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Development and Validation of a Stability-Indicating HPLC Method for the Analysis of Desvenlafaxine Succinate in the Presence of its Acidic Induced Degradation Product in Bulk and Pharmaceutical Preparation

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ABSTRACT

A stability- indicating High Performance Liquid Chromatography (HPLC) method of analysis of desvenlafaxine suucinate(DSV) in the presence of its acidic induced- degradation product in pure and pharmaceutical preparation had been developed and validated. The chromatographic conditions comprised of an isocratic reversed- phase separation on Discovery C18 column. Elution was carried out using acetonitrile: phosphate buffer pH 3.8 (50 : 50 v/v) as a mobile phase at a flow rate of 0.7 ml/min and UV detection at 229 nm. The linear regression analysis data for the calibration plots showed good linear relationship in the concentration range 5-100 µg/ml (r^2 =0.9999). The values of slope and intercept were 34.295 and 12.564 respectively. The method was successfully validated in accordance to ICH guidelines acceptance criteria. The specificity and stability-indicating capabilities of the method was verified by subjecting DSV to acid hydrolytic stress condition. The acid degradation product was confirmed as 4-(1-cyclohexenyl-2-(dimethylamino)ethyl)phenol. Under the chromatographic condition, the degradation product was well resolved from the active pharmaceutical ingredients with significantly different retention time. Thus the proposed method was found to be stability-indicating and can be used for routine analysis of the drug without interference of acidic degradation product. The proposed method was successfully applied for the analysis of pharmaceutical formulation. The validity of the suggested procedures was further assessed by applying the standard addition technique which was found to be satisfactory. The results were statistically analyzed and compared with those obtained by the reported method.

Keywords: HPLC; desvenlafaxine succinate; acidic degradation product: NMR.

INTRODUCTION

Depression is one among the most rampant form of psychiatric disorders and a leading cause for morbidity and mortality[1].

Desvenlafaxine succinate (DSV) is a newer antidepressant drug which is chemically RS-4-[2-dimethylamino-1-(1-hydroxycyclohexyl) ethyl] phenol succinate monohydrate **(Fig. 1)**. The FDA approved desvenlafaxine in February 2008 for the treatment of patients with major depressive disorder [2]. Moreover, desvenlafaxine is also being investigated as the first non-hormonal treatment for vasomotor symptoms attributed to menopause [3]. Venlafaxine is mainly metabolized in the liver to desvenlafaxine by cytochrome P450 2D6 (CYP2D6). Desvenlafaxine is pharmacologically active and is not metabolised by CYP2D6 and is excreted unchanged or after conjugation [4].

There is no reference for determination of desvenlafaxine succinate in official compendia. Several methods have been reported in the literature for simultaneous determination of the DSV with venlafaxine in plasma and biological fluids using HPLC coupled to spectrophotometric [5-8], spectrofluorimetric [9-12] or coulometric detection [13]. Capillary electrophoresis.[14–20], HPLC-ESI/MS [21] and LC-MS/MS [22-27] have been used as well .

To our knowledge, simple UV Spectrophotometric method and difference spectrophotometric method have been developed for the estimation of desvenlafaxine succinate in tablet dosage form[28]. Dimal A. Shah et al reported that the DSV is stable to acid hydrolysis at room temperature and at 70°C [29].

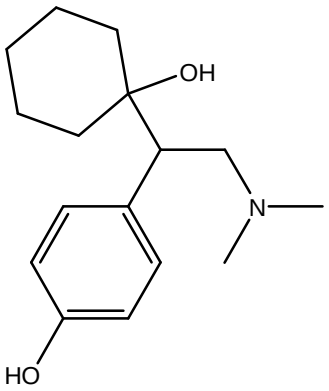


Figure 1: Chemical structure of Desvenlafaxine

The International Conference on Harmonization (ICH) guidelines [30] require the implementation of stress testing procedures for the identification of degradation products that are potentially occurring in drug substances which can help to understand the possible degradation pathway for the drug. Thus in this work, we described forced degradation of DSV under acidic condition and the product was characterized by IR, MS and ^{1}H NMR spectral data. Today, RP-HPLC is the most popular analytical technique for separating complex mixtures in the chemical, pharmaceutical and biotechnological industry. RP-HPLC is the opposite of normal-phase chromatography, with a nonpolar stationary phase and a polar, largely aqueous mobile phase.

