BAB V
KESIMPULAN DAN SARAN

5.1 Kesimpulan

Berdasarkan hasil penelitian yang telah dilakukan maka dapat disimpulan bahwa senyawa asam 3-(3,4-dihidroksifenil)-*N*-*o*-tolil-akrilamida (t.l. 190-193 ^{o}C) dapat disintesis melalui reaksi esterifikasi pada suhu ruang selama 6 jam, klorinasi dengan suhu refluks 80 ^{o}C selama 4 jam, kemudian dilanjutkan dengan reaksi amidasi secara *in situ* menggunkan amina *o*-tolilamin pada suhu ruang selama 1 jam dan tahap terakhir melalui reaksi deasetilasi pada suhu ruang selama 2 jam. Rendemen reaksi tahap akhir (deasetilasi) senyawa asam 3-(3,4-dihidroksifenil)-*N*-*o*-tolil-akrilamida adalah sebesar 57,68%. Hasil uji bioaktivitas terhadap sel Hela memberikan nilai IC_{50} untuk senyawa asam 3-(3,4-dihidroksifenil)-*N*-*o*-tolil-akrilamida sebesar 89,76 µg/mL dan dinyatakan dalam kategori aktif sebagai antikanker.

5.2 Saran

Pada penelitian selanjutnya disarankan untuk pengujian lebih lanjut dengan uji bioktivitas terhadap sel yang lain.

Lampiran 2. Perhitungan Sintesis

1. Sintesis senyawa asam 3-(3,4-diasetoksifenil)akrilat

a. Asam 3-(3,4-dihidroksifenil)akrilat

$$\text{n 3-(3,4-dihidroksi-fenil)akrilat} = \frac{\text{m asam 3-(3,4-dihidroksifenil)akrilat}}{\text{Mr asam 3-(3,4-dihidroksifenil)akrilat}}$$

$$= \frac{0{,}8071 \text{ gram}}{180{,}16 \text{ gram/mol}}$$

$$= 0{,}0045 \text{ mol} = 4{,}5 \text{ mmol}$$

b. Anhidrida asetat

n anhidrida asetat = 5 kali n asam 3-(3,4-dihidroksifenil)akrilat

= 5 x 0,0045 mol

= 0,0225 mol = 22,5 mmol

m anhidrida asetat = n anhidrida asetat x Mr anhidrida asetat

= 0,0225 mol x 102,09 g/mol

= 2,2970 g

$$\rho \text{ anhidrida asetat} = \frac{\text{m anhidrida asetat}}{\text{V anhidrida asetat}}$$

$$\text{V anhidrida asetat} = \frac{\text{m anhidrida asetat}}{\rho \text{ anhidrida asetat}}$$

$$= \frac{2{,}2970 \text{ gram}}{1{,}082 \text{ gram/mL}}$$

$$= 2{,}1229 \text{ mL}$$

c. Piridin

n Piridin = 2,75 kali asam 3-(3,4-diasetoksifenil)akrilat

= 2,75 x 0,0045 mol

= 0,012 mol = 12 mmol

m Piridin = n Piridin x Mr Piridin

= 0,012 mol x 79,10 g/mol

= 0,9492 g

$$V \text{ piridin} = \frac{m \text{ DMAP}}{\rho \text{ DMAP}}$$

$$= \frac{0{,}9492 \text{ gram}}{0{,}9819 \text{ gram/mL}}$$

= 0,966 mL = 1 mL

2. Sintesis senyawa 2-asetoksi-4-(2-klorokarbonilvinil)fenil asetat

a. Asam 3-(3,4-diasetoksifenil)akrilat

$$\text{Asam 3-(3,4-diasetoksifenil)akrilat} = \frac{m \text{ asam 3-(3,4-diasetoksifenil)akrilat}}{Mr \text{ Asam 3-(3,4-diasetoksifenil)akrilat}}$$

$$= \frac{0{,}6 \text{ gram}}{263 \text{ gram/mol}} = 0{,}002 \text{ mol} = 2 \text{ mmol}$$

b. Tionil klorida

n tionil klorida = 5 kali n asam 3-(3,4-diasetoksifenil)akrilat

= 5 x 0,002 mol

= 0,01 mol = 10 mmol

m tionil klorida = n tionil klorida x Mr tionil klorida

= 0,01 mol x 118,97 g/mol

= 1,1897 g

$$\rho \text{ tionil klorida} = \frac{m \text{ tionil klorida}}{V \text{ tionil klorida}}$$

$$V \text{ tionil klorida} = \frac{m \text{ tionil klorida}}{\rho \text{ tionil klorida}}$$

$$= \frac{1,\ 1897 \text{ gram}}{1{,}638 \text{ gram/mL}}$$

= 0,7263 mL

3. Sintesis senyawa 2-asetoksi-4-(2-*o*-tolilkarbamoilvinil)fenil asetat

a. *o*-toililamin

n *o*-toililamin = 1,1 kali n asam 3-(3,4-diasetoksifenil)akrilat

= 1,1 x 0,002 mol

= 0,0022 mol = 2,2 mmol

m *o*-toililamin = n *o*-toililamin x Mr *o*-toililamin

= 0,0022 mol x 107 g/mol

= 0,214 g

$$\rho \text{ } o\text{-toililamin} = \frac{\text{m } o\text{-tolilamin}}{\text{V } o\text{-toililamin}}$$

$$\text{V } o\text{-toililamin} = \frac{\text{m } o\text{-tolilamin}}{\rho \text{ } o\text{-toililamin}}$$

