Supplementary Material (ESI) for Chemical Communications

SUPPLEMENTARY INFORMATION-I

Surfactant Mediated Oxygen Reuptake in Water for Green Aerobic Oxidation: Mass-spectrometric Determination of Discrete Intermediates to Correlate Oxygen Uptake with Oxidation Efficiency

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Index of the Supplementary Information

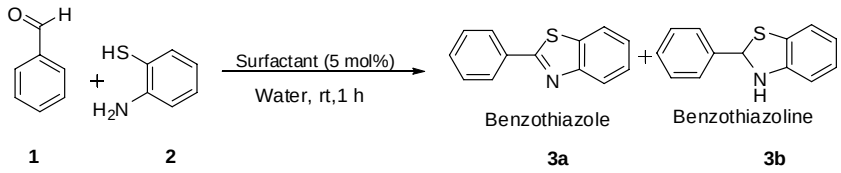
1. General Considerations

The glasswares were thoroughly washed and dried in an oven and the experiments were carried out with required precautions. Chemicals and all solvents were commercially available (Aldrich Chemical, Merck AG, Fluka and S-D Fine Chemicals) and used without further purification. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz NMR spectrometer in $CDCl_3$ with residual undeuterated solvent ($CDCl_3$: 7.26/77.0) using Me_3SiCl as an internal standard. Chemical shifts (δ) are given in ppm and *J* values are given in Hz. ^{13}C NMR spectra were fully decoupled and were referenced to the middle peak of the solvent $CDCl_3$ at 77.00 ppm. Splitting pattern were designated as s, singlet; bs, broad singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet. Mass spectra were recorded on a GCMS- QP 5000 (Shimadzu) [for EI] mass spectrometers. Infra-red (IR) spectra were recorded on Perkin Elmer FT-IR spectrometer in the range 4000-600 cm^{-1} either as neat samples or using KBr for preparing pellets for solid samples.

Open column chromatography, thin layer chromatography (TLC) was performed on Silica gel [CDH silica gel 60-120 mesh, F254 and Merck® silica gel respectively. Mass spectra for mechanistic study were recorded in advance Bruker Daltonics® MALDI-TOF instrument and ESI-MS in advance Thermo Scientific LTQ-XL mass spectrometer. Oxygen content of the medium was determined by the Oxygraph instrument. Evaporation of solvents was performed at reduced pressure, using a Bűchi rotary evaporator.

2. Experimental Procedures

A. Evaluation of catalytic efficiency of various surfactants for oxidative synthesis of benzothiazole and benzothiazoline:benzothiazole selectivity



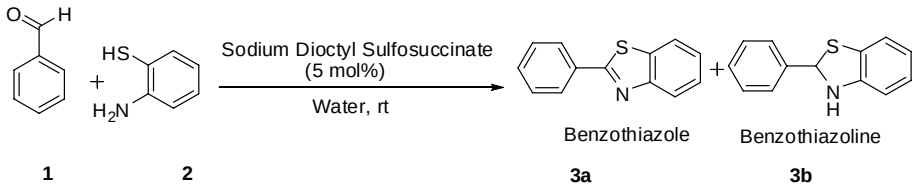
Supplementary Table 1[a]

Entry	Surfactant	1[b]	2[b]	3a[b]	3b[b]	3a/3b[c]	% Yield[d]
1	Nil[e]	08	02	17	72	19:81	--
2	Tween 80	00	00	89	11	89:11	82
3	Triton X-100	00	00	82	18	82:18	76
4	β-Cyclodextrin Hydrate	00	00	70	30	70:30	63
5	Benzalkonium Chloride	00	10	80	10	88:12	66
6	TBAB	00	5.20	87	08	91:09	79
7	CTAB	00	3.61	25	71	26:74	18
8	Hexadecyl Pyridinium Chloride	00	10	83	07	92:08	73
9	SDS	00	00	98	02	98:02	90
10	Sodium Deoxy Cholate	00	00	95	05	95:05	87
11	Sodium Dioctyl Sulfosuccinate	00	00	100	00	100:00	95
12	PEG-600	10	01	60	29	67:33	48
13	PEG-20000	22	04	40	34	54:46	32
14	Span 80	00	00	88	12	88:12	80

[a]Benzaldehyde (0.26 g, 2.5 mmol) was treated with 2-amino thiophenol (0.32 g, 2.5 mmol, 1 equiv) in presence of surfactant (5 mol%) at rt for 1 h. [b]The % conversion was determined by GCMS. [c] Calculated on the basis of total conversion to **3a** and **3b**. [d]Isolated yield of **3a** after column chromatographic purification. [e] Reaction was carried out without any surfactant.

B. Optimization of various reaction parameters for benzothiazoline:benzothiazole selectivity.

I) Time optimization study



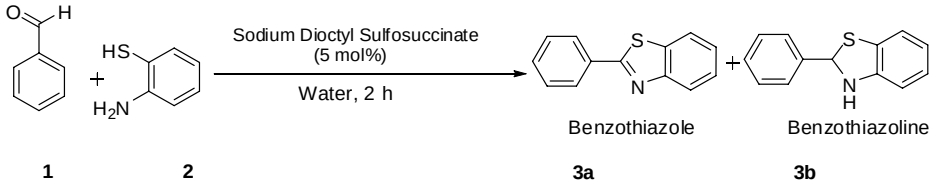
1 2 3a 3b

Supplementary Table 2[a]

Entry	Time (h)	3a[b]	3b[b]	% Yield[c]
1	0.5	98	2	88
2	1	100	0	95
3	2	100	0	95
4	3	100	0	96

[a]Benzaldehyde (0.26 g, 2.5 mmol) was treated with 2-amino thiophenol (0.32 g, 2.5 mmol, 1 equiv) in presence of sodium dioctyl sulfosuccinate (SDOSS) (0.22g, 5 mol%) at rt for different time duration. [b]The % conversion was determined by GCMS.[c] Isolated yield of **3a** after column chromatographic purification.

II) Optimization of temperature



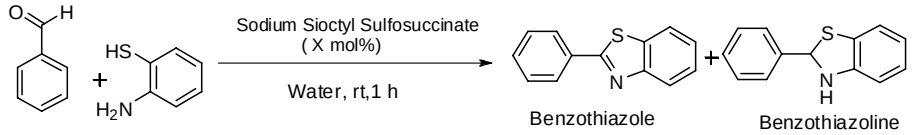
1 2 3a 3b

Supplementary Table 3[a]

Entry	Temp (°C)	Time (min)	3a[b]	3b[b]	%Yield[c]
1	rt (30-35)	60	100	00	95
2	60	15	89	11	84
3	60	30	100	00	94
4	60	45	100	00	94
5	60	60	100	00	94
6	100	15	90	10	85
7	100	30	100	00	95
8	100	45	100	00	95
9	100	60	100	00	95

[a]Benzaldehyde (0.26 g, 2.5 mmol), 2-amino thiophenol (0.32 g, 2.5 mmol, 1 equiv) in presence of SDOSS (0.22g, 5 mol%) at different temperature for 2 h. [b]The % conversion was determined by GCMS. [c] Isolated yield of **3a** after column chromatographic purification.

III) Optimization of the amount of SDOSS



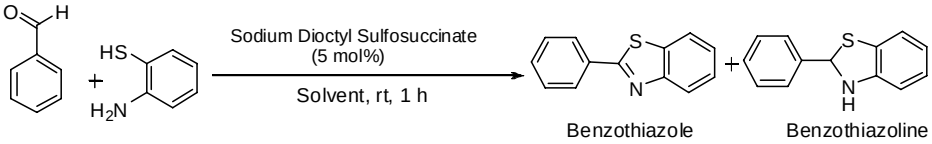
1 2 3a 3b

Supplementary Table 4[a]

Entry	SDOSS (mol%)	Time (h)	3a[b]	3b[b]	%Yield [c]
1	0.5	3	85	15	70
2	1	2.5	83	17	68
3	2	2	85	15	75
4	4	1.5	96	4	89
5	5	1	100	0	95
6	10	1	100	0	95
7	15	1	100	0	96

[a]Benzaldehyde (0.26 g, 2.5 mmol) was treated with 2-amino thiophenol (0.32 g, 2.5 mmol, 1 equiv) using different amount of SDOSS at rt for different time. [b]The % conversion was determined by GCMS. [c]Isolated yield of **3a** after column chromatographic purification.

IV) Solvent study



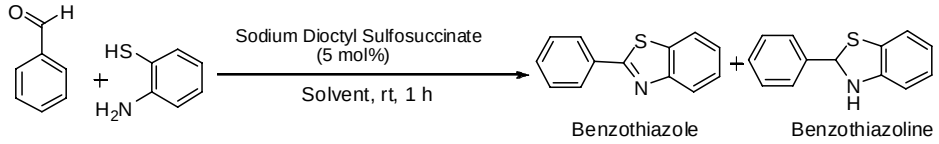
1 2 3a 3b

Supplementary Table 5[a]

Entry	Solvent	1[b]	3a[b]	3b[b]	% Yield[c]
1	Neat [d]	2	17	77	--
2	Water [d,e]	8	17	72	--
3	Water	0	100	0	95
4	THF [d]	66	20	06	--
5	DMF [d]	37	31	31	18
6	Hexane	0	64	32	56
7	Dioxane	31	36	36	32
8	DCM	0	76	23	68
9	Ethanol	5	54	40	47
10	Methanol	14	47	35	38
11	*t* Butanol[d]	3	47	45	37
12	*i* Propanol[d]	2	49	43	38

[a]Benzaldehyde (0.26 g, 2.5 mmol) was treated with 2-amino thiophenol (0.32 g, 2.5 mmol, 1 equiv) using 5 mol % of SDOSS in different solvents at rt for 1 h. [b] The % conversion was determined by GCMS.[c] Isolated yield of **3a** after column chromatographic purification. [d]4, 3, 8, 4, 1, 4, 4 and 5 % 2-amino thiophenol (**2**) was found unreacted for the entries 1, 2, 4, 6, 8, 10, 12, and 13 respectively (GCMS). [e]Reaction was carried out without any surfactant.

V) Detailed GCMS study of Monitoring the Progress of the Reaction under Different Conditions.



1 **2** **3a** **3b**

Supplementary Table 6[a]

Entry	Solvent	Time (min)	1 (%)[b]	2 (%)[b]	3a (%)[b]	3b (%)[b]	3a : 3b[b]
1	**Water**[c]	5	08	01	13	78	14:86
2		10	02	01	14	83	14:86
3		15	03	01	13	83	14:86
4		30	05	02	16	77	17:83
5		60	08	02	17	72	19:81
6	**Water**	5	00	00	39	61	39:61
7		10	00	00	45	56	45:56
8		15	00	01	56	43	57:43
9		30	00	04	66	30	69:31
10		60	00	00	100	00	100:00
11	**Water**[d]	5	00	01	76	23	77:23
12		10	00	00	91	09	91:09
13		15	00	00	95	5	95 : 05
14		30	00	00	100	00	100:00
15		60	00	00	100	00	100:00
16	**Water**[c,d]	5	02	04	16	78	17:83
17		10	00	00	39	61	39:61
18		15	00	00	44	56	44:56
19		30	00	00	45	55	45:55
20		60	00	03	51	46	53:47

21	**Water**[e]	5	07	01	12	80	13:87
22		10	02	04	16	78	17:83
23		15	02	04	17	77	18:82
24		30	01	03	31	65	32:68
25		60	00	03	51	46	53:47
26	**DMF**	5	46	09	22	23	49:51
27		10	36	05	32	26	55:45
28		15	31	03	35	31	53:47
29		30	22	04	40	34	54:46
30		60	22	02	47	29	62:38
31	**Hexane**	5	67	13	13	07	65:35
32		10	14	01	23	62	27:73
33		15	14	01	31	53	37:63
34		30	11	01	59	29	67:33
35		60	00	04	64	32	67:33
36	**DCM**	5	37	41	8	14	36:64
37		10	28	30	16	25	39:61
38		15	15	17	20	48	29:71
39		30	05	08	28	59	32:68
40		60	00	00	76	23	77:23
41	**MeOH**	5	68	17	8	7	53:47
42		10	56	12	20	12	62:38
43		15	51	12	23	14	62:38
44		30	43	12	29	16	64:36
45		60	14	4	47	35	57:23
46	**THF**	5	80	05	08	07	53:47
47		10	77	06	11	05	68:32
48		15	75	06	14	05	74:26
49		30	77	04	13	06	72:28
50		60	66	08	20	06	77:23

[a]The mixture of **1** (0.26 g, 2.5 mmol), **2** (0.32 g, 2.5 mmol, 1 equiv) and SDOSS (5 mol%) in various solvents (5 mL) was stirred magnetically at rt (~35 - 40 °C) for 1 h. [b]The conversion was determined

by GCMS. [c]Reaction was carried out in the absence of SDOSS. [d] Oxygen gas was bubbled through the reaction mixture during the course of reaction. [e] Reaction was carried out in degassed water.

C. Oxygraph study

I) Determination of oxygen contents

Supplementary Table 8[a]

Entry	Catalyst	Catalyst amount (mol%)	Oxygen Content (ng/mL)	Relative Oxygen Content (nmol/mL)
1	NIL[b]	0.0	150.1	0
2	β – Cyclo Dextrin	5.0	218.0	2.12
3	CTAB	5.0	200.0	1.56
4	Sodium deoxycholate	5.0	244.9	2.96
5	Sodium dodecyl sulphate	5.0	251.5	3.17
6	SPAN 80	5.0	225.2	2.35
7	TWEEN 80	5.0	227.2	2.41
8	Tetrabutyl ammonium bromide	5.0	223.2	2.28
9	Triton X 100	5.0	236.1	2.69
10	Sodium dioctyl sulfosuccinate	2.0	246.8	3.02
11	Sodium dioctyl sulfosuccinate	4.0	252.4	3.20
12	Sodium dioctyl sulfosuccinate	5.0	259.2	3.41
13	Sodium dioctyl sulfosuccinate	10.0	260.6	3.45

[a]To an amount of the surfactant (that corresponds to 5 mol % of 1 mmol of benzaldehyde) was added 2 mL of ELGA water and the mixture was stirred for 30 min at rt. One mL of the resultant solution/suspension was used for oxygen content determination by Oxygraph at rt and at 70 RPM upto a total duration of 5 min. [b]One mL of ELGA water was taken and the oxygen content was determined by Oxygraph (microelectrode) at rt and at 70 RPM upto a total duration of 5 min.

D. Typical procedure for cyclocondensation of an aldehyde with 2-aminothiophenol for synthesis of benzothiazoles

Representative experimental procedure for formation of 2-phenyl-benzothiazole: To the magnetically stirred suspension of sodium dioctyl sulfosuccinate (0.22 g, 5 mol%) in water (5 mL) was added to benzaldehyde (0.21 g, 2 mmol) and 2-aminothiophenol (0.25 g, 2 mmol) and the mixture was stirred magnetically at rt. After completion of reaction (TLC, 1 h), the reaction mixture was extracted with EtOAc (3 × 5 mL) and the combined EtOAc extracts were dried ($MgSO_4$) and concentrated under rotary vacuum evaporation. The crude product was recrystallized using methanol to give 2-phenylbenzothiazole (0.4 g, 95%). White solid (lit.,[1] 109-112°C); IR (KBr) ν : 3061, 1505, 1474, 1451, 1429, 1310, 1284, 1221, 1154, 1121, 1067, 960, 762, 686, 617 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.39 (t, 1H, *J* = 7.47 Hz), 7.50 (m, 4H), 7.91 (d, 1H, *J* = 7.83 Hz), 8.07-8.09 (m 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ

121.63, 123.24, 121.73, 125.30, 126.32, 127.57, 129.03, 130.98, 133.63, 135.07, 154.15, 168.08; MS (ESI): m/z = 211 (M^+).

E. Procedure for GCMS study

Determination of benzothiazoline:benzothiazole selectivty during cyclocondensation of benzaldehyde with 2-aminothiophenol in the presence of surfactant:

To the magnetically stirred suspension of surfactant (5 mol%) in water (5 mL) was added benzaldehyde (0.21 g, 2 mmol) and 2-aminothiophenol (0.25 g, 2 mmol) and the mixture was stirred at rt. After completion of reaction (TLC, 1 h), the reaction mixture was diluted with EtOAc (5 mL). An aliquot portion (100 μL) of the supernatent EtOAc layer was taken out and subjected to GCMS to observe the benzothiazoline:benzothiazole selectivity.

Determination of progress of reaction at different time intervals in various aqueous/organic reaction media in terms of benzothiazoline:benzothiazole selectivity by GCMS during cyclocondensation of benzaldehyde with 2-aminothiophenol:

In case of aqueous media, the magnetically stirred suspension of SDOSS (0.22 g, 5 mol%) in water (5 mL) was added benzaldehyde (0.21 g, 2 mmol) and 2-aminothiophenol (0.25 g, 2 mmol) and the mixture was stirred at rt and progress of reaction was monitored by GCMS. At a particular time interval the reaction mixture was diluted with EtOAc (5 mL) and an aliquot portion (500 μL) of the supernatent EtOAc layer was taken out and subjected to GCMS to observe the benzothiazoline:benzothiazole selectivity.

In case of solvent study where organic solvents were used as reaction medium, an aliquot portion (500 μL) samples were directly submitted for GCMS without any further dilution/addition by EtOAc to observe the benzothiazoline:benzothiazole selectivity.

F. Procedure for determination of oxygen content of the medium

Experimental Procedure for the Determination of Oxygen of the Medium in the Presence and Absence of Various surfactants: (Table 7)

Oxygen content of the medium was determined by the Oxygraph instrument. In a blank experiment ultrapure water (1 mL) (27 °C, 18.2 Ω) was taken into the cuvette of the Oxygraph and the oxygen content was recorded (0-5 min) at 70 rpm. Following similar procedure the oxygen content/uptake of freshly prepared 1 mL of 0.02 M solution of the surfactant in ultrapure water (27 °C, 18.2 Ω) was recorded. The actual oxygen content was determined by substracting the blank reading from the corresponding reading of the analyte solution at 5 min and normalised to nmol/mL.

G. Procedure for Ion-fishing using +ve ESI MS for Determination of Oxygen Adduct with Different Surfactants:

An aliquot portion (50 μL) of the solution of surfactant (0.02 M) in 5 mL of water-acetonitrile (1:1) was subjected to (+ve) ESI MS in advance Thermo Scientific LTQ-XL mass spectrometer and the TIC was recorded.

H. Procedure for Determination of Ion Current of SDOSS with its Oxygen Adduct

An aliquot portion (10 μL) of the solution of SDOSS (0.22 g) in 5 mL of water-acetonitrile (1:1) was subjected to (+ve) ESI MS in advance Thermo Scientific LTQ-XL mass spectrometer and the ion current was determined by measuring the area of the ion peak corresponding to oxygen adduct of SDOSS.

I. References:

[1] G. Evindar, R. A. Batey, *J. Org. Chem*. **2006**, *71*, 1802–1808.
[2] T. Itoh, T. Mase, *Org. Lett*. **2007**, *9*, 3687–3689.
[3] A. K. Chakraborti, S. Rudrawar, G. Kaur, L. Sharma, *Synlett* **2004**, 1533–1536.

