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LAMPIRAN 1

CERTIFICATE OF ANALYSIS

SIGMA

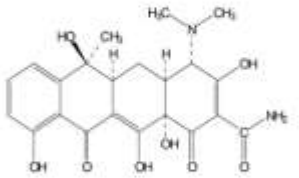
3050 Spruce Street
Saint Louis, Missouri 63103 USA
Telephone 800-325-5832 • (314) 771-5765
Fax (314) 286-7828
e-mail: techserv@sial.com
sigma-aldrich.com

ProductInformation

TETRACYCLINE

Product Number **T 3258**

CAS NUMBER: 60-54-8
SYNONYMS: Abramycin; Abricycline; Agromicina; Ambramicina; Ambramycin; Amycin; Biotetra; Bristacicin Alpha; Cefracycline Suspension; Criseocicina; Democracin; Deschlorobiomycin; Hostacyclin; Liquamycin; 6-methyl-1, 11-dioxy-2-naphthacenecarboxamide; Neocycline; Oletetrin; Omegamycin; Ortycycline; Panmycin; Polycycline; Purocycline; Robitet; Sanclomycine; Sigmamycin; Steclin; Tetrabon; Tetracycline; Tetracycline I; Tetracycline II; SK-Tetracycline, Tetracyn, Tetradecin, Tetravenine, Tsiklomitsin, T-125, Veracin, Vetacyclinum



Product Description
Molecular formula: $C_{22}H_{24}N_2O_8$
Formula weight: 444.4 (anhydrous)
pK_a: 8.3, 10.2 (50% aqueous DMF)[3]
λmax: 220, 268, 355 nm ($E^{1\%}_{1cm}$ = 13, 18.04, 13.32) for free base in 0.1 M HCl[1]
Specific Rotation: -257.9° (0.1 M HCl at 25 °C)[1]
-239° (methanol at 25 °C)[1]

Tetracyclines possess a wide range of antimicrobial activity against gram-positive and gram-negative bacteria. The bacterial ribosome is the site of action of tetracyclines. Access to the ribosomes of gram-negative bacteria is obtained by passive diffusion through hydrophilic pores in the outer cell membrane and then by an energy-dependent active transport system that pumps all tetracyclines through the inner cytoplasmic membrane. This active transport system may require a periplasmic protein carrier. Tetracyclines bind specifically to 30S ribosomes and appear to inhibit protein synthesis by preventing access of aminoacyl tRNA to the acceptor site on the mRNA-ribosome complex. The inhibitory effects of the tetracyclines can be reversed by washing. This suggests that the reversibly bound antibiotic rather than the small portion of irreversibly bound drug is responsible for the antibacterial action.[3]

Preparation Instructions
Tetracycline as a free base is soluble 1 in 2500 of water and 1 in 50 of alcohol; soluble in methanol, but sparingly soluble in acetone; freely soluble in dilute acids and, with decomposition, in solutions of alkali hydroxides, but practically insoluble in chloroform and ether.[2]

The product is soluble in 1 M HCl with heating (50 mg/mL), yielding a clear to slightly hazy yellow to orange-brown solution. Tetracycline undergoes reversible epimerizations in solution to the less active 4-epitetracycline; the degree of epimerization is dependent on pH and is greatest at a pH of approximately 3.

Epimerization has been observed to be the dominant degradation reaction at pH 2.5 to 5. Formation of anhydrotetracycline occurs at a very low pH and oxidation to isotetracycline occurs at alkaline pH. Tetracycline's potency is reduced in solutions with a pH below 2. The pH of a 1% suspension in water may range from 3.0 to 7.0.[3]

Storage/Stability
This product should be stored in the freezer. The product will darken in moist air when exposed to strong sunlight.[2]

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JWM 7/8/2003

Gambar V.1 *Certificate of analysis tetrasiklin*

LAMPIRAN 1
(LANJUTAN)

