

# 安捷伦 6420 液质联用仪检测器打拿极的清洗

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**摘要：**安捷伦 6420 液质联用仪调谐异常，经排查为打拿极有污染，经过清洗后，调谐顺利通过。

**关键词：**6420 液质联用仪，打拿极，污染，清洗，调谐

前面说到单位一台尘封多年的 6420 因为扩项需要使用，经过一天的折腾后，总算能进样分析了。可是出峰是没有出峰的。那先做个调谐吧，调谐过程是一波三折。从我连怎么看喷雾都不知道，到调谐液污染，清洗离子源，重新安装调整挡片…，最后得到一片黄绿色的调谐报告。那还搞了个啥，继续问工程师。

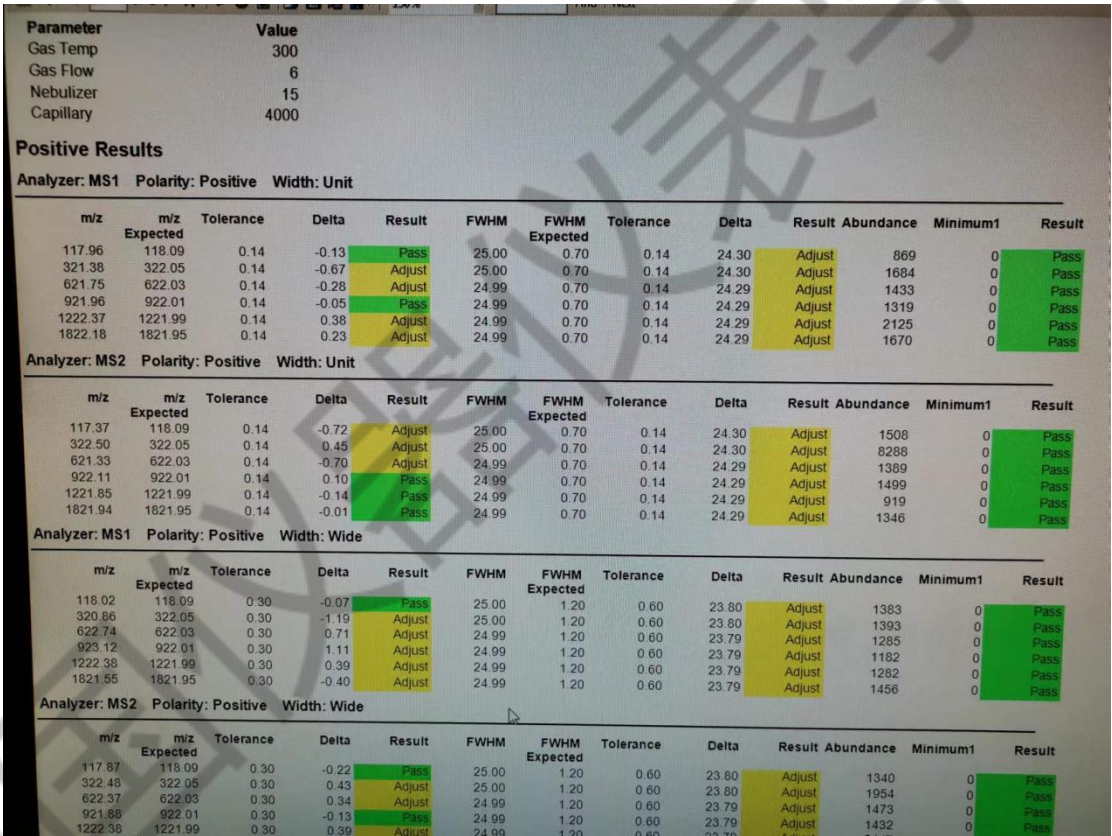


图 0

1. 完成基本操作后，分别在手动调谐模式下，观察质谱图，发现不管 MS1 在调谐液打开还是关闭的情况下，都有很多杂峰，如图 1，图 2。怀疑是打拿极污染，需要清洗。

