EVOLUTION OF PUMPING SYSTEMS IN SUPPORT OF MASS SPECTROMETERS

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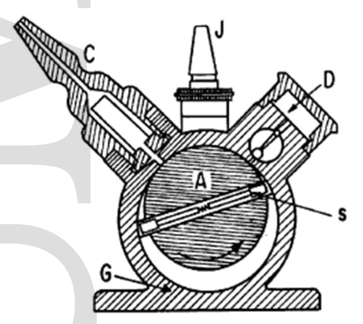
ROUGH VACUUM



*A Gaede Oil Sealed Vacuum Pump (1907)

*B Welch Fore Pump (Patented - 1939 Sold - 1950s - 1960s)

*C Rotary Pump with Oil Trap (1980 - present)

*D Scroll Fore Vacuum Pump (present)

The very nature of mass spectrometry presupposes that conditions to limit both background gas and chemical contamination can be achieved. Hence, two physical concepts must be addressed within the confines of the instrument: gas load and unwanted species.

The history of how mass spectrometrists have approached these concepts is largely the history of vacuum technology.

In this poster, we explore some of the ways chemists and physicists - practitioners of the art and science of mass spectrometry - have addressed the challenges posed by the need to conduct experiments at sub-atmospheric pressures.

THE CHALLENGES

Ensuring the integrity of ion analysis process - i.e., that the identities of the detected ions match the identities of the reactant or product ions - requires minimizing collisions with gas in the analyzer. This means that *pressure* in the analyzer must be *low*; or alternatively, that *vacuum* must be *high*. As instruments have developed, two major approaches have evolved that are related to gas load, in reality these approaches form a continuum of high vacuum conditions.

Static Pumping Systems

typically single chamber, no differential pumping

- Analyzer evacuated to the required level and operated without intentional introduction of additional gas load.
- Sample to be studied is already mounted inside vacuum system.
- This approach was used in all early instruments including those of Thomson and Aston.
- Most instruments from the 1930s -1960s operated in this gas load regime.
- Some modern systems, e.g. MALDI and SIMS, operate in quasi-single-chamber mode.
  - A valved sample entry mechanism permits the analyzer to remain under vacuum while the sample stage is evacuated.
  - When the pressure of the sample stage is compatible with that of the analyzer, the valve is opened, the sample is positioned, the laser or ion beam is turned on, and emitted or sputtered ions are guided through lenses to the analyzer.

Dynamic Pumping Systems

typically dual chamber and differential pumping

The source (sample inlet and ionization apparatus) is housed in a chamber separate from the analyzer.

The two chambers have different pumping systems capable of achieving different levels of vacuum:

- They are connected by flow restrictors and/or valves.
- Vacuum is moderate for the source, high for the analyzer.
- The analyzer is maintained at high vacuum, while the pressure in the source varies with ionization method and sample load.

Many modern techniques require pumping systems capable of handling high gas loads and in fact were made practical only with the development of high speed pumping systems. These include

- Chemical Ionization and APCI
- Liquid Chromatography/Mass Spectrometry
- Electrospray Ionization
- Ion Mobility Mass Spectrometry

VACUUM CONDITIONS & GAS LOADS

High vacuum is attained by using a combination of pumps:

- Fore pumps bring a system from atmospheric pressure to ~10^{-3} torr and continue to operate on gas output of high vacuum pumps
- High vacuum pumps begin operating at ~10^{-3} torr and continue to reduce pressure until ultimate vacuum is attained

Mass analyzers operate at high vacuum (low pressure) (10^{-5} - 10^{-8} torr)

Sources operate between atmosphere and high vacuum:

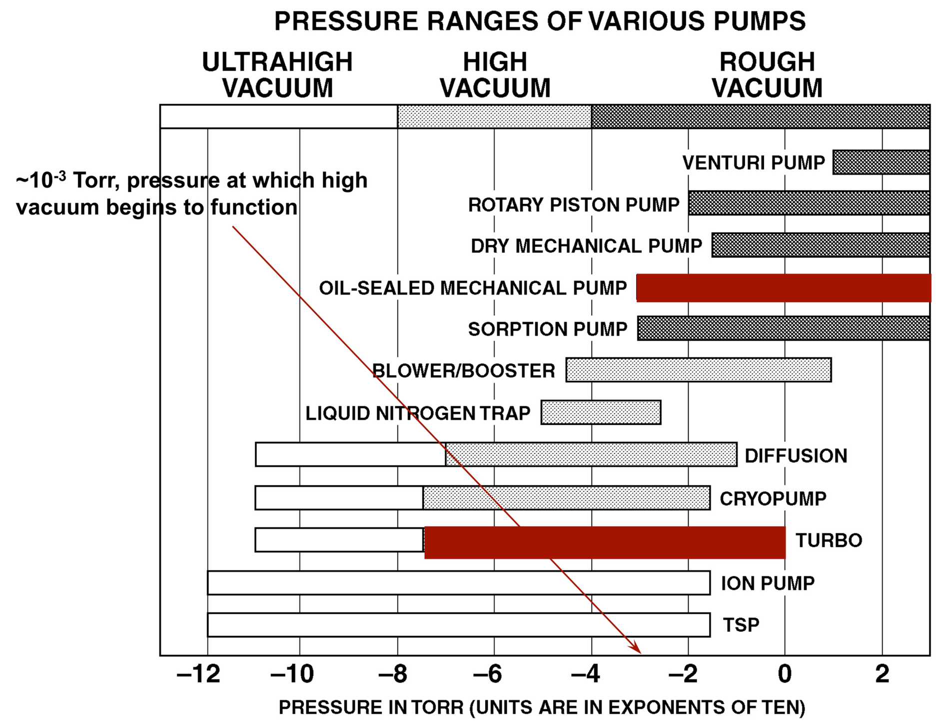
- APCI, ESI – atmospheric pressure
- CI – intermediate vacuum, 1- 0.1 torr
- EI – low vacuum, <10^{-3} torr
- MALDI and SIMS – high vacuum, <10^{-6} torr