Certificate of Analysis

1.00983.4000 Ethanol absolute for analysis EMSURE® ACS,ISO,Reag. Ph Eur
Batch K49462683

	Batch Values	
Purity (GC)	≥ 99.9	%
Identity (IR)	conforms	
Appearance	conforms	
Color	≤ 10	Hazen
Solubility in water	conforms	
Acidity or alkalinity	≤ 20	ppm
Titrable acid	≤ 0.0002	meq/g
Titrable base	≤ 0.0002	meq/g
Density (d 20 °C/20 °C)	0.790 – 0.793	
UV absorption	conforms	
Aldehydes (as Acetaldehyd)	≤ 0.001	%
Fusel oils	conforms	
Substances reducing potassium permanganate (as O)	≤ 0.0002	%
Carbonyl compounds (as CO)	≤ 0.003	%
Readily carbonizable substances	conforms	
Acetone (GC)	≤ 0.001	%
Ethylmethylketone (GC)	≤ 0.02	%
Isoamyl alcohol (GC)	≤ 0.05	%
2-Propanol (GC)	≤ 0.01	%
Higher alcohols (GC)	≤ 0.01	%
Volatile impurities (GC) (Acetaldehyde and Acetal)	≤ 10	ppm
Volatile impurities (GC) (Benzene)	≤ 2	ppm
Volatile impurities (GC) (Methanol)	≤ 100	ppm
Volatile impurities (GC) (Total of other impurities)	≤ 300	ppm
Volatile impurities (GC) (disregard limit)	≤ 9	ppm
Chloride (Cl)	≤ 0.3	ppm
Nitrate (NO_3)	≤ 0.3	ppm
Phosphate (PO_4)	≤ 0.3	ppm
Sulfate (SO_4)	≤ 0.3	ppm
Ag (Silver)	≤ 0.000002	%
Al (Aluminium)	≤ 0.00005	%
As (Arsenic)	≤ 0.000002	%
Au (Gold)	≤ 0.000002	%
Ba (Barium)	≤ 0.00001	%
Be (Beryllium)	≤ 0.000002	%
Bi (Bismuth)	≤ 0.000002	%
Ca (Calcium)	≤ 0.00005	%
Cd (Cadmium)	≤ 0.000005	%
Co (Cobalt)	≤ 0.000002	%

Merck KGaA, Frankfurter Straße 250, 64293 Darmstadt (Germany); +49 6151 72-0
EMD Millipore Corporation - a subsidiary of Merck KGaA, Darmstadt, Germany
400 Summit Drive, Burlington, MA 01803, USA. Phone +1 (781) 533-6000