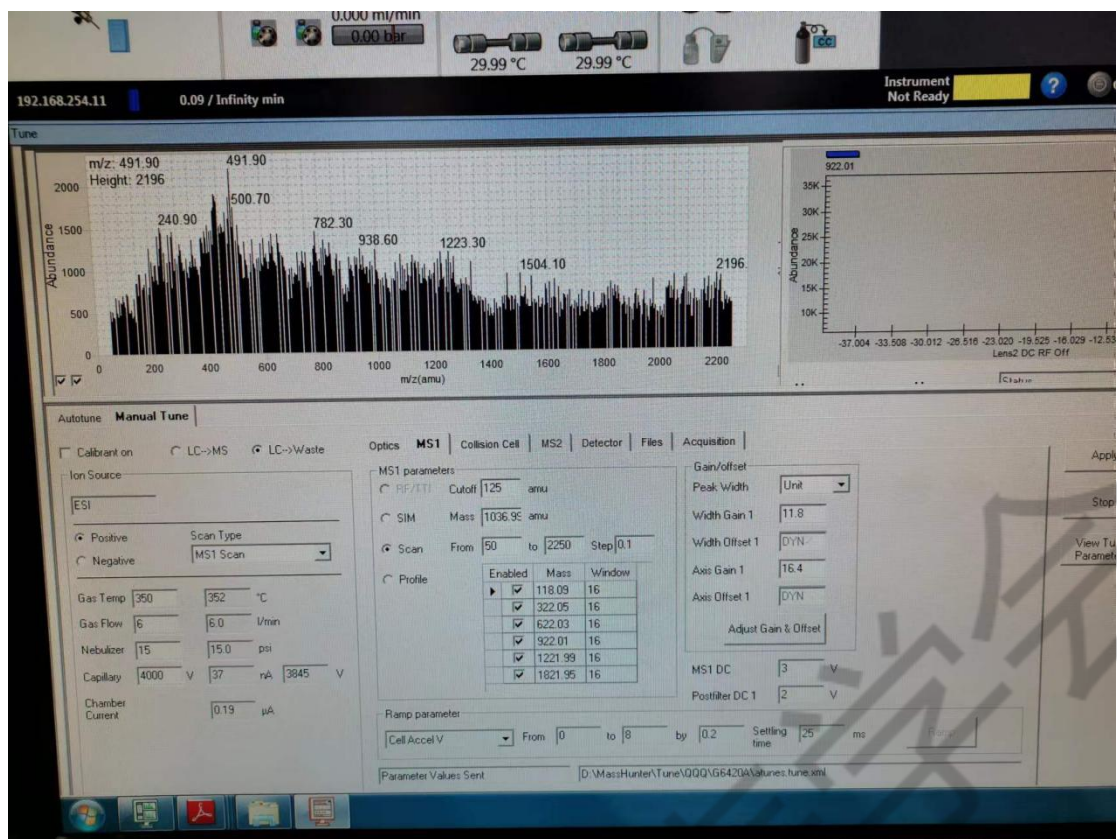


图 1 关闭调谐液下 ms1 扫描

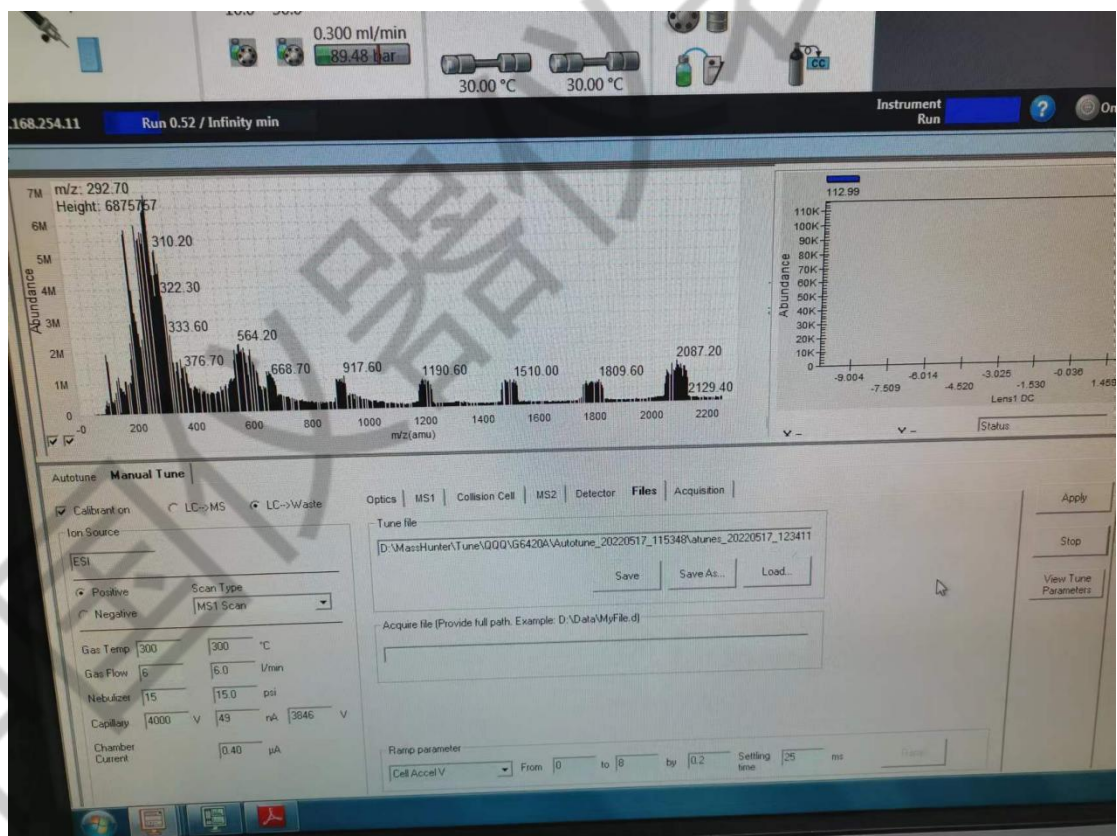


图 2 打开调谐液的 ms1 扫描

2. 工程师建议报修，由他们来做清洗。但是我觉得我可以，因为气质的打拿极，倍增管我是洗过多次的，没有技术难度。工程师听说后，同意指导我做清洗。先关机，放空，取下前盖板，再取下左侧的电源板，如图 3，注意这一坨，后面还有排线，要注意别掰歪了。

