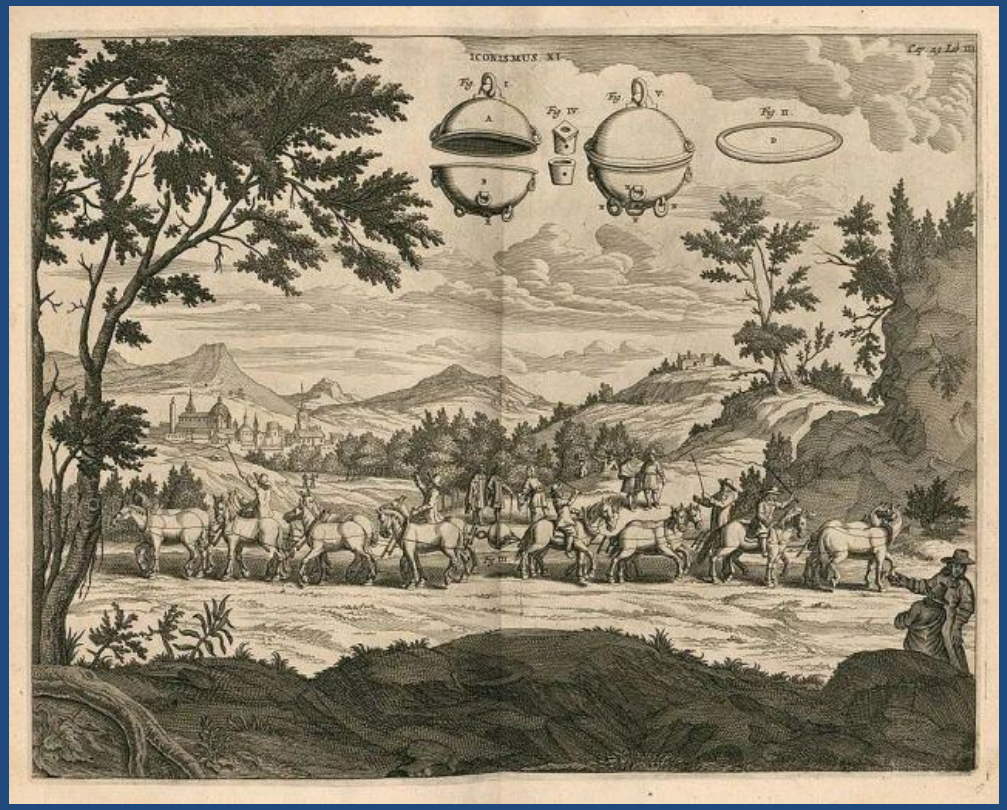
真空技术与四极杆质谱



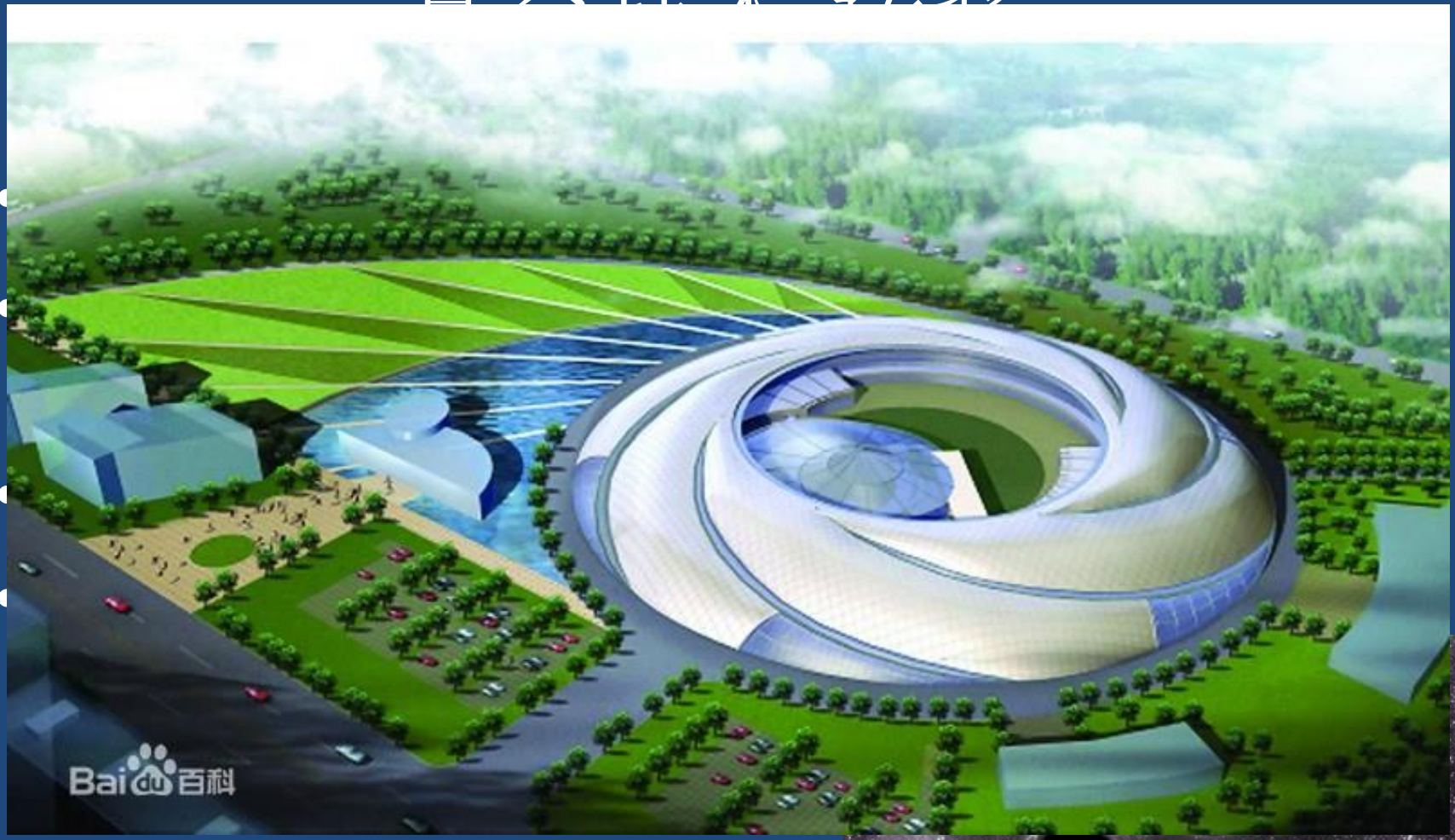
俞熹 2022/6

真空技术-历史与应用

- 古希腊:Vacuum 来自拉丁语 Vascuus
- 亚里士多德 认为 inconceivable
- 1657 Magdeburg Hemispheres Experiment
- 1855 水银置换泵(10Pa)海因里希·盖斯勒
- 1936 ~1937 气镇泵、油扩散泵
  - 冷阴极电离真空计
- 1950’ UHV (离子泵)
- 2000+ XHV (材料表面技术)

克鲁克斯辐射计

真空技术 进步



- 上海光源(432m同步辐射

黑洞合并(模拟图)

真空单位和真空级别

- 1atm=1bar=1000mbar=760Torr =10^5Pa
- 低真空LV　　10^5Pa → 10^2Pa
- 中真空MV　　10^2Pa → 10^{-1}Pa
- 高真空HV　　10^{-1}Pa → 10^{-6}Pa
- UHV(Ultra High Vac.)　　10^{-6}Pa → 10^{-10}Pa
- XHV(eXtreme High Vac.)　$<10^{-10}$Pa