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SUPPLEMENTARY INFORMATION-II

Surfactant Mediated Oxygen Reuptake in Water for Green Aerobic Oxidation: Mass-spectrometric Determination of Discrete Intermediates to Correlate Oxygen Uptake with Oxidation Efficiency

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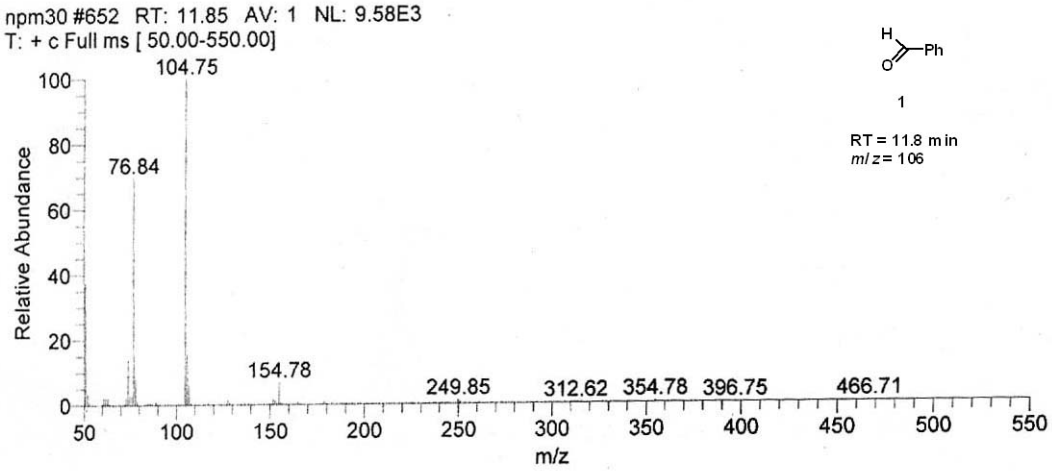
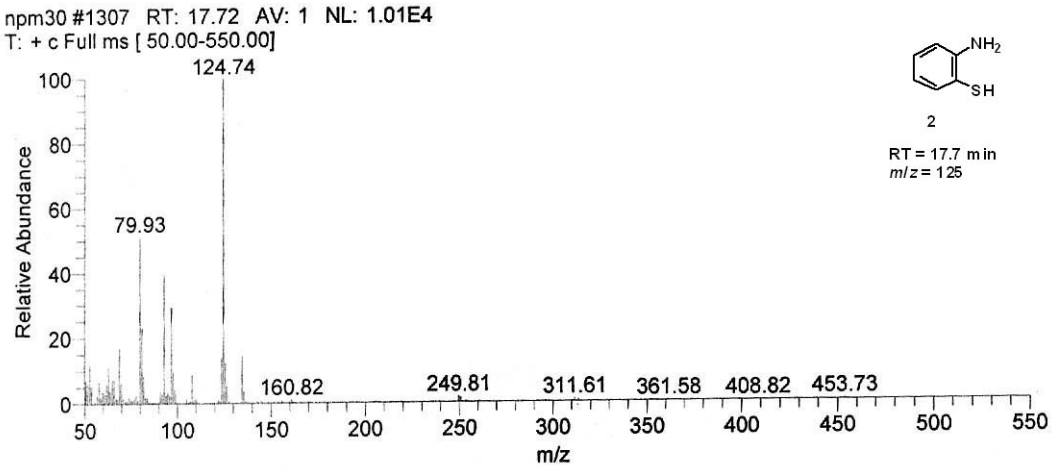
3. Scanned Spectra and supplementary figures

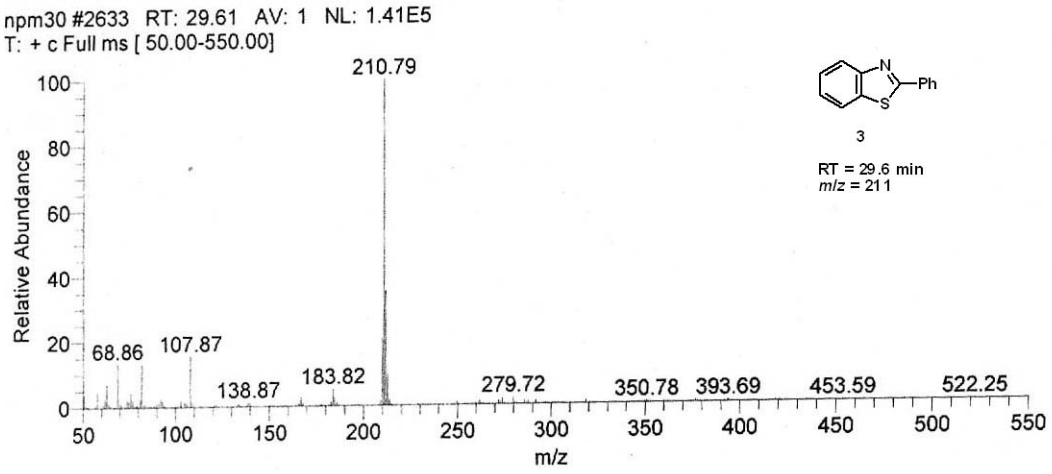
A. GCMS Spectra; Benzaldehyde



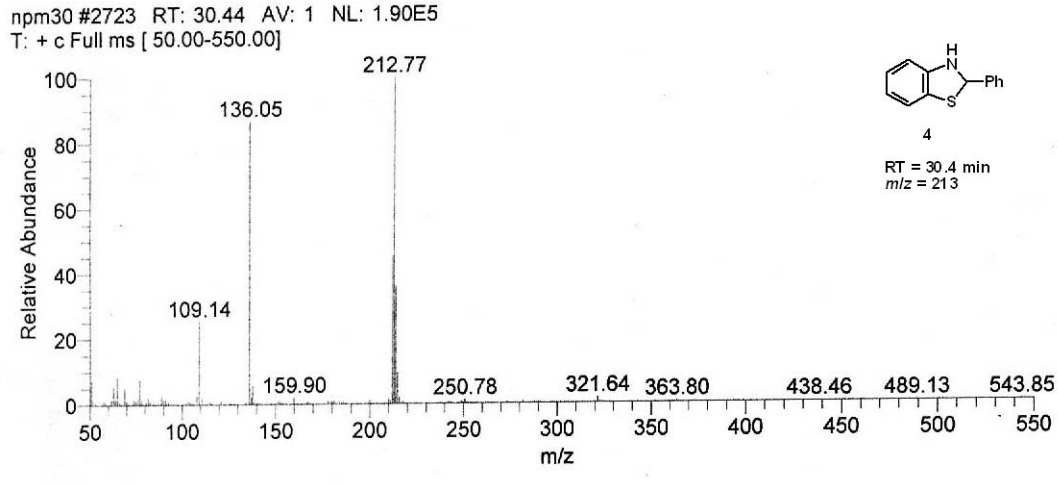
GCMS Spectra; 2-Aminothiophenol



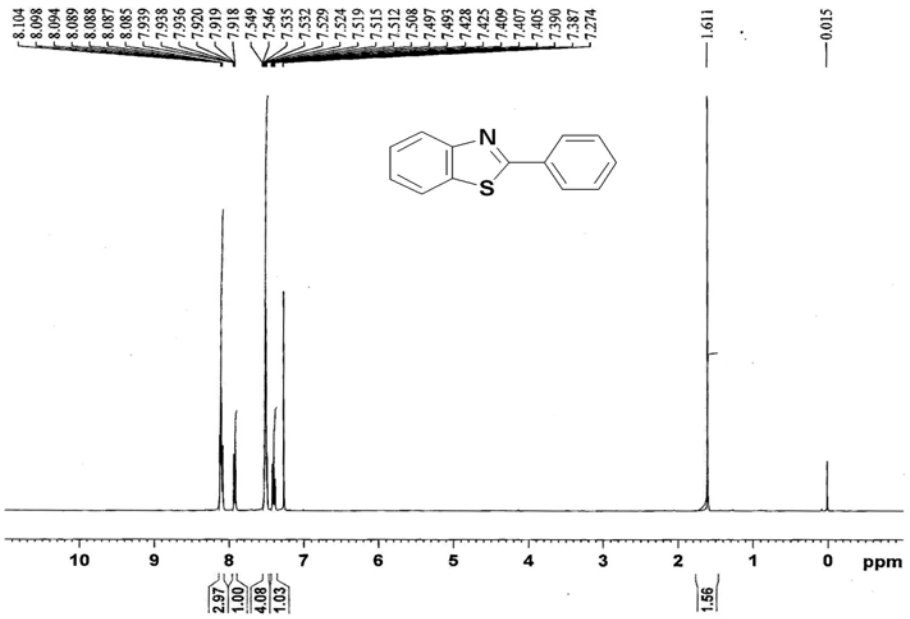
B. GCMS Spectra; 2-Phenyl benzothiazole



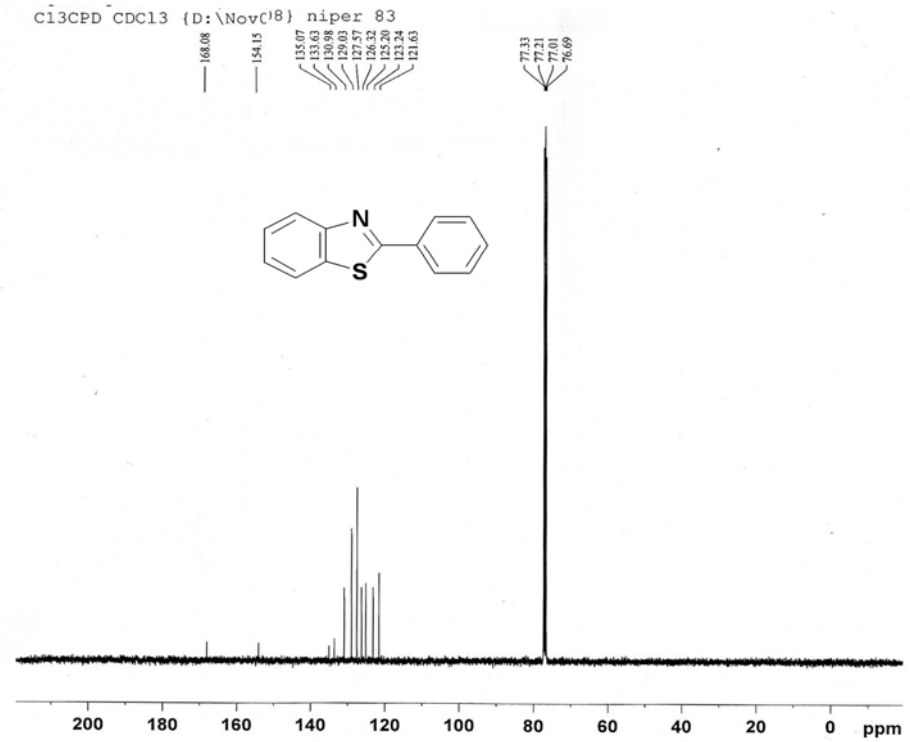
GCMS Spectra; 2-Phenyl benzothiazoline



C. ^{1}H NMR (400 MHz, $CDCl_3$, 27°C, TMS) Spectra; 2-Phenyl benzothiazole

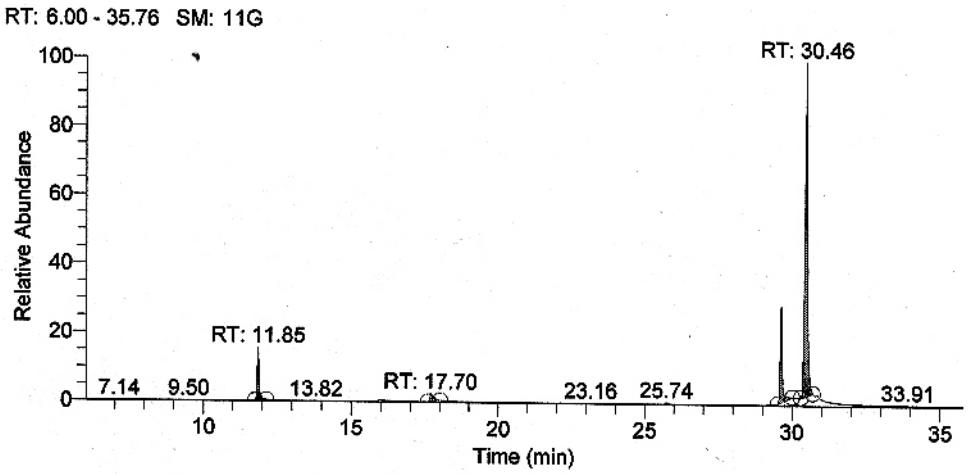


^{13}C NMR (100 MHz, $CDCl_3$, 27°C, TMS); Spectra; 2-Phenyl benzothiazole



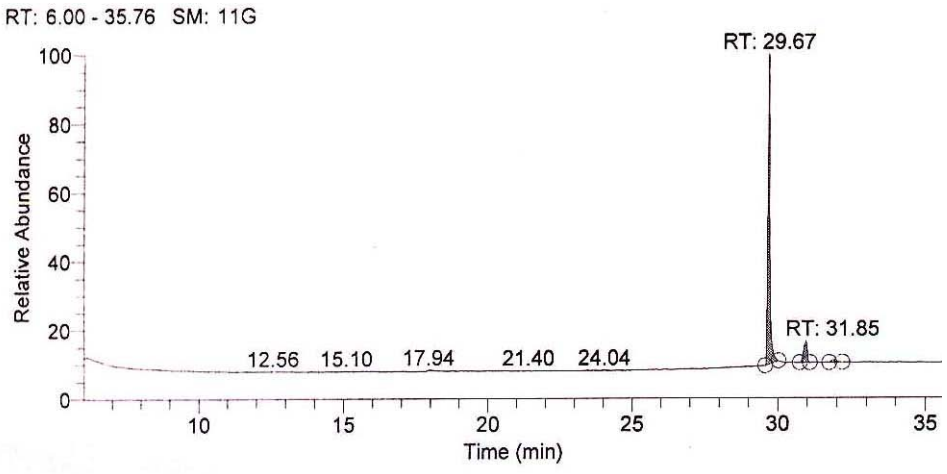
D. GCMS spectra relating screening of various surfactants for the synthesis of 2-phenyl benzothiazole:

GCMS Spectra; Water without surfactant (Entry 1, Table 1)



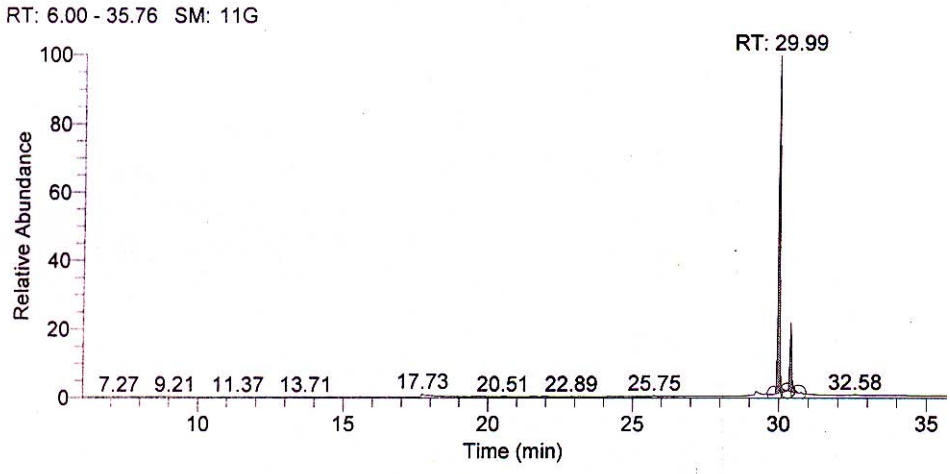
RT	Peak Area	Area %
11.85	487201	8.33
17.70	139809	2.39
29.63	991892	16.95
30.46	4232947	72.34

GCMS Spectra; Water with Tween-40 (Entry 2, Table 1)



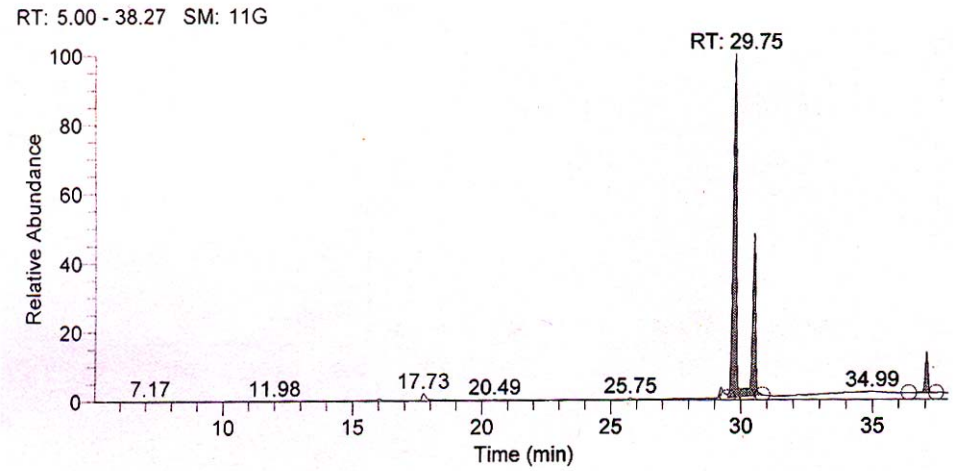
RT	Peak Area	Area %
29.67	1855753	87.65
30.96	227718	10.76
31.85	33644	1.59

GCMS Spectra; Water with Triton-80 (Entry 3, Table 1)



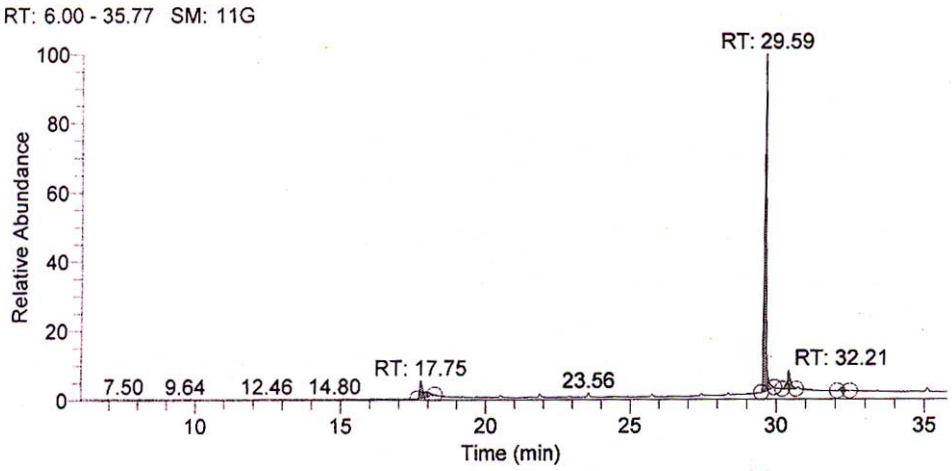
RT	Peak Area	Area %
29.99	7958123	81.54
30.39	1801958	18.46

GCMS Spectra; Water with *β*-Cyclodextrin hydrate (Entry 4, Table 1)



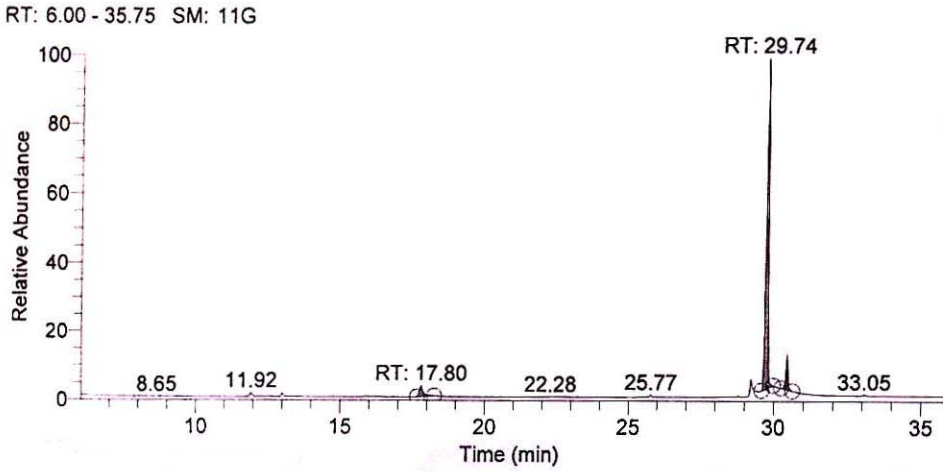
RT	Peak Area	Area %
29.75	506980740	65.73
30.50	212283855	27.52
37.04	52013424	6.74

GCMS Spectra; Water with Benzalkonium chloride (Entry 5, Table 1)



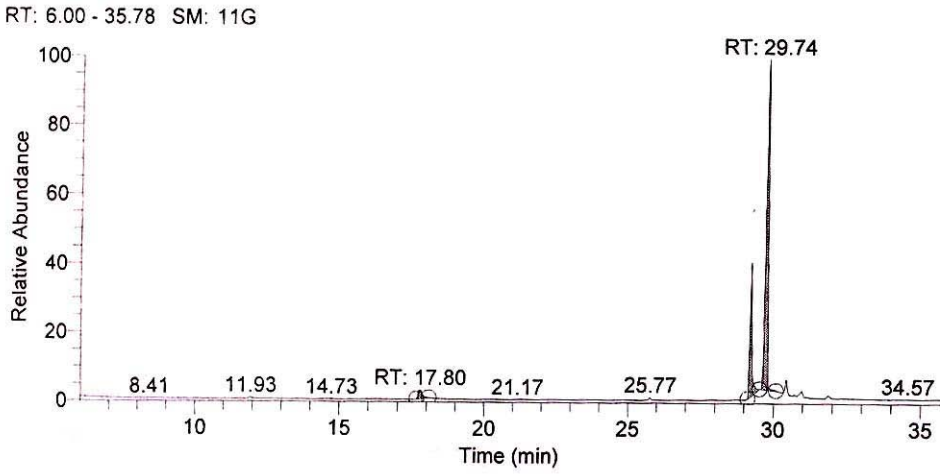
RT	Peak Area	Area %
17.75	122336	10.17
29.59	956644	79.55
30.41	102652	8.54
32.21	20905	1.74

GCMS Spectra; Water with TBAB (Entry 6, Table 1)