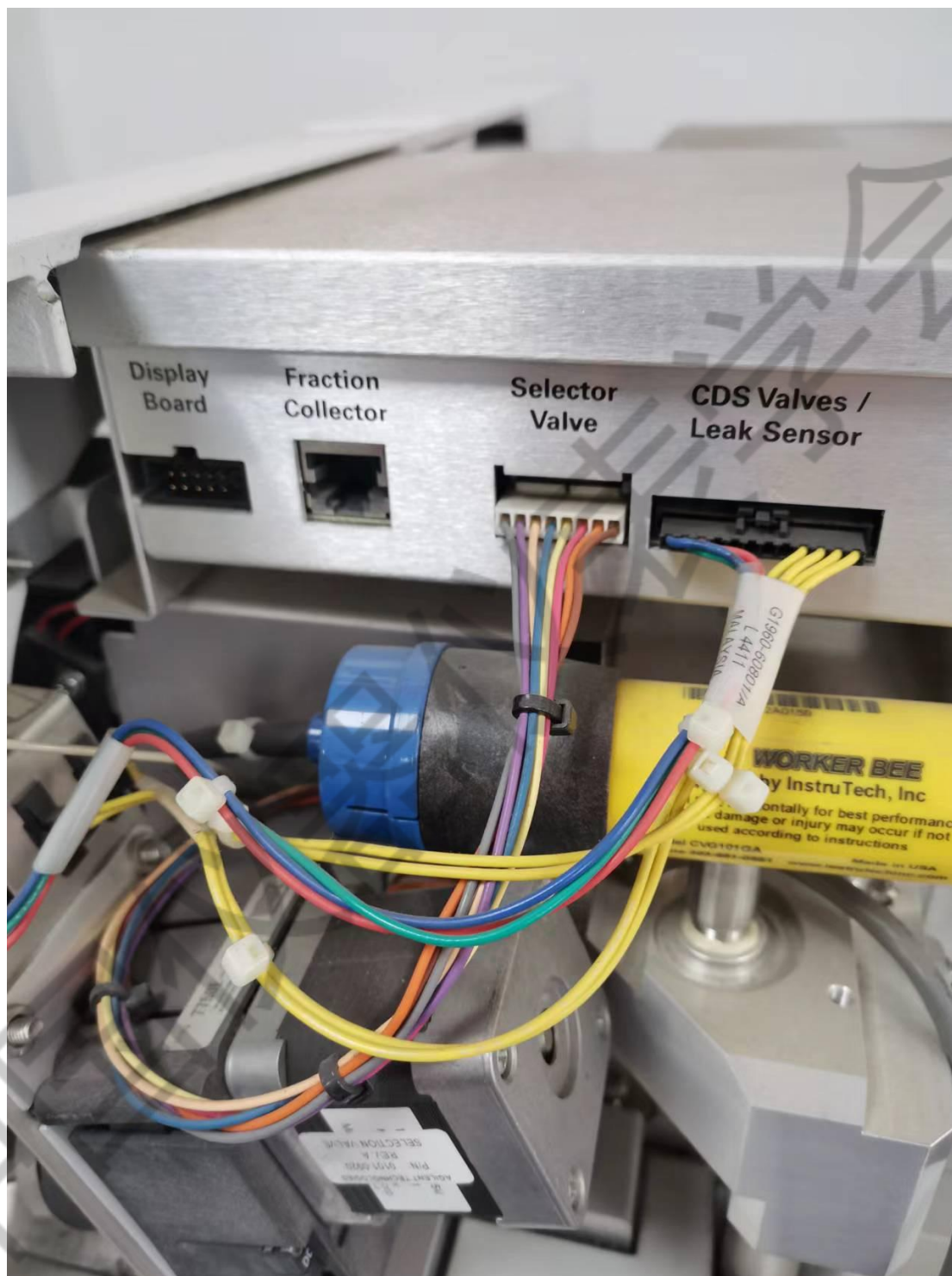


图 3 电源模块

3. 然后拧下真空腔盖板的四个螺丝，取下盖板，注意有一个大的 o 圈，别整断了，如图 4



图 4

4. 右侧就是打拿极和倍增管了，拧下固定的两颗螺丝，拔掉连接线，就可以一同取下打拿极和倍增管，如图 5