$$= \frac{0{,}214 \text{ gram}}{1{,}00 \text{ gram/mL}} = = 0{,}214 \text{ mL}$$

b. Piridin

n Piridin = 3,8 kali asam 3-(3,4-diasetoksifenil)akrilat

= 3,8 x 0,002 mol

= 0,0076 mol = 7,6 mmol

m Piridin = n Piridin x Mr Piridin

= 0,0076 mol x 79,10 g/mol

= 0,6012 g

$$\text{V piridin} = \frac{\text{m DMAP}}{\rho \text{ DMAP}}$$

$$= \frac{0{,}6012 \text{ gram}}{0{,}9819 \text{ gram/mL}}$$

= 0,6 mL

c. Trietilamin

n trietilamin = 3,8 kali asam 3-(3,4-diasetoksifenil)akrilat

= 3,8 x 0,002 mol

= 0,0076 mol = 7,6 mmol

m trietilamin = n trietilamin x Mr trietilamin

= 0,0076 x 101,19 g/mol

= 0,7690 g

$$V \text{ trietilamin} = \frac{m \text{ trietilamin}}{\rho \text{ trietilamin}}$$

$$= \frac{0{,}7690 \text{ gram}}{0{,}7255 \text{ gram/mL}}$$

= 1 mL

4. Sintesis senyawa 3-(3,4-dihidroksifenil)-*N*-*o*-tolilakrilamida

1. Asam asetat 2-asetoksi-4-(2-*o*-tolilkarbamoilvinil)fenil asetat

n Asam Asetat 2-Asetoksi-4-(2-*o*-tolilkarbamoilvinil)fenil asetat =

$$\frac{m \text{ 2-Asetoksi-4-(2-}o\text{-Tolilkarbamoilvinil)fenil asetat}}{Mr \text{ 2-Asetoksi-4-(2-}o\text{-Tolilkarbamoil-Vinil)fenil asetat}} = \frac{0{,}2 \text{ gram}}{353{,}31 \text{ gram/mol}}$$

= 0,00056 mol = 0,56 mmol

2. Pirolidin

n pirolidin = 17,86 kali 2-asetoksi-5-(2-*o*-tolilkarbamoilvinil)fenil asetat

= 17,86 x 0,00056 mol

= 0,01 mol = 10 mmol

m pirolidin = n pirolidin x Mr pirolidin

= 0,01 mol x 71,11 g/mol

= 0,7111 g

$$V_{\text{pirolidin}} = \frac{m_{\text{pirolidin}}}{\rho_{\text{pirolidin}}}$$

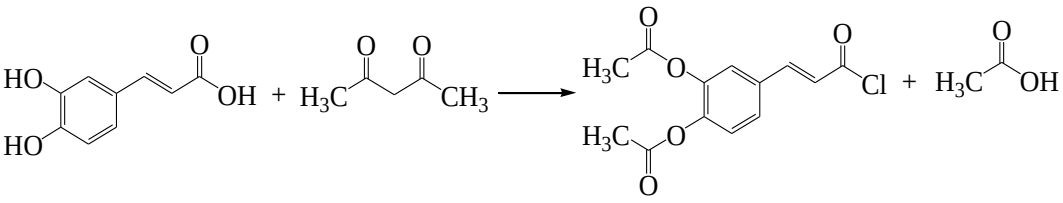
$$= \frac{0{,}7111 \text{ gram}}{0{,}866 \text{ gram/mL}}$$

$$= 0{,}8 \text{ mL}$$

Lampiran 3. Perhitungan Rendemen

1. Sintesis senyawa asam 3-(3,4-diasetoksifenil)akrilat

Persamaan reaksi :



4,5 mmol 22,5 mmol 4,5 mmol

Bobot teoritis = mol produk x Mr asam 3-(3,4-dihidroksifenil)akrilat

= 4,5 mmol x 264,15 g/mol

= 0,0045 mol x 264,15 g/mol

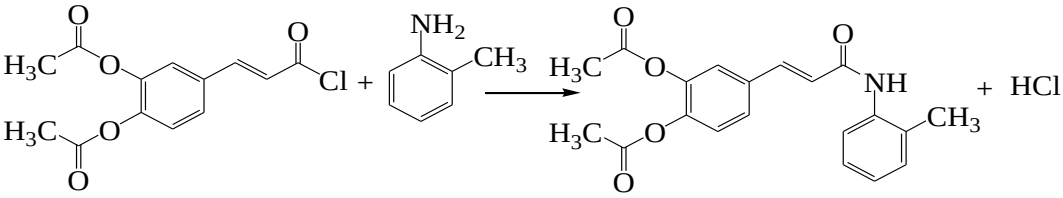
= 11887 g

Rendemen $= \frac{\text{bobot praktek}}{\text{bobot teori}}$ x 100%

$= \frac{0{,}6856}{1{,}1887}$ x 100% = 57,68%

2. Sintesis senyawa 2-asetoksi-4-(2-*o*-tolilkarbamoilvinil)fenil asetat

Persamaan reaksi :



2 mmol 4 mmol 2 mmol

Bobot teoritis = mol produk x Mr senyawa tahap III

= 2 mmol x 353,31 g/mol

= 0,002 mol x 353,31 g/mol

= 0,7066 g

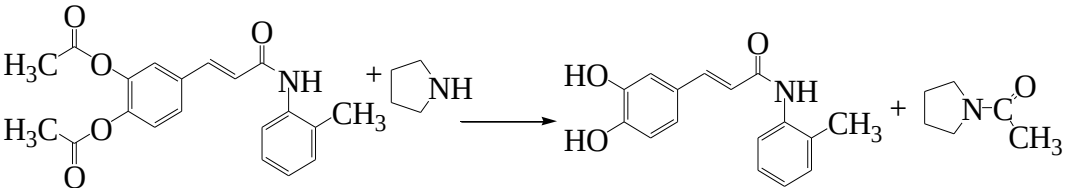
Rendemen $= \frac{\text{bobot praktek}}{\text{bobot teori}}$ x 100%

$= \frac{0,3}{0,7066}$ x 100%

= 42,46%

3. Sintesis senyawa 3-(3,4-dihidroksifenil)-*N*-*o*-tolilakrilamida

Persamaan reaksi :



