RGA Calibration

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Why do we need to calibrate a vacuum gauge?

Instrument is indicating a pressure value of 1 Pa.

How do we know that the pressure of gas is really 1 Pa?

Answer:

We need traceable calibration of the instrument.

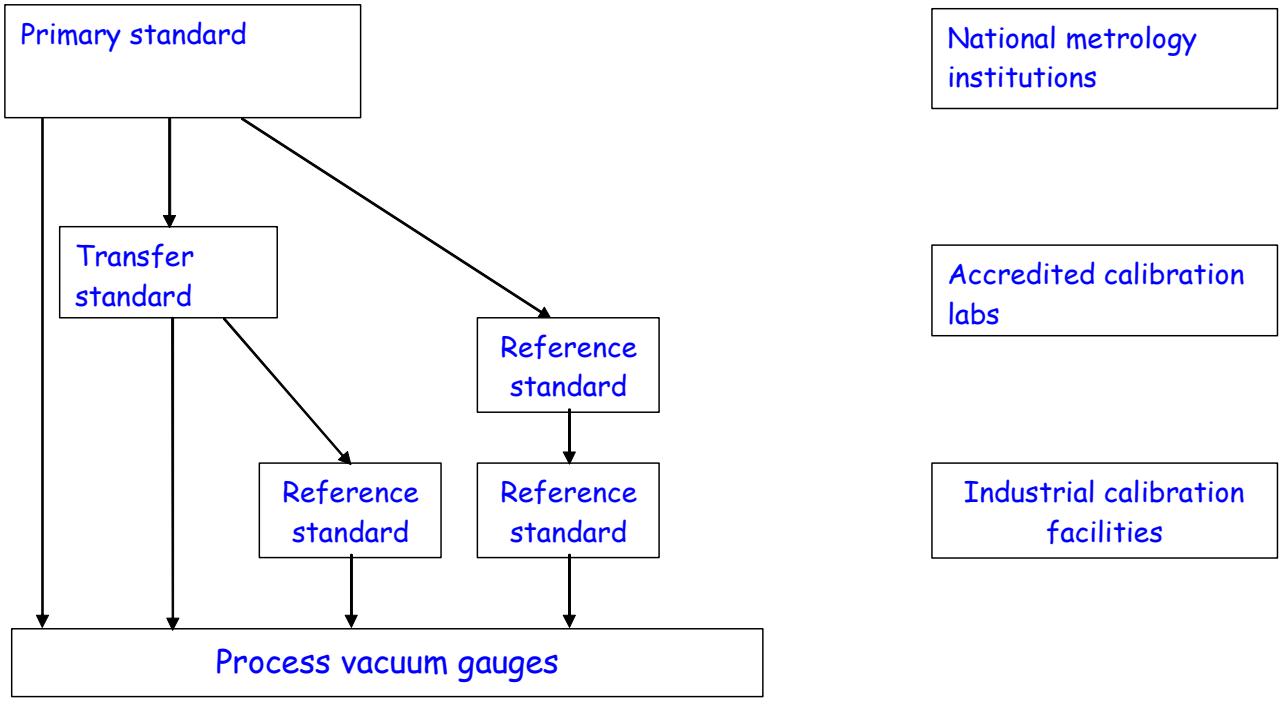
Only traceable calibrations assure comparable measurements in "time" and "space"

Why (and when) should we care about calibrations?

- Process repeatability
- Process transferability
- Instrument interchangeability
- Increased production uptime through prediction and prevention
- To avoid potential health hazard
- ISO 9000 compliance

Traceability chain

- Unbroken link to primary standards
- Measurement uncertainty at each link in the chain is well known



Definition of calibration

(From International vocabulary of basic and general terms in metrology)

- **Calibration** is a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

Pressure determined by the standard Pr	Pressure indicated by the instrument under test P_{DUT}	Error $E=(P_{DUT} - Pr)$	Correction $C=(Pr-P_{DUT})$
1.00000	1.0032	0.0032	-0.0032

Adjustment (of a measuring instrument)

- **Adjustment** is altering the performance of an instrument to ensure that the value it indicates are correct within specified limits