相关理论公式

- Maxwell Boltzman Distribution

$$\sqrt{8kT}$$

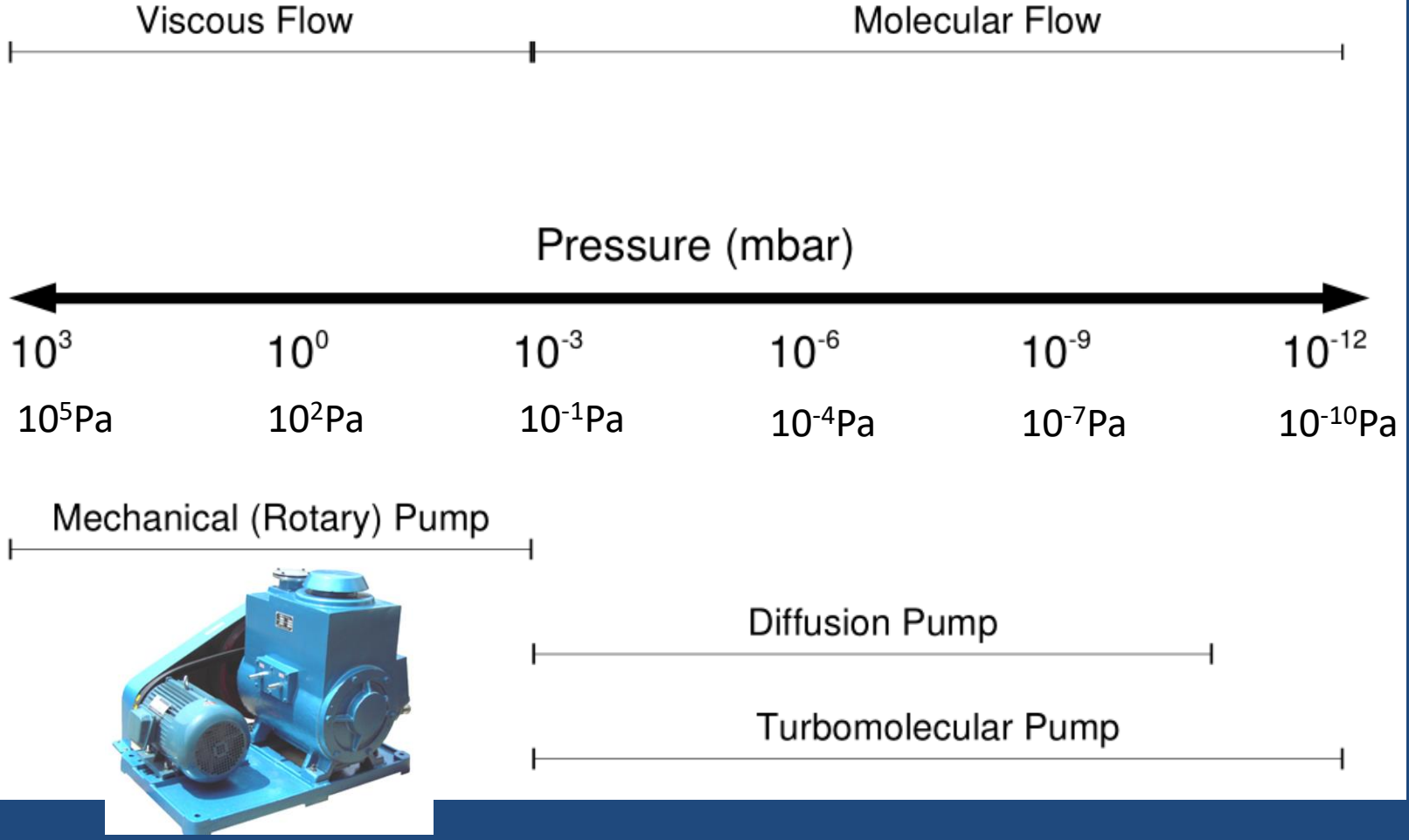
- Mean free

- Impingeme

- N_2为例:

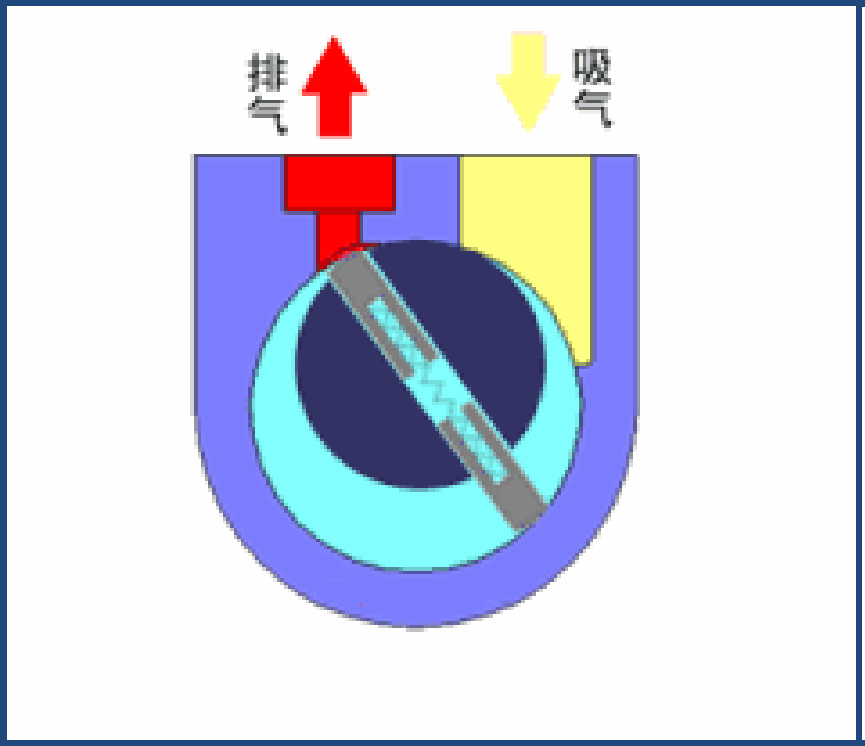
P (mbar)	n (litre^{-1})	λ	J (cm^{-2}s^{-1})
1000	2.5×10^{22}	66 nm	2.9×10^{23}
1	2.5×10^{19}	66 μm	2.9×10^{20}
1×10^{-3}	2.5×10^{16}	66 mm	2.9×10^{17}
1×10^{-6}	2.5×10^{13}	66 m	2.9×10^{14}
1×10^{-10}	2.5×10^{9}	660 km	2.9×10^{10}