0,56 mmol 10 mmol 0,56 mmol

Bobot teoritis = mol produk x Mr senyawa tahap IV

= 0,56 mmol x 221,32 g/mol

= 0,00056 mol x 221,32 g/mol

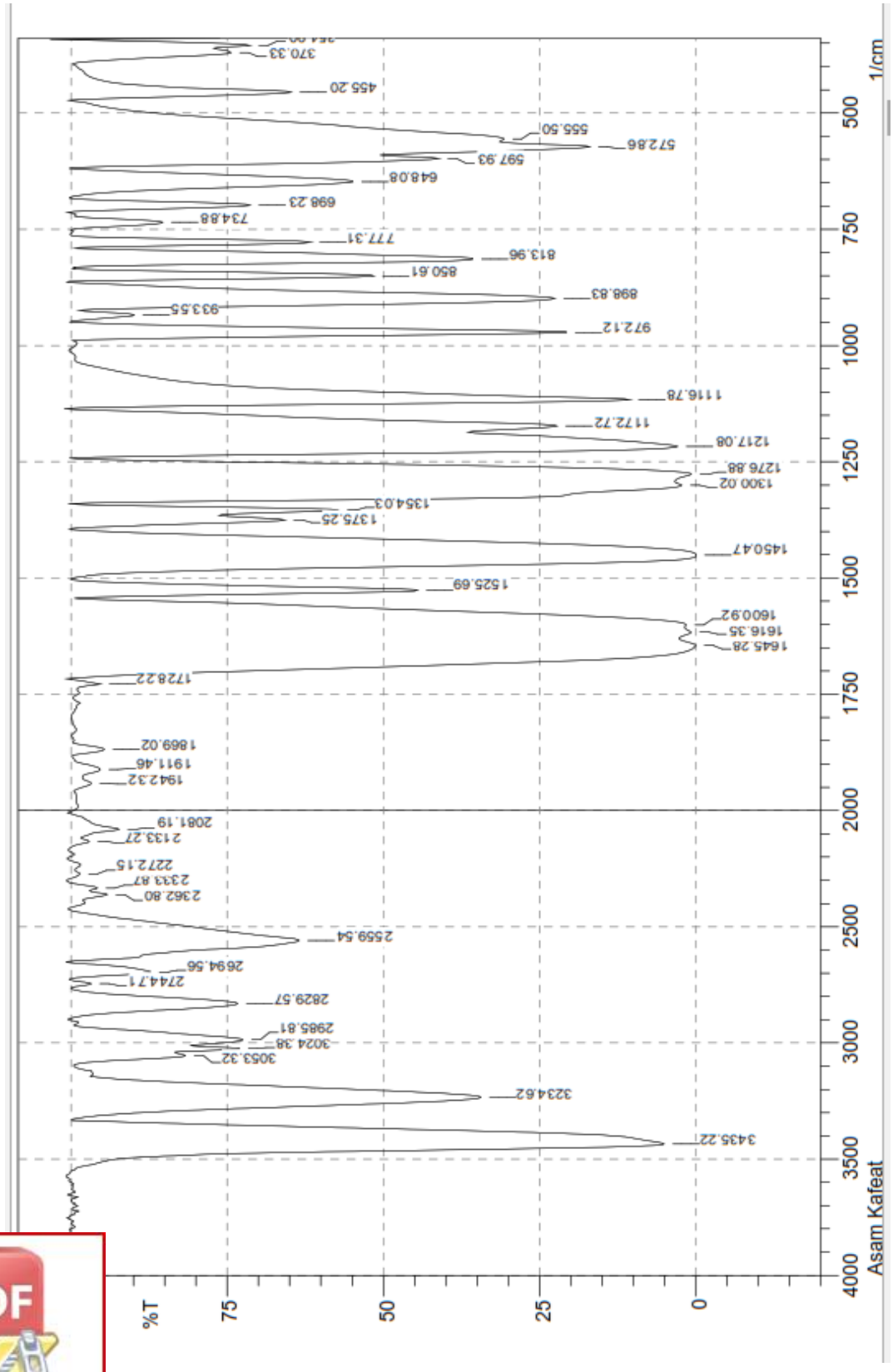
= 0,1239 g

Rendemen $= \frac{\text{bobot