

LAMPIRAN

Lampiran 1. sertifikat COA Betametason

sicor

TEVA API Division

PRODUCT SPECIFICATIONS AND CERTIFICATE OF ANALYSIS		
Page 1 of 4		
Product Name: Betamethasone Dipropionate Microsized Control No.: S433M000817 Quantity: 3.000 KG Manufacturing Site: Sicor - Rhe Manufacturing Date: October 2017		
Order No.: SU701148 Client Packing Order: Quality Market: EUR Original Analysis Date: 13-December-2017 Re Test date: October 2022		
Packaging and storage: Preserve in tight. Store at 25°C, excursions permitted between 15°C and 30°C. Store protect from light.		
TESTS AND METHODS	SPECIFICATIONS	RESULTS*
EU TESTS		
Appearance	white or almost white, crystalline powder	Conforms
Identification by Infrared	the infrared absorption spectrum of the preparation of the test specimen exhibits maxima only at the same wavelengths as that of the corresponding CRS	Corresponds
IR		
Specific Optical Rotation	between +84° and +88° on dried basis	+85°
IR		
Related Substances by HPLC		
Impurity A	not more than 0.10%	Less than 0.05%
Impurity B	not more than 0.15%	Less than 0.05%
Impurity C	not more than 0.20%	Less than 0.05%
Impurity D	not more than 0.15%	Less than 0.05%
Impurity E	not more than 0.10%	Less than 0.05%
Impurity F	not more than 0.10%	Less than 0.05%
Impurity G	not more than 0.10%	Less than 0.05%
Other Impurity Each	not more than 0.10%	Less than 0.05%
Total Impurities	not more than 1.0%	Less than 0.05%
Assay by HPLC	between 98.0% and 102.0% on dried basis	101.0%
IR		

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Lampiran 2. sertifikat COA Deksklorfeniramin maleat

07/11/19
PT. SUPRIYA LIFESCIENCE
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CDP
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Certificate of analysis

Product	Des-chlorpheniramine Maleate USP	Lot/Case No.	KD-129
Batch No.	SLL/DC/0719015 ✓	Date of Manufacturing	July-2019 ✓
Batch Qty.	160.00kg	Date of Expiry	June-2022 ✓
A. R. No.	SLL/QC/VP/19/0857	Date of Release	02/08/2019

S.No.	Test	Specifications	Results
1.	Description	White, odorless, crystalline powder.	A white crystalline powder.
2.	Solubility	Freely soluble in water; soluble in alcohol and in chloroform.	Conforms
3.	i. Test A : IR	The IR absorption spectrum should be concordant with that of Deschlorpheniramine maleate working standard / reference standard.	Complies
	ii. Assay By HPLC	The retention times of the maleic acid and deschlorpheniramine peaks of the sample solution correspond to those of the standard solution, as obtained in the Assay.	Complies
4.	Assay By HPLC on dry basis (% w/w)	98.0 - 102.0	99.90
5.	Residue on Ignition (% w/w)	NMT 0.2	0.05
6.	Organic impurities by HPLC (%)		
	i. Pheniramine	NMT 0.4	BDL
	ii. Any other unspecified impurity	NMT 0.10	BDL
	iii. Total impurities	NMT 1	BDL
7.	Enantiomeric Purity by HPLC (%)	NMT 2	0.35

QA/911/03-02 Effective date 05/08/2015 Page 1 of 2

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Lampiran 3. Uji Selektifitas

Analit	Jarak tempuh analit (cm)	Rf	α
Betametason	1,4	0,8	2,85
Deksklorfeniramin maleat	4	0,28	

