

# 应用ISQ单四级杆气质联用仪检测16种多环芳烃

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## 关键词

- 多环芳烃
- 单四级杆GC-MS
- 定性定量

## 1. 引言

多环芳烃英文全称为polycyclic aromatic hydrocarbon, 简称PAHs, 包括萘、蒽、菲、芘等150余种化合物, 具有致癌作用多为四到六环的稠环化合物。国际癌症研究中心 (IARC) (1976年)列出的94种对实验动物致癌的化合物, 其中15种属于多环芳烃。由于苯并(a)芘是第一个被发现的环境化学致癌物, 且致癌性很强, 故常以苯并(a)芘作为多环芳的代表, 它占全部致癌性多环芳烃的1%-20%。目前, 大多数国家都将多环芳烃列为环境监测的重要内容之一, 中国政府列出的“中国环境优先监测黑名单”中包括7种PAHs; 美国环保总署1979年确定了16种PAHs作为优先监测污染物, 而德国政府强制规定所有在德国出售的电动工具必须经过检验其中不含有过量的PAHs。

ISQ单四级杆气质联用仪是由Thermo Fisher公司于2010年5月在德国慕尼黑Analytica展会上正式发布的最新一代单四级杆气质联用仪。ISQ在继承了Thermo Fisher气质联用一贯的超高灵敏度之外, 真正拥有不泄真空更换整个离子源的独特能力。配备了最新

ExtractaBrite™离子源的系统保证最大的运行时间和无限工作效率, 从而简化客户的日常操作。本文即采用ISQ单四级杆气质联用仪分析16种多环芳烃。

## 2. 实验部分

### 2.1 前处理方法

参照2005/69/EC指令, GS认证等方法, PVC材料和橡胶粉碎后, 称取1.0g, 经索氏提取6-12小时, 或通过微波萃取30分钟, 硅胶柱净化, 用30mL正己烷洗脱。样品浓缩后, 用正己烷定容至10ml, 进行GC/MS分析。

### 2.2 仪器与试剂

ISQ气质联用仪 (赛默飞世尔科技Thermo Fisher Scientific公司)。配置有EI源 (+/-CI源), Trace GC Ultra气质联用系统, 配程序升温进样口, TriPlus自动进样器, 进样体积为1ul。色谱柱为TR-5MS柱 (60m \*0.25mm \*0.25um)。

### 2.3 仪器条件

a. 质谱条件 (采用全扫描选择离子交替扫描):

The screenshot displays the Thermo Xcalibur Instrument Setup interface. The 'Method Setup' section shows the following parameters:

- Method type: Acquisition
- MS transfer line temp.: 250 °C
- Ion source temp.: 200 °C
- Acquisition threshold: 1000
- Ionization mode: EI
- CI gas type: Methane (Port A)
- CI gas flow: 1.00 mL/min
- Run completion: GC run time (selected), Stop after: 10.0 min

The 'Scans' table lists the following data:

Time (min)	Mass List or Range (amu)	Dwell or Scan Times (sec)	Tune File Name
6.50	45-350	0.1	(Last Saved)
	128, 127, 102, 129	0.03, 0.03, 0.03, 0.03	(Last Saved)
7.50	45-350	0.1	(Last Saved)
	153, 151, 152, 150	0.03, 0.03, 0.03, 0.03	(Last Saved)
8.50	45-350	0.1	(Last Saved)
	167, 165, 166, 139	0.03, 0.03, 0.03, 0.03	(Last Saved)
9.30	45-350	0.1	(Last Saved)
	152, 176, 178, 179	0.03, 0.03, 0.03, 0.03	(Last Saved)
10.50	45-350	0.1	(Last Saved)
	101, 200, 202, 203	0.03, 0.03, 0.03, 0.03	(Last Saved)
13.00	45-350	0.1	(Last Saved)
	226, 228, 227, 229, 202	0.03, 0.03, 0.03, 0.03, 0.03	(Last Saved)
16.00	45-350	0.1	(Last Saved)
	126, 250, 252, 253	0.04, 0.05, 0.05, 0.05	(Last Saved)
22.00	45-350	0.1	(Last Saved)
	276, 277, 278, 279, 138, 139, 248, 252	0.03, 0.03, 0.03, 0.03, 0.03, 0.03, 0.03, 0.03	(Last Saved)

