

LAMPIRAN A

BAGAN ALIR SINTESIS 2-(2,3-DIMETILFENILAMINO)METIL BENZOAT

10 mmol Asam Mefenamat (2,412 gram)
50 mmol Kalium Karbonat (6,9 gram)
50 mmol dimetil sulfat (5 ml)
30 ml aseton

diaduk 30 menit (suhu kamar)

Suspensi :
metil mefenamat, kalium karbonat, aseton

refluks 1 jam

Larutan : metil mefenamat, H_2O , aseton

aseton diuapkan
disaring

fase cair : H_2O

Dibuang

padatan :
metil mefenamat,
kalium karbonat

cuci dengan air

padatan :
metil mefenamat

fase cair :
kalium karbonat, H_2O

rekristalisasi
etanol 90%

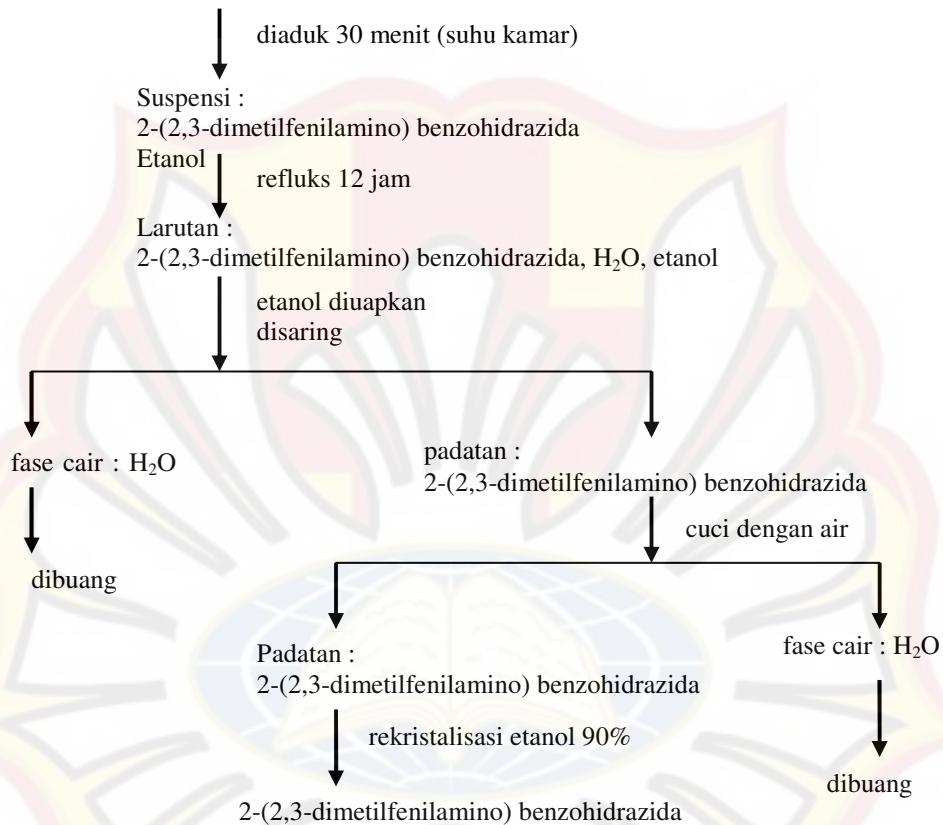
2-(2,3-dimetilfenilamino)metil benzoat

dibuang

LAMPIRAN B

BAGAN ALIR SINTESIS 2-(2,3-DIMETILFENILAMINO) BENZOHIDRAZIDA

4 mmol metil Mefenamat (1,0gram)
Hidrazin hidrat 2ml
etanol



LAMPIRAN C
BAGAN ALIR SINTESIS *N'*-(*P*-BENZILIDEN-2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA

10 mmol 2-(2,3-dimetilfenilamino) benzohidrazida

Benzaldehida 10 mmol (1 ml)

Etanol 10 ml

↓ diaduk 5 menit (suhu kamar)

Kristal :

N'-(*p*-benziliden-(2,3-dimetilfenil) amino benzohidrazida

↓ Microwave 5 menit

Uji KLT tiap 1 menit

↓

Padatan :

N'-(*p*-benziliden-(2,3-dimetilfenil) amino benzohidrazida

↓ cuci dengan air

Padatan :

