

BAB V

KESIMPULAN DAN SARAN

A. Kesimpulan

Berdasarkan dari hasil penelitian yang telah dilakukan dapat diperoleh kesimpulan bahwa :

Pertama, *myricetin* dapat ditingkatkan kelarutannya melalui teknik solubilisasi menggunakan penghantaran variasi konsentrasi *poloxamer F127*. Kadar kelarutan *myricetin* paling tinggi sebesar 66,46 ppm pada proporsi *poloxamer F127* satu setengah diatas cmc.

Kedua, *Myricetin* dengan sistem solubilisasi *poloxamer F127* pada formula 1, formula 2 dan formula 3 memberikan kenaikan kelarutan *myricetin* yang baik. Kelarutan *myricetin* meningkat seiring dengan meningkatnya jumlah *poloxamer F127*. Sehingga nilai tiga variasi konsentrasi *myricetin* yang diperoleh ini dapat disimpulkan baik.

B. Saran

Penelitian ini masih banyak kekurangan, maka perlu dilakukan penelitian lebih lanjut mengenai :

Pertama, perlu dilakukan optimasi *poloxamer* yang lebih beragam.

Kedua, pengembangan dalam bentuk sediaan.

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Lampiran 1. Sertifikat analisis myricetin

TOCRIS
a biotechne brand

Print Date: Jul 20th 2017

Certificate of Analysis

www.tocris.com

Product Name: Myricetin

Catalog No.: 6189

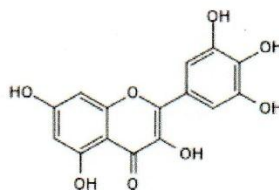
Batch No.: 1

CAS Number: 529-44-2

IUPAC Name: 3,5,7-Trihydroxy-2-(3,4,5-trihydroxyphenyl)-4H-1-benzopyran-4-one

1. PHYSICAL AND CHEMICAL PROPERTIES

Batch Molecular Formula: C₁₅H₁₀O₈·H₂O
Batch Molecular Weight: 336.26
Physical Appearance: Yellow solid
Solubility: DMSO to 100 mM
 ethanol to 50 mM
Storage: Store at -20°C
Batch Molecular Structure:



2. ANALYTICAL DATA

HPLC: Shows 97.7% purity
¹H NMR: Consistent with structure
Mass Spectrum: Consistent with structure
Microanalysis:

	Carbon	Hydrogen	Nitrogen
Theoretical	53.58	3.6	
Found	53.6	3.57	

Caution - Not Fully Tested • Research Use Only • Not For Human or Veterinary Use

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Product Information
www.tocris.com
Product Name: Myricetin

Catalog No.: 6189

Batch No.: 1

CAS Number: 529-44-2

IUPAC Name: 3,5,7-Trihydroxy-2-(3,4,5-trihydroxyphenyl)-4H-1-benzopyran-4-one

Description:

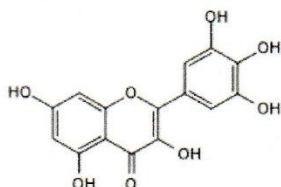
Irreversible TrxR inhibitor (IC_{50} = 0.62 μ M). Exhibits concentration-, time- and NADH-dependent TrxR inhibition. Results in the oxidation of Trx and reduced TrxR activity in vitro in addition to the accumulation of cells in sub-G₁ phase. Reduces neoplastic transformation and induces cell death in cancer cell lines. Chemotherapeutic.

Physical and Chemical Properties:
Batch Molecular Formula: C₁₅H₁₀O₈·H₂O

Batch Molecular Weight: 336.26

Physical Appearance: Yellow solid

Minimum Purity: >97%

Batch Molecular Structure:

Storage: Store at -20°C

CAUTION - This product is light sensitive and we recommend that the solid material and any solutions obtained are protected from exposure to light.

Solubility & Usage Info:

DMSO to 100 mM

ethanol to 50 mM

Stability and Solubility Advice:

Some solutions can be difficult to obtain and can be encouraged by rapid stirring, sonication or gentle warming (in a 45-60°C water bath).

Information concerning product stability, particularly in solution, has rarely been reported and in most cases we can only offer a general guide. Our standard recommendations are:

SOLIDS: Provided storage is as stated on the product label and the vial is kept tightly sealed, the product can be stored for up to 6 months from date of receipt.