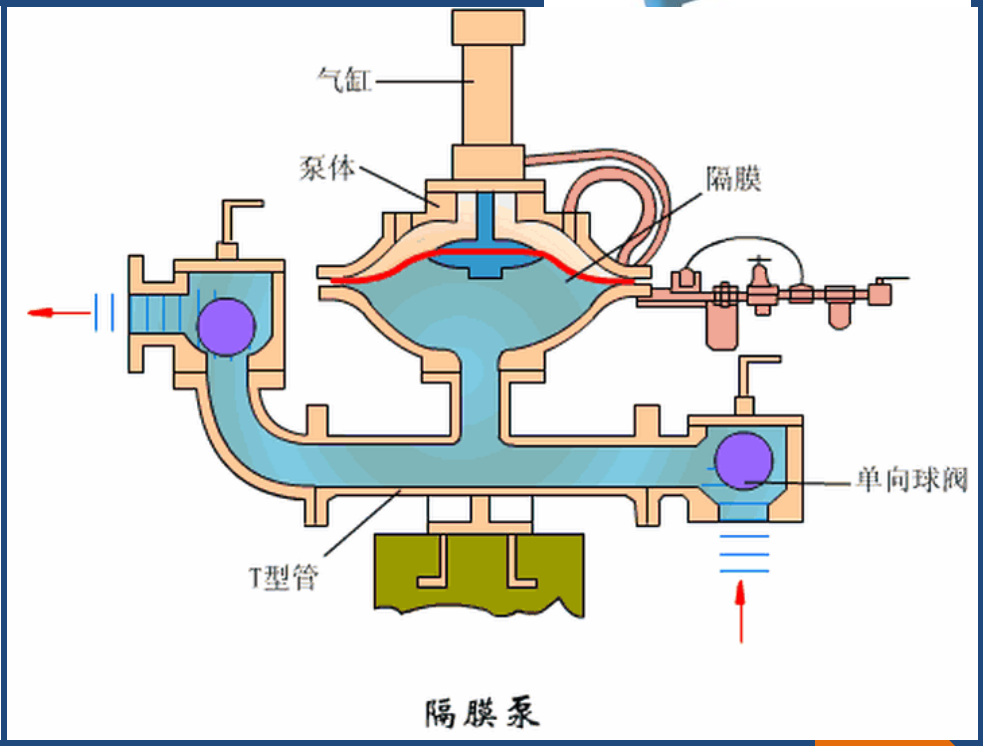
真空泵技术


Viscous Flow
Molecular Flow
Pressure (mbar)
10^{3}
10^{0}
10^{-3}
10^{-6}
10^{-9}
10^{-12}
10^{5}Pa
10^{2}Pa
10^{-1}Pa
10^{-4}Pa
10^{-7}Pa
10^{-10}Pa
Mechanical (Rotary) Pump
Diffusion Pump
Turbomolecular Pump

机械泵Rotary Vane Pump

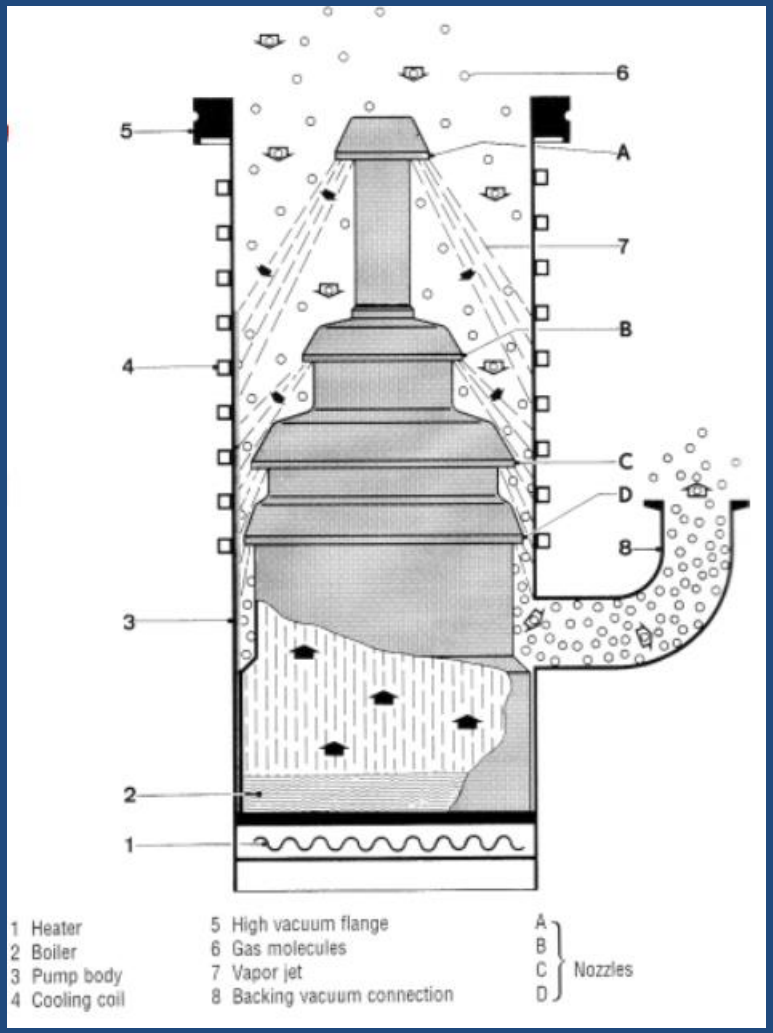
- 旋片式真空泵
- 干式真空泵(隔膜分离泵)





扩散泵Diffusion Pump

- 利用伞状喷嘴喷出的高速工质蒸汽射流将泵送的气体扩散到蒸汽射流中
- 工作蒸汽到达水冷泵壁，冷凝成液体，沿泵壁流回油底壳
- 极限真空7×10^{-5}Pa



涡轮分子泵 Turbomolecular Pump

- 1958 W. Becker发明
- 由泵体、带叶片的转子(即动叶轮)、静叶轮和驱动系统等组成
- 动叶轮外缘的线速度高达气体分子热运动的速度(150～400m/s)
- 性能:压缩比(排气:进气)
- 极限真空10^{-6}Pa

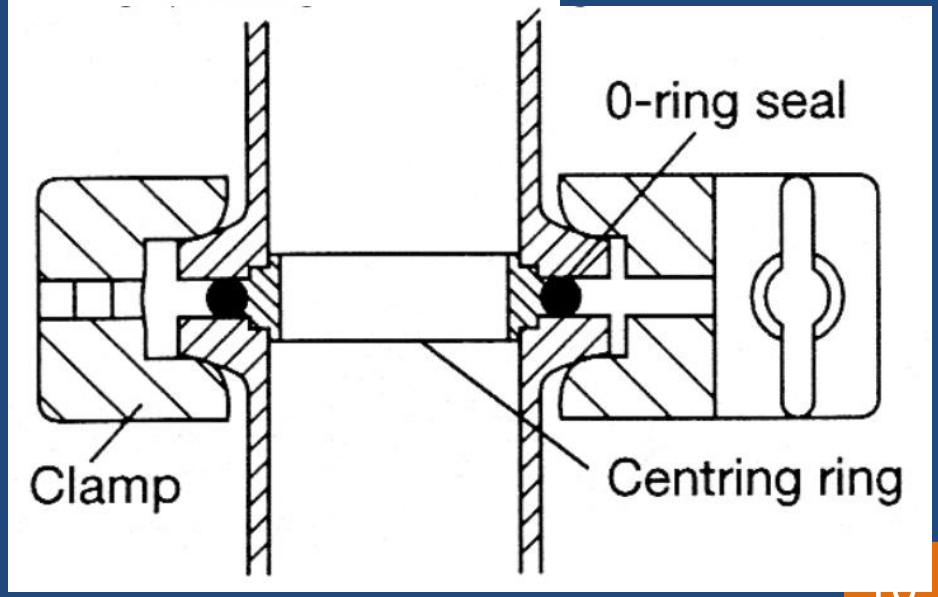
HV管道与连接件(KF)

