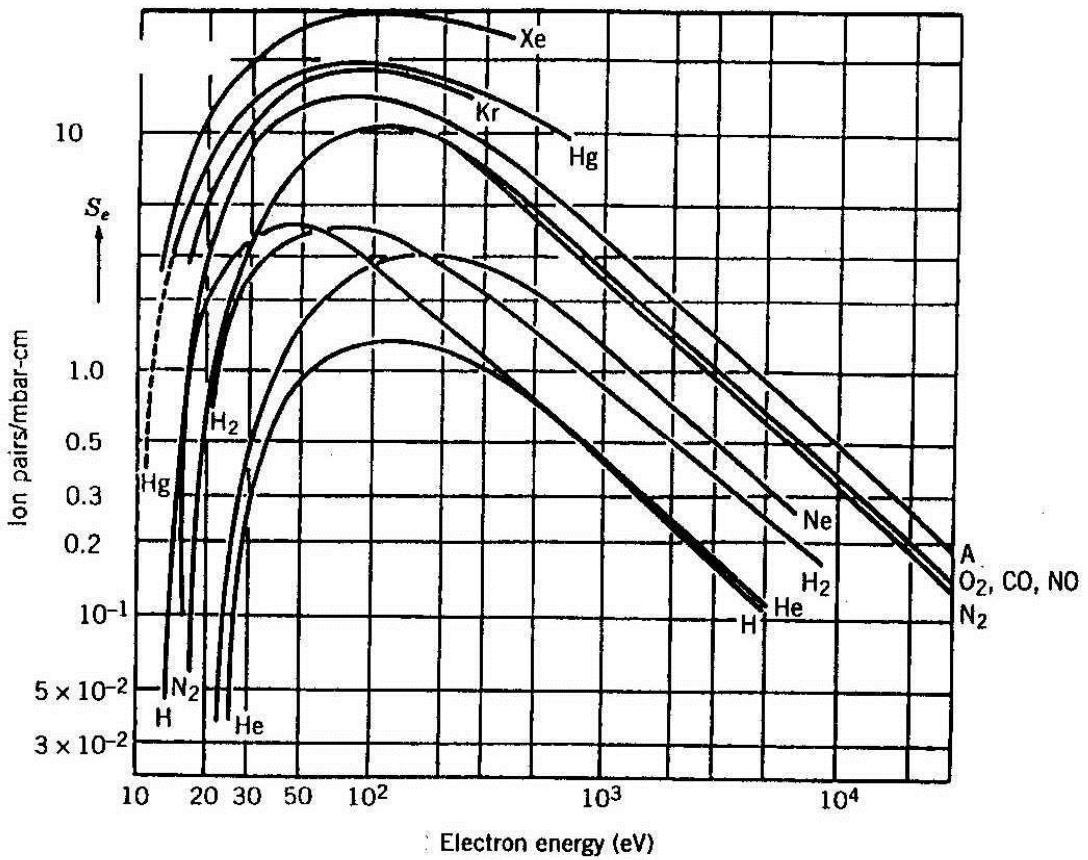
Vacuum gauges II

Karl Jousten, PTB, Berlin

1. Introduction and history
2. Ion gauges with emitting cathodes
3. Limits, accuracy and problems of ion gauges
4. Quadrupole mass spectrometers – work principle
5. Limitations of quadrupole mass spectrometers
6. Summary

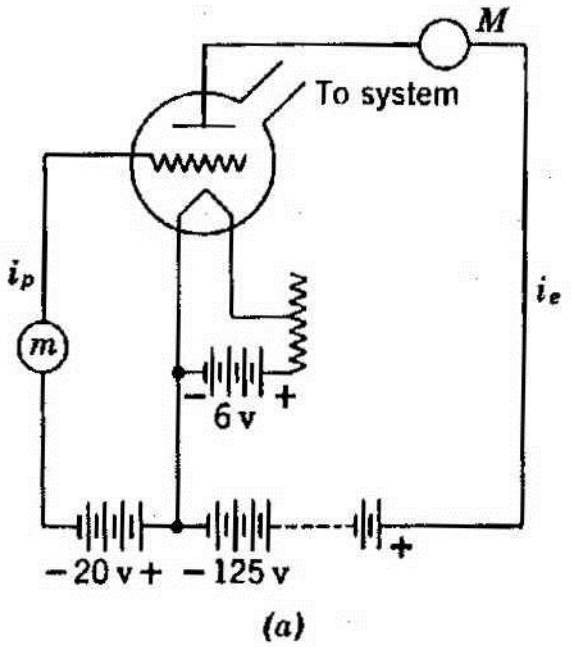
Measurement principle

Ionisation probability of different gas species for electrons between 10 eV and 10 keV

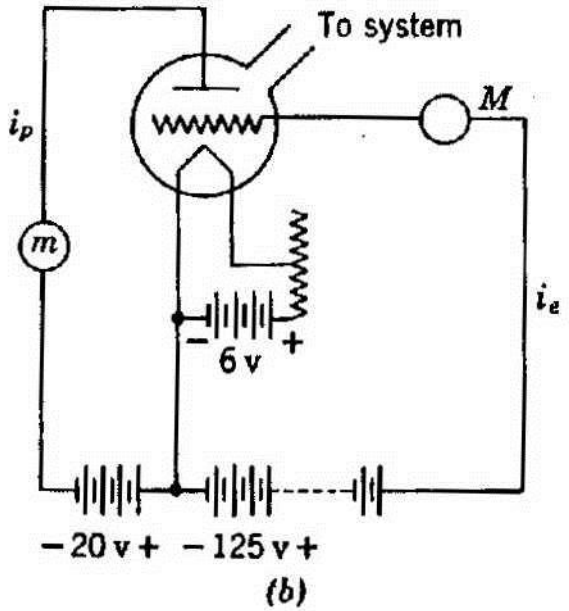


History

The historical vacuum triode gauge originating from the electrical amplifier, the triode, a vacuum electronic device, called the „tube“.



Grid as ion collector



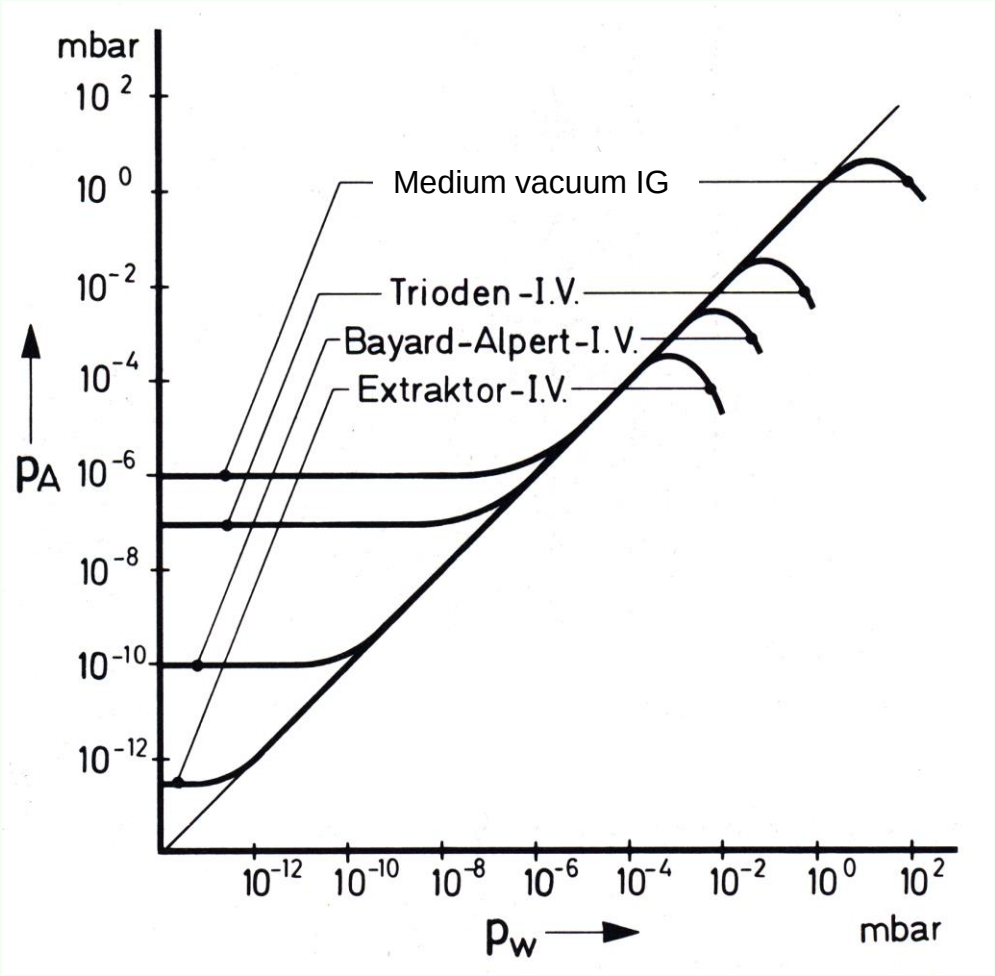
Anode as ion collector

Baeyer 1909

Buckley 1916 useful gauge

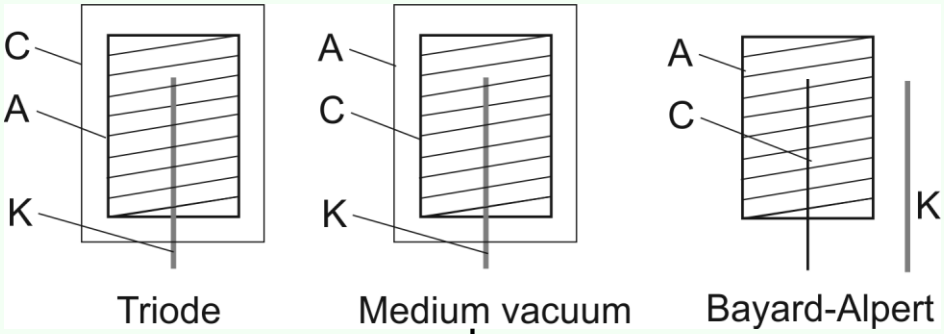
Measurement principle

Ionization gauges for different vacuum ranges

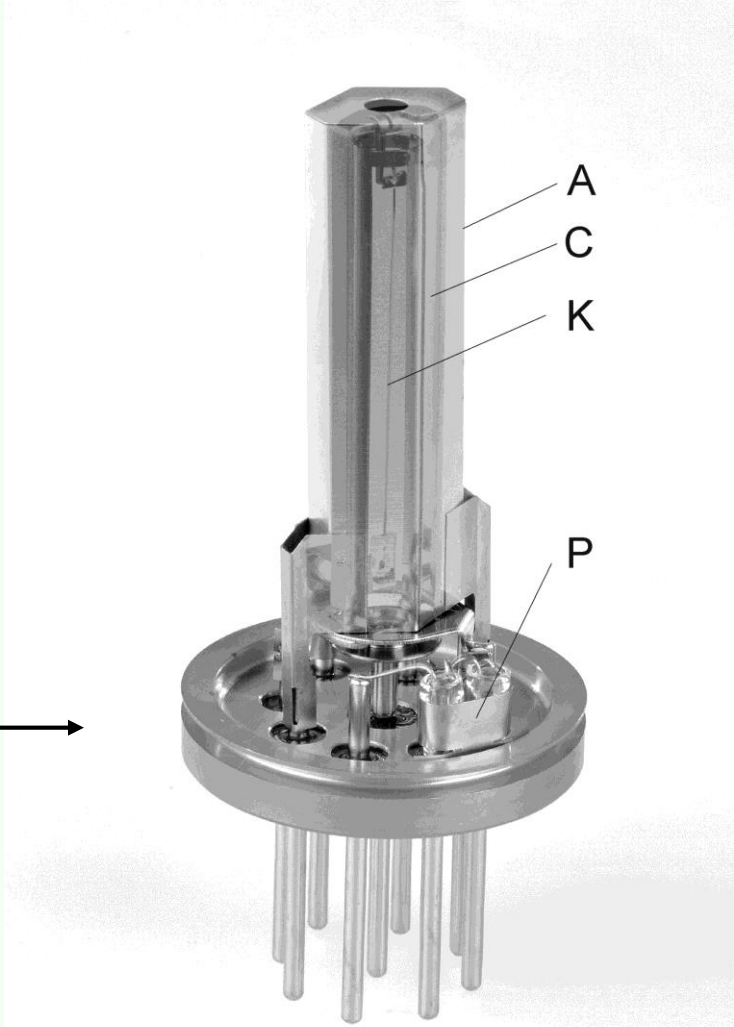


Measurement principle

Ionisation gauges: today‘s electrode design



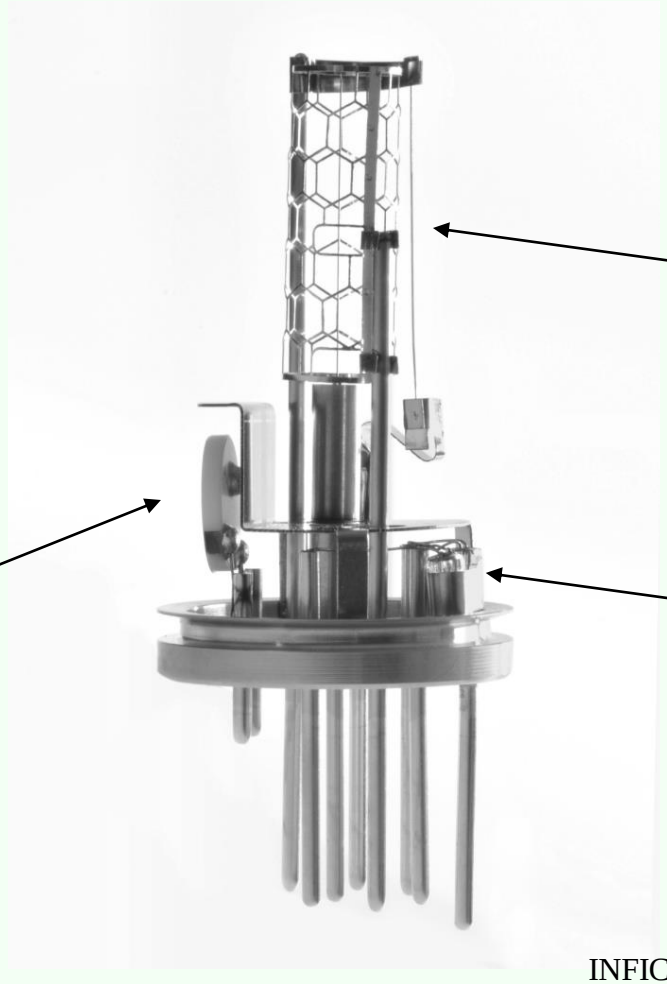
Development of burn-out-proof oxide cathodes allowed medium vacuum IG
Trick: Make the ion path as short as possible