praktek}}{\text{bobot teori}}$ x 100%

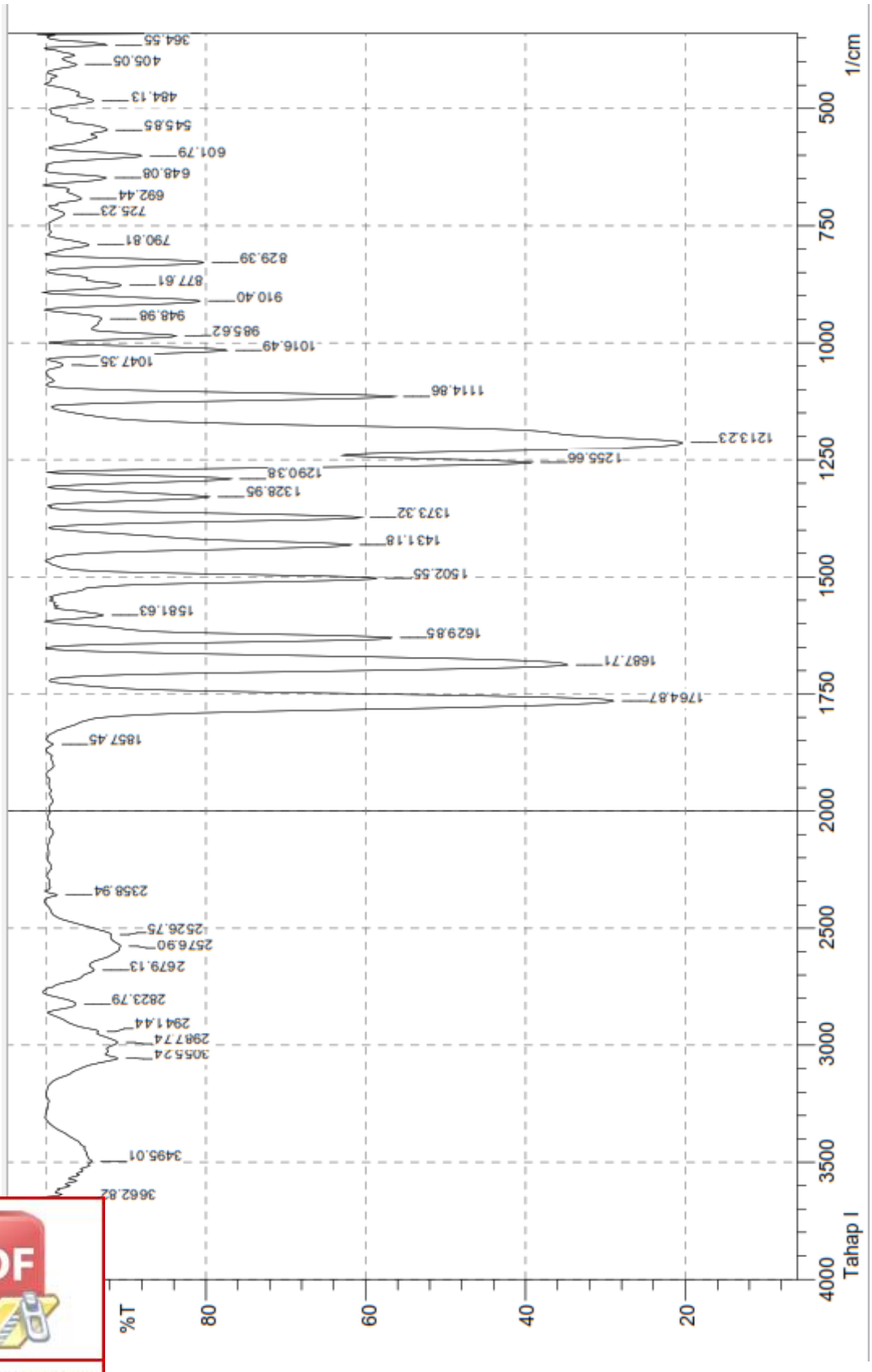
$= \frac{0,0787}{0,1239}$ x 100%

= 63,51%

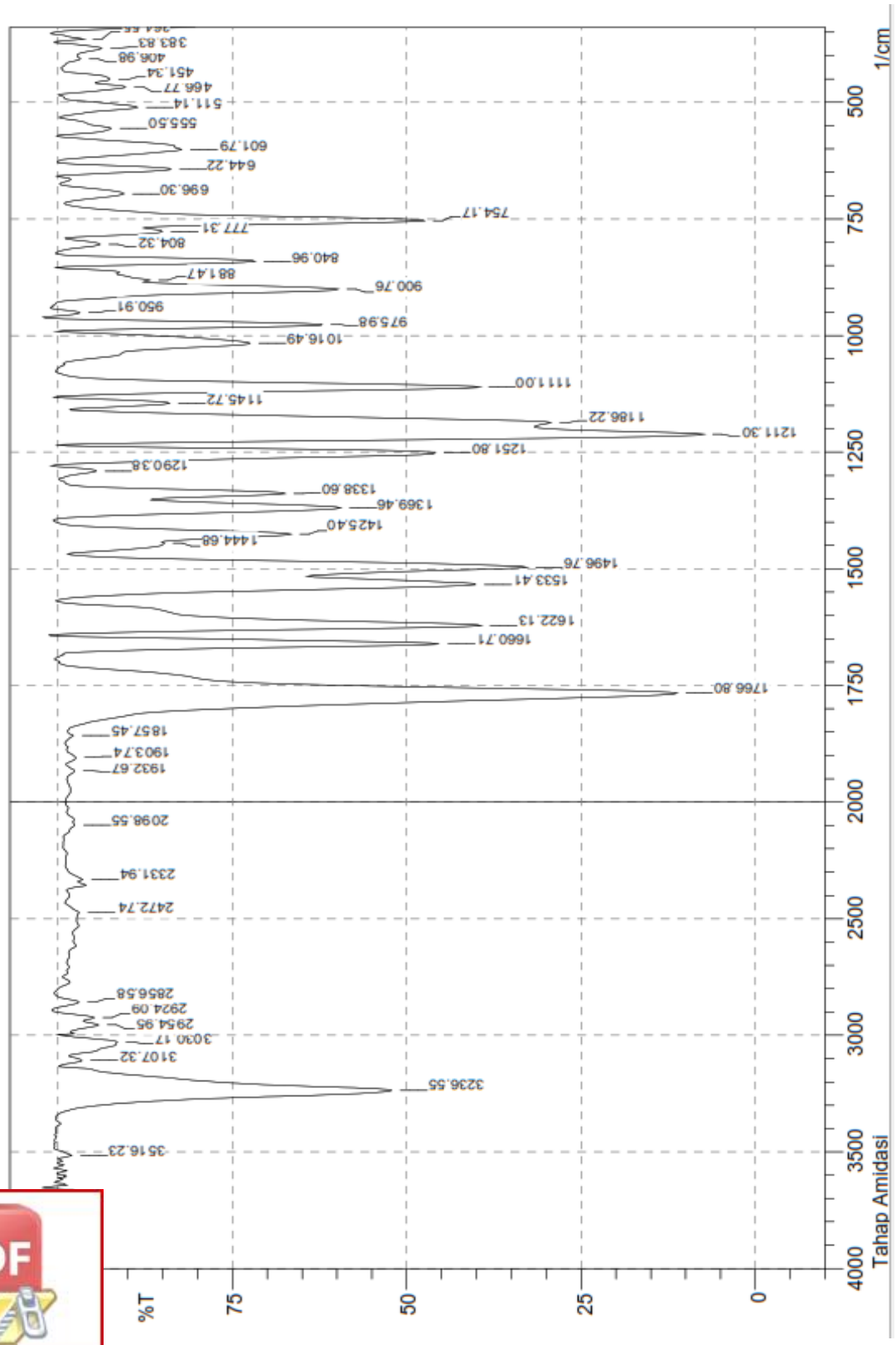
Lampiran 4. Spektrum FT-IR Asam 3-(3,4-dihidroksifenil)akrilat