N'-(*p*-benziliden-(2,3-dimetilfenil) amino benzohidrazida

↓ rekristalisasi
etanol 90%

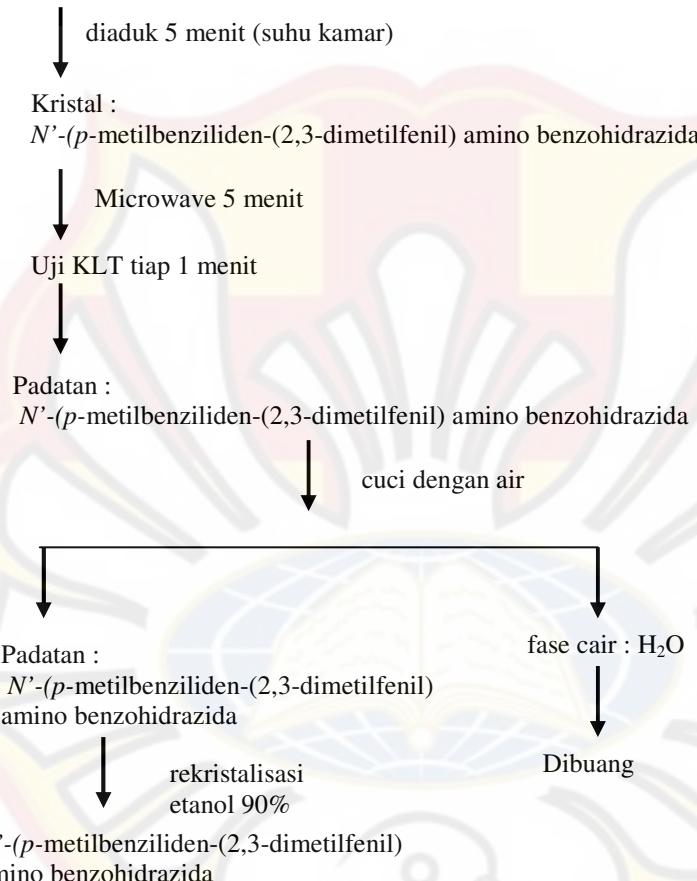
N'-(*p*-benziliden-(2,3-dimetilfenil) amino
benzohidrazida

fase cair : H₂O

↓ Dibuang

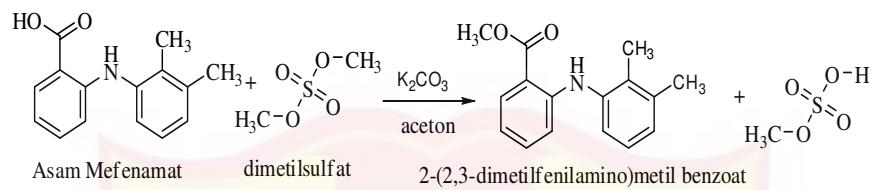
LAMPIRAN D
BAGAN ALIR SINTESIS *N'*-(*P*-METILBENZILIDEN-2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA

10 mmol 2-(2,3-dimetilfenilamino) benzohidrazida
p-metilbenzaldehida 10 mmol (1 ml)
Etanol 10 ml