High / ultrahigh vacuum gauge calibrations

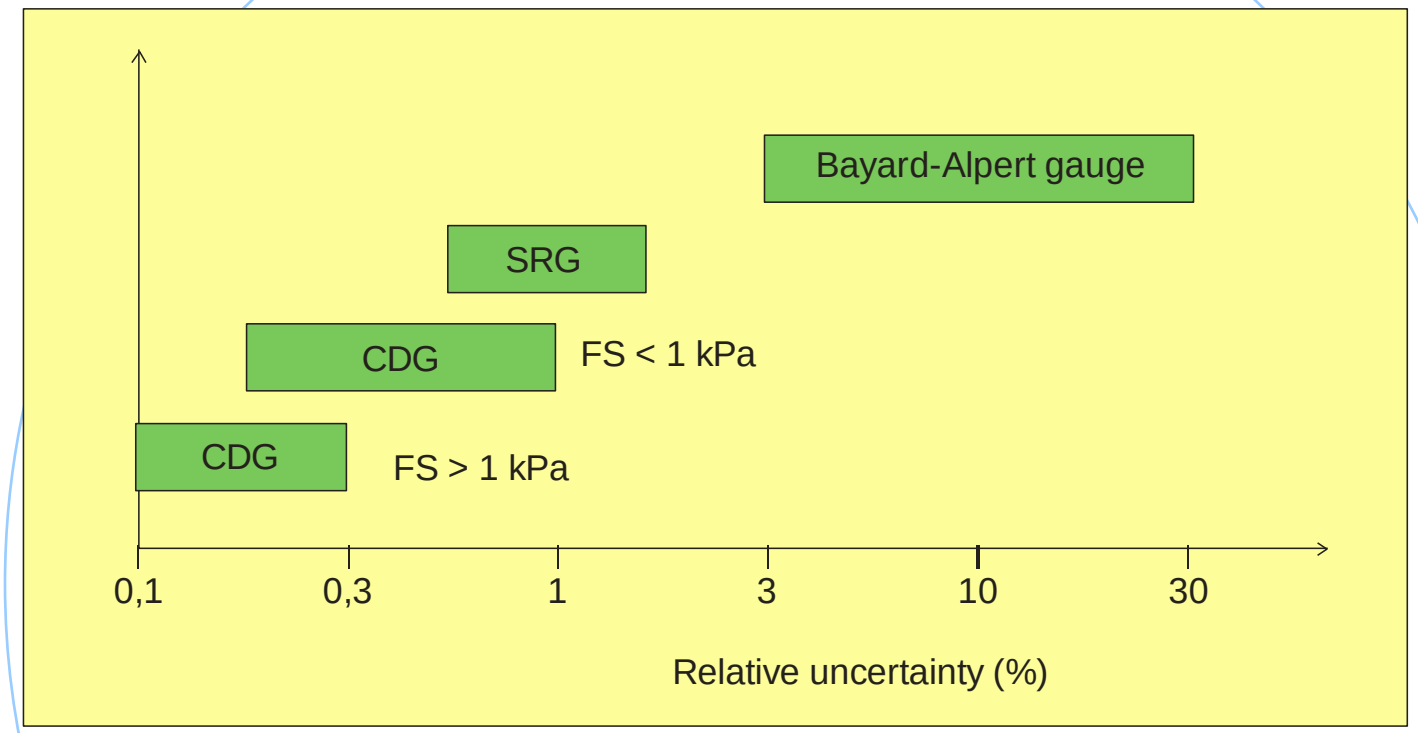
- **Primary level:** generation of known gas pressure by
  - static expansion method
  - dynamic (continous) expansion method

- **Secondary level:** comparison calibrations using reference vac. gauges
  - Capacitance diaphragm gauges
  - Spinning rotor gauge
  - ionization gauge (Bayard-Alpert, extractor...)

Usually there are two main contributions to the uncertainty of HV/UHV gauge:

$$U(p) = \sqrt{U^2(\text{calibration standard}) + U^2(\text{time stability})}$$

Typical time stability (1 year) of reference vacuum gauges



Residual Gas Analyser (RGA)

or

Quadrupole Mass Spectrometer (QMS)

ION SOURCE	MASS FILTER	ION DETECTOR

in general: U(RGA)>U(BAG)

main additional sources of uncertainty:

- **injection of ions into mass filter**
- **transmission of ions through mass filter**
- **....**

Calibration of RGA on primary level

- no routine service at national metrology institutes (NMI) yet
- developement of such service by NMIs was recently initiated:

EMRP IND12 Project
Vacuum metrology for production environments

Joint Research Project (JRP) of a consortium of 7 European NMIs and 5 industrial companies
(http://www.ptb.de/emrp/ind12-home.html).