Courtesy of INFICON AG

Measurement principle

Combined gauges



INFICON BCG 450 courtesy of INFICON AG

Ion gauges with emitting cathodes

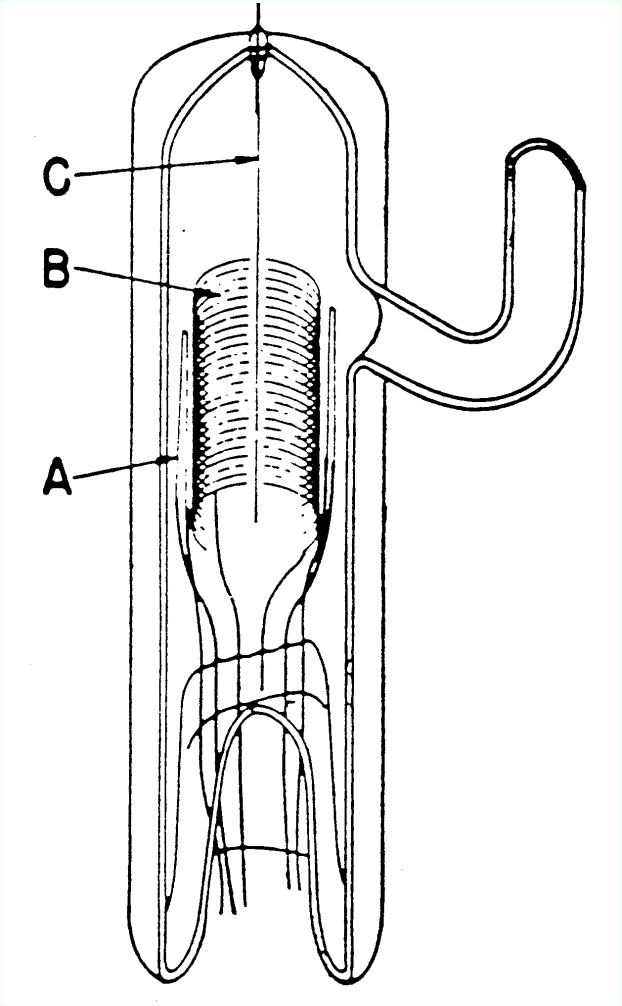
In the first half of the 19th century pumps improved, but the measured pressures with the triode gauges did not, - the readings were “nailed” to about 10^{-7} mbar.

Why?

IVC-1, 1947, Nottingham: X-rays?

The original Bayard-Alpert gauge

reduced the lower limit by 100

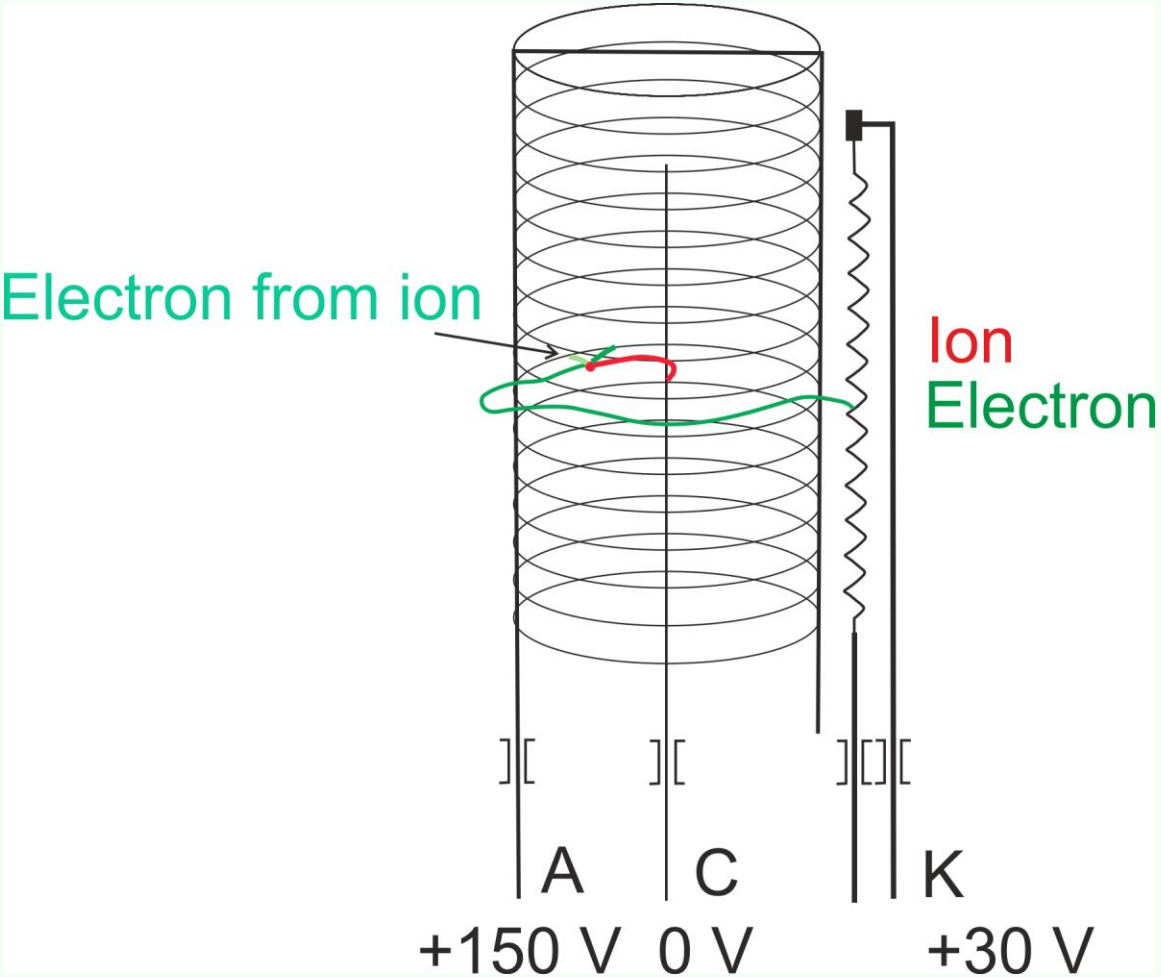


Bayard-Alpert design

Today no glass envelope in Europe: Nude gauge or metal envelope.

Emission current 0.1 mA … 10 mA

Electron path length typical 1.5 *D*

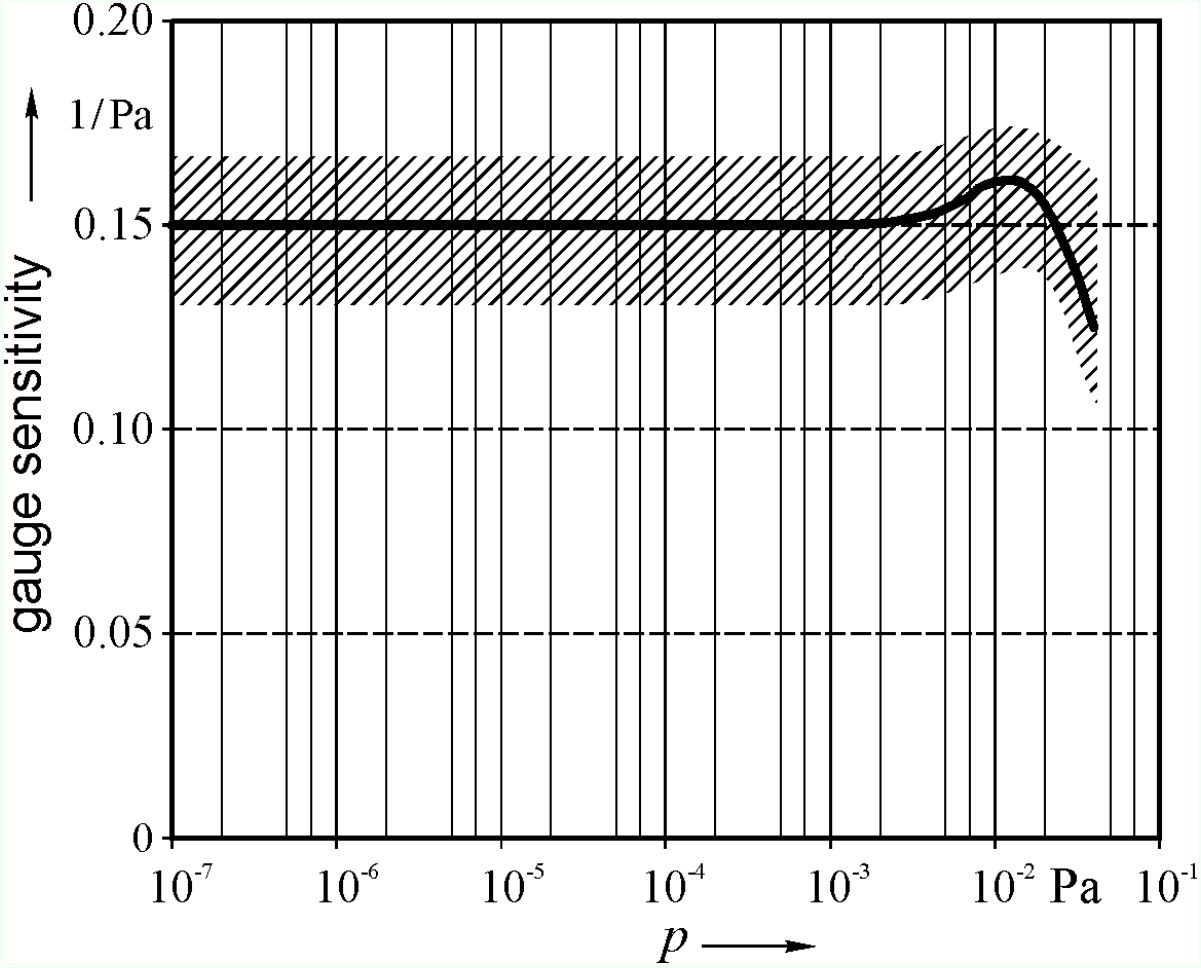


BAG calibration

$$I^+ = SI_e p$$

$$S = \frac{I^+ - I_0}{I_e p}$$

Dropping due to:
*I*ion = 0.002 *I*e @ 0.01 Pa
Mean free path



Ion gauges with emitting cathodes

Disturbing effects in Bayard-Alpert gauges

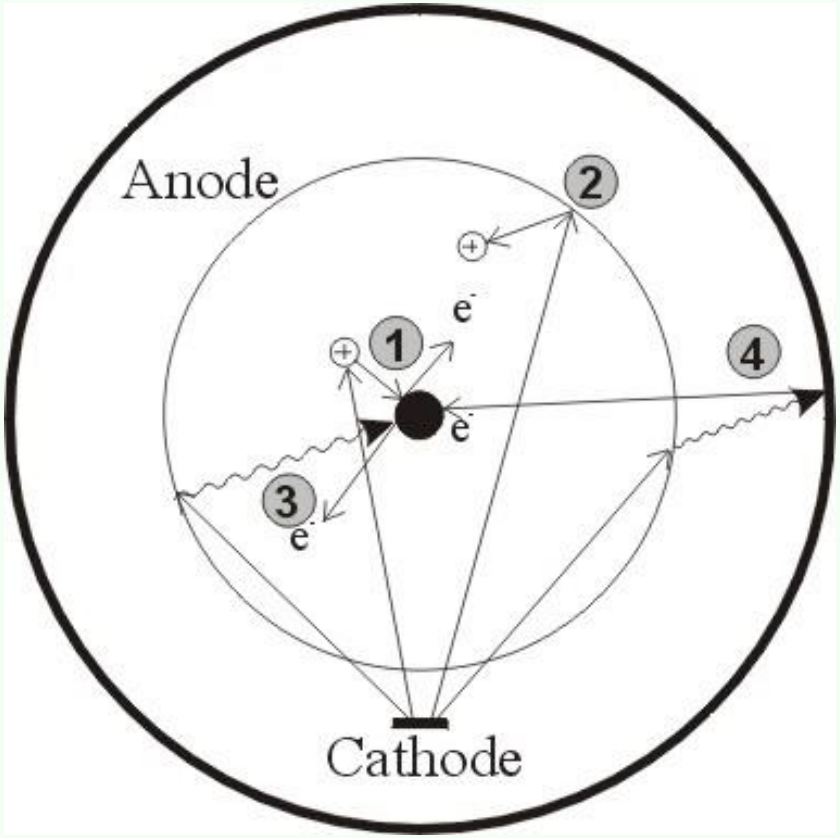
1: The desired ionisation, but secondary electron

2: Electron stimulated desorption

3: X-ray effect

4: Inverse X-ray effect

Other: e.g. Dissociation of molecules at hot cathode



Cross section of BA gauge

How does the number of adsorbate molecules compare with the ones in the gas phase in XHV?

