NEW PRODUCTS AND SERVICES



Soft dB Data Loggers

Scantek, Inc., is pleased to announce the availability of two of the newest instruments from Soft dB, the "Piccolo" and the "Concerto."

The Piccolo is a compact, data logging integrating sound level meter with up to 10 days of continuous measurement and many other features. The introductory

price is \$345.00 which includes windscreen, USB cable, battery, software, case.

The Concerto is a sophisticated four-channel, Class 1, hand-held acoustic data logging measurement system with up to 16 GB of internal memory. The four independent channels can do building acoustics, building vibrations, and soon, sound intensity. The unit starts at about \$10,000.

About Scantek, Inc.

Scantek, Inc., is a distributor for multiple sound and vibration lines, including Norsonic, RION, CESVA, Castle Group, KCF Technologies, Soft dB, Metra Vibration Transducers, DataKustik, BSWA Transducers, and Exttech Sound and Vibration Instruments. Scantek also has an ISO 17025 NIST/NVLAP accredited Calibration Laboratory, --Scantek is committed to providing quality sales, customer repair, service, full instrument rental, and calibration of sound & vibration instrumentation. For more information, call (800) 224-3813 or visit www.scantekinc.com.

ASL F650 Precision Thermometry Bridge

ASL, an Elektron Technology plc company, has introduced the F650 precision thermometry bridge, a significant enhancement to its best selling F600 and F700 ranges. Based on proven and exceptionally stable AC bridge technology, the F650 provides high accuracy temperature measurement and calibration, accurate to +/- 1ppm over the full range (typically +/- 0.25mK, at 0.01°C).

Thanks to the elimination of thermal EMF errors, automatic cancellation of probe and cable reactance effects, the F650 thermometry bridge is ideally suited to national and accredited laboratories, in-house calibration departments, critical process companies and research organizations requiring high accuracy measurement of a ratio, resistance or temperature.

With a temperature range of -200°C to +962°C the F650 excels in the most demanding temperature measurement industries and helps meet ITS90, CVD and EN 60751 standards. The thermometry bridge operates at the lower frequency of 25Hz (50Hz supply) and provides resolution of 0.01mK with inherently low noise.

The F650 has 25Ω and 100Ω internal standard resistors and can also be used with external standard resistors. It has a ratio range of 0 to 4.9999999, or 0 to 500 ohms resistance, with measurement results presented on a large scale, multifunction VFD screen. The mean, max, min, std dev and n sample count can also be displayed with a separate graphical display.

To ensure long term reliability the F650 uses surface mount technology with no mechanical relays.

For further information and to download a datasheet visit: www.aslltd.co.uk/

About ASL

ASL is an expert in precision temperature metrology and calibration equipment. With a 45 year track record, ASL's customers include NASA, Airbus and leading international standards setting laboratory NPL.

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NEW PRODUCTS AND SERVICES

Mitutoyo CRYSTA-Apex® S CMM

The new CRYSTA-Apex® S Coordinate Measuring Machine from Mitutoyo America Corporation brings new levels of performance and economy to the 1.7 μm class of CNC CMMs.

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CRYSTA-Apex® S more than doubles the effective measuring range at a given measurement tolerance as compared to typical CMMs in its class. Additionally, the CRYSTA-Apex® S drive features high-speed (max 519 mm/s) and high acceleration (max 2,309 mm/s²). These advances result in higher throughput for greater productivity and lower total owning and operating costs.

The CRYSTA-Apex® S uses the new UC-400® controller to manage digital servo system control loops for position, speed, and current. This makes it easy to implement various types of control algorithms.

Extreme rigidity helps the CRYSTA-Apex® S maintain accuracy. The Y-axis guide rail is integrated into one side of the granite surface plate. Precision air bearings located on the bottom, front, rear and upper surfaces of the X-axis slider minimize vibration and ensure stability even during high-speed, high-acceleration operation.

Accuracy is further enhanced by an advanced Temperature Compensation System. The System consists of a thermometer unit that measures the temperatures from thermal sensors located on the scale units of the CMM main unit and from a set of workpiece thermal sensors. The temperature data is transferred to the UC-400® machine controller for thermal compensation.

The CRYSTA-Apex® S supports a wide range of probes that offer increased capabilities including the MPP-310Q scanning probe that collects cloud point data at speeds of up to 120 mm/s. Other probes suited for screw depth measurement. Available software options enable the CRYSTA-Apex® S to tackle a wide

variety of measurement applications. Software packages include GEOPAK®, a high-functionality general-purpose measurement program which is at the heart of MCOSMOS® (Mitutoyo Controlled Open System for Modular Operation Support) software. Additional software supported includes: CAT1000S® for freeform surface evaluation; CAT1000P®, an offline teaching program; SCANPAK®, for contour measurement; and a range of programs supporting laser and vision probes.

Additionally, CRYSTA-Apex® S supports MeasurLink STATMeasure Plus®, Mitutoyo's proprietary statistical-processing and process-control program.

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Lista International Corporation presents the Arlink® 8000 Modular Workstation System. Ergonomically designed, these modular workstations offer unlimited flexibility to accommodate changing or future needs. Ideal for a variety of applications, such as assembly, computing environments, service, repair, research and technical work, Arlink 8000 workstations can be assembled and reconfigured faster than other workstations, saving time and money.

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The Arlink 8000 Modular Workstation System is part of Lista International's technical workstation offering. Lista offers the most complete technical workstation/workbench product line on the market – all available through a single source.

To find out more about Lista's Arlink 8000 Modular Workstations, call or write: Lista International Corporation, 106 Lowland Street, Holliston, MA 01746 USA; TEL 1-800-722-3020; FAX 508-626-0353; email sales@listaintl.com. Visit Lista's web site at www.listaintl.com.



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NEW PRODUCTS AND SERVICES

Keithley Launches New Power Supply Product Line

Keithley Instruments, Inc., a world leader in advanced electrical test instruments and systems, announced today the availability of five new general-purpose programmable DC power supplies designed to complement the company's existing line of specialty power supplies and source measurement instruments for component, module, and device characterization and test applications. The Series 2200 family combines superior voltage and current output accuracy at a cost-effective price, flexible operation, and features designed to enhance ease of use in a variety of device characterization or test applications.

The five models in the Series 2200 line offer maximum voltage, current, and power output levels designed to address a wide range of sourcing requirements for characterizing components, circuits, modules, and complete devices:

- Model 2200-20-5: 20V, 5A, 100W
- Model 2200-30-5: 30V, 5A, 150W
- Model 2200-32-3: 32V, 3A, 96W
- Model 2200-60-2: 60V, 2.5A, 150W
- Model 2200-72-1: 72V, 1.2A, 86W

High output accuracy

The voltage output accuracy of Series 2200 power supplies is specified at 0.03%; their current output accuracy is 0.05%. Both specifications are significantly better than those of competitive general-purpose supplies. In addition, their high output (1mV) and measurement (0.1mA) resolution makes them well-suited for characterizing low power circuits and devices in applications such as measuring idle mode and sleep mode currents to confirm devices can meet today's ever-more-challenging goals for energy efficiency. Remote sense terminals on the back panel and less than 5mVp-p noise help ensure that the voltage programmed is the voltage that the supply actually outputs.

Flexible operation

Series 2200 supplies include a variety of features designed to enhance operating versatility. For example, each model provides 40 onboard memory locations for storing frequently used test setups for later recall and reuse. In addition, a built-in list mode function supports the programming and storage of up to seven custom test sequences of up to 80 steps.

Protection for the devices under test (DUTs)

Several Series 2200 features help protect DUTs from damage during testing, including a programmable voltage limit value that prevents the supply from outputting excessive voltage (even if a voltage higher than the limit is entered into the instrument) and a programmable over-voltage function that causes the output to drop to less than 1V if the over-voltage limit is reached. These limits are in addition to the current limit setting function, which controls the level of current that can flow into the DUT.

Superior ease of use

Series 2200 power supplies can be controlled easily over either a standard GPIB or USB interface. The USB interface is test and measurement class (TMC) compliant, so users can employ the standard SCPI command syntax.

Additional information on the Series 2200 family is available on Keithley's website: <http://www.keithley.com/data?asset=55901>.

Rohde & Schwarz ZNB Multiport Network Analysis

The latest generation network analyzer from Rohde & Schwarz now also comes with four test ports and a second internal generator. Users who need to characterize multiport DUTs, mixers and amplifiers will benefit from the extremely wide dynamic range, short measurement times and exceptionally easy operation.

The new four-port R&S ZNB models cover the frequency ranges from 9 kHz to 4.5 GHz or 8.5 GHz. Rohde & Schwarz has designed the powerful instruments for demanding applications in the production and development of RF components with multiple ports. Two internal signal sources and a frequency-converting mode enable comprehensive measurements on mixers or amplifiers. Using mixed-mode S-parameter measurements, the R&S ZNB fully characterizes even balanced DUTs such as SAW filters used in mobile phones.

The four-port network analyzer can also be used to measure high-blocking duplex filters, couplers, splitters or isolators. They need only be connected once to the R&S ZNB. In addition to mixer conversion loss and impedance matching, users can determine virtually any secondary measured quantities, including crosstalk between ports. Thanks to the R&S ZNB's second internal source, intermodulation measurements on amplifiers can be configured with a single, compact instrument – and considerably faster than would be possible using an external generator.

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NEW PRODUCTS AND SERVICES

All R&S ZNB models feature excellent performance characteristics: a wide dynamic range of more than 140 dB, low trace noise, IF bandwidths of 1 Hz to 10 MHz and an output power of up to +13 dBm, which can be lowered electronically by 100 dB. The network analyzer therefore ensures high throughput and short measurement times, which makes manual adjustment significantly easier, even with high signal attenuation. The R&S ZNB offers a sweep time of only 4 ms for 401 points. Its magnitude and phase drift are very low, resulting in excellent temperature and long-term stability. This makes it possible to perform precise measurements without recalibration over an extended period of time.

All instrument functions are accessible in no more than three operating steps via the soft panel. A toolbar and drag & drop functionality allow users to configure the R&S ZNB very quickly. They can switch between measurement setups at the touch of a finger. Traces and measurement channels can be configured and combined as desired. Users can therefore display

results in a clear and straightforward manner even for complex measurements.