SOLUTIONS: We recommend that stock solutions, once prepared, are stored aliquoted in tightly sealed vials at -20°C or below and used within 1 month. Wherever possible solutions should be made up and used on the same day.

References:

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Lampiran 2. Gambar alat dan bahan penelitian

Alat	
 A white analytical balance scale with a stainless steel weighing pan and a digital display showing 0.0000.	 A white and black UV-Vis spectrophotometer with a digital display and a keypad.
Neraca analitik	Spektro UV-Vis
 A blue and white shaking thermostatic water bath with a digital display and a control knob. Several glass beakers are placed on the shaking platform.	 Three glass pipettes with orange rubber bulbs, arranged vertically on a wooden surface.
Shaking Thermostatic Water Bath	Pipet Tetes

Alat

Pipet Volume



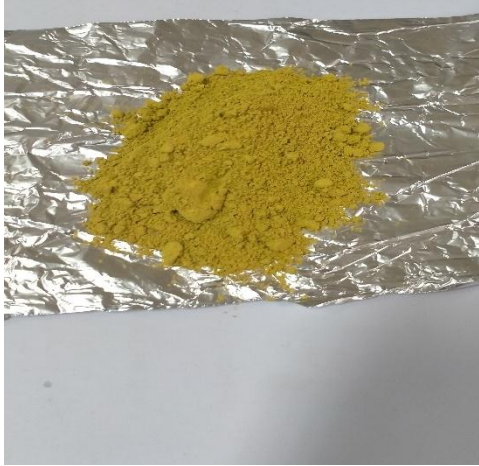
Botol Vial



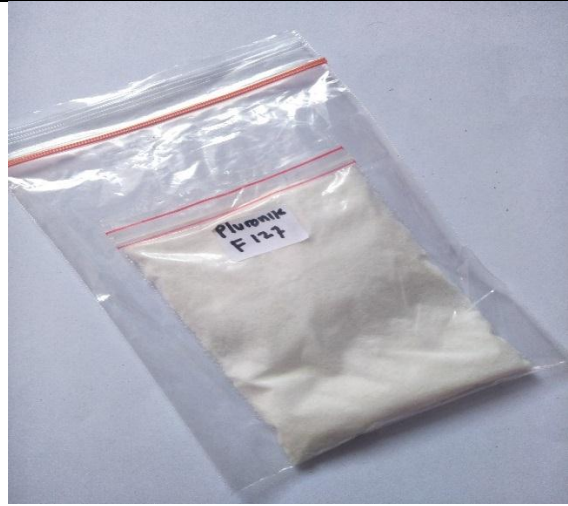
Batang Pengaduk



Sendok Tanduk

Bahan

Serbuk Myricetin



Poloxamer F127



Aquadest

Lampiran 3. Formula Myricetin

A. Formula Myricetin Setelah di Shaker

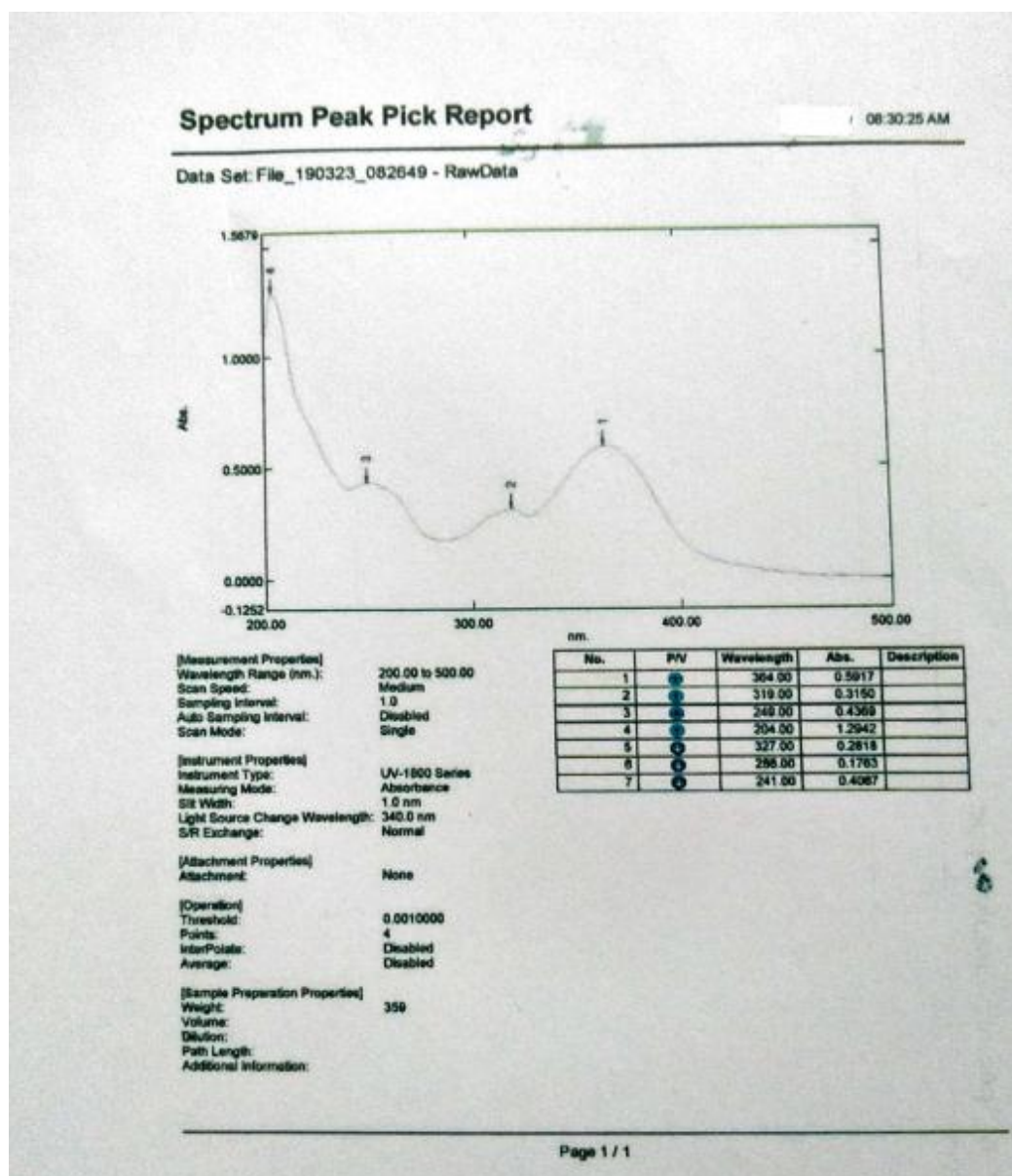


B. Formula Myricetin Setelah Penyaringan Filtrat



