|  | JABATAN KIMIA                              | Kategori         | Arahan Kerja   |
|--|--|------------------|----------------|
|  | PROSEDUR PENGENDALIAN                      | Dokumen No.      | AK/J.K.P/B/01  |
|  | PERALATAN LCMS QQQ BAGI<br>ANALISIS SAMPEL | Tarikh   Semakan | 01.08.2018   1 |
|  |  | Mukasurat:       | 1 dari 20      |

## 1. TUJUAN

Tujuan prosedur ini dtubuhkan adalah untuk memberikan arahan yang jelas bagi langkah atau tatacara bagaimana menjalankan analisis sampel menggunakan peralatan Liquid Chromatography Mass Spectrometer Triple Quadropole

#### 2. SKOP

Terpakai untuk pengguna dari Jabatan Kimia.

#### 3. PROSEDUR ARAHAN

3.1 Ringkasan pengoperasian LC-MSQQQ



|      | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|------|-------------------------|------------------|----------------|
| TINT | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|      | ANALISIS SAMPEL         | Mukasurat:       | 2 dari 20      |

# 3.2 Hidupkan Komputer Perisian

- i. Buka semua peralatan:
- Tekan buka/ tutup butang pada setiap peralatan.
- Lampu pada peralatan itu akan menunjukkan warna `Oren' sebagai tanda didalam keadaan bersedia (seperti gambar rajah di bawah).
- Warna yang berkaitan menunjukkan tanda sebagai:



Gambar rajah 3.2.1 : Binary pump



Gambar rajah 3.2.2 : Sampler



Gambar rajah 3.2.3: Column

ii. Hidupkan komputer(Username: Admin)(Password: 3000hanover)

UV DAD ( If use)



Gambar rajah 3.2.4 : UV DAD

|      | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|------|-------------------------|------------------|----------------|
| TINA | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UNI  | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|      | ANALISIS SAMPEL         | Mukasurat:       | 3 dari 20      |

# 3.3 Penukaran Bahan Pelarut

- Terdapat empat saluran yag digunakan, iaitu A1 A2 B1 B2,
- $\circ$  \* Pelajar hanya dibenarkan menggunakan saluran A1 and B1 sahaja

# Penting:

• A1: Air Tulen (LC-Grade)

\*\*Pertimbangan untuk bahan penambahan campuran ( buffer) :

- Bahan jenis mudah meruap
- Pastikan kepekatan dibawah 10Mm.
- Contoh campuran bahan penambahan:
  - ✓ Ammonium Acetate ( pH 6-7)
  - ✓ Ammonium Formate ( pH 5-6)
  - ✓ Acetic Acid (pH 3.8-5.8)
  - ✓ *Trifluoroacetic Acid* (pH 1-2)
  - ✓ Formic Acid



Gambar rajah 3.3.1 : Saluran A1

\*\* Sekiranya bahan penambah (*Buffer*) bukan meruap perlu digunakan, guna kan bahan penambah dimana anion/ cation nya adalah tidak meruap.

Contoh:

- ✓ Phosphate
- ✓ Sulphates
- ✓ Borates
- o B1 : Bahan pelarut Organik (LC-Grade)
  - ✓ Methanol
  - ✓ Ethanol
  - ✓ Propanol
  - ✓ Isopropanol
  - ✓ Butanol
  - ✓ Acetonitrile
  - ✓ DMSO \* At lower solvent percentages (`10% or less)
  - ✓ Acetic Acid
  - ✓ Acetone



Gambar rajah 3.3.2 : Saluran B1

|      | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|------|-------------------------|------------------|----------------|
| TINA | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|      | ANALISIS SAMPEL         | Mukasurat:       | 4 dari 20      |

## 3.4 Pemasangan Column



Gambar rajah 3.4.1 : Column



Gambar rajah 3.4.2: Column Compartment

 Pastikan kedudukan yang betul sebelum memasang *column*

- Gunakan tangan untuk mengetatkan column dibahagian penyambung plastik.
- Gunakan alatan yang disediakan untuk mengetatkan *Column* dibahagian penyambung besi.

