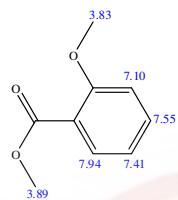
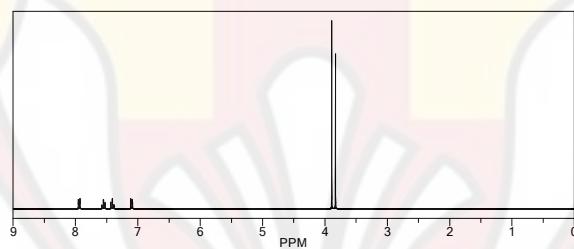


**LAMPIRAN A**  
**ESTIMASI SPEKTRUM RMI-<sup>1</sup>H DIMETIL SALISILAT**



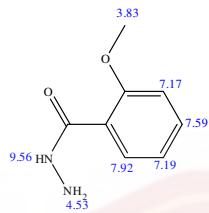
Estimation quality is indicated by color: **good**, **medium**, **rough**



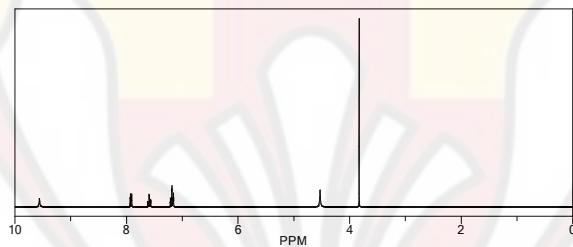
Protocol of the H-1 NMR Prediction:

| Node     | Shift | Base + Inc.                   | Comment (ppm rel. to TMS) |
|----------|-------|-------------------------------|---------------------------|
| CH 7,10  | 7,26  | 1-benzene                     |                           |
|          | -0,49 | 1 -O-C                        |                           |
|          | 0,11  | 1 -C(=O)OC                    |                           |
|          | 0,22  | general corrections           |                           |
| CH 7,94  | 7,26  | 1-benzene                     |                           |
|          | -0,11 | 1 -O-C                        |                           |
|          | 0,71  | 1 -C(=O)OC                    |                           |
|          | 0,08  | general corrections           |                           |
| CH 7,55  | 7,26  | 1-benzene                     |                           |
|          | -0,11 | 1 -O-C                        |                           |
|          | 0,21  | 1 -C(=O)OC                    |                           |
|          | 0,19  | general corrections           |                           |
| CH 7,41  | 7,26  | 1-benzene                     |                           |
|          | -0,44 | 1 -O-C                        |                           |
|          | 0,11  | 1 -C(=O)OC                    |                           |
|          | 0,48  | general corrections           |                           |
| CH3 3,83 | 0,86  | methyl                        |                           |
|          | 2,87  | 1 alpha -O-1:C*C*C*C*C*1      |                           |
|          | 0,10  | general corrections           |                           |
|          | 0,01  |                               |                           |
| CH3 3,89 | 0,86  | methyl                        |                           |
|          | 3,02  | 1 alpha -OC(=O)-1:C*C*C*C*C*1 |                           |
|          | 0,01  | general corrections           |                           |

**LAMPIRAN B**  
**ESTIMASI SPEKTRUM RMI-<sup>1</sup>H 2-METOKSIBENZO HIDRAZIDA**



Estimation quality is indicated by color: good, medium, rough

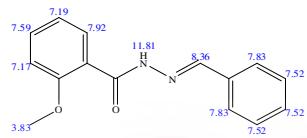


Protocol of the H-1 NMR Prediction:

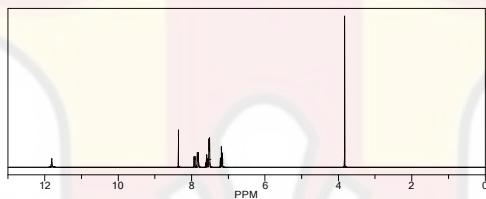
| Node     | Shift | Base + Inc.              | Comment (ppm rel. to TMS) |
|----------|-------|--------------------------|---------------------------|
| NH 9,56  | 8,00  | sec. amide               |                           |
|          | 1,56  | general corrections      |                           |
| NH2 4,53 | 2,00  | amine                    |                           |
|          | 2,53  | general corrections      |                           |
| CH 7,17  | 7,26  | 1-benzene                |                           |
|          | -0,49 | 1 -O-C                   |                           |
|          | 0,18  | 1 -C(=O)N                |                           |
|          | 0,22  | general corrections      |                           |
| CH 7,92  | 7,26  | 1-benzene                |                           |
|          | -0,11 | 1 -O-C                   |                           |
|          | 0,69  | 1 -C(=O)N                |                           |
|          | 0,08  | general corrections      |                           |
| CH 7,59  | 7,26  | 1-benzene                |                           |
|          | -0,11 | 1 -O-C                   |                           |
|          | 0,25  | 1 -C(=O)N                |                           |
|          | 0,19  | general corrections      |                           |
| CH 7,19  | 7,26  | 1-benzene                |                           |
|          | -0,44 | 1 -O-C                   |                           |
|          | 0,18  | 1 -C(=O)N                |                           |
|          | 0,19  | general corrections      |                           |
| CH3 3,83 | 0,86  | methyl                   |                           |
|          | 2,87  | 1 alpha -O-1:C*C*C*C*C*1 |                           |
|          | 0,10  | general corrections      |                           |

## LAMPIRAN C

### ESTIMASI SPEKTRUM RMI-<sup>1</sup>H N'-BENZILIDEN-2-METOKSIBENZO HIDRAZIDA



Estimation quality is indicated by color: good, medium, rough

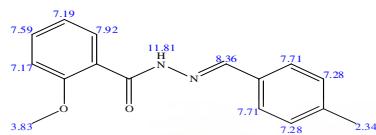


Protocol of the H-1 NMR Prediction:

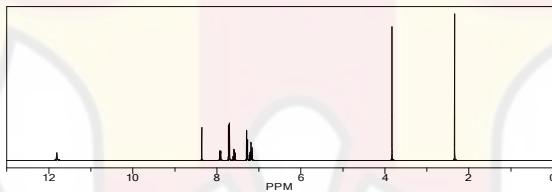
| Node     | Shift | Base + Inc.              | Comment (ppm rel. to TMS) |
|----------|-------|--------------------------|---------------------------|
| NH 11.81 | 8.00  | sec. amide               |                           |
|          | 3.81  | general corrections      |                           |
| CH 7.17  | 7.26  | 1-benzene                |                           |
|          | -0.49 | 1-O-C                    |                           |
|          | 0.18  | 1-C(=O)N                 |                           |
|          | 0.22  | general corrections      |                           |
| CH 7.92  | 7.26  | 1-benzene                |                           |
|          | -0.11 | 1-O-C                    |                           |
|          | 0.69  | 1-C(=O)N                 |                           |
|          | 0.08  | general corrections      |                           |
| CH 7.83  | 7.62  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.21  | general corrections      |                           |
| CH 7.83  | 7.62  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.21  | general corrections      |                           |
| CH 7.52  | 7.29  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.23  | general corrections      |                           |
| CH 7.59  | 7.26  | 1-benzene                |                           |
|          | -0.11 | 1-O-C                    |                           |
|          | 0.25  | 1-C(=O)N                 |                           |
|          | 0.19  | general corrections      |                           |
| CH 7.19  | 7.26  | 1-benzene                |                           |
|          | -0.44 | 1-O-C                    |                           |
|          | 0.18  | 1-C(=O)N                 |                           |
|          | 0.19  | general corrections      |                           |
| CH 7.52  | 7.29  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.23  | general corrections      |                           |
| CH 7.52  | 7.29  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.23  | general corrections      |                           |
| CH3 3.83 | 0.86  | methyl                   |                           |
|          | 2.87  | 1 alpha-O-1:C*C*C*C*C*1  |                           |
|          | 0.10  | general corrections      |                           |
| CH 8.36  | 8.11  | benzylidenimin           |                           |
|          | ?     | 1 unknown substituent(s) |                           |
|          | 0.25  | general corrections      |                           |