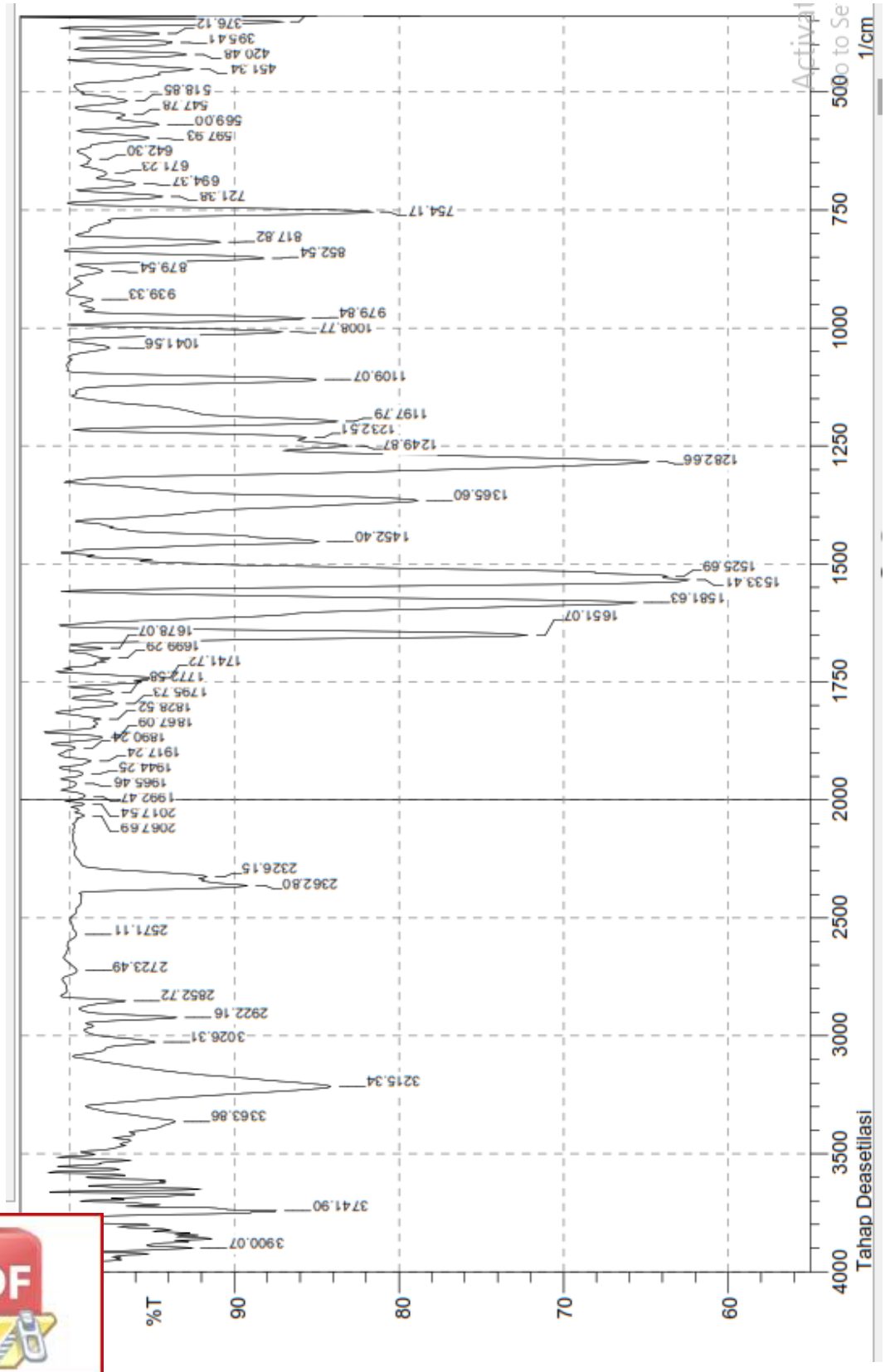
Lampiran 5. Spektrum FT-IR Asam 3-(3,4-diasetoksifenil)akrilat



Lampiran 6. Spektrum FT-IR 2-asetoksi-4-(2-*o*-klorokarbamoilvinil)fenil asetat



Lampiran 7. Spektrum FT-IR 3-(3,4-diasetoksifenil)-*N*-*o*-tolilakrilamida



Lampiran 8. Hasil Uji Bioaktivitas Senyawa 3-(3,4-dihidroksifenil)-*N*-*o*-tolilakrilamida terhadap Sel HeLa

Panjang Gelombang/nm	Media	Media+Sel	Cisplatin	DMSO 3,50%	Konsentrasi Sampel (µg/mL)			
					100,00	10,00	1,00	0,10
570	0,4138	0,6907	0,5239	0,6356	0,4654	0,7153	0,7224	0,7208
	0,4111	0,6905	0,5122	0,6264	0,4607	0,7053	0,7167	0,7132
600	0,5054	0,2643	0,4926	0,2907	0,451	0,1963	0,2025	0,2037
	0,5028	0,2731	0,4849	0,2977	0,4624	0,1979	0,1927	0,2047
Corrected Absorbance	- 0,0917	0,5181	0,1230	0,4366	0,0920	0,6106	0,6116	0,6087
		0,5091	0,1190	0,4204	0,0899	0,5990	0,6165	0,6004