The most common stationary phases used are octadecyldimethyl (C18) phases with silica as the solid support [31].

The aim of the present study was to develop and validate a simple, isocratic RP-HPLC method for the determination of DSV in tablets in the presence of its acid degradation product.

EXPERIMENTAL SECTION

Materials and Reagents:

All chemicals and reagents used were of analytical or pharmaceutical grade. Solvents were of HPLC grade .Analytically pure DSV was purchased from AFINE CHEMICALS LIMITED, Hangzhou, China its purity found to be 99.33 %. D-VENIZ 50 tablets (SUN pharmaceutical ind. Ltd.; each tablet was labeled to contain desvenlafaxine succinate equivalent to 50 mg desvenlafaxine. Acetonitrile, HPLC grade (LAB SCAN), Water, HPLC grade(LAB SCAN), Potassium dihydrogen phosphate(Adwic) and Ortho phosphoric acid were used .

Diluent: A mixture of acetonitrile: Water in the ratio 50:50 v/v.

Instrument used

The HPLC system consisted of Younglin instrument equipped with gradient HPLC pump 9000, mixer and degasser, and 9000 UV-detector ,manual 20 µL loop, autochrome3000 software system controller. The separation is made on Discovery® C-18 column, 5 µm particle size (250 × 4.6 mm). The samples are injected with a 100 µl Agilent analytical syringe. Teflonmembrane filter, pore size 0.45 µm and 47mmdiameter for solvents, Teflon disposable membrane filter pore size 0.45 µm for samples. An ultrasonic, Soniclean 120T, Australia, A digital pH meter and HANNA HI 9321, Portugal were used.

Methodology

Chromatographic conditions

Chromatographic separation was achieved at ambient temperature on a reversed phase column. The mobile phase consisted of acetonitrile : 5 mM potassium dihydrogen phosphate solution (50:50v/v) at a flow rate of 0.7 ml/min. Potassium dihydrogen phosphate solution was prepared by dissolving 680mg KH_2PO_4 in 1000ml HPLC grade water. Final pH of the mobile phase was adjusted to 3.8 by orthophosphoric acid. The mobile phase so prepared was filtered through 0.22 µm nylon membrane filter and degassed by sonication. Flow rate of 0.7ml / min was maintained. Detection was carried out at 229 nm. The injection volume was 20 µl for assay and degradation level.

Preparation of stock solutions

preparation of intact DSV standard solution

An accurate weight of DSV (25 mg) was introduced into a 25 ml volumetric flask, dissolved in and completed to volume with water to prepare a stock solution of concentration (1000µg/ml). Aliquots (5, 6 and 7 ml) of stock solution were diluted with diluent to prepare working standard solutions of DSV of concentration100, 120 and 140µg/ml.

Preparation of Acid Induced Degradation Product.
Phosphoric acid (1ml) was added dropwise to a solution of DSV (500mg) in diethyl ether (10ml) in ice bath during addition and keeping the stirring for 10 hours at room temperature. The pH of the reaction mixtures was adjusted to 7.5 with 10% sodium carbonate solution where the product was precipitated , the diethyl ether was allowed to evaporate and then the formed product was filtered, dried and recrystallized from acetonitrile.

The Deg. product was accurately weighed (25 mg), transferred to a 100 ml volumetric flask, dissolved and diluted up to the mark with the diluent (250µg/ml).

Preparation of D-VENIZ 50 tablets solution:
Twenty tablets were accurately weighed and finely powdered. An amount of powder equivalent to 25 mg DSV was transferred to 100 ml volumetric flask containing 70 ml water, sonicated for 15 minutes, diluted up to the mark with water and filtered discarding the first 15 ml. An aliquot (25ml) was transferred to 50 ml volumetric flask and complete to volume with acetonitrile to prepare tablet solution of concentration (125µg/ml).

Method Validation
System suitability
According to the USP 28, the system suitability tests are an integral part of chromatographic analysis and should be used to verify that the resolution and reproducibility of the system are adequate for the analysis to be performed.

To ascertain the effectiveness of the method developed in this study, system suitability tests were performed on standard stock solutions of DSV and its acidic degradation product.

Linearity and Limits of Detection and Quantitation
Accurately measured aliquots of working standard solution DSV (100µg/ml) equivalent to (50-1000µg)were transferred separately into a series of 10 ml volumetric flasks and diluted up to the mark with diluent. Twenty micro liters for each solution was injected in duplicate injection in to Discovery C18 column and the chromatogram was recorded at a flow rate of 0.7 ml/min.; the eluent was monitored at 229 nm. The area under peaks (AUPs) were recorded and used for the construction of calibration curve. A regression equation was computed.