Lampiran 4. Pembuatan kurva kalibrasi dan validasi metode

1. Penentuan panjang gelombang



2. Operating Time Myricetin

Kinetics Data Print Report

03/11/2020 04:13:17 PM

Time (Minute)	RawData ...
0.000	0.417
1.000	0.418
2.000	0.417
3.000	0.417
4.000	0.417
5.000	0.418
6.000	0.418
7.000	0.418
8.000	0.418
9.000	0.418
10.000	0.418
11.000	0.417
12.000	0.418
13.000	0.418
14.000	0.418
15.000	0.418
16.000	0.418
17.000	0.418
18.000	0.418
19.000	0.419
20.000	0.418
21.000	0.419
22.000	0.419
23.000	0.418
24.000	0.418
25.000	0.418
26.000	0.418
27.000	0.418
28.000	0.418
29.000	0.418
30.000	0.418

3. Linieritas

Penimbangan *myricetin* :

Kertas kosong : 0,2812 g

Kertas kosong + isi : 0,2921 g

Kertas sisa : 0,2815 g

Zat aktif : 0,0106 g

Membuat larutan induk sebesar 106 ppm dengan menimbang 10,6 mg *myricetin* ditambahkan aquadestilata sampai 100 mL, selanjutnya dibuat seri konsentrasi :

1) 4 ppm

$$\begin{aligned} V_1 \times C_1 &= V_2 \times C_2 \\ 0,4 \text{ ml} \times 100 \text{ ppm} &= 10 \text{ ml} \times C_2 \\ C_2 &= 4 \text{ ppm} \end{aligned}$$

2) 6 ppm

$$\begin{aligned} V_1 \times C_1 &= V_2 \times C_2 \\ 0,6 \text{ ml} \times 100 \text{ ppm} &= 10 \text{ ml} \times C_2 \\ C_2 &= 6 \text{ ppm} \end{aligned}$$