Alatan yang disediakan:
 Untuk ketatkan – mengikut arah jam
 Untuk longgarkan- mengikut lawan arah jam



Gambar rajah 3.4.3 : Alatan yang disediakan

|        | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|--------|-------------------------|------------------|----------------|
| T IN / | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI   | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|        | ANALISIS SAMPEL         | Mukasurat:       | 5 dari 20      |

# 3.5 Perisian (Data Aquisition)

o Klik `Data Acquisition' icon



• Paparan seperti di bawah akan dipaparkan



#### 3.6 Pembersihan sistem dan column

- \* Penting untuk menjaga ketahanan column dan juga sistem instrumentasi
- ✓ Klik kanan di *Binary Pump* kekunci
- ✓ Pilih `*Control*' → tetapan untuk masa, komposisi, dan pengeluaran pelarut → Ok
   \* Digalakkan untuk menggunakan masa yang panjang bagi tujuan pembersihan column
- ✓ Klik kanan di Binary Pump tab
- ✓ Pilih `*Purge On'* \*Periksa column untuk memastikan tiada kebocoran pelarut



|    | JABATAN KIMIA                                    | Kategori         | Arahan Kerja   |
|----|--|------------------|----------------|
| UM | PROSEDUR PENGENDALIAN<br>PERALATAN LCMS QQQ BAGI | Dokumen No.      | AK/J.K.P/B/01  |
|    |  | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL                                  | Mukasurat:       | 6 dari 20      |

# 3.7 Merekod prosedure *Qualitative Analysis*:

*Method editor* → *Worklist/ sample* → *Data Processing* 

a) *HIP Sampler* (rujuk gambar rajah di bawah)