LOD and LOQ were calculated using following equation as per ICH guidelines. LOD = 3.3 ×σ /S and LOQ = 10 ×σ /S, where σ is the standard deviation of y-intercept of regression line and S is the slope of the calibration curve.

Precision
Pure samples of DSV (24, 66& 90 µg/ml) were analyzed over different days to obtain inter-day(intermediate precision, n = 3) and within the same day to obtain intra-day precision (repeatability, n = 3) , then the RSDs % values were calculated

Accuracy
Aliquot portions of DSV working standard solution (120 µg /ml) equivalent to (120-840 µg) were transferred into a series of 10 ml volumetric flasks and complete to volume with diluent.

The assay was completed as described under linearity procedure . The concentration was calculated using the corresponding regression equation and the recovery percentages were calculated.

Selectivity

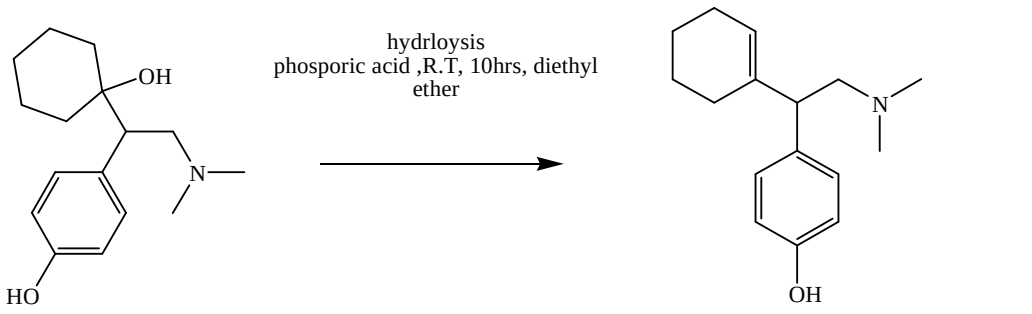
Accurately measured aliquots of working solution of DSV (100µg/ml) and Deg stock solution(250 µg/ml) equivalent to (50-850µg) and ((850-50 µg) respectively were transferred into a series of 10 ml volumetric flasks and the volumes were completed with the diluent to prepare different mixtures containing 5-95% of degradation products. The prepared mixtures were analyzed as previously described.

Robustness

In this study, the chromatographic parameters (retention time t_R, capacity factor K and tailing T) were evaluated using DSV and degradation solutions changing flow rate (0.6 ,0.7 and 0.8 ml/min), acetonitrile percentage in mobile phase(48,50,52%) and pH (3.6.3.8 and 4.0) of mobile phase .

Analysis of marketed formulation (D-VENIZ 50)

An aliquots equivalent to (62.5 & 315 µg) transferred to 10 ml volumetric flasks and completed to volume with the diluent. The solutions were chromatographed as mentioned previously. The experiment was repeated applying the standard addition technique. The recovered percentages of labeled & added DSV were calculated using regression equation.



Desvenlafaxine
(4-(2-(dimethylamino)-1-(1-hydroxycyclohexyl)ethyl)phenol)

Dehydro desvenlafaxine
(4-(1-cyclohexenyl-2-(dimethylamino)ethyl)phenol)

Scheme1: Possible degradation pathway of DSV

RESULTS AND DISCUSSION

Elucidation of the Degradation Products

In this work , it was found that the reaction of DSV in 2M HCL at 100 ^{0}C for 24h produce an additional late eluting peak and produce only 20-30% of degradation product. The hypothetical acid degraded product for desvenlafaxine would be result by the dehydration of cyclohexanol .The degradation product is less polar in nature and eluted slower than desvenlafaxine (**Fig2).**

As reviewed in the literatures reaction of venlafaxine, its dehydro derivative was synthesized by acid-catalyzed dehydration with sulfuric acid[32]. Thus the dehydro derivative of desvenlafaxine was successfully prepared by using phosphoric acid as strong dehydrating agent as shown in scheme 1.