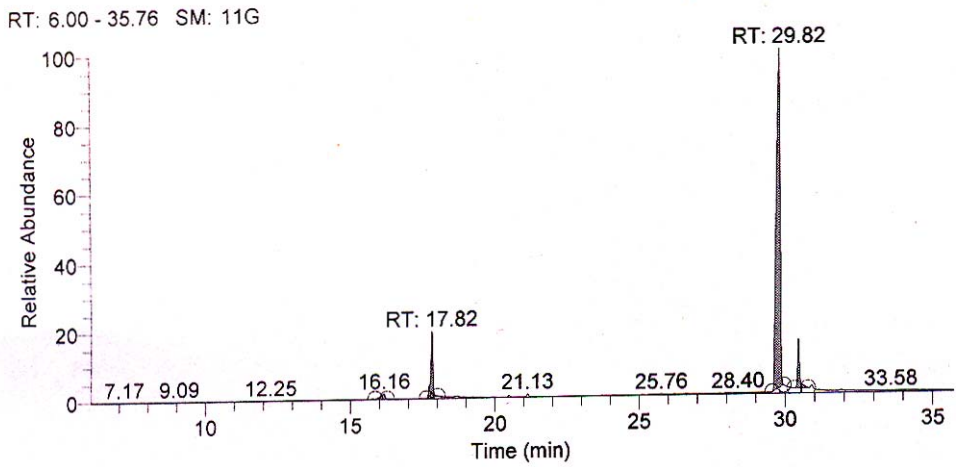
RT	Peak Area	Area %
17.80	560088	5.20
29.74	9324398	86.62
30.44	880740	8.18

GCMS Spectra; Water with CTAB (Entry 7, Table 1)



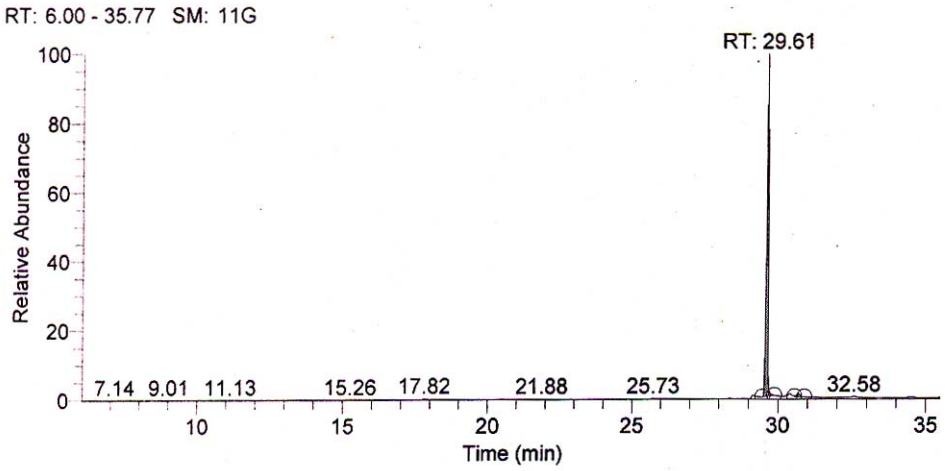
RT	Peak Area	Area %
17.80	455263	3.61
29.21	3201317	25.36
29.74	8967202	71.03

GCMS Spectra; Water with Hexadecyl pyridinium chloride (Entry 8, Table 1)



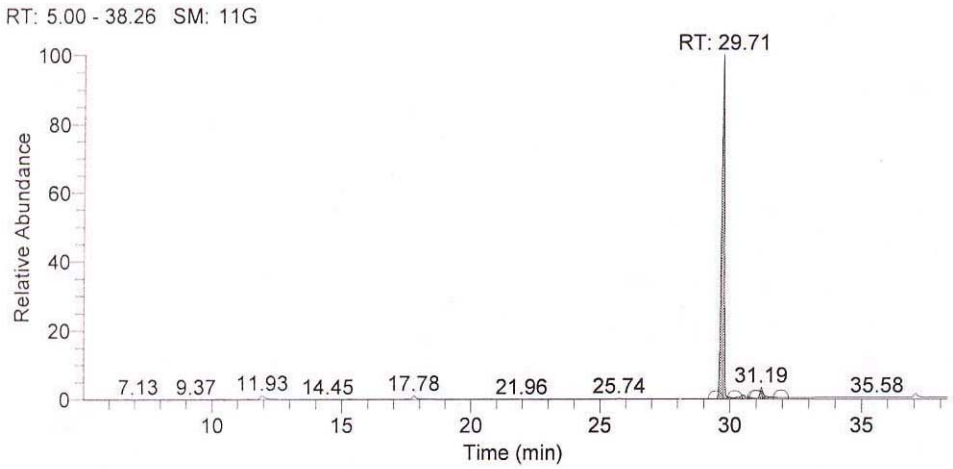
RT	Peak Area	Area %
16.16	14374610	1.44
17.82	93789382	9.43
29.82	822447489	82.67
30.46	64255736	6.46

GCMS Spectra; Water with Sodium dodecylsulfate (Entry 9, Table 1)



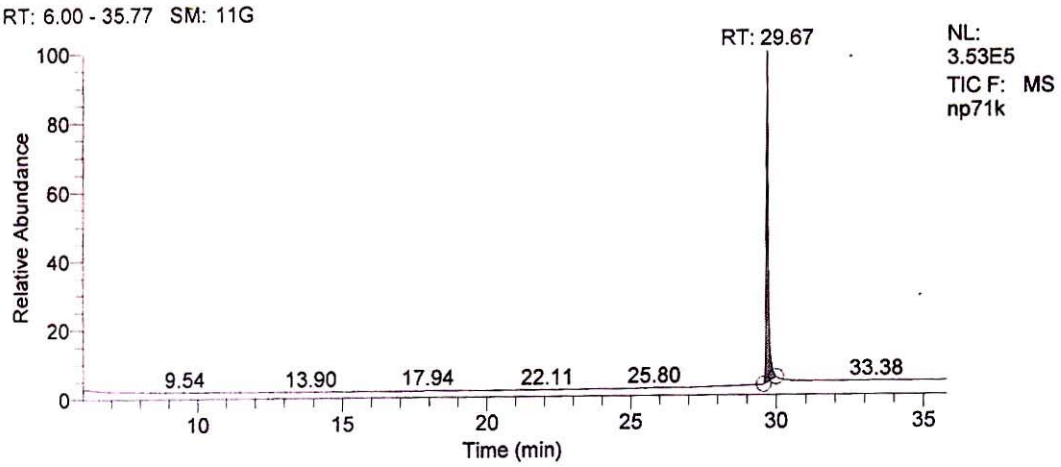
RT	Peak Area	Area %
29.61	6940910	98.13
30.73	132164	1.87

GCMS Spectra; Water with Sodium deoxycholate (Entry 10, Table 1)



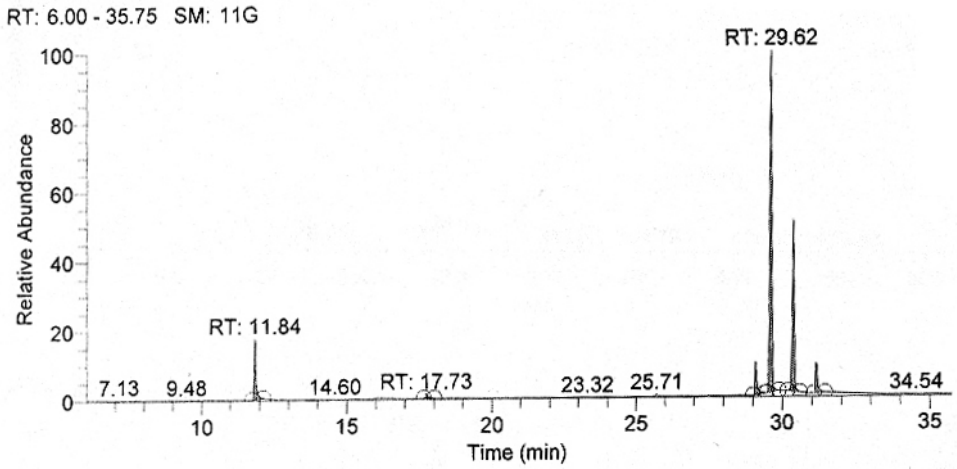
RT	Peak Area	Area %
29.71	7093187	94.14
30.49	156382	2.08
31.19	285406	3.79

GCMS Spectra; Water with Sodium dioctyl sulfosuccinate (Entry 11, Table 1)



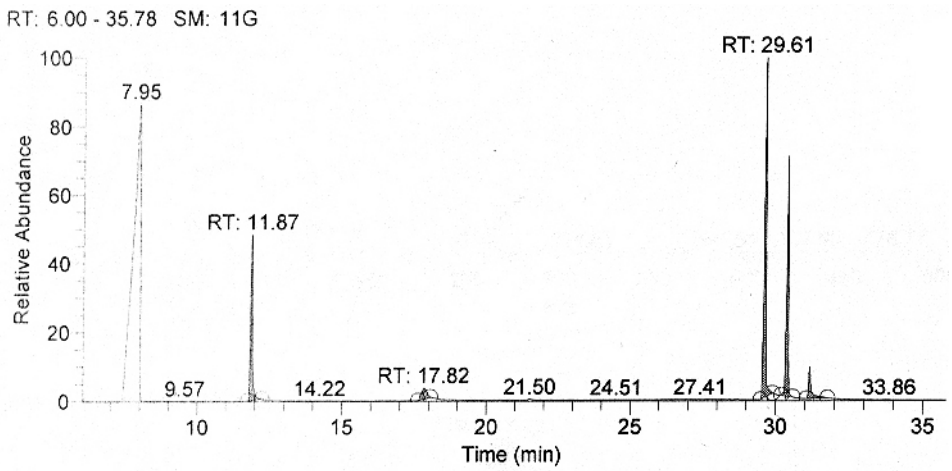
RT	Peak Area	Area %
29.67	1681407	100.00

GCMS Spectra; Water with PEG-600 (Entry 12, Table 1)



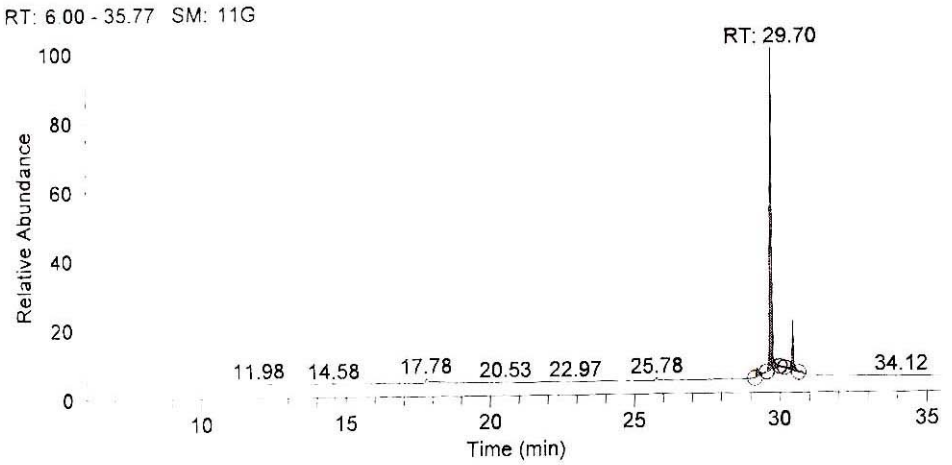
RT	Peak Area	Area %
11.84	406133	10.11
17.73	43158	1.07
29.10	219736	5.47
29.62	2105535	52.39
30.38	1017757	25.32
31.16	226544	5.64

GCMS Spectra; Water with PEG-20000 (Entry 13, Table 1)



RT	Peak Area	Area %
11.87	319786	22.34
17.82	59978	4.19
29.61	571837	39.95
30.38	396956	27.73
31.15	82928	5.79

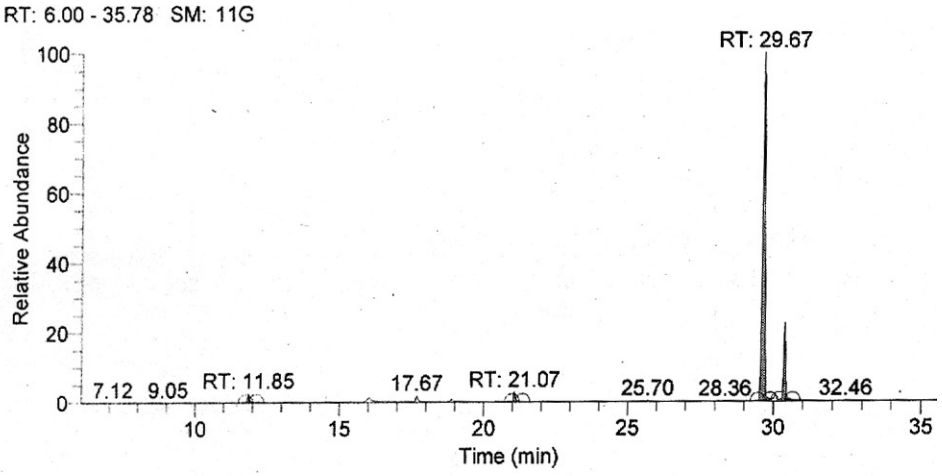
GCMS Spectra; Water with SPAN-80 (Entry 14, Table 1)



RT	Peak Area	Area %
29.22	266253	3.96
29.70	5664681	84.18
30.43	798651	11.87

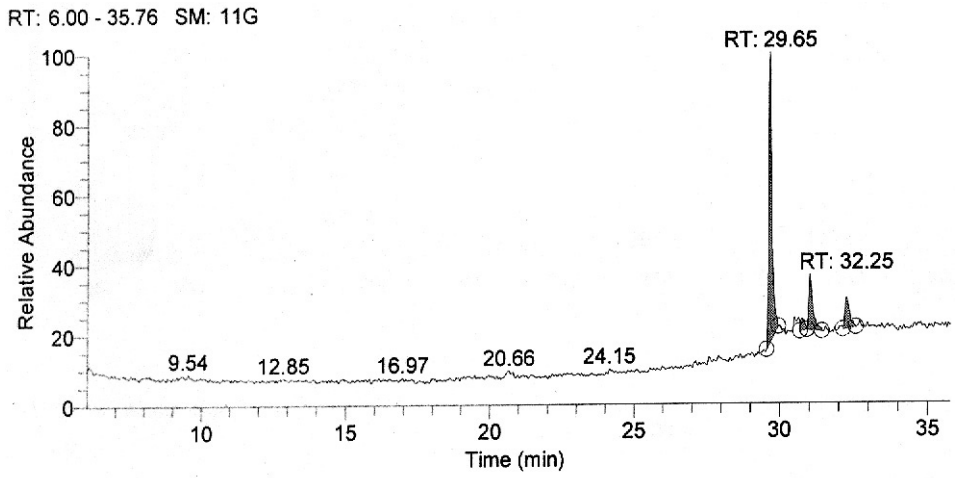
E. GCMS spectra relating optimization of Sodium dioctyl sulfosuccinate amount

GCMS Spectra; 0.5 mol % of SDOSS (Entry 1, Table 4)



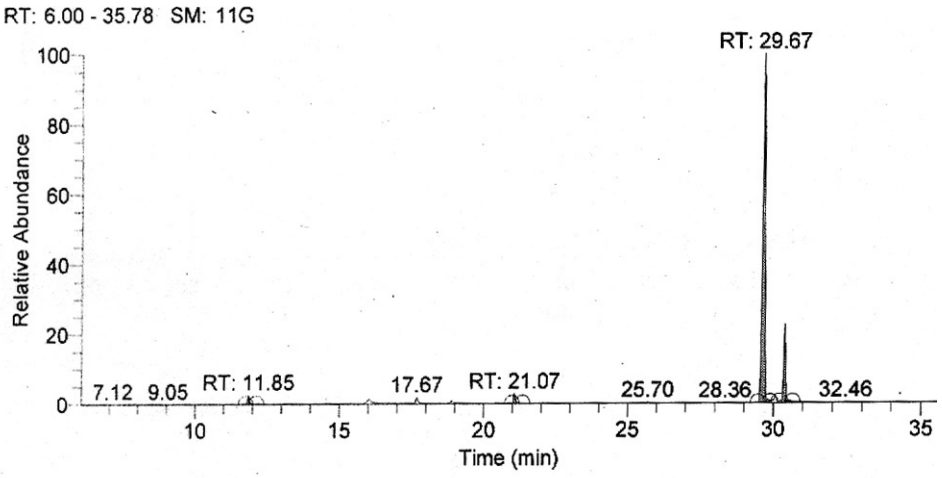
RT	Peak Area	Area %
11.85	128456	1.89
21.07	125819	1.86
29.67	5556740	81.95
30.38	969870	14.30

GCMS Spectra; 1mol % of SDOSS (Entry 2, Table 4)



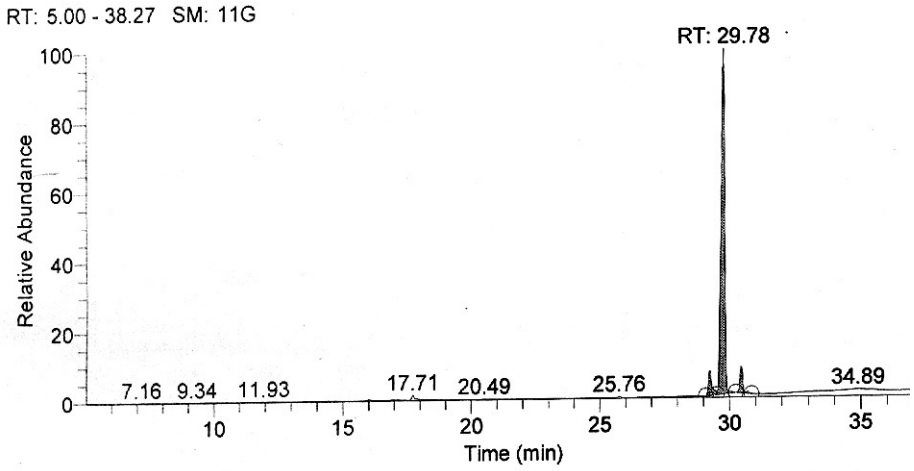
RT	Peak Area	Area %
29.65	28902	67.54
30.74	1658	3.88
31.01	7396	17.28
32.25	4836	11.30

GCMS Spectra; 2 mol % of SDOSS (Entry 3, Table 4)



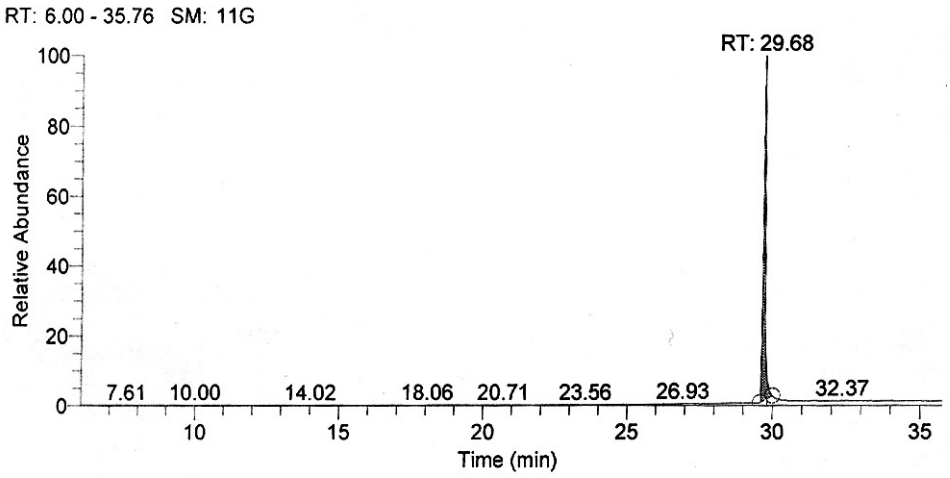
RT	Peak Area	Area %
11.85	128456	1.89
21.07	125819	1.86
29.67	5556740	81.95
30.38	969870	14.30

GCMS Spectra; 4 mol % of SDOSS (Entry 4, Table 4)



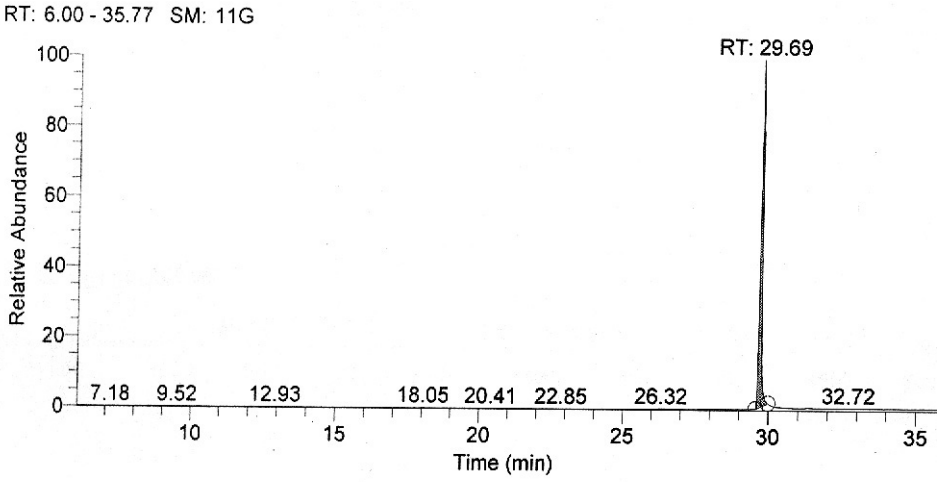
RT	Peak Area	Area %
29.25	44876135	6.15
29.78	644665795	88.37
30.45	39949423	5.48

