133, Bd National - BP 231 - 92503 Rueil Malmaison Cedex - France
Tél +33 (0)1 47089250 Fax +33 (0)1 47083239 http://www.riber.com
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Reference 9000™

High Resolution Beam Flux Calibration Module

The introduction of the Reference 9000™ calibration module complements the full range of products for MBE offered by RIBER. This new technology takes the sting out of flux measurement problems by accurately calibrating effusion cells with reproducibility better than 0.2% of the beam equivalent pressure (B.E.P.).

1. Introduction

Reproducibility of MBE growth is extremely dependent on the reproducibility of source fluxes. In order to define the optimum process parameters, a flux gauge (movable or installed on the manipulator) is commonly used as a calibration tool. However, the beam equivalent pressure (B.E.P.) read out is affected by the behavior of system materials, especially II-VI, and by the ageing of the gauge itself that may result in a dramatic drift of the measurement and thereby, hinder the reproducibility of material composition in layers. Therefore, frequent and time-consuming recalibrations are necessary. This point is even more critical in the case of quaternary alloys. Responding to this need for high-resolution routine calibration, RIBER, in collaboration with the University of Ulm, has developed the Reference 9000™.

This high-resolution beam flux calibration module meets the ultimate trends of advanced MBE in terms of accuracy, stability and above all, reproducibility of material fluxes. Reference 9000™ has been designed in modular system and consists of:

- a high accuracy electrometer
- an external flux reference system.

The high accuracy electrometer permits to improve the beam pressure reading for achieving relative calibration (see page 2). An absolute calibration (Fig. 1) is possible when using the external flux reference system in addition to the electrometer (see page 3).

These two sub-units can be ordered and operated separately. Reference 9000™ is directly retrofitable to all existing commercial and homemade MBE systems.

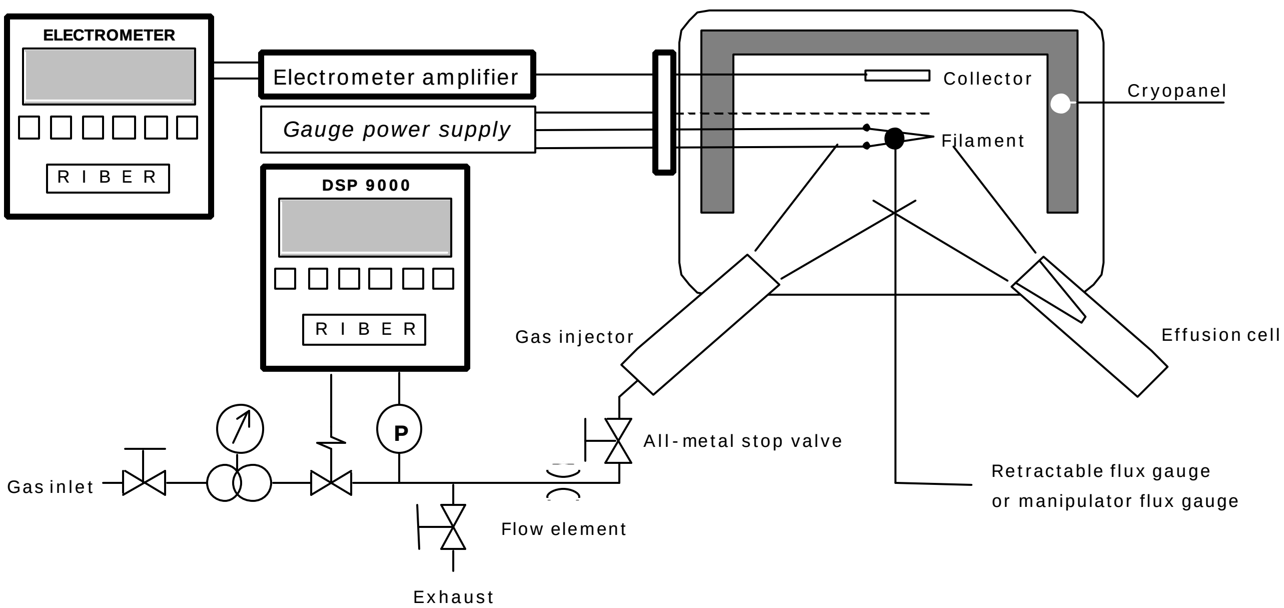


Fig. 1: Absolute calibration set-up

2. High accuracy electrometer

On standard configurations, the beam equivalent pressure measurement is performed with the conventional 2-digits-precision gauge controller supplied with the flux gauge (e.g. GP 307 or 350). The pressure measurement accuracy is **± 3 % of reading in the two decades** of current used during a process (1mA, 10 mA).