Anode surface of typically 10 cm^2: 10^{16} molecules at one monolayer.

To be compared with density at 10^{-8} Pa: 10^6 molecules/cm^3 . In the gauge about 10 cm^3: 10^7 molecules.

10^9 more molecules on the surface: A huge reservoir to annoy you !!!

⟶ Degassing feature of IG

Not too small I_e (self-cleaning)

Towards XHV measurements

$$I^+ = I_e (n + n_{gen}) \sigma_{e,gas} \Delta l \, c_{ion,C} + I^-_{x-ray} - I^-_{tocollector} + I_{ESD}$$

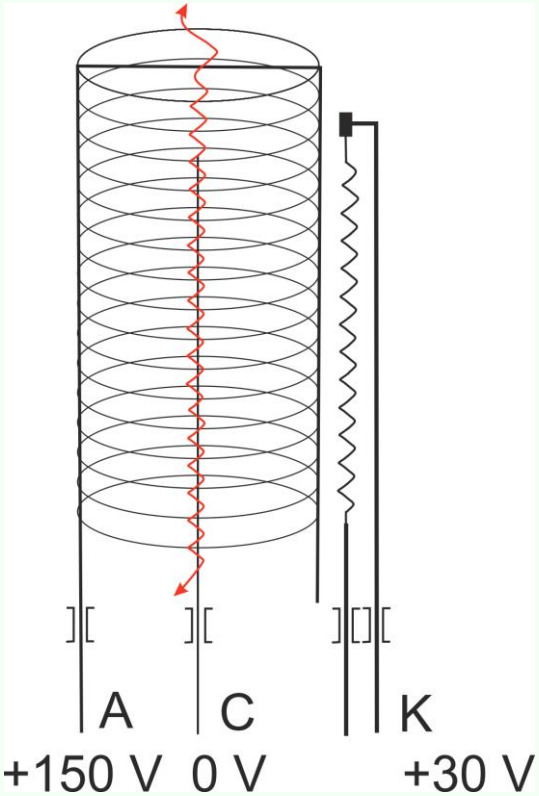
Strategies to decrease low pressure limit of emissive cathode ion gauges

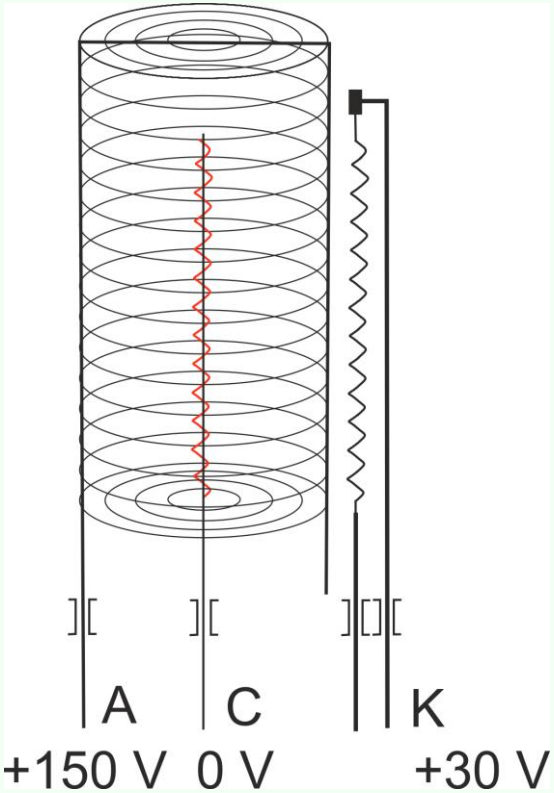
1. Increase Δl and
2. Increase ion collection efficiency $c_{ion,C}$
3. Reduce X-ray effect $I_{x\text{-}ray}$
4. Cancel forward and inverse X-ray effect
5. Reduce ESD ions and separate them from gas phase ions I_{ESD}
6. Reduce outgassing of the gauge n_{gen}
7. Amplify ion current / Reduce noise

2. Increase ion collection efficiency

- **Close the cylindrical grid anode**

Closed cylindrical grids have higher sensitivities than open grid structures.



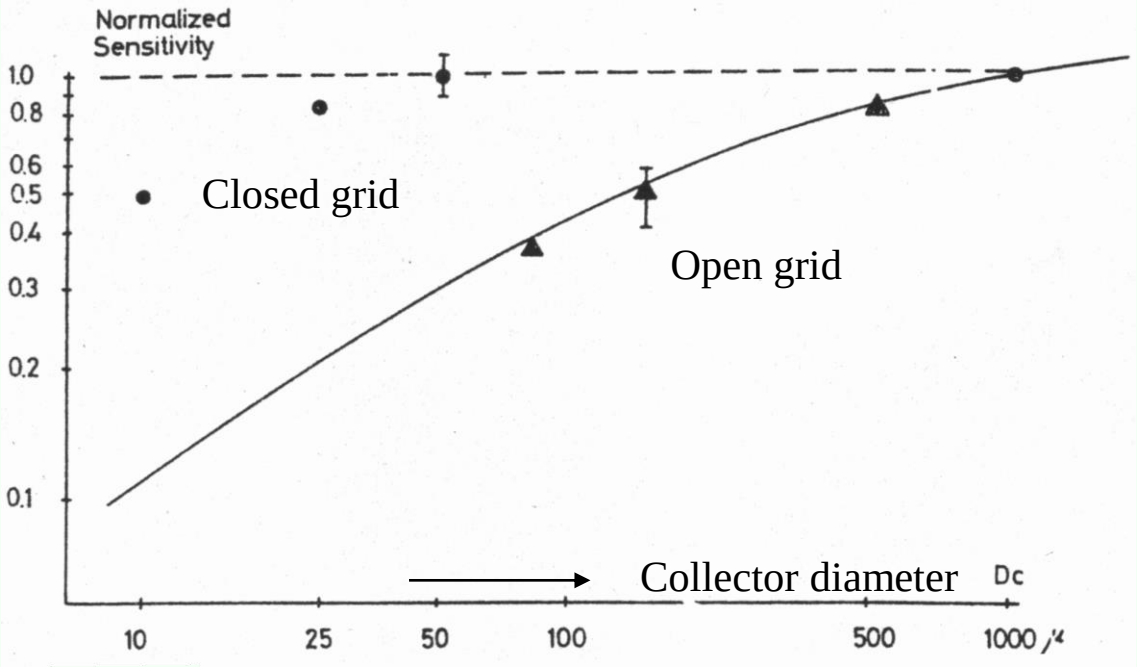


2. Increase ion collection efficiency

•Close the cylindrical grid anode

Closed cylindrical grids have higher sensitivities than open grid structures.

The smaller the collector diameter, the larger the effect.
e.g. Benvenuti, 1977: 25 mm diameter and closed grid, 40 mm length, 80 µm collector by a factor of 3 higher sensitivity compared to open one.



Benvenuti, Hauer, Nucl. Instr. and Meth. 140 (1977),453…460.

2. Increase ion collection efficiency

Even closed grids let ions escape

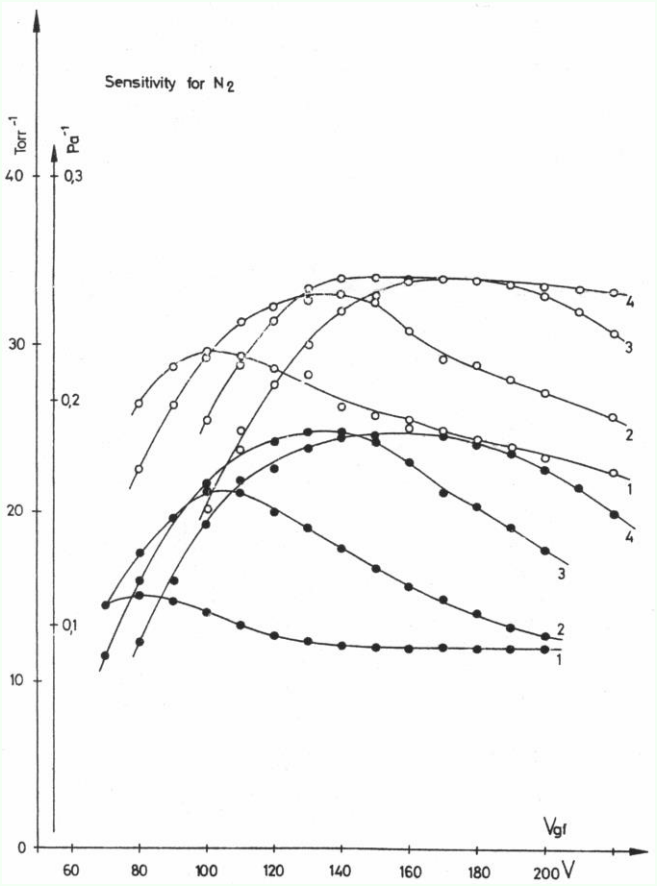
↓

Optimize filament potential V_f and grid-to-filament voltage V_{gf}

.

Collector diameter 50 µm

Collector diameter 10 µm



V_f

200 V

150 V

100 V

50 V

Benvenuti, Hauer, Nucl. Instr. and Meth. 140 (1977),453…460.

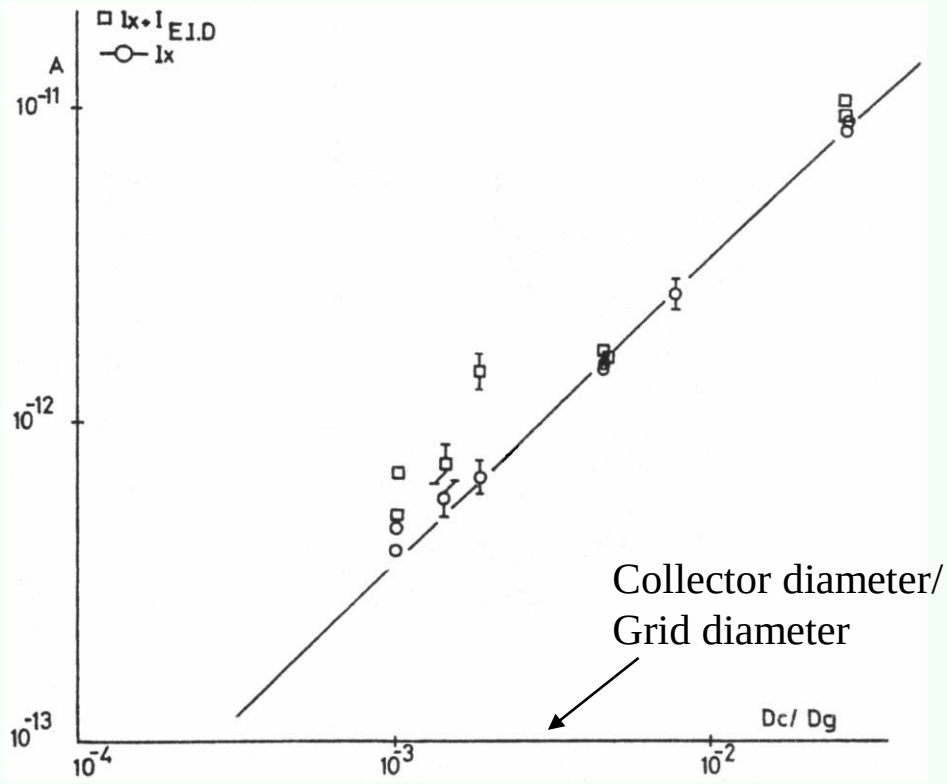
3. Reduce X-ray effect

In the BAG: Reduce the collector diameter

$I_{\text{x-ray}} \propto D$

Drawback: Due to conservation of angular momentum ions may spiral around the collector wire until leaving the grid.

Solution: ***Closed grids are a necessity, but sensitivity decreases anyhow.***



Benvenuti, Hauer, Nucl. Instr. and Meth. 140 (1977),453…460.