1. Faktor Retensi

$$R_f = \frac{\text{Jarak tempuh analit}}{\text{Jarak tempuh pengembang}}$$

$$R_f \text{ Deksklorfeniramin maleat} = \frac{1,4}{5} = 0,28$$

$$R_f \text{ Betametason} = \frac{4}{5} = 0,8$$

2. Faktor Selektivitas (α)

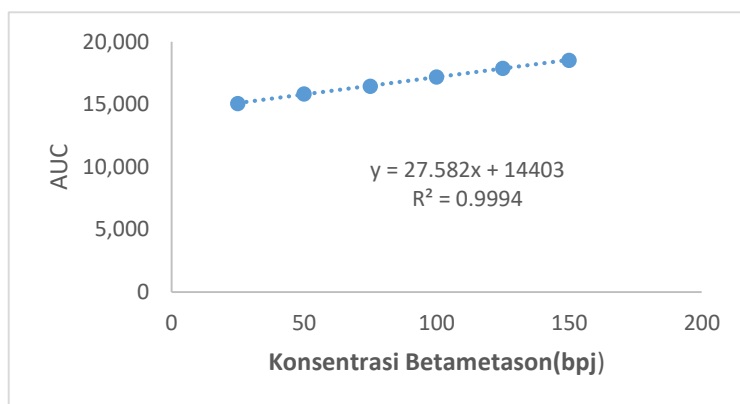
$$\alpha = \frac{R_f \text{ Betametason}}{R_f \text{ Deksklorfeniramin maleat}}$$

$$\alpha = \frac{0,8}{0,28} = 2,857142857$$

Lampiran 4. Kurva Kalibrasi

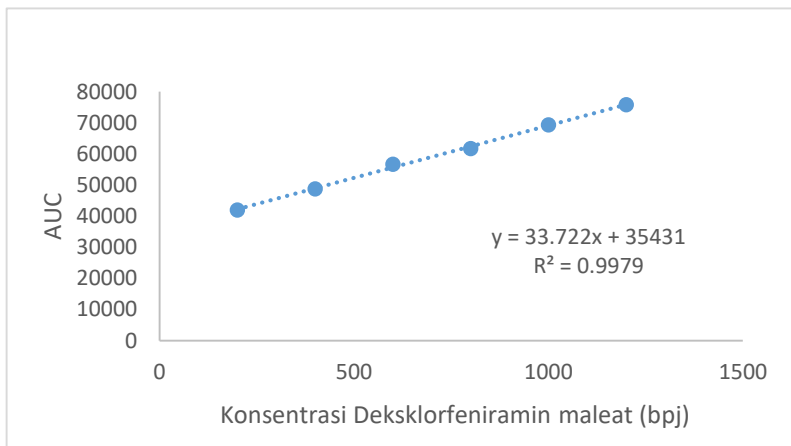
Kurva kalibrasi Betametason

Konsentrasi (bpj)	AUC
25	15064
50	15815,886
75	16447,563
100	17185,785
125	17876,17
150	18507,402



Kurva kalibrasi Deksklorfeniramin maleat

Konsentrasi (bpj)	AUC
200	41944.312
400	48751.907
600	56685.505
800	61692.555
1000	69350.889
1200	75793.759



**Hasil perhitungan Batas Deteksi dan Batas Kuantisasi
Betametason**

No	Konsesntrasi (bpj)	AUC (y)	y' (bx+a)	y-y'	(y-y') ²
1	25	15064,404	15092,55	-28	792,20
2	50	15815,886	15782,1	34	1141,49
3	75	16447,563	16471,65	-24	580,18
4	100	17185,785	17161,2	25	604,42
5	125	17876,17	17850,75	25	646,18
6	150	18507,402	18540,3	-33	1082,28
	87,5				$\Sigma = 4846,75$

Batas Deteksi (BD) = 3,78608 bpj

Batas Kuantisasi (BK) = 12,62029 bpj

Sx0 = 1,2620

Vx0 = 0,0144%

$$S_{y/x} = \sqrt{\frac{\sum(y-y')^2}{n-2}} = \sqrt{\frac{4846,75}{4}} = 34,8093$$

$$BD = \frac{3 \times sy/x}{slope} = \frac{3 \times 34,8093}{27,582} = 3,78608 \text{ bpj}$$

$$BK = \frac{10 \times sy/x}{slope} = \frac{10 \times 34,8093}{27,582} = 12,62029 \text{ bpj}$$

$$S_{x0} = \frac{sy/x}{slope} = \frac{34,8093}{27,582} = 1,2620$$

$$V_{x0} = \frac{S_{x0}}{\bar{x}} = \frac{1,2620}{87,5} \times 100\% = 0,0144\%$$

**Hasil perhitungan Batas Deteksi dan Batas Kuantisasi
Deksklorfeniramin maleat**

No	Konsesentrasi (bpj)	AUC (y)	y' (bx+a)	y-y'	(y-y') ²
1	200	41944,312	42175,4	-231	53401,66
2	400	48751,907	48919,8	-168	28188,05
3	600	56685,505	55664,2	1021	1043063,90
4	800	61692,555	62408,6	-716	512720,44
5	1000	69350,889	69153	198	39160,05
6	1200	75793,759	75897,4	104	10741,45
	700				$\Sigma = 1687275,58$

