CONTOH PERHITUNGAN PERSENTASE HASIL SINTESIS

I. Perhitungan berat teoritis

  a. Ammonium tiosianat (BM : 76,12 g/mol)

    Penimbangan : 1,14 gram

    mmol ammonium tiosianat : $\dfrac{1,14 \times 1000}{76,12} = 14,98 \sim 15$

  b. Benzoil klorida (BM : 140,57 g/mol, berat jenis : 1,2070 g/cm^3)

    Volume : 1,2 ml

    mmol benzoil klorida : $\dfrac{12 \times 1,2070}{140,57} \times 1000 = 10,3 \sim 10$ mmol

  c. Anilin (BM : 93,13 g/mol, berat jenis : 1,02 g/cm^3)

    Volume : 0,9 ml

    mmol anilin : $\dfrac{0,9 \times 1,02}{93,13} \times 1000 = 9,9 \sim 10$ mmol

II. Perhitungan persentase hasil sintesis berdasarkan mmol teoritis

  Persentase hasil N-fenil-N'-benzoiltiourea :

	ammonium tiosianat +	benzoil klorida		benzoilisotiosianat +	NH$_4$Cl
awal	15 mmol	10 mmol		0	0
reaksi	10 mmol	10 mmol	-	10 mmol	10 mmol +
sisa	5 mmol	0		10 mmo	10 mmol

	Benzoilisotiosianat +	anilin		N-fenil-N'-benzoiltiourea	
Awal	10 mmo	10 mmol		0	
reaksi	10 mmol	10 mmol	-	10 mmol	+
sisa	0	0		10 mmol	

Perhitungan persentase hasil sintesis :

BM N-fenil-N'-benzoiltiourea teoritis = 256,32

Sintesis I :   Massa praktis = 2,11 gram

Mmol praktis = $\frac{2,11}{256,32}$ x 1000 = 8,23 mmol

Mmol teoritis = 9,88 mmol

% hasil   = $\frac{8,23}{9,88}$ x 100 % = 83,23 % ≈ 83 %

Sintesis II :   Massa praktis = 2,12 gram

Mmol praktis = $\frac{2,12}{256,32}$ x 1000 = 8,27 mmol

% hasil   = $\frac{8,27}{9,88}$ x 100 % = 83,70 % ≈ 84 %

Sintesis III : Massa praktis = 2,11 gram

Mmol praktis = 8,23 mmol

% hasil   = $\frac{8,23}{9,88}$ x 100% = 83,23 % ≈ 83 %

Persentase hasil rata-rata : $\frac{83\% + 84\% + 83\%}{3}$ = 83,33 %

LAMPIRAN B
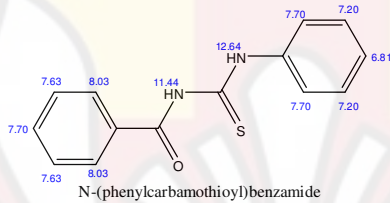# SKEMA KERJA SINTESIS SENYAWA TURUNAN N-FENIL-N'-BENZOILTIOUREA

Dalam beaker glass 50 ml :
- Benzoil klorida 10 mmol (1,16 ml)   - PEG 400 3-4 tetes (0,18 g)
- Amonium tiosianat 10 mmol (0,8 g)   - CH₂Cl₂ 5 ml
- Silika Gel 4 g

↓ diaduk hingga berwarna kuning

Padatan :
- Benzoilisotiosianat
- Silika gel
- PEG 400

↓ ditambah 0,9 ml anilin dalam 2 ml diklorometana*
diaduk ad homogen, diuapkan dalam lemari asam
dimasukkan *microwave* (3 X 0,5 menit) 160 watt, dinginkan

Padatan :
- N-fenil-N'-benzoiltiourea   - NH₄Cl
- Asam benzoat   - Silika gel

↓ dicuci (4 X 10 ml diklorometana)

Padatan (dibuang) :
Silika gel

Filtrat :
- NH₄Cl   - H₂O
- N-fenil-N'-benzoiltiourea - Asam benzoat

↓ diuapkan

Padatan :
- N-fenil-N'-benzoiltiourea
- Asam benzoat

↓ dicuci dengan Na₂CO₃ 10%, HCl

Filtrat (dibuang) :
- Na benzoat
- H₂O

Padatan N-fenil-N'-benzoiltiourea

Kristal N-fenil-N'-benzoiltiourea

Uji kemurnian :
- KLT
- Titik leleh

Identifikasi struktur :
- Spektrofotometri UV-Vis
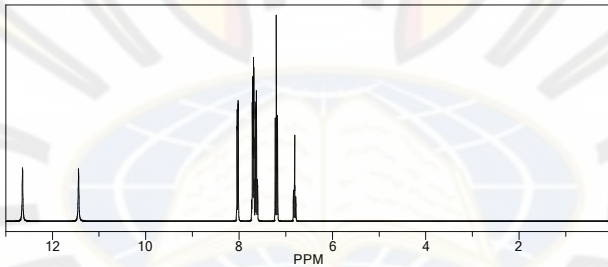- Spektrofotometri IR
- Spektrofotometri ¹H-NMR