Project is funded:

45% by the European Metrology Research Programme – EMRP
55% from national funds of participating countries

JRP start date and duration: September 2011 – 3 years
JRP Coordinator: Dr. Karl Jousten, PTB

JRP Partners:

PTB, Germany
INRIM, Italy
IMT, Slovenia
LNE, France
CMI, Czech Republik
CEM, Spain
TUBITAK-UME, Turkey

Nonfunded Partners:

INFICON Ltd, Liechtenstein
VACOM GmbH, Germany
Danfoss A/S, Denmark
Lazzero Tecnologie S.r.I, Italy
INFICON GmbH, Germany

Research exelence grant – REG
University in Genova

R&D work of JRP IND -12 is divided into three workpackages:

- *WP 1: Dynamic vacuum calibration facility (PTB)*
- *WP 2: Leak measurement and testing (INRIM)*
- *WP 3: Partial pressure and outgassing rate measurement (IMT)*

WP 3: Partial pressure and outgassing rate measurement

Workpackage addresses the "**Traceability** for partial pressure measurement and outgassing rate measurement and materials characterisation in industry"

Main aims are:

• to provide dedicated calibration facilities for quadrupole mass spectrometers (QMS)

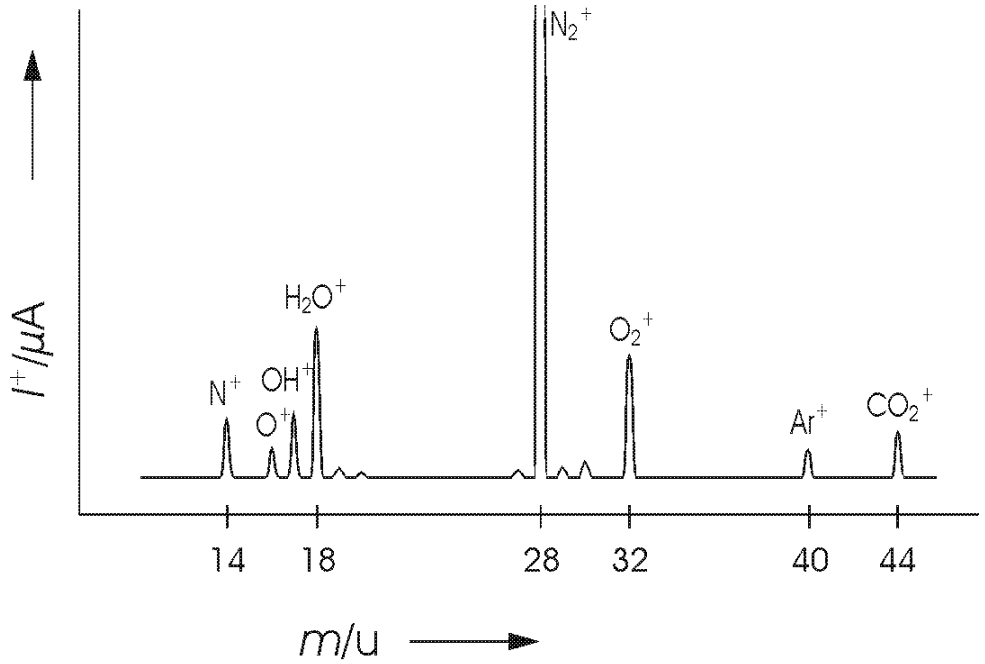
• to investigate factors influencing metrological characteristics of QMS

• to develop model systems for outgassing rate measurements applying different methods and provide measurement traceability of these methods

• to investigate different materials (like high vacuum compatible polymers, nanostructures, porous silicon, zeolite...) as a possible reference materials for validation of outgassing rate measurements in industrial environment

Measurement result of RGA

Gas spectrum: measured ion current as a function of mass number (m/e)



Basic quantities to be calibrated:

- mass scale
- sensitivity
- "cracking" pattern (fragmentation factor)

Mass scale

– can be easily checked (and calibrated) by the user using common gas peaks present in vacuum systems

2	H_2^+
12	C^+
18	H_2O^+
28	CO^+ (N_2^+)
44	CO_2^+

- ***Definition of sensitivity of QMS***

  (from ISO 14291-Vacuum technology — Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers – currently at Committee Draft stage*)*

sensitivity
S

ratio of the change in spectrum peak height (ion current) $[I - I_o]$ to the corresponding change in partial pressure $[p - p_o]$

$$S(p) = \frac{I - I_0}{p - p_0} \qquad (1)$$

I is the ion current measured at partial pressure p and I_o is the ion current measured at residual pressure p_o. The unit of sensitivity is A/Pa. S depends on pressure p.