Batas Deteksi (BD) = 57,7791 bpj

Batas Kuantisasi (BK) = 192,5970 bpj

S_{x0} = 19,2597

V_{x 0} = 0,0275%

$$S_{y/x} = \sqrt{\frac{\Sigma(y-y')^2}{n-2}} = \sqrt{\frac{1687275,58}{4}} = 649,4758$$

$$BD = \frac{3 \times s_{y/x}}{slope} = \frac{3 \times 649,4758}{33,722} = 57,7791 \text{ bpj}$$

$$BK = \frac{10 \times s_{y/x}}{slope} = \frac{10 \times 649,4758}{33,722} = 192,5970 \text{ bpj}$$

$$S_{x0} = \frac{s_{y/x}}{slope} = \frac{649,4758}{33,722} = 19,2597$$

$$V_{x0} = \frac{S_{x0}}{\bar{x}} = \frac{19,2597}{700} \times 100\% = 0,0275\%$$

Lampiran 5. Hasil Akurasi Betametason

Konsentrasi (%)	AUC	Konsentrasi teoritis (mg/mL)	Bobot pengukuran (mg/mL)	% recovery
80	15409,818	0,4	0,3650	91,2567%
	15448,768		0,3791	94,7871%
	15391.354		0,3583	89,5832%
Rata-rata				91,8757%
100	15689,333	0,5	0,4663	93.2733%
	15674,354		0,4609	92.1872%
	15660,726		0,4559	91.1990%
Rata-rata				92,2198%
120	16060,487	0,6	0.6009	100.1551%
	16110,236		0.6189	103.1612%
	16014,638		0.5843	97.3846%
Rata-rata				100,2336%

Contoh perhitungan :

Persamaan regresi linear :

$$y = 27,582x + 14403$$

$$15409,818 = 27,582x + 14403$$

$$27582x = 15409,818 - 14403$$

$$x = 36.50271916 \text{ bpj}$$

$$\% \text{ recovery} = \frac{\text{konsentrasi pengukuran}}{\text{konsentrasi teoritis}} \times 100\%$$

$$= \frac{0,3650}{0,4} \times 100\%$$

$$= 91,2567\%$$

Hasil Akurasi Deksklorfeniramin maleat

Konsentrasi (%)	AUC	Konsentrasi teoritis (mg/mL)	Bobot pengukuran (mg)	% recovery
80	46947,484	3,2	3,4151	106,7226%
	46418,09		3,2581	101,8167%
	45734,836		3,0555	95,4851%
Rata-rata				101,3415%
100	49148,969	4	4,0679	101,6989%
	49226,286		4,0908	102,2721%
	49370,475		4,1336	103,3411%
Rata-rata				102,4374%
120	52214,806	4,8	4,9771	103,689%
	52951,17		5,1954	108,238%
	53378,655		5,3222	110,879%
Rata-rata				107.6029%

Contoh perhitungan :

Persamaan regresi linear :

$$y = 33.722x + 35431$$

$$33.722x = 46947,484 - 35431$$

$$x = 341,5124 \text{ bpj}$$

$$\% \text{ recovery} = \frac{\text{konsentrasi pengukuran}}{\text{konsentrasi teoritis}} \times 100\%$$

$$= \frac{3,4151}{3,2} \times 100\%$$

$$= 106,7226\%$$

Lampiran 6. Hasil Presisi Betametason

AUC (y)	Konsentrasi (X)	(X-X')	(X-X') ²	SD	%RSD
15689,333	46,6366	0,4941	0,2442		
15674,354	46,0936	-0,0488	0,0023		
15660,726	45,5995	-0,5429	0,2948	0,5568	1,2069
15670,496	45,9537	-0,1887	0,0356		
15668,375	45,8768	-0,8177	0,6687		
15690,931	46,6946	0,5521	0,3048		
Rata-rata	46,1425		$\Sigma = 1,5506$		