The 'Groups' table on the right shows the following data:

Time (min)	Total Scan Time (sec)
6.50	0.24
7.50	0.24
8.50	0.24
9.30	0.24
10.50	0.24
13.00	0.274
16.00	0.31
22.00	0.376

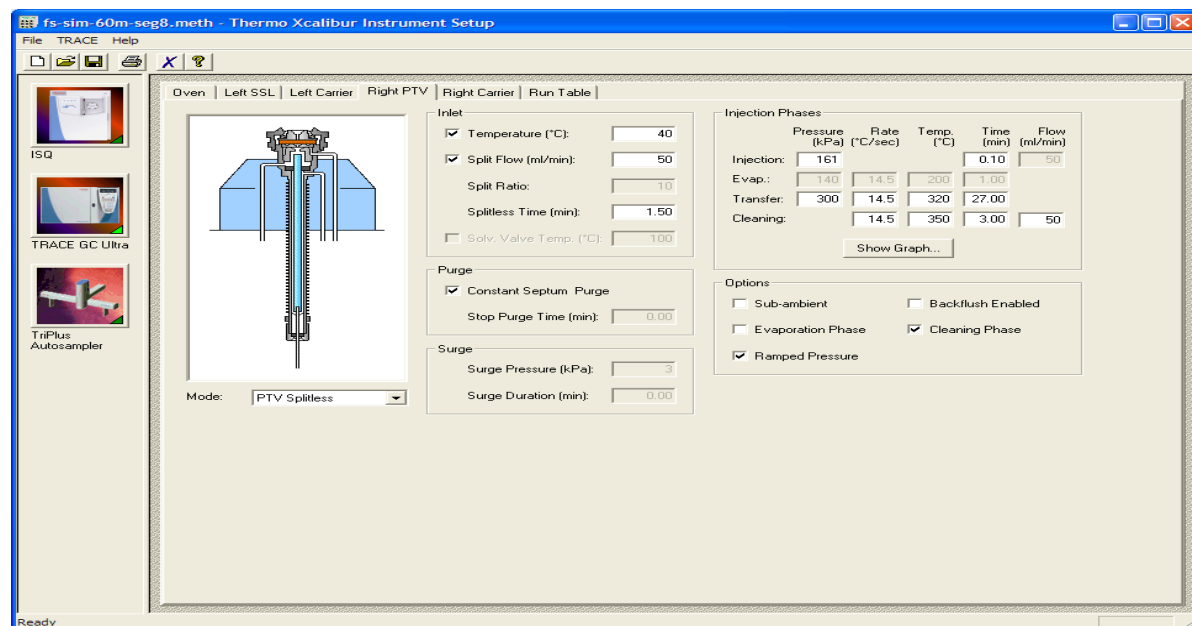
The interface also includes a sidebar with icons for ISQ, TRACE GC Ultra, and TriPlus Autosampler, and a bottom status bar showing 'Ready'.

b. 气相方法：

柱温箱：40°C起始保持0.5min，以40°C/min的速率升至250 °C，再以6.5 °C/min的速率升至300°C，保持18min。

载气：梯度流速方法，1.3ml/min起始保持15min，然后以1ml/min的速率升至2ml/min，再保持15min。

PTV进样口参数设置如下：



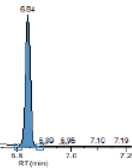
### 3. 结果与讨论

#### 3.1 色谱分离

图1为16种1000pg PAHs的色谱图，按照保留时间依次列出。用QuanLab Form™软件（Thermo Scientific）处理标准品和未知样品。QuanLab Form™可以确定1个定量离子和2个定性离子的比例，从而更准确的完成定性定量的工作。

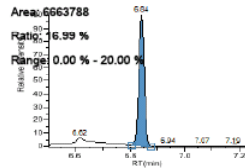
## Naphthalene

Quan m/z: 128.00  
Total Area: 39211965  
Peak Area: 39211965  
RT: 6.84min (6.84)  
Amount: 1000.000  
TAmount: 1000.000



