

理化分析中心实验室 仪器与方法介绍

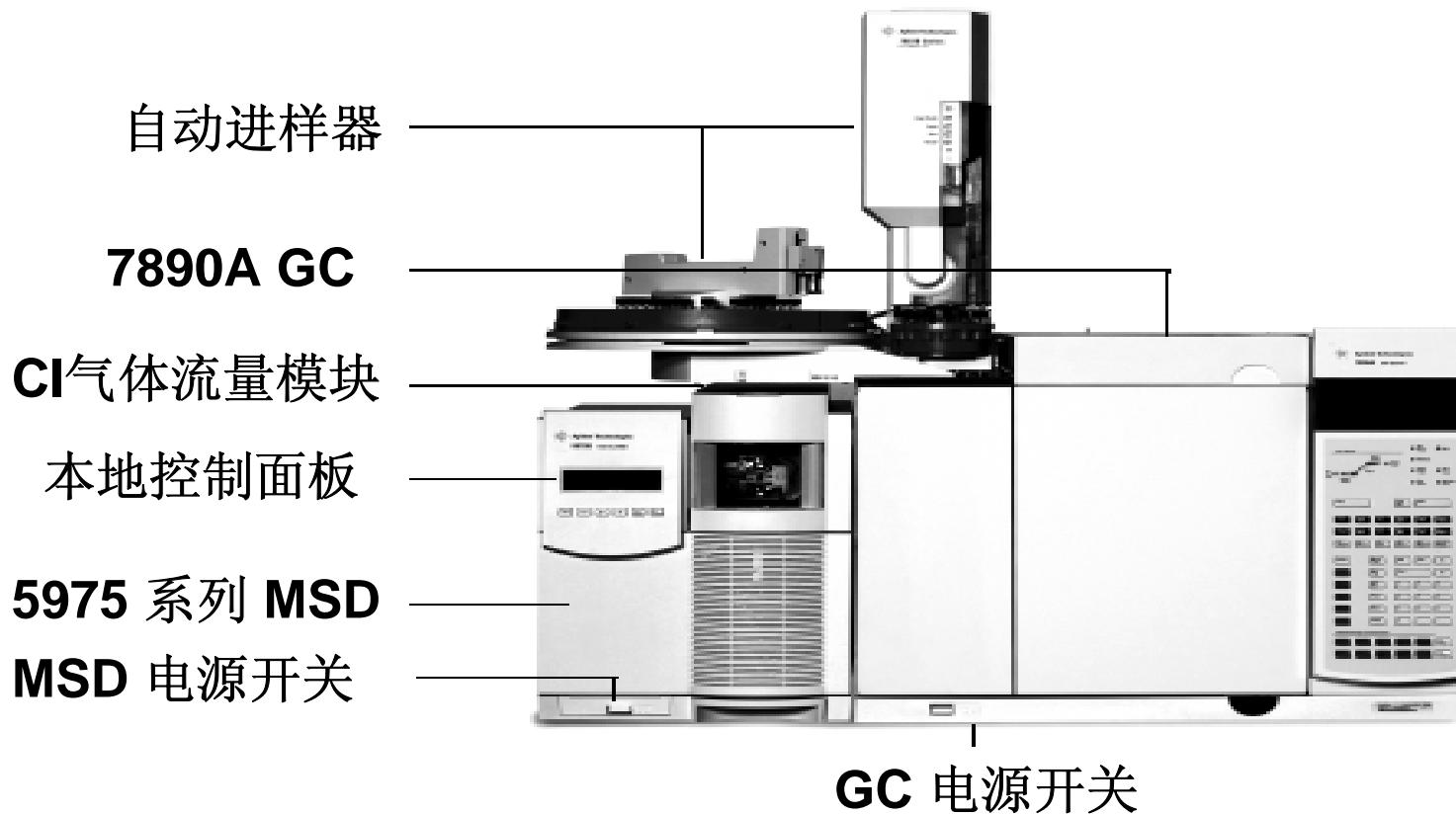
张 倩

2010.09

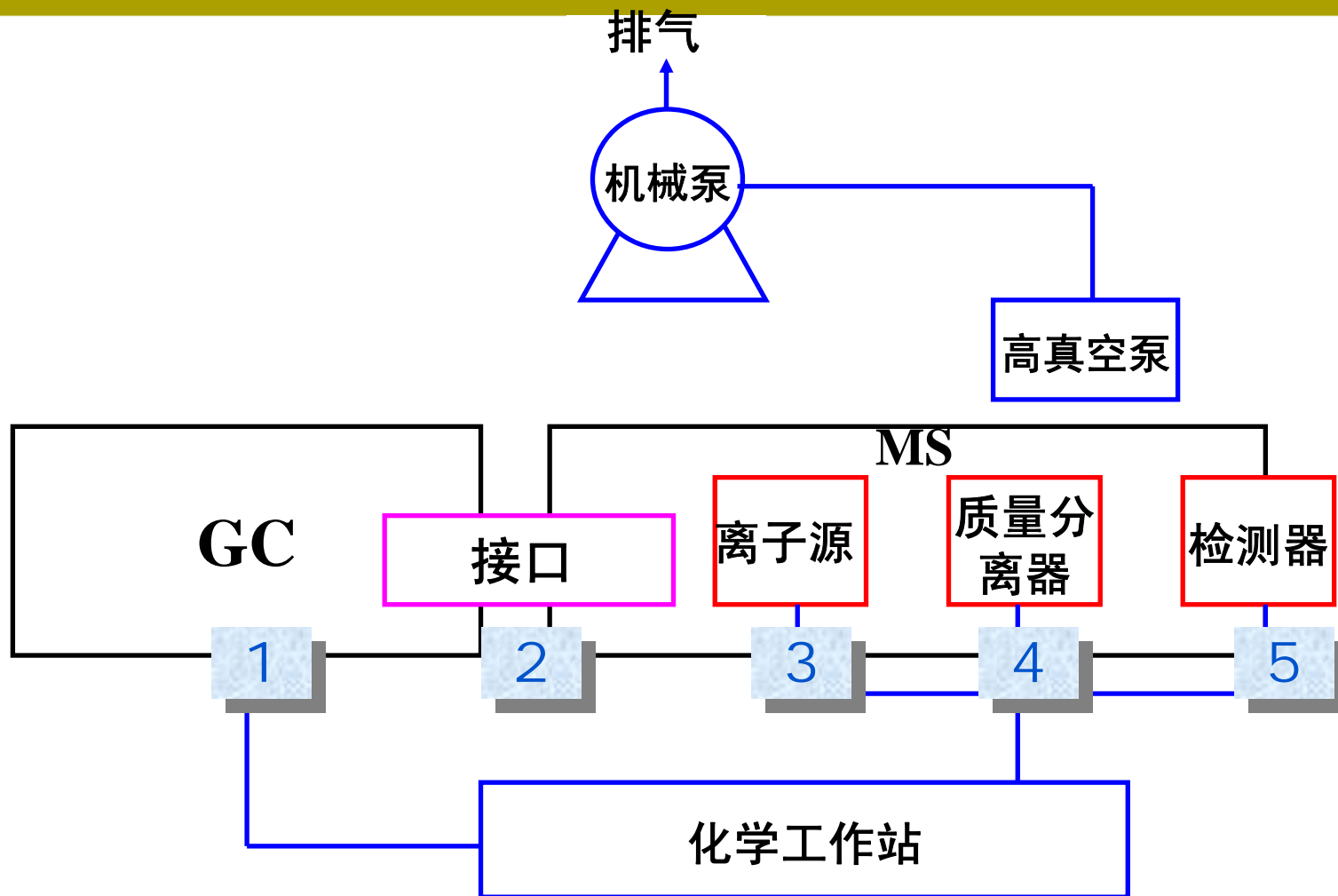
气相色谱与质谱联用仪（GC-MS）

- 一、仪器结构及工作原理
- 二、操作规程
- 三、数据处理