* pada sintesi N-(4-klorofenil)-N'-benzoiltiourea ditambah 1,27 gr 4-kloroanilin dalam 3 ml diklorometana

* pada sintesis N-(3,4-diklorofenil)-N'-benzoiltiourea ditambah 1,62 gr 3,4-dikloroanilin dalam 3 ml diklorometana

LAMPIRAN C

ESTIMASI RMI-^{1}H SENYAWA N-FENIL-N'-BENZOILTIOUREA

ChemNMR [1]H Estimation

N-(phenylcarbamothioyl)benzamide

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


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Protocol of the H-1 NMR Prediction:

Node    Shift    Base + Inc.   Comment (ppm rel. to TMS)

NH  12.64         4.00         aromatic C-NH
                  8.64           general corrections
NH  11.44         8.00         sec. amide
                  3.44           general corrections
CH   7.70         7.26         1-benzene
                 -0.80           1 -N
                  1.24           general corrections
CH   8.03         7.26         1-benzene
                  0.69           1 -C(=O)N
                  0.08           general corrections
CH   7.70         7.26         1-benzene
                 -0.80           1 -N
                  1.24           general corrections
CH   8.03         7.26         1-benzene
                  0.69           1 -C(=O)N
                  0.08           general corrections
CH   7.20         7.26         1-benzene
                 -0.25           1 -N
                  0.19           general corrections
CH   7.63         7.26         1-benzene
                  0.18           1 -C(=O)N
                  0.19           general corrections
CH   7.20         7.26         1-benzene
                 -0.25           1 -N
                  0.19           general corrections
CH   7.63         7.26         1-benzene
                  0.18           1 -C(=O)N
                  0.19           general corrections
CH   6.81         7.26         1-benzene
                 -0.64           1 -N
                  0.19           general corrections
CH   7.70         7.26         1-benzene
                  0.25           1 -C(=O)N
                  0.19           general corrections
```