3) 8 ppm

$$\begin{aligned} V_1 \times C_1 &= V_2 \times C_2 \\ 0,8 \text{ ml} \times 100 \text{ ppm} &= 10 \text{ ml} \times C_2 \\ C_2 &= 8 \text{ ppm} \end{aligned}$$

4) 10 ppm

$$\begin{aligned} V_1 \times C_1 &= V_2 \times C_2 \\ 1,0 \text{ ml} \times 100 \text{ ppm} &= 10 \text{ ml} \times C_2 \\ C_2 &= 10 \text{ ppm} \end{aligned}$$

5) 12 ppm

$$\begin{aligned} V_1 \times C_1 &= V_2 \times C_2 \\ 1,2 \text{ ml} \times 100 \text{ ppm} &= 10 \text{ ml} \times C_2 \\ C_2 &= 12 \text{ ppm} \end{aligned}$$

Konsentrasi standar (ppm)	Absorbansi
4	0,355
6	0,465
8	0,58
10	0,682
12	0,793

Persamaan regresi linier antara konsentrasi (ppm) dan serapan diperoleh :

$$a = 0,1378$$

$$b = 0,0547$$

$$r = 0,9999$$

$$y = 0,1378 + 0,0547x$$

keterangan :

x = Konsentrasi (ppm)

y = Serapan

4. Akurasi

KONSENTRASI	REPLIKASI	ABS	KONSENTRASI	SEBENARNYA	%		
80%	1	0,603	8,0992	8	101%	101,52%	101,11%
	2	0,605	8,1264	8	102%		
	3	0,606	8,1400	8	102%		
100%	1	0,761	10,2504	10	103%	102,82%	
	2	0,763	10,2777	10	103%		
	3	0,766	10,3185	10	103%		
120%	1	0,89	12,0068	12	100%	99,00%	
	2	0,87	11,7345	12	98%		
	3	0,882	11,8979	12	99%		

$$\% \text{ recovery} = \frac{\text{konsentrasi terukur}}{\text{konsentrasi sebenarnya}} \times 100\%$$

Hasil dari akurasi didapatkan rata-rata % recovery yaitu 101,52%, 102,82% dan 99,00%.

Rata-rata % yaitu 101,11%.

5. Presisi

KONSENTRASI (PPM)	ABS	KONSENTRASI
6	0,602	8,0856
6	0,608	8,1673
6	0,612	8,2217
6	0,617	8,2898
6	0,621	8,3443
6	0,628	8,4396
RATA-RATA		8,258044
SD		0,127044
CV		0,015384

$$SD = \sqrt{\frac{\sum(x - \bar{x})^2}{n - 1}}$$

$$CV = \frac{SD}{\bar{X}}$$

Hasil presisi didapatkan nilai SD sebesar 0,1270 dan nilai CVnya 0,01%.

6. Penentuan LOD dan LOQ

Konsentrasi (ppm)	Absorbansi (abs)	Y'	Y-Y'	(Y-Y') ²
4	0,355	0,356400	-0,0014	0,000001960
6	0,465	0,465700	-0,0007	0,000000490
8	0,580	0,575000	0,0050	0,000025000
10	0,682	0,684300	-0,0023	0,000005290
12	0,793	0,793600	-0,0006	0,000000360
TOTAL				0,00003310

Nilai y' diperoleh dari substitusi konsentrasi dalam persamaan $y' = 0,1131 + 0,0543x$ dengan x adalah konsentrasi (ppm) dan y' adalah serapan.

- $\hat{y} = 0,1378 + 0,0547x$
 $\hat{y} = 0,1378 + 0,0547 \times 4$
 $\hat{y} = 0,3566$
- $\hat{y} = 0,1378 + 0,0547x$
 $\hat{y} = 0,1378 + 0,0547 \times 6$
 $\hat{y} = 0,466$
- $\hat{y} = 0,1378 + 0,0547x$