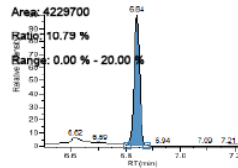
Qual m/z: 127.00

Area: 6663788  
Ratio: 16.99 %  
Range: 0.00 % - 20.00 %

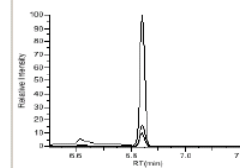


Qual m/z: 129.00

Area: 4229700  
Ratio: 10.79 %  
Range: 0.00 % - 20.00 %

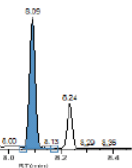


RT: 6.84 | data02



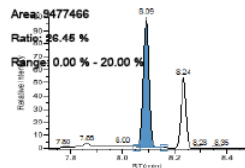
## Acenaphthylene

Quan m/z: 152.00  
Total Area: 35830080  
Peak Area: 35830080  
RT: 8.09min (8.09)  
Amount: 1000.000  
TAmount: 1000.000



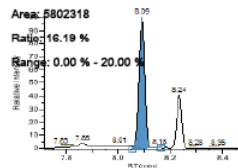
Qual m/z: 151.00

Area: 9477466  
Ratio: 26.46 %  
Range: 0.00 % - 20.00 %

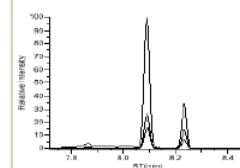


Qual m/z: 150.00

Area: 5802318  
Ratio: 16.19 %  
Range: 0.00 % - 20.00 %

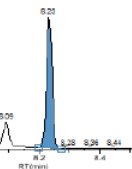


RT: 8.09 | data02



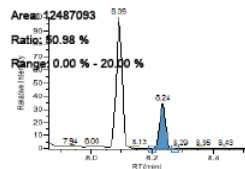
## Acenaphthene

Quan m/z: 153.00  
Total Area: 24493412  
Peak Area: 24493412  
RT: 8.23min (8.23)  
Amount: 1000.000  
TAmount: 1000.000



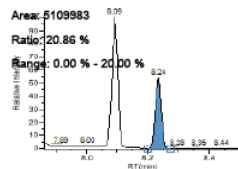
Qual m/z: 152.00

Area: 12487093  
Ratio: 50.98 %  
Range: 0.00 % - 20.00 %

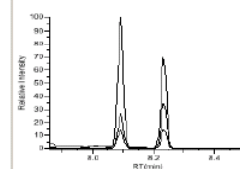


Qual m/z: 151.00

Area: 5109983  
Ratio: 20.86 %  
Range: 0.00 % - 20.00 %

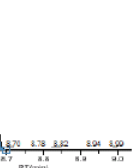


RT: 8.23 | data02



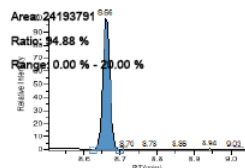
## Fluorene

Quan m/z: 166.00  
Total Area: 25498362  
Peak Area: 25498362  
RT: 8.66min (8.66)  
Amount: 1000.000  
TAmount: 1000.000



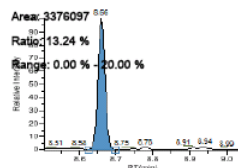
Qual m/z: 165.00

Area: 24193791  
Ratio: 94.88 %  
Range: 0.00 % - 20.00 %

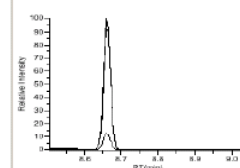


Qual m/z: 167.00

Area: 3376097  
Ratio: 13.24 %  
Range: 0.00 % - 20.00 %

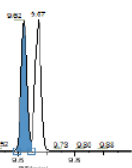


RT: 8.66 | data02



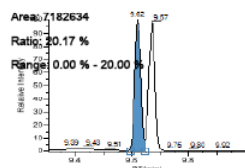
## Phenanthrene

Quan m/z: 178.00  
Total Area: 35608800  
Peak Area: 35608800  
RT: 9.62min (9.62)  
Amount: 1000.000  
TAmount: 1000.000