一、GC-MS仪器结构



GC-MS内部连接示意图



1. 气相色谱 (GC) 部分

➤ 载气系统

用于整个系统样品传送。

➤ 进样系统

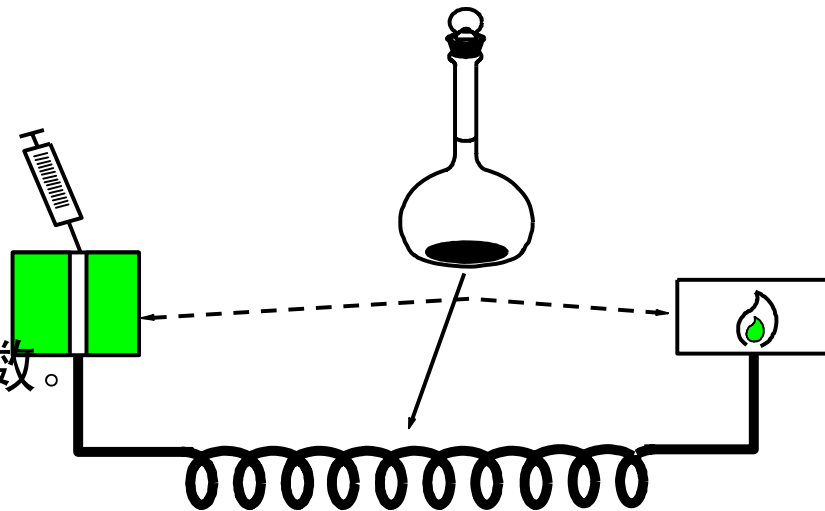
包括液态进样器和气化室，当样品为液体时，被注射到气化室，快速气化后由载体携带进入色谱柱。