- KF(Klein Flange)标准,适合 低→高真空

波纹管

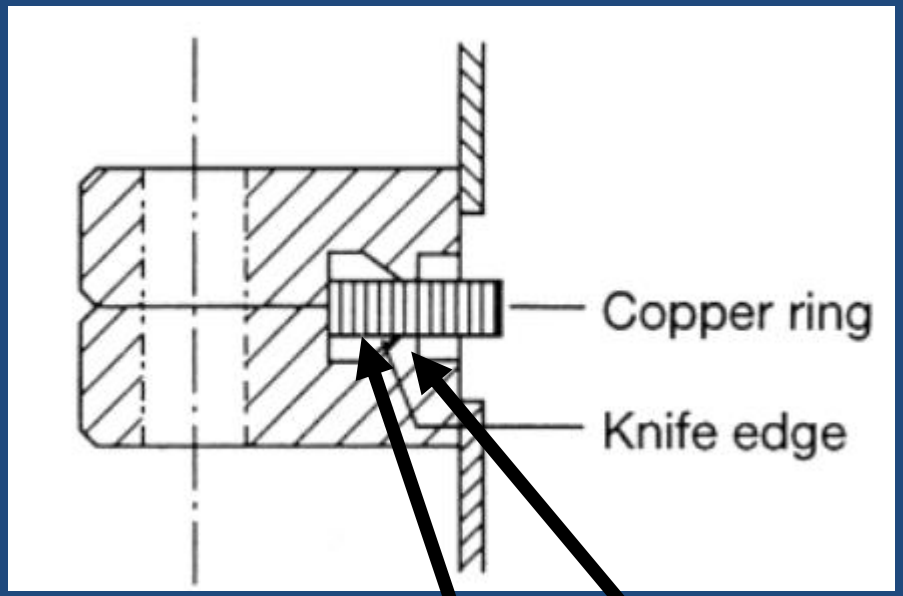
转接件

连接件

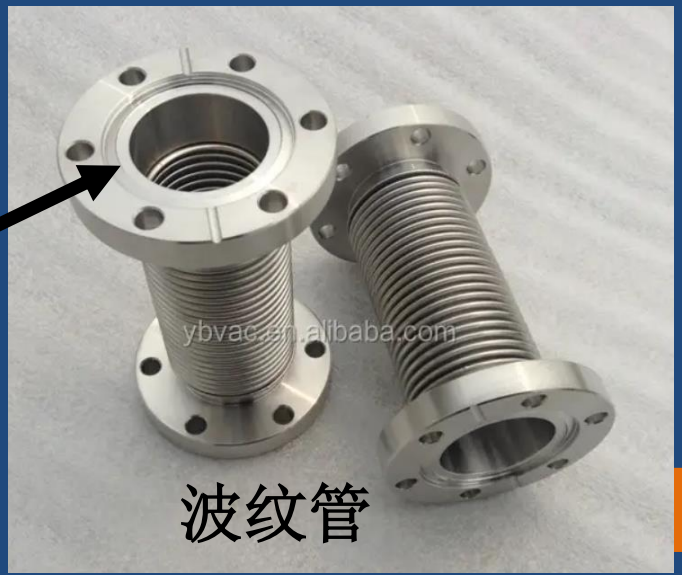


UHV管道与连接件(CF)

- CF(ConFlat)标准,适合UHV




转接件

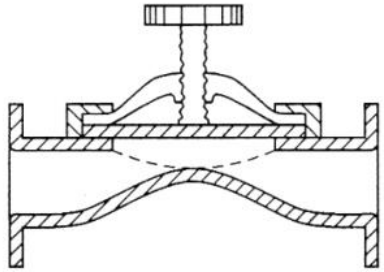


波纹管

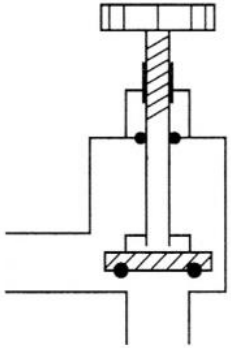
铜密封环

刀口

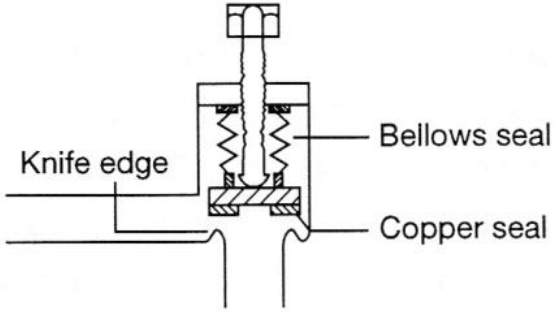
真空阀门Vacuum Valves



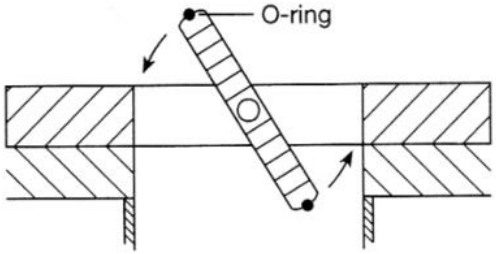
Diaphragm valve
(typically on backing lines)



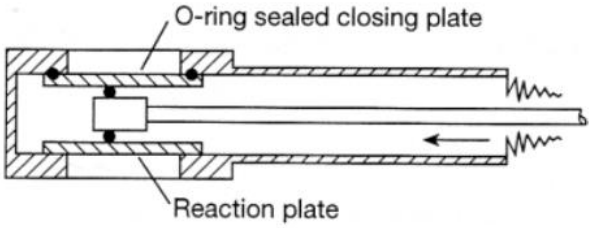
HV 90° valve



UHV 90° valve

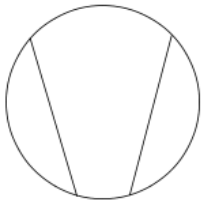


Butterfly valve
(high conductance)

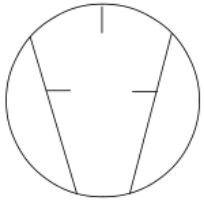


Gate valve
(high conductance + UHV)