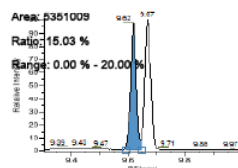
Qual m/z: 176.00

Area: 7182634  
Ratio: 20.17 %  
Range: 0.00 % - 20.00 %

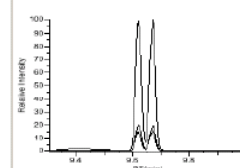


Qual m/z: 179.00

Area: 5351009  
Ratio: 15.03 %  
Range: 0.00 % - 20.00 %

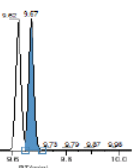


RT: 9.62 | data02



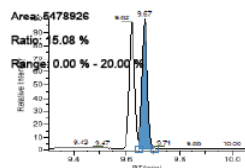
## Anthracene

Quan m/z: 178.00  
Total Area: 36322551  
Peak Area: 36322551  
RT: 9.67min (9.67)  
Amount: 1000.000  
TAmount: 1000.000



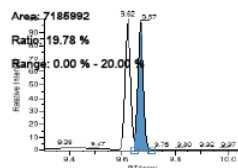
Qual m/z: 179.00

Area: 5478926  
Ratio: 15.08 %  
Range: 0.00 % - 20.00 %

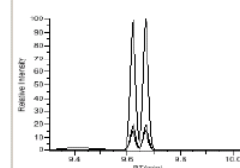


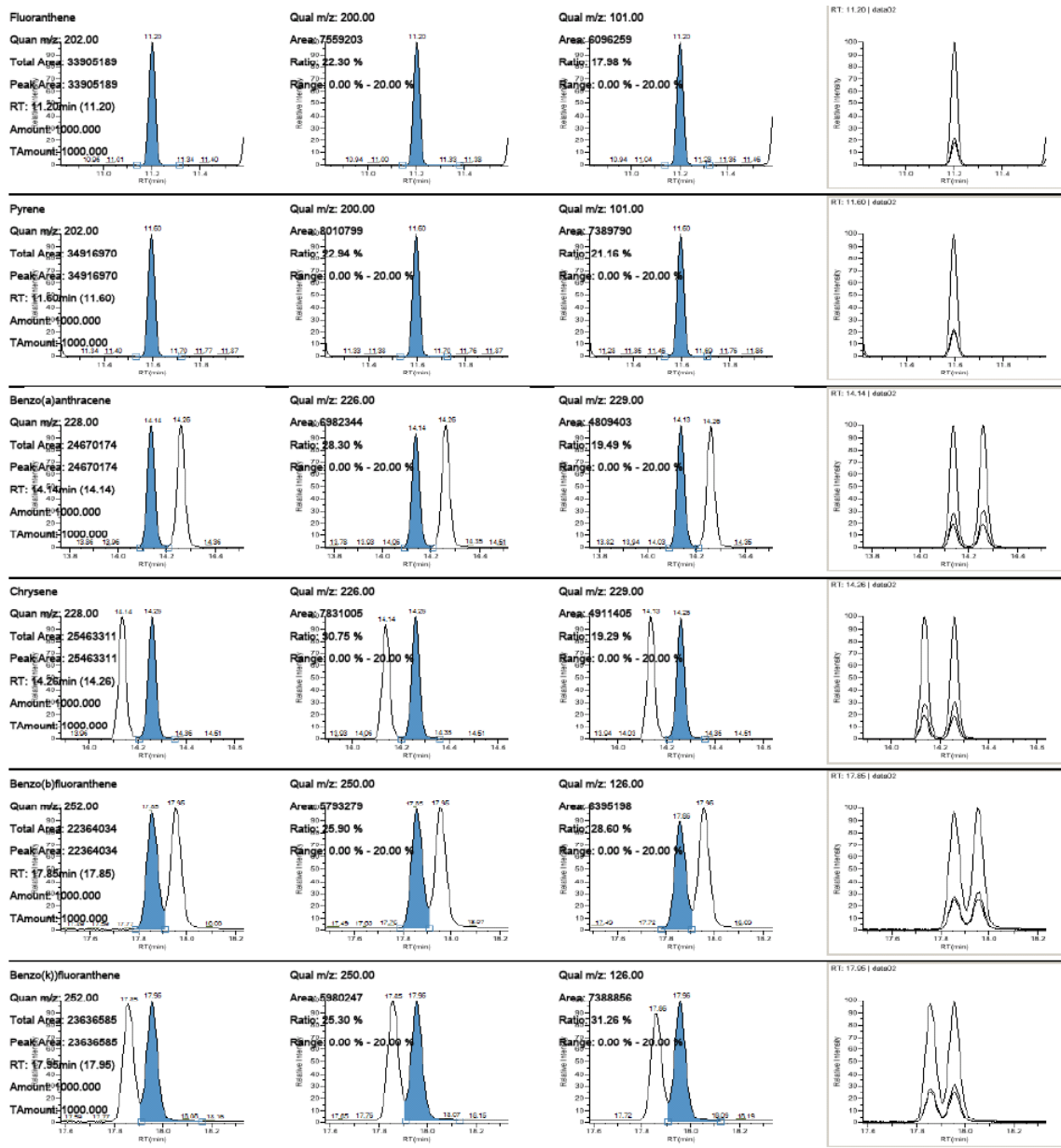
Qual m/z: 176.00

Area: 7189992  
Ratio: 19.78 %  
Range: 0.00 % - 20.00 %