The prepared acidic degradation product was elucidated by infra red (IR) spectrum where the absorption band at 3170.97 corresponding to OH of cyclohexanol disappeared, and appearance new absorption band at 1616 corresponding to (C=C).In ^{1}H NMR, in the 1H NMR spectrum, a singlet signal at 5.508 ppm corresponding to alkene proton with one proton integration was observed, the mass spectrum of degradation product showed a peak at m/z 245 representing the molecular ion peak.

Based on these spectral data, the acidic degradation product was confirmed as 4-(1-cyclohexenyl-2-(dimethylamino)ethyl)phenol.

Method of development

The Chromatographic conditions were optimized to separate the degradation product from drug. Several mobile phase compositions with different pH-values were employed including ammonium acetate buffers (pH 3, 4) which showed noise in chromatographic base line at retention time of DSV . The good separation was achieved using a mobile phase comprised of acetonitrile: 5mM potassium dihydrogen phosphate buffer (50;50, v/v) the pH was adjusted to 3.8 with orthophosphoric acid . The peaks were well defined, resolved and free from tailing. The percentage of acetonitrile was found to be critical in achieving the separation between the compounds. A percentage more than 55 % decrease resolution. The elution order was the degradation product at t_R = 4.6 min. and DSV t_R = 3.5 min. at 229 nm with a flow rate of 0.7 mL/min((**Figs. 3 & 4**).

System suitability

The system suitability parameters are presented in (**table 1**). All parameters met the recommended criteria of the ICH guidelines on validation of chromatographic methods, i.e., N should be > 2000, T should be < 2, K'>2 and R_S >2.

Validation of the method

Linearity

The linear relationship between area under the peak and concentration of DSV was obtained in the range of 5-100 μg.ml^{-1} and the regression equation was computed. The values of correlation coefficient, slope and intercept were 0.9999 , 34.295 and 12.564 respectively.

LOD and LOQ were found to be 1.146μg/ml and 3.476μg/ml respectively. The analytical data of the calibration curve including standard deviations for the slope and intercept (S_b, S_a) are also summarized in (**Table 2).**

Precision

The results of repeatability and intermediate precision experiments are shown in (**Table 3**). The developed method was found to be precise as the RSD% was < 2%, as recommended by ICH guidelines.

Robustness

Variations of flow rate (±0.1), mobile phase pH (±0.2) and acetonitrile percentage in mobile phase (±2.0) resulted in no significant changes, showing that this method is robust for small variations (**Table 4**).

Recovery studies

As shown from data in (**Table 5**) , The suggested method could be applied as a stability-indicating method for the determination of DSV in laboratory prepared mixtures containing 5-95 % (w/w) of its main degradant with mean percentage recovery of 101.63 ± 0.316 (n = 7)). Moreover, the chromatograms of the samples were checked for the appearance of any extra peaks. No chromatographic interference from any of the excipients was found at the retention times of the examined drug(**Fig. 5**). Good recoveries were obtained for the determination of DSV in bulk, where the mean percentage accuracies were 99.56±1.095. Also the proposed HPLC method could be successfully applied for the quantitative assay of DSV in its pharmaceutical formulation with good percentage recoveries. Results of standard addition and recovery experiment assessed the validity of the method as the percentage recovery of tablet and added were 98.51±0.44 and 99.36±1.27 respectively (**Table3**).

Table 1: System suitability tests

Parameter	Obtained value
Resolution (R)[a]	3.09
Tailing factor (T)[a]	1.633
Relative retention (α) time (selectivity)[a]	1.391
Column capacity (K)[a]	4.496
Column efficiency (N)[a]	5086
Height equivalent to theoretical plate(HETP)	0.005

Table 2:Results obtained by the proposed HPLC method for the determination of DSV in presence of its acid degradation product.

Item	value
Wavelength of detection	229nm
Linearity range	5-100 μg/ml
Slope	34.295
Intercept	12.564
Regression coefficient (r^2)	0.9999
Standard deviation of slope(S_b)	0.147
Standard deviation of the intercept(S_a)	8.937
LOD	1.147
LOQ	3.476
Confidence limit of the slope	33.859 -34.678
Confidence limit of the intercept	-10.316 -39.312
Standard error of the estimation	11.912
Recovery results	
1)Drug in bulk	99.56±1.0
2)Drug in dosage form	98.51±0.44
3) Drug added	99.36±1.27

Statistical comparison between the results obtained using the proposed HPLC method and the reported method [27] show no significant difference between them (**Table 6**).