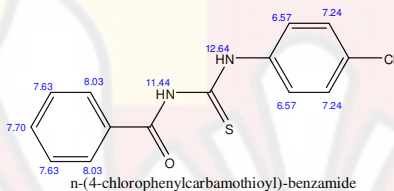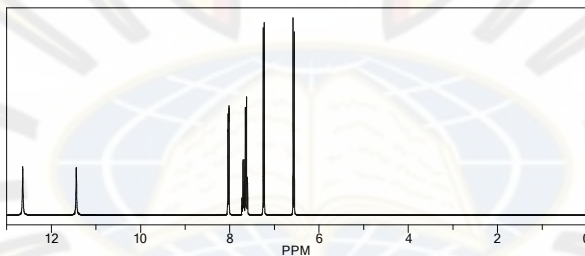
LAMPIRAN D
ESTIMASI RMI-[1]H SENYAWA N-(4-KLOROFENIL)-N'-BENZOILTIOUREA

ChemNMR [1]H Estimation

n-(4-chlorophenylcarbamothioyl)-benzamide

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  12.64        4.00           aromatic C-NH
                 8.64               general corrections
NH  11.44        8.00           sec. amide
                 3.44               general corrections
CH  7.24         7.26           1-benzene
                 0.01               1 -Cl
                -0.25               1 -N
                 0.22               general corrections
CH  6.57         7.26           1-benzene
                -0.06               1 -Cl
                -0.80               1 -N
                 0.17               general corrections
CH  8.03         7.26           1-benzene
                 0.69               1 -C(=O)N
                 0.08               general corrections
CH  7.24         7.26           1-benzene
                 0.01               1 -Cl
                -0.25               1 -N
                 0.22               general corrections
CH  6.57         7.26           1-benzene
                -0.06               1 -Cl
                -0.80               1 -N
                 0.17               general corrections
CH  8.03         7.26           1-benzene
                 0.69               1 -C(=O)N
                 0.08               general corrections
CH  7.63         7.26           1-benzene
                 0.18               1 -C(=O)N
                 0.19               general corrections
CH  7.63         7.26           1-benzene
                 0.18               1 -C(=O)N
                 0.19               general corrections
CH  7.70         7.26           1-benzene
                 0.25               1 -C(=O)N
                 0.19               general corrections
```
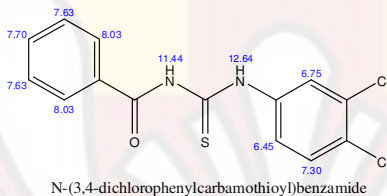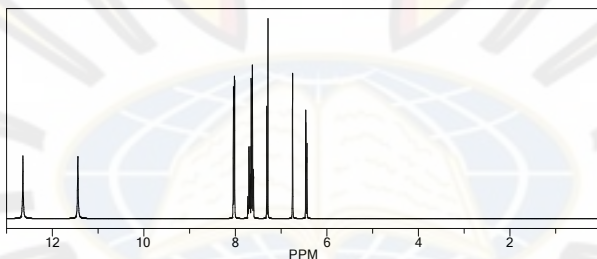
LAMPIRAN E
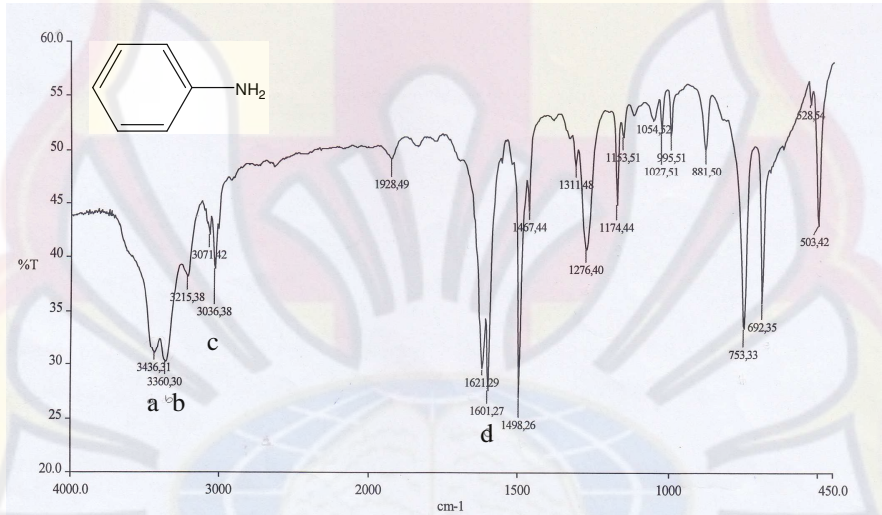# ESTIMASI RMI-[1]H SENYAWA N-(3,4-DIKLOROFENIL)-N'-BENZOILTIOUREA