GCMS Spectra; 5 mol % of SDOSS (Entry 5, Table 4)



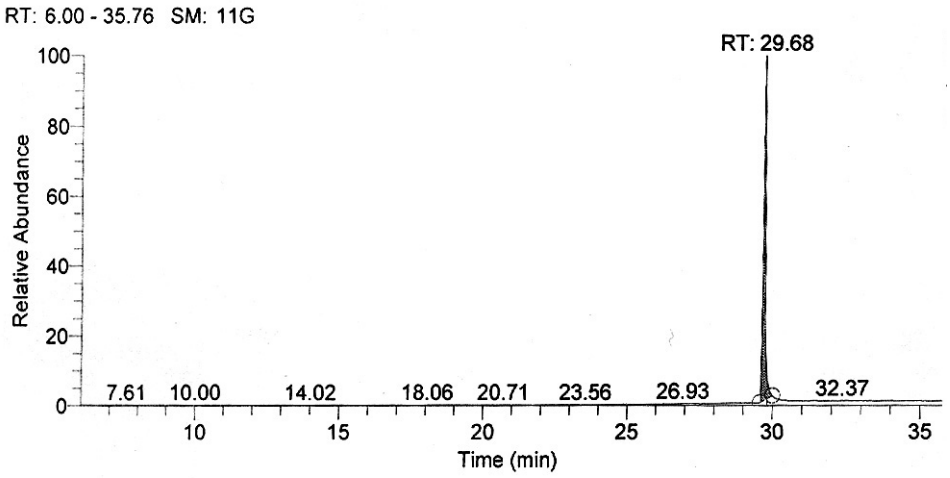
RT	Peak Area	Area %
29.68	895134	100.00

GCMS Spectra; 10 mol % of SDOSS (GCMS Spectra; Entry 6, Table 4)



RT	Peak Area	Area %
29.69	1504968	100.00

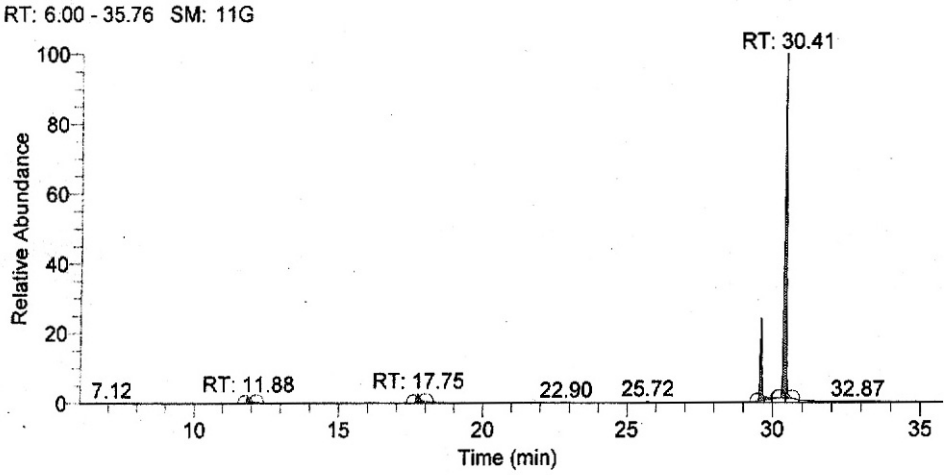
GCMS Spectra; 15 mol % of SDOSS (Entry 7, Table 4)



RT	Peak Area	Area %
29.68	895134	100.00

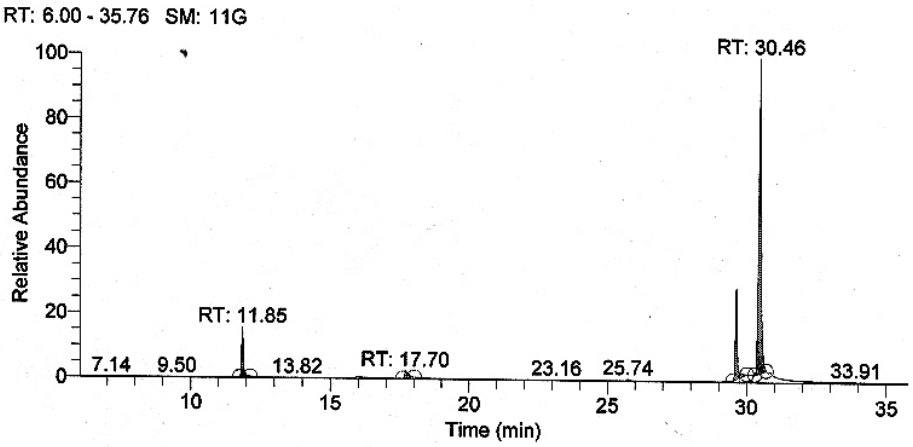
F. GCMS spectra relating solvent study

GCMS Spectra; SDOSS without Water (Entry 1, Table 5)



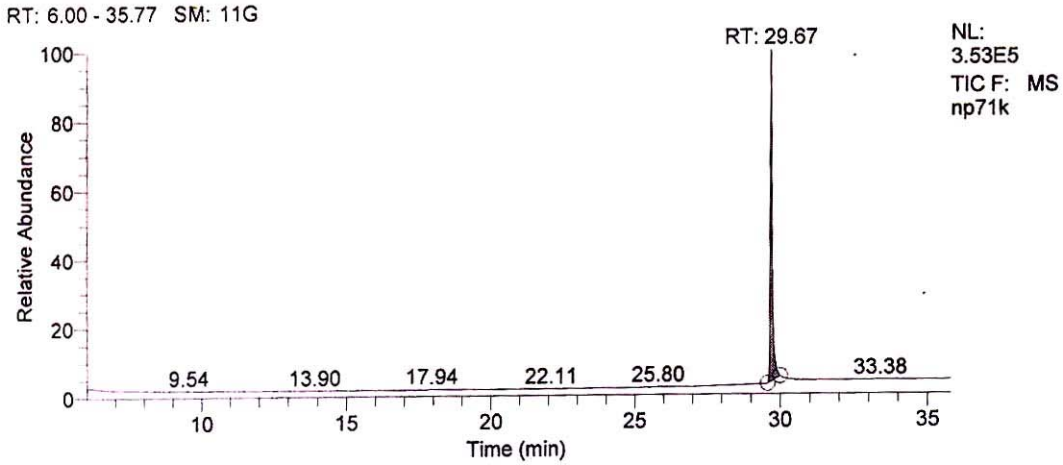
RT	Peak Area	Area %
11.88	57209	1.97
17.75	107530	3.70
29.59	503758	17.32
30.41	2239748	77.01

GCMS Spectra; Water without SDOSS (Entry 2, Table 5)



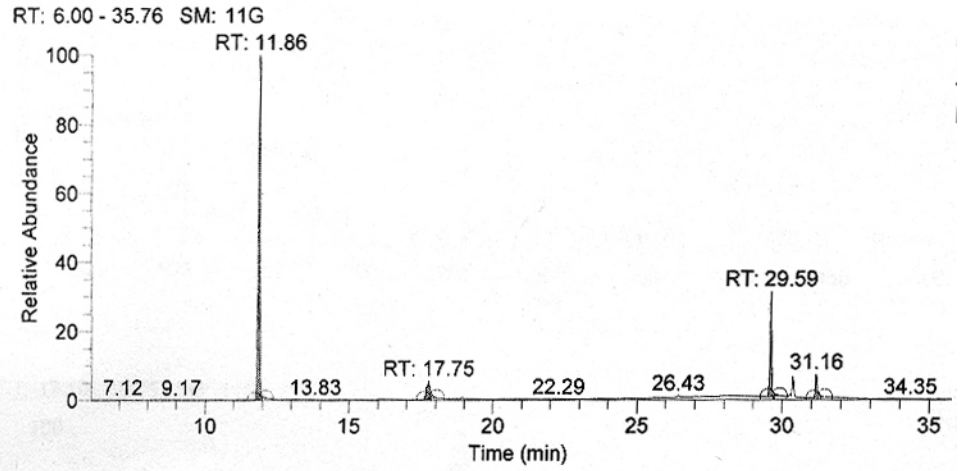
RT	Peak Area	Area %
11.85	487201	8.33
17.70	139809	2.39
29.63	991892	16.95
30.46	4232947	72.34

GCMS Spectra; SDOSS with Water (Entry 3, Table 5)



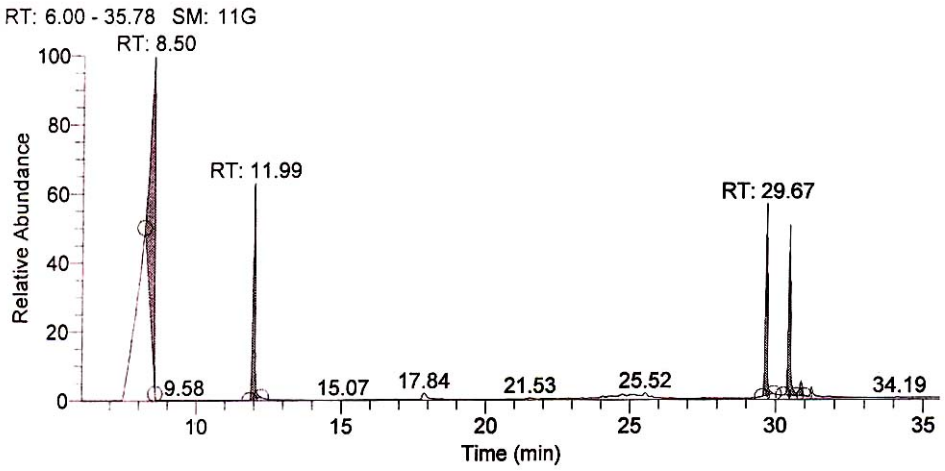
RT	Peak Area	Area %
29.67	1681407	100.00

GCMS Spectra; SDOSS with THF (Entry 4, Table 5)



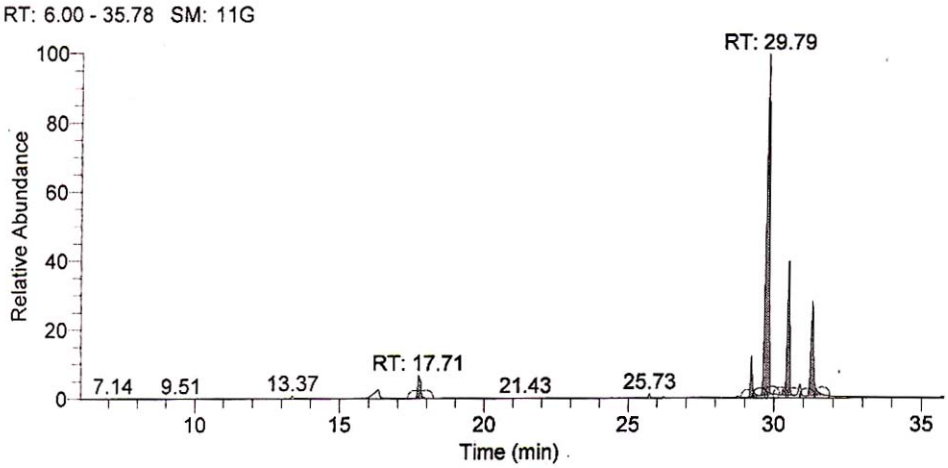
RT	Peak Area	Area %
11.86	814887	66.34
17.75	98408	8.01
29.59	245011	19.95
31.16	70026	5.70

GCMS Spectra; SDOSS with DMF (Entry 5, Table 5)



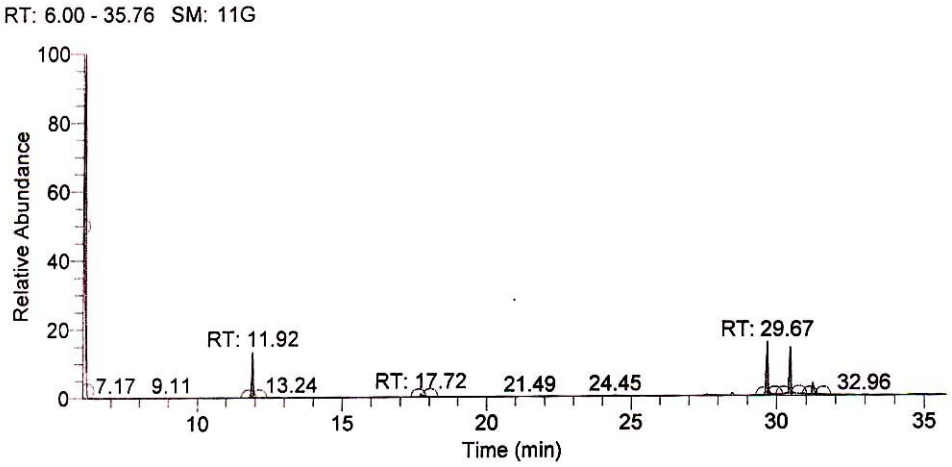
RT	Peak Area	Area %
8.50	8006719	54.67
11.99	2545167	17.38
29.67	1987866	13.57
30.45	1869631	12.77
30.86	236518	1.61

GCMS Spectra; SDOSS with Hexane (Entry 6, Table 5)



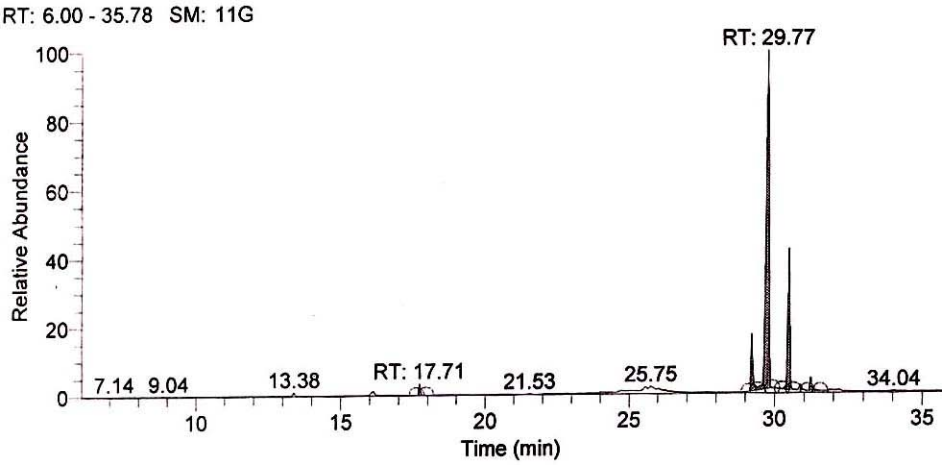
RT	Peak Area	Area %
17.71	1085719	3.81
29.23	1264072	4.43
29.79	16934482	59.39
30.50	5048792	17.71
31.30	4181807	14.67

GCMS Spectra; SDOSS with Dioxane (Entry 7, Table 5)



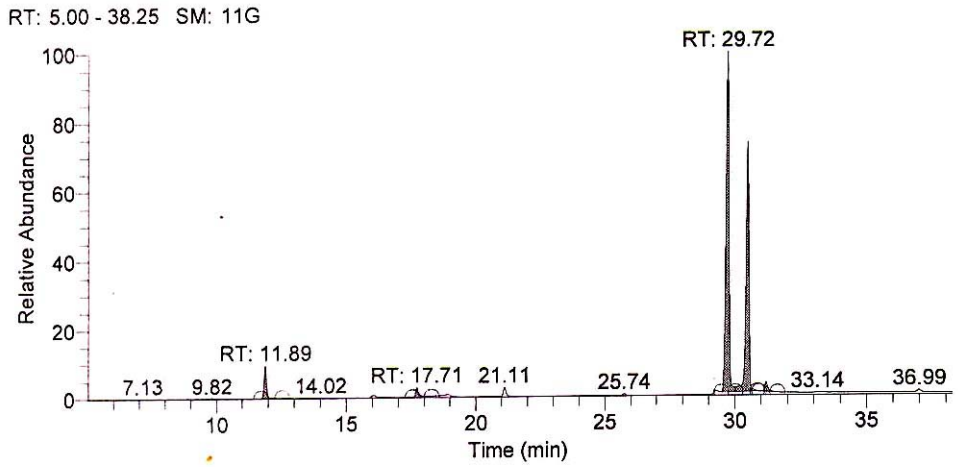
RT	Peak Area	Area %
6.09	12046092	61.67
11.92	2047730	10.48
17.72	236492	1.21
29.67	2360259	12.08
30.45	2196561	11.25
31.22	645155	3.30

GCMS Spectra; SDOSS with DCM (Entry 8, Table 5)



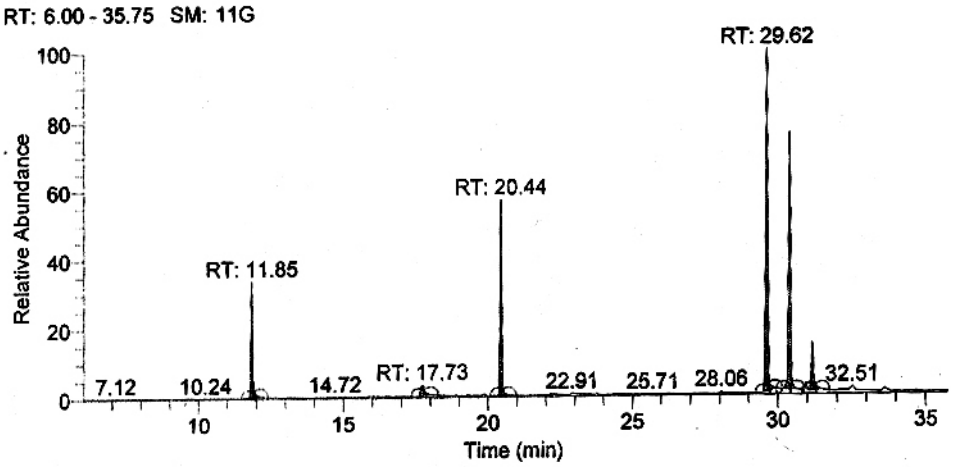
RT	Peak Area	Area %
17.71	254281	1.42
29.21	1434947	8.01
29.77	12108602	67.58
30.49	3725016	20.79
31.22	394283	2.20

GCMS Spectra; SDOSS with EtOH (Entry 9, Table 5)



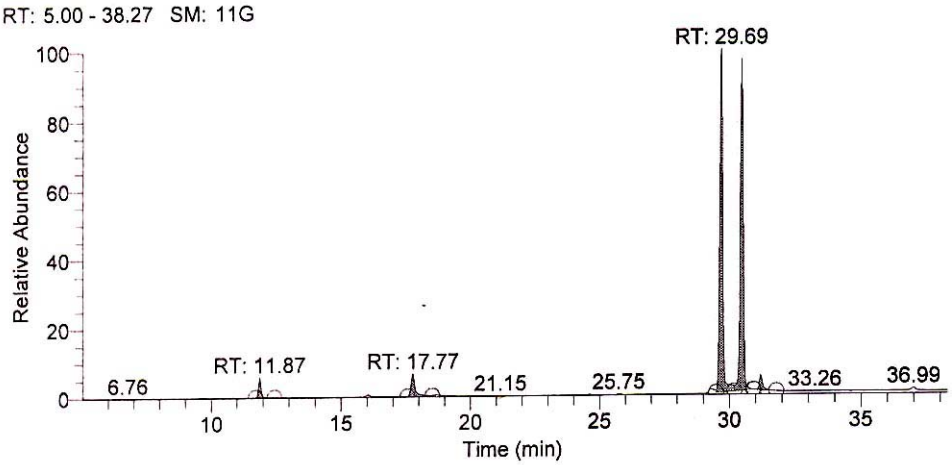
RT	Peak Area	Area %
11.89	684349	4.60
17.71	308252	2.07
29.72	7798570	52.40
30.48	5851114	39.31
31.20	241750	1.62

GCMS Spectra; SDOSS with MeOH (Entry 10, Table 5)



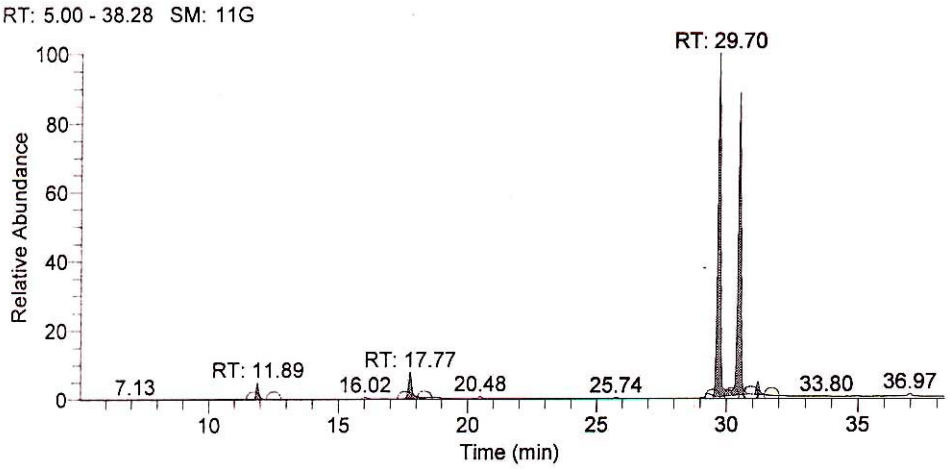
RT	Peak Area	Area %
11.85	714748	11.21
17.73	162481	2.55
20.44	1128773	17.70
29.62	2259313	35.42
30.39	1740956	27.29
31.16	372328	5.84