| Agilent MassHu<br>File View Si | inter Workstation Data Acquisition<br>ample Worklist Method T                | ools Help             |                     |               |               |           |                       |                        |   |                                |  | _ 0 X            |
|--------------------------------|--|-----------------------|---------------------|---------------|---------------|-----------|-----------------------|------------------------|---|--------------------------------|--|------------------|
| Context: Acquisi               | tion V Lavout: Default/c   | ve) het 💌 🚺 🗸         |                     | Method: svik  | in flushing m |           |                       |                        | ▼ Worklist:                               |                                |  | • 📾              |
| For Hele Dever Cl              | Light Cayour Delaur(s  | yshiye 🕐              |                     |               | in_nushingin  |           |                       |                        |   | Ne westfirst leaded   DAMaadd  | unted Anibia Bushine and DA Free second    | - 362.1 CR   NUM |
| For Help, Press F1             |  |                       |                     |               |               |           |                       |                        |   | No worklist loaded. D:\IviassH | unter\\syikin_tiusning.m   D:\- Free space | 202.1 GB   NUM   |
| ; Instrument Status            |  |                       |                     |               |               |           |                       | ×                      | ; Actuals                                 |                                |  | x                |
| 🧼 HiP                          | Sampler 🛛 ? 🗕 🗐  | 🍐 Binary              | / Pump 🛛 ? 🗕 🔳      | 🥒 🛛 Column Co | omp. ? 🗕 🗖    |           | QQQ ?                 | _ =                    | Parameter                                 | Value                          |  |                  |
|                                |  |                       |                     |               | T.U.          | 0.0       |                       |                        | QQQ: Instrument State                     | background_acquisition         |  |                  |
|                                |  |                       | NOT READY           |               | Idle          | $\odot$   | Not Ready             | _                      | QQQ: High Vac                             | 2.44E+0 100<br>4.46E-5 Torr    |  |                  |
| 4                              |  |                       |                     |               | 0             |           | ŀ                     |                        | QQQ: Sheath Gas Temp                      | 298 °C                         |  |                  |
|                                |  | A2 B2                 | 2                   |               | 4             | Ū         | AJS ESI               |                        | QQQ: Collision Gas                        | on                             |  |                  |
|                                | 5 Aul 0  |                       |                     |               | _             |           |                       |                        | QQQ: Error State                          | no_error                       |  |                  |
|                                | 3.0µL  | 0.00 100.             | .00                 |               |               |           |                       |                        | QQQ: MS1 Heater                           | 100 °C                         |  |                  |
|                                |  |                       | 5 000 ml /min       |               |               |           | J                     |                        | QQQ: MS 2 Heater                          | 100 °C                         |  |                  |
|                                |  |                       |                     |               |               |           | <b>1</b> 00           |                        | QQQ: Turbo 1 Speed                        | 100.0 %                        |  |                  |
|                                | <u></u>  |                       |                     |               | ufu           |           | CC                    |                        | QQQ: Turbo 2 Speed                        | 100.0 %                        |  |                  |
| Temperat                       | ture 19°C  |                       |                     | 19.85 °C      | 18.08 °C      |           |                       |                        | QQQ. Sriedin Gas Flow                     | 11.2 vmin                      |  |                  |
| (                              | 0.00 / 0.00  |                       |                     |               |               | Ins<br>No | trument               | 🜔 😑 On 🔞 Of            | f   |                                |  |                  |
| Chromatogram P                 | lot  |                       |                     |               |               | ,         | Spectrum Plot         |                        |   |                                |  | x                |
|                                |  |                       |                     |               |               |           | QQQ Spectrum MS       | 1: MS2 Scan, AJS I     | ESI (+), 8506.85                          |                                |  |                  |
|                                |  |                       | TIC                 |               |               |           | <sup>50</sup> Area: 0 |                        |   | 550.0                          |  |                  |
| 1E/-                           |  |                       |                     |               |               |           | m/#/40.000            |                        | ***                                       |                                | ,  |                  |
| 8000000                        |  |                       |                     |               |               |           | Height: 46            |                        |   |                                |  |                  |
| 700000                         |  |                       |                     |               |               |           | 2 30 Resolution: 0    |                        |   |                                |  |                  |
| 6000000                        |  |                       |                     |               |               |           | Time: 550.0           |                        |   |                                |  |                  |
| 5000000                        |  |                       |                     |               |               |           | ₽ 20                  |                        |   |                                |  |                  |
| 4000000                        |  |                       |                     |               |               |           |                       |                        |   |                                |  |                  |
| 3000000-                       |  |                       |                     |               |               |           | 10                    |                        |   |                                |  |                  |