However, a reproducibility of solid cells beam flux better than 0.2% is only possible if the ion gauge collector current is calibrated and monitored by the RIBER 5-digits electrometer (Fig. 2). This beam pressure readout electronics provides a collector current measurement with an accuracy and a stability **better than ± 0.1 % of reading on each decade**.

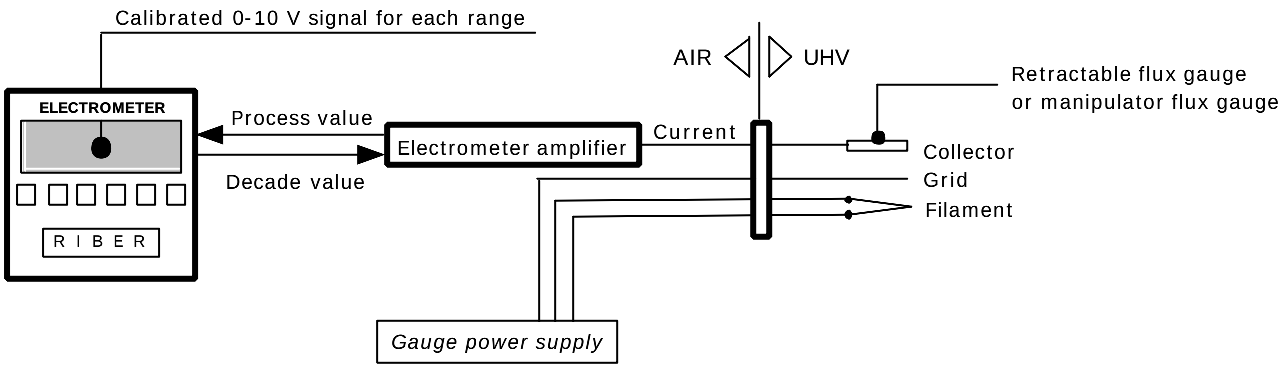


Fig. 2

The RIBER 5-digits electrometer consists of a high accuracy electrometer amplifier unit and a high-resolution digital control unit conditioning the signal coming from the amplifier. The high accuracy amplifier unit is linked to the collector output through a short-shielded BNC wire and reads an analog signal (collector current). This current is then amplified into an output analog signal through one of five sensitivity ranges, from 1nA to 10 µA corresponding to five decades of collector current, and dependent on the partial pressures inside the UHV chamber, selected either manually or in autoranging mode from the control unit.

This signal is fed to the DSP electrometer for A/D 16-bit conversion and 5-digit display. The controller handles the analog signal with independent and automatic calibration for all measurement ranges. B.E.P. value readout is either in nA or µA, format XXXXX from .00000 to 999.99.

The controller unit features a RS 232 terminal interface for range selection commands to the amplifier, and can be operated either in local mode by a menu-based dialogue, or in remote mode via a host computer using high speed RS 422 network interface and ANSI X3.28 industrial communication protocol.