GCMS Spectra; SDOSS with *t*-BuOH (Entry 11, Table 5)



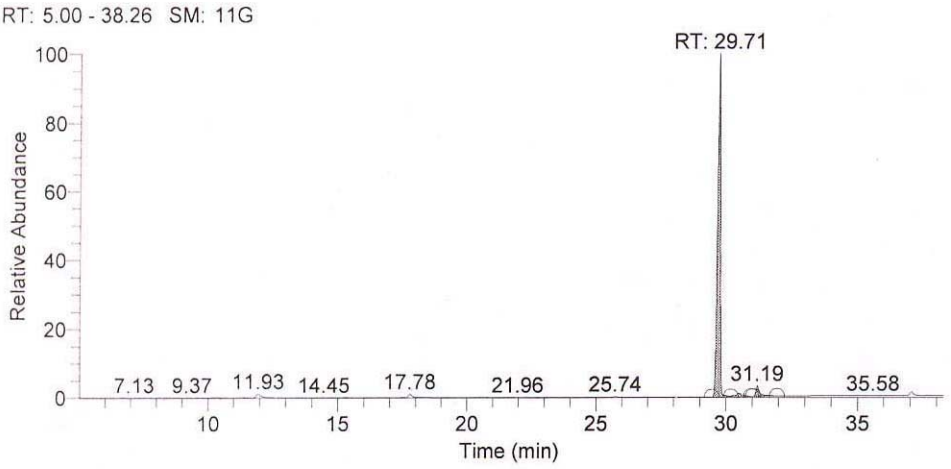
RT	Peak Area	Area %
11.87	297830	2.51
17.77	554829	4.68
29.69	5409981	45.66
30.47	5316086	44.86
31.19	270394	2.28

GCMS Spectra; SDOSS with *i*-Propanol (Entry 12, Table 5)



RT	Peak Area	Area %
11.89	316713	2.34
17.77	697440	5.15
29.70	6517762	48.17
30.48	5735894	42.39
31.20	263108	1.94

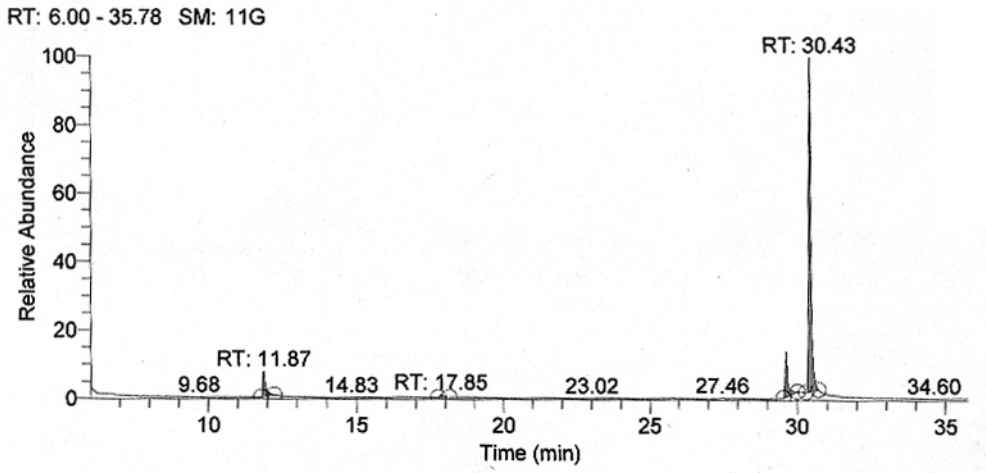
GCMS Spectra; SDOSS with CF_3CH_2OH (Entry 13, Table 5)



RT	Peak Area	Area %
29.71	7093187	94.14
30.49	156382	2.08
31.19	285406	3.79

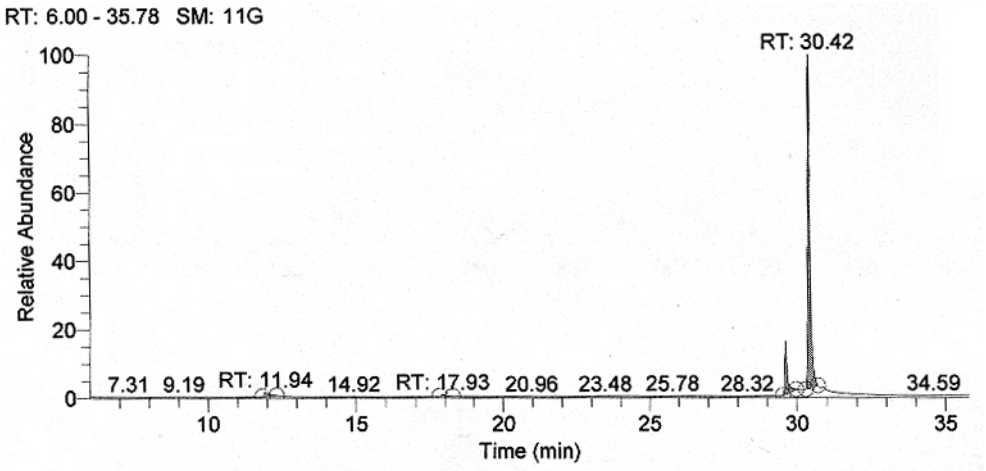
G. GCMS spectra relating detail study in selected solvent

GCMS Spectra; Water as reaction medium without SDOSS; Sample withdrawn after 5 min. (Entry 1, Table 6)



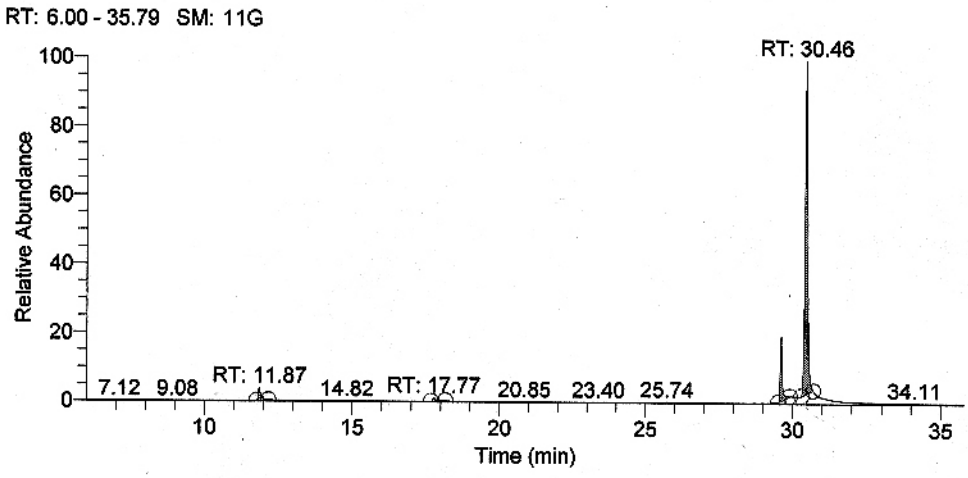
RT	Peak Area	Area %
11.87	180369	8.15
17.85	25393	1.15
29.62	276679	12.50
30.43	1730965	78.20

GCMS Spectra; Water as reaction medium without SDOSS; Sample withdrawn after 10 min. (Entry 2, Table 6)



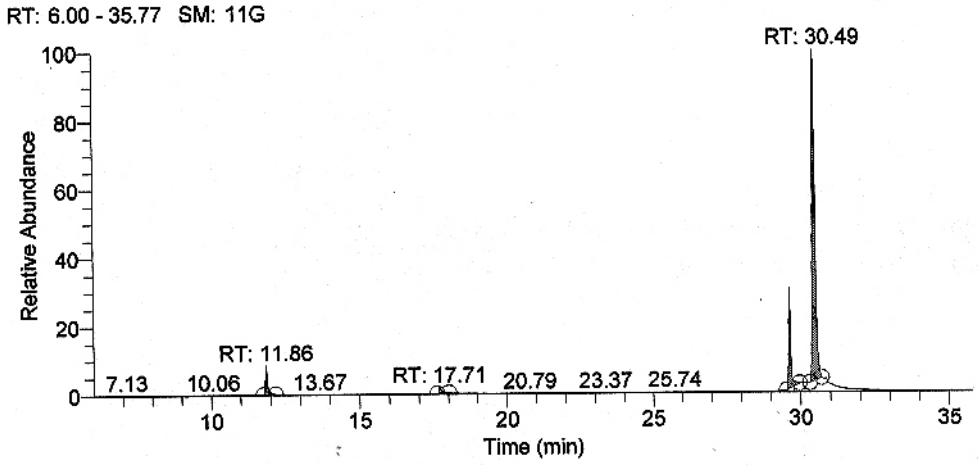
RT	Peak Area	Area %
11.94	40792	2.29
17.93	16625	0.93
29.62	255167	14.33
30.42	1467972	82.44

GCMS Spectra; Water as reaction medium without SDOSS; Sample withdrawn after 15 min. (Entry 3, Table 6)



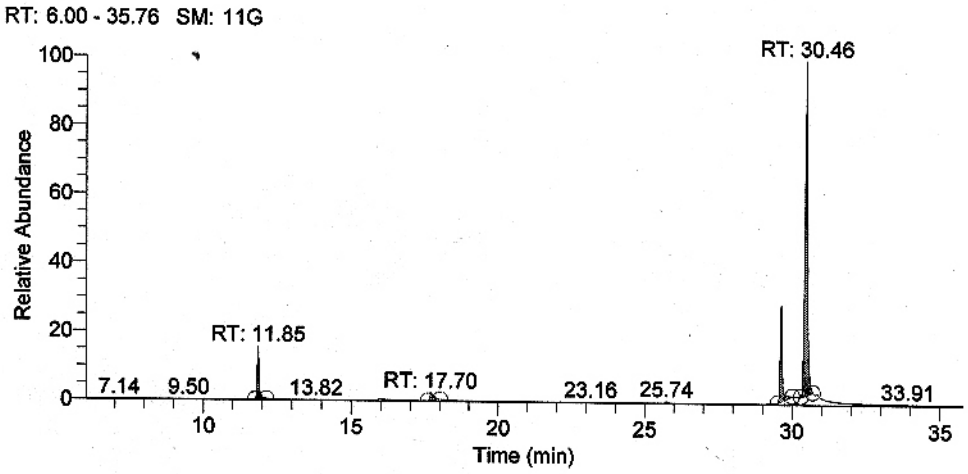
RT	Peak Area	Area %
11.87	154732	3.21
17.77	56065	1.16
29.62	619541	12.86
30.46	3987421	82.77

GCMS Spectra; Water as reaction medium without SDOSS; Sample withdrawn after 30 min. (Entry 4, Table 6)



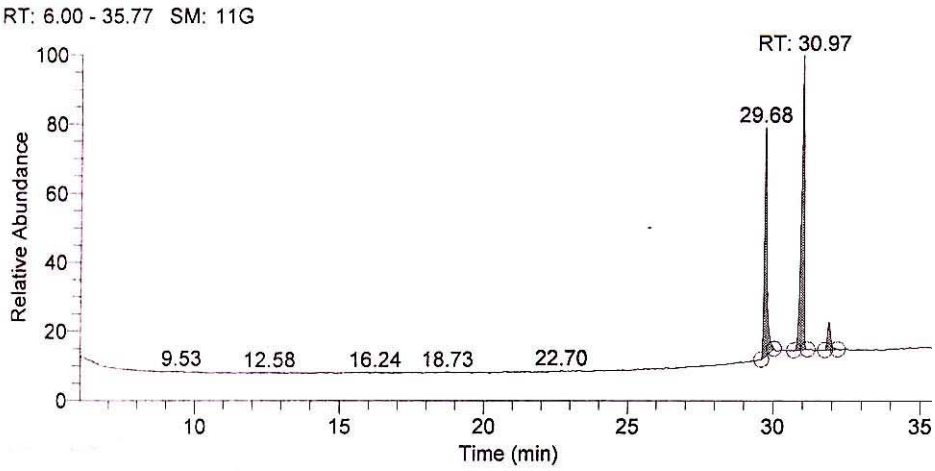
RT	Peak Area	Area %
11.86	341940	4.86
17.71	117872	1.68
29.63	1169980	16.63
30.49	5406104	76.84

GCMS Spectra; Water as reaction medium without SDOSS; Sample withdrawn after 60 min. (Entry 5, Table 6)



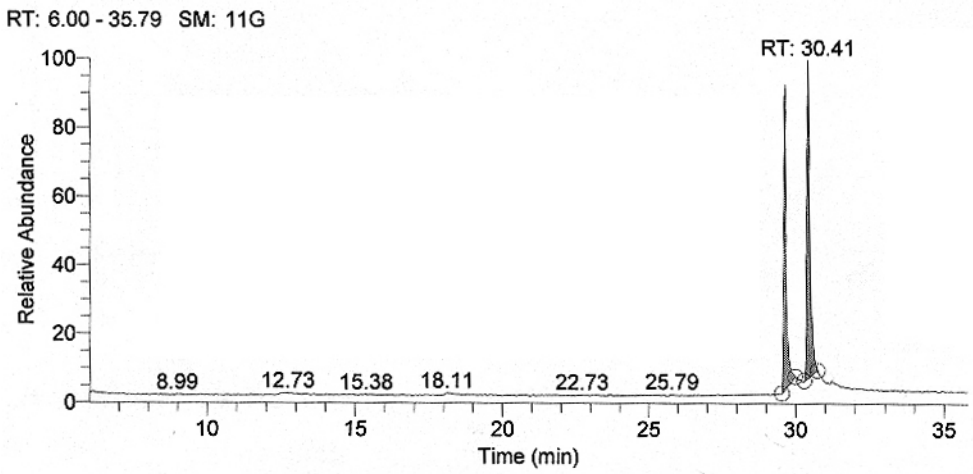
RT	Peak Area	Area %
11.85	487201	8.33
17.70	139809	2.39
29.63	991892	16.95
30.46	4232947	72.34

GCMS Spectra; Water as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 6, Table 6)



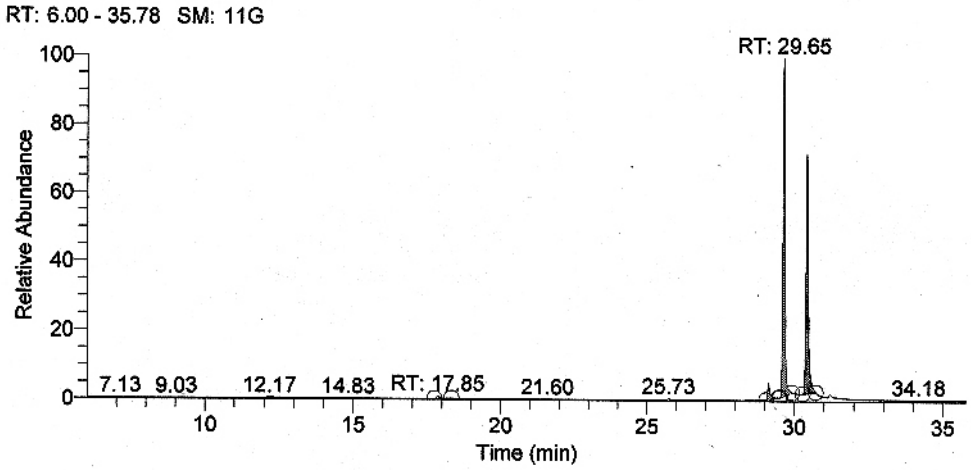
RT	Peak Area	Area %
29.68	537670	38.94
30.97	759623	55.01
31.86	83632	6.06

GCMS Spectra; Water as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 7, Table 6)



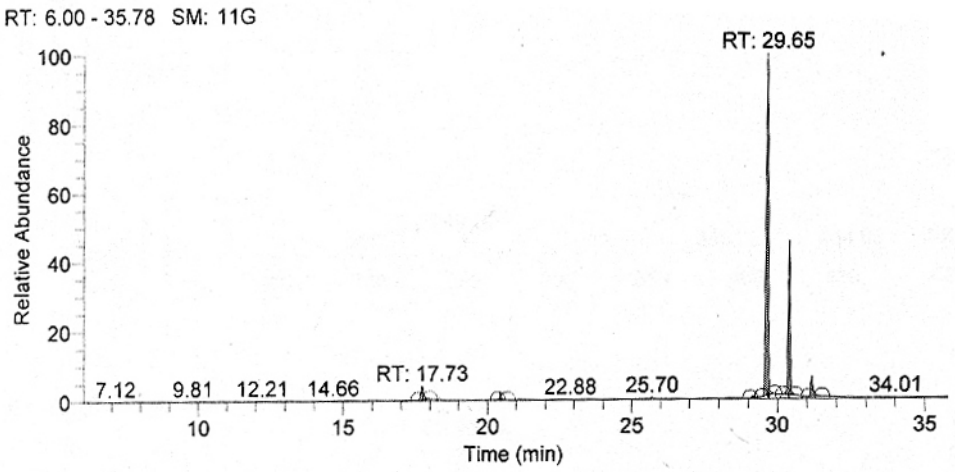
RT	Peak Area	Area %
29.62	245732	44.54
30.41	305951	55.46

GCMS Spectra; Water as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 8, Table 6)



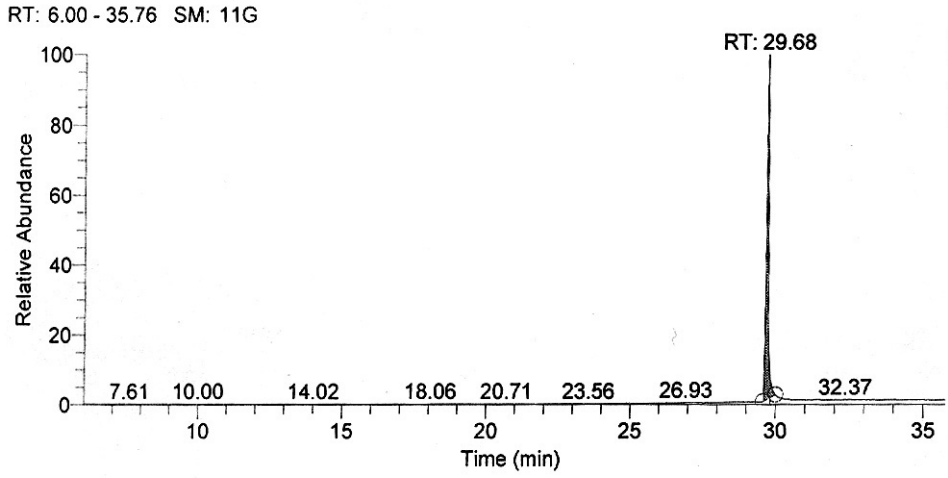
RT	Peak Area	Area %
17.85	30298	0.86
29.14	117208	3.32
29.65	1865638	52.89
30.43	1514377	42.93

GCMS Spectra; Water as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 9, Table 6)



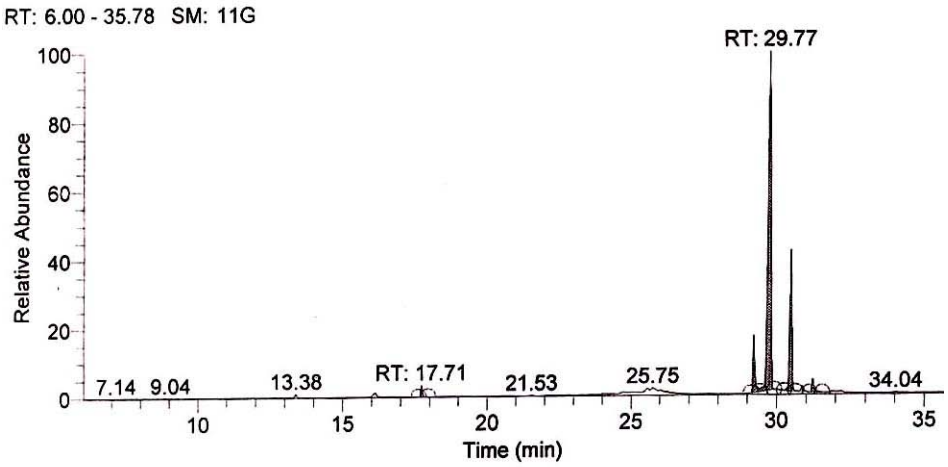
RT	Peak Area	Area %
17.73	262070	3.77
20.44	88648	1.27
29.14	32876	0.47
29.65	4547402	65.33
30.39	1754488	25.21
31.16	274846	3.95

GCMS Spectra; Water as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 10, Table 6)