| 1000000-                       |  |                       |                     |               |               |           |                       |                        |   |                                |  |                  |
|                                | 8504.5   | 8505                  | 2505.5              | 8508          | 8508.5        | min       | 0 100                 | 200 30                 | 0 400                                     | 500 600 700                    | 800 900                                    | 1000             |
| سعر                            |  |                       |                     |               |               |           | VV                    |                        |   | m/z(amu)                       |  |                  |
| Method Editor                  |  |                       | 2) (111)            | IID complex 7 | Tab           |           |                       |                        |   |                                |  | ×                |
|                                | B gjikin flushing.m  |                       | 2) KIK I            | The sumpler i | ub            |           |                       |                        |   |                                |  |                  |
| Properties D                   | HiP Sampler HiP Sampler Pre  | etreatment Binary Pun | np Column Comp. QQQ |               |               |           |                       |                        |   |                                |  |                  |
|                                | $\smile$   |                       |                     |               |               |           |                       |                        |   |                                | HiP Sampler (G4226A                        |                  |
| and the second second          |  |                       |                     |               |               |           |                       |                        |   |                                |  |                  |
| Injection Mode                 |  |                       | 3) Masukl           | an jumlah     |               |           |                       |                        |   |                                |  |                  |
| Injectio                       | n volume: 5.00 ‡ μL  | ノ                     | inje                | ction         |               |           |                       |                        |   |                                |  | <u>^</u>         |
|                                | C Standard injection   |                       |                     |               |               |           | Draw spe              | ed: 100.0 *            | uL/min                                    |                                |  |                  |
|                                | <ul> <li>Standard nijectron</li> <li>A leiseties with seedle weel</li> </ul> |                       | * Maksim            | um 6.00µl     |               |           |                       |                        |   |                                |  |                  |
|                                | <ul> <li>Injection with needle wasi</li> </ul>                               | n                     |                     | •             |               |           | Eject spe             | ed: 100.0 ,            | µL/min                                    |                                |  |                  |
|                                |  |                       |                     |               |               |           | Draw positi           | on: 0.0 🔅              | mm  |                                |  |                  |
| Needle wash                    |  |                       |                     |               |               |           | Equilibration tir     | me: 2.0 🕻              | sec                                       |                                |  |                  |
| -                              | Mode: Flush Port   | *                     | Advanced            |               |               |           |                       |                        |   |                                |  |                  |
|                                | Time: 20 *   |                       | Autonecu            |               |               |           |                       |                        |   |                                |  |                  |
|                                | nine. 3.0 ,  | 300                   |                     |               |               |           | Canada fluch aut fa   | alar: 50               | · General Standard and Stand              |                                |  |                  |
|                                | Location:  |                       |                     |               |               |           | Sample Ilusti out la  | 0.0                    | <ul> <li>unes injection volume</li> </ul> |                                |  |                  |
|                                | Repeat: 3 *  | times                 |                     |               |               |           |                       | Vial/Vell hott         | om sensing                                |                                |  |                  |
|                                |  |                       | 1                   |               |               |           |                       |                        | un acriaing                               |                                |  |                  |
| Stoptime                       | Posttime   |                       | High throughput     |               |               |           |                       |                        |   |                                |  |                  |
|                                |  |                       |                     |               |               |           |                       |                        |   |                                |  |                  |
| As Pumpl                       | As Pumplificator     Off     Automatic delay volume reduction                |                       |                     |               |               |           |                       |                        |   |                                |  |                  |
| C                              | 1.00 ( min )   | 1.00 ° m              | n 1) Klik /         | Method Edito  | or Tab        |           |                       |                        |   |                                |  |                  |
|                                |  |                       | ·                   |               |               |           |                       | nable overlanned inion | tion                                      |                                |  |                  |
|                                |  |                       | 7                   |               |               |           |                       | anabio ovenapped hijeu | uvii                                      |                                |  |                  |
| Me                             | ethod Edi  | tor \                 | Norklist            | Sam           | ole Ru        | ın l      |                       |                        |   |                                |  |                  |