➤ 色谱柱

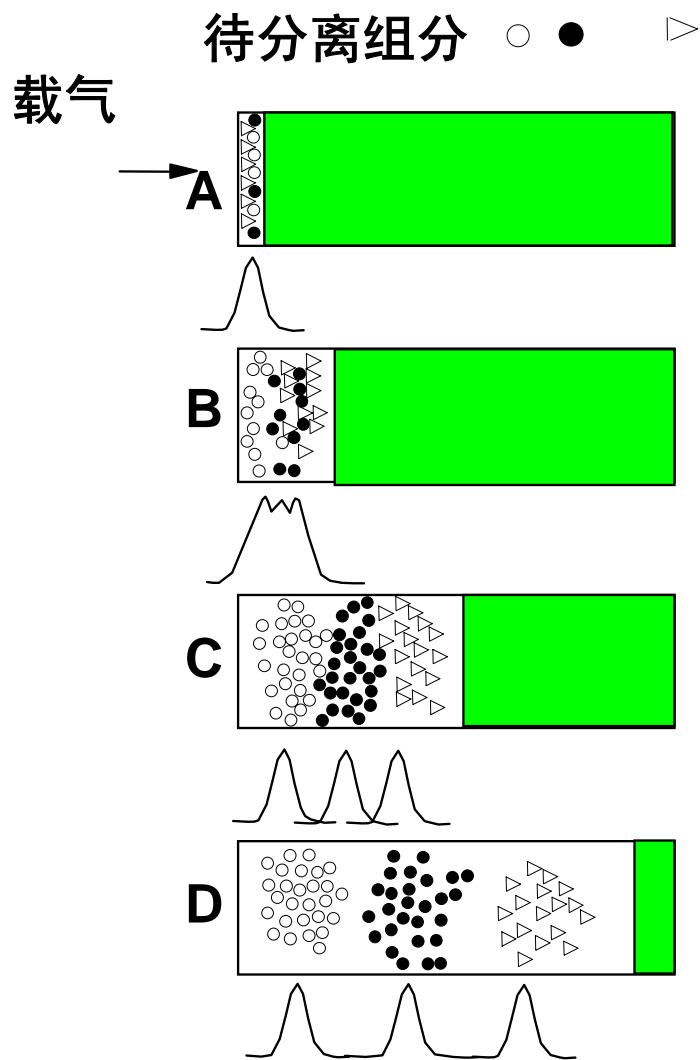
实现样品组分的分离。

➤ 温度控制系统

温度是试样气化和色谱分离的重要参数。



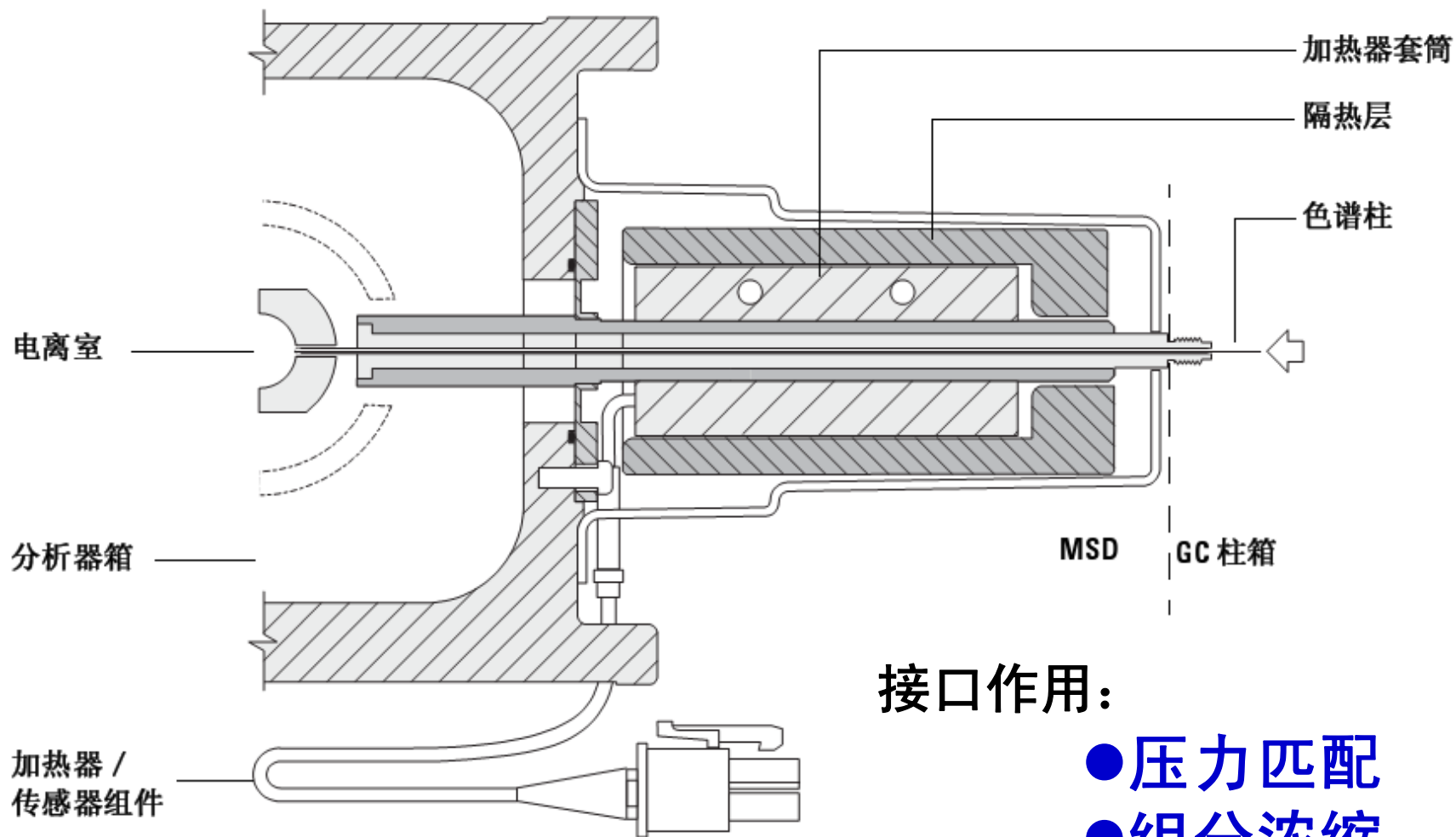
色谱分离过程



色谱系统组成:

- **固定相:** 填入玻璃管或不锈钢管内静止不动的固体或液体;
 - 色谱柱: 装有固定相的管子 (玻璃管或不锈钢管)
- **流动相:** 携带样品流过色谱柱的气体。

2. MSD接口



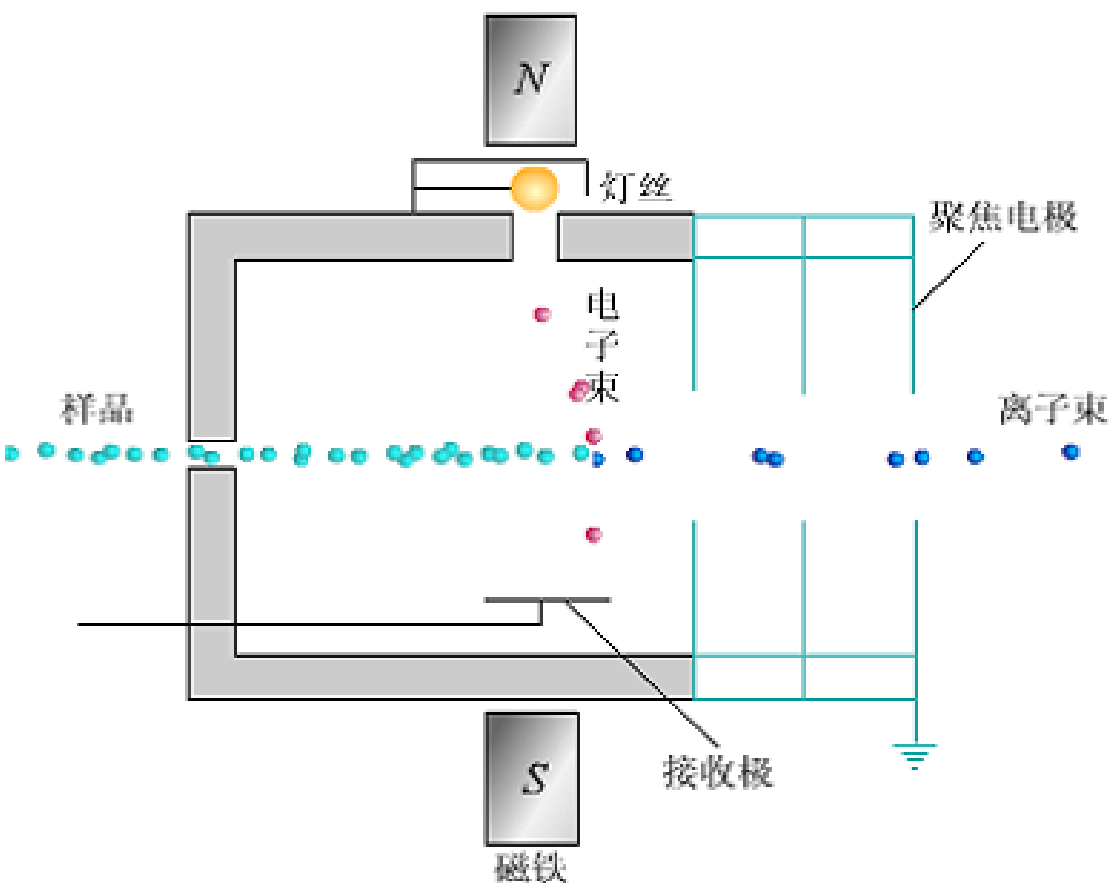
接口作用:

- 压力匹配
- 组分浓缩

3. 离子源

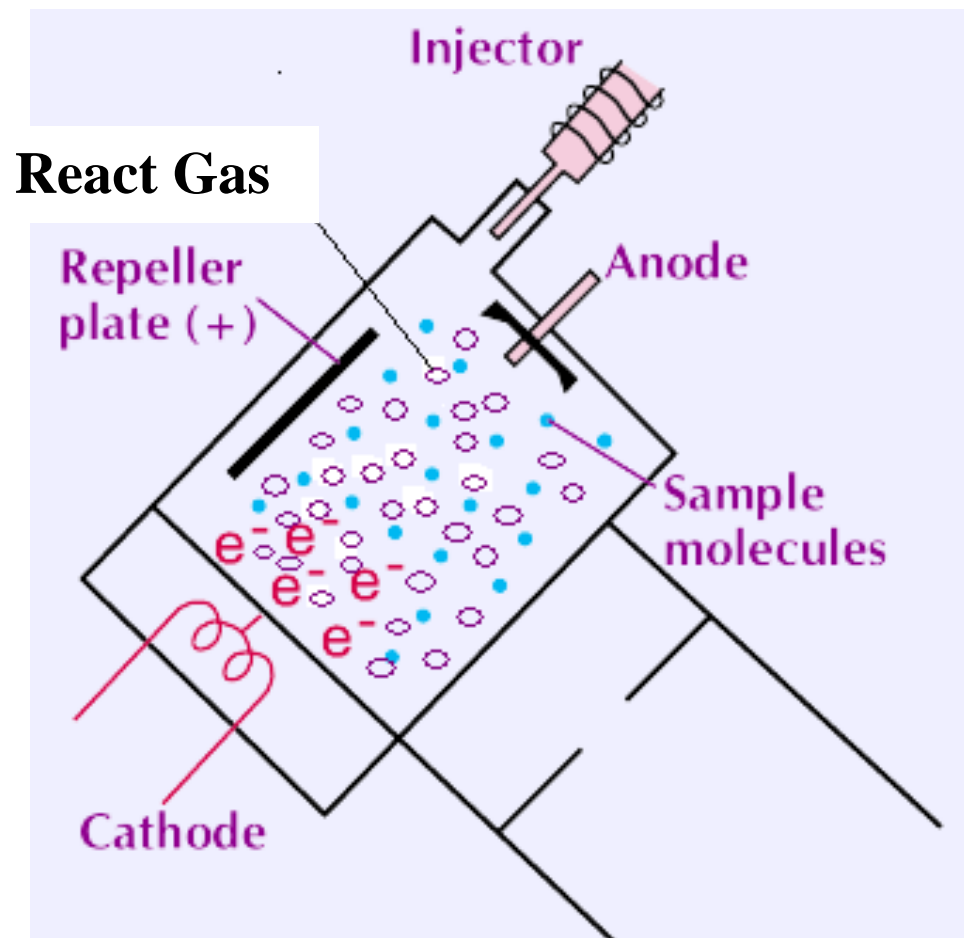
电子轰击源

(Electron Ionization EI)EI源



由GC进入的样品，以气体形式进入离子源，由灯丝发出的电子与样品分子发生碰撞使样品分子电离。在70eV电子碰撞作用下，有机物分子可能被打掉一个电子形成分子离子，也可能会发生化学键的断裂形成碎片离子。

化学电离源 (Chemical Impact) CI源



反应气体一般为甲烷、氨气、正丁烷等。

高能电子首先与反应气碰撞，然后再与样品发生分子-离子反应，产生的碎片相对较少，称为一种更软的电离。

以甲烷反应气为例介绍:

➤ 在电子轰击下，甲烷首先被电离:



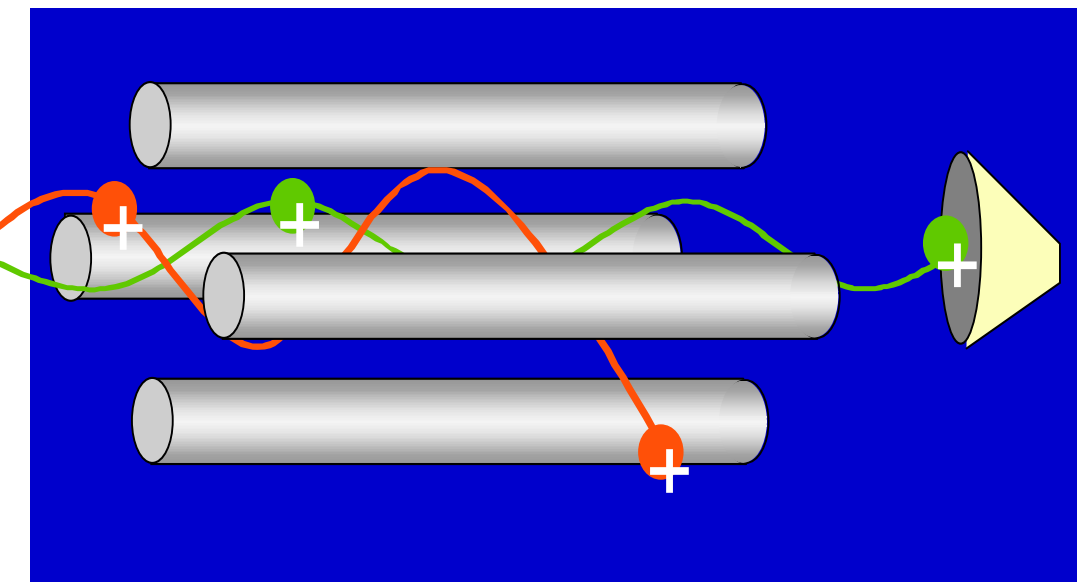
➤ 甲烷离子与分子进行反应，生成加合离子:



➤ 加合离子与样品分子反应:



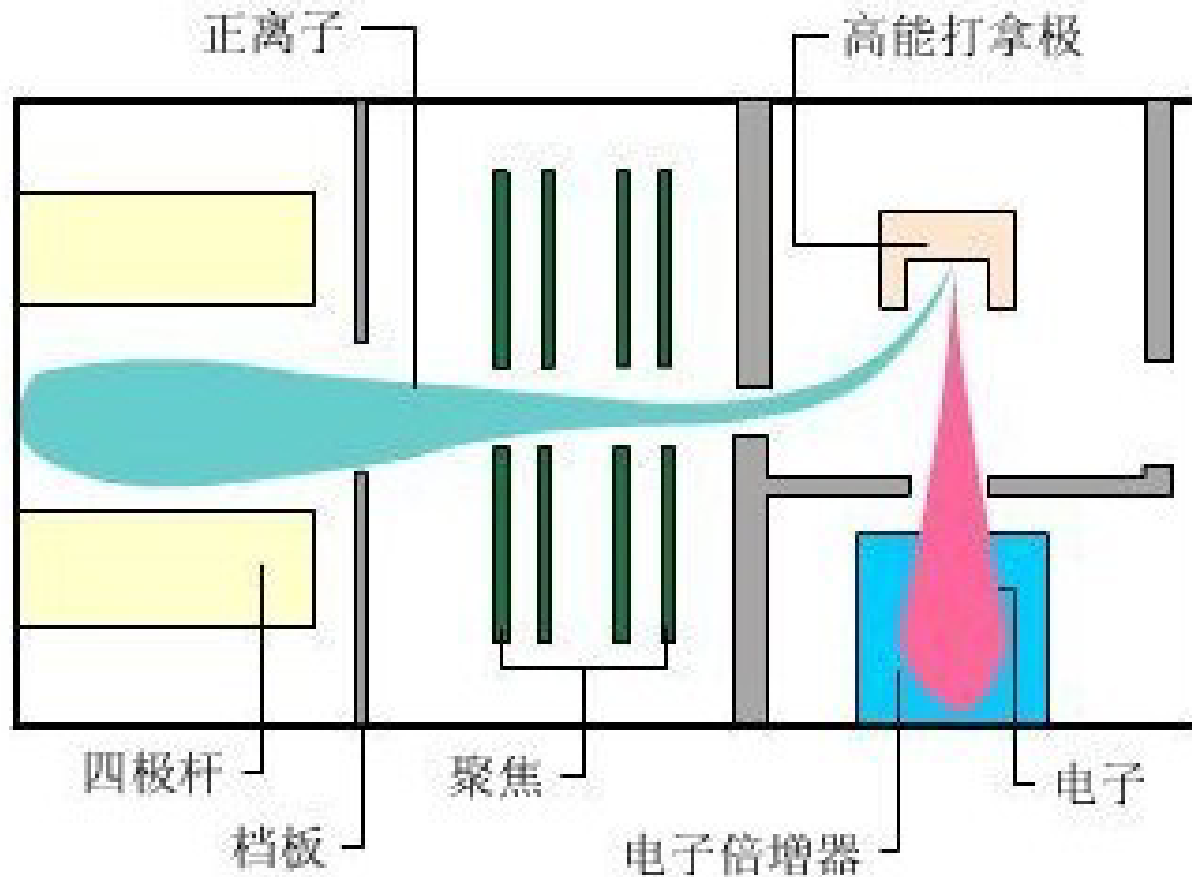
4. 四极杆质量分析器



过程：在两个相对的极杆之间加电压 $(U+V\cos\omega t)$ ，在另两个相对的极杆上加 $-(U+V\cos\omega t)$ 。离子进入可变电场后，只有具合适的曲率半径的离子可以通过中心小孔到达检测器。改谱 U 和 V 并保持 U/V 比值一定，可实现不同 m/z 离子的检测。

作用：将不同碎片按质荷比 m/z 分开

5. 检测器——电子倍增器



➔由四级杆出来的离子打到高能打拿级产生电子，电子经电子倍增器产生的电信号被送入计算机存储，这些信号经计算机处理后可以得到色谱图，质谱图及其他各种信息。

二、操作规程

点击File》》 New Method File, 建立分析方法文件。

The screenshot displays the GCMS Real Time Analysis software interface. The title bar reads "GCMS Real Time Analysis (Admin) - [Acquisition - Disabled, 060918-3.qgd(Live1), 060909.qgd]". The menu bar includes File, Edit, View, Method, Instrument, Acquisition, Data, Tools, Window, and Help. The File menu is open, showing options such as "New Method File", "Open Method File...", "Close Method File", "Save Method File", "Save Method File As...", "Save Method As Template...", "Load Method Parameters...", "Open Reference Data File...", "Close Reference Data File", "Select Project(Folder)...", "File Search...", "System Administration...", "Audit Trail...", "Print Setup...", "Print Method File", "Method File Properties...", and a list of recent method files. The main window shows a chromatogram with a yellow background and a grid. The x-axis is labeled "Time: 0:00 / 10:00 min" and "Scan #: 0". The y-axis is labeled "Intensity: 0" and "Group: 1". The baseline is flat at "Base Peak Intensity: 0". The x-axis has major ticks at 4.5, 5.0, 5.5, 6.0, 6.5, 7.0, 7.5, 8.0, and 8.5. The y-axis has a major tick at 50. The bottom right corner shows a small inset chromatogram with a y-axis labeled "Split" and a x-axis labeled "min".

GC参数设定

The screenshot displays the GCMS Real Time Analysis software interface. The top menu bar includes File, Edit, View, Method, Instrument, Acquisition, Data, Tools, Window, and Help. The toolbar contains various icons for file operations and analysis. The main window is divided into several sections:

- Acquisition Panel (Left):** Contains buttons for Top, Method, Sample Logon, Standby, Start, Stop, Method Detail, Snapshot, and Data.
- Chromatogram (Top Center):** Shows a plot of intensity versus time (min) with a yellow background. The x-axis ranges from 0.0 to 45.0 minutes. The y-axis ranges from 0.0 to 2.0. A single peak is visible at approximately 15.0 minutes.
- Parameters Panel (Bottom Left):** Displays various acquisition parameters:
 - Inj. Port: SP12-PT
 - Inj. Mast Port: (blank)
 - Column Oven Temp: 50.0 °C
 - Injection Temp: 200.0 °C
 - Injection Mode: Split
 - Split Ratio: 10.0
 - Flow Control Mode: Linear Velocity
 - Pressure: 300.2 kPa
 - Total Flow: 25.0 mL/min
 - Column Flow: 3.61 mL/min
 - Linear Velocity: 40.3 m/sec
 - Sample Flow: 3.0 mL/min
- Program Panel (Bottom Right):** Shows the temperature program settings:
 - Program: Column Oven Temperature
 - Table with columns: Run, Final Temperature, Hold
 - Total Program Time: 40.37 min