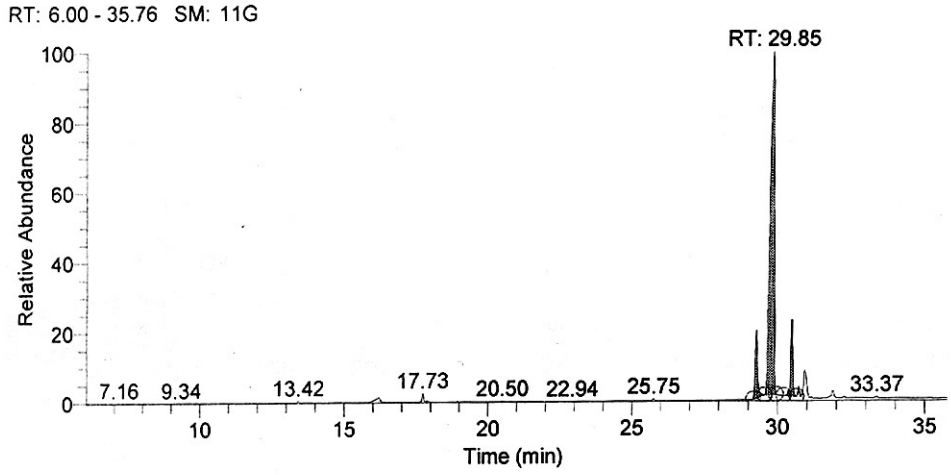
RT	Peak Area	Area %
29.68	895134	100.00

GCMS Spectra; Water as reaction medium with SDOSS in presence of oxygen bubbling; Sample withdrawn after 5 min. (Entry 11, Table 6)



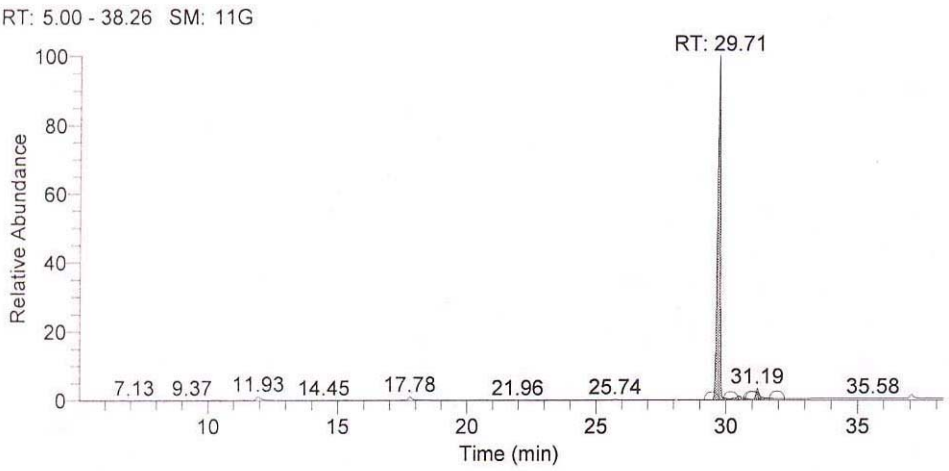
RT	Peak Area	Area %
17.71	254281	1.42
29.21	1434947	8.01
29.77	12108602	67.58
30.49	3725016	20.79
31.22	394283	2.20

GCMS Spectra; Water as reaction medium with SDOSS in presence of oxygen bubbling; Sample withdrawn after 10 min. (Entry 12, Table 6)



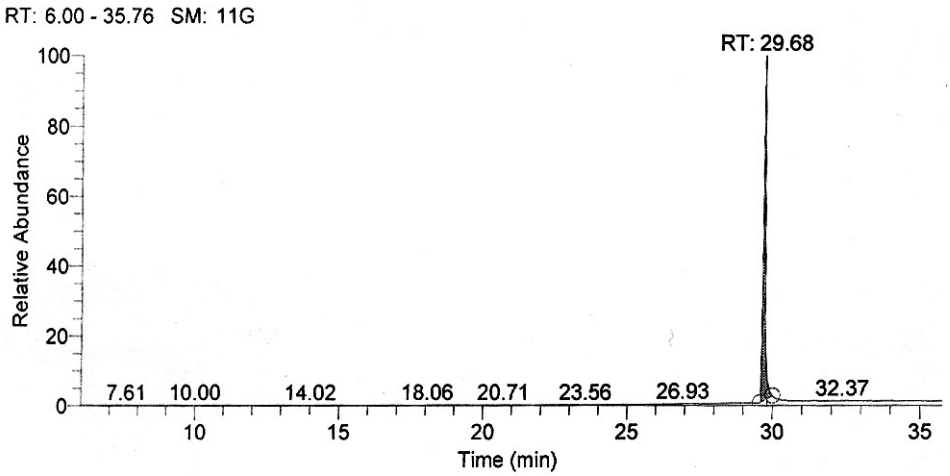
RT	Peak Area	Area %
29.28	109666348	9.17
29.85	977609663	81.79
30.49	108031737	9.04

GCMS Spectra; Water as reaction medium with SDOSS in presence of oxygen bubbling; Sample withdrawn after 15 min. (Entry 13, Table 6)



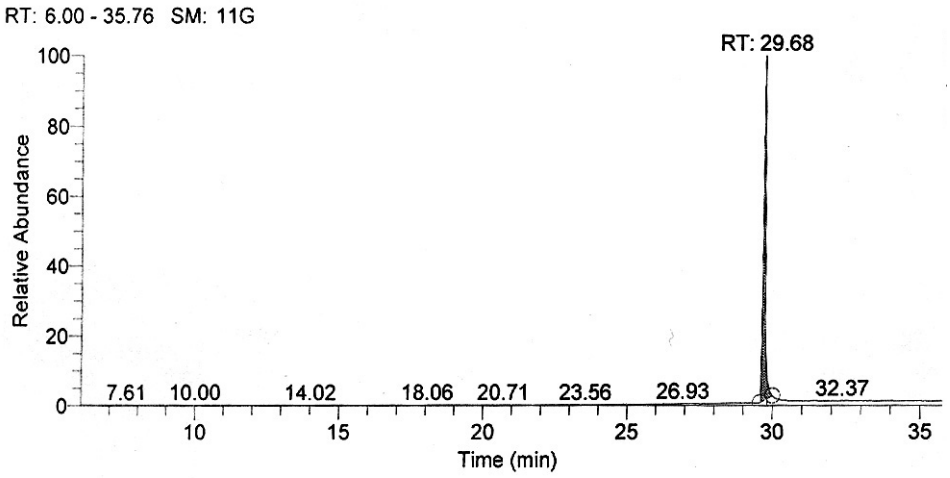
RT	Peak Area	Area %
29.71	7093187	94.14
30.49	156382	2.08
31.19	285406	3.79

GCMS Spectra; Water as reaction medium with SDOSS in presence of oxygen bubbling; Sample withdrawn after 30 min. (Entry 14, Table 6)



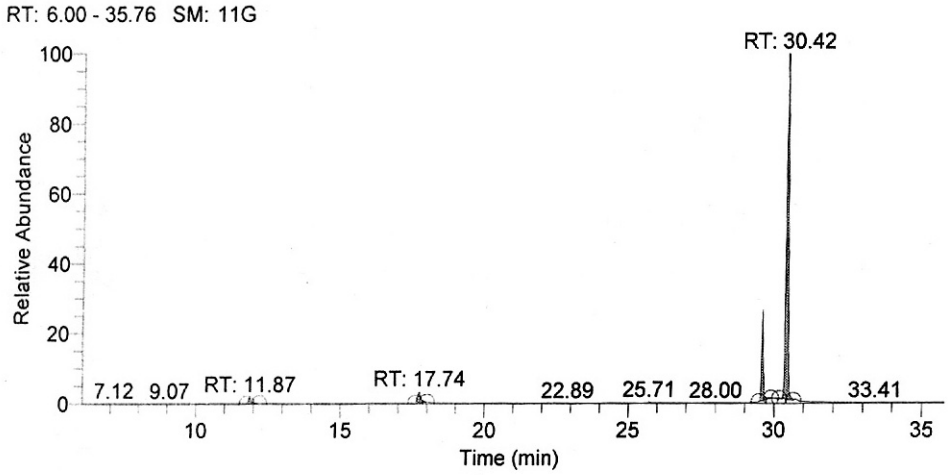
RT	Peak Area	Area %
29.68	895134	100.00

GCMS Spectra; Water as reaction medium with SDOSS in presence of oxygen bubbling; Sample withdrawn after 60 min. (Entry 15, Table 6)



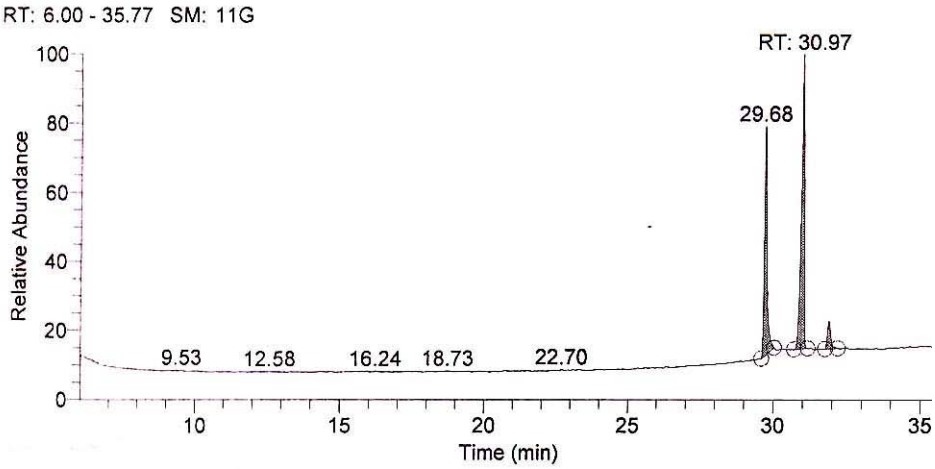
RT	Peak Area	Area %
29.68	895134	100.00

GCMS Spectra; Water as reaction medium without SDOSS in presence of oxygen bubbling; Sample withdrawn after 5 min. (Entry 16, Table 6)



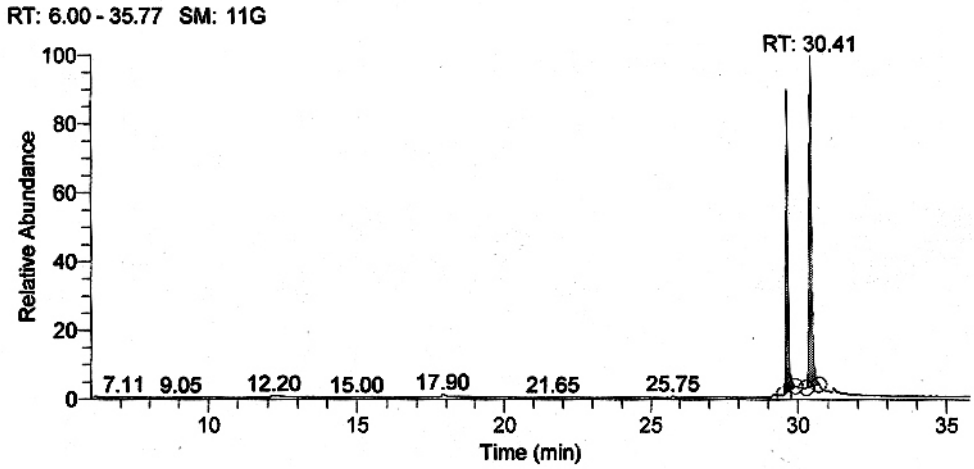
RT	Peak Area	Area %
11.87	67952	1.84
17.74	148920	4.02
29.60	600261	16.22
30.42	2883992	77.92

GCMS Spectra; Water as reaction medium without SDOSS in presence of oxygen bubbling; Sample withdrawn after 10 min. (Entry 17, Table 6)



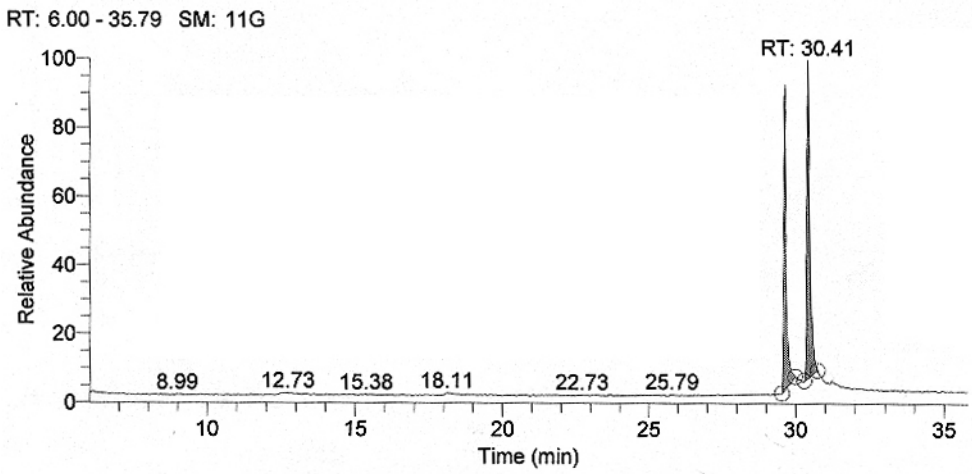
RT	Peak Area	Area %
29.68	537670	38.94
30.97	759623	55.01
31.86	83632	6.06

GCMS Spectra; Water as reaction medium without SDOSS in presence of oxygen bubbling; Sample withdrawn after 15 min. (Entry 18, Table 6)



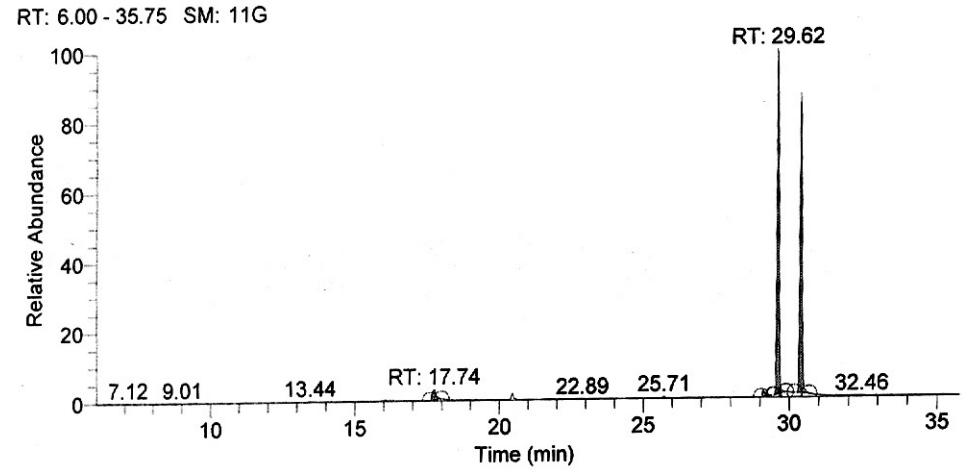
RT	Peak Area	Area %
29.63	789313	44.34
30.41	990937	55.66

GCMS Spectra; Water as reaction medium without SDOSS in presence of oxygen bubbling; Sample withdrawn after 30 min. (Entry 19, Table 6)



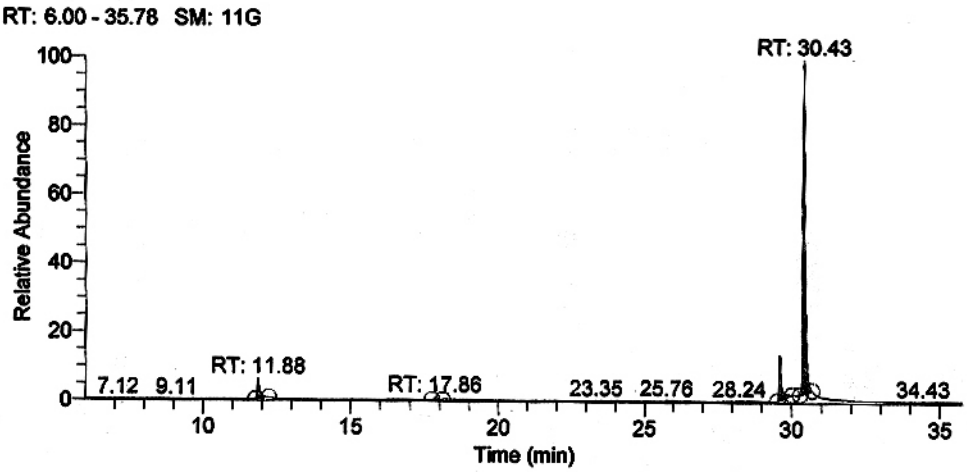
RT	Peak Area	Area %
29.62	245732	44.54
30.41	305951	55.46

GCMS Spectra; Water as reaction medium without SDOSS in presence of oxygen bubbling; Sample withdrawn after 60 min. (Entry 20, Table 6)



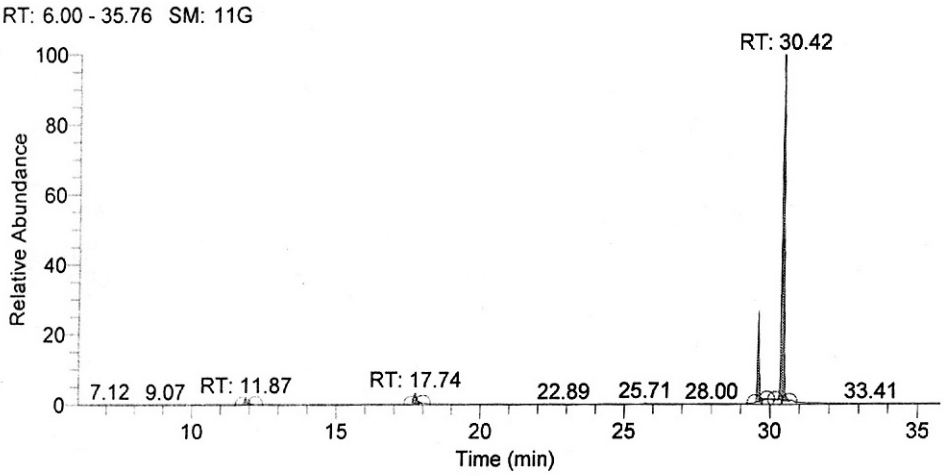
RT	Peak Area	Area %
17.74	125326	3.19
29.12	50092	1.27
29.62	1963786	49.93
30.40	1793911	45.61

GCMS Spectra; Degassed water as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 21, Table 6)



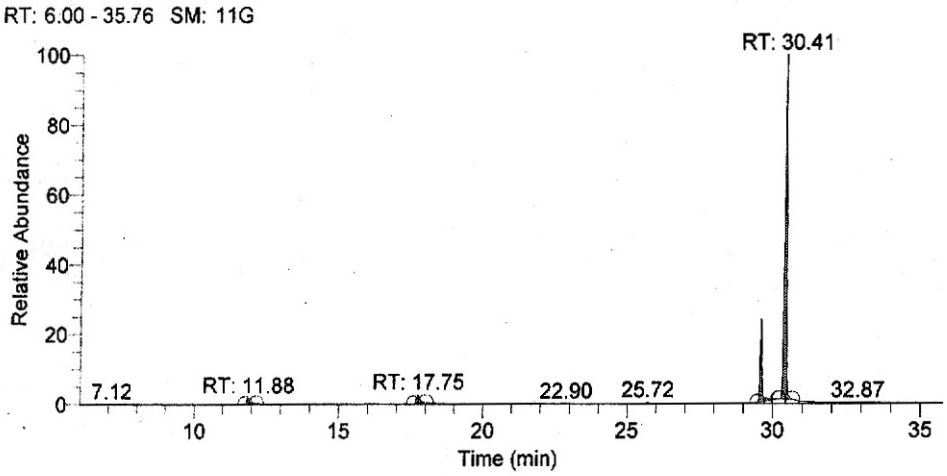
RT	Peak Area	Area %
11.88	143831	6.50
17.86	28139	1.27
29.62	275553	12.45
30.43	1766077	79.78

GCMS Spectra; Degassed water as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 22, Table 6)



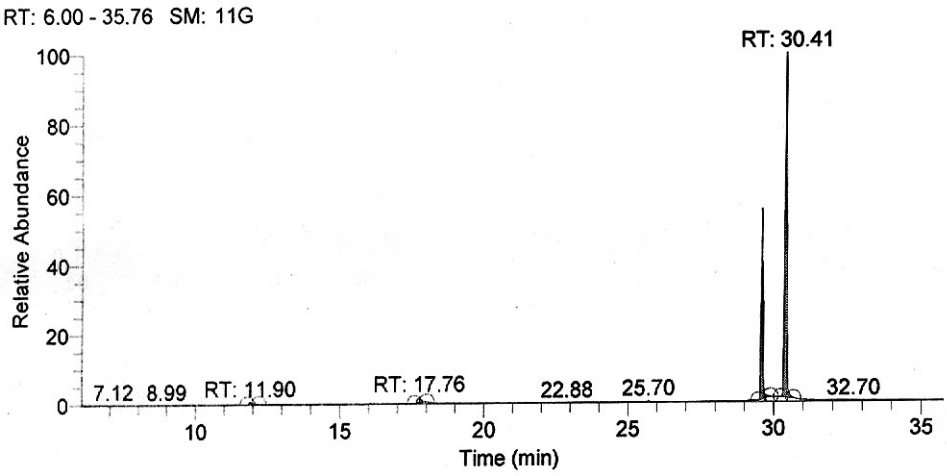
RT	Peak Area	Area %
11.87	67952	1.84
17.74	148920	4.02
29.60	600261	16.22
30.42	2883992	77.92