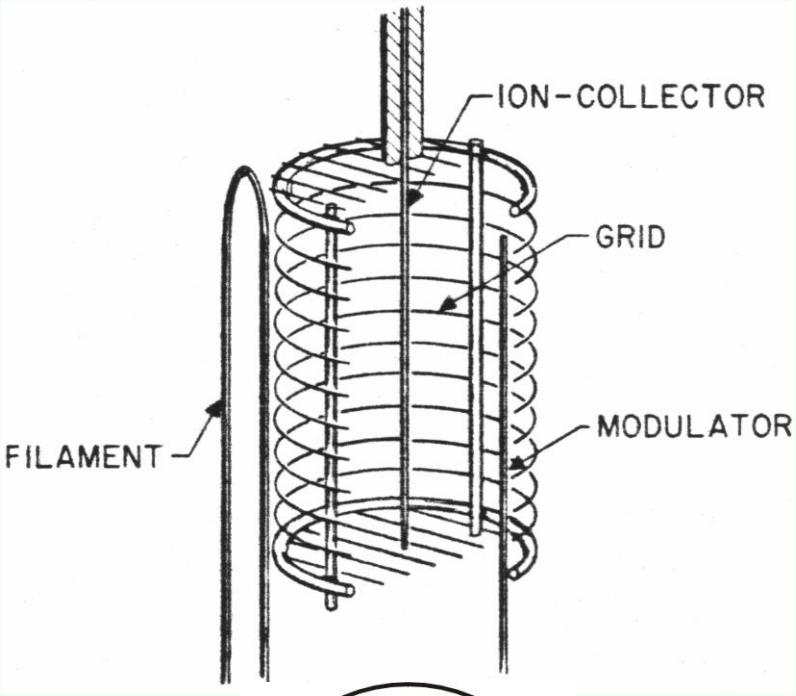
3. Reduce X-ray effect

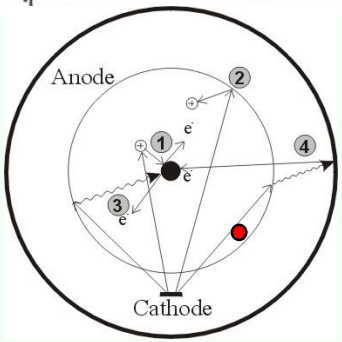
In the BAG: Measure and subtract the X-ray current.

Modulator (Redhead) switches between grid (collects no ions) and collector potential (collects ions).

Drawback: Also X-ray current is modulated due to the change of electron trajectories.

Result: Higher complexity, commercially used, today rarely.





3. Reduce X-ray effect

Idea of construction: Decouple ion collection from ionisation region, so that there is no line of sight for X-rays* from grid to collector.

The extractor gauge

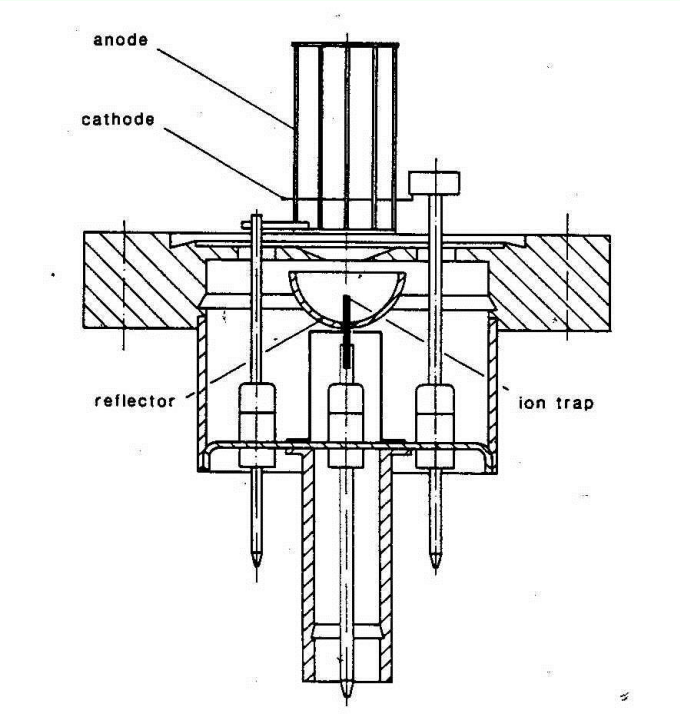
The Helmer gauge

The Ion Spectroscopy gauge

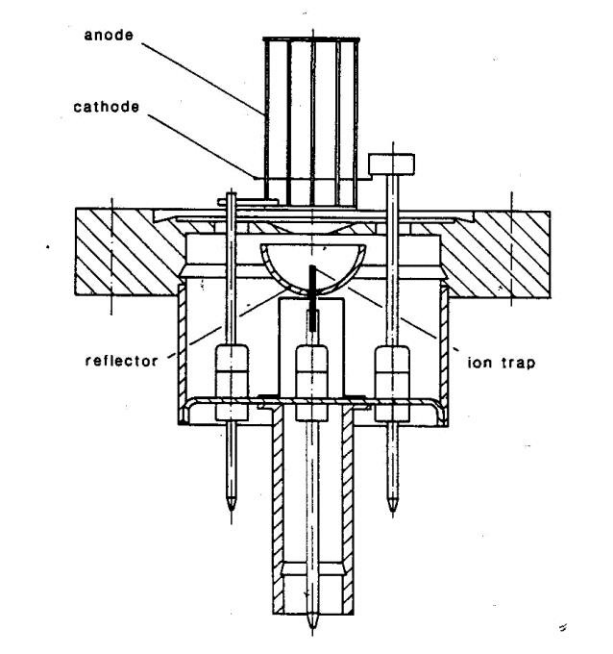
The Oshima gauge

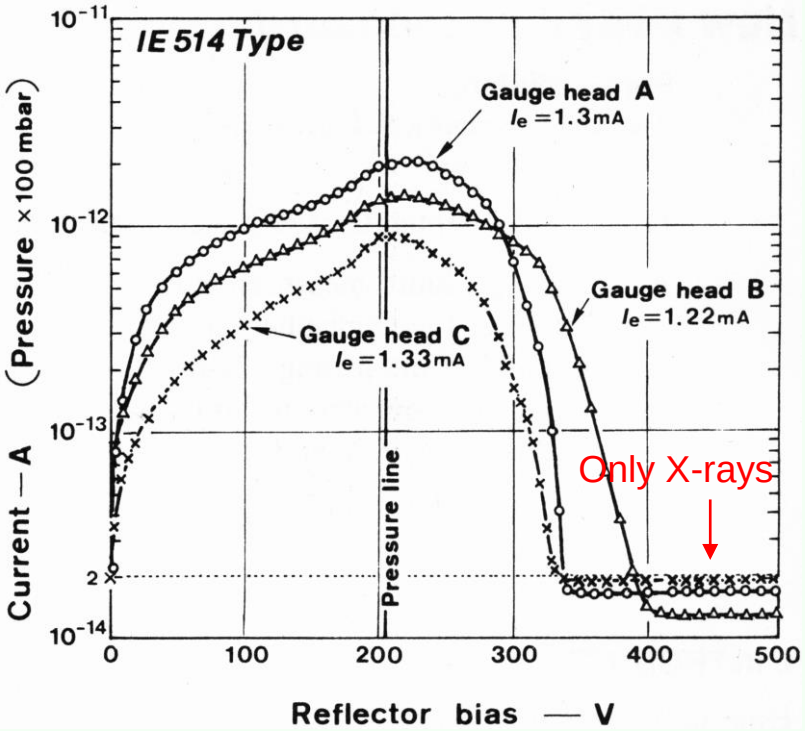
The AxTran gauge

* X-rays have high reflection coefficient from most metals!



3. Reduce X-ray effect





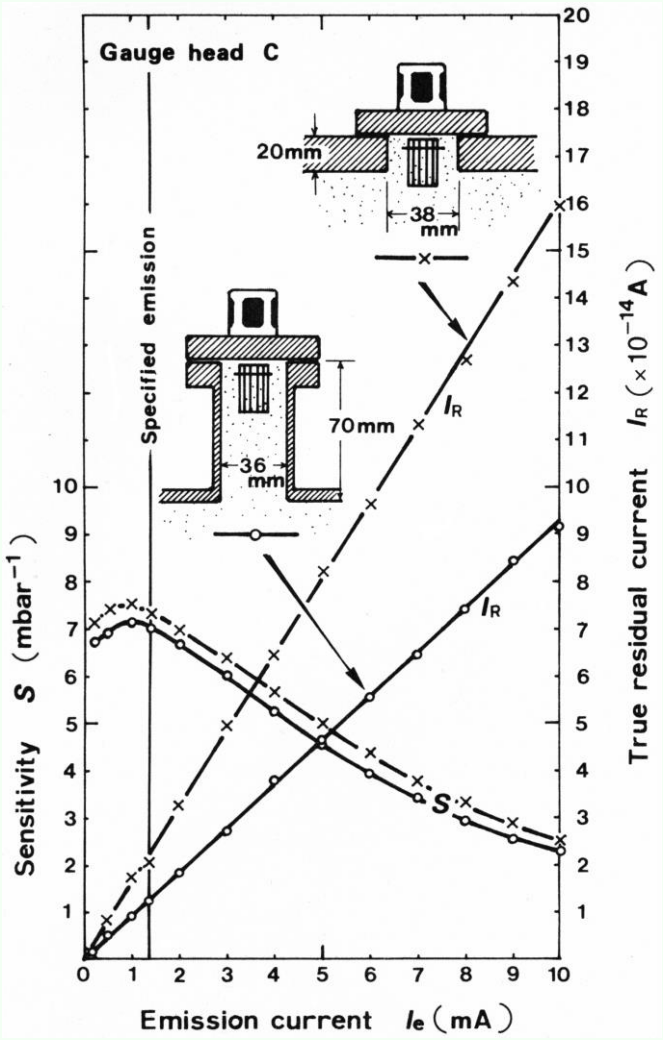
F. Watanabe, JVST A 9 (1991), 2744…2746.

3. Reduce X-ray effect

Tubulation changes the sensitivity by 5%, but also the X-ray limit by 70%, probably due to the inverse X-ray effect.

4. Cancel forward and inverse X-ray effect

Unstable because of changing SEY with surface changes.
Same material of tube and collector for the sake of stability, or vary tube potential.



F. Watanabe, JVST A 9 (1991), 2744…2746.

5. Reduce ESD ions and separate them from gas phase ions

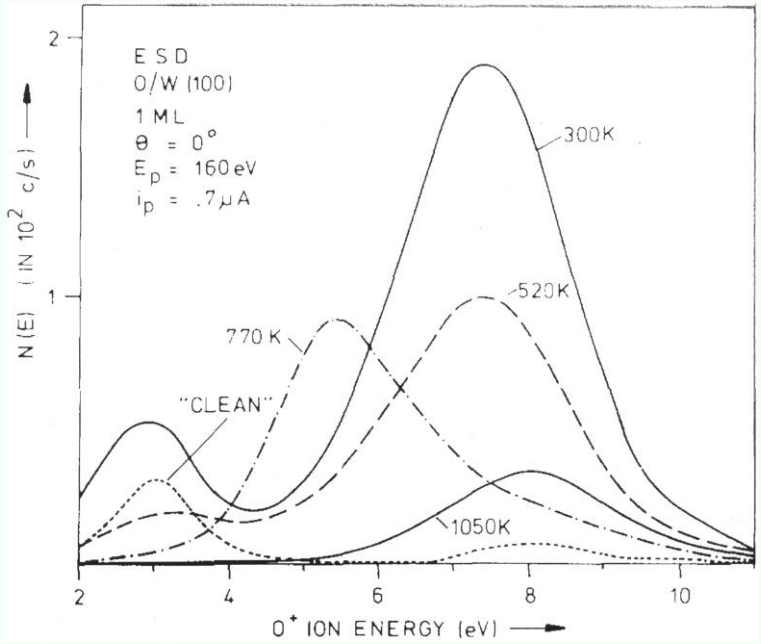
Effect: ESD ions have an higher energy than ions generated in the gas phase for two reasons.

Reason 1: Higher initial energy

Reason 2: Electrostatic potential inside the grid.

Idea: Analyze the energy of the ions before reaching the collector.