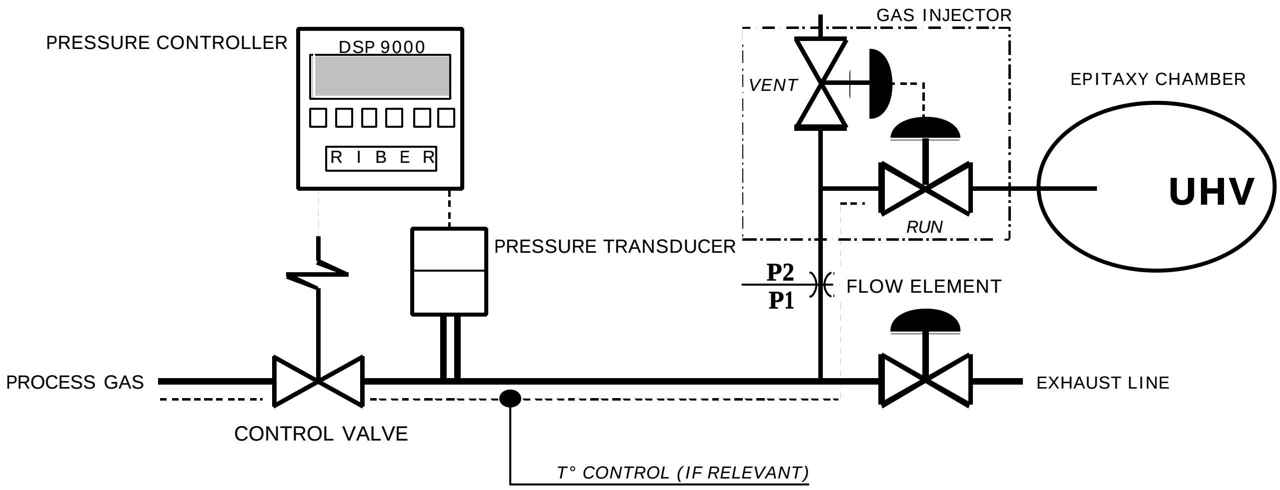
NOTE

A standard power supply with high emission current stability to supply the Bayard-Alpert flux gauge is required in addition (often part of the existing MBE system).

3. External flux reference system

To break away from drift problems, the flux gauge must be calibrated against a very accurate and reproducible pressure standard given by a gas injection line. The use of an external, well-defined reference is not only extremely beneficial to the reproducibility of a given process, but also for the transfer of data from one MBE system to another. In the field of gas flow control, ultimate accuracy and reproducibility are provided by pressure control.

Better than mass flow controllers, which for gas-based doping applications are often operated outside their operational range, which lead to poor response, overshoot, day-to-day drift, or clogging, pressure control permits efficient regulation of very low flow rates (down to the 10^{-3} sccm range) and is easily capable of operating at errors below 0. 2 % of maximum flow over a long period of time. Moreover, thanks to a 3-decade dynamic range, pressure control enables to rid the user from dilution problems (no need for carrier gas or hydrogenated mixtures). This technique is based on the control of the gas source pressure upstream of a calibrated leak using a high sensitivity pressure sensor. The gas flow Q is determined by the pressure drop P across the orifice of conductance C, with the relation Q = C x P. Pressure control also requires precise control by electronics; to that effect, RIBER has developed a specific 16-bit PID regulator called DSP 9000.



A pressure control loop comprises:

- A pressure transducer, which measures the pressure of the gas behind a flow element (fixed leak) setting the conductance C of the gas line (as a constant), and thereby linearizing the function $Q_{flow} = C \times P_1$ (upstream pressure), with P_2 (downstream pressure) much lower than P_1. Since the flow through the leak is directly proportional to pressure in the gas line, flux control is achieved by control of that pressure P1.

- A RIBER DSP 9000 pressure controller, controlling regulation parameters. DSP 9000 is a flexible PID controller with features designed for simple and easy operation. Ultimate accuracy, stability and reproducibility are achieved by setting of parameters: setpoint, linear and non-linear ramps, real time PID-optimization, valve bias, etc. It could be operated either in local mode by a menu-based dialogue or in remote mode via a host computer using the RS-422/RS 232-network interface.

- A proportioning, electromagnetically operated control valve controlled with the DSP 9000, giving both fast response (less than 0.1 s) and long-time stability.

NOTE

The exhaust line is designed to quickly empty the gas line upstream of the (very low conductance) flow element at the time of purging. The vent line is used for setting the gas flows before injection into the process chamber. The vent line is connected downstream of the flow element. The pressure inside this tubing is equivalent to the chamber pressure during the process in order to reduce switching transients.

3.1. Performance

Due to the high standard of the DSP controller, the performance data are determined by the selection of the pressure transducer.