GCMS Spectra; Degassed water as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 23, Table 6)



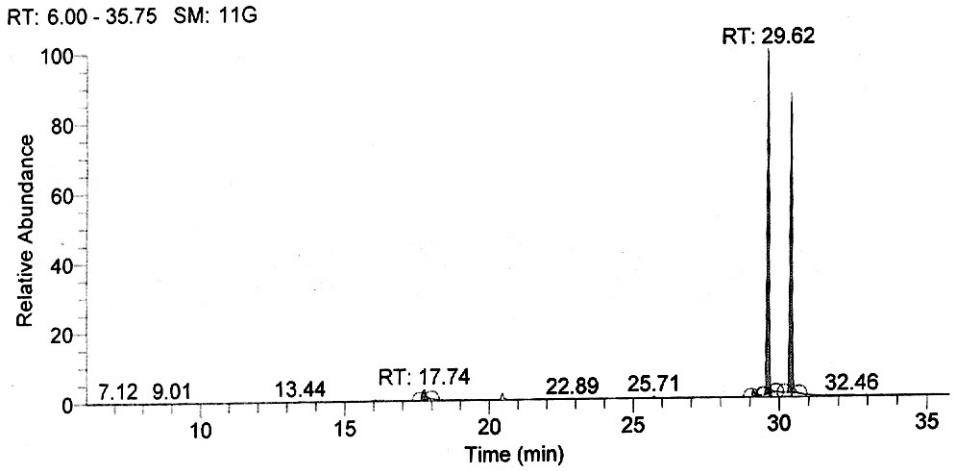
RT	Peak Area	Area %
11.88	57209	1.97
17.75	107530	3.70
29.59	503758	17.32
30.41	2239748	77.01

GCMS Spectra; Degassed water as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 24, Table 6)



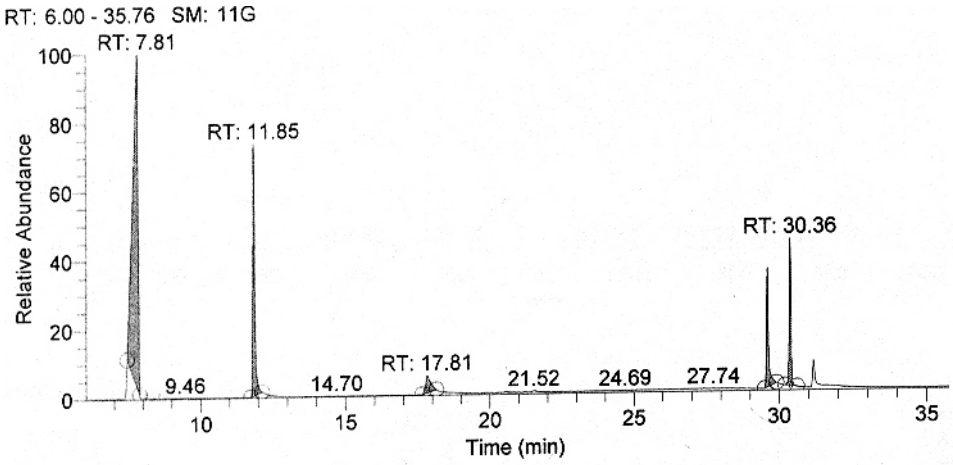
RT	Peak Area	Area %
11.90	25050	0.97
17.76	69704	2.69
29.60	814053	31.46
30.41	1679146	64.88

GCMS Spectra; Degassed water as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 25, Table 6)



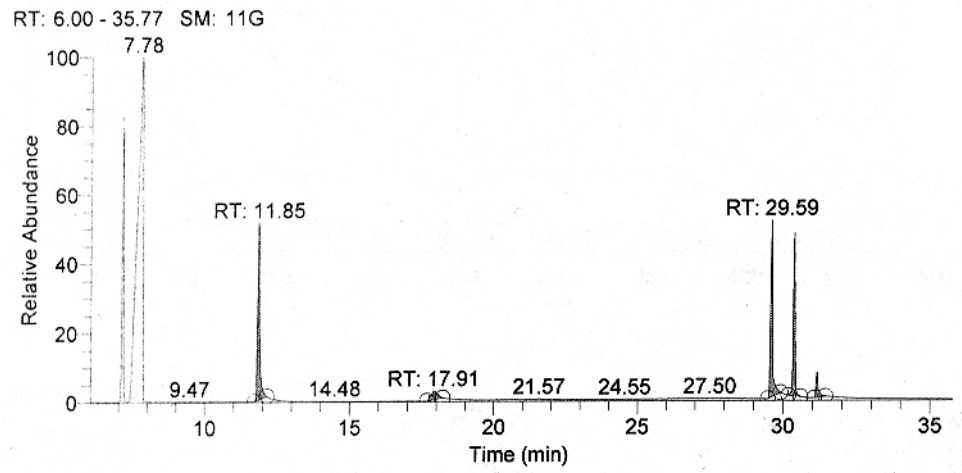
RT	Peak Area	Area %
17.74	125326	3.19
29.12	50092	1.27
29.62	1963786	49.93
[illegible]	1793911	45.61

GCMS Spectra; DMF as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 26, Table 6)



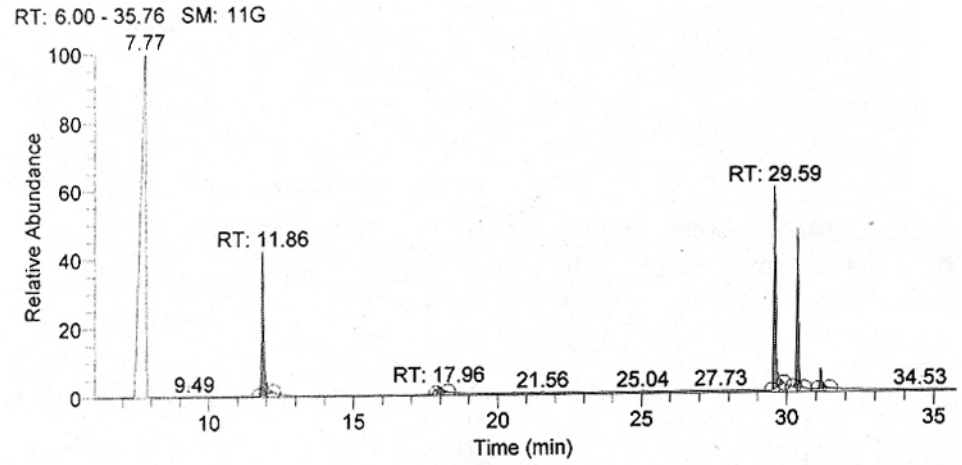
RT	Peak Area	Area %
7.81	1035739	62.16
11.85	290570	17.44
17.81	55062	3.30
29.59	141074	8.47
30.36	143716	8.63

GCMS Spectra; DMF as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 27, Table 6)



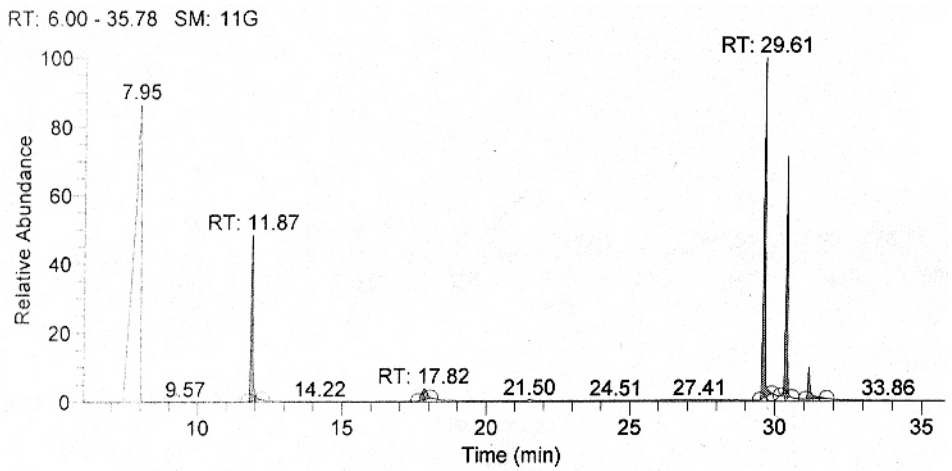
RT	Peak Area	Area %
11.85	208377	34.57
17.91	30424	5.05
29.59	183999	30.52
30.36	149212	24.75
31.15	30783	5.11

GCMS Spectra; DMF as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 28, Table 6)



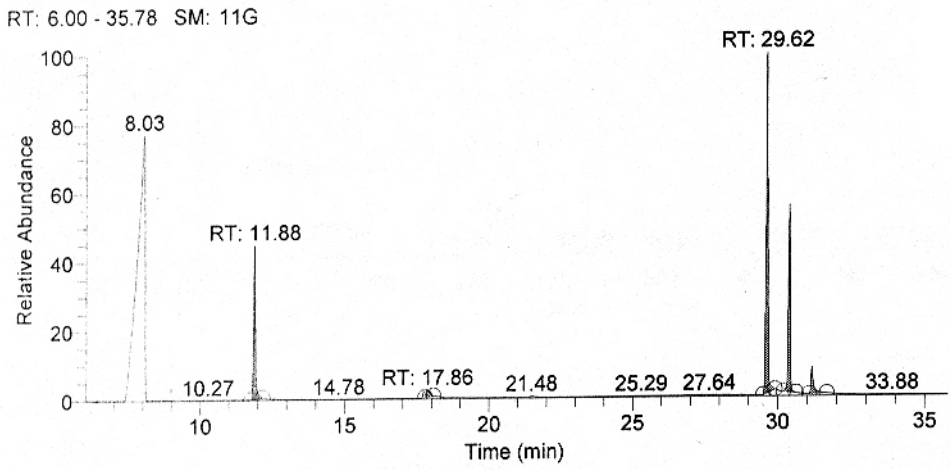
RT	Peak Area	Area %
11.86	164663	30.83
17.96	17000	3.18
29.59	187272	35.07
30.36	140713	26.35
31.15	24397	4.57

GCMS Spectra; DMF as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 29, Table 6)



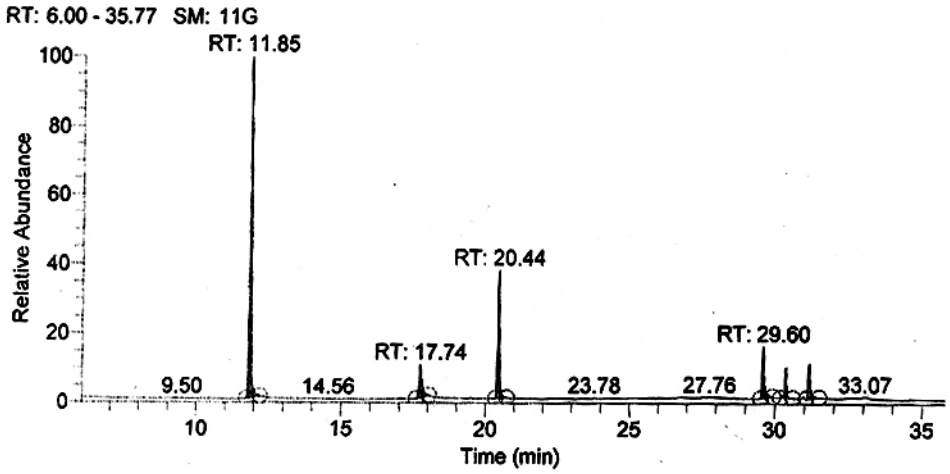
RT	Peak Area	Area %
11.87	319786	22.34
17.82	59978	4.19
29.61	571837	39.95
30.38	396956	27.73
31.15	82928	5.79

GCMS Spectra; DMF as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 30, Table 6)



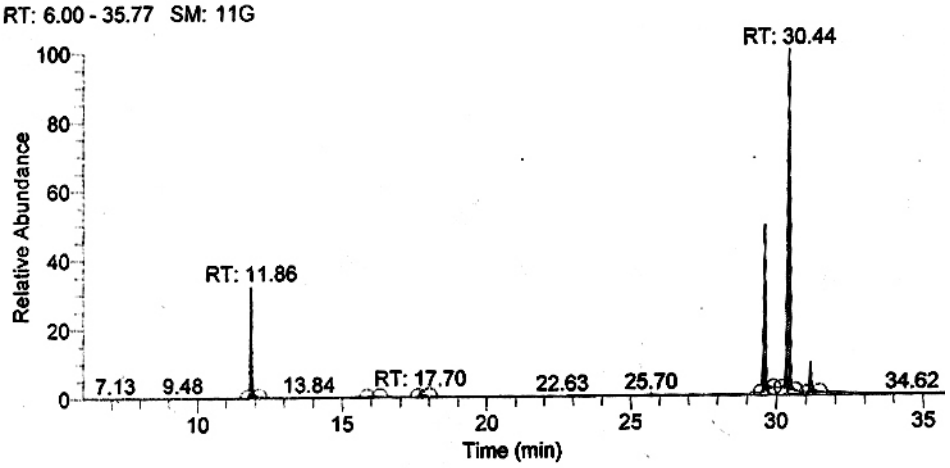
RT	Peak Area	Area %
11.88	381754	21.63
17.86	35744	2.03
29.62	825660	46.78
30.39	430063	24.37
31.16	91801	5.20

GCMS Spectra; Hexane as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 31, Table 6)



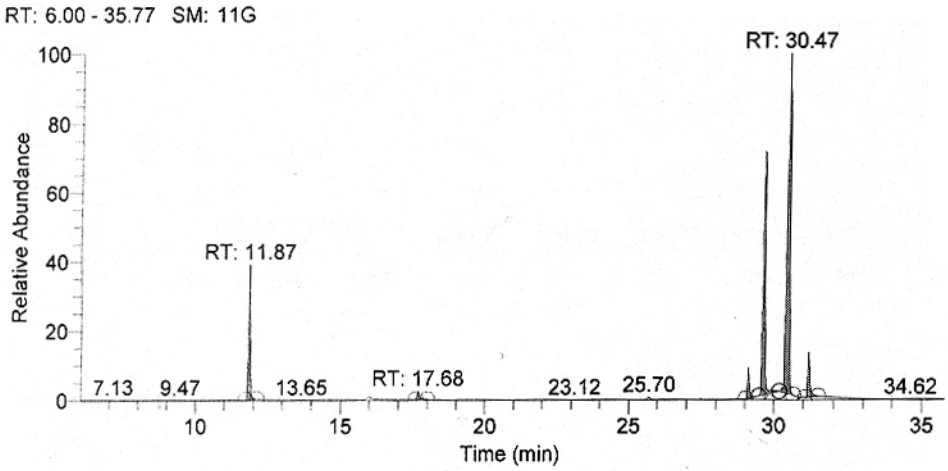
RT	Peak Area	Area %
11.85	1182019	49.48
17.74	220191	9.22
20.44	478848	20.04
29.60	230151	9.63
30.37	120115	5.03
31.16	157683	6.60

GCMS Spectra; Hexane as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 32, Table 6)



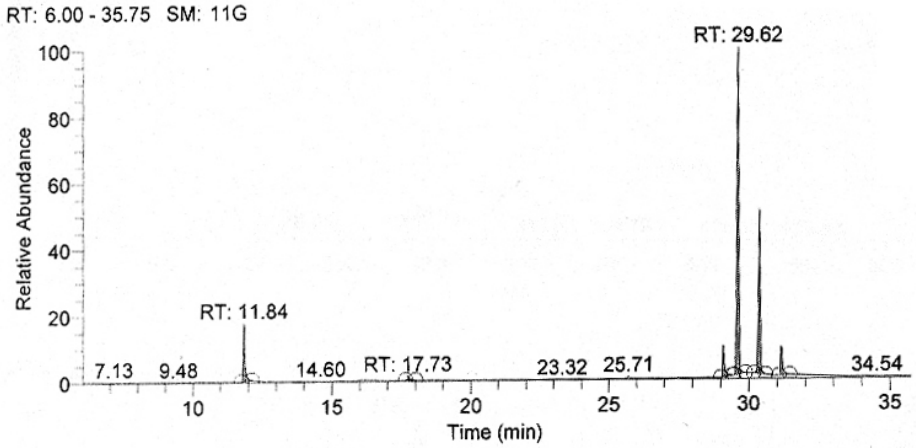
RT	Peak Area	Area %
11.86	1171483	13.62
15.95	35266	0.41
17.70	93330	1.09
29.61	1875746	21.81
30.44	5015526	58.32
31.16	408693	4.75

GCMS Spectra; Hexane as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 33, Table 6)



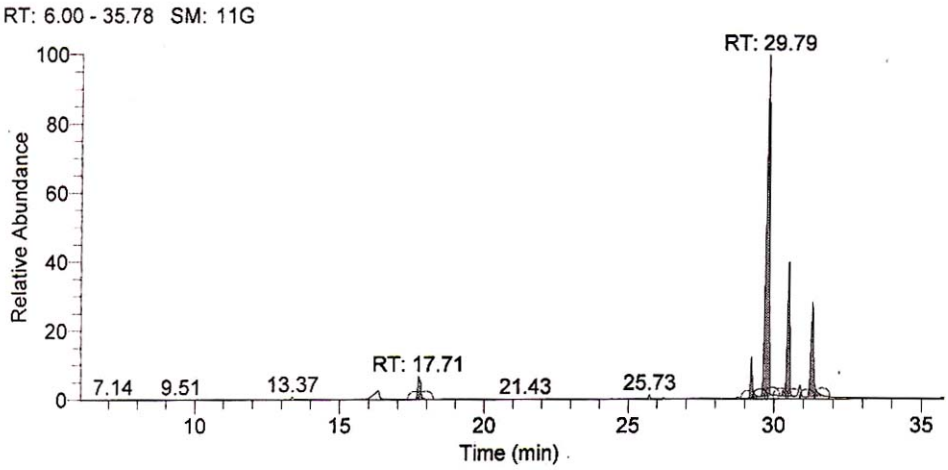
RT	Peak Area	Area %
11.87	1908342	13.21
17.68	159075	1.10
29.12	452981	3.14
29.64	4134174	28.61
30.47	7050404	48.80
31.17	742912	5.14

GCMS Spectra; Hexane as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 34, Table 6)



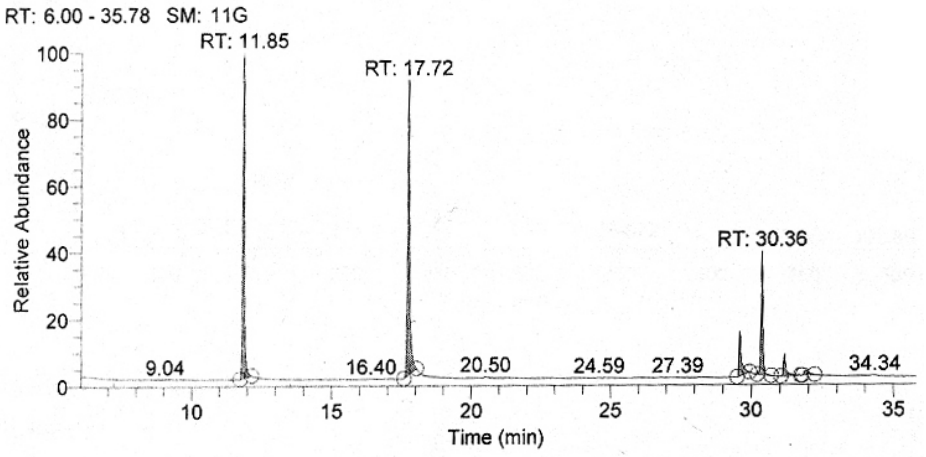
RT	Peak Area	Area %
11.84	406133	10.11
17.73	43158	1.07
29.10	219736	5.47
29.62	2105535	52.39
30.38	1017757	25.32
31.16	226544	5.64

GCMS Spectra; Hexane as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 35, Table 6)