Gambar rajah 3.7.1 : Paparan untuk penetapan prosedur (Hip Sampler)

Tetapan lain tidak boleh ditukar selain mendapat kebenaran dari kakitangan yang bertanggungjawab.

|      | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|------|-------------------------|------------------|----------------|
| TINA | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|      | ANALISIS SAMPEL         | Mukasurat:       | 7 dari 20      |

b) Binary Pump



Gambar rajah 3.7.2 : Paparan untuk penetapan prosedur (Binary Pump)

|      | JABATAN KIMIA                              | Kategori         | Arahan Kerja   |
|------|--|------------------|----------------|
| ТІЛЛ | PROSEDUR PENGENDALIAN                      | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI | PERALATAN LCMS QQQ BAGI<br>ANALISIS SAMPEL | Tarikh   Semakan | 01.08.2018   1 |
|      |  | Mukasurat:       | 8 dari 20      |

c) QQQ (Mass Spectrometer)



Gambar rajah 3.7.3 : Paparan untuk penetapan prosedur (MS QQQ)

|   |  | JA   | BATAN KIMIA   |   | Kategori   | Arahan Kerja  |
|---|--|--|---|---|--|---|
| т   | TN /T  | PROSEI   | DUR PENGENDA  | LIAN  | Dokumen No.  | AK/J.K.P/B/01   |
|   | JIVI   | PERALA   | TAN LCMS QQQ  | BAGI  | Tarikh   Semakan   | 01.08.2018   1  |
|   |  | AN   | ALISIS SAMPEL   | ı   | Mukasurat:   | 9 dari 20   |
| 3.8 M   | enyimpan<br>°  | <b>prosedure</b><br>Setelah semua                                  | a maklumat diması   | ukan, untuk   | menyimpan prosedur   | e/method yang   |
|   | K  | telah di edit :<br>lik ` <i>Save As' —</i>                         | → Cipta satu dokı   | umen <i>eg</i> : (D   | 0rAzman_iman)>   | ok  |
|   | 0  | Untuk Membı  | ika dokumen   |   |  |   |
|   | К  | lik `Open folder'  | > Cari  | Fail —>   | ok   |   |
| Agilent MassHunter Workstation Data A<br>File View Sample Worklist Me   | Acquisition<br>thod Tools Help                               |  |   |   |  |   |
| Context: Acquisition   Layout: For Help Press F1                        | Default(sys).lyt 🔹 🔚   |  | ethod: syikin_flushing.m  |   | Worklist:  | Inaded Dr\MassHunter\_\svikin flushing m Dr\- Free snare: 2621 GB NI  |
| Instrument Status   |  |  |   |   | X Actuals  | House   |
| C C C C C C C C C C C C C C C C C C C                                   |  | Idle     Idle       0.00     0.100 mL/min       7.68 bar     19.98 | Comp. 2 ↓ 1<br>Comp. 2 ↓ 1<br>Comp | Idle<br>AJS ESI   | QQ2         Instrument State         backgroup           QQ2         Rough Vac         2.42E40           QQ2         High Vac         4.44E55           QQ2         Sheaht Gas Temp         300 °C           QQ2         Collision Gas         on           QQ2         Collision Gas         on           QQ2         Collision Gas         on           QQ2         State         no_error           QQ2         MS1 Heater         100 °C           QQ2         Tubol Speed         1000 %           QQ2         Speed         1000 %           QQ2         Speed         100 %           QQ2         Sheath Gas Flow         11 10 limit | nd_soquisition<br>Torr<br>Forr  |
| Chromatogram Plot   |  |  | ×   | Idle Spectrum Plot  |  |   |
| 167<br>900000<br>900000<br>7000000<br>200000<br>200000                  |  |  |   | OQO Spectrum MS 11:MS2<br>Area. 0<br>m/ <del>AFAR,R1 7 14 40</del><br>40 FVHM: 900.0<br>Height: 46<br>9 30 Resolution: 0<br>Time: 550.0<br>9 20 | Scan, AJS ESI (+), 8512.69<br>550 0<br>N. H.   | <b>111-14 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177 - 177</b> - <b>17</b> |
| 4000000 -<br>3000000 -<br>2000000 -                                     |  | Kekunci  |   | 10  |  |   |
| 8510  | 8510.5   | Simpanan   | 8512 8512.5 min   | 0<br>100 200  | 300 400 500 6<br>m/z(amu)  | 00 700 800 900 1000   |
| Method Editor   |  |  |   |   |  |   |
| Properties DA HiP Sampler HiP S   | n<br>Sampler Pretreatment Binary I                           |  | Buka Kekunci Fai<br>Simpanan  |   |  | Column Comp. (G1316C)   |
| Temperature<br>Left<br>Not Controlled<br>200 1 10<br>C As Detector Cell | Right:<br>Not Controlled<br>C 200 [ *C<br>C As Detector Cell | Advanced   | ☑ when front door open<br>Left:<br>☑ With any temperature<br>☑ When temperature is within   |   | C 10   | Flight<br>(th any temperature<br>then temperature is within   |

Gambar rajah 3.8.1 : Paparan untuk penyimpanan prosedure dan pembukaan fail simpanan

Timetable (empty)

|         | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|---------|-------------------------|------------------|----------------|
| T IN /T | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|         | ANALISIS SAMPEL         | Mukasurat:       | 10 dari 20     |

#### 3.9 Mencipta/ Menyimpan rekod analisa sampel

#### a) Untuk bilangan sampel yang banyak

 $\circ$  Method editor  $\rightarrow$  Worklist/sample run  $\rightarrow$  Data Processing

\* Sekiranya bilangan sample banyak, pelajar boleh menggunakan `Worklist'



Gambar rajah 3.9.1 : Paparan untuk penyediaan worklist

|      | JABATAN KIMIA                              | Kategori         | Arahan Kerja   |
|------|--|------------------|----------------|
| TINA | PROSEDUR PENGENDALIAN                      | Dokumen No.      | AK/J.K.P/B/01  |
| UIVI | PERALATAN LCMS QQQ BAGI<br>ANALISIS SAMPEL | Tarikh   Semakan | 01.08.2018   1 |
|      |  | Mukasurat:       | 11 dari 20     |

o Penyimpanan Worklist: Setelah maklumat diisi,

Klik *Save As*  $\longrightarrow$  cipta fail *Cth* : (DrAzman\_iman worklist)  $\longrightarrow$  ok