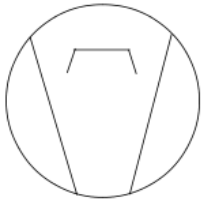
常用真空器件符号



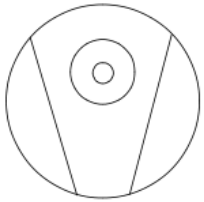
Vacuum Pump, General



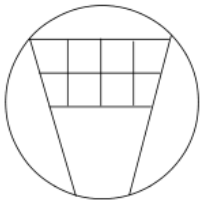
Rotary Vane Pump



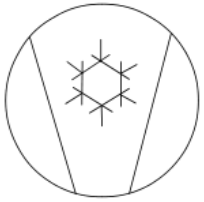
Diffusion Pump



Turbomolecular Pump

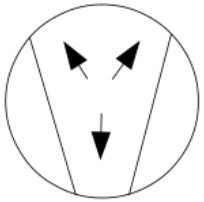


Adsorption Pump

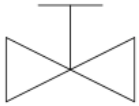


Cryopump

Sputter-Ion Pump

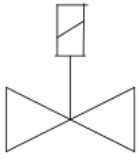


Getter Pump

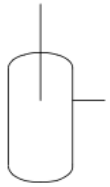


Manual Valve

Variable Leak Valve



Electromag. Valve

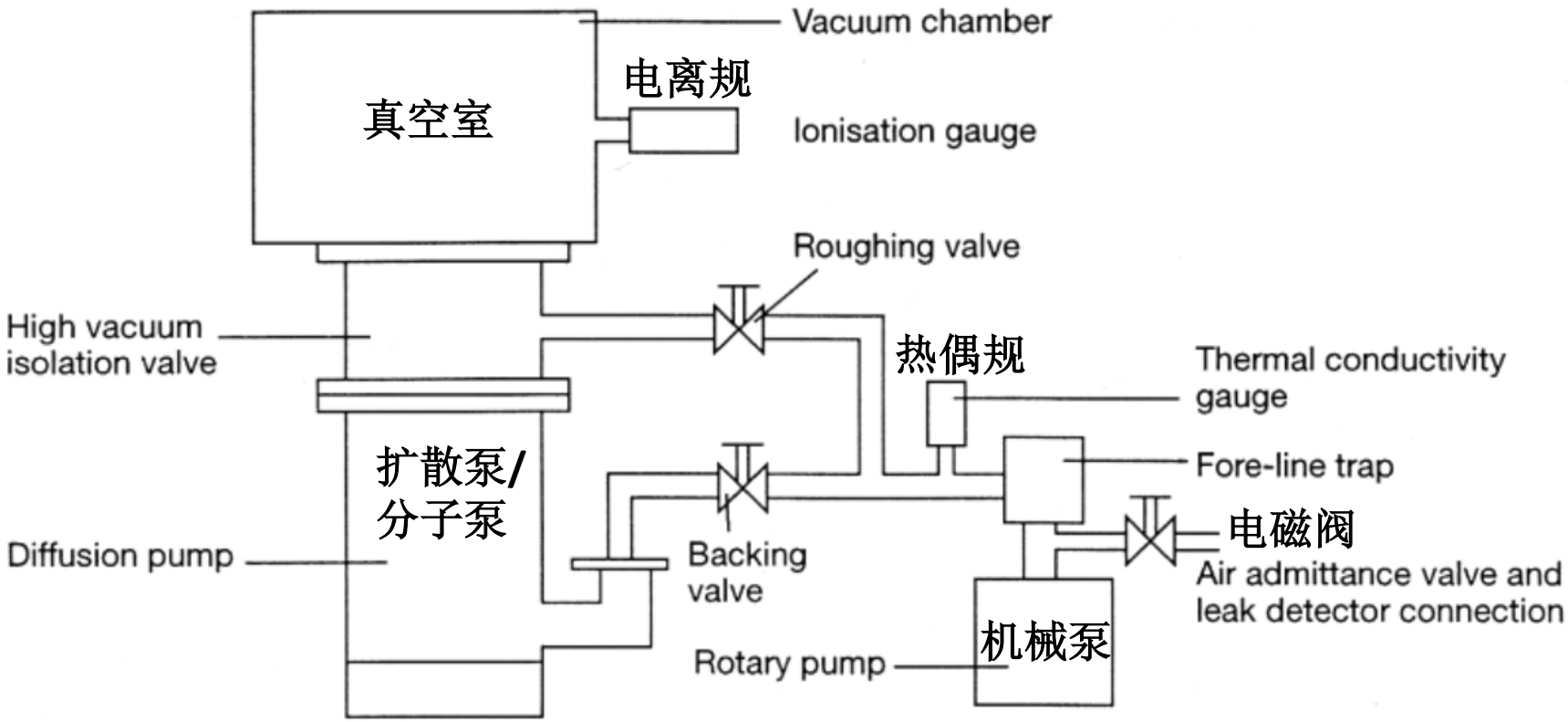


Cold Trap

Sorption Trap

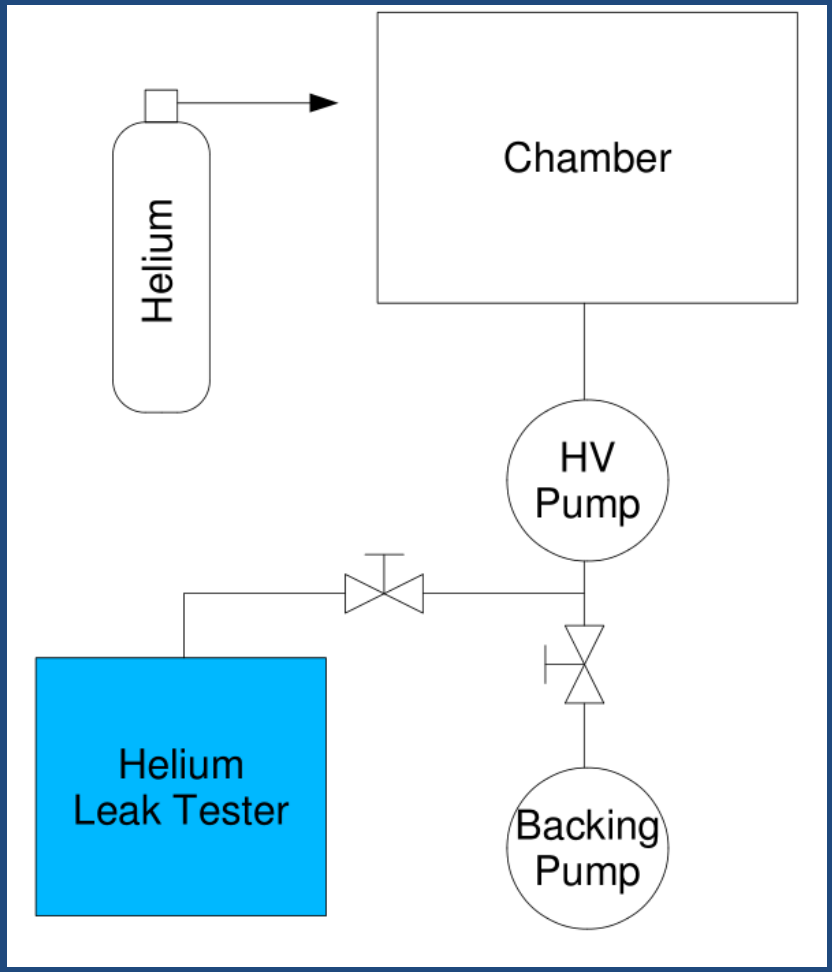
Vacuum Gauge

标准高真空系统


Vacuum chamber
真空室
电离规
Ionisation gauge
Roughing valve
High vacuum
isolation valve
热偶规
Thermal conductivity
gauge
扩散泵/
分子泵
Fore-line trap
Diffusion pump
Backing
valve
电磁阀
Air admittance valve and
leak detector connection
机械泵
Rotary pump

真空系统检漏

- 简单检漏:有疑问处喷洒丙酮看压力变化
- 通过氦气检漏
- 氦气质谱检测
- 响应时间~10s



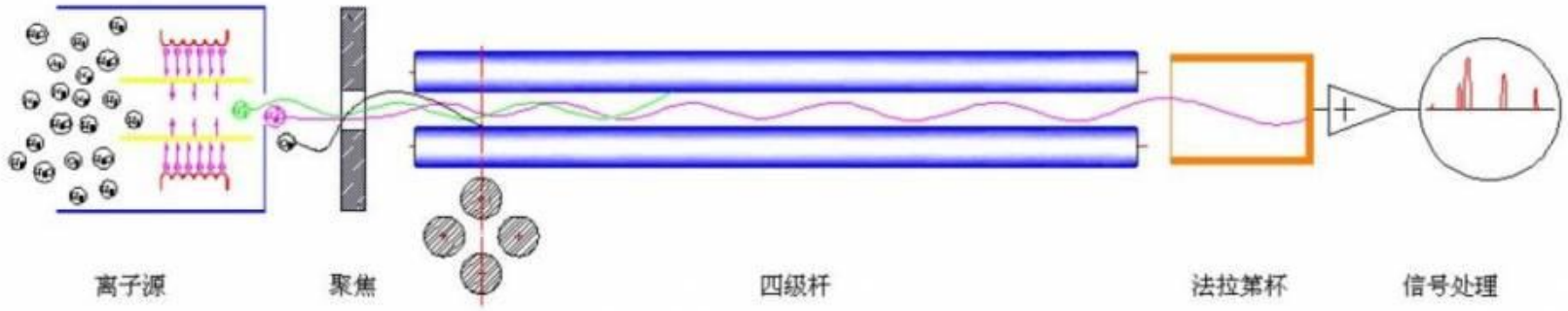
实验装置

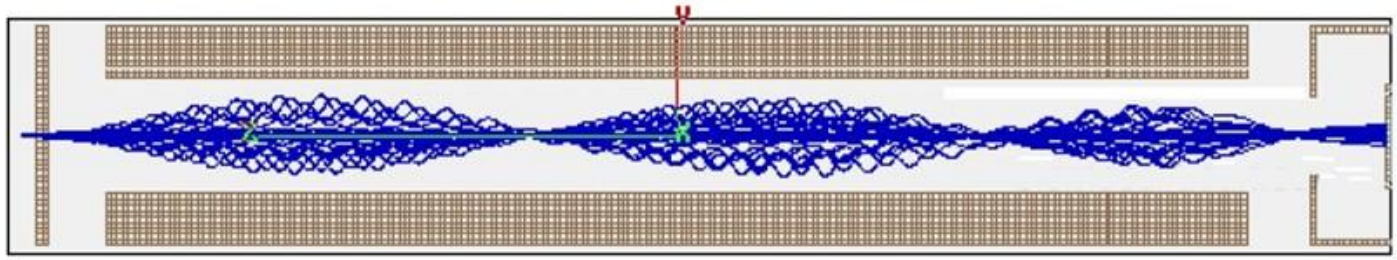

KYKY
高真空实验系统
上海实路真空技术工程有限公司
Shanghai Solution Vacuum Technology Co.,Ltd
WWW.SHSVAC.COM