图5 注意倍增管上还有两条电线

5. 单独拆下倍增管，打拿极是在整个左侧一坨的中间，要拆下来需要动到多颗螺丝，而我又没合适的螺丝刀，那就这样吧。如图6

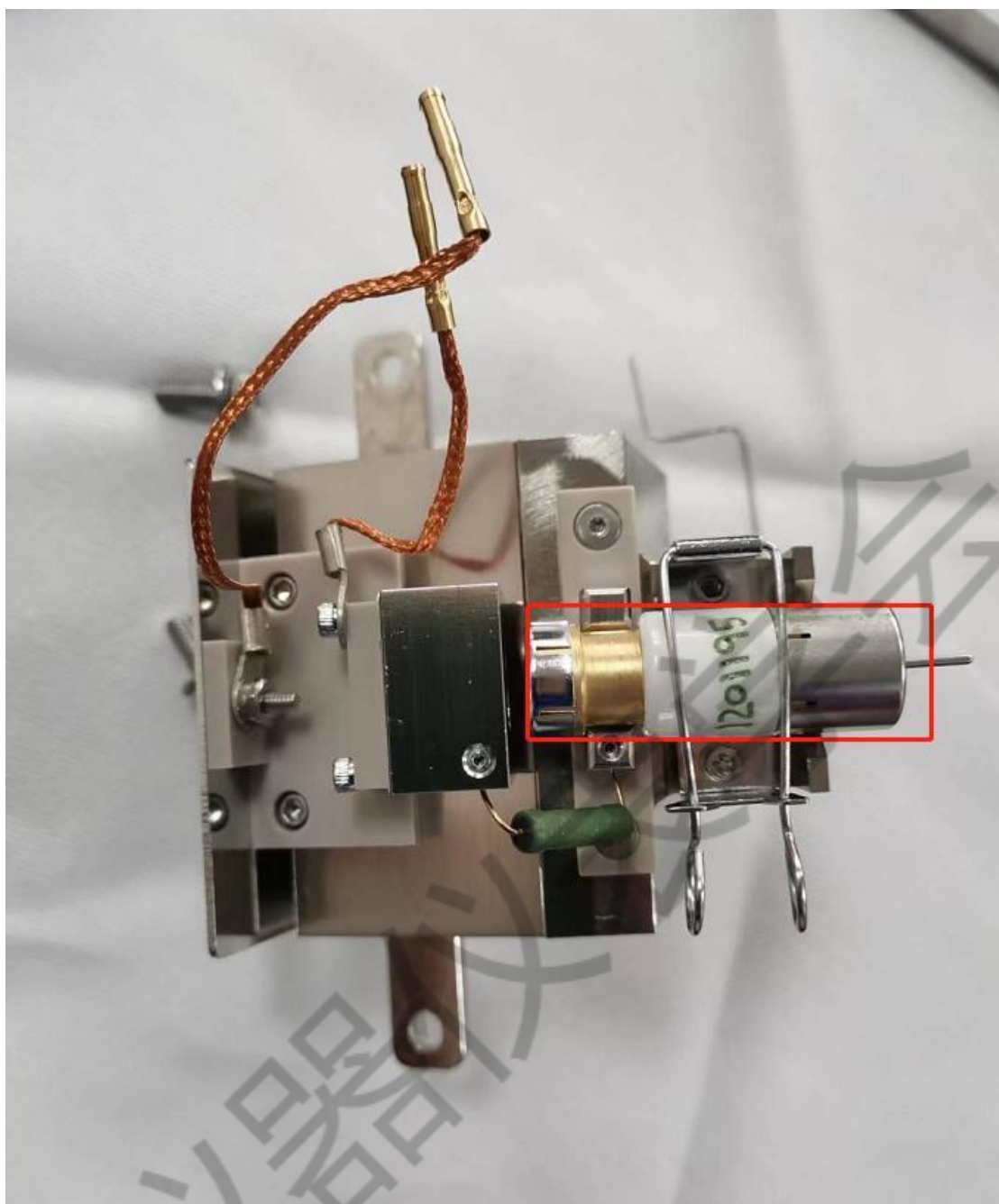


图 6

6. 分开 EM 后，把打拿极一大坨放到甲醇中超声，注意上面有个绿色的电阻，那个被浸润到甲醇中。手拿着，放甲醇中超声，如图 7