Table 3: Precision studies

Concn. µg/ml	Intra-day		Inter-day	
	Recovery%±SD	RSD%	Recovery%±SD	RSD%
24	101.40±0.29	0.291	100.49±0.92	0.918
66	99.45 ± 0.31	0.310	99.01±0.463	0.468
90	100.04±1.82	1.814	100.59±0.33	0.325

Table 4 Robustness studies

Factor	Retention time(t_R)	Capacity factor(K)	Tailing factor(T)
Flow rate			
0.6	4.10	5.31	1.70
0.7	3.53	4.43	1.60
0.8	3.07	3.72	1.63
mean±SD	3.57±0.52	4.49±0.80	1.64±0.051
% of acetonitrile in mobile phase			
48	3.40	4.23	1.5
50	3.53	4.43	1.6
52	3.45	4.31	1.88
mean±SD	3.46±0.07	4.32±0.10	1.66±0.20
PH of mobile phase			
3.6	3.53	4.43	1.60
3.8	3.53	4.43	1.60
4.0	3.62	4.57	1.71
mean±SD	3.56±0.05	4.48±0.08	1.63±0.06

Table 5. Results of analysis of laboratory-prepared mixtures containing different percentages of DSV and its acidic degradation product (Deg) by the proposed HPLC method

Conc. DSV(µg/ml)	**[a]Deg. %**	**Conc. Found(DSV)**	**Recovery%**
5	5	5.08	101.60
25	30	25.51	102.04
35	40	35.63	101.80
45	50	45.75	101.67
55	60	55.84	101.53
65	70	66.15	101.77
85	95	85.87	101.02
mean±SD			101.63±0.316

a Calculated with respect to intact weight (degradation products-drug mixture wt/wt %).

Table (6): Tests of significance for the HPLC method proposed for the determination of DSV and its degradation product

Statistical Term	Reference method**	HPLC method
Mean	100.11	99.56
S.D.±	0.557	1.095
S.E.±	0.249	0.447
%RSD	0.311	1.099
n	6	6
V	0.310	1.199
t (2.201)*		1.075
F (4.95)*		3.867

** Figures in parentheses are the theoretical t and F values at (p= 0.05).*
*** spectrophotometric method[28].*

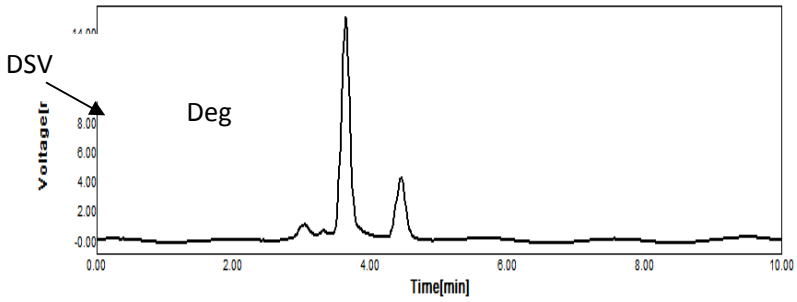


Fig2: Chromatogram of acid-treated (2M HCl at 100^{0}C for 24 h) Desvenlafaxine.

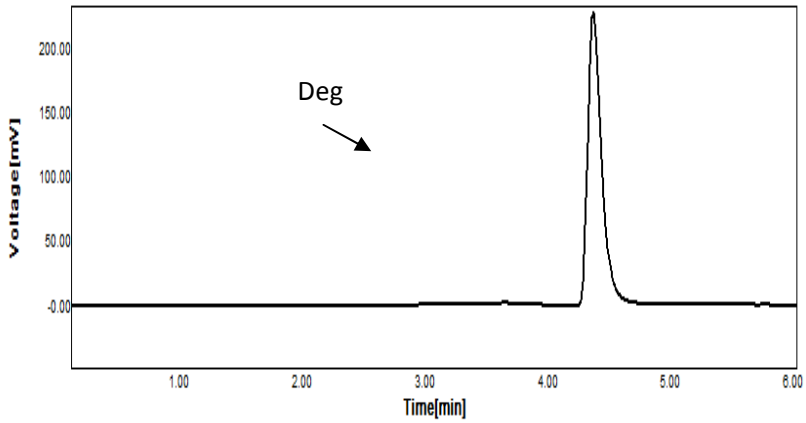


Fig3: The simple chromatogram of acidic degradation product(dehydro-desvenlafaxine)