Contoh Perhitungan :

$$\begin{aligned}
 \text{SD} &= \sqrt{\frac{\Sigma(x-x)^2}{n-1}} \\
 &= \sqrt{\frac{1,5506}{5}} = 0,5568
 \end{aligned}$$

$$\begin{aligned}
 \text{RSD} &= \frac{\text{SD}}{\text{x rata-rata}} \times 100\% \\
 &= \frac{0,5568}{46,1425} \times 100\% = 1,2069\%
 \end{aligned}$$

Hasil Presisi Deksklorfeniramin maleat

AUC (y)	Konsentrasi (X)	(Xi-X')	(Xi-X) ²	SD	%RSD
49148,969	406,7958	-6,0191	36,23037	5,2781	1,2785
49226,286	409,0886	-3,7263	13,88598		
49370,475	413,3644	0,5494	0,301866		
49662,028	422,0102	9,1952	84,55178		
49302,392	411,3454	-1,4695	2,159505		
49401,534	414,2854	1,4704	2,162236		
Rata-rata	412,8149		$\Sigma = 139,2917$		

Contoh Perhitungan :

$$\begin{aligned}
 \text{SD} &= \sqrt{\frac{\sum (x-x)^2}{n-1}} \\
 &= \sqrt{\frac{139,2917}{5}} = 5,2781
 \end{aligned}$$

$$\begin{aligned}
 \text{RSD} &= \frac{\text{SD}}{\text{x rata-rata}} \times 100\% \\
 &= \frac{5,2781}{412,8149} \times 100\% = 1,2785\%
 \end{aligned}$$

Lampiran 7. Penetapan Kadar

Hasil AUC Betametason

Sampel	Rata-rata AUC	Rata-rata Bobot (mg)	FK	Rata-rata Kadar (%)	Kadar tablet (mg/tab)
1	15761,766	0,4926	0,9976	98,2959%	0,4914
2	15762,501	0,4928	0,9976	98,3490%	0,4917

Contoh Perhitungan :

Persamaan garis regresi linier yang dipakai $y = 27.582x + 14403$

$$y = 27.582x + 14403$$

$$15761,766 = 27,582x + 14403$$

$$27582x = 15761,766 - 14403$$

$$x = 49,2627 \text{ bpj}$$

$$\text{Bobot terukur} = \text{Konsentrasi(X)} \times \text{Volume}$$

$$= 49,2627 \text{ bpj} \times 10 \text{ mL}$$

$$= 492,627 \text{ } \mu\text{g} = 0,4926 \text{ mg}$$

$$\text{Faktor Koreksi} = \frac{100}{\% \text{ recovery}}$$

$$= \frac{100}{100,2336} = 0,9976$$

$$\text{Kadar sampel Betametason} = \frac{\text{Bobot terukur}}{\text{Bobot teori}} \times \text{Fk} \times 100\%$$

$$= \frac{0,4926}{0,5} \times 0,9976 \times 100\%$$

$$= 98,2890\%$$

Hasil AUC Deksklorfeniramin maleat

Sampel	Rata-rata AUC	Rata-rata Bobot (mg)	FK	Rata-rata Kadar (%)	Kadar tablet (mg/2tab)
1	49344,034	4.1258	0,9867	101,7796%	4,0711
2	49312,559	4.1164	0,9867	101,5494%	4,0619

Contoh Perhitungan :

Persamaan garis regresi linier yang dipakai $y = 33.722x + 35431$

$$y = 33.722x + 35431$$

$$49344,034 = 33.722x + 35431$$

$$33.722x = 49344,034 - 35431$$

$$x = 412,5803 \text{ bpj}$$

$$\begin{aligned} \text{Bobot terukur} &= \text{Konsentrasi(X)} \times \text{Volume} \\ &= 412,5803 \text{ bpj} \times 10 \text{ mL} \\ &= 4125,803 \text{ } \mu\text{g} = 4,1258 \text{ mg} \end{aligned}$$

$$\begin{aligned} \text{Faktor Koreksi} &= \frac{100}{\% \text{ recovery}} \\ &= \frac{100}{101,3415} = 0,9867 \end{aligned}$$

$$\begin{aligned} \text{Kadar sampel Dex-CTM} &= \frac{\text{Bobot terukur}}{\text{Bobot teori}} \times \text{Fk} \times 100\% \\ &= \frac{4,1258}{4} \times 0,9867 \times 100\% \\ &= 101,7731\% \end{aligned}$$