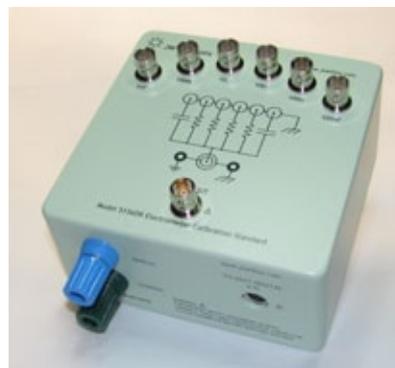
The R&S ZNB with four test ports up to 4.5 GHz or 8.5 GHz is now available from Rohde & Schwarz: <http://www.rohde-schwarz.com>.

JW Solutions - 5156DR Electrometer Calibration Standard

With the Keithley model 5156 being discontinued in 2004, there has been a continued interest in supporting a variety of high resistance – low current meters including models 6430, 6485, 6487, 6514, 6517A and many more. The demand for a direct, drop-in replacement has steadily increased and has driven the development of the new model 5156DR.

The model 5156DR is smaller in size and offers improved specifications as a result of improvements in commercially available high resistance technology combined with additional research, unique design & development efforts and extensive testing.

The model 5156DR can be ordered



directly online and is normally in stock. Each unit is delivered with a certificate of conformance and instruction manual. NIST traceable certificate can be ordered separately.

Custom values and stand alone resistor standards are available upon request. Call (407) 615-1950 or email info@jswilley.com for more information. <http://www.jswilley.com>

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Preparation and Use of an Ice-Point Bath

By Jerry L. Eldred

Training Objective: To demonstrate the basic methods of creating an ice-point bath.

Introduction

Calibration of many types of temperature sensors and thermometers require an accurate 0.0 °C reference. Throughout history, the point at which water freezes has been the center of temperature measurement; and has been measured using an ice-point bath. But since inception of the International Practical Temperature Scale of 1948 (IPTS-48), and modern technology's need for greater accuracy, the ice-point bath was replaced by the more accurate and reproducible Triple Point of Water Cell at +0.010 Degrees Celsius.

A proper ice-point bath is made up of fine shaved ice with just enough water to fill in the voids. Using pure water, uncertainties of +/-0.010 °C can easily be achieved; using good technique, uncertainties may be improved to +/-0.002°C; and under the most ideal conditions, uncertainties as low as +/-0.0001 °C have been produced. However, inattention to important details (such as water purity) will easily offset bath temperature by enough to render it invalid.

So although ice-point bath uncertainties do not approach those of the Triple Point of Water (as low as +/-0.000070°C), the simplicity and low cost of a well made ice-point bath continue to make it a useful alternative where it meets uncertainty needs. For this reason it will likely be used for many years to come.



Figure 1. Freshly shaved ice using high power food processor/blender (consistency of 'snow').

Ingredient / Equipment List

- Distilled Water, Reagent Grade IV or purer
- Plastic Ice Cube Trays, new
- Plastic Ice Bin, new with cover (optional)
- Stirring Rod (stainless steel or plastic), similar diameter to temperature sensor, for making insertion wells and stirring the ice-point bath
- Latex or Plastic Gloves
- Laboratory Stand to hold temperature sensor as needed
- Utensils, Plastic or Stainless steel (for handling ice, as needed)
- Dewar Flask, Cylindrical (about 8 cm inner diameter and 36 cm inner depth)
- Dewar Flask, Large Cylindrical (about 16 cm inner diameter and 40 – 60 cm inner depth) optional
- Rubber stopper to thermally seal the Dewar flask.
- Siphon tube, about 24 – 36 inches (as needed)
- Ice Shaver, new (a high power blender with food processor attachment is a low cost alternative for infrequent use; at least 6A/120V recommended). A lab grade shaver is recommended for heavy use.

Note: "new" means never been used for any other purpose; thus minimizing possibility of cross-contamination.

Preparation of the Ice

Thoroughly rinse equipment and utensils with pure (distilled) water prior to use. Plastic or latex gloves should be worn for the entire process.

Commercial block ice made from distilled water may be used, and is considered adequate for most applications. When using block ice, rinse surface with distilled water, use only the outer parts of the block that appear perfectly clear. Contaminants may concentrate at the center of the block, and should not be used. Discard any translucent, opaque or discolored areas.

Locally made ice cubes made from Reagent Grade IV distilled water (or better), and frozen in new plastic ice cube trays may be used. Follow a similar procedure to the above, using only clear ice cubes, discarding any with observable impurities.

Water purity is a primary contributor to ice-point bath accuracy. Tap water should not be used, as the level of contaminants is variable and unknown. If tap water must be used, it is presumed to be of lesser and unknown accuracy. If possible in such circumstances, verify the water's resistivity; at 10 °C its resistivity should be greater than $0.5 \times 10^6 \Omega \cdot m$. Additionally, inspect for a clear, colorless appearance. Translucent or milky ice should not be used, as ice-point accuracy is certainly degraded.

Pre-chill plastic ice bin and food processor attachment in a freezer; and liquid distilled water in refrigerator.

If using block ice, break into chunks that will fit into food processor (unless using ice cubes). Then shave in the food processor to a fine, snow-like texture (crystals should not exceed 1 mm in diameter), as shown in Fig. 1.

Dump the shaved ice into the pre-chilled plastic ice bin, and store in freezer while each batch of ice is being shaved (to preserve its frozen state).

Preparation of the Ice-Point Bath

The ice-point bath is prepared in a vacuum sealed Dewar flask to maximize thermal stability and isolation. Optionally, the Dewar flask is inserted into a larger, outer Dewar flask (or plastic ice bucket) to improve thermal isolation.

Fill the Dewar flask about one-third full with cold, distilled water. Add crushed ice until the flask is full (leaving room to place the stopper on the flask while it stabilizes. Insert the siphon tube until its tip is at the bottom of the flask.

Stir the mixture of ice and water ("slush"), and pack down. When the slush is depressed, the top should be wet ice (not dry snow or water). Periodically stir the mixture, press the top and drain excess water with the siphon tube.

Note: Periodic removal of excess water with siphon tube is critical for maintaining temperature accuracy of the ice-point bath. Since water is

most dense at 4 °C, the melted ice will sink to the bottom of the flask as it warms to that temperature. For that reason, regular removal of excess water from the bottom of the bath is critical.

Using the remaining shaved ice and cold water, fill a large, outer Dewar flask (or ice bucket) to an appropriate level with slush to allow the smaller flask to be fully inserted, and provide further thermal isolation (Fig. 2).



Figure 2. Fully prepared ice bath. Smaller dewar flask (center) is placed in a secondary bath in larger outer dewar flask to provide maximum thermal isolation and stability.

Once the ice-point bath has been prepared and inserted in the outer flask, cover with a stopper and allow it to stabilize for at least fifteen minutes.

Preparation of Temperature Sensor for Ice-Point Calibration

Clean the temperature sensor with distilled water before insertion into the ice-point bath.

Insert the temperature sensor into a secondary chilled bath (made from distilled water) to pre-cool it prior to ice-point calibration (this may be done in the larger, outer flask when used).

Leave the temperature sensor in the secondary bath long enough to stabilize near ice-point temperature.

Clean the stirring rod and place it in the secondary pre-cooling bath and allow it to pre-cool.

Calibration of the Temperature Sensor in the Ice-Point Bath

Remove the stopper from the ice-point bath immediately prior to making calibration measurements.

Press ice-point bath surface to check for excess water, and remove as needed using the siphon tube. Stir the bath to assure consistent texture (proper mixture of shaved ice and water), then re-pack.

Insert pre-cooled stirring rod to an adequate depth to form an insertion well for the temperature sensor. Return the stirring rod to the pre-cooling bath when done.

Insert the sensor to an appropriate depth and repack the slush around it. Ensure that the end of the sensor is completely surrounded by slush. Sensor should not reach the bottom of the ice-point bath, or to liquid that may form near the bottom (liquid accumulation should be periodically removed with the siphon). Leave the sensor in the ice-point bath for at least 15 to 20 minutes to reach thermal equilibrium, before making measurements.

Note: Silicon rubber insulator or a rubber laboratory stopper with appropriate sized hole in the center may be used to insulate the top of the ice-point bath.

Monitor sensor for at least three readings spaced about two to three minutes apart, and observe there is no drift in measured temperature. Once stable readings have been observed, make calibrated measurements as needed, and remove from the ice point bath.

Some calibrations may require using an external monitor to validate ice-point bath temperature. Where this is a requirement, the sensor being calibrated and external monitor should both be allowed to stabilize together (as described above) with probe tips in close proximity. When an external monitor is used, its measured temperature is to be used as the actual calibration temperature.

Notes About IEC/ISO17025 Compliance

This procedure is not intended to fully cover IEC/ISO17025 requirements. But a basic requirement when using an ice-point bath in the context of accredited calibrations (or where documented measurement uncertainty is needed) is an appropriate external monitor to assure the actual temperature of the ice-point bath. For example, if measuring zero degree offset of a thermocouple sensor, an RTD or SPRT of adequate accuracy needs to be placed in the bath and properly included as a part of the measurement uncertainty.

Also, although this procedure is intended as a convenient tool to assist metrologists in basics of ice-point bath preparation, IEC/ISO17025 requires that they be prepared and used in accordance with national or international standard methods (listed in Technical References below).

Technical References

1. Measurement Standards Laboratory of New Zealand, "Technical Guide 1 - The Ice Point," <http://www.msl.iir.cri.nz/>.
2. B. W. Mangun, "Reproducibility of the Temperature of the Ice Point in Routine Measurements," NIST Technical Note 1411, June 1995.
3. "Standard Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature," ASTM E563-02.
4. BIPM "3.2.1.1 Preparation of the Ice Point," *Techniques For Approximating the International Temperature Scale of 1990*.

Jerry L. Eldred is Lead Metrologist for Evans Analytical Group (EAG) Calibration Services in Austin, Texas.



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An Introduction to Mass Metrology in Vacuum

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Mass metrology carried out in atmospheric pressure air is suitable for most every critical application. However, mass metrology in vacuum is important in several current research projects. Performing mass measurements in a vacuum environment eliminates the need for air buoyancy corrections and hence allows a more precise determination of true mass. In addition, the major experiments for redefining the kilogram in terms of a physical constant nature, the watt balance and the Avogadro project, both operate in vacuum. A kilogram that redefined by these experiments will require making the transfer from mass in vacuum to mass in air. Therefore, understanding mass metrology in a vacuum environment is essential for doing the research necessary to maintain world-class mass standards. In this paper, we present an introduction to the techniques and apparatus needed for vacuum mass metrology and discuss some of the work in this area being done at NIST¹.