- *UltraHigh© performance can be obtained by the following set-up: RIBER DSP 9000© controller + MKS™ 120 pressure transducer (range 0-1 Torr) + MKS™ 248 control valve.*

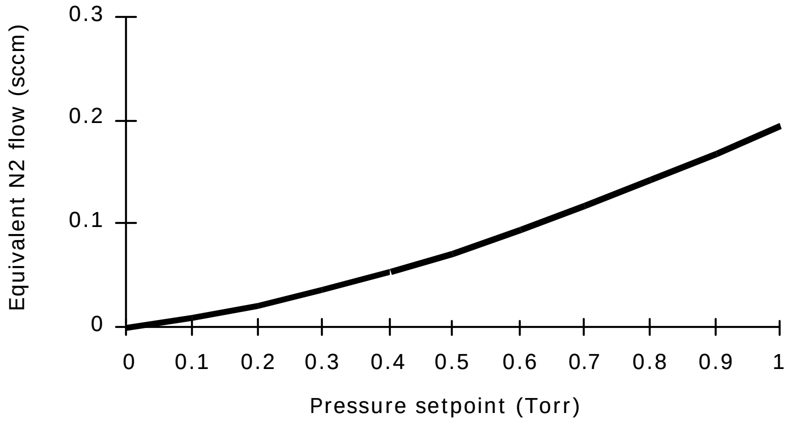
Full scale (f.s.) 1 Torr	**1st decade (¯ 0.1 Torr)**	**2nd decade (¯ 0.01 Torr)**	**3rd decade (¯ 0.001 Torr)**
Uncertainty (0.08% reading) *	8 X 10^{-5} Torr	8 X 10^{-6} Torr	8 X 10^{-7} Torr
Resolution (0.0001% f.s.)	1 X 10^{-6} Torr	1 X 10^{-6} Torr	1 X 10^{-6} Torr
Zero coefficient (0.0004% f.s.)**	4 X 10^{-6} Torr	4 X 10^{-6} Torr	4 X 10^{-6} Torr
Span coefficient (0.002% reading)**	2 X 10^{-6} Torr	2 X 10^{-7} Torr	2 X 10^{-8} Torr
Total uncertainty (% reading)	**0.09 %**	**0.13 %**	**0.58 %**

* Includes non-linearity, hysteresis, and non-repeatability.
** With ± 1°C ambient temperature change.

3.2. How to convert setpoint into flowrate?

- Graphs of gas flow vs. pressure setpoint for a 0.35 mm flow element; with 1 Torr pressure transducer full scale, 0.35 mm flow element and methane as calibration gas

P (Torr)	Q (sccm)
0	0
0.1	0.01
0.2	0.022
0.3	0.036
0.4	0.052
0.5	0.072
0.6	0.093
0.7	0.117
0.8	0.142
0.9	0.168
1	0.194



These measurements have been performed in a RIBER CBE 32 growth chamber evacuated by a 1500 l/s (N_2) turbopump. The gas flows have been read out through a UNIT mass flow meter. Since the test gas is nitrogen, users have to convert the equivalent gas flows with specific correction factor, i.e. actual flow of gas X = tabulated N_2 flow x C (X), with C = 0.71 for methane.

4. Absolute calibration procedure

The calibration procedure of the effusion cells consists in the following steps:

4.1. Gas injection

Gas (e.g. methane) flow rate is tuned to obtain a B.E.P. in the exact order of magnitude of the requested solid source beam pressure (e.g. $5x10^{-7}$ Torr for indium); sensitivity of this adjustment is strictly dependent on the flow element size. The flow rate value (or pressure displayed on the DSP 9000 pressure controller) is considered as the **reference gas flux** for this calibration procedure. This reference gas flux has the performance given page 4. After operation, the background pressure remains low because of the efficient condensation of methane on the LN_2 cooled cryopanels.

NOTE

Due to the very low amount of gas involved, there is no need for additional turbo- or cryopumping. However, a standard rotary vane pump must pump the exhaust line (please consult RIBER for full assistance).