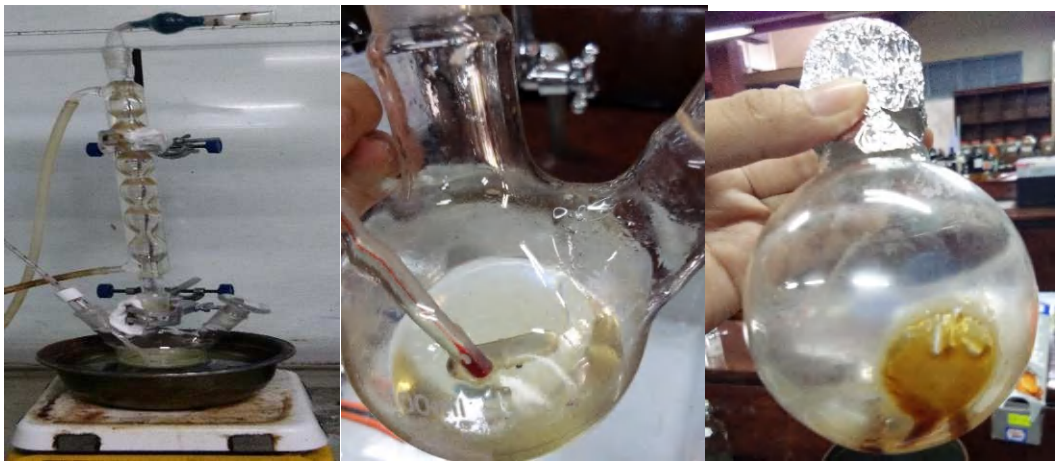
Nilai IC 50 SP-KAF : 89,76 µg/mL

Sampel	**IC_{50} (µg/mL)**
SP-KAF	89,76

Lampiran 9. Dokumentasi Hasil Penelitian

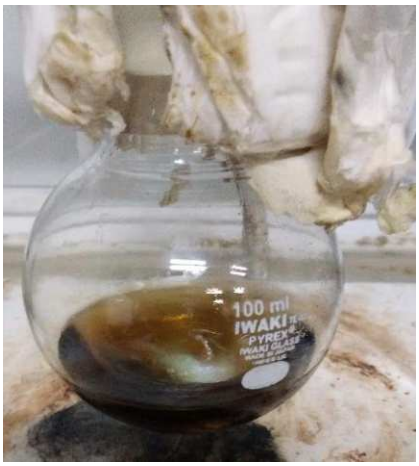
(a) (b) (c)

Tahap sintesis senyawa 1(a) pengadukan campuran reaksi (prekursor + piridin + anhidrida asetat) pada suhu ruang. (b) Campuran reaksi ditambahkan akuades. (c) Kristal senyawa tahap I.



(a) (b) (c)

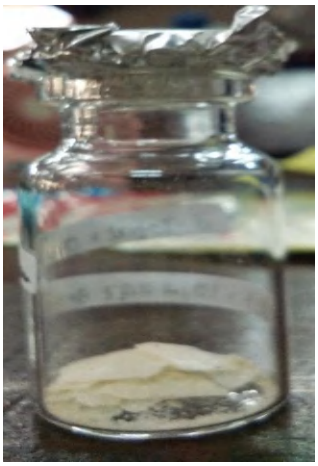
Tahap klorinasi (a) Proses refluks pada tahap sintesis senyawa 2 (b) hasil campuran reaksi setelah refluks (c) hasil yang diperoleh setelah evaporasi.