Introduction

Vacuum mass metrology enables the most precise experimental measurement of the “true mass” [1] of a standard mass artifact by eliminating the need for the air buoyancy force correction and its associated uncertainty. Vacuum mass metrology has received great interest in connection with the internationally coordinated efforts to redefine the SI unit of mass, the kilogram, in terms of an unvarying physical constant. In this regard, two experiments performed in vacuum have emerged as leading candidates: the watt balance, which measures the Planck constant [2, 3] and the Avogadro constant project [4]. Both of these experiments will produce a vacuum-reference definition of the kilogram that will have to be tied to the current International Prototype Kilogram (IPK) [5, 6] in order to maintain continuity and stability for mass metrology that is practiced in air. Several efforts are underway to develop a *mise en pratique*, or practical method of disseminating the vacuum-based kilogram to air [7, 8, 9] all of which require high precision mass balances to operate within an easily reproducible vacuum environment.

The ceaseless demands of the semiconductor industry over the past 40 years have enabled vacuum technology to evolve to the point where high quality materials and components are readily available world-wide at reasonable expense. There are so many off-the-shelf choices of plumbing components, construction materials, pumps, pressure gauges, sealing systems, valves, and feedthroughs that even the most well intentioned designer can make critical mistakes that will compromise the performance of the vacuum system and ultimately affect the quality of the mass measurements made within. It is our purpose to present the basics of vacuum technology required for sound design and

construction of a vacuum system suitable for use in mass metrology. We will assume that a high precision, vacuum compatible mass balance is available and that provisions exist for mounting it in the vacuum chamber.

The Vacuum Environment

Degree of Vacuum

“Vacuum” is a quantitative term used to describe the absence of air pressure within a given volume. It is inversely related to the quantitative term “pressure,” which is a measure of force per unit area. Therefore, we cannot talk about “measuring vacuum,” as it is not a quantitative term, but instead we must measure pressure either directly or indirectly. The SI unit of pressure [10] is the pascal, which is defined as one newton per square meter ($1 \text{ Pa} = 1 \text{ N/m}^2$). There are many non-SI pressure units, including the torr and millibar ($1 \text{ Torr} = 133.322 \text{ Pa}$, $1 \text{ mbar} = 100 \text{ Pa}$) in common use, but this paper will use the pascal and other SI units exclusively. The terms “high vacuum” and “low pressure” are used in common parlance with nebulous meanings. Table 1 relates common understanding of degree of vacuum to quantitative pressure values [11].

In order to calculate the degree of vacuum required for mass metrology, we consider the fact that air or any other gas will exert a buoyant force on a mass artifact that is equal to the weight of the volume of gas that is displaced by the artifact. Assuming the density of stainless steel is 8.0 g/cm^3 , the volume of a 1kg stainless steel mass artifact is approximately 125 cm^3 ; in atmospheric pressure air, the weight of this displaced volume is about 0.15 g. In order to reduce the buoyant force to a negligible level, its magnitude should be much smaller than the uncertainty of the artifact’s

¹ Certain commercial equipment, instruments, or materials are identified in this document. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

Degree of Vacuum (abbreviation)	Pressure Range (Pa)
Low	$10^5 > P > 3.3 \times 10^3$
Medium	$3.3 \times 10^3 \geq P > 10^{-1}$
High (HV)	$10^{-1} \geq P > 10^{-4}$
Very high	$10^{-4} \geq P > 10^{-7}$
Ultrahigh (UHV)	$10^{-7} \geq P > 10^{-10}$
Extreme high vacuum (XHV)	$10^{-10} > P$

Table 1. Pressure ranges for degree of vacuum.

mass measurement. Assuming the expanded uncertainty [12] on a 1 kg artifact is approximately 50 μg , and that a buoyancy contribution of one percent or less of this value is negligible, the ideal gas equation can be used to calculate the desired pressure within the vacuum vessel, which turns out to be about 0.1 Pa (or less). This is the upper limit of the high vacuum (HV) regime, as shown in Table 1 above.

Another important consideration for vacuum mass measurement is the number density and species of the gas molecules inside the weighing chamber. Assuming a temperature of 22 °C, a pressure of 10^{-1} Pa, and a sticking probability of unity, a monolayer air will form on the chamber surface in about 2.5 ms. Air molecules are very weakly physisorbed (desorption energy less than 40 MJ/kg mole) onto a metal surface such as found on the inside of a vacuum chamber and are easily pumped away. At 10^{-1} Pa, there is a density of about 10^{13} molecules/cm³, which is composed mostly of water vapor for an unbaked chamber that has recently been exposed to the atmosphere (atmospheric air at 22 °C and 50% relative humidity has a water vapor partial pressure of about 840 Pa). Water molecules are strongly chemisorbed (desorption energy of 96 MJ/kg mole) to a metal surface and have residence times on the order of 30 hours. At 10^{-1} Pa a monolayer of water adsorbs to a metal surface in less than 2 ms. Up to 100 monolayers of water may build up on the surface when exposed to atmospheric pressure air [13] but the binding energy between water monolayers is much less than between water and the metal surface, meaning that almost all but the last monolayer will readily pump away. One hundred monolayers of water molecules on a 1 kg weight can add more than 50 μg to the total mass of the artifact; in addition, multiple monolayers of water on the other metallic parts of the balance's weighing mechanism will add mass in proportion to their area as well. For this reason, it is necessary to allow the vacuum chamber to pump until all but the last monolayer has been removed, which may take several days at room temperature (22 °C). This can be monitored by making multiple weighings over time until the artifact's mass is stable to the desired level. Note that the artifact will appear to lose mass as water desorbs and is pumped away. The most effective way of ridding a metal chamber of adsorbed water is by raising the temperature to

over 100 °C for an extended period of time; even a slightly elevated temperature of 70 °C will hasten the desorption process. However, this is not possible or advisable for many mass-in-vacuum measurement systems due to the delicate construction of the balance. Most elastomeric seals will tolerate baking temperatures to at least 80 °C, with some materials such as Viton® A [14] able to operate at temperatures well in excess of 100 °C.

The considerations presented above suggest that the vacuum balance and mass artifacts under test be subjected to several days of pumping prior to making mass measurements in order to minimize the amount of adsorbed water on the balance and artifacts' surfaces.

Vacuum System Design and Construction

Several factors must be considered when choosing the vacuum technology used in a given application. Among these are desired ultimate pressure, required degree of cleanliness, whether or not the system will be baked, frequency of cycling to atmospheric pressure, the need to insert and/or remove things while under vacuum, and of course monetary budget. This final criterion is critical; vacuum design is very much driven by the "level of effort" required to achieve system goals. One can build an inadequate system by spending too little or too much for the wrong technology for a given application.

The equilibrium pressure within a vacuum chamber is a balance between the flow of gas into the chamber and the rate at which it is removed. The time required to evacuate the chamber to a given pressure depends upon the chamber volume, the pumping speed of the pump(s), the conductance of the components (tubes, valves, etc.) between the pump(s) and the vacuum chamber, and the influx of gas through the mechanisms of leaks in the chamber, outgassing of materials, virtual leaks (trapped gas within tiny volumes that slowly pumps away), and permeation of atmospheric gases through seals. Vacuum weighing introduces the additional gas load due to the mass balance, which in most cases is unknown and difficult to estimate. The manufacturer of the mass balance that is intended for use in vacuum should take appropriate steps

to eliminate as many sources of gas within the balance as is practicable. These steps may include eliminating the application of paint on the outside surface, using high vacuum compatible insulation on wires, eliminating pockets of trapped gas by venting screws, and minimizing the use of “gassy” materials such as plastics, elastomers, and certain lubricants. Given the ambiguity with which the mass balance gas load is known, it is prudent to conservatively estimate the gas load so that sufficient pumping can be incorporated to permit an equilibrium pressure in the desired range. This paper cannot give an in-depth treatment of all the vacuum system design issues, but references will be given throughout so reader can delve deeper into any particular topic.

Materials for Vacuum Systems

First and foremost, a vacuum chamber must be strong enough to support the differential weight of the earth’s atmosphere, which is about 10 N/cm² (15 lb/in²). Modern vacuum chambers are typically made from metal, either stainless steel or an aluminum alloy, and may be 5 mm or more thick depending on the surface area. The ASME has published standards for the wall thickness of cylindrical vessels under external pressure [15]; easy to use charts that provide wall thickness as a function of material and diameter for cylindrical and spherical chambers may be found in the references [16, 17]. Engineers for commercial vacuum component manufacturers can also assist in selecting the right material and thickness for a given chamber. Both aluminum and stainless steel offer high strength, cleanliness, weldability for easy modification, and world standard “building block” component systems that make system design easy. The glass vacuum systems that were common prior to 1970 are exceedingly rare nowadays except for highly specialized applications or demonstrations that use glass bell jars. There are many grades of stainless steel, not all of which are suited for use in vacuum chamber construction [18]. The properties of good weldability, high temperature bakeability, relatively low cost, and low contaminant outgassing make 304 stainless steel a good choice. A variety of weldable flanges and other fittings are available in a wide range of standardized sizes and sealing systems [19], making component selection very versatile and convenient. Aluminum of the 6000 series is also used for vacuum chamber construction but is generally restricted to medium and high vacuum systems that use elastomer sealed flanges; ultrahigh vacuum aluminum and stainless steel bimetal seals are also available at greatly increased cost [20].

In theory, a vacuum chamber can be made in any shape, though easy to fabricate vessels such as right circular cylinders are most common. In applications where minimizing surface area (and hence outgassing) is important, spherically shaped chambers are used. Special surface treatments such as electropolishing provide a

high luster finish that further reduces surface area. This is important for ultrahigh vacuum applications, but not critical for high vacuum applications. A variety of coating processes are also available to reduce outgassing [21] or enhance pumping, but again, these are geared to UHV applications.

Every vacuum system contains an experiment that must be monitored and interacted with from outside the vacuum environment. Environmental variables such as temperature and pressure, electrical connectors, and various types of motion feedthroughs are some commonly used items that need some way attaching to the vacuum chamber. As mentioned earlier, ports and flanges of many shapes, sized, and functions are commercially available to interface to a vacuum chamber through standard fittings. The designer should consider the type and quantity of feedthroughs and ports needed for temperature, pressure, and humidity measurements before the chamber is constructed.