LAMPIRAN E

PERHITUNGAN HASIL SINTESIS 2-(2,3-DIMETILFENILAMINO)METIL BENZOAT



Berat molekul Asam Mefenamat = 241,2 g/mol

$n = g/BM$

$0,01\text{ mol} = g/241,2\text{ g/mol}$

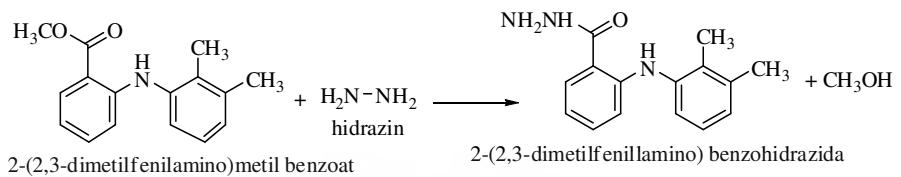
$G = 2,412 \text{ g}$

Berat Molekul Dimetilsulfat = 126 g/mol

$BJ = 1,325 \text{ g/ml} \quad \rightarrow \quad vol = G/1,325$

$G = 6,3 \text{ g} \quad \quad \quad = 44,75 \text{ ml} \approx 5,0 \text{ ml}$

LAMPIRAN F
**PERHITUNGAN HASIL SINTESIS 2-(2,3-DIMETILFENILAMINO)
BENZOHIDRAZIDA**



Berat molekul 2-(2,3-dimetilfenilamino) benzohidrazida = 255 g/mol

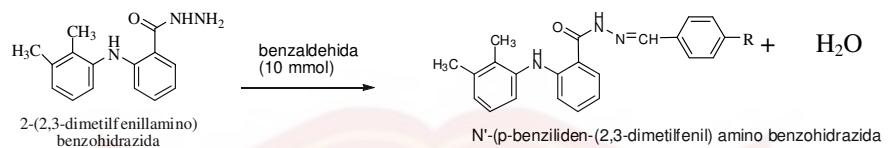
$$N = g/BM$$

$$0,004 \text{ mol} = \frac{\text{g}}{255 \text{ g/mol}}$$

G = 1,02 g

LAMPIRAN G

PERHITUNGAN HASIL SINTESIS *N'*(*P*-BENZILIDEN-(2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA



Berat molekul 2-(2,3-dimethylfenilamino) benzohidrazida = 255 g/mol

N = g/BM

0,01 mol = g/255 g/mol

G = 2,55 g

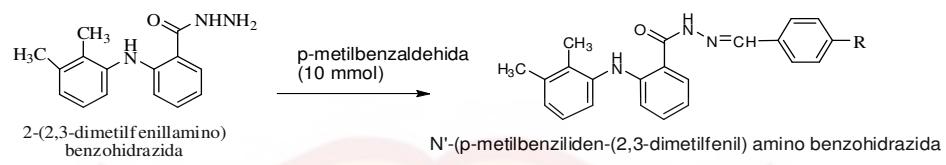
Berat molekul benzaldehida = 106,12 g/mol

N = g/mol

0,01 mol = g/106,12 g/mol

G = 1,06 g ≈ 1,06 ml

LAMPIRAN H
PERHITUNGAN HASIL SINTESIS N'(P-METILBENZILIDEN-(2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA



LAMPIRAN I

PERHITUNGAN RANDEMEN HASIL 2-(2,3-DIMETILFENILAMINO)METIL BENZOAT

$$\text{Randemen hasil} = \frac{\text{berat praktis}}{\text{berat teoritis}} \times 100\%$$

a. 2-(2,3-dimetilfenilamino) metil benzoat

Sintesis 1 : berat praktis = 2,26 g

Berat teoritis = 2,55 g

Presentase hasil

$$= \frac{2,26}{2,55} \times 100\% = 88,63\%$$

Sintesis II : presentase hasil = 85,49 % (2,18 g)

Sintesis III : presentase hasil = 86,66 % (2,21 g)

$$\text{Presentase hasil rata-rata} : \frac{88,63\% + 85,49\% + 86,66\%}{3} = 86,93\%$$

LAMPIRAN J
PERHITUNGAN RANDEMEN HASIL 2-(2,3-DIMETILFENILAMINO) BENZOHIDRAZIDA

$$\text{Randemen hasil} = \frac{\text{berat praktis}}{\text{berat teoritis}} \times 100\%$$

b. 2-(2,3-dimetilfenilamino) benzohidrazida

Sintesis I : berat praktis = 0,78 g

Berat teoritis = 1,02 g

Presentase hasil

$$= \frac{0,78}{1,02} \times 100\% = 76\%$$

Sintesis II : presentase hasil = 73 % (0,75 g)

Sintesis III : presentase hasil = 68 % (0,70 g)

Presentase hasil rata-rata : $\frac{76\% + 73\% + 68\%}{3} = 72\%$

LAMPIRAN K
PERHITUNGAN RANDEMEN HASIL *N'*-(*P*-BENZILIDEN-(2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA

$$\text{Randemen hasil} = \frac{\text{berat praktis}}{\text{berat teoritis}} \times 100\%$$

c. *N'*-(*p*-benziliden-(2,3-dimetilfenil)amino benzohidrazida

Sintesis I : berat praktis = 2,52 g

Berat teoritis = 3,32 g

Presentase hasil

$$= \frac{2,52}{3,32} \times 100\% = 76\%$$

Sintesis II : presentase hasil = 75 % (2,48 g)