4.2. B.E.P. measurement

The B.E.P. corresponding to the reference flux is measured with the flux gauge provided with the MBE system (manipulator flux gauge or retractable flux gauge) and the RIBER high accuracy 5-digits electrometer (see page 2). It is very likely that the beam equivalent pressure measured with this readout electronics varies with time due to the ageing of the gauge; drifts in the order of 10% are commonly observed.

However, for a given reference gas flux, the relative beam equivalent pressure measurement can be used for further calibration of the effusion cells. The user can measure the process value, compare it with previous data and correct the changed sensitivity of the ion gauge.

4.3. Effusion cell calibration

Once the reference gas flow is switched off, the user can adjust the effusion cell temperature required in order to obtain the relative beam equivalent pressure measurement that matches the reference gas flux.

Reference gas flux

$$Sensitivity(t) = \frac{I\ collector(t)}{I\ emission(t) \times P}$$

P ref = Fixed (external standard)

↓

New sensitivity

$$Pressure\ x = \frac{I\ collector(t)}{I\ emission(t) \times S(t)}$$

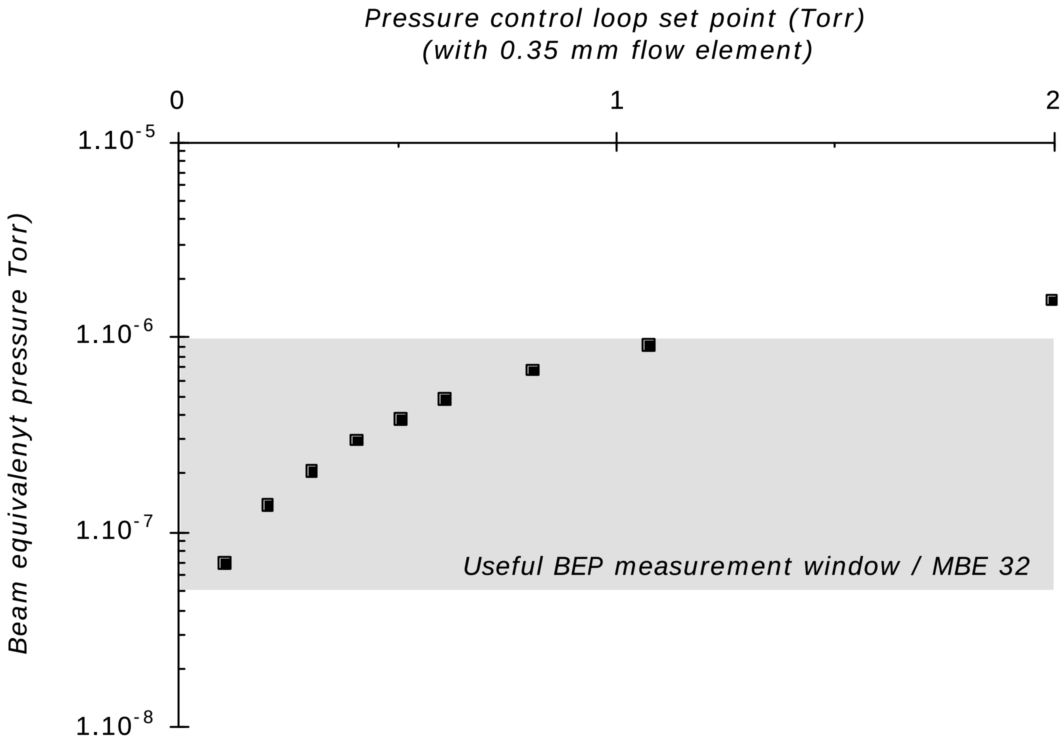
Pressure x = Beam equivalent pressure

↓

Effusion cell calibration

4.4. Example of B.E.P. vs. Calibration flow relationship

The following picture (values on corresponding table) provides example of a curve obtained on a MBE 32 system equipped with a Reference 9000© absolute calibration module (0.35 mm ID flow element). Beam pressure typically varies from 6.9 x 10^{-8} Torr to 1.8 x 10^{-6} Torr when the pressure setpoint of the pressure control loop varies from 0.1 to 2 Torr. Smaller beam pressures (< 6.9 x 10^{-8} Torr) can also easily be obtained by using a 0.2 mm flow element.