Electrical grounding is a very important safety and performance consideration. A metal chamber should be ground to a “true” ground (earth) that doesn’t fluctuate with electrical noise from other instruments. Safety grounds found on U.S. 3-pronged outlets are typically inadequate. Direct connections to a metal cold water pipe that is buried in the earth is best. All other instruments in use should have the same ground as the chamber to avoid ground loops.

Sealing Options

Mass metrology requires a high vacuum system that can be exposed frequently to atmospheric pressure. The bakeability of the chamber is limited by the ability of the mass balance, artifacts, and seals to withstand heat. In general, it is not advisable to expose high precision mass comparators to elevated temperatures. At least one manufacturer [22] of these instruments specifies the operating temperature range to be between 17 °C and 30 °C (with an upper limit of 27 °C for some models), so effective baking of the chamber to remove adsorbed water is not possible. Given these constraints, elastomer sealed vacuum flanges are sufficient using either aluminum or 304 stainless steel construction.

Elastomer O-rings vary in their chemical composition. Viton® A, Neoprene, Kalrez® [23] and Buna N (also called Nitrile) are common elastomers used for vacuum seals [24]. Of these, Viton® A, a fluoroelastomer, is generally considered to be the “cleanest” in terms of outgassing rate and the most versatile, as it can be used at elevated temperatures of up to 150 °C. Viton® A and many other elastomers are hygroscopic, absorbing moisture from the air at atmospheric pressure and then releasing it under vacuum. It will also allow the permeation of atmospheric gasses into the chamber while under vacuum; for these reasons, the water outgassing rate of elastomer seals limits the ultimate pressure of the vacuum chamber and plays a large role in pump specifications. The outgassing rate

of elastomer seals can be reduced by prebaking O-rings to 100 °C [25]. Unlike metal sealed systems, water cannot be eradicated from elastomer sealed systems due to the constant influx from permeation of atmospheric water. For this reason, water is the primary component of the elastomer sealed high vacuum environment. This is an important consideration in mass metrology, as water adsorbed onto mass artifacts is the subject of much research [26, 27, 28, 29].

The ISO and associated “Quick Flange” (QF, KF, or NW) sealing systems are convenient for use in high vacuum chamber construction and are available through many manufacturers. Photos of each of these sealing systems are shown in Figure 1. Both ISO and QF systems use an elastomer O-ring held in place by a metal retaining ring; the retaining ring/O-ring assembly is placed between two flat flanges and clamped together via nuts and bolts, claw clamps, or in the case of QF, special clamps that encompass the perimeter of the mating flanges. The clamps should be tightened to the specified torque values for ISO clamps. For QF clamps, there is a wing nut or a “T” shaped nut that is used to tighten the clamp; this should never be tightened any more than finger tight. In no situation should tools like pliers or a wrench be used to tighten these fittings. Over-tightening can cause the O-ring to exceed the compression ratio specified for sealing and lead to a faulty seal. Vacuum grease should not be applied to the O-rings of these seals; no sealing issues should arise if all flange surfaces are clean, parallel, and free of burrs, scratches, and other defects. The use of grease may actually inhibit a good seal, as the proper O-ring compression may not be achieved with the specified clamping torque. In general, the use of vacuum grease should be avoided as it can migrate into the vacuum chamber and possibly contaminate the weights, comparator parts, or other delicate instrumentation.

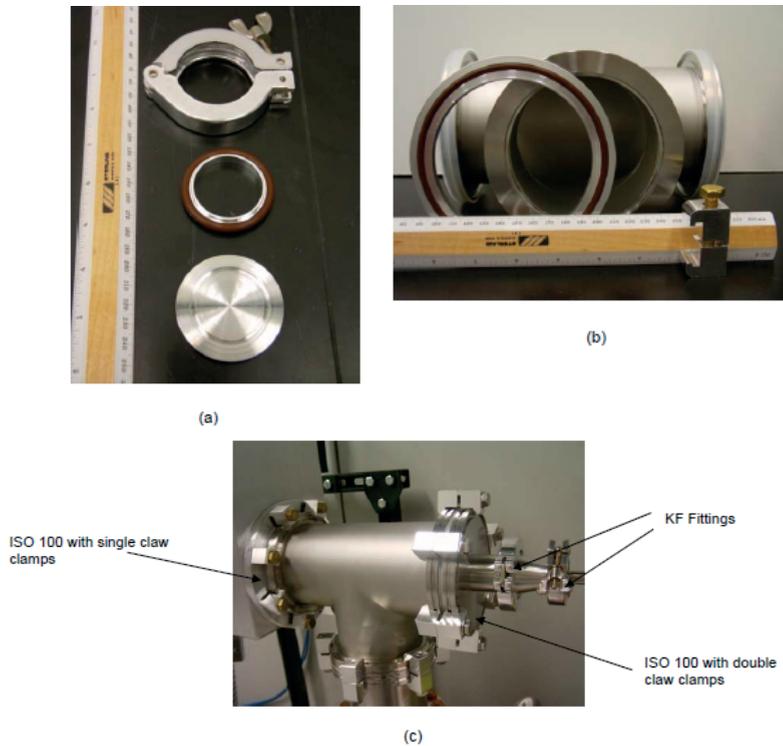


Figure 1. Photos of elastomer sealing systems: (a) KF or NW, showing (top to bottom) clamp, retaining ring and gasket, and blank fitting; (b) ISO 100, showing (left to right) retaining ring and gasket, tee-flange, and double claw clamp; and (c) a vacuum tee in the authors’ lab having both NW and ISO 100 flanges.

Sources of Gas in Vacuum Systems

There are many sources of gas in a real vacuum chamber. The following are the most common sources [30]:

- Outgassing from adsorbed layers
- True leaks (holes and cracks)
- Porosity of metals
- Virtual leaks (pockets of gas trapped by screws, spacers, etc.)
- Permeation from internal surfaces (elastomers, metals, etc.)
- Outgassing of surface contamination (hydrocarbons)
- Pressure gauges (especially hot cathode)
- Backstreaming from the pumping system

It is beyond the scope of this paper to fully describe each of these, and the reader is encouraged to consult the references listed at the end. A diagram showing the major sources and sinks of gas in a vacuum chamber is shown in Figure 2. In an unbaked metal chamber with metal seals and sufficient pumping, it is possible to remove most gas molecules except the chemisorbed water on the chamber walls. This typically results in a base pressure of approximately 10^{-6} Pa. If elastomer seals are used, a steady stream of atmospheric gases will permeated the O-rings and will limit the base pressure to around 10^{-5} Pa, and higher than this depending on the number and size of the O-rings. For an unbaked chamber, water will desorb from the metal surfaces within the chamber at a very slow rate, thus adding to the gas load of the elastomers. In order to minimize the outgassing rate inside the chamber,

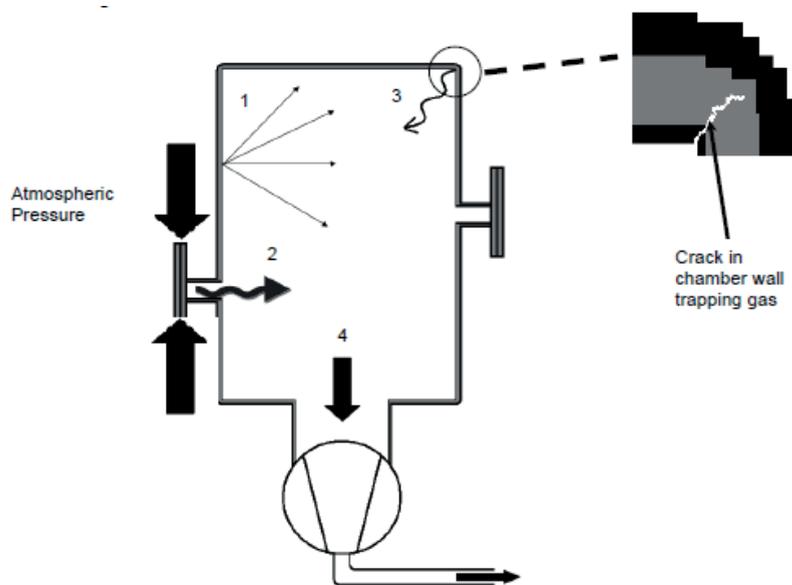


Figure 2. Illustration of the major sources and sinks of gas in a vacuum chamber; arrows represent the flow of gas molecules (1) desorption and outgassing from surfaces; (2) permeation from the atmosphere through seals; (3) “virtual leaks” of trapped gas; and (4) evacuation through chamber pump(s).

the size and number of elastomer gaskets should be kept to a minimum, as the outgassing rate is proportional to the total surface area of all the gaskets combined. Don't use large diameter ports where smaller ports will do. Generally it is a good idea to have a few spare ports for future considerations, but too many will increase the chamber's gas load if elastomer seals are involved.

A general expression of the equilibrium pressure within the chamber, P_{eq} is:

$$P_{eq} = P_{ult} + \frac{Q_{og}}{S_{net}} \quad (1)$$

In (1), P_{ult} is the ultimate pressure (lowest possible) for the pump, Q_{og} is the total outgassing rate in $\text{PA m}^3/\text{s}$, and S_{net} is the net pumping speed for the system in m^3/s . This equation tells us that there are two ways to lower the equilibrium pressure in the chamber: by reducing the outgassing rate and/or by increasing the net pumping speed.

Contaminants such as hydrocarbons from pump oil, dirt, and fingerprints will all outgas and limit the chamber's ultimate pressure. To minimize

contamination, the inside surface of the vacuum chamber should be cleaned with an alkali detergent and thoroughly rinsed with deionized water. Any parts on the inside of the chamber should be cleaned with detergent and deionized water or reagent grade organic solvents such as acetone and ethyl alcohol if grease or oil is present. Note that parts should be completely dried after cleaning and prior to installing within the vacuum chamber. Any solvent trapped in the vacuum chamber during pumping will present an enormous gas load to the pump and will greatly lengthen the time needed to reach an acceptable pressure. For this reason, elastomers should never be cleaned with solvents, as they are absorbed by the elastomer and will outgas in the vacuum chamber during pumping. In order to keep a vacuum chamber clean, precautionary measures need to be taken. The laboratory environment should be kept clean of dust and dirt that could migrate into the vacuum chamber when a port is opened. The length of time a chamber is open to the atmosphere should be minimized

to reduce the quantity of gas that is adsorbed and absorbed by chamber components. While working on the inside of the chamber or on any parts that will be put inside the chamber, clean, lint-free gloves should be worn to prevent transfer of dirt and oil from fingers and hands. Latex or nitrile gloves such as those worn in the health profession work well, and it is best to remember that gloves are only as clean as the last item they touched. For prolonged work inside a vacuum chamber, change gloves often. Powdered gloves should never be worn. If vacuum ports must be open for long periods of time, then it is prudent to cover them with a plastic protective cap or with clean aluminum foil. For high vacuum applications such as mass measurements, commercial aluminum foil is sufficiently clean; however ultra-high vacuum applications require a higher degree of cleanliness, and traces of oils used for lubricating the foil during the rolling process may contaminate the vacuum system and limit the achievable base pressure. Venting from vacuum to atmospheric pressure should be done with a clean, dry, non-reactive gas. Filtered air and nitrogen are good choices.