$$\hat{y} = 0,1378 + 0,0547 \times 8$$

$$\hat{y} = 0,5754$$

$$4. \hat{y} = 0,1378 + 0,0547x$$

$$\hat{y} = 0,1378 + 0,0547 \times 10$$

$$\hat{y} = 0,6848$$

$$5. \hat{y} = 0,1378 + 0,0547x$$

$$\hat{y} = 0,1378 + 0,0547 \times 12$$

$$\hat{y} = 0,7942$$

$$S_{x/y} = \frac{\sqrt{\sum (y - \hat{y})^2}}{n-2}$$

$S_{x/y}$ = Simpangan baku residual

$\sum (y - \hat{y})^2$ = Jumlah kuadrat total residual

N = Jumlah data

$$S_{x/y} = \frac{\sqrt{0,00003310}}{5-2}$$

$$= 0,0033$$

$$LOD = 3,3 \times \frac{S_{x/y}}{b}$$

$$= 3,3 \times \frac{0,0033}{0,0547}$$

$$= 0,1990 \text{ ppm}$$

$$y = a + bx$$

$$= 0,1378 + 0,0547x$$

$$= 0,1378 + 0,0547 (0,1990)$$

$$= 0,1378 + 0,0108$$

$$= 0,1486$$

$$\text{Serapan LOD} = 0,1486$$

$$LOQ = 10 \times \frac{S_{x/y}}{b}$$

$$= 10 \times \frac{0,0033}{0,0547}$$

$$= 0,6032 \text{ ppm}$$

$$y = a + bx$$

$$= 0,1378 + 0,0547x$$

$$= 0,1378 + 0,0547 (0,6032)$$

$$= 0,1378 + 0,0329$$

$$= 0,1707$$

$$\text{Serapan LOQ} = 0,1707$$

Lampiran 5. Kadar *myricetin* terlarut dalam air

Penimbangan *myricetin* :

Kertas kosong : 0,2812 g

Kertas kosong + isi : 0,5812 g

Kertas sisa : 0,2815 g

Zat aktif : 0,300 g

Membuat *myricetin* dengan pelarut air 3000 ppm dengan menimbang 0,300 gram *myricetin* ditambahkan aquadest sampai 100 mL, selanjutnya dibaca absorbansinya :

Hasil : 0,234 (Abs)

$$y = a + bx$$

$$0,234 = 0,1131 + 0,0543x$$

$$x = \frac{0,234 - 0,1131}{0,0543}$$

$$x = 2,2265 \text{ ppm}$$

Lampiran 6. Kadar *myricetin* terlarut dalam perbandingan campuran

Kadar zat terlarut			
Aquadest	<i>Pluronic F127</i> (gram)	<i>Myricetin</i> (gram)	Kadar (ppm)
Ad 100	0,017	0,300	5,191
Ad 100	0,035	0,300	6,591
Ad 100	0,052	0,300	6,646

Kadar zat terlarut *myricetin* :

$$V1 \times C1 = V2 \times C2$$

$$5 \text{ ml} \times 3000 \text{ ppm} = 10 \text{ ml} \times C2$$

$$C2 = 1.500 \text{ ppm}$$

Formula 1 : (Abs = 0,395)

$$y = a + bx$$

$$0,395 = 0,1131 + 0,0543x$$

$$x = \frac{0,395 - 0,1131}{0,0543}$$

$$x = 5,1915 \text{ ppm}$$

Formula 2 : (Abs = 0,471)

$$y = a + bx$$

$$0,471 = 0,1131 + 0,0543x$$

$$x = \frac{0,471 - 0,1131}{0,0543}$$

$$x = 6,5911 \text{ ppm}$$

Formula 3 : (Abs = 0,474)

$$y = a + bx$$

$$0,474 = 0,1131 + 0,0543x$$

$$x = \frac{0,474 - 0,1131}{0,0543}$$

$$x = 6,6464 \text{ ppm}$$

Lampiran 6. Data analisis *One Way* ANOVA hasil solubilisasi

Descriptive Statistics

	N	Mean	Std. Deviation	Minimum	Maximum
Absorbansi	9	,4469	,03899	,39	,48

One-Sample Kolmogorov-Smirnov Test

		Absorbansi
N		9
Normal Parameters ^{a,b}	Mean	,4469
	Std. Deviation	,03899
	Absolute	,373
Most Extreme Differences	Positive	,237
	Negative	-,373
Kolmogorov-Smirnov Z		1,118
Asymp. Sig. (2-tailed)		,164

a. Test distribution is Normal.

b. Calculated from data.

ANOVA

absorbansi

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	,012	2	,006	1476,027	,000
Within Groups	,000	6	,000		
Total	,012	8			

Test of Homogeneity of Variances

absorbansi

Levene Statistic	df1	df2	Sig.
1,061	2	6	,403