Page 1 of 2

Dipindai dengan CamScanner

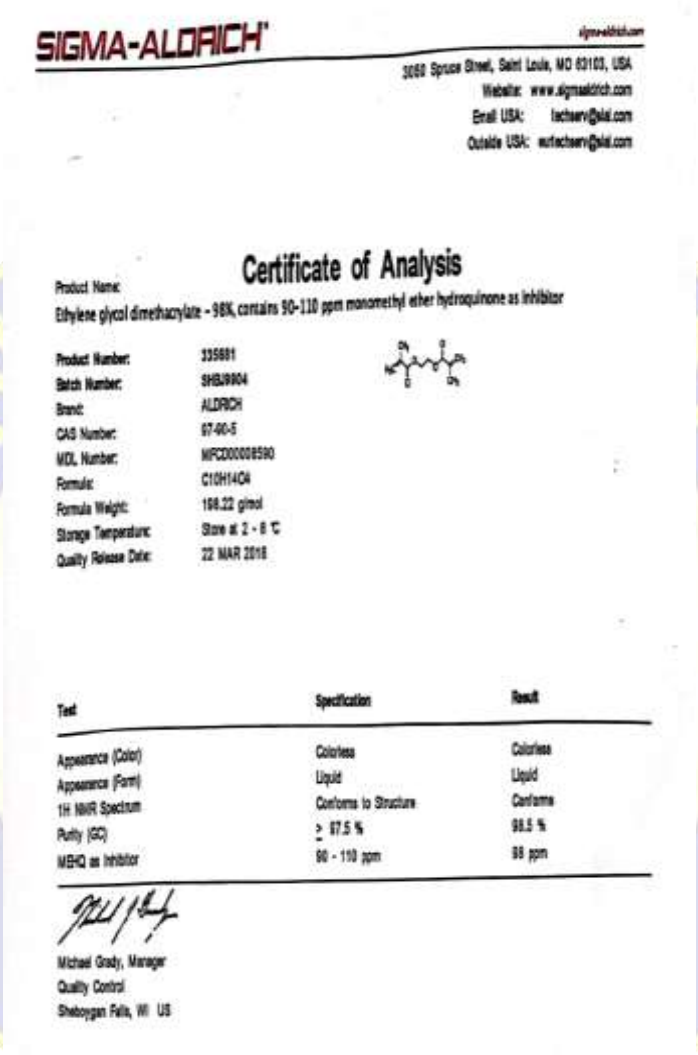
Certificate of Analysis

1.00983.4000 Ethanol absolute for analysis EMSURE® ACS,ISO,Reag. Ph Eur
Batch K49462683

Cr (Chromium)	≤ 0.000002	%
Cu (Copper)	≤ 0.000002	%
Fe (Iron)	≤ 0.00001	%
Ga (Gallium)	≤ 0.000002	%
In (Indium)	≤ 0.000002	%
Li (Lithium)	≤ 0.000002	%
Mg (Magnesium)	≤ 0.00001	%
Mn (Manganese)	≤ 0.000002	%
Mo (Molybdenum)	≤ 0.000002	%
Ni (Nickel)	≤ 0.000002	%
Pb (Lead)	≤ 0.00001	%
Pt (Platinum)	≤ 0.000003	%
Sb (Antimony)	≤ 0.000002	%
Sn (Tin)	≤ 0.00001	%
Ti (Titanium)	≤ 0.000002	%
Tl (Thallium)	≤ 0.000002	%
V (Vanadium)	≤ 0.000002	%
Zn (Zinc)	≤ 0.00001	%
Zr (Zirconium)	≤ 0.000002	%
Evaporation residue	≤ 0.0005	%

Gambar V.2 *Certificate of analysis* etanol

LAMPIRAN 1
(LANJUTAN)



SIGMA-ALDRICH

3050 Spruce Street, Saint Louis, MO 63103, USA
Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Certificate of Analysis

Product Name:
Ethylene glycol dimethacrylate - 98%, contains 90-110 ppm monomethyl ether hydroquinone as inhibitor

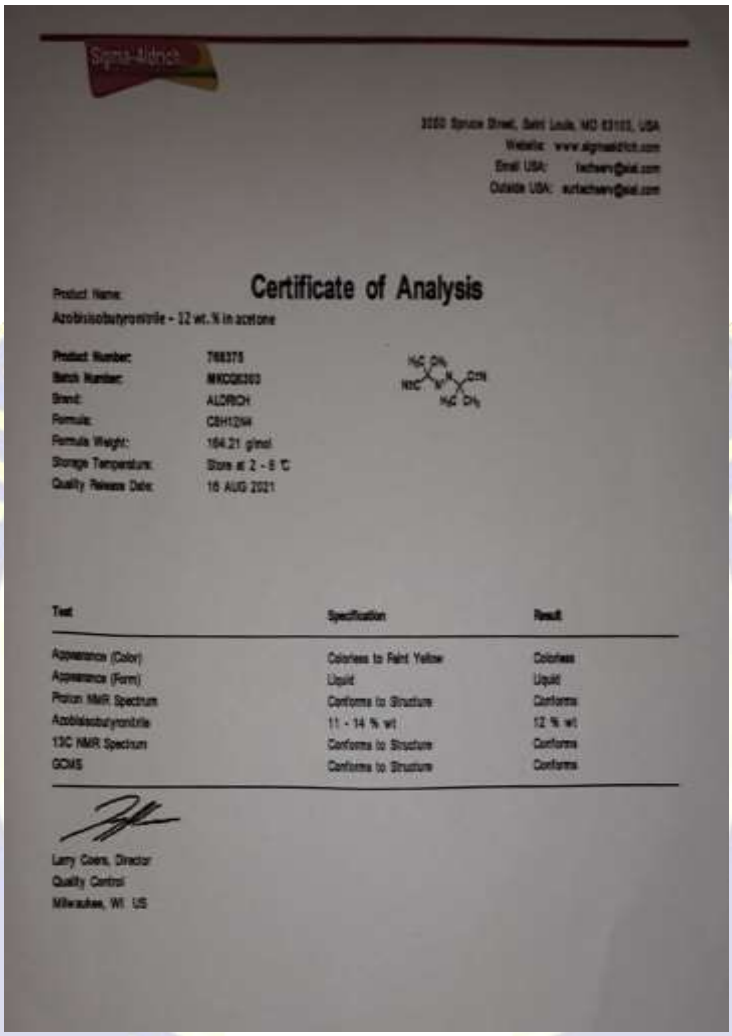
Product Number:	335681
Batch Number:	SHBJ9904
Brand:	ALDRICH
CAS Number:	97-90-5
MDL Number:	MFCD00008590
Formula:	C10H14O4
Formula Weight:	198.22 g/mol
Storage Temperature:	Store at 2 - 8 °C
Quality Release Date:	22 MAR 2018