RT: 9.67 | data02





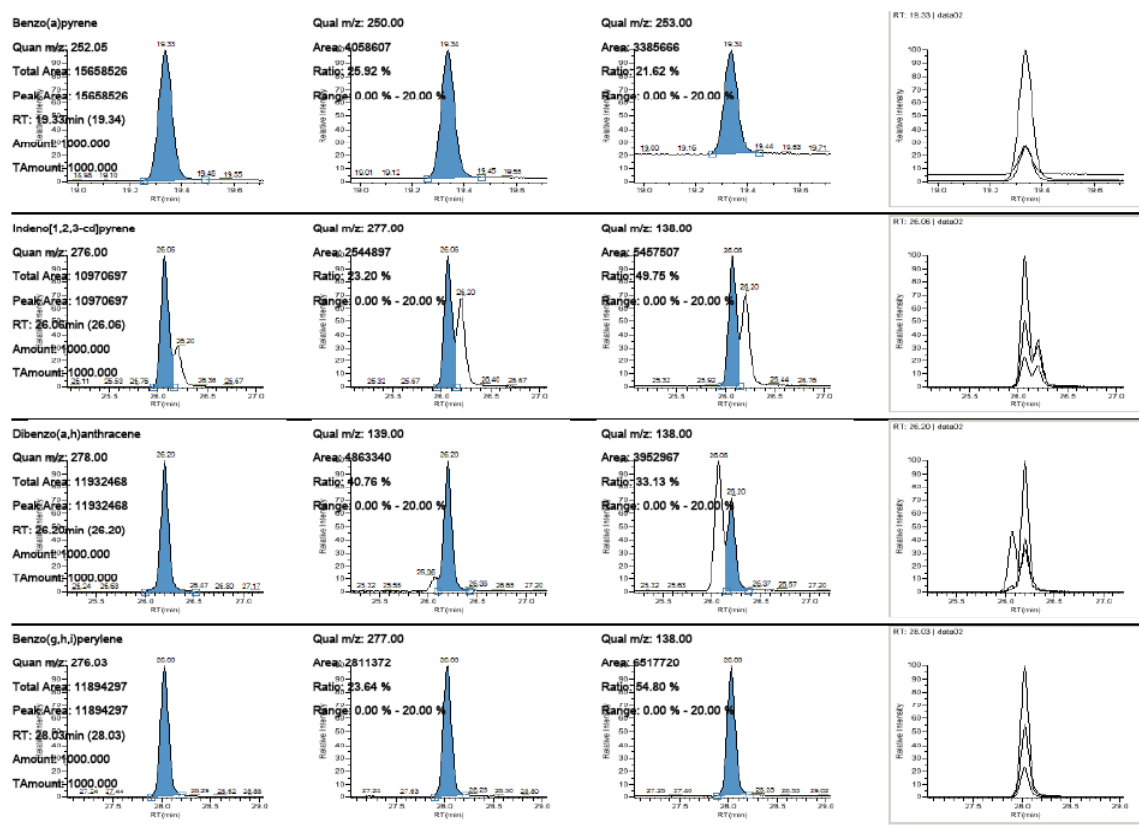
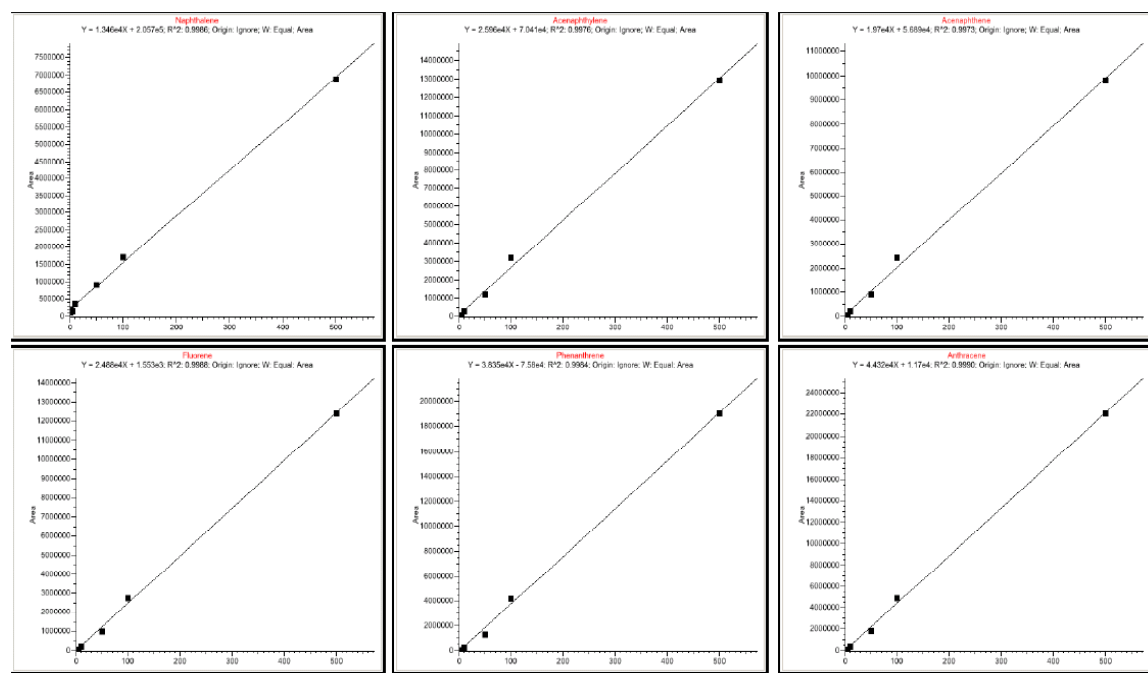


图1：16种PAHs的标准色谱图，包括1个定量离子色谱图和2个定性离子色谱图，以及3个离子的叠加谱图。

### 3.2 标准曲线及方法学验证

配制7个梯度浓度的的混标溶液，图2为16种PAHs6个浓度点的线性方程，浓度设置分别为1pg, 2pg, 5pg, 10pg, 50pg, 100pg500pg, R2均大于0.995，表明线性良好。配制高中低浓度的混标（5ppb, 50ppb和500ppb），重复进样5次检测精密度，最终各化合物的精密度在1.01-5.53%之间，方法学的验证表明此方法快速有效。