To calibrate the sensitivity one has to generate a known change of partial pressure and measure a change in ion current

- *each gas has different sensitivity!*

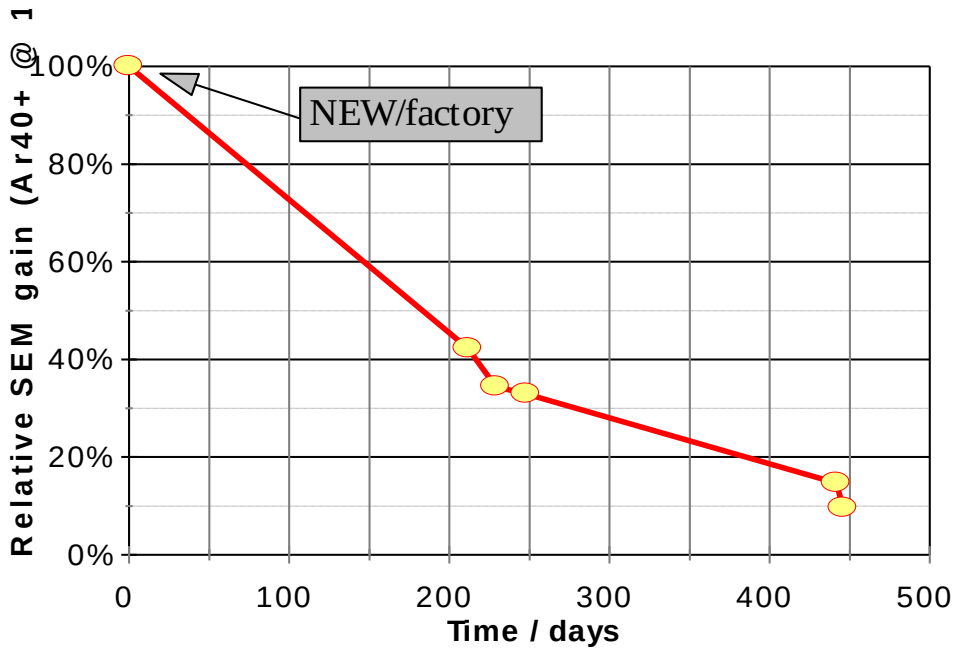
Sensitivity depends on "adjustable" parameters:

- *electron emission current*
- *electron energy*
- *mass resolution setting*

When calibrating RGA sensitivity these parameters shall be specified (and reported in a calibration certificate)

If RGA is used with secondary electron multiplier (SEM) the sensitivity depends on the gain of SEM

– SEM gain can vary considerably with time (aging of SEM!)



If SEM is not stable than it has no sense to calibrate in NMI the sensitivity of RGA operating with SEM, but only with Faraday cup detector.

Possible methods for calibration of RGA sensitivity

J. Vac. Sci. Technol. A 11(3), May/Jun 1993

American Vacuum Society Recommended Practice

Recommended Practice for the Calibration of Mass Spectrometers for Partial Pressure Analysis

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(Received 1 October 1991; accepted 9 January 1993)

This Recommended Practice addresses issues involved in the use of partial pressure analyzers (PPAs) for quantitative analysis and describes recommended apparatus and procedures for determining resolution and sensitivity of a PPA so that the instrument can be used quantitatively for partial pressure, partial flow and gas composition analysis. This updates

Three methods for calibration of QMS are discussed in AVS RP

- direct comparison with ionization gauge or SRG
- pressure divider system
- orifice flow (primary) system