The extractor gauge
The Helmer gauge
The Ion Spectroscopy gauge
The AxTran gauge

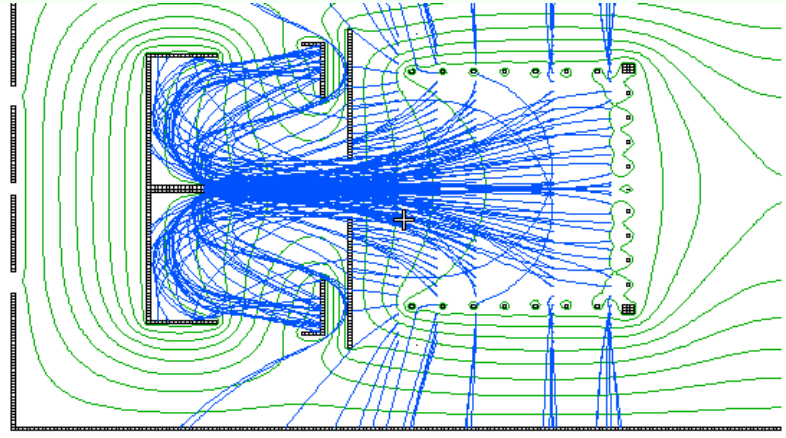


S. Prigge, H. Niehus, E. Bauer, Surf. Sci. 75 (1978), 635.

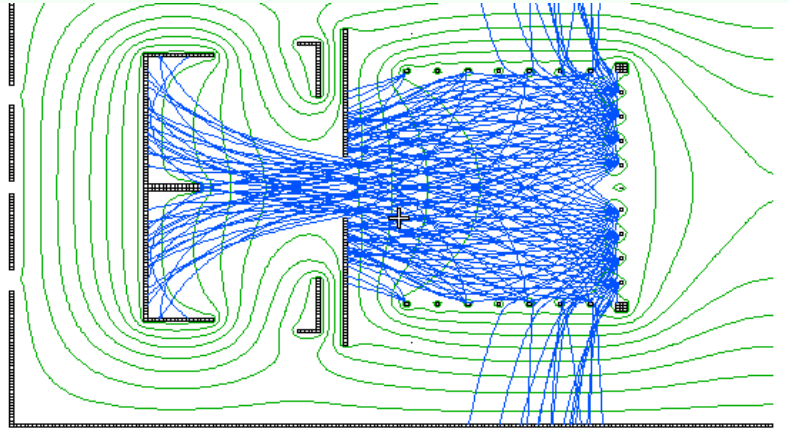
5. Reduce ESD ions and separate them from gas phase ions

The extractor gauge

Ionisation from gas phase



Ionisation from surface



SIMION applied by O.F. Kieler, University of Magdeburg

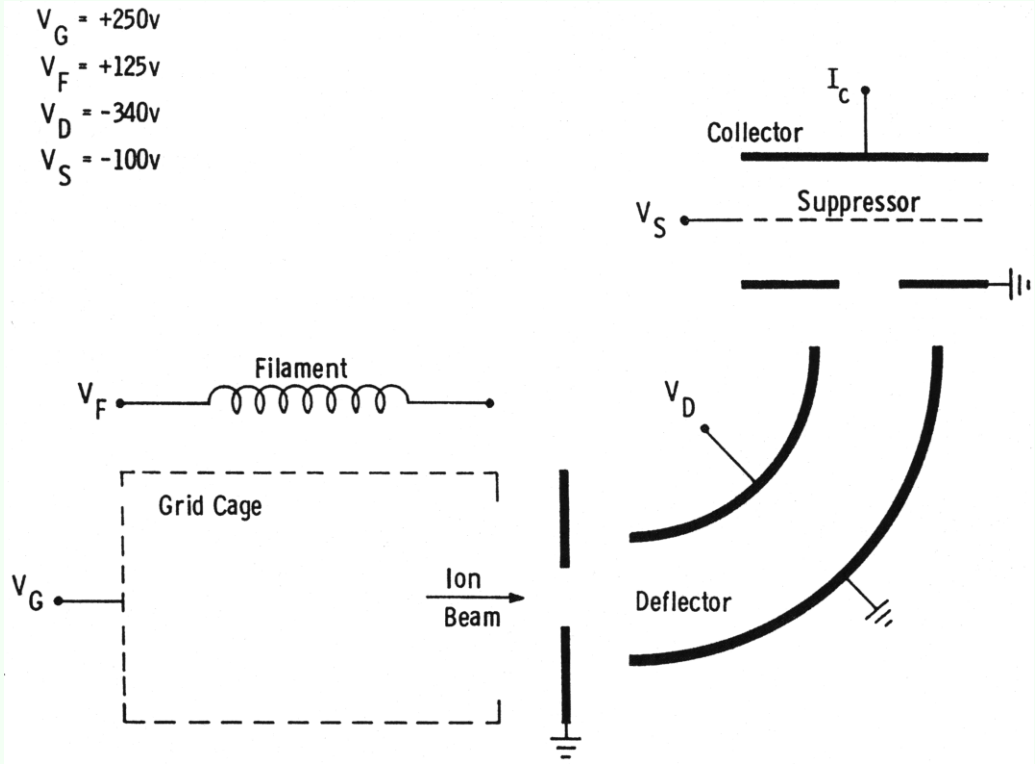
5. Reduce ESD ions and separate them from gas phase ions

The Helmer gauge

J.C. Helmer and W. Hayward, Rev. Sci. Instr. 37 (1966), 1652.

90° for reduction of X-rays, later also to reduce ESD ions.

Measured 7×10^{-11} Pa, but capable of 3×10^{-12} Pa.



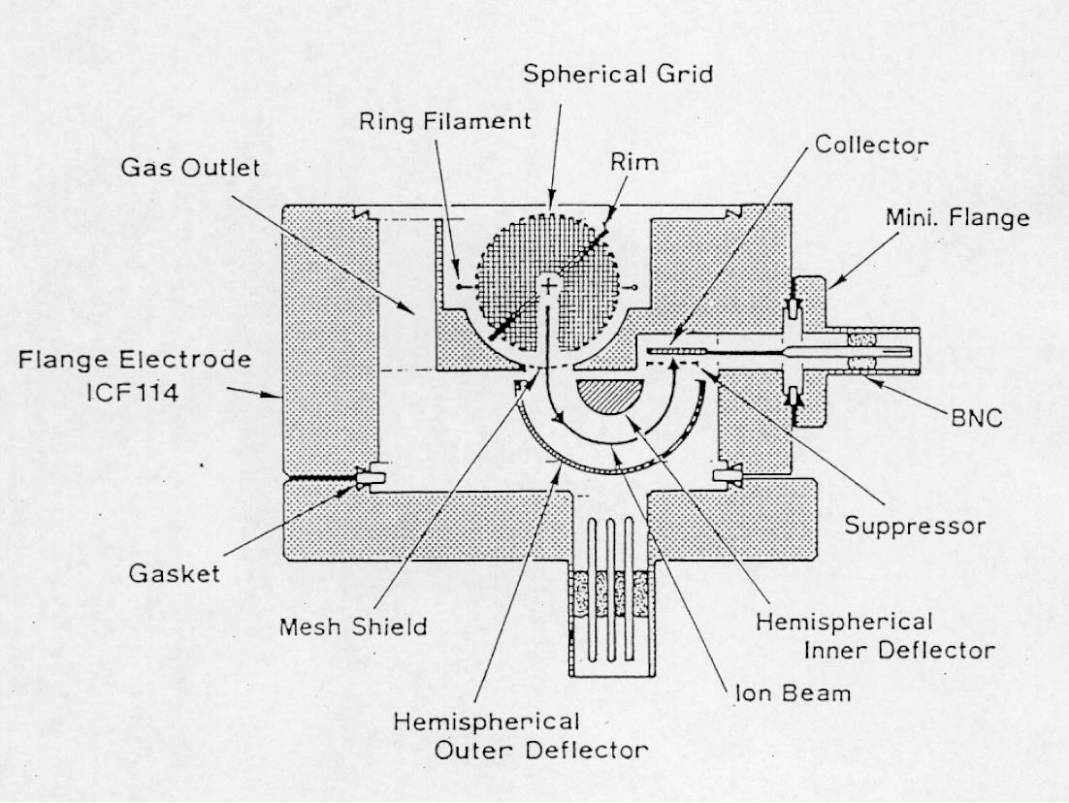
5. Reduce ESD ions and separate them from gas phase ions

The Ion Spectroscopy Gauge by Fumio Watanabe

Spherical grid and ring filament for enhanced space charge

Heated grid

3D-Hemispherical analyser for full collection efficiency

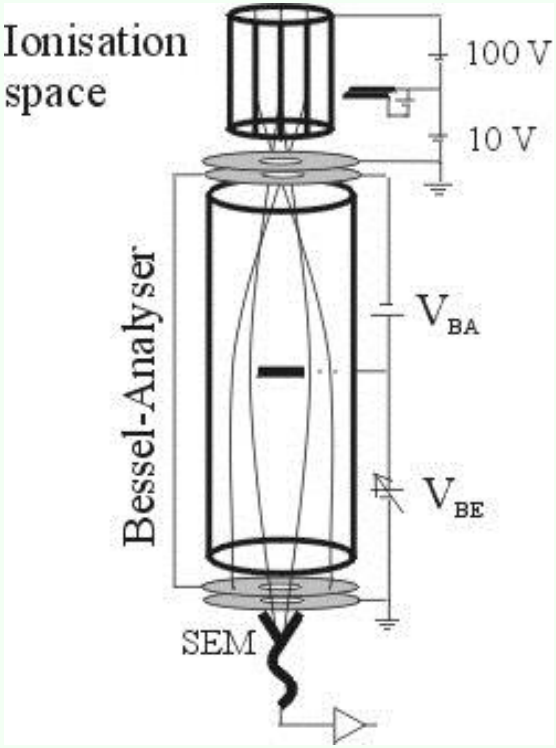


5. Reduce ESD ions and separate them from gas phase ions

The AxTran gauge by ULVAC

X-ray limit 4E-11 Pa nitrogen equivalent

Lowest detectable pressure 3E-12 Pa



5. Reduce ESD ions and separate them from gas phase ions

Idea: Reduce the number of ESD ions by

•Cleaning the grid (electron bombardment)

•Choosing suitable material (platinum-iridium alloy, platinum clad molybdenum, gold. Less good experience with W-re alloy or Au-plated SS)

•Using a clean vacuum system (no hydrocarbons)

5. Reduce ESD ions and separate them from gas phase ions

But there are limits !!!

Desorbed by electrons hitting the grid surface are:

1.	Ionized molecules	*Can be separated*
2.	Ionized fragmented molecules	*Can be separated*
3.	Neutral molecules*	***Cannot** be separated!!!*
4.	Fragmented neutral molecules	*Difficult to separate (by cracking pattern)*

* Neutrals from a Pt-Ir grid dominate after bake-out!

6. Reduce outgassing of the gauge

What outgasses thermally?

- The filament itself
- Material heated by the hot filament
- Anode material heated by electrons (10 mA, 100 V generates 1 Watt !!)
- All other materials in the gauge (that are more or less at “room” temperature)

6. Reduce outgassing of the gauge

Strategies:

a. Choose suitable filament material

b. Reduce heating power of filament

c. Reduce emission current

d. Use cold cathodes

e. Use materials inside gauge and for its tubulation of low emissivity, high thermal conductivity and high heat capacity

f. Degas materials before operating the gauge

7. Amplify ion current / Reduce noise

Secondary electron amplifiers were extensively used by many investigators, even ion counting techniques.

Problems: Outgassing, stability.

Also Lock-in technique was used (Watanabe, 1989).

Comparison of the types of ionisation gauges

Max. deviations in % from 1 st calibration within 6 months

	EXG	**BAG1**	**BAG2**	**IMG1**	**IMG2**
N_2	-2.5	-4.3	-3.2	-6.2	+5.9
Ar	-1.9	-3.8	+3.8	-2.4	+3.1
He	-5.9	-4.4	-3.6	+8.4	-5.0
H_2	+9.4	-1.9	-3.6	-1.0	-1.3

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.

Comparison of the types of ionisation gauges

Outgassing rates in Pa L/s

EXG at 1.5mA	BAG1 at 4mA	BAG2 at 1mA	IMG1/IMG2
$2.4 \cdot 10^{-8}$	$8.1 \cdot 10^{-8}$	$3.0 \cdot 10^{-8}$	none

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.

Comparison of the types of ionisation gauges

Pumping speeds in L/s

Gas	IMG1	IMG2	BAG1 at 4mA	BAG2 at 1mA	BAG2 at 10mA
N_2	0.045	0.065	0.019	-	0.045
Ar	0.2	0.21	0.067	0.037	0.23

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.

Applications

Problems special to accerators:

Radiation (Example: IG close to photon absorber)

Strong magnetic fields (shielding necessary)

RF radiation (Example: RF cavity -> shield)

Applications

Problem: IG measures density, not pressure

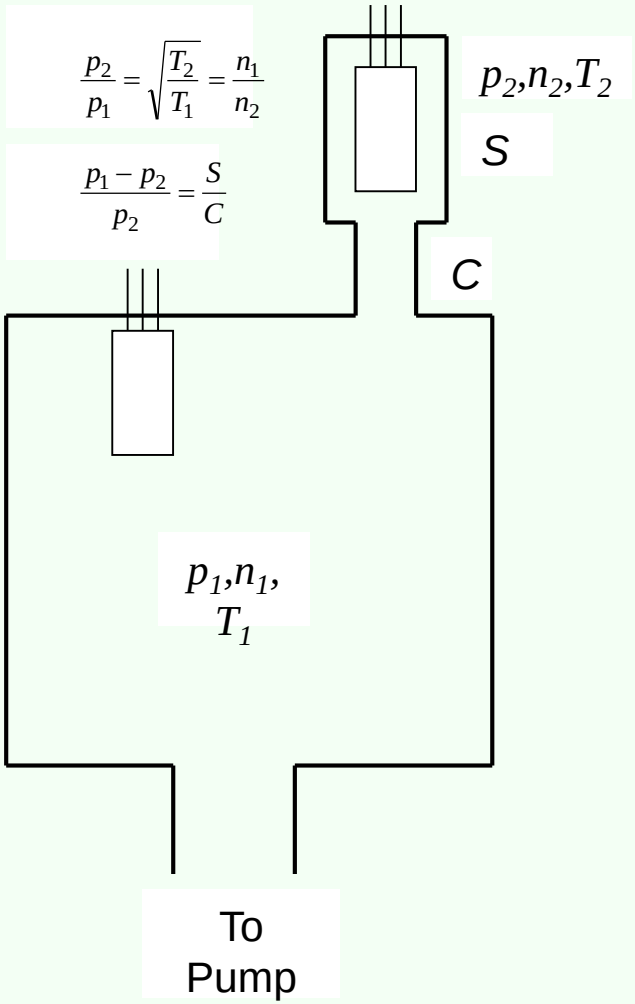
Sealed-off chamber, cool it down.

$$\frac{p_2}{p_1} = \frac{T_2}{T_1} = \frac{77}{300} = 0.257$$

But IG will have same reading!