ChemNMR [1]H Estimation

N-(3,4-dichlorophenylcarbamothioyl)benzamide

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


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Protocol of the H-1 NMR Prediction:

Node    Shift    Base + Inc.   Comment (ppm rel. to TMS)

NH  12.64        4.00          aromatic C-NH
                 8.64              general corrections
NH  11.44        8.00          sec. amide
                 3.44              general corrections
CH  6.75         7.26          1-benzene
                 0.01              1 -Cl
                -0.06              1 -Cl
                -0.80              1 -N
                 0.34              general corrections
CH  7.30         7.26          1-benzene
                -0.06              1 -Cl
                 0.01              1 -Cl
                -0.25              1 -N
                 0.34              general corrections
CH  6.45         7.26          1-benzene
                -0.12              1 -Cl
                -0.06              1 -Cl
                -0.80              1 -N
                 0.17              general corrections
CH  8.03         7.26          1-benzene
                 0.69              1 -C(=O)N
                 0.08              general corrections
CH  8.03         7.26          1-benzene
                 0.69              1 -C(=O)N
                 0.08              general corrections
CH  7.63         7.26          1-benzene
                 0.18              1 -C(=O)N
                 0.19              general corrections
CH  7.63         7.26          1-benzene
                 0.18              1 -C(=O)N
                 0.19              general corrections
CH  7.70         7.26          1-benzene
                 0.25              1 -C(=O)N
                 0.19              general corrections
```

LAMPIRAN F
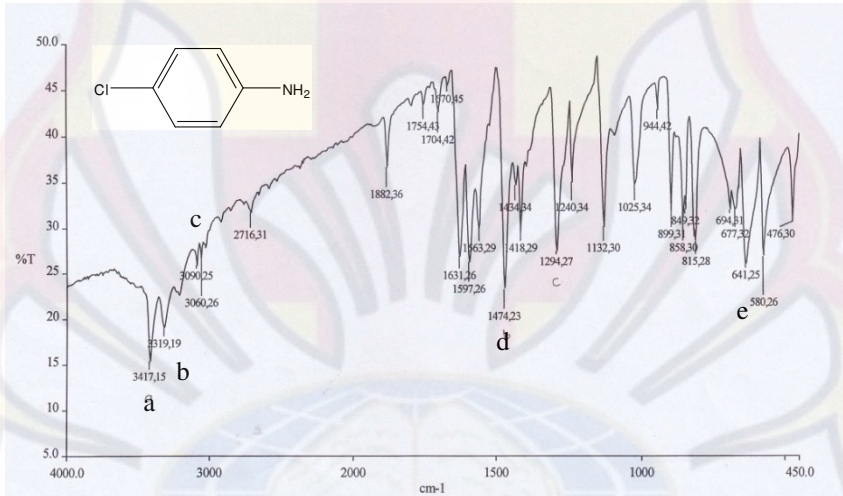## SPEKTRUM INFRAMERAH SENYAWA ANILIN DENGAN MENGGUNAKAN PELLET KBR

TABEL SERAPAN INFRAMERAH SENYAWA ANILIN

Ikatan	Bilangan gelombang (cm⁻¹)		No. puncak
	Pustaka *	Anilin	
$-NH_2$	3000 – 3700	3436, 3360	a, b
Csp^2-H	3000 – 3300	3036	c
C=C aromatis	1450 – 1600	1601	d

Keterangan : * Pavia *et al* 2001, Fessenden 1986, Hart 2003 , Williams 1966

LAMPIRAN G
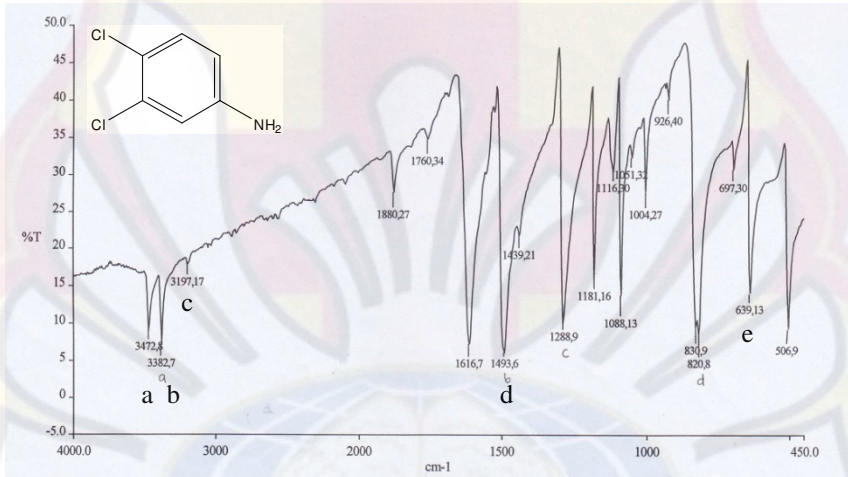## SPEKTRUM INFRAMERAH SENYAWA 4-KLOROANILIN
DENGAN MENGGUNAKAN PELLET KBR

TABEL SERAPAN INFRAMERAH SENYAWA 4-KLOROANILIN

Ikatan	Bilangan gelombang (cm^{-1})		No. puncak
	Pustaka *	4-kloroanilin	
$-NH_2$	3000 – 3700	3417, 3319	a, b
Csp^2-H	3000 – 3300	3090	c
C=C aromatis	1450 – 1600	1474	d
C–Cl	600 – 800	641	e

Keterangan : * Pavia *et al* 2001, Fessenden 1986, Hart 2003, Williams 1966.

LAMPIRAN H
SPEKTRUM INFRAMERAH SENYAWA 3,4-DIKLOROANILIN
DENGAN MENGGUNAKAN PELLET KBR

TABEL SERAPAN INFRAMERAH SENYAWA 3,4-DIKLOROANILIN

Ikatan	Bilangan gelombang (cm^{-1})		No. puncak
	Pustaka *	3,4-dikloroanilin	
–NH$_2$	3000 – 3700	3472, 3382	a, b
Csp^2–H	3000 – 3300	3197	c
C=C aromatis	1450 – 1600	1493	d
C–Cl	600 – 800	639	e

Keterangan : * Pavia *et al* 2001, Fessenden 1986, Hart 2003, Williams 1966.