Proses penambahan amina *o*-tolilamina

Proses pencucian

Kristal setelah evaporasi



Kristal senyawa 3

Kristal senyawa 4

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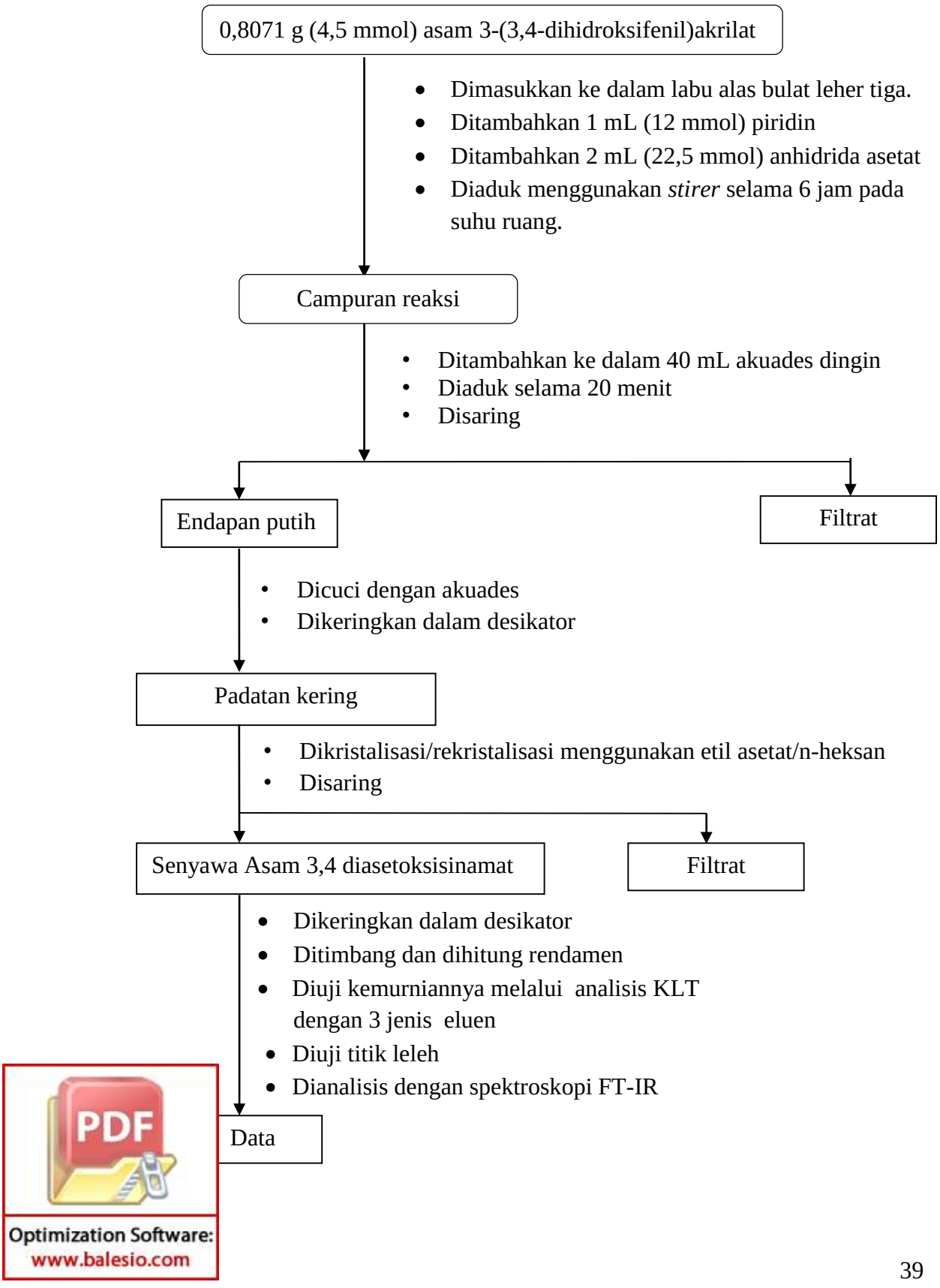
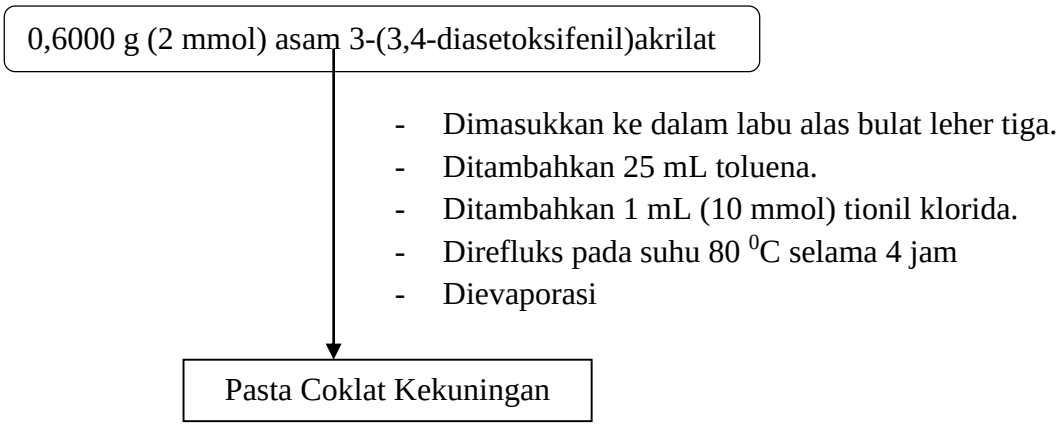
Lampiran 1. Bagan Prosedur Penelitian

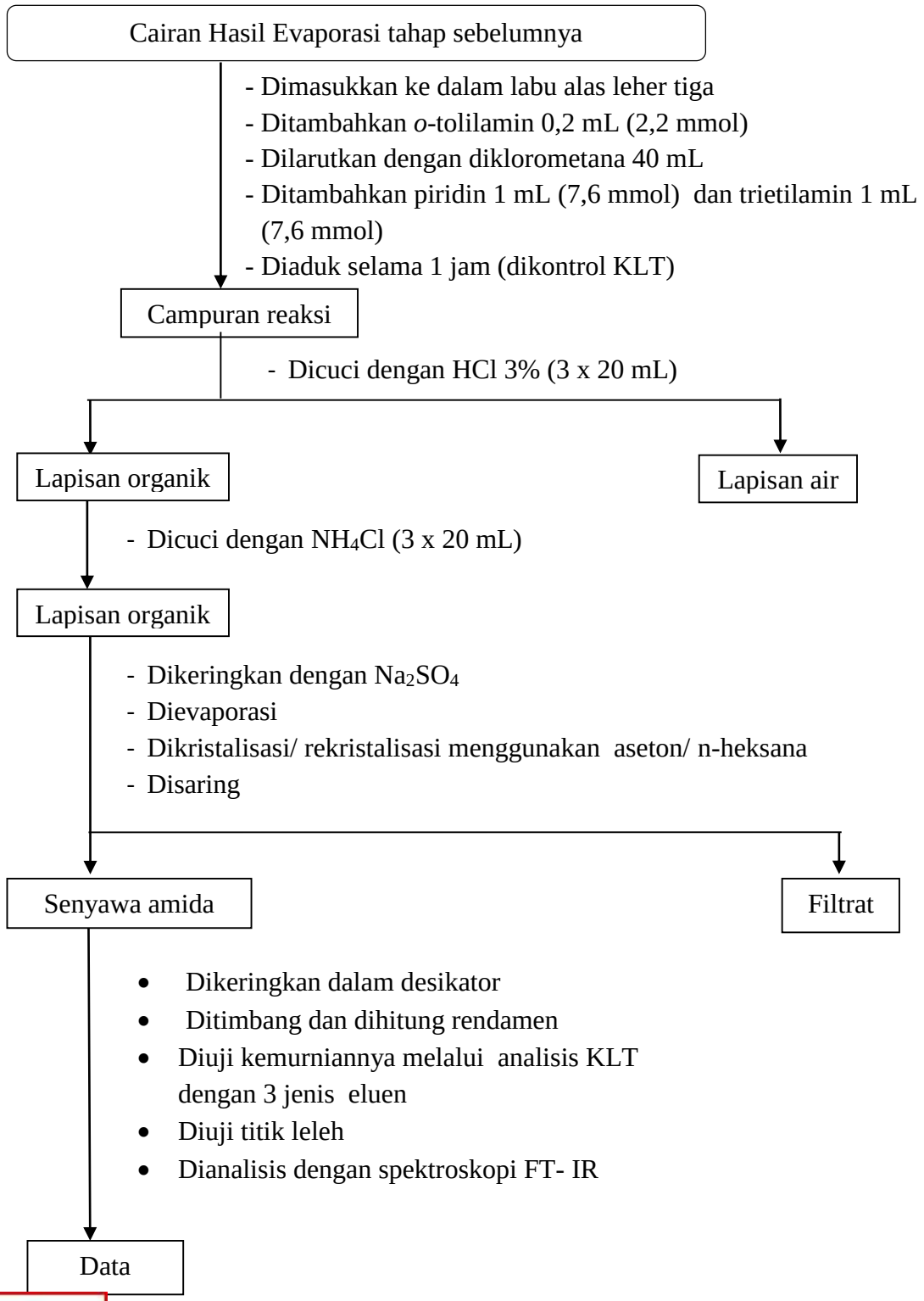
A. Prosedur Asetilasi (Tahap 1)