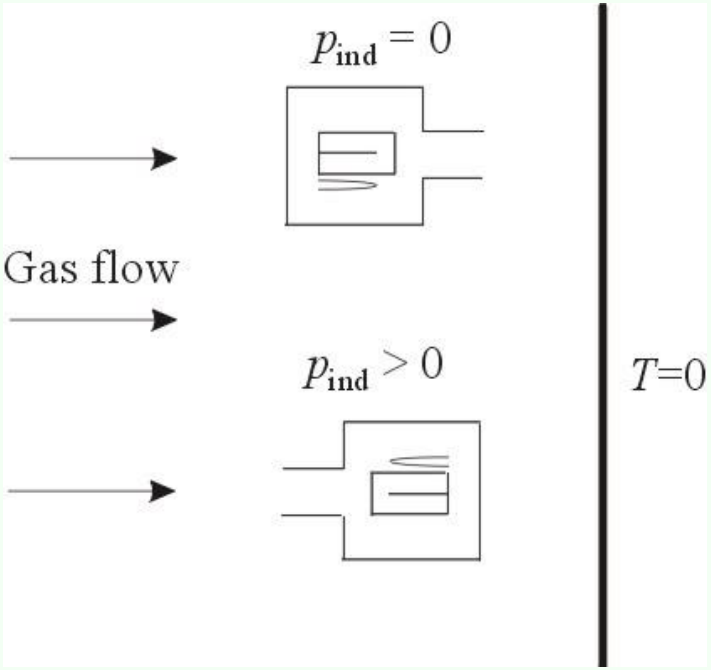
Applications

The effects of tubulation, conductance, pumping speed, and thermal transpiration



Applications

Orientation of a gauge



Quadrupole mass spectrometers

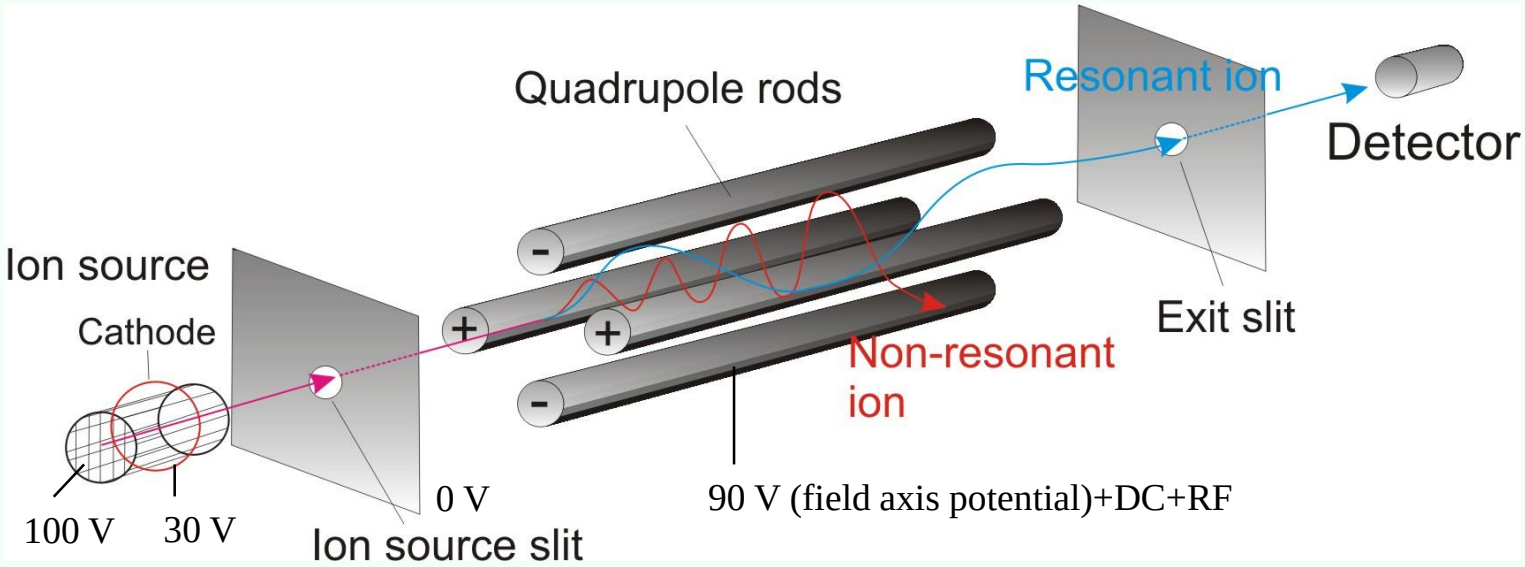
Advantages of mass spectrometers:

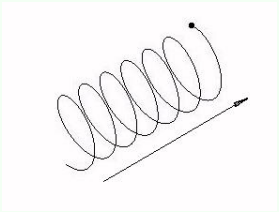
- Residual gas is automatically analyzed
- X-ray effect is intrinsically reduced
- High sensitivity (multiplier 1E-10 Pa, off-axis multiplier for XHV 1E-12 Pa)

Disadvantages of mass spectrometers:

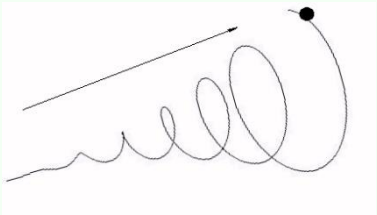
- Producing significantly residual gases
- Changes gas composition
- Still ESD effect (QMS with energy analyzer before quad, e.g. Bessel-box)
- Higher outgassing than total pressure gauge
- Difficult to calibrate

Quadrupole mass spectrometers





Resonant ion



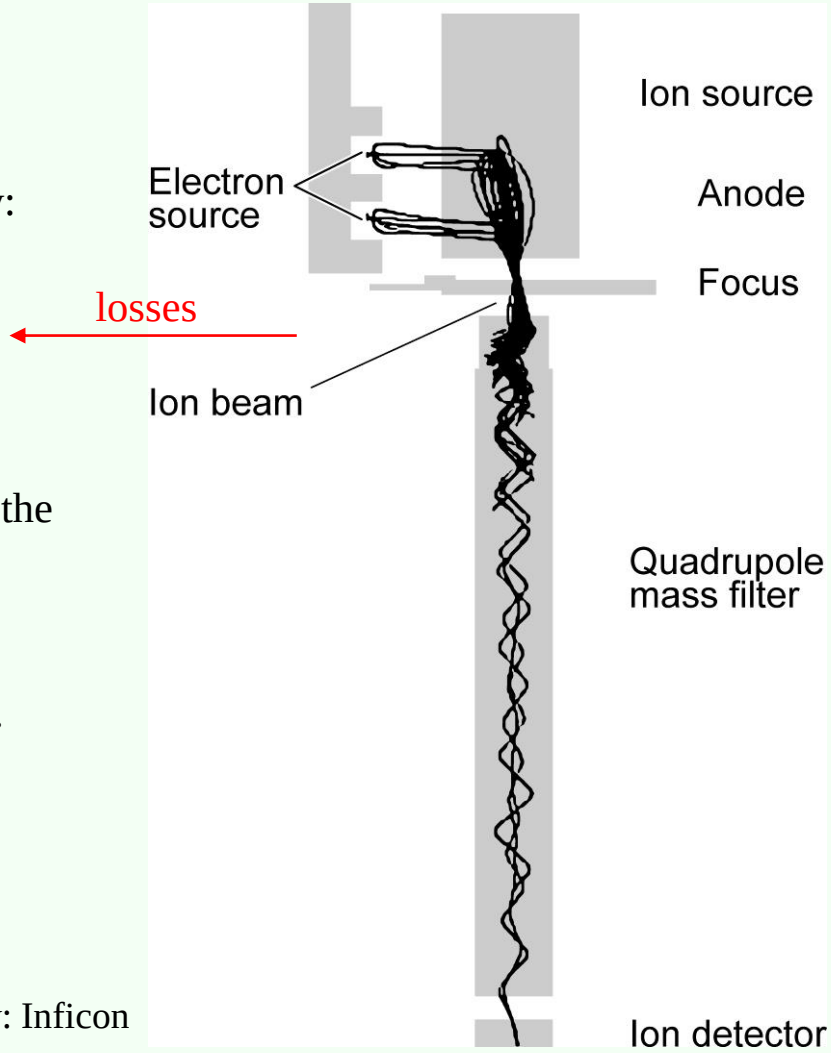
Non-resonant ion

Quadrupole mass spectrometers

Extraction and transmission probability:

Sensitivity of QMS: 10^{-6} A/Pa compared to ionization gauge of about 10^{-4} A/Pa

Ideally, oscillations are independent of the energy of the ion (energy spread is not critical, simple ion source feasible).
But fringe fields have the higher effect the longer the flight time through them.
→ Ion energy does matter!



Courtesy: Inficon

Quadrupole mass spectrometers

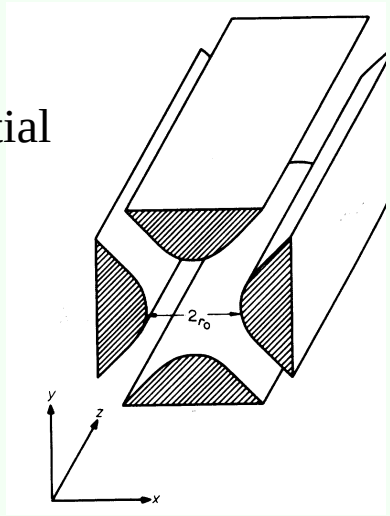
$$V = +\left[U + u\cos(\omega t)\right] + FA \qquad FA \text{ field axis potential}$$

$$V = -\left[U + u\cos(\omega t)\right] + FA$$

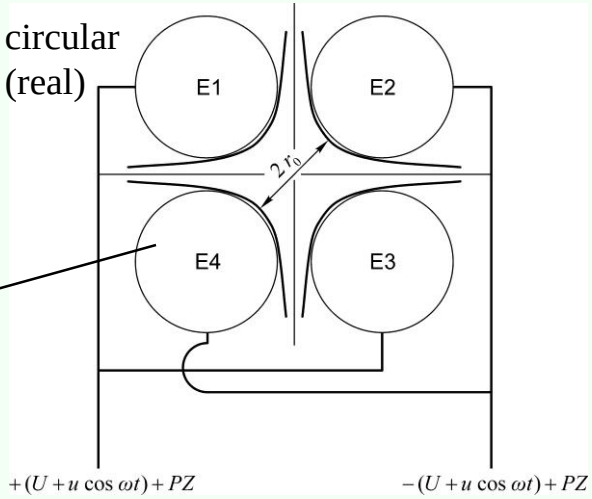
Stability characterized by two parameters (Mathieu's equations):

$$a = 0.194\frac{zU}{mr_0^2 f^2}$$

$$q = 0.097\frac{zu}{mr_0^2 f^2}$$



$$r_{\text{cyl}} = 1.147 r_0$$



Quadrupole mass spectrometers

Stability characterized by two parameters (Mathieu‘s equations):

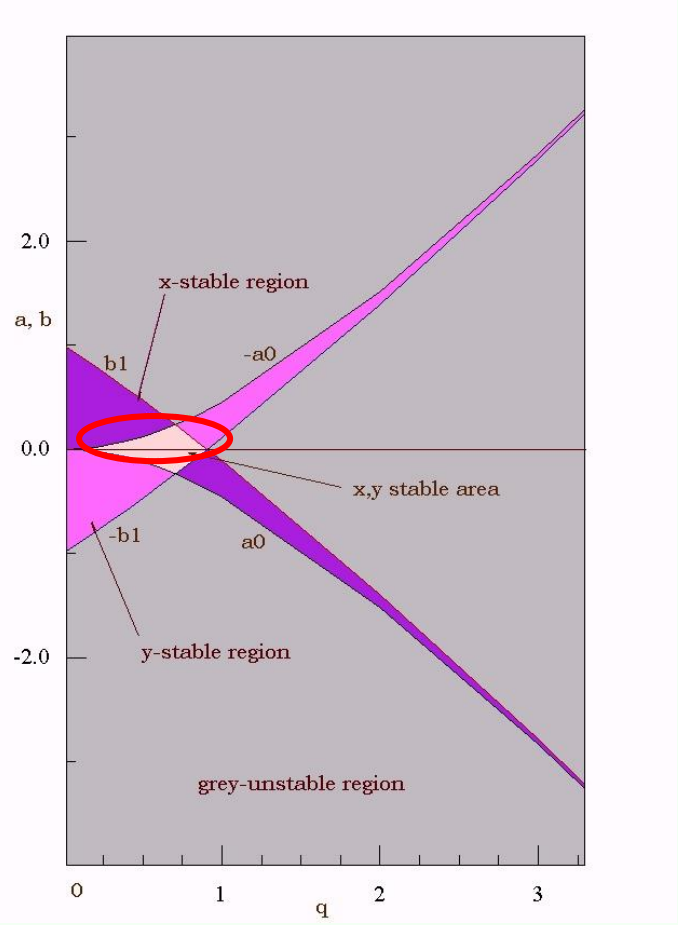
$$a = 0.194 \frac{zU}{mr_0^2 f^2}$$

$$q = 0.097 \frac{zu}{mr_0^2 f^2}$$

Solved as eigenvalue problem

Stable states for oscillatory motion for special *a* and *q*

Symmetry between x and y



Quadrupole mass spectrometers

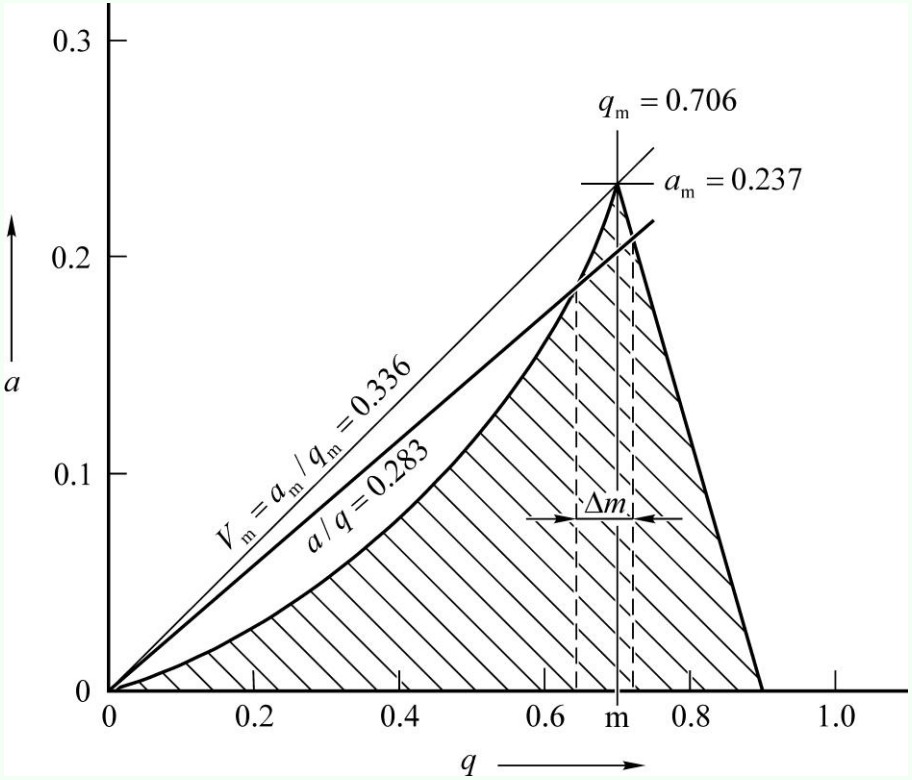
Stability characterized by two parameters (Mathieu‘s equations):