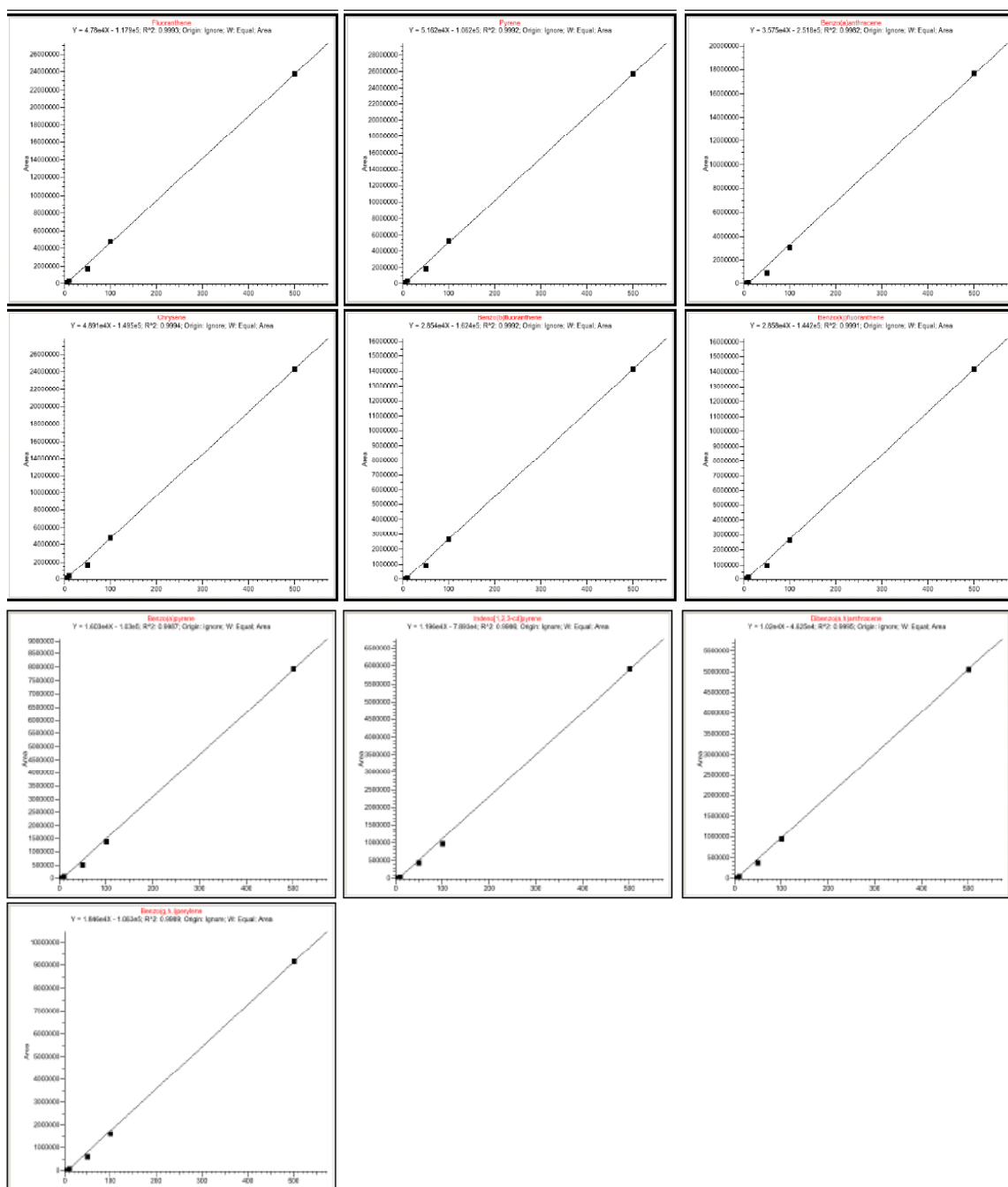


图2 各化合物的标准曲线

#### 4. 总结

Thermo Fisher新款的ISQ单四级杆气质联用仪，拥有超高的抗污染能力和超高的灵敏度，本文即采用ISQ单四级杆气质联用仪检测16种PAHs，方法简便、高效、快速、准确、灵敏度高、实用性强。采用全扫描选择离子扫描方式可以同时完成定性定量的工作，使定性结果更准确可靠。采用PTV进样口可以在进样口进行一次程序升温，使16种PAHs在进样口即有一个初步的分离，从而使PAHs在色谱柱上的分离效果更好。

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

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