

Certificate of Analysis

Product Name: Demeclocycline hydrochloride hydrate
VETRANAL™, analytical standard

Product Number: 46161

Batch Number: BCCC8240

Brand: Sigma-Aldrich

CAS Number: 64-73-3

Formula: $C_{21}H_{21}ClN_2O_8 \cdot HCl \cdot xH_2O$

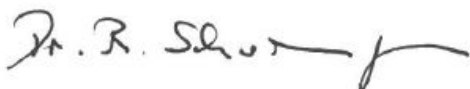
Formula Weight: 501.31

Storage Temperature: 2-8 C

Expiration Date: JAN 2025

Quality Release Date: 20 FEB 2020

TEST	SPECIFICATION	RESULT
APPEARANCE (COLOR)	WHITE TO YELLOW AND FAINT BEIGE TO LIGHT BEIGE AND FAINT BROWN TO LIGHT BROWN	LIGHT YELLOW TO LIGHT BEIGE
APPEARANCE (FORM)	POWDER OR CRYSTALS	POWDER
PURITY (HPLC AREA %)	≥ 90.0 %	93.0 %
SPECIFIC ROTATION (20/D)	-265 TO -245 DEGREES	-264 DEGREES
CONCENTRATION	C=0.5 IN 0.1N H2SO4	--
WATER	≤ 3.0 %	2.7 %
PROTON NMR SPECTRUM	CONFORMS TO STRUCTURE	CONFORMS



Dr. Reinhold Schwenninger
Quality Assurance
Buchs, Switzerland

Sigma-Aldrich warrants that at the time of the quality release or subsequent retest date this product conformed to the information contained in this publication. The current specification sheet may be available at Sigma-Aldrich.com. For further inquiries, please contact Technical Service. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

Certificate of Analysis

Dionex Ultimate 3000	
Pump : LPG-3400RS	Injection Time : 20.11.19 10:49
Autosampler : WPS-3000	Processed By : n.a.
Detector : VWD-3400	Vial Number : GA2
Column : Supelco Ascentis Express C18, 2.7 um	Column S/N : -
Column Dim. : 100 x 2.1 mm	Sample Type : Unknown
Mobile Phase :	Injection Volume : 1.0 µl
%A : Acetonitril	Flow : 0.50 ml/min
%B : %B	Column Temp. (°C) : n.a.
%C : HPS pH 2.4	Run Time : 15.00 min
%D : %D	
Gradient : see Figure 1	
Sample Prep. : 1 mg sample in 1 ml acetonitrile : water 1 : 1	

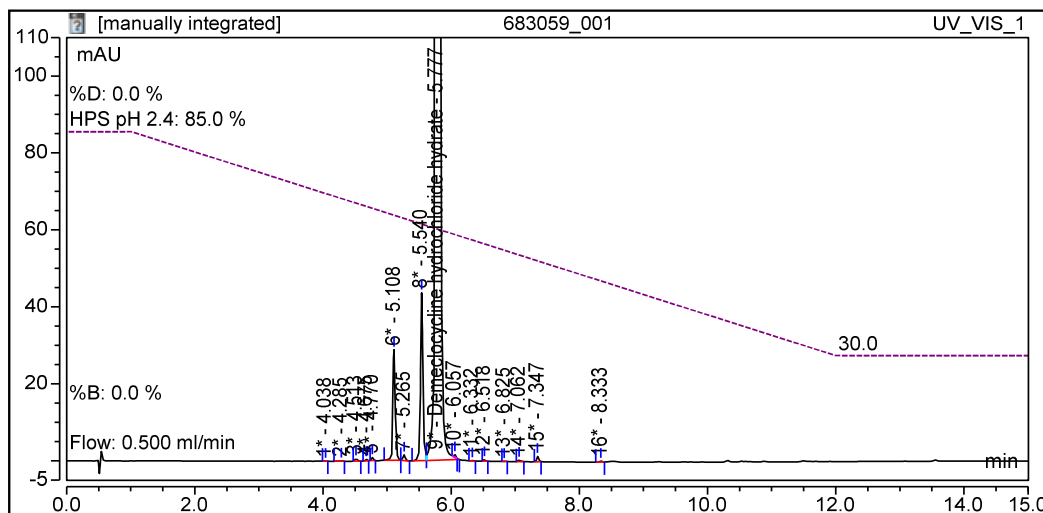


Figure 1: Zoomed Chromatogram

No.	Ret.Time min	Peak Name	Area mAU*min	Height mAU	Amount	Rel.Area %
1	4.038		0.00069	0.02342	n.a.	0.00
2	4.285		0.00705	0.11011	n.a.	0.01
3	4.513		0.02958	0.66403	n.a.	0.06
4	4.675		0.01537	0.42321	n.a.	0.03
5	4.770		0.03214	0.80111	n.a.	0.07
6	5.108		1.22268	28.68455	n.a.	2.55
7	5.265		0.05679	1.52683	n.a.	0.12
8	5.540		1.87533	43.61068	n.a.	3.91
9	5.777	Demeclocycline hydrochloride	44.63128	778.00034	n.a.	92.98
10	6.057		0.02396	0.71404	n.a.	0.05
11	6.332		0.00348	0.09868	n.a.	0.01
12	6.518		0.01510	0.42904	n.a.	0.03
13	6.825		0.00172	0.06149	n.a.	0.00
14	7.062		0.01782	0.40959	n.a.	0.04
15	7.347		0.05582	1.43945	n.a.	0.12
16	8.333		0.01144	0.25705	n.a.	0.02
Total:			48.00024	857.25362		100.00

Table 1: Integration