"The rate at which molecules enter into the chamber's gas phase from all these sources is called the chamber's gas load. In a tight vacuum system, a major contributor to gas-phase molecules is wall desorption."

\- Kurt J. Lesker Company

Typical Gas Loads

Origin of Gas	Flow Rate (typical units)	Flow Rate (molecules/sec)
Capillary Column GC	1 atm.cc/sec	4.1×10^{17}
Packed Column GC	10 atm.cc/sec	4.1×10^{18}
Microbore LC	2 µL/min (liquid)	1.1×10^{18}
ESI Capillary Interface	90 atm.cc/min	3.7×10^{19}
SIMS	nominal	nominal
MALDI	nominal	nominal



The Question of Units

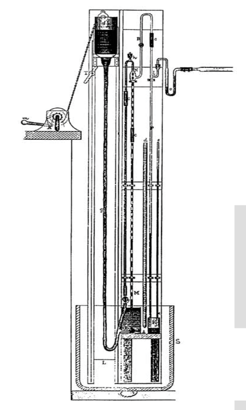
There are several systems of units in common usage for description of pressue and vacuum. Torr is the unit based on classical barometric pressures (mm Hg) while Pascal (Pa) is the SI unit and mbar is related to an alternative statement of atmospheric pressures.

Torr	Pa	mBar	Atm
1	133.3	1.333	1.316E-03
7.50E-03	1	0.01	1.02E-05
0.7501	100	1	1.02E-03
760	1.01E+05	1.01E+03	1

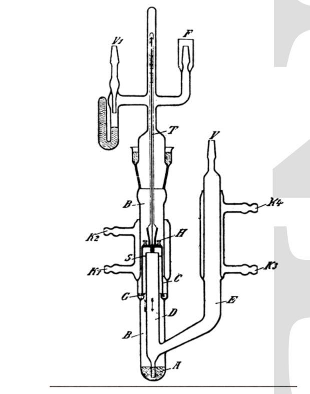
Other Devices that Assist in Maintaining Vacuum Conditions

- Chemical and Physical Scavengers
- Cold Traps, Cryosystems
- Flow Restrictors
- Differential Pumping
- Valves

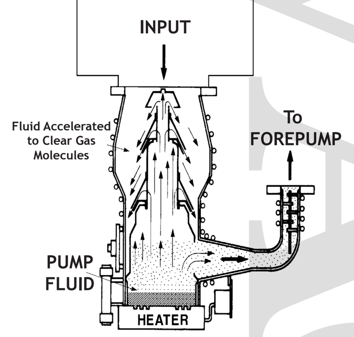
FINE VACUUM



*1 Crookes High Vacuum Pumping System (1870)



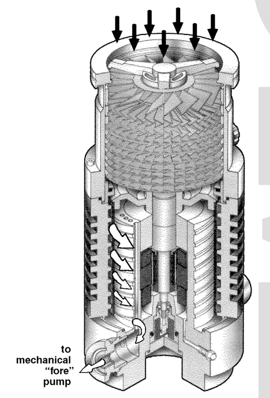
*2 First Mercury Diffusion Pump Wolfgang Gaede (1913)
Bunsen Burner Heat Source



*3 Multi-stage self-fractionating Mercury Diffusion Pump (Louis Malter - 1937)

*4 **Oil-based Diffusion Pumps**

- Silicone diffusion pump fluid Dow Corning – 1956
- *Fomblin* (perfluoropolyether vacuum fluids) Montedison – 1967
- DC 704 and DC 705 – tetramethyl, tetraphenyl trisiloxane pump fluids (Dow) – c.1970



*5 Turbo-molecular High Vacuum Pump (1980 - present)

Thomson's Parabola
*A1

Dempster's 180° Magnet
*A2

Sector Magnet, Double Focusing
*B3 - B4 - C4

Bendix Time-of-Flight
*B3

Quadrupole Mass Filter
Other TOF
*B4 - C4 - C5

Reflector TOF, Ion Trap
Tandem Analyzers: QqQ, QqTOF
*C5 - D5

Static Pumping Systems Predominate EXCEPT MALDI and SIMS

Dynamic Pumping Systems Predominate

1900 — 1910 — 1920 — 1930 — 1940 — 1950 — 1960 — 1970 — 1980 — 1990 — 2000

Discharge Ionization

Electron Ionization

GC/MS, SIMS

Chemical Ionization

LC/MS

FAB
Thermospray

ESI, MALDI

IMS

* *Starred letter-number combinations refer to fore-high vacuum configurations associated with the noted instrument developments.*