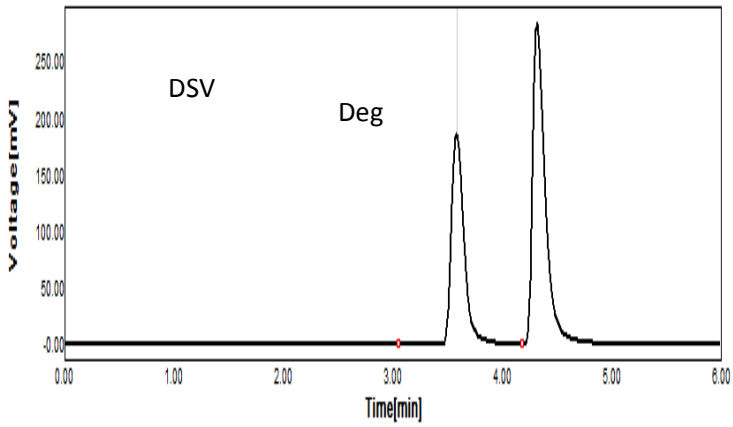


Fig 4: The simple chromatogram DSV and acidic degradation Product

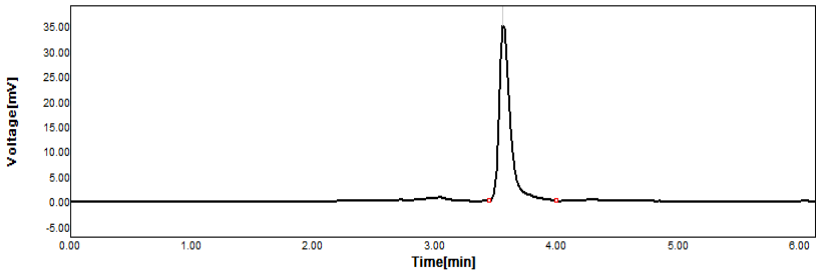


Fig 5: The simple chromatogram sample DSV

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18-Jul-11 11:33:40

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: A.GABR
Analyzed	: 18-Jul-11 11:29:16 AM
Sample Name	: DE
Sample ID	:
Customer Name	: YASMEEN ABDALLA
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Method File	: C:\GCMSsolution\Data\Project1\HG Trial.qgm
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Report File	:
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Modified	: 18-Jul-11 11:30:44 AM

Method

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Event Time	:0.50sec
Scan Speed	: 625
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End m/z	:350.00
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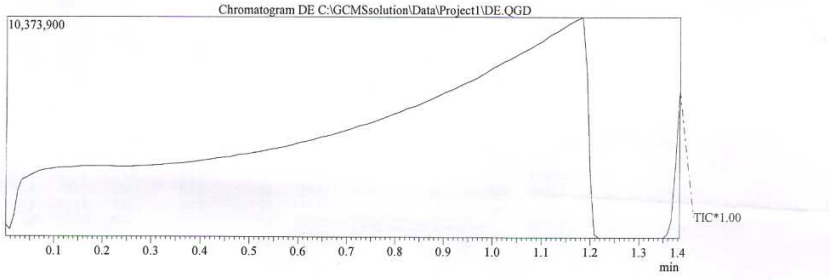
Chromatogram DE C:\GCMSsolution\Data\Project1\DE.QGD