Test	Specification	Result
Appearance (Color)	Colorless	Colorless
Appearance (Form)	Liquid	Liquid
1H NMR Spectrum	Conforms to Structure	Conforms
Purity (GC)	≥ 97.5 %	98.5 %
MEHQ as Inhibitor	90 - 110 ppm	99 ppm

Michael Grady, Manager
Quality Control
Sheboygan Falls, WI US

Gambar V.3 *Certificate of analysis* EGDMA

LAMPIRAN 1
(LANJUTAN)



Certificate of Analysis

Product Name:
Azobisisobutyronitrile - 12 wt. % in acetone

Product Number:	788378
Batch Number:	MKCQ6303
Brand:	ALDRICH
Formula:	$C_8H_{12}N_4$
Formula Weight:	164.21 g/mol
Storage Temperature:	Store at 2 - 8 °C
Quality Release Date:	16 AUG 2021

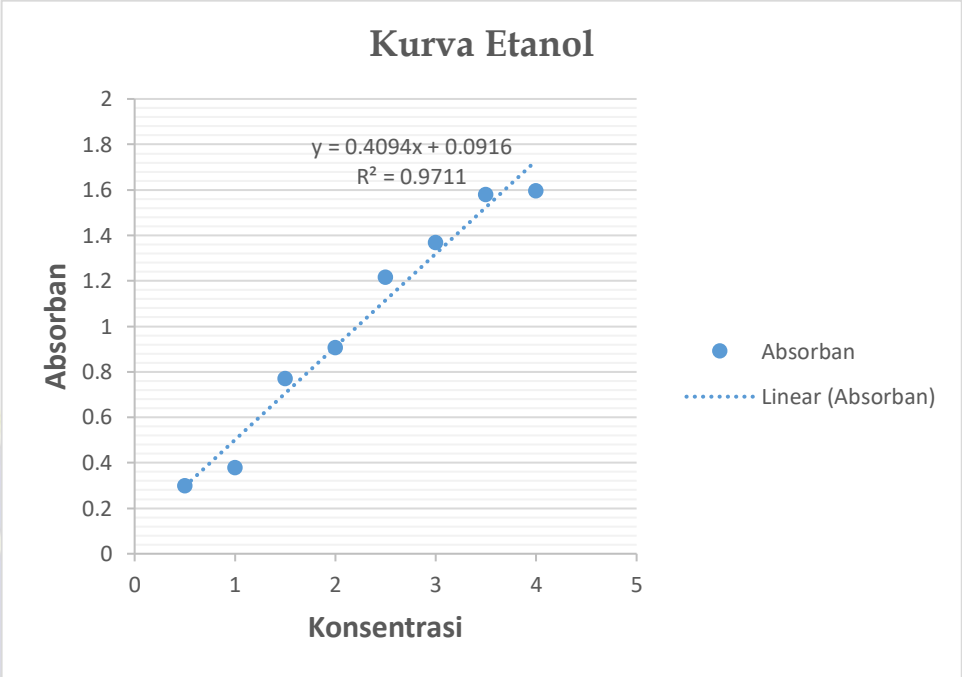
Test	Specification	Result
Appearance (Color)	Colorless to Faint Yellow	Colorless
Appearance (Form)	Liquid	Liquid
Proton NMR Spectrum	Conforms to Structure	Conforms
Azobisisobutyronitrile	11 - 14 % wt	12 % wt
13C NMR Spectrum	Conforms to Structure	Conforms
GCMS	Conforms to Structure	Conforms

Larry Coers, Director
Quality Control
Milwaukee, WI US

Gambar V.4 *Certificate of analysis* AIBN

LAMPIRAN 2

GRAFIK KURVA KALIBRASI PADA PELARUT ETANOL



Gambar V.5 Grafik Kurva Kalibrasi Dari Larutan Standar Tetrasiklin Pada Pelarut Etanol

LAMPIRAN 3

DATA PERHITUNGAN UJI PRESISI

Tabel V.1

Data Perhitungan %RSD Uji Presisi Untuk Pelarut Etanol

Konsentrasi (ppm)	Absorban	X
1	0,353	0,985
1	0,354	0,987
1	0,355	0,989
1	0,354	0,987
1	0,355	0,989
1	0,356	0,991
	Σ	5,928
	$\bar{X}$	0,988