$$a = 0.194 \frac{zU}{mr_0^2 f^2}$$

$$q = 0.097 \frac{zu}{mr_0^2 f^2}$$

$$\frac{a}{q} = \frac{2U}{u}$$ *All* ions!

$$\frac{2U}{u} < 0.336$$ Detection possible



All masses/ions lie on common straight line. Line defines min and max of mass filter.

Quadrupole mass spectrometers

Close to the tip of stability diagram the transmission probability is proportional to the resolution:

$$T \propto R = \frac{m}{\Delta m} = \frac{1.5064}{1 - \frac{U/V}{(U/V)_{\max}}}$$

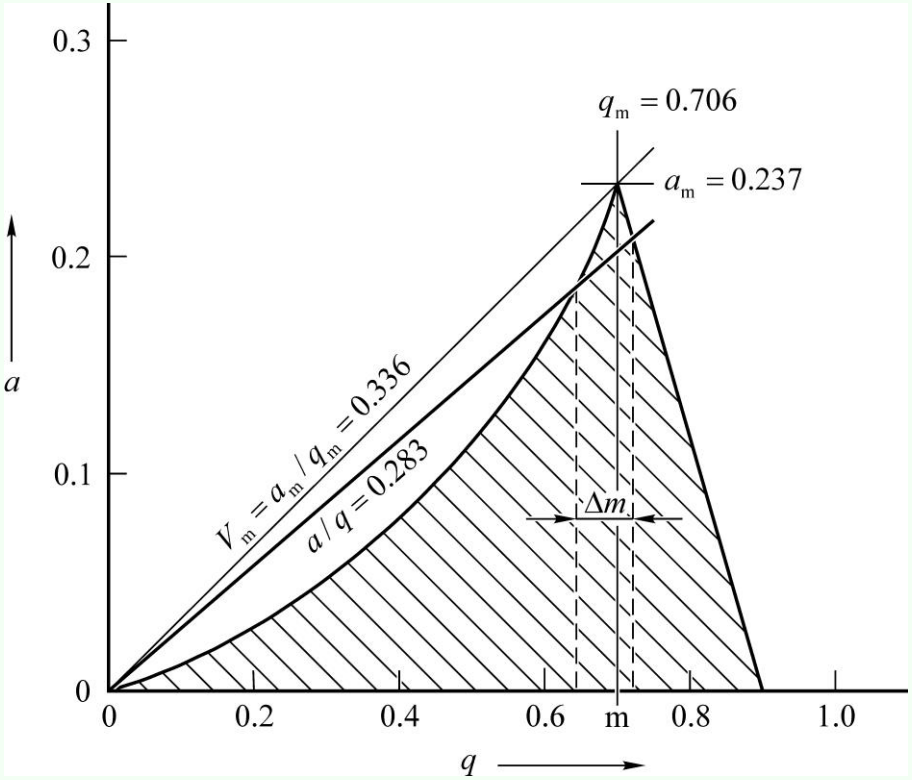
For R=100 the voltage ratio is 0.9849

For R=110 this ratio 0.9863

This difference of 0.14% corresponds to a 10% peak change!

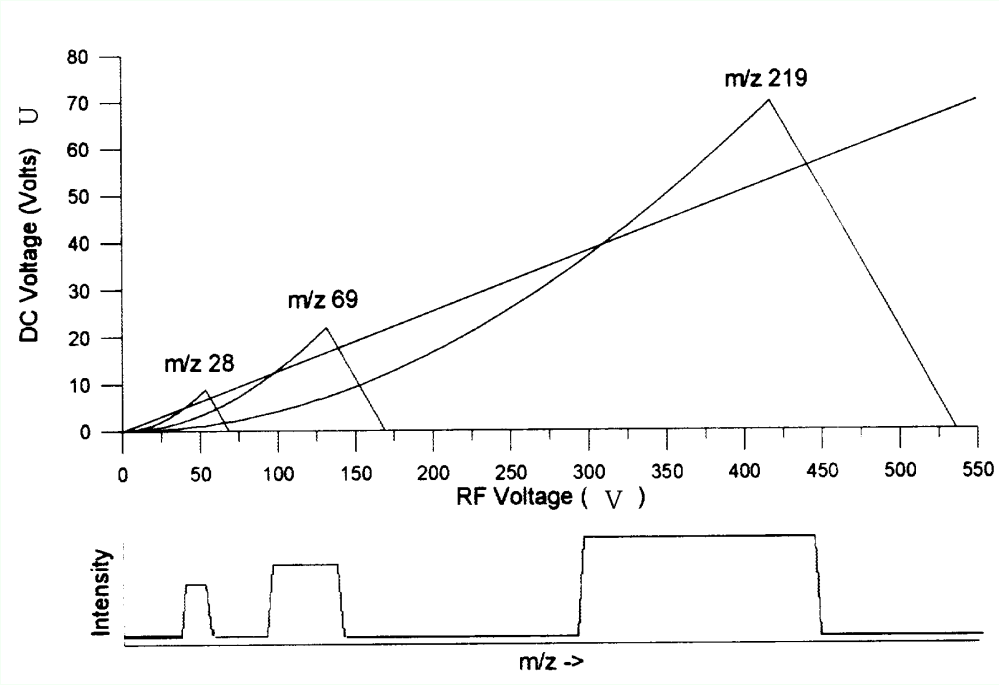
Similar stability of U/V is required for peak stability.

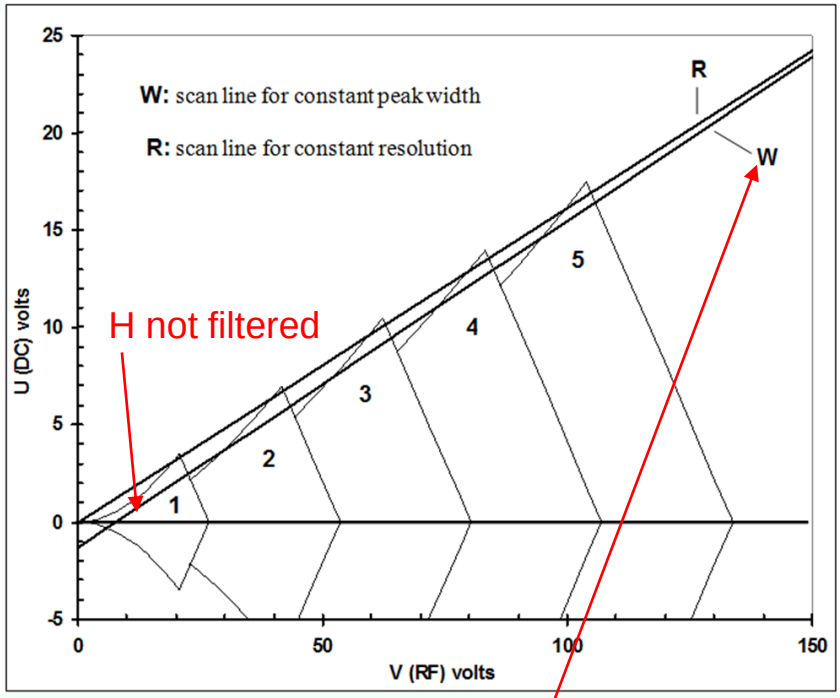
$T \propto$ area above line / below line



Quadrupole mass spectrometers

To detect higher mass *U*(DC) and *u*(RF) must be increased



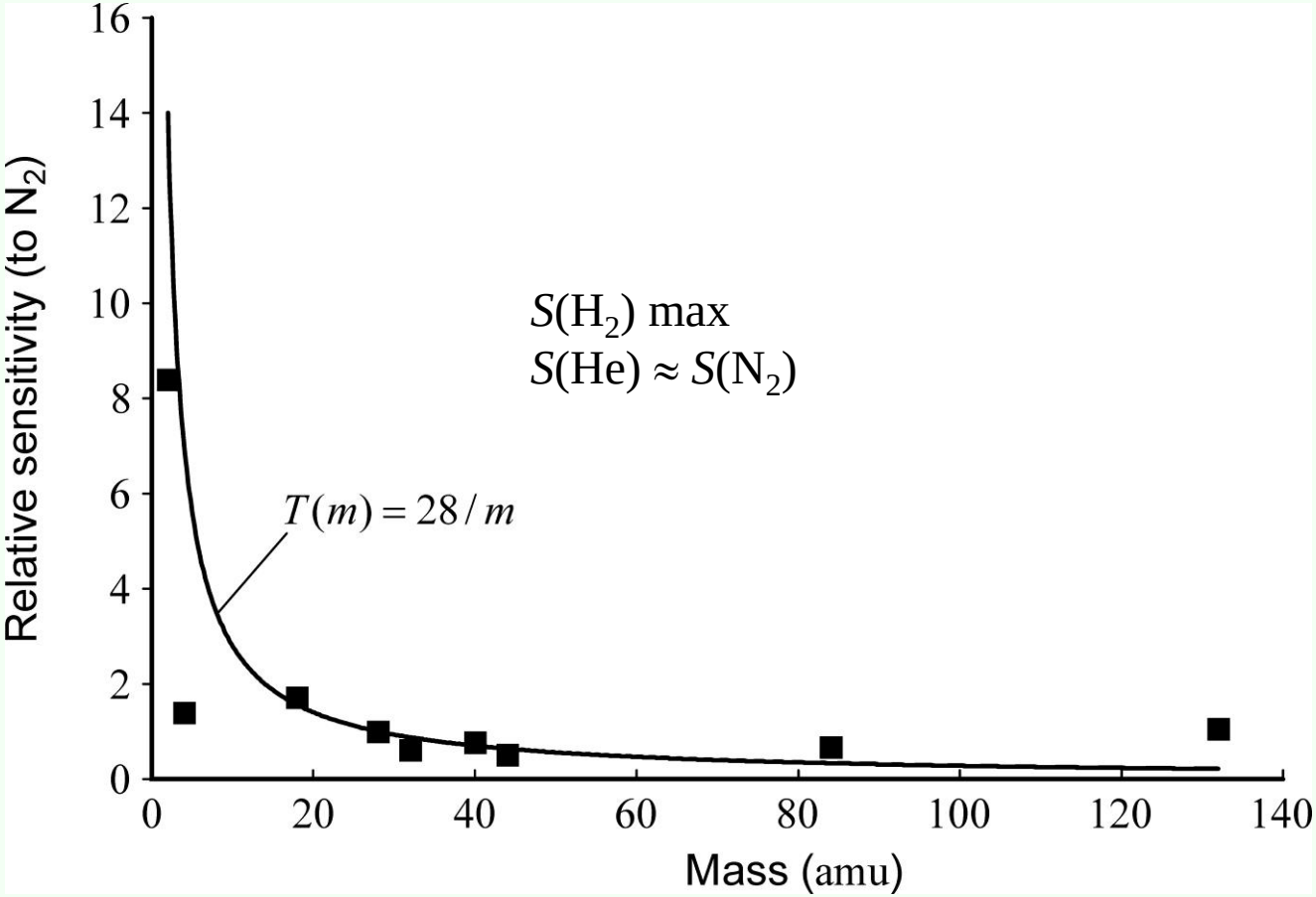


U(DC)/*u*(RF) constant

U(DC)/*u*(RF) not constant

$$\frac{m}{\Delta m}T = const \qquad \Delta m = 1 \quad \rightarrow \quad T = \frac{const}{m}$$