操作指南

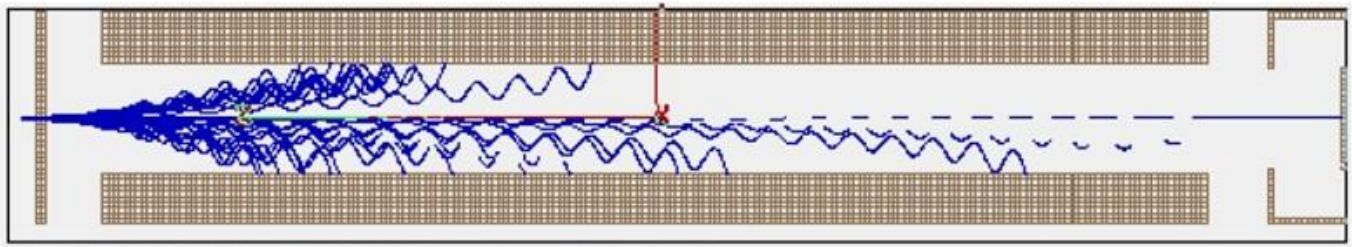

备用开关2
备用开关1
机械泵启停
总电源开关
急 停
KYKY
中科科仪
TCDP-II 分子泵驱动控制器
TCDP-II PUMP DRIVE-CONTROLLER

四极杆质谱的基本原理





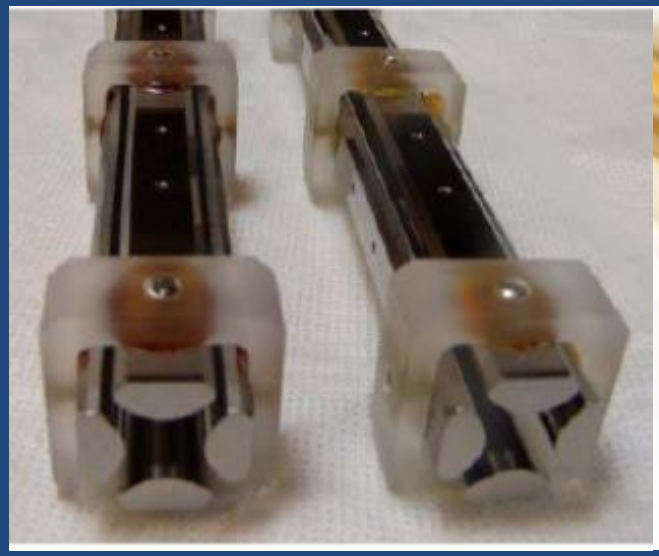
离子顺利的通过四极杆质量分析器



离子没有能够顺利的通过四极杆，撞死在四极杆的表面上

电极外形

- 双曲型
- 圆柱面



马绍(Mathieu)方程

- 当质量为 m，电荷为 e 的离子，运动方程为:

$$mx'' + (2e/r_0^2)(U + V_0 \cos \omega t)x = 0$$

$$my'' - (2e/r_0^2)(U + V_0 \cos \omega t)y = 0$$

- 令

$$\xi = \frac{\omega t}{2}, \quad a = \frac{8eU}{m\omega^2 r_0^2}, \quad q = \frac{4eV_0}{m\omega^2 r_0^2}$$

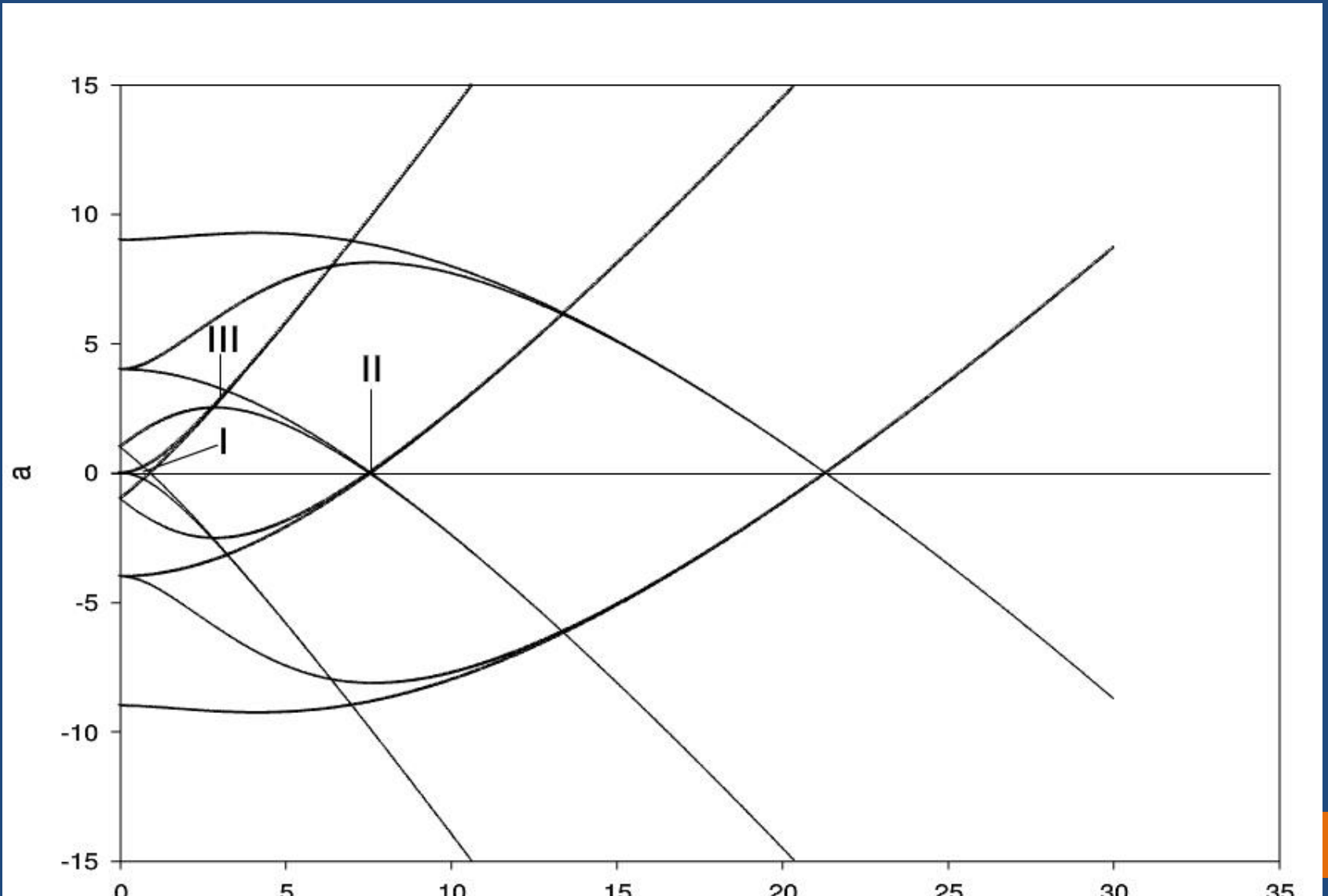
- 可得马绍方程基本形式:

$$\frac{\partial^2 x}{\partial \xi^2} + (a + 2q\cos 2\xi)x = 0$$

$$\frac{\partial^2 y}{\partial \xi^2} - (a + 2q\cos 2\xi)y = 0$$

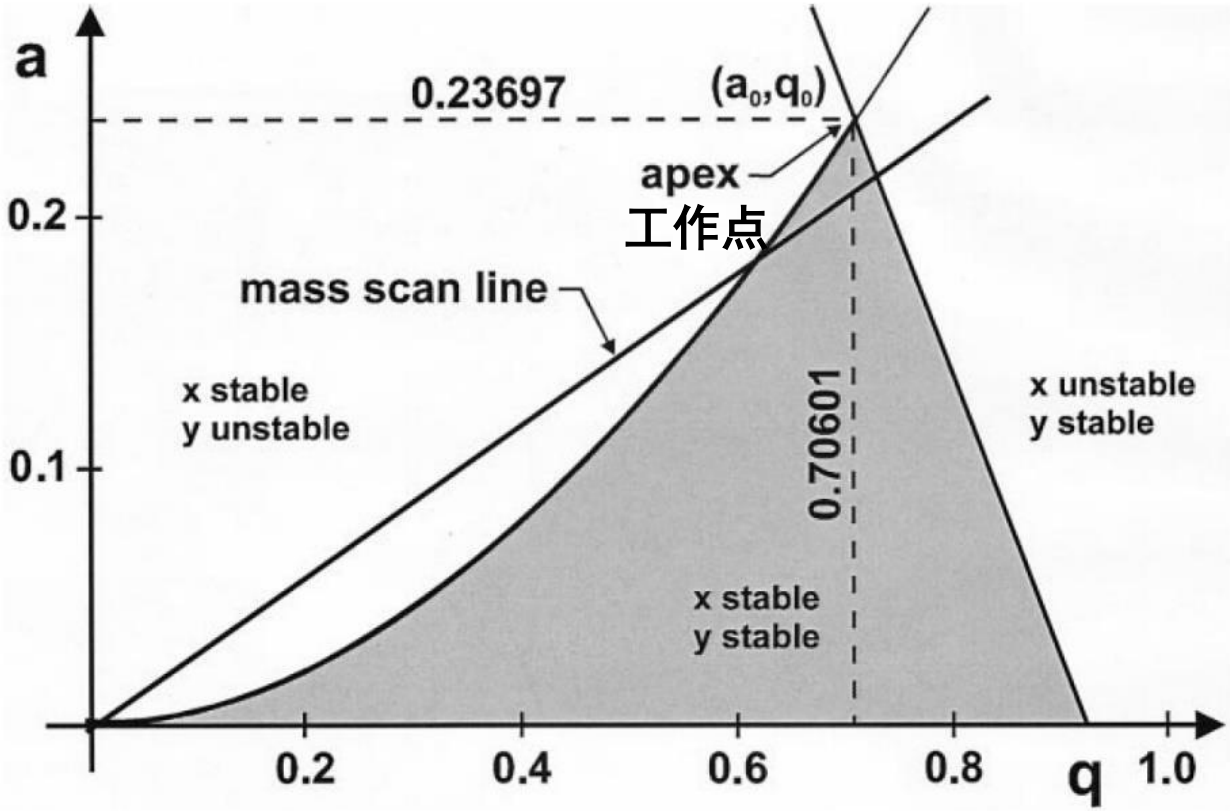
- 离子需在 x 和 y 方向都稳定才能通过

马绍方程稳定区



稳定区与扫描线

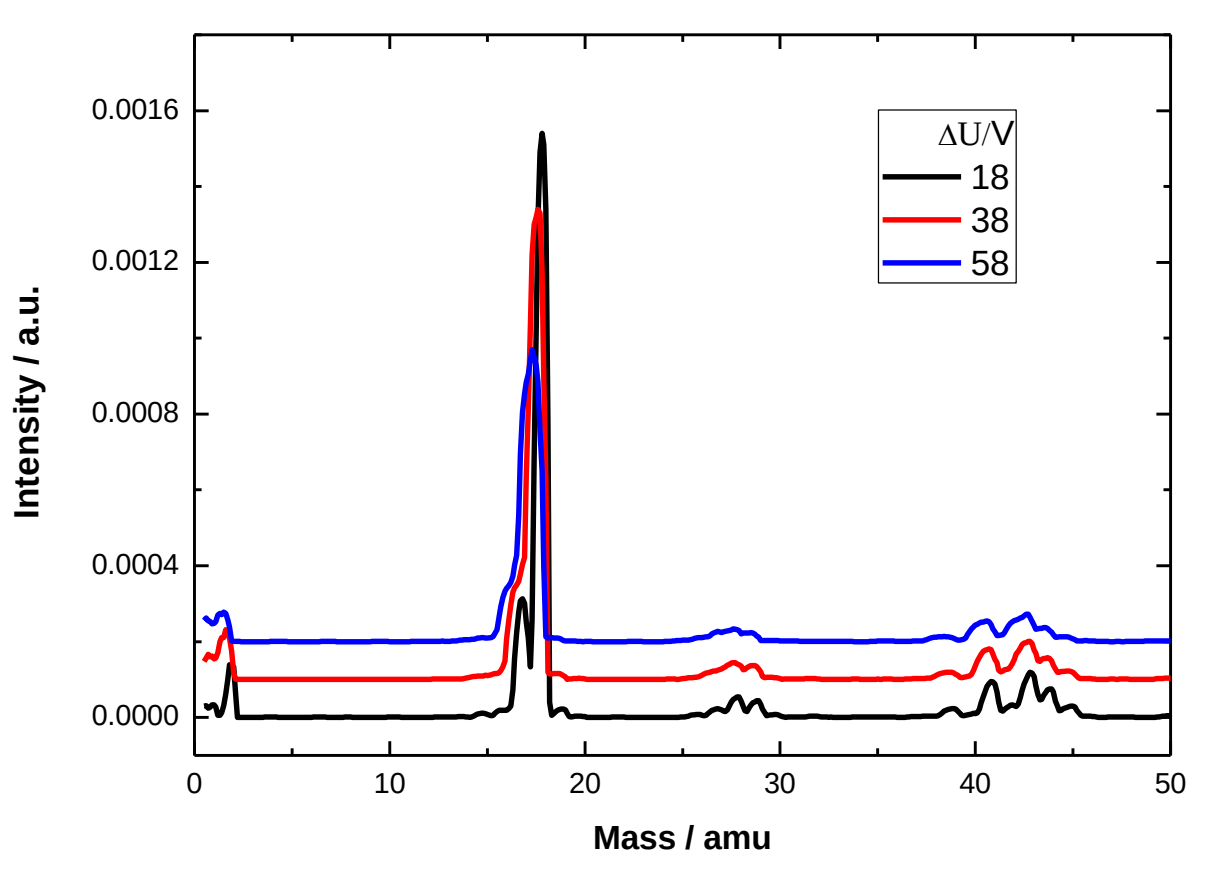
- 近原点放大后的第一稳定区



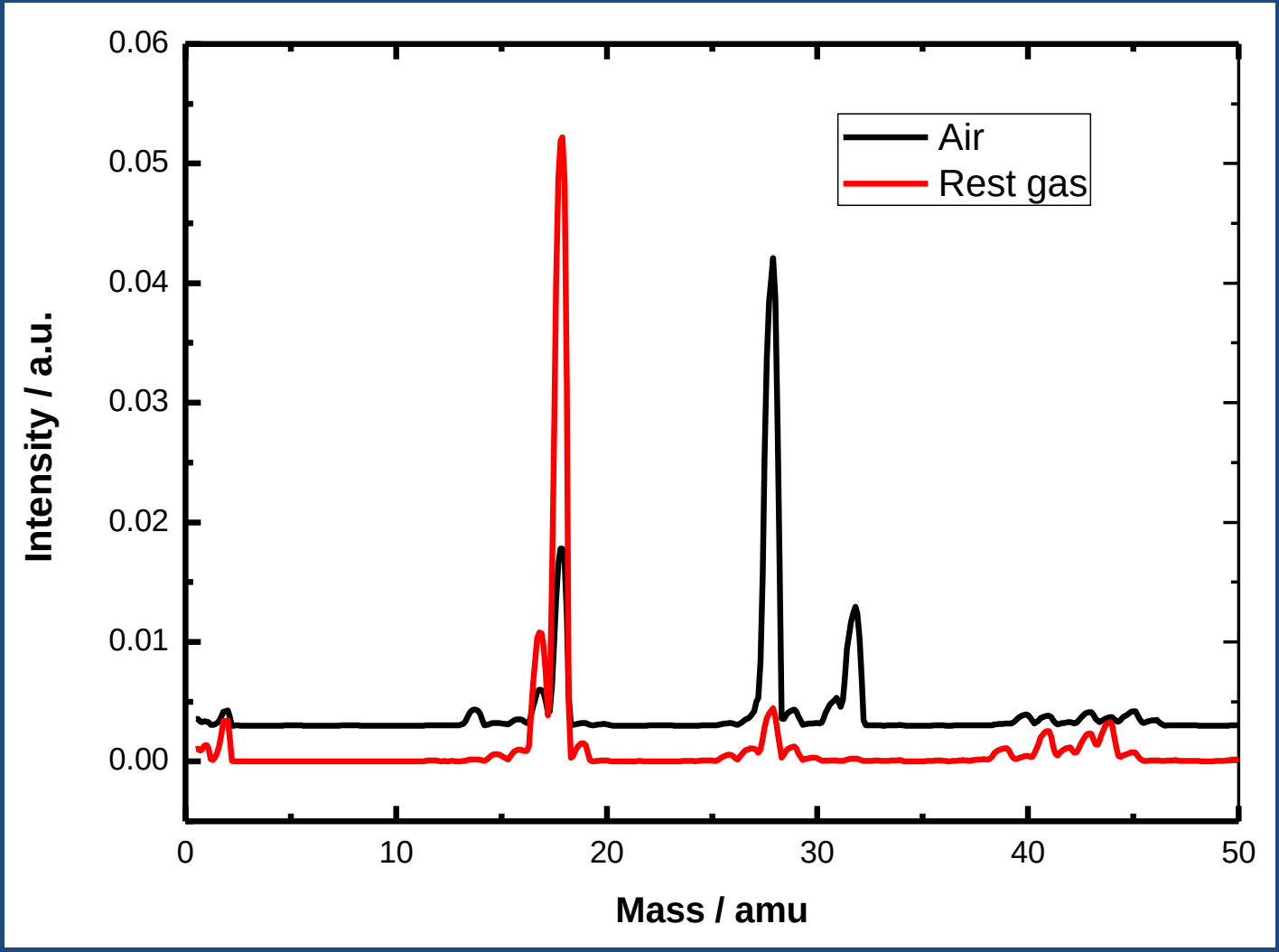
近原点放大后的第一稳定区

参数研究

- 改变四极杆上所加电场的交、直流分量的比例