- 进样口温度
- 色谱柱温度
- 程序升温
- 进样模式
- 流量控制方法
- 流量控制参数
- 色谱柱参数

MS参数设定

GCMS Real Time Analysis (Admin) - [Acquisition - method-ET-2000S25.qgm, 040918-3.qgd(Lin1), 080903.qgt]

File Edit View Method Instrument Acquisition Data Tools Window Help

Acquisition

Sample Name : 080918-3
Sample ID :
Data Description :

Line1-MS Rel. Time 0:00:49.00 min Scan 0-1 Intensity 0 Group 1 C: 9.77

Intensity(10,000,000) Max Intensity

Base Peak m/z: 0.00 Base Peak Intensity: 0

GCMS-082010

Ion Source Temp. : 200 °C
Interface Temp. : 250 °C
Solvent Cut Time : 2.5 min
Micro Scan Width : 0 μ

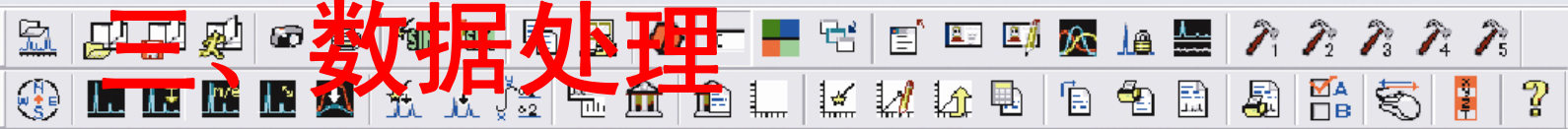
Detector Voltage : Relative to the Tuning Result Absolute
0 kV
Threshold : 700

Use MS Program : Program Time : 49.17 min
Group#1 = BasePeak1

	Start Time (min)	End Time (min)	Acq. Mode	Event Time (sec)	Scan Speed	Start m/z	End m/z	CSI m/z
1	3.00	49.00	Scan	0.50	1400	40.00	100.00	
2	0.00	0.00	Scan	0.00	0	0.00	0.00	

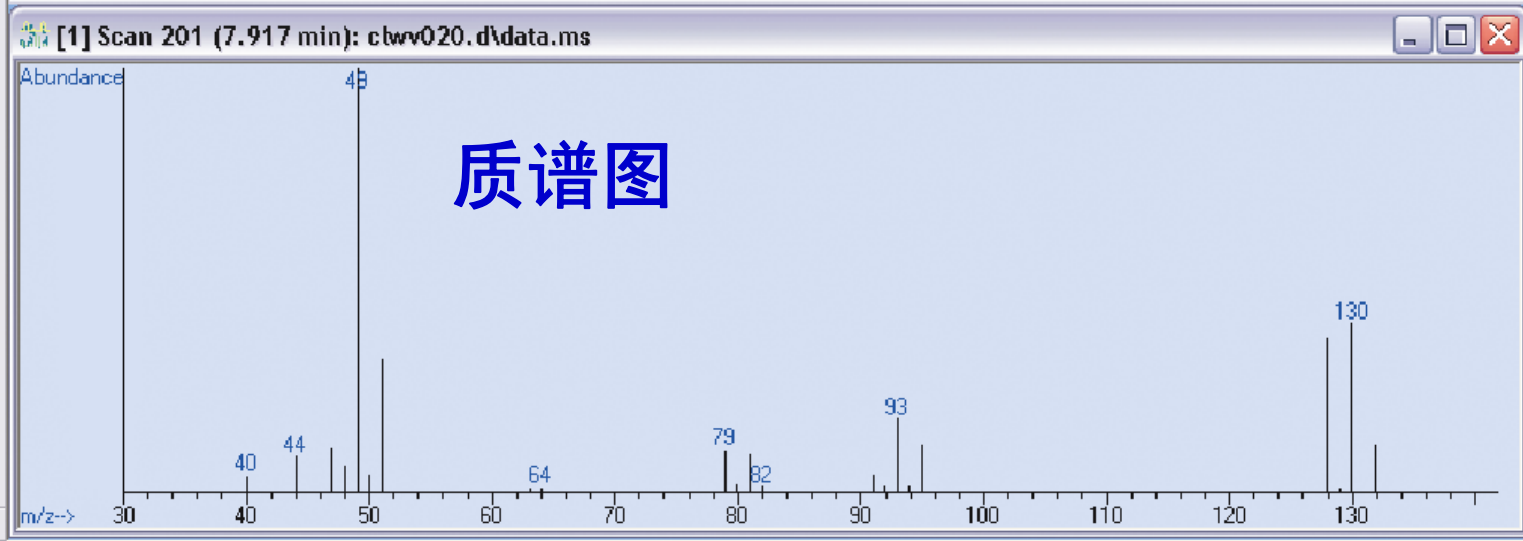
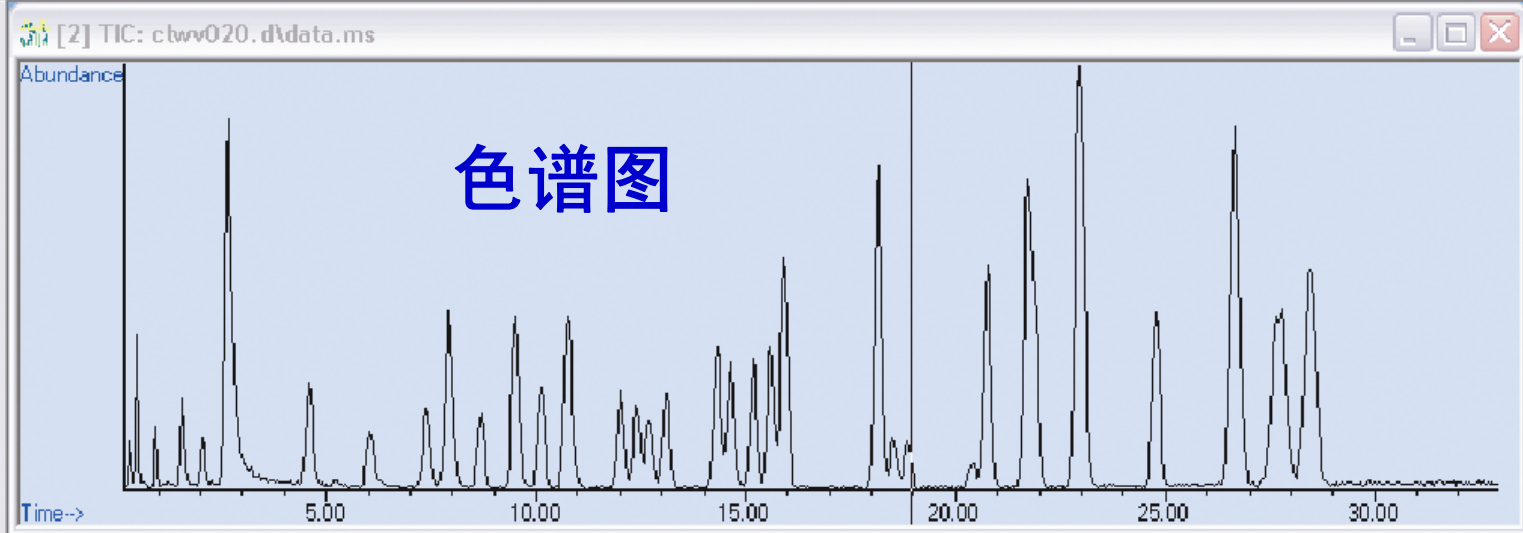
- 接口温度
- 离子源温度
- 溶剂切除时间
- 检测器电压
- 数据采集方式
- 阈值
- 扫描时间和速度
- 扫描范围 (SCAN)
- 选择离子 (SIM)