Pumping Considerations

Types of Vacuum Pumps

The purpose of a pumping system is to remove gas from the vacuum chamber. The rate of gas removal should be much greater than the flow rate of gas into the chamber in order to achieve the desired pressure in a reasonable amount of time. There is no single pump that can evacuate a chamber from atmospheric pressure down to high vacuum; instead, a combination of different pumping technologies must be used. Initial evacuation from atmospheric pressure down to about 1 Pa, sometimes referred to as “roughing,” can be obtained using a variety of primary or “mechanical pumps” including oil sealed rotary vane pumps, piston

pumps, diaphragm pumps, or scroll pumps. For details on each of these designs, the reader is referred to recent texts on vacuum technology [31, 32, 33]. Other than the oil sealed rotary vane pump, the other pumps mentioned above are “dry pumps,” meaning they are oil free; this is a big advantage in that these pumps do not require the use of a trap to prevent condensable oil vapors from backstreaming from the pump to the vacuum chamber. Contamination from pump oil backstreaming can be catastrophic for the vacuum chamber and any equipment inside the chamber, such as mass balances and weights.

In choosing a primary pump, the variables are pumping speed, cleanliness, and cost. For mass metrology in vacuum, the use of any type of primary pump containing sealing oil is *strongly* discouraged due to the possibility of oil contamination from backstreaming. Scroll pumps [34] have the highest available pumping speed of any dry roughing pump, are compact, and are competitively priced with oil sealed rotary vane pumps. Maintenance of the tip seals must be performed yearly for heavily used scroll pumps, but replacement tip seal maintenance kits are available from vendors and are easily installed. An isolation valve between the scroll pump and the vacuum chamber (or high vacuum pump) must be part of the design of the pumping system in order to prevent possible particulate migration from the scroll pump to the vacuum chamber in the event of loss of electrical power [35].

A scroll pump has an ultimate pressure of about 5 Pa; to achieve lower pressures, it is necessary to use a high vacuum pump in combination with the scroll pump. Modern turbomolecular pumps having a molecular drag stage [36], i.e., turbodrag pumps, are efficient, clean, reliable, quiet, and can be mounted in any orientation. Turbodrag pumps come in many sizes and are available in pumping speeds from a few liters per second to many thousands of liters per second. Depending on the size of the chamber and the desired pressure, a typical turbodrag pump for a mass-invacuum application will have a nominal pumping speed of 100 L/s to 500 L/s. Turbodrag pumps compress the gas at their inlets and require a backing stage to remove the compressed gas from their exhaust ports. The design shown in Figure 3 uses the same primary chamber pump as the turbomolecular pump’s backing stage.

There are other types of high vacuum pumps, but they are impractical for vacuum mass metrology. Diffusion pumps use jets of oil vapor to transfer momentum to gas molecules in order to pump them away. Cold traps are necessary to prevent oil contamination of the vacuum chamber. They also require high temperatures to vaporize the oil, which may cause thermal gradients in the vacuum system. Unlike the turbomolecular and diffusion pumps which physically move molecules from the vacuum chamber to a rough pump, ion pumps use an ion burial gettering process to remove gas molecules from the chamber. The molecules are buried in the pumping elements which have a finite ability

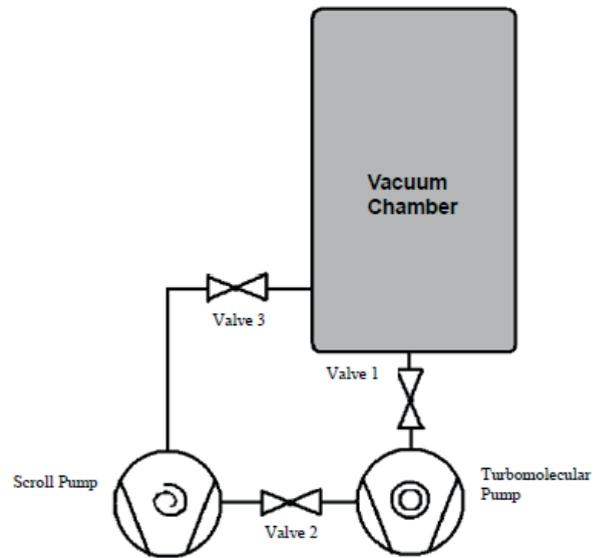


Figure 3. A vacuum chamber using a scroll pump as both primary pump and backing pump for a turbomolecular pump.

to pump gas. Therefore, ion pumps are practical only for very small gas loads, corresponding to chamber pressures typically less than 10^{-5} Pa. In addition, potentially lethal voltages of several thousand volts and very high magnetic fields are required to operate ion pumps.

Conductance

The efficacy of a given pump in evacuating a metal chamber depends not only on the design of the pump, but also on the “plumbing” used to connect the pump to the chamber. The diameters of the connecting chamber port and connecting pipes, the number and severity of bends in the connecting hardware (such as with elbows), and the total length of pipe between the chamber and the pump affect the overall pumping speed through their *conductance* [37, 38, 39].

Conductance is a measure of the speed with which a unit volume of gas is moved through a given passageway; in reference [24], Dushman refers to conductance as the rate of flow per unit difference of pressure. The units of conductance are the same as those of pumping speed, that is, volume per unit time (e.g. or m^3/s or L/s). The larger the diameter (cross sectional area) of the connecting pipe, the higher the conductance, and the faster the gas molecules will be pumped away. For good conductance, the pipe connecting the high vacuum pump with the chamber should have a wide diameter, no bends, and be as short as possible. In general, it is good practice to use connecting hardware having about the same diameter as the high vacuum pump’s inlet port. Practically, this is limited to diameters of around 100 mm (about 4 inches)

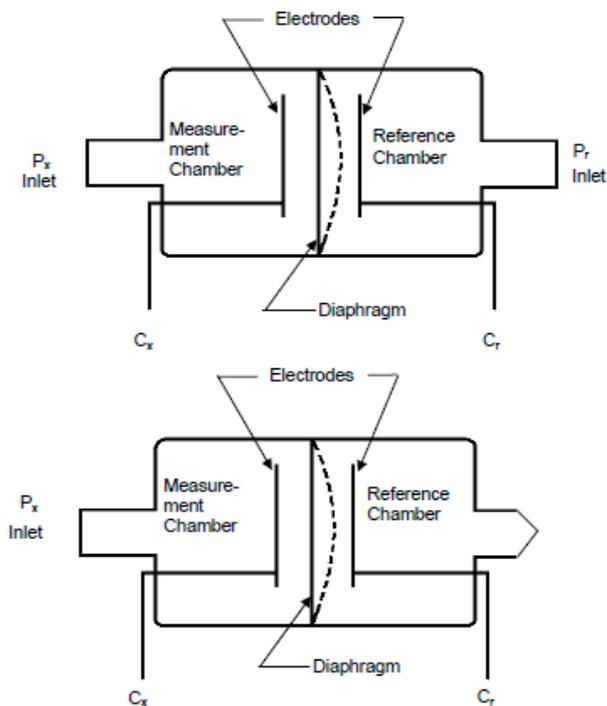


Figure 4. Diagrams of differential (top) and absolute (bottom) capacitance diaphragm gauges. P_x and P_r are the unknown and reference pressures respectively. C_x and C_r are the capacitor plates on the unknown and reference sides [40].

for ISO-100 fittings. Small diameter connecting hardware in effect “chokes” the high vacuum pump, and the effect on pumping speed is analogous to draining a swimming pool with a drinking straw.

Conductance can be maximized by fastening the inlet port of the high vacuum pump directly to an equal diameter port on the vacuum chamber. However, this is a risky proposition with mass metrology in that vibrations from the pump may couple into the sensitive mass comparator and introduce a source of instability. While it is good vacuum design practice to keep the pumps as close as possible to the vacuum chamber, vibration isolation measures may need to be considered, such as adding a bellows between the pump and chamber or simply locating the pump a moderate distance away from the chamber. Davidson [9] has eliminated the vibration problem by using a turbomolecular pump having magnetically levitated blades. Using a wide diameter pipe (100 mm is usually sufficient) to locate the pump a few meters from the chamber will reduce the effect of vibrations and heat that are generated by both the primary and high vacuum pumps. The connecting pipe should have as few bends as possible, as each bend reduces the total conductance. The effective conductance between pump and chamber may be

calculated for a given vacuum system, but the analytical expressions change as a function of gas flow regime. The formulae for making these calculations may be found in any of the vacuum technology texts appearing in the reference section. In most cases, the above sources of gas, along with component conductances and pumping speeds can be calculated, measured, or estimated with sufficient accuracy in order to create an effective design.

Measuring Pressure

Types of pressure gauges

Pressure is force per unit area, and is commonly measured over 16 decades from about 10^9 Pa to 10^{-7} Pa. To make accurate measurements over such a wide range requires many different technologies. Pressure gauges may be direct or indirect reading. In direct reading gauges, a non-gas dependent change in some physical property of the gauge occurs in response to the application of pressure. An example of this type is the capacitance diaphragm gauge shown in Figure 4 [40], in which a thin metal diaphragm stretched between two electrodes deflects away from its equilibrium position with the application of pressure; the deflection changes the capacitance between the diaphragm and the electrodes, and this change is converted to a pressure indication by conditioning electronics. Indirect reading gauges respond to some other quantity that is proportional to pressure and frequently dependent on gas species. An example of this is the thermocouple gauge which measures heat flow as a function of pressure [41]. The type of pressure gauge used in a particular application depends on the pressure range of interest, the desired accuracy of the measurement, compatibility with other apparatus, and of course monetary cost. Table 2 below lists some of the more common gauges that are useful for mass in vacuum metrology along with some of their characteristics.