## LAMPIRAN D

### ESTIMASI SPEKTRUM RMI $^1\text{H}$ N'-{(4-METILBENZILIDEN)-2-METOKSIBENZO}HIDRAZIDA



Estimation quality is indicated by color: good, medium, rough



Protocol of the H-1 NMR Prediction:

| Node     | Shift | Base + Inc.               | Comment (ppm rel. to TMS) |
|----------|-------|---------------------------|---------------------------|
| NH 11.81 | 8.00  | sec. amide                |                           |
|          | 3.81  | general corrections       |                           |
| CH 7.17  | 7.26  | 1-benzene                 |                           |
|          | -0.49 | 1 -O-C                    |                           |
|          | 0.18  | 1 -C(=O)N                 |                           |
|          | 0.22  | general corrections       |                           |
| CH 7.92  | 7.26  | 1-benzene                 |                           |
|          | -0.11 | 1 -O-C                    |                           |
|          | 0.69  | 1 -C(=O)N                 |                           |
|          | 0.08  | general corrections       |                           |
| CH 7.71  | 7.62  | benzylidenimin            |                           |
|          | ?     | 1 unknown substituent(s)  |                           |
|          | -0.12 | 1 -C from 1-benzene       |                           |
|          | 0.21  | general corrections       |                           |
| CH 7.28  | 7.29  | benzylidenimin            |                           |
|          | ?     | 1 unknown substituent(s)  |                           |
|          | -0.20 | 1 -C from 1-benzene       |                           |
|          | 0.19  | general corrections       |                           |
| CH 7.71  | 7.62  | benzylidenimin            |                           |
|          | ?     | 1 unknown substituent(s)  |                           |
|          | -0.12 | 1 -C from 1-benzene       |                           |
|          | 0.21  | general corrections       |                           |
| CH 7.28  | 7.29  | benzylidenimin            |                           |
|          | ?     | 1 unknown substituent(s)  |                           |
|          | -0.20 | 1 -C from 1-benzene       |                           |
|          | 0.19  | general corrections       |                           |
| CH 7.59  | 7.26  | 1-benzene                 |                           |
|          | -0.1  | 1 -O-C                    |                           |
|          | 0.25  | 1 -C(=O)N                 |                           |
|          | 0.19  | general corrections       |                           |
| CH 7.19  | 7.26  | 1-benzene                 |                           |
|          | -0.44 | 1 -O-C                    |                           |
|          | 0.18  | 1 -C(=O)N                 |                           |
|          | 0.19  | general corrections       |                           |
| CH3 3.83 | 0.86  | methyl                    |                           |
|          | 2.87  | 1 alpha -O- 1:C*C*C*C*C*1 |                           |
|          | 0.10  | general corrections       |                           |
| CH 8.36  | 8.11  | benzylidenimin            |                           |
|          | ?     | 1 unknown substituent(s)  |                           |
|          | 0.25  | general corrections       |                           |
| CH3 2.34 | 0.86  | methyl                    |                           |
|          | 1.49  | 1 alpha -1:C*C*C*C*C*1    |                           |
|          | -0.01 | general corrections       |                           |

## LAMPIRAN E

### CONTOH PERHITUNGAN PERSENTASE HASIL SINTESIS

#### I. Perhitungan berat teoritis

a. Asam salisilat (BM : 138,134 g/mol)

Penimbangan : 11,04 gram

mol                  asam                  salisilat                  :

$$\frac{11,04 \text{ gram}}{138,134} = 0,079 \text{ mol} = 0,08 \text{ mol}$$

b. Dimetil sulfat (BM : 126,19 g/mol, berat jenis : 1,33 g/cm<sup>3</sup>)

Volume : 20 ml

$$\text{mol dimetil sulfat} : \frac{20ml \times 1,33}{126,19} = 0,2 \text{ mol}$$

c. Hidrazin (BM : 32 g/mol, berat jenis : 1,03 g/cm<sup>3</sup>)

Volume : 0,64 ml = 1ml

$$\text{mol hidrazin} : \frac{0,64ml \times 1,03}{32} = 0,02 \text{ mol}$$

d. Benzaldehida (BM : 106,12 g/mol, berat jenis : 1,05 g/cm<sup>3</sup>)

Volume : 2,03 ml

$$\text{mol benzaldehida} : \frac{2,03 \times 1,05}{106,12} = 0,02 \text{ mol}$$

II. Perhitungan persentase hasil sintesis berdasarkan mmol teoritis

Persentase hasil : metil-2-metoksibenzoat

|                                 |               |                        |               |
|---------------------------------|---------------|------------------------|---------------|
| asam salisilat + dimetil sulfat | $\rightarrow$ | metil-2-metoksibenzoat | + asam sulfat |
| awal                            | 0,08 mol      | 0,2 mol                | 0             |
| reaksi                          | 0,08 mol      | 0,08 mol               | 0,08 mol      |
| sisa                            | 0             | 0,12 mol               | 0,08 mol      |