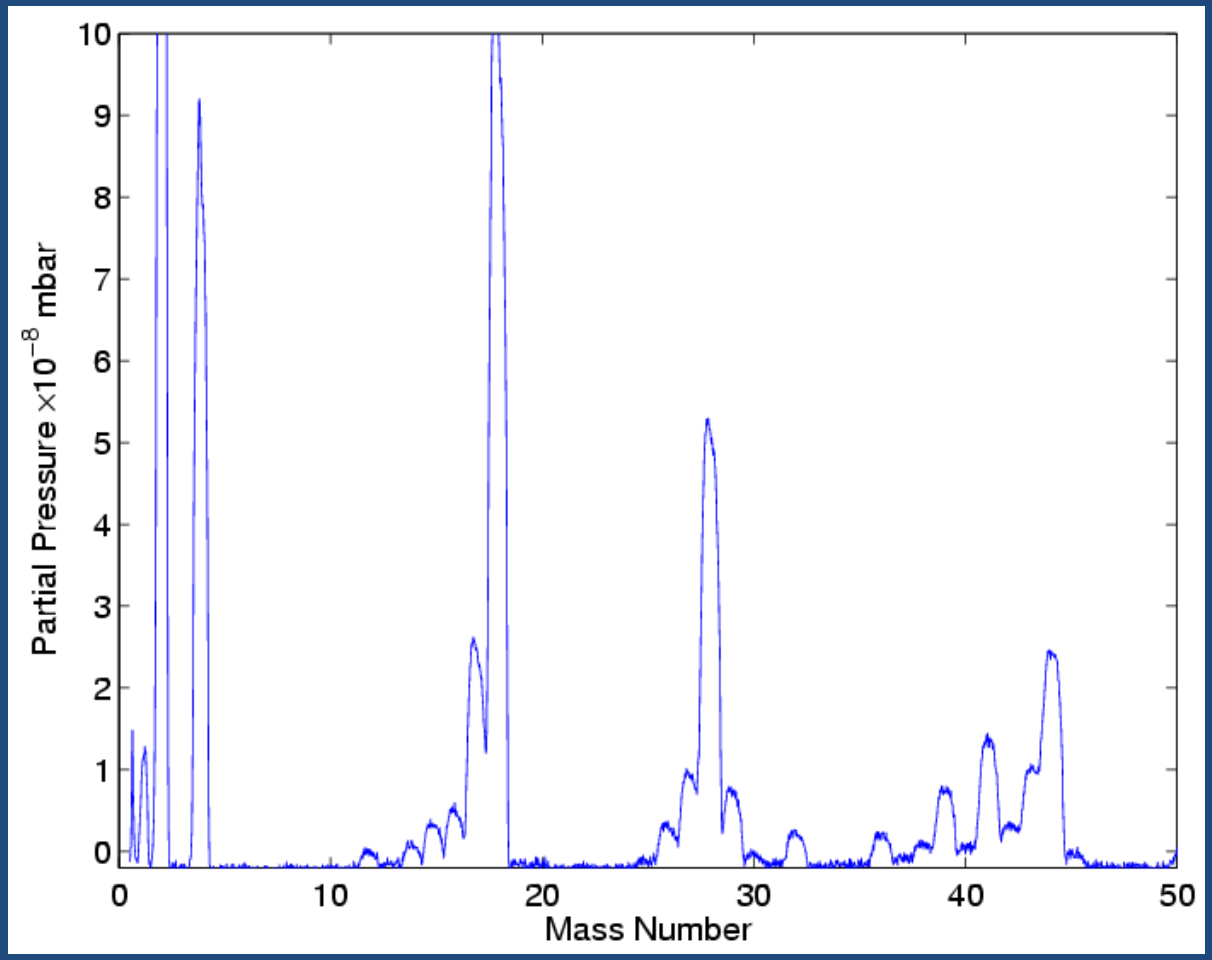


剩余气体质谱和空气质谱



如何测定未知样品

- 如何读谱?
- 如何定标?



实验室注意事项

- 检查冷却水
- 检查机械泵位置
- 先到初真空后再开启分子泵
- 通过系统菜单关闭分子泵
- 真空一般原则:先开后关,后开先关
- 离开前检查：仪器、水、电、门窗

请提问！

期盼早日复课！

参考资料

- 高真空技术基础
- 四极质谱气体组份分析仪 使用说明书
- 化学系徐国宾课件：四极杆质谱原理和技术
- “Basic Vacuum Technology”, A. Chambers, R.K. Fitch and B.S. Halliday.
- “A Users Guide to Vacuum Technology”, O’Hanlon.