Pressure ratio $R_p = P_{high}/P_{low}$

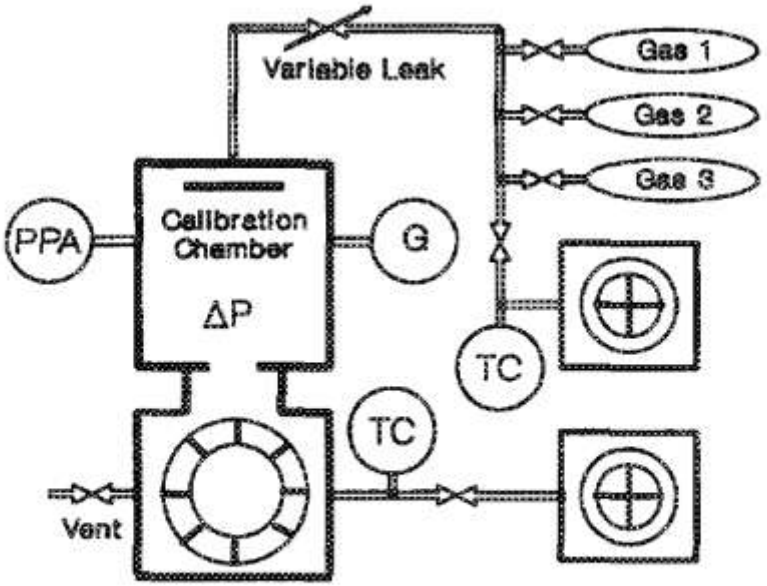

FIG. 7. System for calibration of PPAs by direct comparison of the PPA ion signal with a pressure reading on a calibrated MDG or IG.

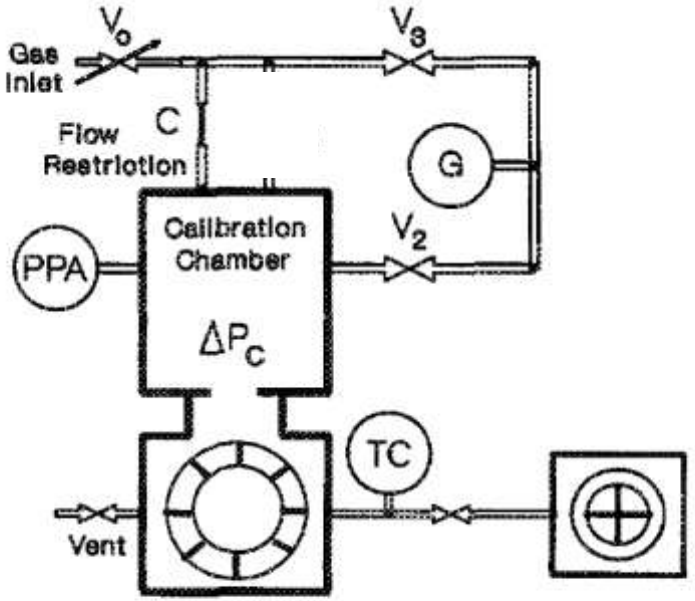

FIG. 8. Pressure-divider calibration system. Pressure gauge *G* is a MDG or CM transfer standard gauge.