---

$$\text{BM teoritis} = 166 \text{ g/mol}$$

$$\text{Massa teoritis} = 0,08 \text{ mol} \times 166 \text{ g/mol} = 13,28 \text{ gram}$$

$$\text{Massa praktis} = 12,04 \text{ gram}$$

$$\% \text{ hasil} = \frac{12,04}{13,28} \times 100\% = 90,67\% = 91\%$$

## LAMPIRAN F

### UJI DENGAN FeCl<sub>3</sub> PADA SENYAWA HASIL SINTESIS

Uji dengan FeCl<sub>3</sub> berguna untuk mengetahui apakah gugus OH fenolik masih terdapat dalam struktur senyawa hasil sintesis. Uji ini dilakukan dengan melarutkan sejumlah zat dengan etanol kemudian diteteskan FeCl<sub>3</sub>. Bila larutan berubah warna menjadi ungu/biru tua, maka senyawa tersebut memiliki gugus OH fenolik pada strukturnya. Hasil uji dengan FeCl<sub>3</sub> senyawa hasil sintesis dapat dilihat pada tabel di bawah ini.

Tabel Identifikasi Gugus OH Fenolik dengan FeCl<sub>3</sub>

| Senyawa        | Hasil uji dengan FeCl <sub>3</sub> | Gugus OH fenolik |
|----------------|------------------------------------|------------------|
| Asam salisilat | Ungu                               | +                |
| Senyawa 1      | Kuning                             | -                |
| Senyawa 2      | Kuning                             | -                |
| Senyawa 3a     | Kuning                             | -                |
| Senyawa 3b     | Kuning                             | -                |

Keterangan:

+ : terbentuk warna ungu tua.

- : tidak terbentuk warna ungu

## LAMPIRAN G

### KESEMPURNAAN HASIL SINTESIS TURUNAN N'-BENZILIDEN-2-METOKSIBENZO HIDRAZIDA

Pada reaksi tahap ketiga, waktu yang dibutuhkan untuk mendapatkan hasil yang sempurna dengan pemanasan menggunakan *microwave* dan daya yang digunakan 240 watt, yaitu tertera pada tabel dibawah ini.

Tabel Persentase Hasil Sintesis N'benziliden-2-metoksibenzohidrazida pada Waktu Pemanasan yang Berbeda

| Daya     | Waktu Pemanasan | Persentase Hasil |
|----------|-----------------|------------------|
| 160 watt | 5 x 2 menit     | 54%              |
| 240 watt | 1 x 2 menit     | 70%              |
| 240 watt | 2 x 2 menit     | 74%              |
| 240 watt | 3 x 2 menit     | 74%              |

Berdasarkan tabel persentase hasil sintesis N'benziliden-2-metoksibenzohidrazida diatas pada waktu pemanasan yang berbeda-beda, maka dipilih waktu reaksi 4 menit karena pada pemanasan selama 6 menit diperoleh persentase hasil yang sama (74%).

## LAMPIRAN H

### CONTOH PERHITUNGAN KONVERSI INDEKS BIAS

perhitungan konversi indeks bias  $n_D^{20}$  pada hasil sintesis metil 2-metoksibenzoat dan 2-metoksibenzohidrazida.

Dengan rumus :

$$n_D^t = n_D^{t'} + 0,00045(t' - t)$$

dimana :

$n_D^{t'}$  = indeks bias pada temperatur tabel ( $20\text{ }^\circ\text{C}$ )

$t'$  = temperatur tabel ( $20\text{ }^\circ\text{C}$ )

$n_D^t$  = indeks bias pada temperatur percobaan

$t$  = temperatur percobaan

➤ Dimetil Salisilat (tahap 1)

| No. Replikasi | Indeks bias | Rata-rata |
|---------------|-------------|-----------|
| 1             | 1,5179      |           |
| 2             | 1,5176      | 1,5177    |
| 3             | 1,5177      |           |

Suhu  $20\text{ }^\circ\text{C} = \text{suhu kamar} + 0,00045 (30-20)$

$$= 1,5177 + 0,00045 (15)$$

$$= 1,52445$$

➤ 2-metoksibenzohidrazida (tahap 2)

| No. Replikasi | Indeks bias | Rata-rata |
|---------------|-------------|-----------|
| 1             | 1,4826      |           |
| 2             | 1,4827      | 1,4826    |
| 3             | 1,4825      |           |

Suhu 20°C = suhu kamar + 0,00045 (30-20)

$$= 1,4826 + 0,00045 (15)$$

$$= 1,48935$$