Sintesis III : presentase hasil = 70 % (2,35 g)

Presentase hasil rata-rata : $\frac{76\% + 75\% + 70\%}{3} = 74\%$

LAMPIRAN L
**PERHITUNGAN RANDEMEN HASIL N'-(P-METILBENZILIDEN-
(2,3-DIMETILFENIL) AMINO BENZOHIDRAZIDA**

$$\text{Randemen hasil} = \frac{\text{berat praktis}}{\text{berat teoritis}} \times 100\%$$

a. *N'*-(*p*-benziliden-(2,3-dimetilfenil)amino benzohidrazida

Sintesis I : berat praktis = 4,39 g

Berat teoritis = 4,21 g

Presentase hasil

$$= 4,39 \quad \quad \quad x \ 100 \% \quad = \ 82 \%$$

Sintesis II : presentase hasil = 86 % (4,21 g)

Sintesis III : presentase hasil = 83 % (4,34 g)

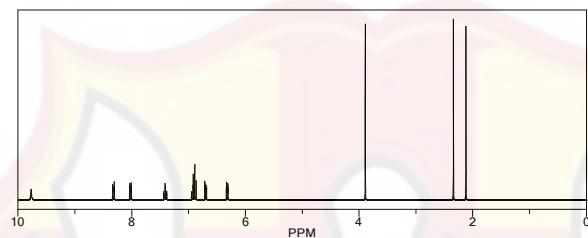
$$\text{Presentase hasil rata-rata : } \frac{82 \% + 86 \% + 83 \%}{3} = 84 \%$$

LAMPIRAN M
DATA ESTIMASI SENYAWA 2-(2,3-
DIMETILFENILAMINO)METIL BENZOAT

ChemNMR ¹H Estimation



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



Protocol of the H-1 NMR Prediction:

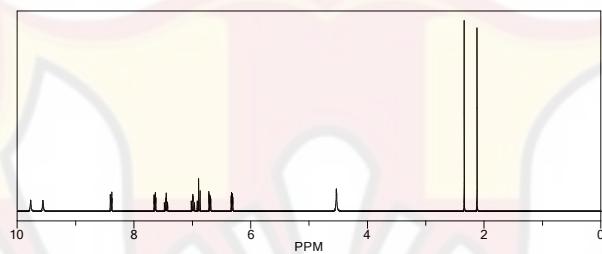
| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|----------|-------|-------------------------------|---------------------------|
| NH 9,77 | 4,00 | aromatic C-NH | |
| | -5,77 | general corrections | |
| CH 6,32 | 7,26 | 1-benzene | |
| | -0,80 | 1 -N | |
| | -0,12 | 1 -C | |
| | -0,19 | 1 -C | |
| | 0,17 | general corrections | |
| CH 8,32 | 7,26 | 1-benzene | |
| | -0,80 | 1 -N | |
| | 0,11 | 1 -(C(=O)OC | |
| | 1,75 | general corrections | |
| CH 8,02 | 7,26 | 1-benzene | |
| | -0,25 | 1 - | |
| | 0,71 | 1 -(C(=O)OC | |
| | 0,30 | general corrections | |
| CH 6,70 | 7,26 | 1-benzene | |
| | -0,64 | 1 -N | |
| | -0,12 | 1 -C | |
| | -0,20 | 1 -C | |
| | 0,40 | general corrections | |
| CH 6,89 | 7,26 | 1-benzene | |
| | -0,25 | 1 -N | |
| | -0,19 | 1 -C | |
| | 0,12 | 1 -C | |
| | 0,19 | general corrections | |
| CH 7,41 | 7,26 | 1-benzene | |
| | -0,25 | 1 -N | |
| | 0,21 | 1 -(C(=O)OC | |
| | 0,19 | general corrections | |
| CH 6,92 | 7,26 | 1-benzene | |
| | -0,64 | 1 -N | |
| | 0,11 | 1 -(C(=O)OC | |
| | 0,19 | 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 | |
| CH3 2,12 | 0,86 | methyl | |
| | 1,49 | 1 alpha -1:C*C*C*C*C*1 | |
| | -0,23 | general corrections | |
| CH3 2,34 | 0,86 | methyl | |
| | 1,49 | 1 alpha -1:C*C*C*C*C*1 | |
| | -0,01 | general corrections | |