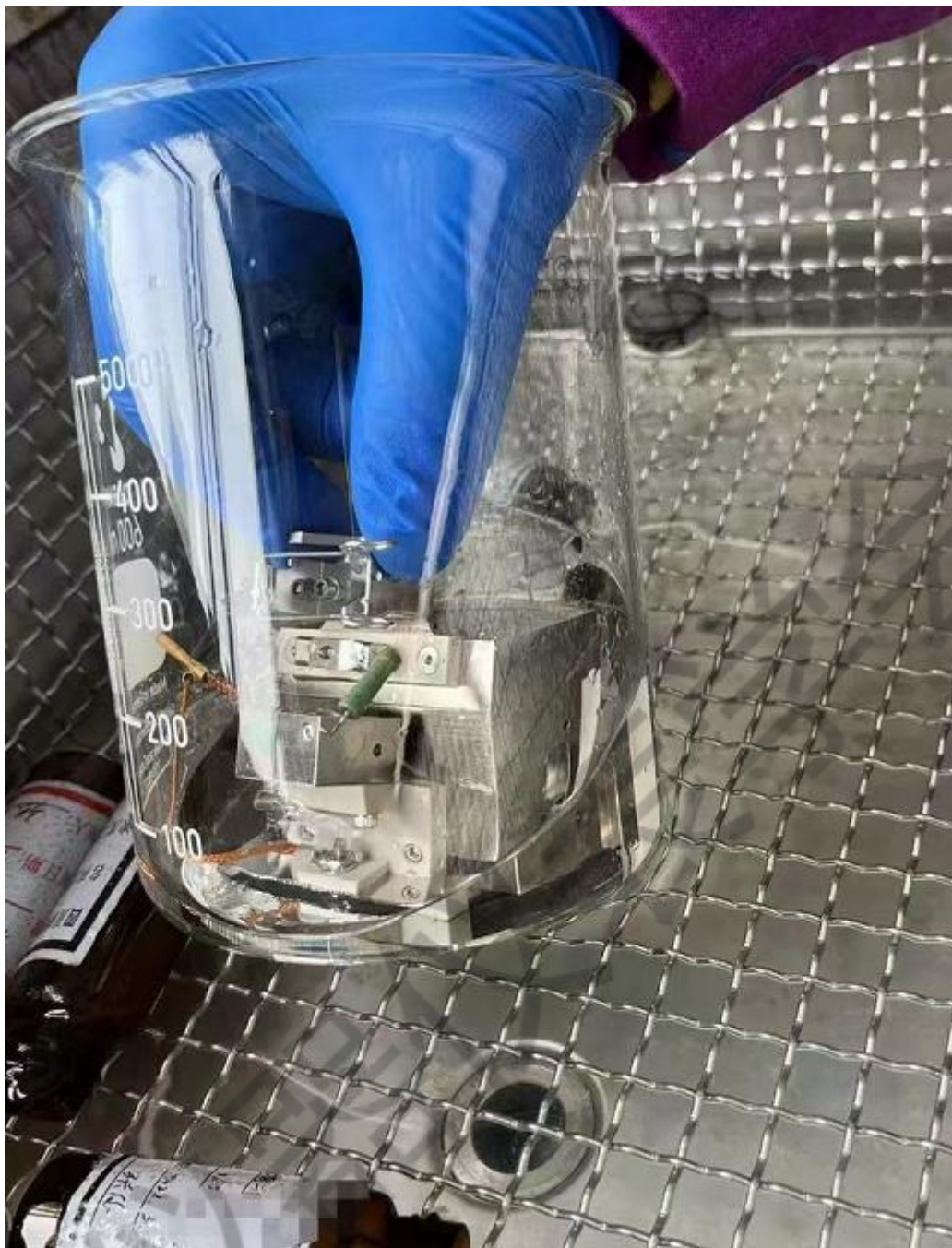


图 7

7. 超声两次后，放烧杯里，再盖上无尘布，放 GC 柱温箱里，80℃烘干。烘干打拿极时，顺便把 EM 洗洗，直接放甲醇里晃晃，用甲醇冲洗一下就可以，然后也放入柱温箱，一起烘干。如图 8，洗好的。