Pressure Gauge Calibration

Table 2 presents accuracy estimates for uncalibrated gauges, and these estimates can vary widely according to gauge type [42]. Thermal conductivity gauges and ionization gauges may be inaccurate by as much as a factor of two. For proper use, any gauge should be calibrated prior to making measurements. A calibration will provide the user with a pressure-dependent correction factor to apply to the gauge’s indicated pressure value, as well as an uncertainty estimate on the corrected pressure values *at the time of calibration*. The uncertainty estimate is critical, and the user should not expect better results than the calibration uncertainty dictates. The user must always operate the pressure gauge in a manner consistent with its calibration; failure to do so will void the calibration. Calibration conditions to observe include using the exact same hardware as was used for the calibration (e.g., one can’t mix and match gauge controllers with gauge heads

Type of Gauge	Pressure Range	Direct/ Indirect	Gas Species Dependent?	Principle	Accuracy (uncalibrated)	Cost
Thermo-couple	1 kPa – 0.01 Pa	Indirect	Yes	Heat Transfer	± 20%	Low
Convection Enhanced Pirani	100 kPa – 0.1 Pa	Indirect	Yes	Heat Transfer	± 20%	Low
Capacitance Diaphragm	100 kPa – 10 ⁻³ Pa	Direct	No	Pressure Dependent Capacitance Change	< ± 1%	High
Piezoresistive	100 kPa – 10 Pa	Direct	No	Pressure Dependent Change in Resistance	< ± 1%	Medium
Resonant Silicon Gauge	100 kPa – 100 Pa	Direct	No	Pressure Dependent Change in Resonator Frequency	± 0.01%	Very High
Hot Cathode (Bayard-Alpert)	10 ⁻¹ Pa – 10 ⁻⁷ Pa	Indirect	Yes	Ionization of Gas Molecules	± 25%	Medium
Cold Cathode	1 Pa – 10 ⁻⁷ Pa	Indirect	Yes	Ionization of Gas Molecules	± 25%	Medium
“Wide Range”	100 kPa – 10 Pa	Indirect	Yes, over at least part of the full range.	Combination of thermal conductivity gauge or piezo resistive gauge with hot or cold cathode gauge	< 10% above 1 Pa; ± 20% below 1 Pa	Medium

Table 2. Pressure gauges suitable for mass metrology in vacuum.

in general), proper operating parameters, the same gas as the gauge was calibrated with, mounting the gauge in the proper orientation, and observing the safe pressure range of the gauge. Even with proper calibration, the response of many pressure gauges tends to drift away from calibration with time and use. Several factors influence this drift including exposure to atmospheric pressure, accidental over pressurization, rough handling, or inherent limitations of the gauge’s technology. For instance, inexpensive thermocouple gauges may not be stable to within 20% even over the course of the calibration! It is essential to choose a gauge and calibration service that is appropriate for the application. Many vendors offer calibrations that are traceable [43] to the National Institute of Standards and Technology (NIST) or other National Metrology Institutes. Only history and experience can determine how frequently your pressure gauges need to be calibrated. It cannot be overstated that when choosing a calibration service, it is prudent to be mindful of the required uncertainty for the pressure measurement application. For mass-in-vacuum metrology, the calculation above determined that the upper limit of pressure is 0.1 Pa. To measure the difference between 0.1 Pa and 0.09 Pa, it’s necessary to have better than 10% uncertainty. Typically, lower calibration uncertainty translates into higher gauge cost. In this case, paying for a gauge that can hold a 0.1% calibration uncertainty is not necessary when 1% is more than adequate.

Pressure Gauge Recommendations

The pressure measurement requirements for mass-in-vacuum call for a clean, stable gauge to monitor pressure during evacuation and to have less than 10% uncertainty at the pressure of interest where the mass metrology will be performed. In addition to uncertainty constraints, the gauge should not generate any contaminants or an excessive amount of heat. A convection enhanced Pirani gauge is adequate to a pressure of 0.1 Pa, but may be unreliable at lower pressures. In the same way, thermocouple gauges suffer from large uncertainties at their lowest range, around 0.1 Pa. Ionization gauges, both hot cathode and cold cathode, are at their high pressure limits at 0.1 Pa and are greatly influenced by space charge effects. Furthermore, there is evidence that contamination produced by the ionization process in a cold cathode gauge can influence mass measurements [44].

Capacitance manometers, or capacitance diaphragm gauges (CDG) can cover the pressure range between 10⁵ to 10⁻³ Pa, respond directly to pressure, and when calibrated have an uncertainty of 0.5% or less [45]. Multiple gauge heads are needed to cover the entire range from atmospheric pressure down to 0.1 Pa or less. This can be done using a minimum of two gauge heads having full scale ranges of 133 kPa and 133 Pa. For pressures from atmospheric to 10 Pa, a convection enhanced Pirani gauge will perform sufficiently and may save money over the use of a 133 kPa CDG. Regarding the use of CDG’s,

both differential and absolute models are available, but a differential CDG requires a separate pump to provide a low reference pressure in the high vacuum regime. Absolute CDG's have a gettering material incorporated into the reference pressure side of the diaphragm that maintains a high vacuum reference pressure.

Figure 4 shows diagrams for both absolute and differential models. One drawback of both absolute and differential CDG's is that they must be zeroed prior to use by applying high vacuum to their measurement ports. If the vacuum chamber is not capable of doing this, then a small auxiliary ion pump can be used to provide this "hard zero" when necessary. A scheme for doing this with a small ion pump is shown in Figure 5.

In summary, the types of pressure gauges to be used are a trade-off between accuracy, cleanliness, and cost. We recommend that a less accurate (and less expensive) device such as a convection enhanced Pirani gauge or a piezoresistive gauge be used to measure pressure from 100 kPa down to 10 Pa, in conjunction with a more accurate absolute capacitance diaphragm gauge of 133 Pa full scale range to measure pressure down to 10^{-2} Pa and possibly lower. A differential CDG may be used if a reference "zero" pressure is readily available. For the Pirani gauge, it is important to follow the manufacturer's instructions for initial setup, which is usually a check of the gauge readings at atmospheric pressure and at "zero pressure" (less than 10^{-2} Pa). Convection enhanced Pirani gauges need to be mounted so that the axis of the gauge is horizontal. It is also important to use the Pirani gauge to measure pressures only for the gas with which the gauge was calibrated (air or nitrogen usually) as catastrophic mistakes (falsely low or high pressure readings) can occur when using a gas having

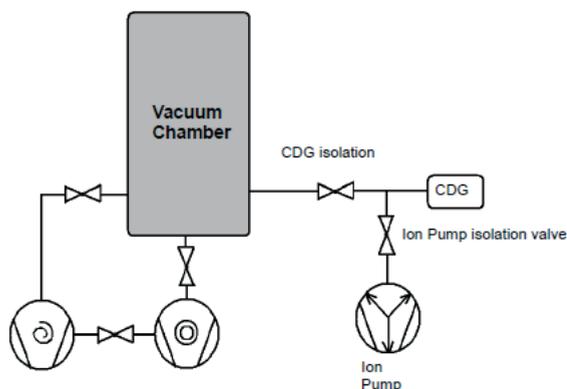


Figure 5. Auxiliary pumping system for zeroing a capacitance diaphragm gauge (CDG) prior to use. The ion pump need only have a pumping speed of 1 or 2 L/s. The CDG is first pumped down to 0.1 Pa or less by the vacuum chamber pump (CDG isolation valve open); the isolation valve is closed and the ion pump is opened to provide a hard zero sufficient for zeroing the CDG.

a different thermal conductivity than the calibration gas. Capacitance diaphragm gauges need to be properly zeroed prior to use. Their response is independent of the gas used, however, they can be damaged if they are over pressurized; follow the manufacturer's instructions regarding this issue.

Vacuum Mass Metrology at NIST

NIST is substantially involved in the efforts to redefine the kilogram and to develop a method of dissemination for the new definition. The following are brief descriptions of each of these experiments.

The Watt Balance and Avogadro Experiments Link to the Kilogram

The decision has been made to redefine the kilogram in terms of an exact value for the Planck constant [46]. As mentioned in the introduction of this paper, the watt balance and Avogadro experiments are being used to provide accurate values of the Planck constant, which would then be used to define the kilogram.

The watt balance relates mechanical and electrical powers by comparing the gravitational force on a one kilogram mass to an electromagnetic force expressed in terms of the Josephson and quantum Hall effects allowing the kilogram to be linked to Planck's constant. The Avogadro project is an international collaboration focused on measuring the Avogadro constant by relating the kilogram to the mass of a fixed number of atoms through accurate measurement of the number of atoms in a sphere of silicon weighing one kilogram.

Since the Avogadro and Planck's constants are theoretically related, measurement of one constant with one experiment can be used to calculate the other constant. For instance, measurement of the Avogadro constant from the Avogadro project can be used to calculate a value of the Planck constant which in turn can be compared to the value of the Planck constant obtained from the watt balance experiment.

Under the new definition, the mass of a kilogram artifact can be determined by placing it in a watt balance and measuring the value of the Planck constant it produces. This value, in comparison to the fixed SI value of the Planck constant will determine the mass calibration value for the artifact. However, this calibration will yield a mass value that is only valid in the vacuum environment of the watt balance; some means of correcting the vacuum calibration for use in air is necessary. NIST is currently involved in research to develop an instrument that is capable of directly comparing the known mass of a kilogram in a vacuum environment to an unknown mass of another kilogram in air using the same high precision mass comparator [8]. In this scheme, a vessel containing two adjacent chambers, one under vacuum and one at atmospheric pressure, is used. A high precision mass comparator is contained in the vacuum

vessel; this comparator can weigh an artifact in the usual way in the vacuum vessel, but can also weigh an artifact in the atmospheric pressure chamber by connecting the comparator to the artifact across the air vacuum boundary via magnetic levitation. This is illustrated in Figure 6.

The complete scenario of calibrating a mass standard in a watt balance under vacuum, transporting it under vacuum to the magnetic levitation system, and then using the magnetic levitation system to transfer the vacuum

calibration to a mass artifact in air is shown in Figure 7. This represents a possible method of disseminating the new SI definition of the kilogram to national laboratories, which then pass it on to the mass community through conventional mass metrology in air. It must be emphasized that all of these steps will be transparent to the mass metrology community that relies on calibration of primary standards by national laboratories. There will be no change in procedures or quality of calibrations as a result of the new SI kg definition.