LAMPIRAN N
DATA ESTIMASI 2-(2,3-DIMETILFENILAMINO)
BENZOHIDRAZIDA

ChemNMR ¹H Estimation



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



Protocol of the H-1 NMR Prediction:

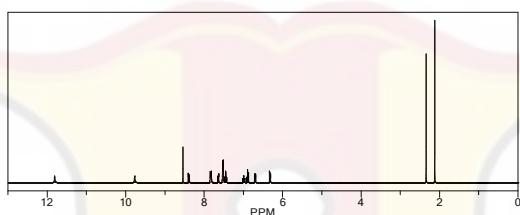
| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|----------|-------|----------------------|---------------------------|
| NH 9,56 | 8,00 | sec. amide | |
| | 1,56 | general corrections | |
| NH2 4,53 | 2,00 | amine | |
| | 2,53 | general corrections | |
| NH 9,77 | 4,00 | aromatic C-NH | |
| | 5,77 | general corrections | |
| CH 6,32 | 7,26 | 1-benzene | |
| | 0,80 | 1 -N | |
| | -0,12 | 1 -C | |
| | -0,19 | 1 -C | |
| | 0,17 | general corrections | |
| CH 8,39 | 7,26 | 1-benzene | |
| | -0,80 | 1 -H | |
| | 0,18 | 1 -(C(=O))N | |
| | 1,75 | general corrections | |
| CH 7,64 | 7,26 | 1-benzene | |
| | -0,25 | 1 -N | |
| | 0,69 | 1 -(C(=O))N | |
| | -0,06 | general corrections | |
| CH 6,70 | 7,26 | 1-benzene | |
| | -0,64 | 1 -H | |
| | -0,12 | 1 -C | |
| | -0,20 | 1 -C | |
| | 0,40 | general corrections | |
| CH 6,89 | 7,26 | 1-benzene | |
| | -0,25 | 1 -N | |
| | -0,19 | 1 -C | |
| | 0,12 | 1 -C | |
| | 0,19 | general corrections | |
| CH 7,45 | 7,26 | 1-benzene | |
| | -0,25 | 1 -N | |
| | 0,25 | 1 -(C(=O))N | |
| | 0,19 | general corrections | |
| CH 6,99 | 7,26 | 1-benzene | |
| | -0,64 | 1 -N | |
| | 0,18 | 1 -(C(=O))N | |
| | 0,19 | general corrections | |
| CH3 2,12 | 0,86 | methyl | |
| | 1,49 | 1 alpha -1:C*C*C*C*1 | |
| | -0,23 | general corrections | |
| CH3 2,34 | 0,86 | methyl | |
| | 1,49 | 1 alpha -1:C*C*C*C*1 | |
| | -0,01 | general corrections | |

LAMPIRAN O
DATA ESTIMASI N'-(*P*-BENZILIDEN-2,3-DIMETILFENIL)
AMINO BENZOVIDRAZIDA

ChemNMR ¹H Estimation



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

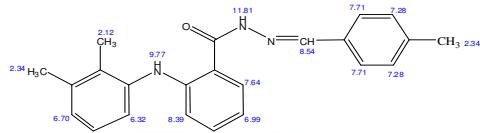


Protocol of the H-1 NMR Prediction:

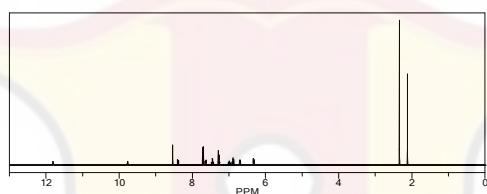
| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|------|-------|---|---|
| NH | 11,81 | 8,00 3,81 | sec. amide general corrections |
| NH | 9,77 | 4,00 5,77 | aromatic C-NH general corrections |
| CH | 6,32 | 7,26 -0,80 -0,12 0,19 0,17 | 1-benzene 1-N 1-C 1-C general corrections |
| CH | 8,39 | 7,26 -0,80 0,18 1,75 | 1-benzene 1-N 1-C(=O)N general corrections |
| CH | 7,64 | 7,26 -0,25 0,69 -0,08 | 1-benzene 1-N 1-C(=O)N general corrections |
| CH | 7,83 | 7,62 ? | benzylidenimin 1 unknown substituent(s) |
| CH | 7,52 | 7,29 ? | benzylidenimin 1 unknown substituent(s) |
| CH | 7,83 | 7,62 ? | benzylidenimin 1 unknown substituent(s) |
| CH | 6,70 | 7,26 -0,64 -0,12 -0,20 0,40 | 1-benzene 1-N 1-C 1-C general corrections |
| CH | 7,52 | 7,29 ? | benzylidenimin 1 unknown substituent(s) |
| CH | 6,89 | 7,26 -0,25 -0,19 -0,12 0,19 | 1-benzene 1-N 1-C 1-C general corrections |
| CH | 7,45 | 7,26 -0,25 0,25 -0,19 0,19 | 1-benzene 1-N 1-C(=O)N general corrections |
| CH | 6,99 | 7,26 -0,64 0,18 0,19 | 1-benzene 1-N 1-C(=O)N general corrections |
| CH | 8,54 | 8,11 ? | benzylidenimin 1 unknown substituent(s) |
| CH3 | 2,12 | 0,86 1,49 -0,23 | methy... -1:C*C*C*C*C*1 general corrections |
| CH3 | 2,34 | 0,86 1,49 -0,01 | methy... -1:C*C*C*C*C*1 general corrections |
| H | 7,52 | 7,29 ? | benzylidenimin 1 unknown substituent(s) |
| | | 0,23 | general corrections |

LAMPIRAN P
DATA ESTIMASI *N'*-(*P*-METILBENZILIDEN-2,3-DIMETILFENIL) AMINO BENZOVIDRAZIDA

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 |
| NH | 9.77 | 4.00 | aromatic -NH |
| | | 1.77 | general corrections |
| CH | 6.32 | 7.26 | 1-benzene |
| | | -0.80 | 1 -N |
| | | -0.12 | 1 -C |
| | | -0.19 | 1 -C |
| | | 0.17 | general corrections |
| CH | 8.39 | 7.26 | 1-benzene |
| | | -0.80 | 1 -N |
| | | 0.18 | 1 -C(=O)N |
| | | 1.75 | general corrections |
| CH | 7.64 | 7.26 | 1-benzene |
| | | -0.25 | 1 -N |
| | | 0.69 | 1 -C(=O)N |
| | | -0.06 | general corrections |
| CH | 7.71 | 7.62 | benzylideneamin |
| | | ? | 1 unknown substituent(s) |
| | | -0.12 | 1 -C from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.28 | 7.29 | benzylideneamin |
| | | ? | 1 unknown substituent(s) |
| | | -0.20 | 1 -N |
| | | 0.19 | general corrections |
| CH | 7.71 | 7.62 | benzylideneamin |
| | | ? | 1 unknown substituent(s) |
| | | -0.12 | 1 -C from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.28 | 7.29 | benzylideneamin |
| | | ? | 1 unknown substituent(s) |
| | | -0.20 | 1 -C from 1-benzene |
| | | 0.19 | general corrections |
| CH | 6.70 | 7.26 | 1-benzene |
| | | -0.64 | 1 -N |
| | | -0.12 | 1 -C |
| | | -0.20 | 1 -C |
| | | 0.40 | general corrections |
| CH | 6.89 | 7.24 | 1-benzene |
| | | -0.25 | 1 -N |
| | | -0.19 | 1 -C |
| | | -0.12 | 1 -C |
| | | 0.19 | general corrections |
| CH | 7.45 | 7.26 | 1-benzene |
| | | -0.25 | 1 -N |
| | | 0.25 | 1 -C(=O)N |
| | | 0.19 | general corrections |
| CH | 6.99 | 7.26 | 1-benzene |
| | | -0.64 | 1 -N |
| | | 0.18 | 1 -C(=O)N |
| | | 0.19 | general corrections |
| CH | 8.54 | 8.11 | benzylideneamin |
| | | ? | 1 unknown substituent(s) |
| | | 0.43 | general corrections |
| CH3 | 2.12 | 0.86 | methylene |
| | | 1.49 | 1 alpha -1'C=C'C*C*1 |
| | | -0.23 | general corrections |
| CH3 | 2.34 | 0.86 | methylene |
| | | 1.49 | 1 alpha -1'C=C'C*C*1 |
| | | -0.01 | general corrections |
| CH3 | 2.34 | 0.86 | methylene |
| | | 1.49 | 1 alpha -1'C=C'C*C*1 |
| | | -0.01 | general corrections |