• Membuka fail:

Kllik `*Open Worklist'* —> Cari fail —> ok



Gambar rajah 3.9.2 : Paparan untuk penyimpanan dan buka fail simpanan bagi sampel banyak.

| UM | JABATAN KIMIA           | Kategori                  | Arahan Kerja   |
|----|-------------------------|---------------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No. AK/J.K.P/B/01 |                |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan          | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:                | 12 dari 20     |

## b) Analisa untuk satu sampel sahaja



Gambar rajah 3.9.3 :Paparan untuk penyimpanan data bagi satu sampel

| UM | JABATAN KIMIA Kategori  |                    | Arahan Kerja   |
|----|-------------------------|--------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.        | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan   | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat: 13 dari | 13 dari 20     |

#### 3.10 Proses Analisis Sampel

Setelah semua maklumat prosedure dan juga senarai sampel telah dibuat, sila semak semua maklumat yang telah dimasukkan dari (3.3 – 3.12) sebelum menekan butang mula.

Tunggu sehingga semua peralatan instrument dalam keadaan bersedia dimana warna 'Hijau' akan ditunjukkan atau dalam keadaan *Idle*, sila rujuk gambar rajah



Gambar rajah 3.10.1 : ikon untuk mula dan berhenti

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja                    |
|----|-------------------------|------------------|---------------------------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01                   |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | AK/J.K.P/B/01<br>01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 14 dari 20                      |

# 3.11 Analisis Data

3.11.1 Memproses data, sila rujuk gambar rajah



Gambar rajah 3.11.1

i. Membuka fail data (sila rujuk gambar rajah di bawah)

|                                     | gilent MassHunte   | r Quantative   | e Analysis E | 1.04.00 - a | azeana10april.m  |
|-------------------------------------|--|----------------|--------------|-------------|--|
| File<br>7                           | Edit View Find I   | (dentify Chror | matograms S  | pectra Meth | hod Wizards Actions Configuration Tools Help   |
| 12                                  | Open Data File 🗙   | Ctrl+O         | 89.          | ୯ -  ା 🌚 🔤  | 🎦 🖸 🔼 🏛 💾 二 🖄 🕼 🏙 🏨 🚇 🕼 🖉 🖄 🎟 🛆 📲  |
| 慶                                   | Refresh Data File  | 12             |              | ×           | Chromatogram Results   |
|                                     | Save Results   | Cth            |              | *           | 🛛 🕶 🛊 । 🔍 🗄 🚧 । ४४ 🕂 🔥 👁 । 🗩 🕑 🕑 🔹 🖃 🔛 🔝 🗛 🖄 🗞 🖏 🎇 👔                                 |
|                                     | Close Data File  |                |              |             |  |
|                                     | Close All  |                | A            |             |  |
| ð                                   | Print  | •              |              | Clik        | K File and Open Data File  |
|                                     | Export   | Þ              |              |             |  |
|                                     | Exit   |                |              |             |  |
|                                     |  |                |              |             | IIL MS Spectrum Results  |
|                                     |  |                |              |             | ।  |
| : <b>B</b>                          | Method Explorer: a   | azeana10april  | m            | ×           | ≝ ┹ ┿ ‡ ! Q Œ ৠ थ <mark>☆</mark> ॒ ▲ ! O C = 6 ¥ <u>1</u> ₩ ⊮ 品 <b>%</b> % % 除 ! ⊯ ! |
| •                                   | Method Explorer: a   | azeana10april  | m            | ×           | ▼  |
|                                     | Method Explorer: a<br>Chromatogram<br>Spectrum                         | azeana10april  | m            | ×           | ▼  |
| : : :<br>• : :<br>Extra             | Method Explorer: a<br>Chromatogram<br>Spectrum<br>act (MS)             | azeana10april. | m            | ×           |  |
| ÷ €<br>• C<br>• S<br>Extr.<br>Extr. | Method Explorer: a<br>Chromatogram<br>Spectrum<br>act (MS)<br>act (MS) | azeana10april. | m            | ×           | ▼  |

Gambar rajah 3.11.2

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|----|-------------------------|------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 15 dari 20     |

ii. Clik data :contoh LC6006.d untuk dapatkan Chromatogram.