Conclusions

We have reviewed vacuum technology design considerations that are germane to mass measurement in vacuum and recommend the following: 1) Based on the need to minimize the air buoyancy correction of the mass measurements a *maximum* chamber pressure of 0.1 Pa should be used for mass metrology. Other factors, such as the amount of water adsorbed onto mass artifacts may dictate that a working pressure lower than 0.1 Pa be used. 2) Scroll pumps are a good choice for initial rough pumping of a vacuum chamber as well as backing a high vacuum pump. They are oil-free, competitively priced and comparable in performance to oil-sealed rotary vane pumps. 3) Turbomolecular drag pumps are the best option for producing high vacuum for mass metrology. They are clean, come in a variety of pumping speeds, and enable the use of oil free backing pumps. 4) A convection enhanced Pirani gauge is economical and sufficiently accurate for measuring pressure from atmospheric pressure to 10 Pa during initial chamber evacuation. For measuring pressure below 10 Pa, a calibrated capacitance diaphragm gauge offers the necessary accuracy, has good stability, and is contaminant-free. A 133 Pa, full scale calibrated CDG can accurately measure pressures down to 10^{-3} Pa with an expanded uncertainty of less than 1%.

Realizing the new SI definition of the kilogram involves a transfer of an artifact's mass as measured in vacuum to atmospheric pressure air. The two are different due to the adsorption of contaminate molecules, mostly water, onto the mass artifact in air. NIST is working on a new method of bridging the vacuum to air boundary using a magnetic levitation technique that uses the same high precision mass comparator to weigh artifacts in vacuum and air.

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It is a pleasure to acknowledge Rick Seifarth of the Mass and Force group at NIST and Jay Hendricks of the Pressure and Vacuum group at NIST for critical readings of this manuscript and for offering suggestions for its improvement. We are also indebted to Dr. Bruce R.F. Kendall, Professor Emeritus at The Pennsylvania State University, for many years of teaching and discussions related to vacuum technology.

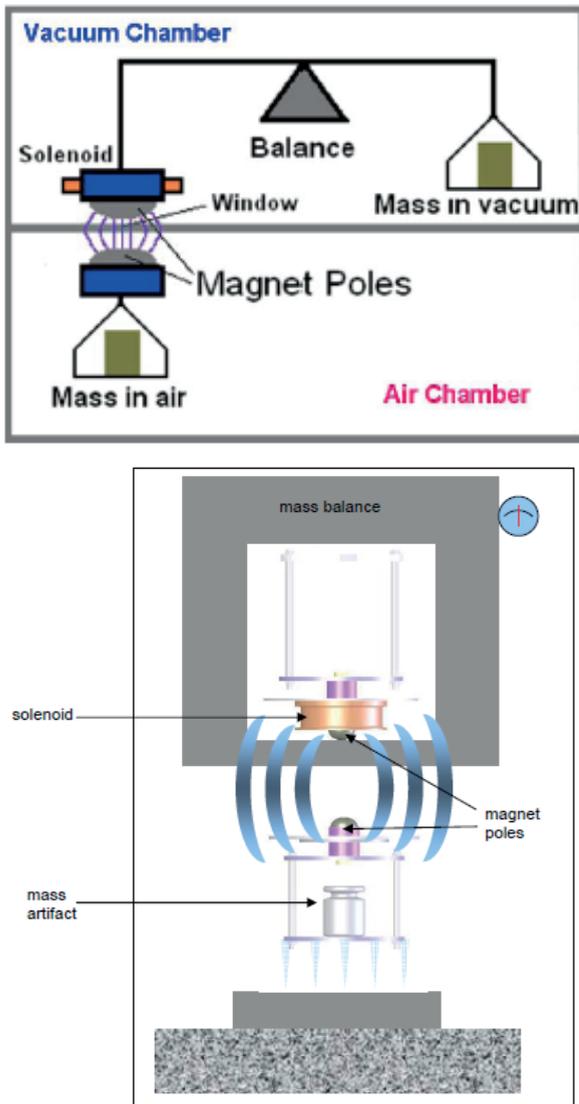


Figure 6. Illustrations of vacuum to air transfer of kilogram calibration using magnetic levitation. The top illustration shows a standard mass in the vacuum chamber being compared to a mass in the air chamber using the same mass balance. The bottom illustration shows the levitation of a mass artifact; the upper magnetic pole is connected to the mass balance.

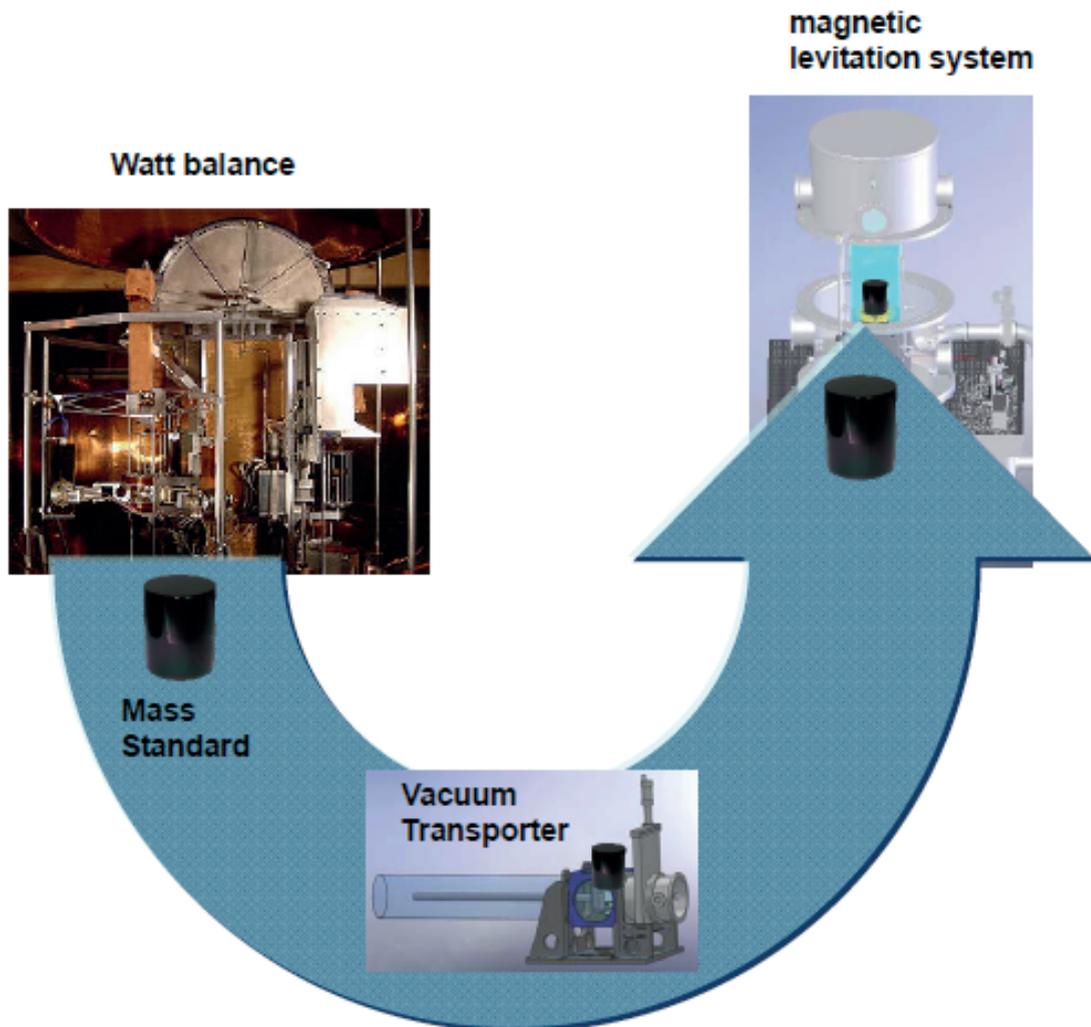


Figure 7. Scenario linking watt balance kilogram realization to an unknown mass in air.

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Effective Communication Between Customers and Their Calibration Labs

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A customer may send equipment to a calibration vendor for calibration with the expectation of quickly receiving it calibrated. The only instructions sent with the equipment may be “please calibrate this.” Calibrations are often delayed because of missing or incomplete information. Additionally, customers may not receive equipment calibrated for how they intend to use it. Using the requirements of ISO/IEC 17025 and feedback from calibration vendors, we created a formalized process and a standardized form for requesting calibration services. We’ve found these to be effective tools to streamline outsourced calibration and equipment services.

Introduction and Viewpoint

This paper is written from the perspective of an internal calibration lab which directly services only the departments within its own company and is also in direct contact with outside calibration vendors. While it is recognized that some companies’ communications go through multiple departments (both for the customer and for the provider), these experiences and solutions may also be applied to any lab needing outsourced calibration services. It is imperative that both the customer and the vendor take equal responsibility for communicating with one another to ensure the customer’s requirements are met. Both the customer and vendor should be familiar with the requirements of ISO/IEC 17025:2005 section 4.4 Review of requests, tenders and contracts, 4.6 Purchasing services and supplies, and Section 4.7 Service to the customer. This paper addresses common frustrations between customers and their external calibration labs and offers solutions for effective communication.

Our Background

In the process of meeting the calibration requirements of our internal departments, we interact with external calibration vendors. Our Testing Labs are accredited to ISO/IEC 17025. While our Calibration Lab is not accredited on its own to ISO/IEC 17025:2005, it must still meet the requirements of ISO/IEC 17025. Throughout our ongoing interaction with calibration vendors, we’ve both encountered repeated common frustrations and communication issues. By looking at those issues, we developed some standardized practices to minimize, and hopefully eliminate, problems, confusion, and repeat-work and to ensure smooth, timely, and complete calibrations and service.

Understanding Our Needs

In the beginning, we thought that just by finding an ISO/IEC 17025-accredited calibration vendor, sending them our equipment, and saying “please calibrate this,” that they would know what we wanted and that our calibration requirements would be satisfied. In return, we’d get a calibration certificate and a sticker. It was only after multiple phone calls, delays, or reviewing calibration reports and finding that our requirements weren’t being fulfilled that we realized that we needed to better communicate our calibration requirements to our vendors. By completely understanding our own requirements, we were able to better communicate with our calibration service providers so that we would receive what was required the first time. By communicating specific requirements upfront, it has saved time, money, and resources for both the supplier (vendor) and the customer (us).