Setpoint (Torr)	P mini (Torr)	P maxi (Torr)	Error (%)	Beam equivalent pressure (Torr)
0.1	0.1015	0.1017	0.18	6.9 x 10^{-8}
0.2	0.2019	0.2021	0.10	1.4 x 10^{-7}
0.3	0.3009	0.3011	0.07	2.1 x 10^{-7}
0.4	0.4053	0.4055	0.05	3.0 x 10^{-7}
0.5	0.5052	0.5054	0.04	3.8 x 10^{-7}
0.6	0.6068	0.6071	0.05	4.6 x 10^{-7}
0.8	0.8071	0.8073	0.02	6.4 x 10^{-7}
1.0	1.0710	1.0710	0.00	8.8 x 10^{-7}
2.0	2.0700	2.0700	0.00	1.8 x 10^{-6}

Acknowledgments

RIBER wants to deeply thank Pr. Harald Heinecke's team of Ulm University for their scientific and technical support during the development of the Reference 9000™ high-resolution beam flux calibration module. The product is protected by the European patent n° EP 0458077B1.

5. Specifications

5.1. DSP 9000 Electrometer

Compatible gauge	*Bayard-Alpert type*
Number of channels	*1*
Amplifier / Input signal	*1 nA - 10 mA full scale in 5 ranges*
Amplifier / Output signal	*0-10 V calibrated and zeroed for each range*
Range selection	*Manual and autoranging / stored calibration factors for each range*
Microprocessor	*Full 16 bit*

5.2. DSP 9000 Pressure Controller

Compatible sensors	*All sensors with 0-10 VDC linear output*
Compatible valves	*All control valves with 0-140 mA linear input*
Number of channels	*1*
Microprocessor	*Full 16 bit-PID algorithm and MKS compatible emulation mode*
Input signal	*0 to 10 VDC from pressure transducer, BNC connector*
Output signal	*0 to 150 mA @ 100 ohm, DB9 female connector to control valve*
Accuracy	*± 0.0001% reading*
Response time	*40 ms*
Digital bias output	*0 to 100 mA. Adjustable, optical help by green/red LED*
Zero point	*Adjustable, optical help by green/red LED*

5.3. Common Features

User interface	*Front panel and RS-422 (DB25 female connector) serial port*
Baudrate	*19200 Baud*
Packaging	*19" rack based enclosure. 132.2 mm (3U) high x 500 mm depth*
Power Input	*220-240 VAC. 50/60 Hz. 30 W (+ power for pressure transducer)*
Operating temperature	*Clean room temperature ± 1°C*

Product development is continuous and RIBER reserves the right to make modifications in specification and manufacture without notice.

6. Ordering information

6.1. High resolution electrometer kit

High resolution electrometer kit including:

- High resolution electrometer
- electrometer amplifier
- necessary cables (length 9 m.).
- front panel with provision for absolute calibration module upgrade.

P/N .... On request

NOTE

Power supply for the flux gauge is not included.

6.2. Reference 9000™ absolute calibration module

Reference 9000™ absolute calibration module including:

- One (1) UHP gas introduction line including:
  - *Necessary manually operated shut-off valves, negative pressure regulator, and tubing*
  - *One (1) pressure control loop (MKS 248 control valve, MKS 120 pressure transducer)*
  - *One (1) set of 0.35 mm flow elements.*

- One (1) electronic control rack including:
  - *One (1) RIBER high resolution pressure controller DSP 9000*
  - *One (1) ± 15 VDC @ 750 mA pressure transducer power supply*
  - *Necessary interconnecting cables (length 9m)*
  - *One (1) high-resolution electrometer, with amplifier and necessary cables (length 9m).*

P/N .... On request

NOTE

High-pressure control assembly, including cross purge and safety devices, as well as exhaust pumping group is not included.

6.2. Gas injector

- One (1) bakeable gas injector, with manual all-metal shut-off valve (CF 16)

P/N .... On request

For more information, contact RIBER at:
RIBER - BP 231 - 92503 Rueil-Malmaison Cedex - France
Phone: +33 (0)1 47 08 92 50 - Fax : +33 (0)1 47 08 32 39
E-mail: info@riber.com