SD = $\frac{n.\Sigma x^2-(\Sigma x)^2}{n\,(n-1)}$

SD = $\frac{6.5,928-(35,141)^2}{6\,(6-1)}$

SD = $\frac{35,568-35,141}{30}$

SD = $\frac{0,427}{30} = 0,014$

%RSD = $\frac{SD}{\bar{x}} x100$

%RSD = $\frac{0,014}{0,988} x100$ = 1,417%

% Ketelitian alat = $100\% - \frac{SD}{x}$ =$100\% - \frac{0,014}{0,988}$

Ketelitian alat = 99,986%

LAMPIRAN 4

DATA PERHITUNGAN UJI AKURASI

Tabel V.2

Perhitungan Data Akurasi Pada Pelarut Etanol

Pelarut	Konsentrasi (ppm)	Absorban	Kadar hasil (ppm)	%Recovery
Etanol	3,2	1,27	3,32	103%
	4	1,594	4,11	102%
	4,8	1,912	4,89	101,8%

Konsentrasi Hasil hitung $=\frac{absorban+0,091}{0,409}$

• Konsentrasi 3,2 ppm

Konsentrasi Hasil Hitung$=\frac{1,27+0,091}{0,409}=3,32$

%Recovery$=\frac{3,32}{3,2}$ x 100% = 103%

• Konsentrasi 4 ppm

Konsentrasi Hasil Hitung$=\frac{1,594+0,091}{0,409}=4,11$

%Recovery$=\frac{4,11}{4}$ x 100% = 102%

• Konsentrasi 4,8 ppm

Konsentrasi Hasil Hitung$=\frac{1,912+0,091}{0,409}=4,89$ %Recovery$=\frac{4,89}{4,8}$ x 100% = 101,8%

LAMPIRAN 5

PENENTUAN LOD dan LOQ

Tabel V.3

Hasil Penentuan Nilai LOD dan LOQ Larutan Tetrasiklin dalam Pelarut Etanol

X (ppm)	Y	Yi	Y-Yi	(Y-Yi)²
0,5	0,297	0,295	0,002	0,000004
1	0,378	0,5	-0,462	0,213
1,5	0,768	0,704	0,064	0,0040
2	0,904	0,909	-0,005	0,00025
2,5	1,124	1,13	0,011	0,00012
3	1,368	1,318	0,05	0,0025
3,5	1,579	1,522	0,057	0,0032
4	1,594	1,727	-0,133	0,017
			Σ	0,239

Persamaan garis regresi Y= 0,409x + 0,091

Contoh perhitungan Yi (Absorban hitungan)

Y = bx + a

Y = 0,409x + 0,091

Y = 0,409x . 1 ppm + 0,091

Y = 0,409 + 0,091 Y = 0,5

$$SB = \sqrt{\frac{\Sigma(Y-Yi)^2}{n-2}} = \sqrt{\frac{0,239}{8-2}} = 0,081$$

$$LOD = \frac{3\ x\ SB}{Slope} = \frac{3\ x\ 0,081}{0,409} = 0,594\ ppm \qquad LOQ = \frac{10\ x\ SB}{Slope} = \frac{10\ x\ 0,081}{0,409} = 1,980\ ppm$$