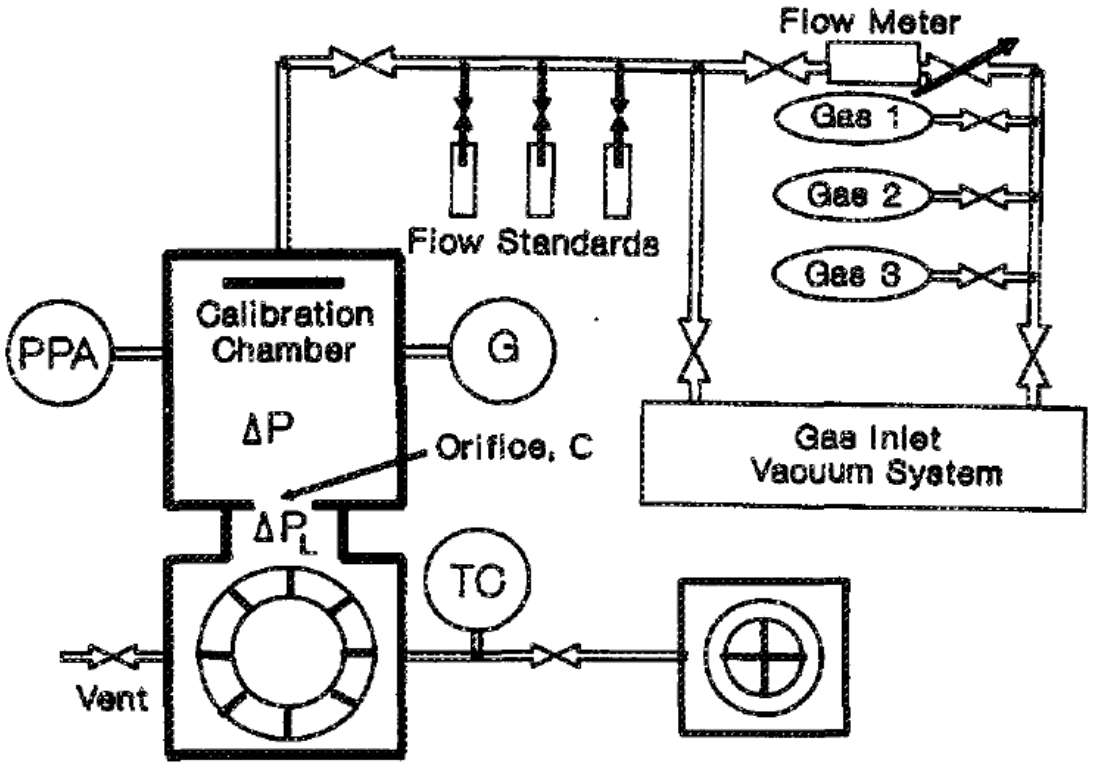

FIG. 9. Direct-flow calibration test stand for determining the sensitivity of a PPA in reference to ion source pressure calculated from $P = F/C$ where C is the conductance of the test chamber.

Direct comparison method:

- with SRG:
  - $P_{min}=10^{-6}$ mbar
  - U=3-5% (if SRG is calibrated only for N_2)
  - very good time stability
- with ionization gauge
  - $P_{min}=10^{-12}$ mbar (Extractor)
  - U= 5-20%
  - time stability can be problematic
  - even if ion gauge is calibrated in-situ with SRG, the linearity can be an issue

Pressure divider system:

- $P_{min}=10^{-9}$ mbar – 10^{-10} mbar

Traceability can be realized via SRG

- U=3-5% (if SRG is calibrated only for N_2)
- very good time stability of SRG
- excelent linearity of SRG in the range from 10^{-6}mbar – 10^{-3}mbar
- it is not a "primary method“

Orifice flow system:

- calibration pressure is calculated from measured gas flow Q and conductance of the orifice C("primary method")

$$p=Q/C$$

Calibration systems within IND-12 Project

- PTB will develop a dedicated "orifice flow" calibration facility for QMS calibration
  - system will enable mixing of three gasses
- IMT plans to build a pressure divider system
- INRIM, CMI and CEM will calibrate QMS on their existing dynamic expansion primary systems
- TUBITAK-UME and LNE will use direct comparison method with ionization gauge

RGA instruments that are available in different NMIs:

- *Pfeiffer Vacuum QMG 422* *(2 instruments)*
- *Pfeiffer Vacuum Prisma* *(3 instruments)*
- *MKS Instruments Evision+* *(1 instrument)*
- *INFICON TRANSPECTOR* *(2 instruments)*
- *Hiden Analytical HALO 100* *(1 instrument)*

- For stability study a unified measurement procedure is under discussion

- We want to compare performance of different instruments
  - comparable settings of influential parameters
    - electron emission current
    - electron energy
    - mass resolution setting

- Open question is a minimum set of gases to be used for sensitivity calibration
  - H_2, He, N_2, Ar, Kr?, SF_6

We at NMIs have systems and know how to generate known partial pressure of gas in calibration chamber

But we lack experience and information

- how QMS are operated in industry...
- what are needs of industry regarding calibration ranges, gases, uncertainty...

So we decided to organize a workshop dedicated to calibration of RGAs

Aims of the WS are:

- to exchange the information on the practical experience of the use of QMS
- to present the users from the industry the activities within the JRP
- to get additional information about the specific needs of industrial users
- to share the information with the manufacturers of QMS about their recommendations for the practical use of QMS for achieving best performances

Title of the workshop:

"Measurement characteristics and use of QMS for vacuum applications"

Dates: **April 13 - 16, 2012**
Location: **Lake Bled, Slovenia**

http://www.slovenia.si/
http://www.slovenia.info/

http://www.bled.si/

Thank you for your attention!

And welcome to the QMS workshop in 2012.

http://ind12-qms.imt.si