Quadrupole mass spectrometers



Quadrupole mass spectrometers

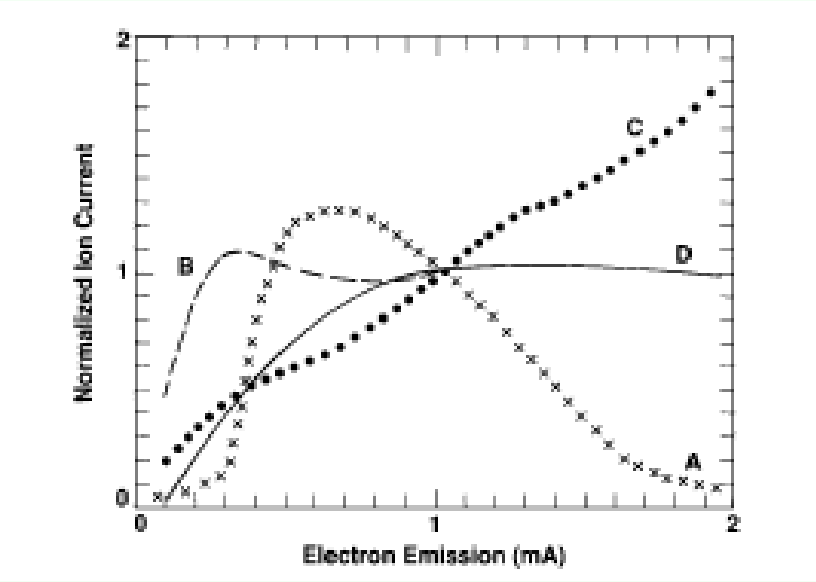
From ion gauges we are used to that

$$I^+ = SI_e p$$

For QMS →

Reason:

More electrons → negative space charge in ion source ⟶ ion extraction efficiency to the quadrupole and/or transmission probability through the quadrupole is decreased



Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854

Quadrupole mass spectrometers

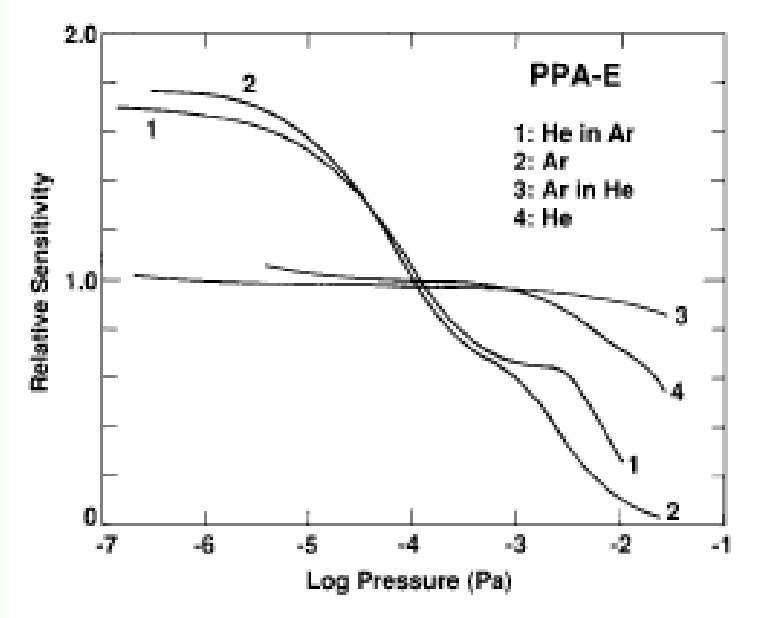
Sensitivity on gas species for different QMS. Huge differences and hardly related to ionization probability!

Gas species	A	B	C	D	E
Ar	1,96	1,33	1,56	2,08	1,23
CH_4	1,18	0,96	1,23	0,77	0,86
CO_2	1,47	0,77	1,39	1,52	0,94
N_2O	1,27	0,47	0,91	0,90	0,63
CO	1,04	0,99	1,05	1,00	0,99
N_2	1,00	1,00	1,00	1,00	1,00
H_2	0,61	1,09	1,42	0,31	1,54
He	0,10	0,35	0,31	0,48	0,40

Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854

Quadrupole mass spectrometers

Tracer kept constant at 1E-6 Pa.
Matrix gas was increased.



Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854

Quadrupole mass spectrometers

Calibration for gas combinations

About 20 relevant gas species (H_2, He, Ne, Ar, Kr, Xe, N_2, O_2, CH_4 + higher hydrocarbons, CO, CO_2, H_2O, NO_x, SiH4, …, SF6, dry air) for total pressure gauges

More relevant gases for QMS (even proteins etc.), 100?

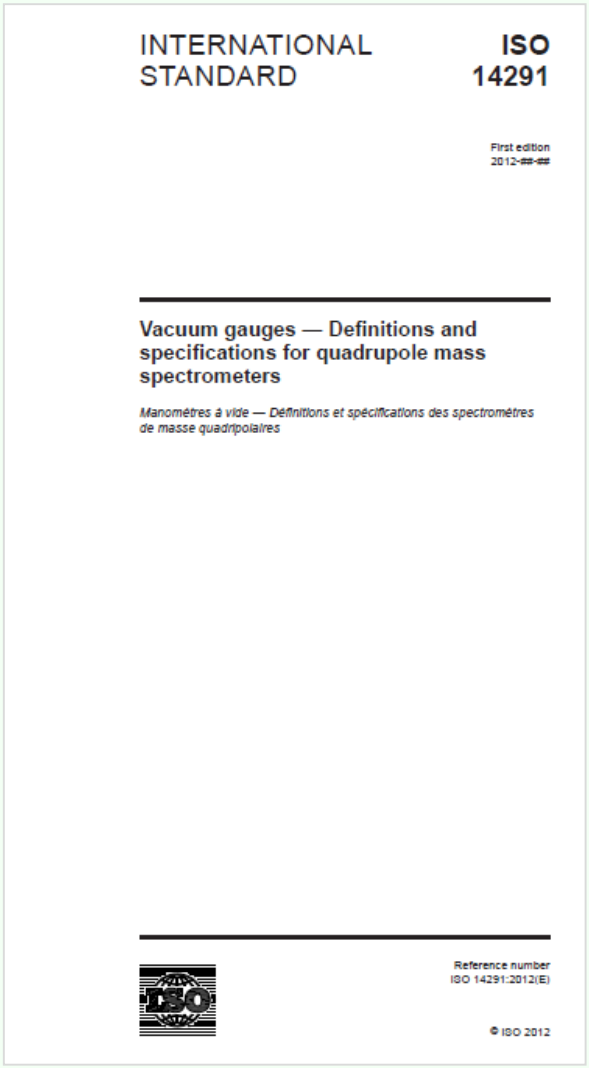
For 100: $1.27 \cdot 10^{30}$ possibilities of combination of gas species!

For 30: $1.07 \cdot 10^{9}$ possibilities of combination of gas species!

For 10: 1023 possibilities of combination of gas species!

Impossible task!!!

Quadrupole mass spectrometers



INTERNATIONAL STANDARD

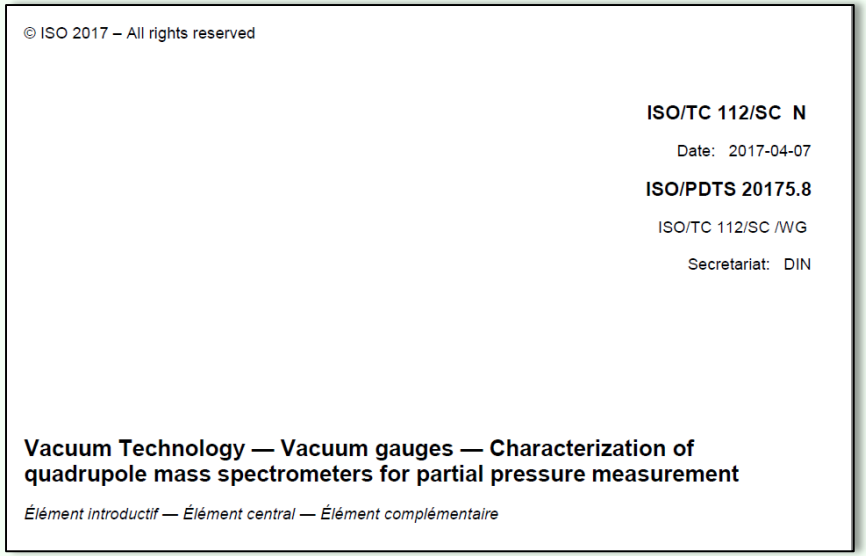
ISO 14291

First edition
2012-##-##

Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers

Manomètres à vide — Définitions et spécifications des spectromètres de masse quadripolaires

Reference number
ISO 14291:2012(E)



ISO/TC 112/SC N

Date: 2017-04-07

ISO/PDTS 20175.8

ISO/TC 112/SC /WG

Secretariat: DIN

Vacuum Technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement

Élément introductif — Élément central — Élément complémentaire

Quadrupole mass spectrometers

ISO 14291

Mass resolving power: $R = \frac{m}{\Delta m}$

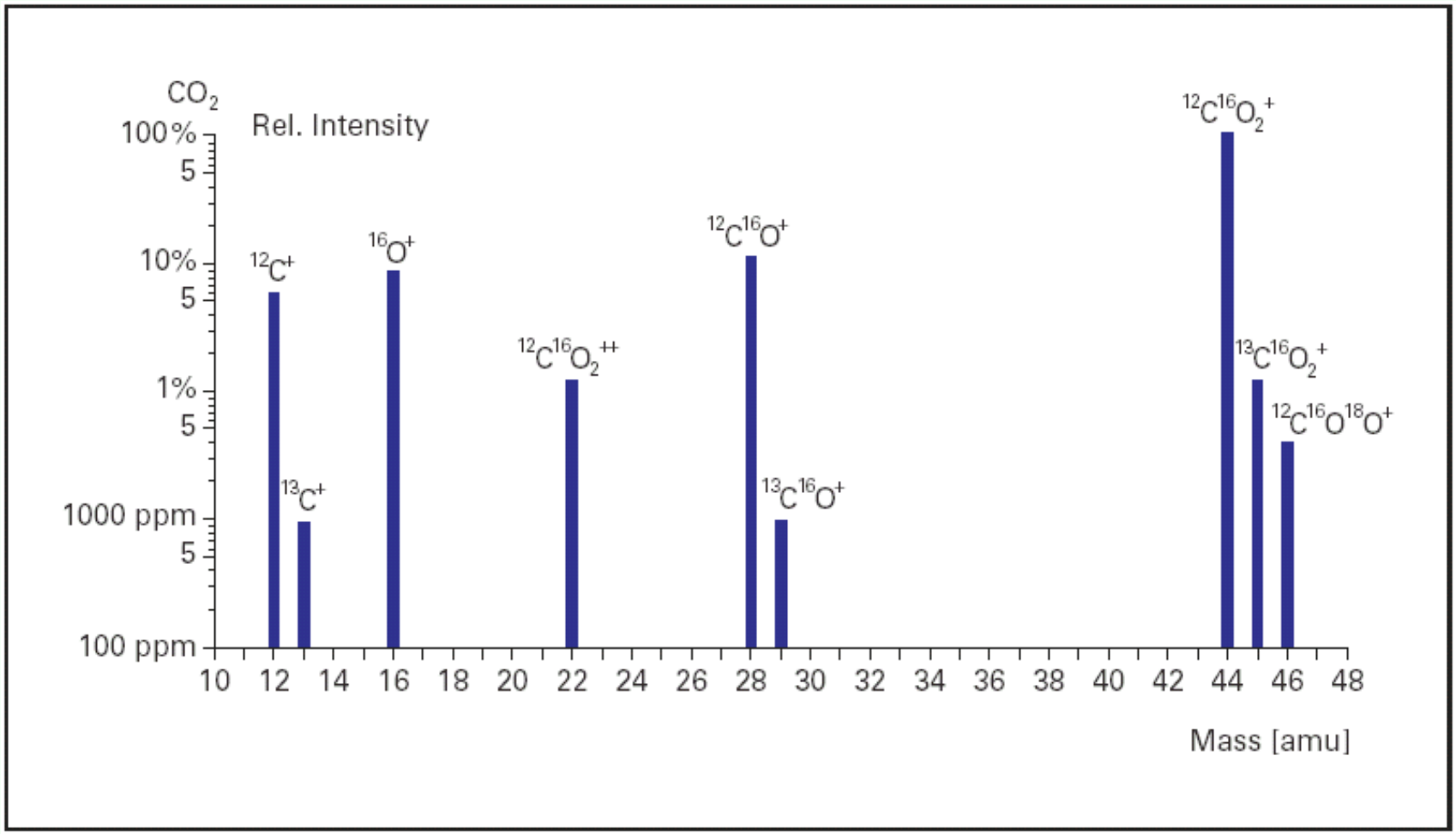
Mass number stability: $\frac{\Delta m_{\text{peak}}}{\Delta t}$

Minimum detectable partial pressure: $MDPP = \frac{3\sigma_I}{S}$

Minumum detectable concentration: $MDC_x = \frac{MDPP_x}{p_{\text{carrier}}}$

Fragmentation factor: $f_i = \frac{I_i}{I_0}$

Quadrupole mass spectrometers



Vacuum gauges II

We have discussed

Ionization gauges with emissive cathode

Effects in emissive cathode gauges

Low pressure limits of ion gauges

Problems in applications

Work principles of quadrupole mass spectrometers

Limits of QMS

Calibration and standards

Vacuum gauges II

Questions?