

LAMPIRAN A

CONTOH PERHITUNGAN PERSENTASE HASIL SINTESIS

I. Perhitungan berat teoritis

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

Penimbangan : 11,04 gram

$$\text{Mol asam salisilat} : \frac{11,04 \text{ gram}}{138,134} = 0,079 \text{ mol}$$

b. Dimetil sulfat (BM : 126,19g/mol, berat jenis : 1,33g/cm³)

Volume : 20 ml

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

c. Hidrazin (BM : 32g/mol, berat jenis : 1,05 g/ cm³)

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 :

Asam salisilat + dimetil sulfat → dimetil salisilat + asam sulfat

| | | | | |
|--------|------------|----------|----------|----------|
| Awal | : 0,08 mol | 0,2 mol | 0 | 0 |
| Reaksi | : 0,08 mol | 0,08 mol | 0,08 mol | 0,08 mol |
| Sisa | : 0 | 0,12 mol | 0,08 mol | 0,08 mol |

BM teoritis = 166 g/mol

Massa teoritis = 0,08 mol x 166 g/mol = 13,28 gram

Massa Praktis = 12,04 gram

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

LAMPIRAN B
CONTOH PERHITUNGAN KONVERSI INDEKS BIAS

Perhitungan konversi indeks bias n_D^{20} pada hasil sintesis dimetil salisilat dan 2-metoksibenzohidrazida.

Dengan rumus :

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

Dimana :

n_D^t = indeks bias pada temperatur tabel (20 °C)

t = temperatur tabel (20 °C)

$n_D^{t'}$ = indeks bias pada temperatur percobaan

t' = temperatur percobaan

➤ Dimetil salisilat (tahap satu)

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

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

$$= 1,5177 + 0,00045 (10)$$

$$= 1,5132$$

➤ 2-metoksibenzohidrazida (tahap dua)

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

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

$$= 1,4826 + 0,00045 (10)$$

$$= 1,4871$$

LAMPIRAN C
UJI DENGAN FeCl_3 PADA SENYAWA HASIL SINTESIS

Uji dengan FeCl_3 berguna untuk mengetahui apakah gugus OH fenolik masih terdapat dalam struktur senyawa hasil sintesis. Uji ini dilakukan dengan melarutkan sejumlah zat dengan etanol kemudian ditetaskan FeCl_3 . Bila larutan berubah warna menjadi ungu/biru tua, maka senyawa tersebut memiliki gugus OH fenolik pada strukturnya. Hasil uji dengan FeCl_3 senyawa hasil sintesis dapat dilihat pada tabel dibawah ini :

Tabel identifikasi gugus OH fenolik dengan FeCl_3

| Senyawa | Hasil uji dengan FeCl_3 | Gugus OH fenolik |
|-------------------|----------------------------------|------------------|
| Asam Salisilat | Ungu | + |
| Dimetil salisilat | Kuning | - |
| Senyawa tiga A | Kuning | - |
| Senyawa tiga B | Ungu | + |

Keterangan :

+ = terbentuk warna ungu tua

- = tidak terbentuk warna ungu

LAMPIRAN D

KESEMPURNAAN HASIL SINTESIS SENYAWA TIGA A

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 senyawa tiga A
dengan waktu pemanasan yang berbeda

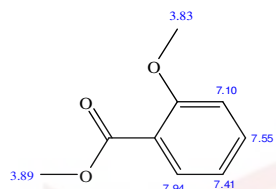
| Waktu pemanasan | Persentase hasil (%) |
|-----------------|------------------------|
| 1x2 menit | 70 |
| 2x2 menit | 74 |
| 3x2 menit | 74 |
| 4x2 menit | 74 |

Berdasarkan table persentase hasil sintesis senyawa tiga A diatas pada waktu pemanasan yang berbeda-beda, maka dipilih waktu waktu pemanasan 2x2 menit karena pada pemanasan selama 3x2 menit diperoleh persentase hasil yang sama (74%).

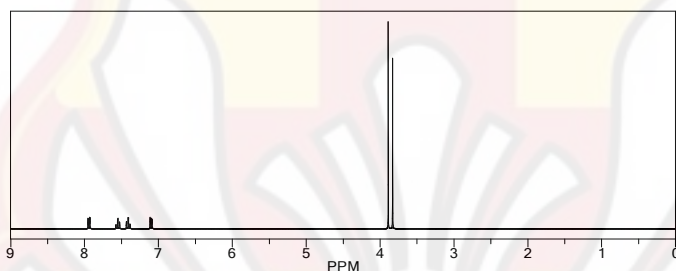
LAMPIRAN E

ESTIMASI DIMETIL SALISILAT

ChemNMR ¹H Estimation



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



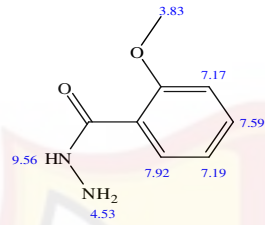
Protocol of the H-1 NMR Prediction:

| 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 |
| CH3 | 3.89 | 0.86 | methyl |
| | | 3.02 | 1 alpha -OC(=O)-1:C*C*C*C*C*1 |
| | | 0.01 | general corrections |