图 8

8. 烘干后，转回仪器，抽真空。然后手动调谐看看，哇哦，挺不错的，杂峰少了很多。MS1 和 MS2 扫描都好多了如图 9，图 10

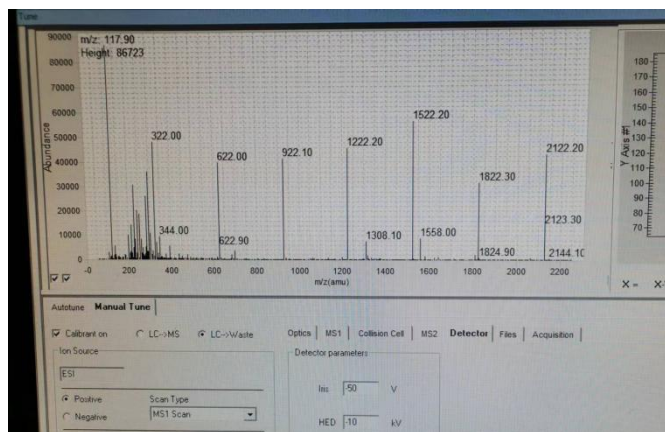


图 9 清洗后 MS1 扫描

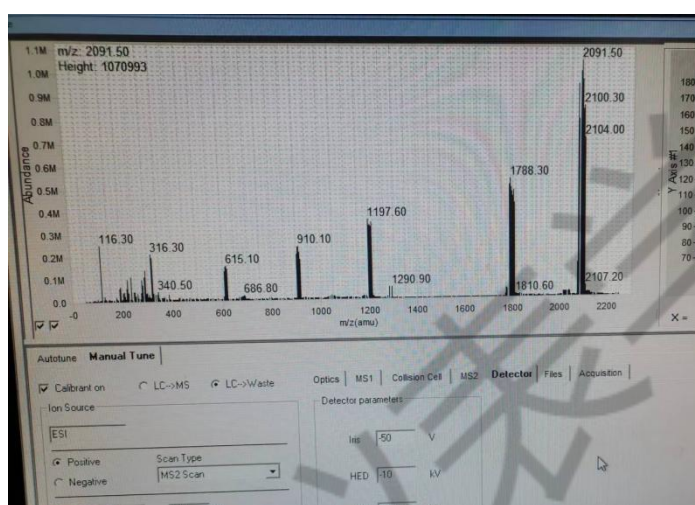


图 10 清洗后 MS2 扫描

9. 做个调谐，忐忑的等待结果，看到了绿油油的一片，心情真好，调谐总算顺利过了，正模式全绿，负模式只有一个黄，如图 11，图 12。仪器正常了，那及可以含泪开始做方法验证了。

Source Parameters

| Parameter | Value |
|-----------|-------|
| Gas Temp  | 300   |
| Gas Flow  | 5     |
| Nebulizer | 15    |
| Capillary | 4000  |

Positive Results

Analyzer: M31 Polarity: Positive Width: Unit

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 118.04 | 118.04   | 0.10      | -0.00 | Pass   | 0.67 | 0.70          | 0.14      | -0.03 | Pass   | 11962     | 0       | Pass   |
| 102.05 | 102.05   | 0.10      | -0.01 | Pass   | 0.66 | 0.70          | 0.14      | -0.04 | Pass   | 8910      | 0       | Pass   |
| 82.05  | 82.05    | 0.10      | -0.01 | Pass   | 0.67 | 0.70          | 0.14      | -0.03 | Pass   | 5819      | 0       | Pass   |
| 62.05  | 62.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | -0.00 | Pass   | 4053      | 0       | Pass   |
| 129.07 | 129.07   | 0.10      | -0.01 | Pass   | 0.69 | 0.70          | 0.14      | -0.01 | Pass   | 4547      | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.00 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1204      | 0       | Pass   |

Analyzer: M32 Polarity: Positive Width: Unit

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 118.04 | 118.04   | 0.10      | -0.01 | Pass   | 0.68 | 0.70          | 0.14      | -0.02 | Pass   | 12815     | 0       | Pass   |
| 102.05 | 102.05   | 0.10      | -0.01 | Pass   | 0.71 | 0.70          | 0.14      | 0.01  | Pass   | 4637      | 0       | Pass   |
| 82.05  | 82.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 11034     | 0       | Pass   |
| 62.05  | 62.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 2948      | 0       | Pass   |
| 129.07 | 129.07   | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 2525      | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.00 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 747       | 0       | Pass   |

Analyzer: M33 Polarity: Positive Width: Wide

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 117.10 | 117.10   | 0.00      | -0.02 | Pass   | 1.06 | 1.20          | 0.00      | -0.14 | Pass   | 14545     | 0       | Pass   |
| 121.07 | 121.07   | 0.00      | -0.08 | Pass   | 1.27 | 1.20          | 0.00      | 0.07  | Pass   | 11034     | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.02 | Pass   | 1.14 | 1.20          | 0.00      | -0.06 | Pass   | 7176      | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.03 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 4541      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 9527      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 1932      | 0       | Pass   |

Analyzer: M32 Polarity: Positive Width: Wide

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 118.07 | 118.07   | 0.00      | -0.02 | Pass   | 1.18 | 1.20          | 0.00      | -0.02 | Pass   | 15419     | 0       | Pass   |
| 102.05 | 102.05   | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 10819     | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 1.21 | 1.20          | 0.00      | 0.01  | Pass   | 16332     | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 7520      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 6709      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.20 | 1.20          | 0.00      | 0.00  | Pass   | 9011      | 0       | Pass   |

Analyzer: M31 Polarity: Positive Width: Widest

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 117.10 | 117.10   | 0.00      | -0.02 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 20034     | 0       | Pass   |
| 121.07 | 121.07   | 0.00      | -0.08 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 10337     | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.02 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 5654      | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.03 | Pass   | 2.38 | 2.50          | 0.00      | -0.12 | Pass   | 10514     | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 2.38 | 2.50          | 0.00      | -0.12 | Pass   | 9812      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.52 | 2.50          | 0.00      | 0.02  | Pass   | 11037     | 0       | Pass   |