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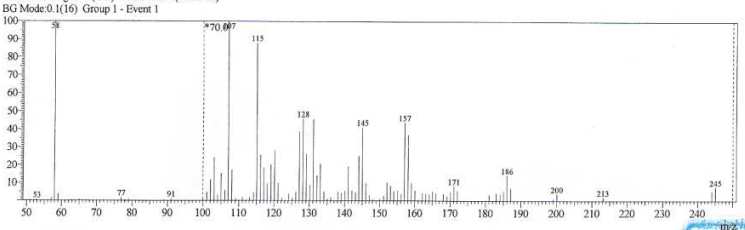
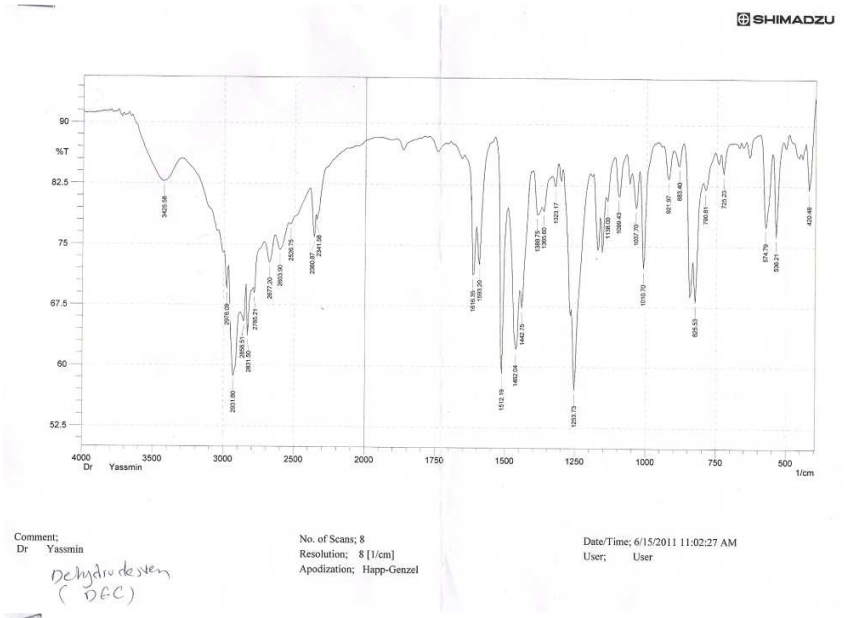


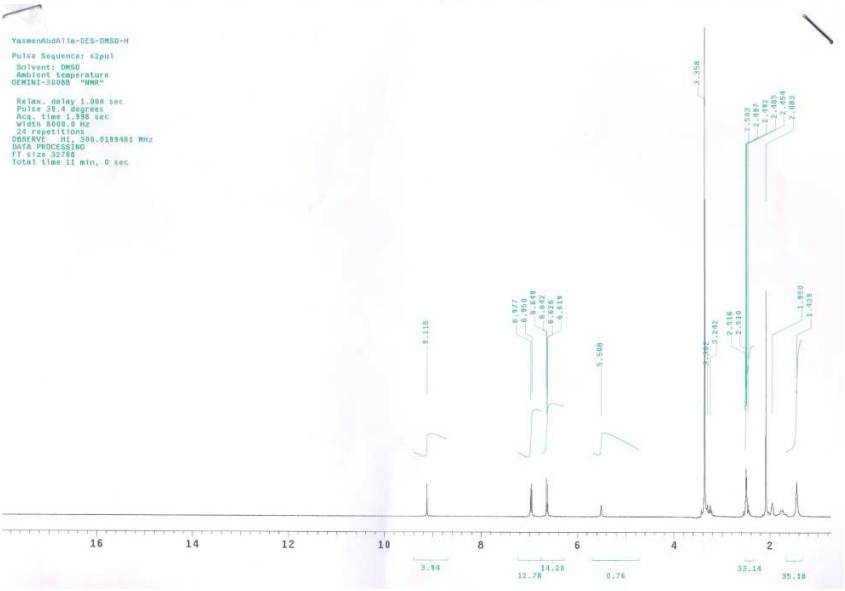
Mass Table

Line#:1 R.Time:1.1(Scan#:132)
MassPeaks:123
RawMode:Single 1.1(132) BasePeak:58(4515562)
BG Mode:0.1(16) Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.00	1392	0.03	4	53.00	23769	0.53	7	56.05	16417	0.36
2	51.00	17727	0.39	5	54.05	2587	0.06	8	57.05	74655	1.65
3	52.00	7996	0.18	6	55.00	21055	0.47	9	58.00	451556	100.00

1 / 2


SHIMADZU
%T
90
82.5
75
67.5
60
52.5
4000
3500
3000
2500
2000
1750
1500
1250
1000
750
500
1/cm
Dr Yassmin
Comment;
Dr Yassmin
Dehydrodesten
(DEC)
No. of Scans; 8
Resolution; 8 [1/cm]
Apodization; Happ-Genzel
Date/Time; 6/15/2011 11:02:27 AM
User; User


YasmenAbdAlla-DES-DMSO-H
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
GEMINI-300BB "NMR"
Relax. delay 1.000 sec
Pulse 30.4 degrees
Acq. time 1.598 sec
Width 8000.0 Hz
24 repetitions
OBSERVE H1, 300.0199481 MHz
DATA PROCESSING
FT size 32768
Total time 11 min, 0 sec
16
14
12
10
8
6
4
2
3.94
14.28
12.78
0.76
33.14
35.18