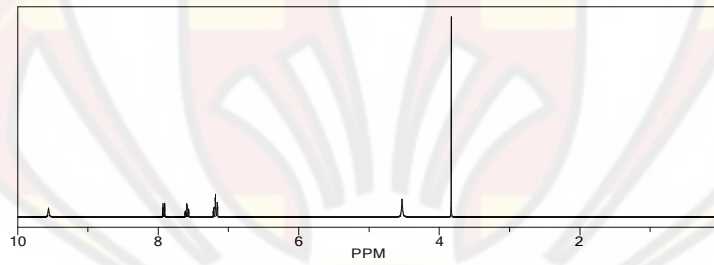
LAMPIRAN F

ESTIMASI 2-METOKSIBENZOHIIDRAZIDA

ChemNMR ¹H Estimation



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

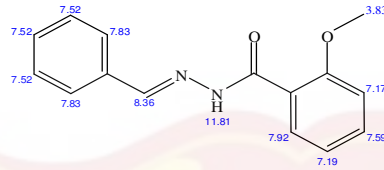


Protocol of the H-1 NMR Prediction:

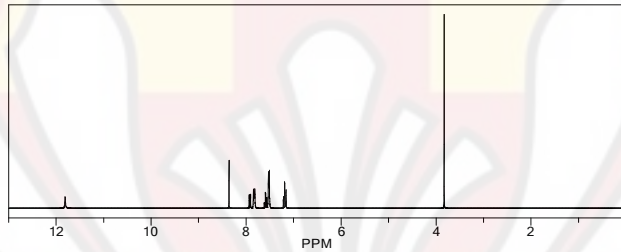
| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|------|-------|-------------|---------------------------|
| NH | 9.56 | 8.00 | sec. amide |
| | | -1.56 | general corrections |
| NH2 | 4.53 | 2.00 | amine |
| | | 2.53 | general corrections |
| CH | 7.17 | 7.26 | 1-benzene |
| | | -0.49 | 1 -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 G
ESTIMASI N'BENZILIDEN-2-
METOKSIBENZOHIIDRAZIDA

ChemNMR ¹H Estimation



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

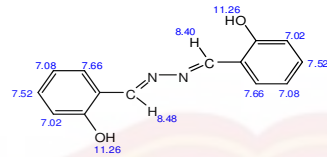


Protocol of the H-1 NMR Prediction:

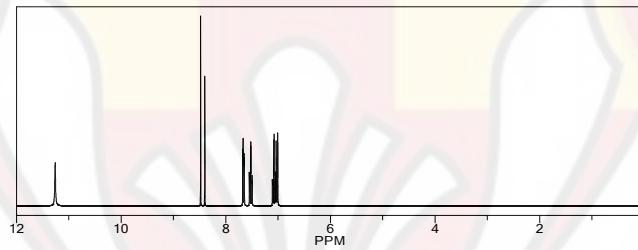
| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|------|-------|-------------|-----------------------------|
| NH | 11.81 | 8.00 | sec. amide |
| | | 3.81 | general corrections |
| CH | 7.17 | 7.26 | 1-benzene |
| | | -0.49 | 1 -O-C |
| | | 0.18 | 1 -C(=O)N |
| | | 0.22 | general corrections |
| 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-C*1 |
| | | 0.10 | general corrections |
| CH | 8.36 | 8.11 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | 0.25 | general corrections |

LAMPIRAN H
ESTIMASI 1,2-BIS-(2-HIDROKSIBENZILIDEN)-
HIDRAZIN

ChemNMR ¹H Estimation



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) |
|------|-------|-------------|---------------------------|
| OH | 11.26 | 5.00 | aromatic C-OH |
| | | 6.26 | general corrections |
| OH | 11.26 | 5.00 | aromatic C-OH |
| | | 6.26 | general corrections |
| CH | 7.02 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.53 | 1 -O from 1-benzene |
| | | 0.26 | general corrections |
| CH | 7.02 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.53 | 1 -O from 1-benzene |
| | | 0.26 | general corrections |
| CH | 7.66 | 7.62 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.66 | 7.62 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.52 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.40 | general corrections |
| CH | 7.52 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.40 | general corrections |
| CH | 7.08 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.44 | 1 -O from 1-benzene |
| | | 0.23 | general corrections |
| CH | 7.08 | 7.29 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | -0.44 | 1 -O from 1-benzene |
| | | 0.23 | general corrections |
| H | 8.40 | 8.11 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | 0.29 | general corrections |
| H | 8.48 | 8.11 | benzylidenimin |
| | | ? | 1 unknown substituent(s) |
| | | 0.37 | general corrections |