| 🔛 Agilent MassHunter Qualitative Analysis B.04.00      | azeana10april.m                   |   |                         |       |
|--|-----------------------------------|---|-------------------------|-------|
| File Edit View Find Identify Chromatograms Spectra Met | hod Wizards Actions Configuration | Tools Help                              |                         |       |
| : 🖻 😂 🔲 🗀 🗇 🖓 - 🗹 👿 🖾 🔊 - (? - ) 🧬                     | لـ 🕂 🏨 🄬 🔊 🐔 💒                    | 🗠 🐼 🛍 🏨 🎧 🕼 🔊 📈                         | H⊞ & A                  |       |
| 🐕 Data Navigator 🛛 🗙 🗙                                 | A Chromatogram Results            |   |                         |       |
| Sort by Data File 👻                                    | Z ↔ ‡ Q ∓ ₩ V                     | A 9 C 8 - H X                           | 🗛 🎊 🔎 🔭 % 🍡 🖄 🐱 Minutes | - 3   |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   |   |                         |       |
|  |                                   | III Open Data File                      |                         |       |
|  | MS Spectrum Results               |   |                         |       |
|  | Z ↔ \$ Q T \$ 2                   | Look in: 🛅 Data LC6                     | 6001 · LC6234 🔄 💽 🚺 📑 🥅 |       |
|  |                                   | MH LC6001.d                             | MH I                    | LCG   |
|  |                                   | MH LC6002.d                             | MH I                    | 106   |
|  |                                   | Documents MH LC6004.d                   | IN I                    | LC6   |
|  |                                   | MH LC6005.d                             | Gen :                   |       |
| Method Explorer: azeana10april.m ×                     |                                   | MH LLSUUSId<br>MH LC6007.d              |                         | 06 4  |
| Chromatogram   |                                   | Documents MH LC6008.d                   |                         | 00 u. |
| = Spectrum   |                                   | MH LC6009.d                             |                         |       |
| Extract (MS)   |                                   | Desktop                                 |                         | LC6   |
| Extract (MS/MS)  |                                   | LC6012 exported                         | d data 🗸 MH             | LC6   |
| Extract (UV)   |                                   | СССО13.d                                | (PH)                    | LCG   |
| Deconvolute: Resolved Isotope                          |                                   | My Computer MH LC6014.d                 | MH I                    | LC6   |
| Extraction Data Format                                 |                                   |   |                         | 2     |
| + General  |                                   | My Network File name: LC60              | UU6.d Open              |       |
| + Reports  |                                   | Places Files of type: Data              | a Files (".d) Cancel    | _     |
| Find Compounds   |                                   | Ontions                                 | Help                    |       |
| Find Compounds by Formula                              |                                   | C Load worklist method                  | Sample Information      | _     |
| Identify Compounds                                     |                                   | <ul> <li>Load results method</li> </ul> | Sample Name : P1        |       |

Gambar rajah 3.11.3

iii. Untuk mendapatkan Chromatogram yang kemas.