LAMPIRAN 6

HASIL SINTESIS DAN EKSTRAKSI

Tabel V.4

Hasil sintesis polimer MIP dan NIP

Polimer (cetakan:monomer:pengikat)	Massa (gram)
MIP	4.7529
NIP	4.3264

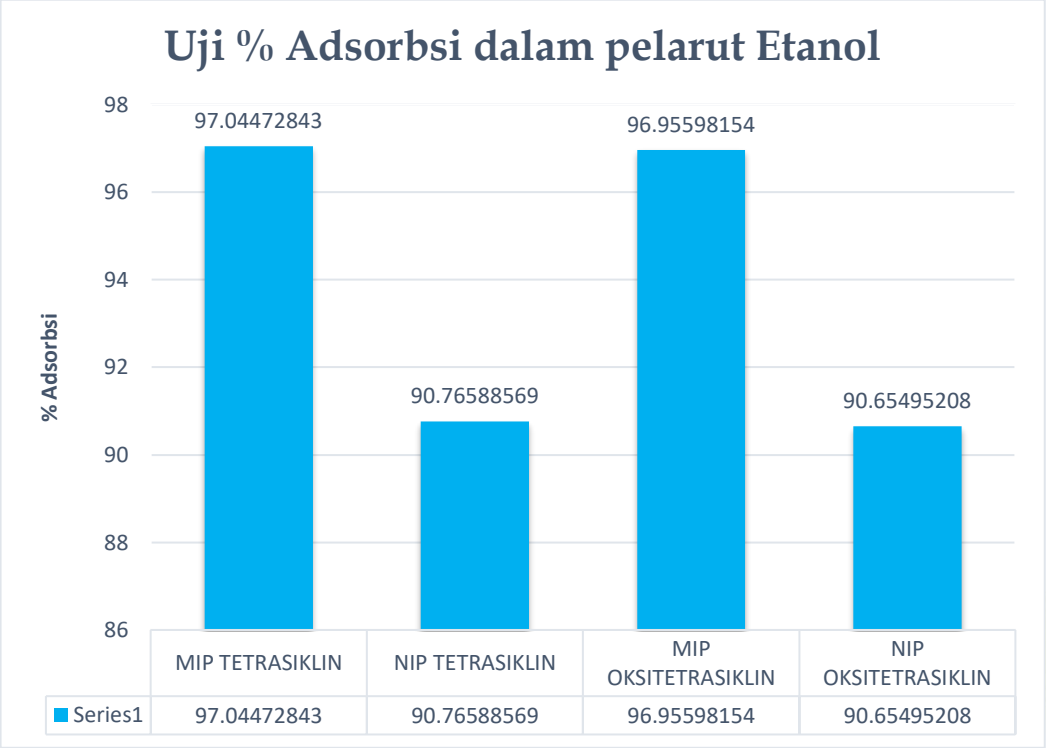
Tabel V.5

Hasil ekstraksi cetakan MIP

Panjang Gelombang	**Absorbansi**		
	MIP Sebelum Ekstraksi	**MIP 3x Ekstraksi**	**MIP 4x Ekstraksi**
380	0.449	0.016	0.001