Analyzer: M32 Polarity: Positive Width: Widest

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 118.02 | 118.02   | 0.00      | -0.01 | Pass   | 2.51 | 2.50          | 0.00      | 0.01  | Pass   | 26401     | 0       | Pass   |
| 102.05 | 102.05   | 0.00      | -0.01 | Pass   | 2.50 | 2.50          | 0.00      | 0.00  | Pass   | 15419     | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 2.50 | 2.50          | 0.00      | 0.00  | Pass   | 15402     | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 2.50 | 2.50          | 0.00      | 0.00  | Pass   | 7520      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 2.50 | 2.50          | 0.00      | 0.00  | Pass   | 21736     | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.50 | 2.50          | 0.00      | 0.00  | Pass   | 21736     | 0       | Pass   |

图 11

Source Parameters

| Parameter | Value |
|-----------|-------|
| Gas Temp  | 300   |
| Gas Flow  | 5     |
| Nebulizer | 15    |
| Capillary | 3000  |

Negative Results

Analyzer: M31 Polarity: Negative Width: Unit

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.10      | -0.00 | Pass   | 0.69 | 0.70          | 0.14      | -0.01 | Pass   | 1584      | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1584      | 0       | Pass   |
| 82.05  | 82.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1584      | 0       | Pass   |
| 62.05  | 62.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1584      | 0       | Pass   |
| 129.07 | 129.07   | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1584      | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 1584      | 0       | Pass   |

Analyzer: M32 Polarity: Negative Width: Unit

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.10      | -0.01 | Pass   | 0.67 | 0.70          | 0.14      | -0.03 | Pass   | 21139     | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.01 | Pass   | 0.68 | 0.70          | 0.14      | -0.02 | Pass   | 1989      | 0       | Pass   |
| 82.05  | 82.05    | 0.10      | -0.01 | Pass   | 0.69 | 0.70          | 0.14      | -0.01 | Pass   | 20732     | 0       | Pass   |
| 62.05  | 62.05    | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 24622     | 0       | Pass   |
| 129.07 | 129.07   | 0.10      | -0.01 | Pass   | 0.67 | 0.70          | 0.14      | -0.03 | Pass   | 14670     | 0       | Pass   |
| 101.06 | 101.06   | 0.10      | -0.01 | Pass   | 0.70 | 0.70          | 0.14      | 0.00  | Pass   | 14670     | 0       | Pass   |

Analyzer: M31 Polarity: Negative Width: Wide

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.00      | -0.01 | Pass   | 1.18 | 1.20          | 0.00      | -0.02 | Pass   | 1697      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2132      | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 24622     | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 24622     | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 24622     | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 24622     | 0       | Pass   |

Analyzer: M32 Polarity: Negative Width: Wide

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 1.19 | 1.20          | 0.00      | -0.01 | Pass   | 2031      | 0       | Pass   |

Analyzer: M31 Polarity: Negative Width: Widest

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |

Analyzer: M32 Polarity: Negative Width: Widest

| m/z    | Expected | Tolerance | Delta | Result | FWHM | FWHM Expected | Tolerance | Delta | Result | Abundance | Minimum | Result |
|--------|----------|-----------|-------|--------|------|---------------|-----------|-------|--------|-----------|---------|--------|
| 112.91 | 112.91   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 82.05  | 82.05    | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 62.05  | 62.05    | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 129.07 | 129.07   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |
| 101.06 | 101.06   | 0.00      | -0.01 | Pass   | 2.40 | 2.50          | 0.00      | -0.10 | Pass   | 2031      | 0       | Pass   |

图 12

注意：拆仪器一定要断电，拔掉电源插头。打拿极倍增管烘干后，转回仪器，一定注意所有的连接线都装好，插稳，各个部件位置要装对。不然就像我一下，装好抽真空后，调谐时莫名其妙的报了错误。而且真空读数还是错了，比实际值低了2个数量级。又放空，重新安装后才可行。