| 🚟 Agilent MassHunter Qualitative Analysis B.04.00  | 🛱 Agilent MassHunter Qualitative Analysis B.04.00 - azeana10april.m  |          |   |   |  |  |  |
|--|--|----------|---|---|--|--|--|
| : File Edit View Find Identify Chromatograms Spectra Method Wizards Actions Configuration Tools Help |  |          |   |   |  |  |  |
| 🛯 🖉 📓 🖿 🎒 🖓 • 🗹 💟 🖾 🔊 • 🕅 •  |  |          |   |   |  |  |  |
| 🏠 Data Navigator 🛛 🗧   | 🗄 🛆 Chromatogram Results   |          |   |   |  |  |  |
| Sort by Data File  ✓ US006.d  ✓ User Chromatograms  ✓ V ter Chromatograms  ✓ V ter Chromatograms     | 4  | -        | 🛏 🔟 🗘 🏄 🖉 🧞 % 🇞 🕅                             | 🧯 Minutes 📼 🎿                                   |  |  |  |
| Background Spectra     Compounds     Matched Sequences   | 0.9-<br>0.8-<br>0.7-   |          |   |   |  |  |  |
|  |  |          | Extract MS Spectrum                           |   |  |  |  |
| Clik pada Chromatogram un  | tuk  |          | Extract MS Spectrum to Background             |   |  |  |  |
|  |  |          | Extract UV Spectrum                           | W   |  |  |  |
| "smooth Chromatogram"  |  |          | Extract Peak Spectrum                         |   |  |  |  |
| C C  | man provide a second se | -        | Extract MS Peak Spectrum from UV Peaks        |   |  |  |  |
|  | -0.1-  |          | Extract Chromatograms                         | de de de de                                     |  |  |  |
|  | 1 2 3 4 5  |          | Extract Defined Chromatograms                 | 12 13 14 15 16<br>%) vs. Acquisition Time (min) |  |  |  |
|  | Spectrum Preview   |          | ose Highlighted Chromatograms                 |   |  |  |  |
|  |  | Q        | Integrate Chromatogram                        |   |  |  |  |
|  |  | -        | Integrate and Extract Peak Spectra            |   |  |  |  |
|  |  |          | Subtract Apy Chromatogram                     |   |  |  |  |
|  |  |          | Calculate Signal-to-Noise                     |   |  |  |  |
|  |  | Δ.       | Integration Peak List                         | -   |  |  |  |
| Method Explorer: azeana10apil.m  |  |          | Adjust Peak Threshold                         |   |  |  |  |
| Chromatogram   |  | v        | Set Apphor                                    | -   |  |  |  |
| Spectrum   |  | -        | Clear Anchor                                  |   |  |  |  |
| Extract (MS)   |  |          | Assign Ranges to                              |   |  |  |  |
| Extract (MS/MS)  |  |          | Copy to User Chromatograms                    |   |  |  |  |
| Extract (UV)   |  | ×        | Clear Results                                 |   |  |  |  |
| Deconvolute: Resolved Isotope  |  | ×        | Delete  |   |  |  |  |
|  |  |          |   |   |  |  |  |
| Extraction Data Format   |  | $\times$ | Delete Peak                                   |   |  |  |  |
| Extraction Data Format   General   | LIL MS Spectrum Results  | ×<br>Q   | Delete Peak<br>                               |   |  |  |  |
| Extraction Data Format  General  Reports   | <u>   </u> MS Spectrum Results<br> : ♪ ↔ ↓ ○ ① 秋 ♡ ○ 6   | ×<br>Q   | Delete Peak<br>Unzoom<br>Assign Random Colors | 3   |  |  |  |

Gambar rajah 3.11.4

iv. Untuk mendapatkan Extract MS Spectrum "highlight peak" yang berkenaan.



Gambar rajah 3.11.5



## v. Keputusan.

Gambar rajah 3.11.6

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|----|-------------------------|------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 17 dari 20     |

# vi. Untuk "Extract MS Spectrum to background"



## Gambar rajah 3.11.7

vii. Keputusan.



Gambar rajah 3.11.8

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|----|-------------------------|------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 18 dari 20     |

viii. Untuk mendapatkan Spectrum asal tanpa spectrum Background.



Gambar rajah 3.11.9

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|----|-------------------------|------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 19 dari 20     |

# 3.11.2 Mencetak Data

i. Rujuk gambar rajah di bawah



Gambar rajah 3.11.2.1



Gambar rajah 3.11.2.2

| UM | JABATAN KIMIA           | Kategori         | Arahan Kerja   |
|----|-------------------------|------------------|----------------|
|    | PROSEDUR PENGENDALIAN   | Dokumen No.      | AK/J.K.P/B/01  |
|    | PERALATAN LCMS QQQ BAGI | Tarikh   Semakan | 01.08.2018   1 |
|    | ANALISIS SAMPEL         | Mukasurat:       | 20 dari 20     |

| SEMAKAN | DISEDIAKAN  | DISEMAK           | TARIKH    | CATATAN          |
|---------|---|-------------------|-----------|------------------|
| 1       | Siti Nur Faridatul<br>Asyikin Binti Said<br>Nor Lela Binti Md Ali<br>Mohamad Akasah | Norzalida Zakaria | 1.08.2018 | Isu kali pertama |