LAMPIRAN 7

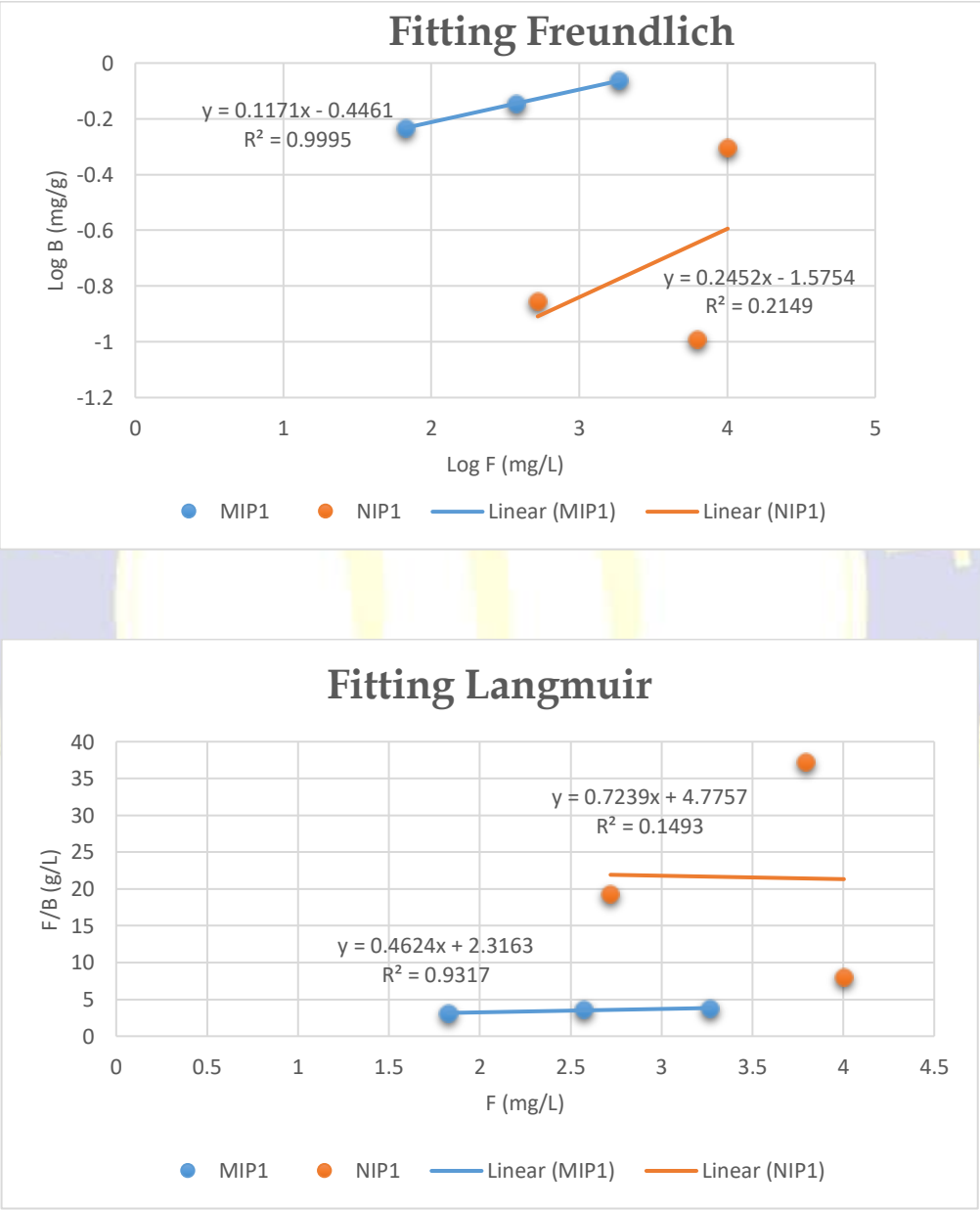
HASIL KEMAMPUAN ADSORPSI



Gambar V.6 Grafik perbandingan persen MIP dan NIP

LAMPIRAN 8

KURVA ISOTERM FREUNDLICH DAN LANGMUIR



Gambar V.7 Hasil kapasitas adsorpsi MIP dan NIP

LAMPIRAN 8
(LANJUTAN)

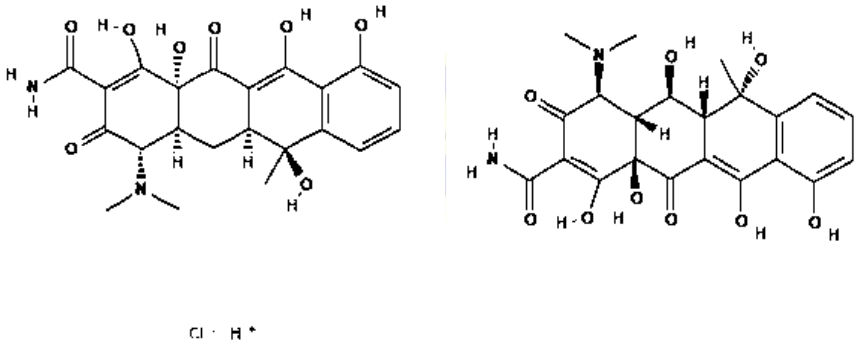
Tabel V.6

Hasil Evaluasi Kapasitas Adsorpsi Menggunakan Isoterm Freundlich dan Langmuir

Polimer	Isoterm Freundlich				Isoterm Langmuir		
	$\frac{1}{n}$	n	K_F (mg/g)	R^2	Q_m	K_e	R^2
MIP	0,1171	8,5397	0,35801	0,9995	2,16262	0,19962	0,9317
NIP	0,2452	4,0783	0,02658	0,2149	1,38140	0,15157	0,1493

LAMPIRAN 9

PERBEDAAN STRUKTUR KIMIA dan HASIL SELEKTIVITAS



(a) (b)