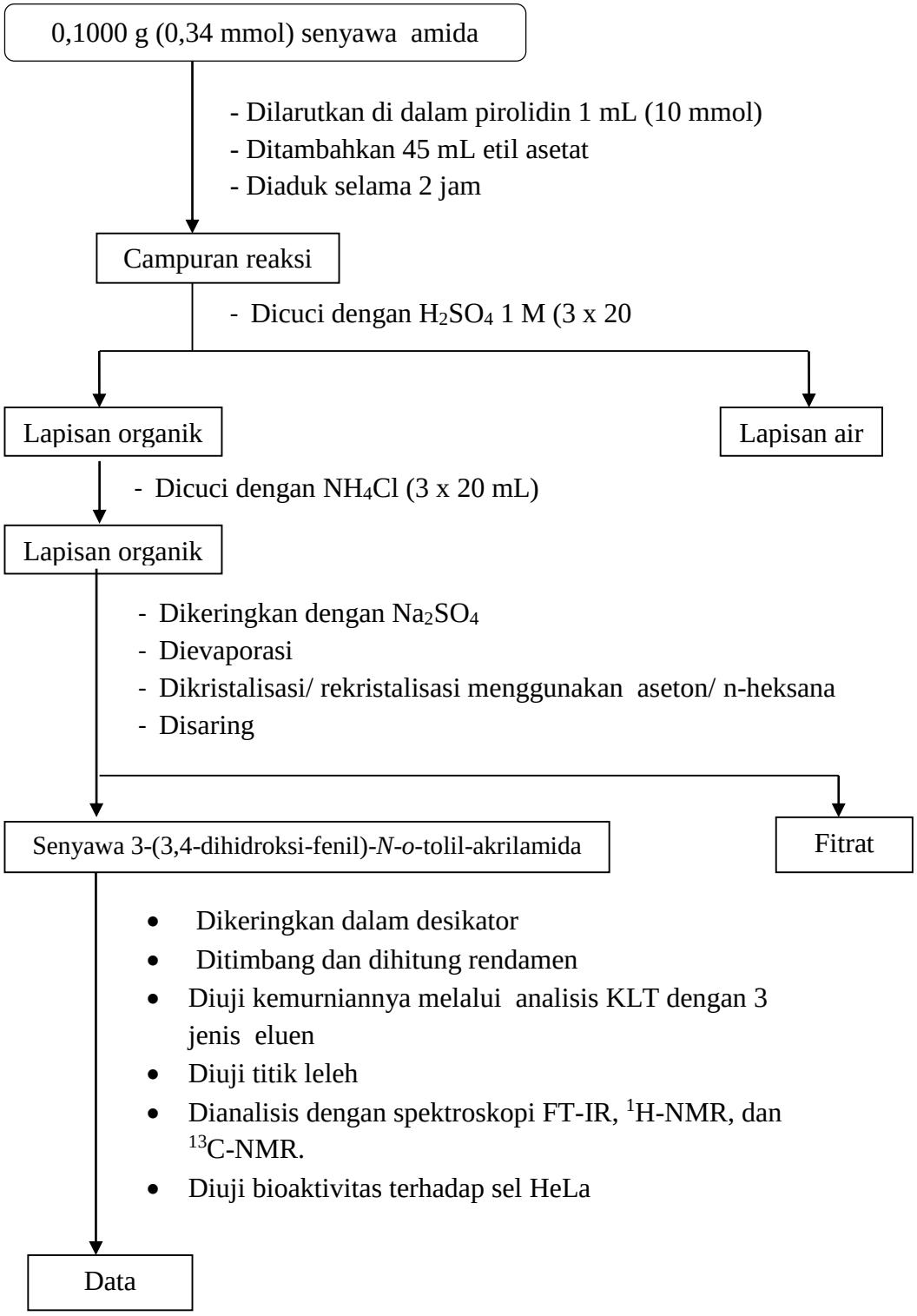
B. Prosedur Klorinasi (Tahap II)



C. Tahap Amidasi (Tahap III)



D. Tahap Deasetilasi (Tahap IV)



E. Uji Bioaktivitas Senyawa Terhadap Sel HeLa