What Were The Issues?

What were the frequent and common issues that we encountered and what did we need to know?

As part of our accreditation, we’re required to use accredited calibration vendors. In the beginning, after finding an accredited vendor, we send the equipment to them and ask them to calibrate it or give us an ISO 17025-calibration.

Some vendors might call back and ask if:

Do we want an ISO 17025-accredited calibration?

- Do we need to just meet the requirements of ANSI Z540, MIL STD 45662A, or something else?
- In what units should the data be reported? (e.g., the gauge reports in inches of water, inches of mercury, psig, kpa, but you only need it to report in psig.)
- Do we want As Found & As Left data?
- Do we need any data or just a Pass/Fail?

- How many data points do we want – single points or multiple points?
- Do we want all parameters calibrated or just some?
- Do we want the entire range calibrated or only what is used?
- What are our accuracy requirements – are they to calibrate to manufacturer’s specs or is there another standard that applies?
- Is there a method or procedure that needs to be followed? (Are we better off finding a calibration vendor accredited to calibrate to a specific method, such as ASTM E4, rather than each of the parameters that may make up the requirements of ASTM E4.)
- Do we want uncertainty data?
- Should they immediately notify us if it needs repair or adjustment or is found to be out-of-tolerance?
- Is a calibration or repair warranty available? Is it needed?
- Did we send accessories with the equipment that needs to be returned, such as instrument manual, power cord, other cables, etc?
- What is our calibration interval?
- Is a rush calibration needed? Can they provide a rush calibration and if so, how much extra is it?
- How would we be paying? What were their accepted methods of payment? What are their payment terms? Is there tax-exempt status that needs to be documented?
- What turn-around time do we need?
- How do we want the equipment shipped back? Is there a shipping account number we’d like it charged to? Do we need it shipped back in the packaging in which it was sent?
- To who’s attention should it be returned?

Our Answers

How did we address these questions? Some of the answers we needed to find out ourselves, such as:

- How our people were using the equipment and their accuracy needs. In some cases we found that they only used it for some but not all parameters, or that the accuracy needs were different from the manufacturer specs – maybe tighter or looser.
- We also sought input from the equipment users as to how frequently they needed it calibrated.
- We also researched calibration vendors and compared costs and services to insure that we both stayed on budget and received the calibrations we needed. For example, some calibration vendors stated they charge extra for calibration

measurement uncertainty data versus their stated "best measurement uncertainty capability." How would the uncertainty data help us? How do we use the uncertainty data? One of the ways we use uncertainty data is to make sure that a piece of equipment can meet the accuracy requirements for which it is used. We also use the uncertainty data in developing the uncertainty budget for the next step in the traceability chain. Another way we use uncertainty data is to evaluate between calibrating, for example, a digital multi-meter, in-house versus sending it out. In this instance, we found that by calibrating it in-house, we didn’t have the reference standard accurate enough to be able to have an uncertainty that was lower than the accuracy specification at certain parameters or levels. So, after finding out that the user did need it at that parameter and level, we researched vendors to whom we could send it. Herein lies our need for the actual *uncertainty data* for a calibration and not the vendor’s "best measurement uncertainty capability."

The Solution: The Calibration / Service Request Letter

We found we were being asked the same questions and giving the same types of answers each time we sent something out. We progressed from writing new calibration request letters for each time equipment were sent out for calibration to developing a template that could be used for any outsourced service – calibration or repair. Sending out equipment for calibration for us has two steps; Step 1 is completed before the equipment is sent out, Step 2 is completed upon equipment receipt back from outsourced calibration or service.

Step 1, Page 1

Retrieve a copy of our Outsourced Service Log template (Figure 1) from our controlled document system. Whether we’ve previously used the selected vendor or not, we check their scope of accreditation each to make sure they are still accredited for that equipment or parameter. We also review their status on our Approved Vendor List. ISO/IEC 17025:2005, Section 4.6.4 *"The laboratory shall evaluate suppliers of critical consumables, supplies and services which affect the quality of testing and calibration, and shall maintain records of these evaluations and list those approved."* We record their accreditation scope status, accreditation provider & number, date (this form was) completed and by whom, and date the equipment is sent out. Any required operational or accuracy checks are performed and recorded in the respective equipment file. If necessary, contact the vendor for return authorization, turn-around estimate, quote for purchase order, and/or discuss calibration details.

Step 1, Page 2

The second page is the Service Request Letter template (Figure 2): complete the mailing/shipping section, including "Attention To." Enter in equipment information, including instrument type, manufacturer, model, serial number, our property ID, calibration interval, and the requirements and/or specifications for calibration. Using check boxes, select the requirements for ISO/IEC 17025-accredited calibration, As Found & As Left data, uncertainty data (for our equipment's calibration), attention for return, return shipping vendor and account, payment information, return of shipping box, accessories sent with equipment, etc. We include a copy of this letter on company letterhead with the equipment sent to the calibration provider, even in addition to copies of service request letters/quotes/forms provided by the vendor.

Step 2

Upon receipt of equipment back from calibration:

- We check the operation of the equipment. ISO/IEC 17025:2005, section 5.5.9 *"When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and*

shown to be satisfactory before the equipment is returned to service."

- We review the calibration certificate for compliance to the original service/calibration request.
 - Are all parameters calibrated?
 - Are the accuracy requirements met?
 - Was everything returned – did you get back what was sent out?
 - Do the data make sense – are there calculation errors?
 - Are all Passes or Fails, truly Passes or Fails? For example, how are you using measurement uncertainty and/or guard-banding when making compliance decisions?
 - Does the calibration certificate meet the requirements of ISO/IEC 17025:2005, 5.10 Reporting the Results?
 - Does the reported equipment ID match the ID of the equipment sent in?
 - Does the calibration sticker have the appropriate information and is the information correct, ID? Calibration Date? Calibration Due Date?

OUTSOURCED SERVICE LOG	
Equipment ID _____	
1. VENDOR STATUS	
Vendor's Name: _____	Location: _____
Does Vendor's Scope of Accreditation include parameters to be calibrated?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Is Vendor on the Approved Calibration Vendors List?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Comments: _____	
Date Completed: _____	Form Completed By: _____
Date Equipment sent out: _____	
2. CALIBRATION DATA STATUS	
A. Date Equipment Received: _____	
B. Vendor Calibration Method: _____	
C. Comments: _____	
D. Measurement Uncertainty: Select:	<input type="checkbox"/> See certificate or <input type="checkbox"/> ± _____ or <input type="checkbox"/> _____
E. OK FOR TESTING:	<input type="checkbox"/> Yes <input type="checkbox"/> No (If No, then comment:) _____
Date of Calibration: _____	
Due date of next Calibration: _____	
Form Completed By: _____	Date: _____
LIMS Updated By: _____	Date: _____
Peer Reviewed By: _____	Date: _____

Figure 1. The Outsourced Service Log is attached with the calibration certificate and other documents as necessary.

**<Company Logo
goes here>**

Date: _____

Company: _____
Address: _____
City, State, Zip: _____
Phone: _____ Ext: _____

ATTN: _____

Enclosed is / are the following _____ for Calibration / Repair / Other _____

Instrument Type: _____ Model: _____ Calibration interval: _____
Serial number: _____ NSF Property number: _____
Calibrate to Manufacturer's Specs / Standard / Other (provided): _____

Instrument Type: _____ Model: _____ Calibration interval: _____
Serial number: _____ NSF Property number: _____
Calibrate to Manufacturer's Specs / Standard / Other (provided): _____

Please:

Provide an ISO/IEC 17025-accredited calibration including As Found, and if necessary, As Left, data

Provide uncertainty data

_____ <use for other comments or remarks>

Return to Attn: _____

Return via FedEx / UPS / Ground / Air / Pre-Pay & Add / Other, Account # _____

Please return in shipping box provided.

For payment: Contact me for payment info / Charge to card on file / Refer to PO# / Other: _____

Please let me know if you encounter problems or delays.

Best regards,

<Name>
<Title>
<Company>
<Address>
<Phone/Fax/e-mail>

Figure 2. The Service Request Letter template. Where the enclosed instruments are listed, repeat the groupings for as many pieces of equipment are to be sent to one vendor at a time.

Step 2, Section 2

On Section 2 of our Outsourced Service Log (Figure 1), we enter the date the equipment was received back, the vendor's calibration method, any comments about calibration (adjustments or repairs made, in spec, out-of-tolerances, etc.), measurement uncertainty, is the unit ok for testing, date of calibration, due date of calibration, name of person completing the form and date form completed, name of person and date the LIMS/equipment tracking system updated, and name of person and date who peer reviewed this calibration. The calibration certificate and related calibration documents are scanned to pdf and attached to this Outsourced Service Request template & letter. Appropriate trending or control chart data are recorded. The pdf document is then stored in the equipment's electronic file.

What If?

What happens if you find that you did not receive something requested or agreed upon in the contract? First, immediately contact the service provider. Perhaps it was an oversight, it's in the mail, or there's an error that can be corrected on an amended certificate. Be open to discussing the issue. Perhaps it's something that you and/or they can learn from to improve in the future. That feedback is an important part of continuous improvement. If you find that you're still having difficulty or not receiving what you need, let them know you'll be contacting their accreditation body. Sometimes that is necessary.

Conclusion

We recognized we were repeating the same process each time we contacted a vendor. By developing a standardized form that can quickly complete, we have found that this process and template streamline our service requests. We find that we haven't forgotten to ask/request any pertinent information. We have less rework and have saved time and money in call backs & recalibrations. We've received positive feedback from our vendors that they appreciate the standardized information and wish more of their customers were as knowledgeable and forthcoming with information, as well.

Acknowledgements

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Heather Wade is a graduate of the University of Michigan with a B.S. in Biology. She has worked as a microbiologist, an extraction and analytical chemist, and a physical test engineer before finding her way into the field of metrology. She is an American Society for Quality-Certified Calibration Technician (ASQ-CCT) and is Calibration Officer at NSF International, in Ann Arbor, MI, USA. She is currently Secretary of ASQ-Measurement Quality Division and will begin her term as Chair-Elect in 2012. She has participated in the updating of the ASQ-CCT exam. She has had articles published in ASQ-MQD's newsletter, "The Standard." She has presented papers at both Measurement Science Conference and NCSL International conference. She is an active committee member of NCSLI's Testing Lab committee and Test Equipment Asset Management committee and subcommittee on Delay Dating.

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