Gambar V.8

Perbedaan struktur kimia (a) tetrasiklin (b) oksitetrasiklin

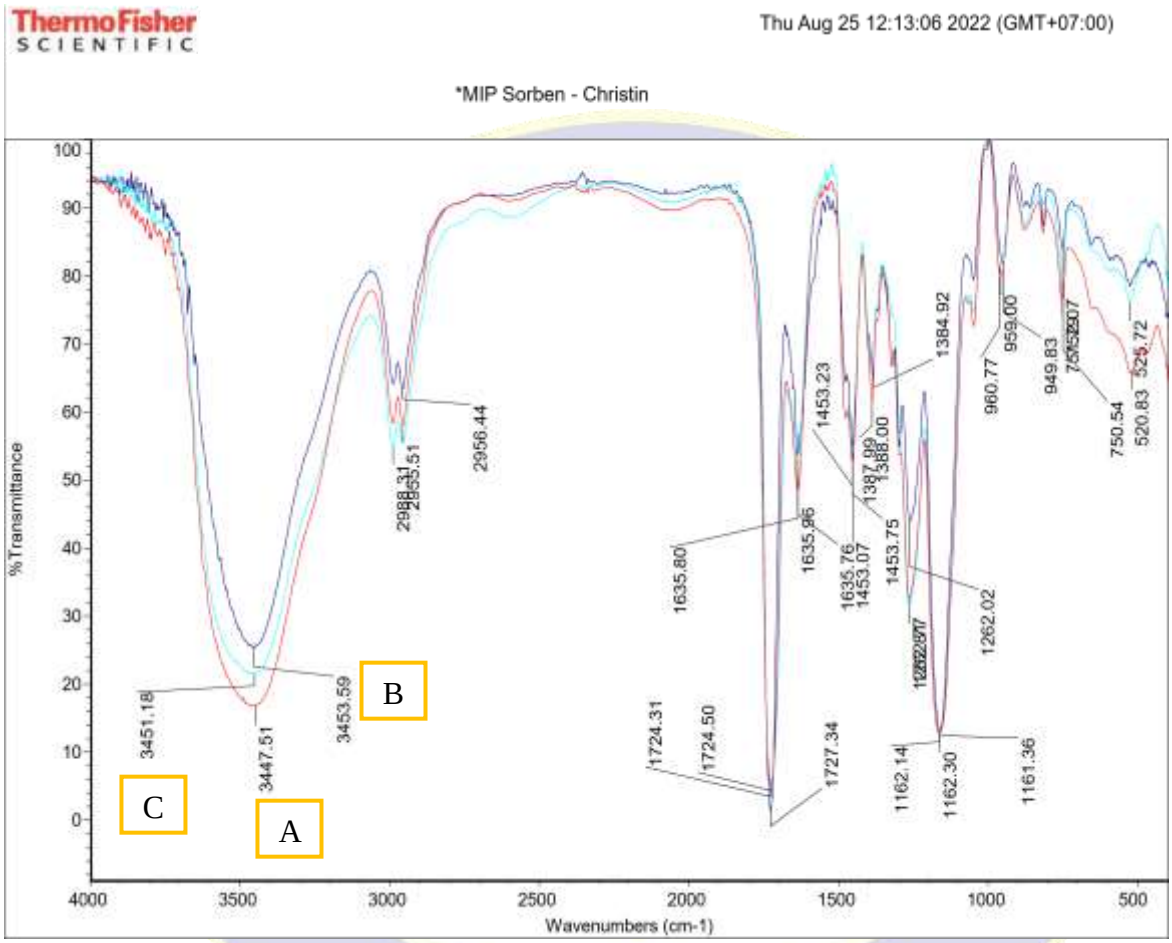
Tabel V.7

Hasil penentuan selektivitas

Analit		**Tetrasiklin**	**Oksitetrasiklin**
K_D (ml/g)	MIP	16,4189	15,9256
	NIP	4,91470	4,85042
Imprinting Factor		3,34077	3,28335

LAMPIRAN 10

SPEKTRUM FTIR



Gambar V.9

Spektrum Inframerah (a) MIP sebelum ekstraksi (b) MIP setelah ekstraksi (c) NIP

LAMPIRAN 10
(LANJUTAN)

Tabel V.8
Hasil analisis FTIR sorben MIP sebelum soxhlet, MIP soxhlet dan NIP

Bilangan Gelombang (cm^{-1})			Gugus Fungsi
MIP sebelum ekstraksi	**MIP setelah ekstraksi**	**NIP**	
1162.30	1161.36	1162.14	C-O Streching
1388.00	1321.17	1453.07	C-H Streching
1635.96	1453.75	1635.80	C=C Streching
1724.50	1635.76	1724.31	C=O Streching
2955.51	2956.44	2955.55	C-H Streching
2989.00	2988.09	2988.31	C-H Streching
3447.51	3453.59	3451.18	O-H Bending

LAMPIRAN 11

DOKUMENTASI PENELITIAN

a. Melarutkan tetrasiklin

b. Bahan yang digunakan tetrasiklin

c. Proses sonikasi

d. Polimer yang terbentuk setelah dimasukan ke dalam oven

e. Polimer setelah penggerusan dan pengayakan

f. Alat ekstraksi Soxhlet

g. Polimer dibungkus dengan kertas saring

Gambar V.10 Dokumentasi Penelitian

LAMPIRAN 11
(LANJUTAN)

h. Penggantian pelarut kedalam labu alas bulat

I Hasil sorben

j. Hasil ekstraksi absorban ke dua

k. Proses pembilasan

l. Alat lain yang digunakan

Gambar V.10 Dokumentasi Penelitian