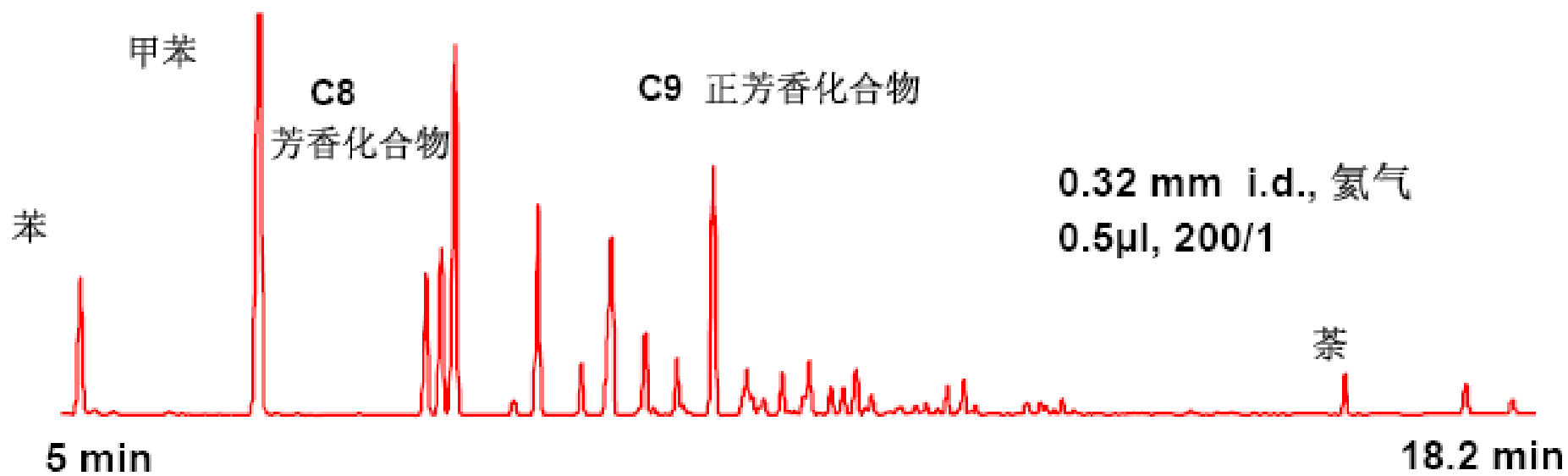
数据处理



Execute

- Browse
 - Canoscan
 - cmprents
 - compaq
 - compaqsettings
 - CPQAPPS
 - DATA
 - Database
 - dmi
 - docs
 - Documents and Setting
 - env
 - EnvDemo
 - bnadata
 - bnalist.m
 - gcdata
 - gcmetho
 - voadata
 - blwv002.d
 - clwv020.d**
 - clwv050.d
 - clwv100.d
 - clwv150.d
 - clwv200.d
 - dlwv002.d
 - mlwv014.d
 - nlwv014.d
 - slwv001.d
 - slwv014.d
 - tlwv001.d
 - tlwv002.d
- voalist.m





质谱图一定性分析