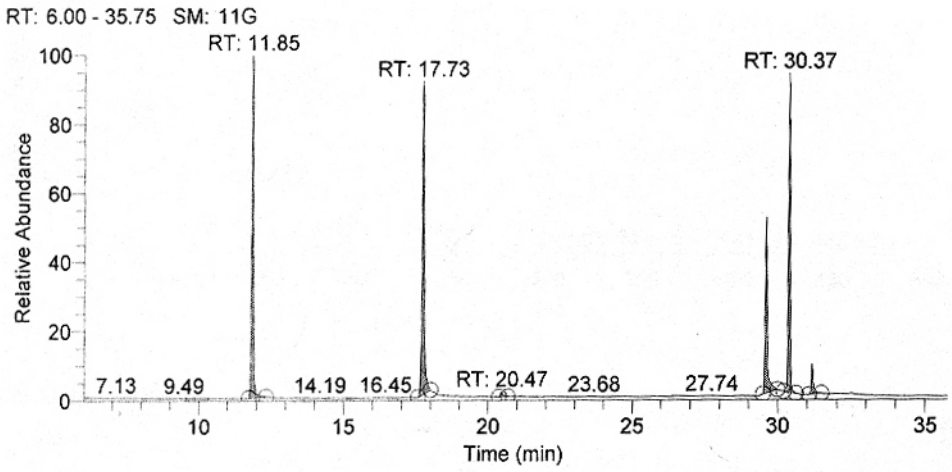
RT	Peak Area	Area %
17.71	1085719	3.81
29.23	1264072	4.43
29.79	16934482	59.39
30.50	5048792	17.71
31.30	4181807	14.67

GCMS Spectra; DCM as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 36, Table 6)



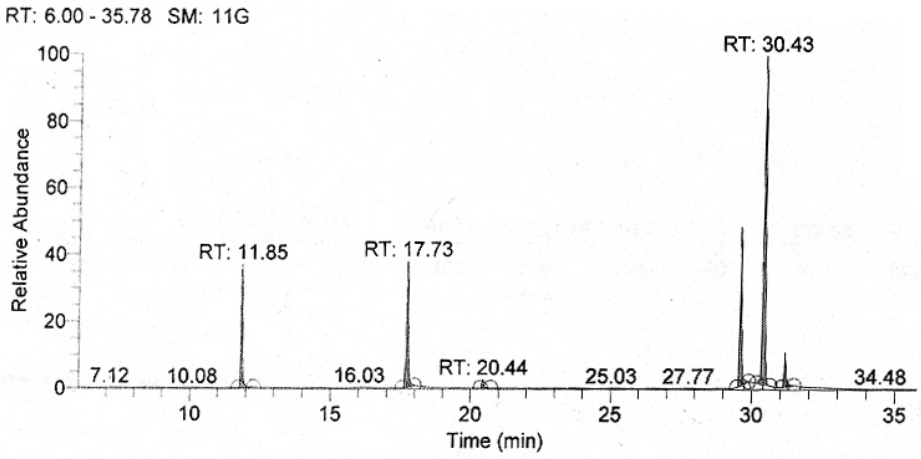
RT	Peak Area	Area %
11.85	832268	35.01
17.72	929162	39.09
29.59	170227	7.16
30.36	324898	13.67
31.16	90298	3.80
31.88	30108	1.27

GCMS Spectra; DCM as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 37, Table 6)



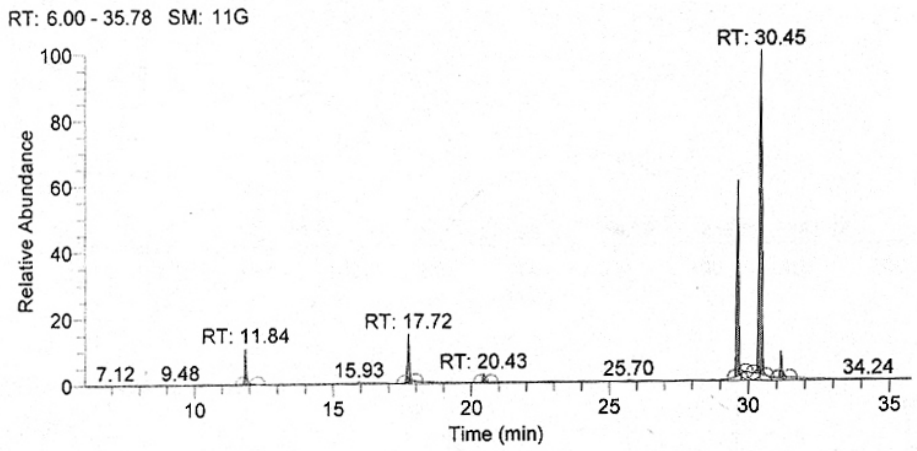
RT	Peak Area	Area %
11.85	1108205	27.29
17.73	1182009	29.11
20.47	21319	0.52
29.59	633705	15.60
30.37	990302	24.38
31.16	125623	3.09

GCMS Spectra; DCM as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 38, Table 6)



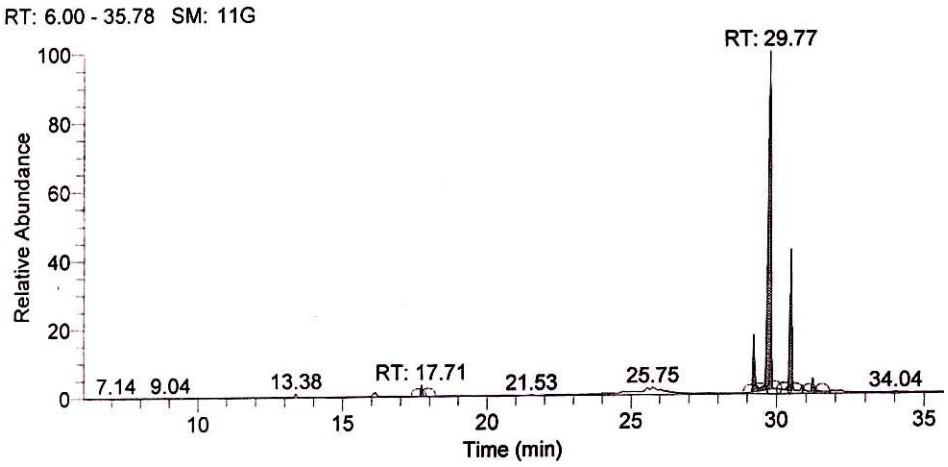
RT	Peak Area	Area %
11.85	1019147	13.64
17.73	1226568	16.42
20.44	84644	1.13
29.60	1371424	18.36
30.43	3415289	45.71
31.16	354343	4.74

GCMS Spectra; DCM as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 39, Table 6)



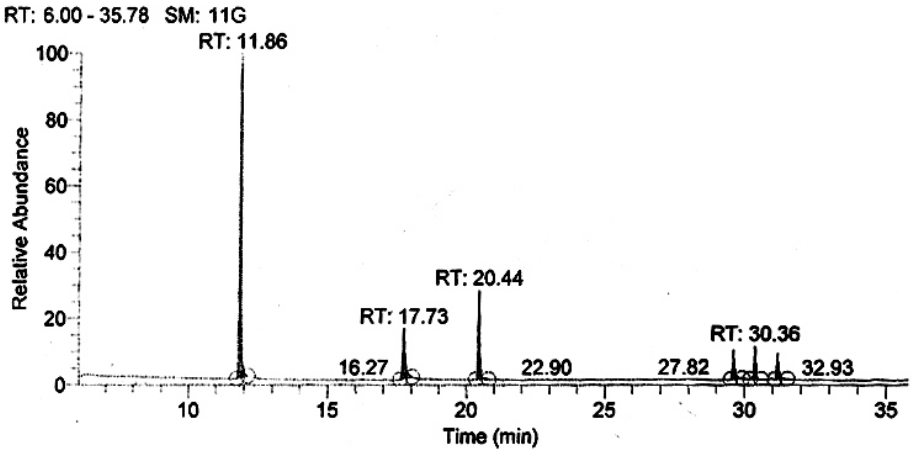
RT	Peak Area	Area %
11.84	513560	4.65
17.72	853066	7.72
20.43	129282	1.17
29.62	2906341	26.31
30.45	6222671	56.33
31.16	421654	3.82

GCMS Spectra; DCM as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 40, Table 6)



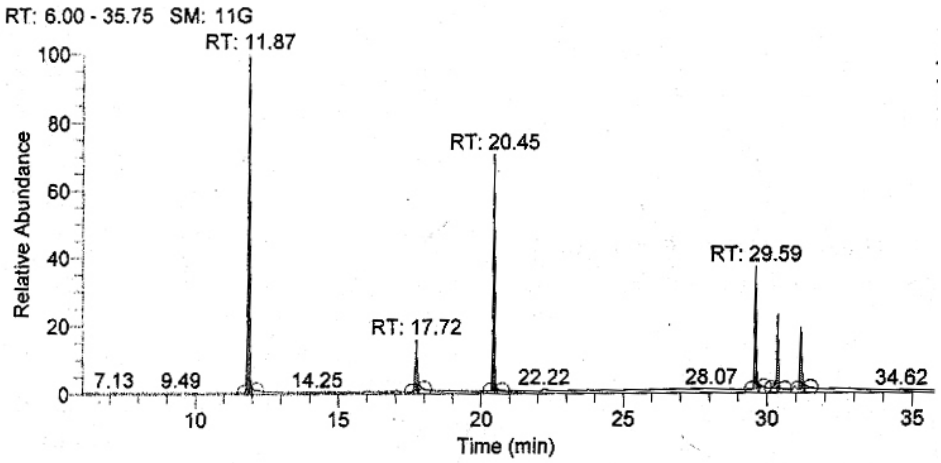
RT	Peak Area	Area %
17.71	254281	1.42
29.21	1434947	8.01
29.77	12108602	67.58
30.49	3725016	20.79
31.22	394283	2.20

GCMS Spectra; MeOH as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 41, Table 6)



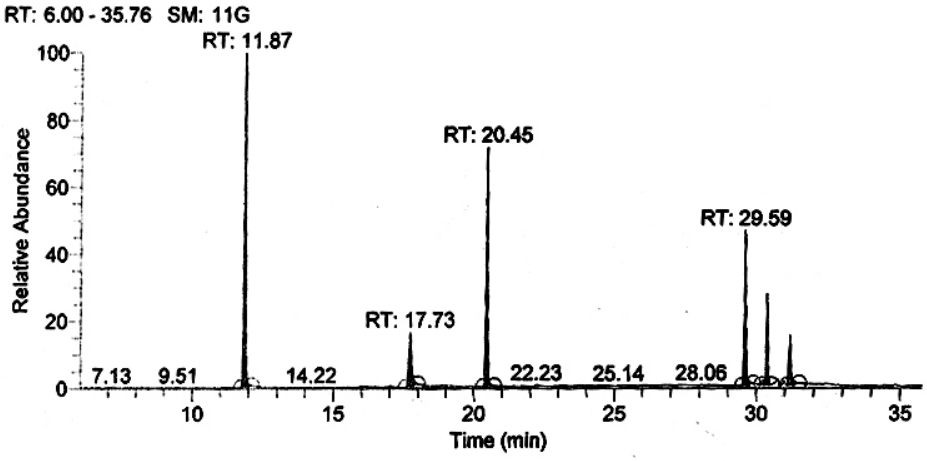
RT	Peak Area	Area %
11.86	1829227	52.89
17.73	450181	13.02
20.44	556766	16.10
29.60	226046	6.54
30.36	201292	5.82
31.16	194881	5.64

GCMS Spectra; MeOH as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 42, Table 6)



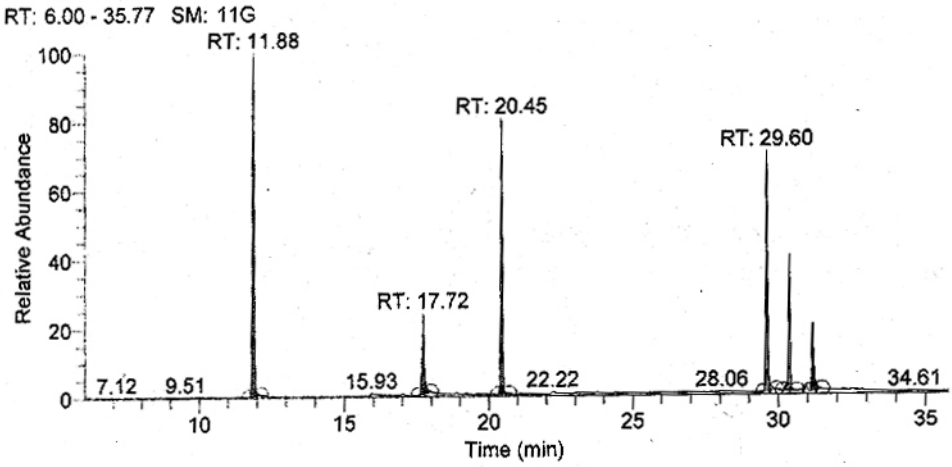
RT	Peak Area	Area %
11.87	2092184	38.00
17.72	444211	8.07
20.45	1337103	24.29
29.59	743927	13.51
30.36	441870	8.03
31.16	446431	8.11

GCMS Spectra; MeOH as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 43, Table 6)



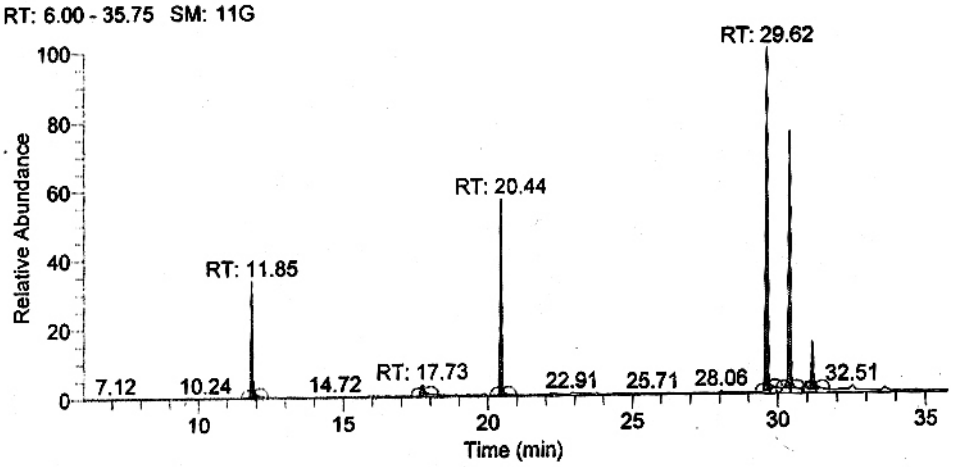
RT	Peak Area	Area %
11.87	1658366	35.56
17.73	391910	8.40
20.45	1119786	24.01
29.59	752243	16.13
30.36	439028	9.41
31.16	301904	6.47

GCMS Spectra; MeOH as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 44, Table 6)



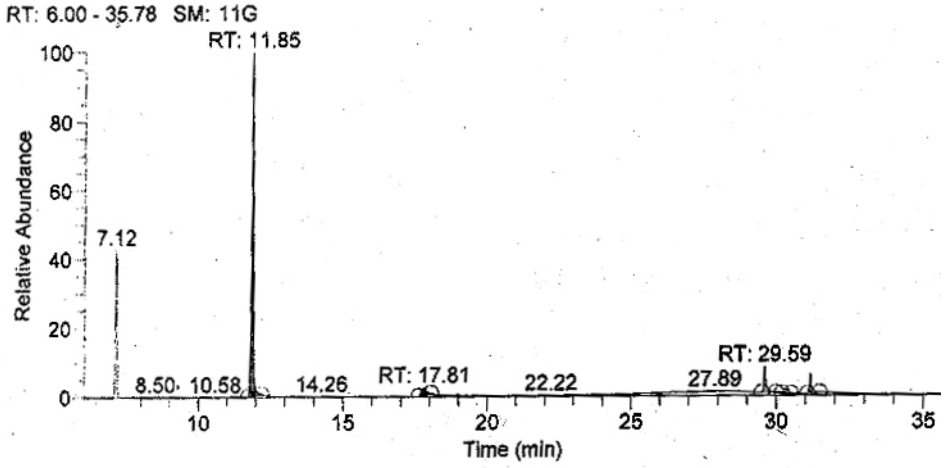
RT	Peak Area	Area %
11.88	2305353	30.65
17.72	658645	8.76
20.45	1661237	22.08
29.60	1532001	20.37
30.37	847324	11.26
31.16	517746	6.88

GCMS Spectra; MeOH as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 45, Table 6)



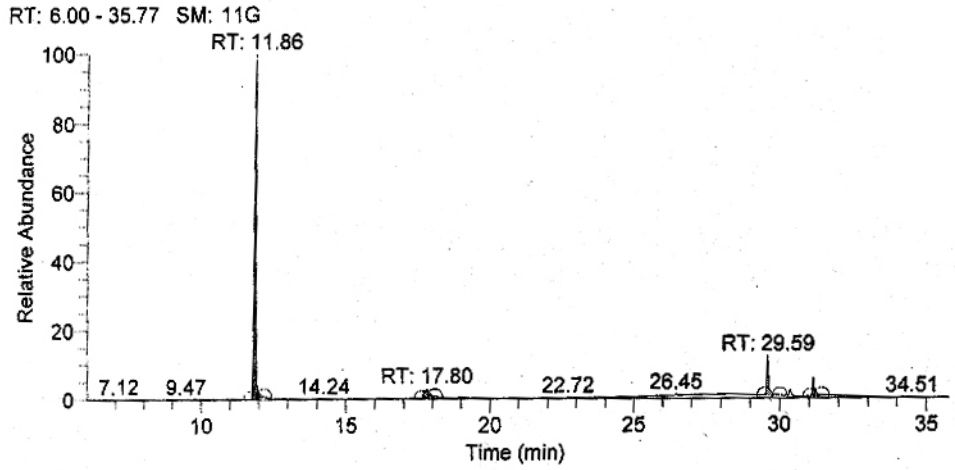
RT	Peak Area	Area %
11.85	714748	11.21
17.73	162481	2.55
20.44	1128773	17.70
29.62	2259313	35.42
30.39	1740956	27.29
31.16	372328	5.84

GCMS Spectra; THF as reaction medium with SDOSS; Sample withdrawn after 5 min. (Entry 46, Table 6)



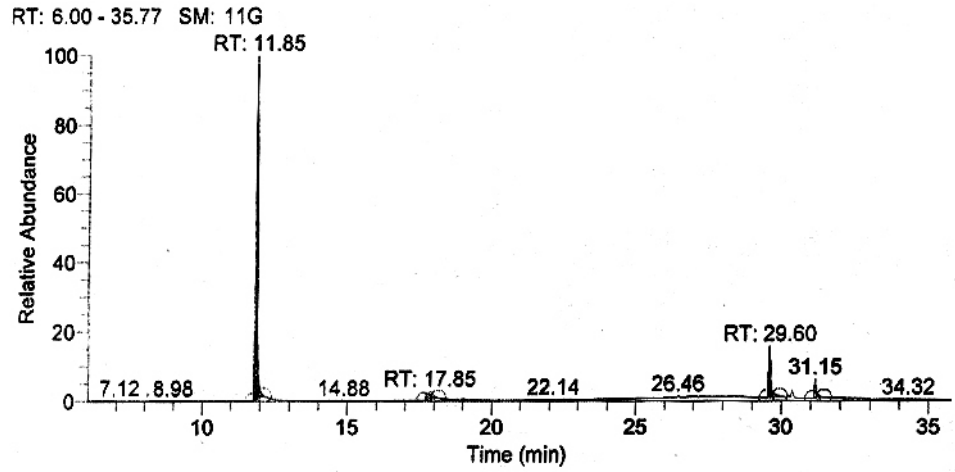
RT	Peak Area	Area %
11.85	581888	79.50
17.81	38907	5.32
29.59	56140	7.67
30.37	11491	1.57
31.15	43541	5.95

GCMS Spectra; THF as reaction medium with SDOSS; Sample withdrawn after 10 min. (Entry 47, Table 6)



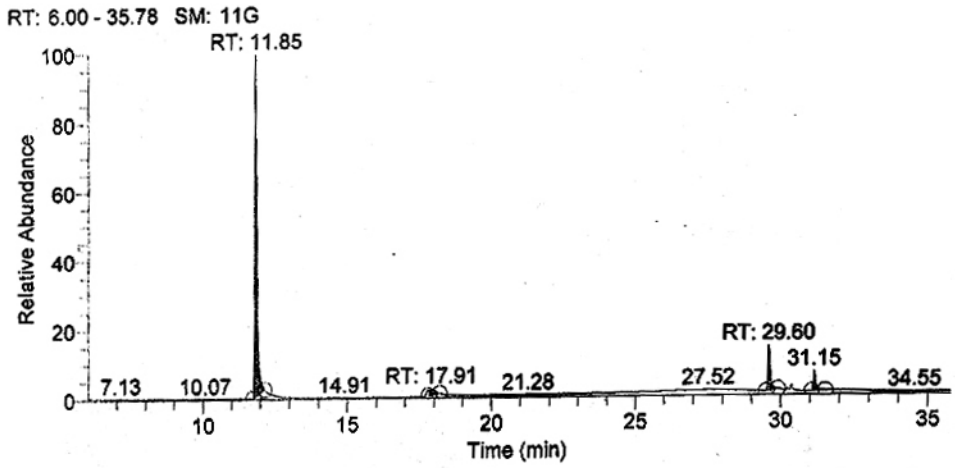
RT	Peak Area	Area %
11.86	570590	77.29
17.80	47358	6.42
29.59	80838	10.95
31.14	39444	5.34

GCMS Spectra; THF as reaction medium with SDOSS; Sample withdrawn after 15 min. (Entry 48, Table 6)



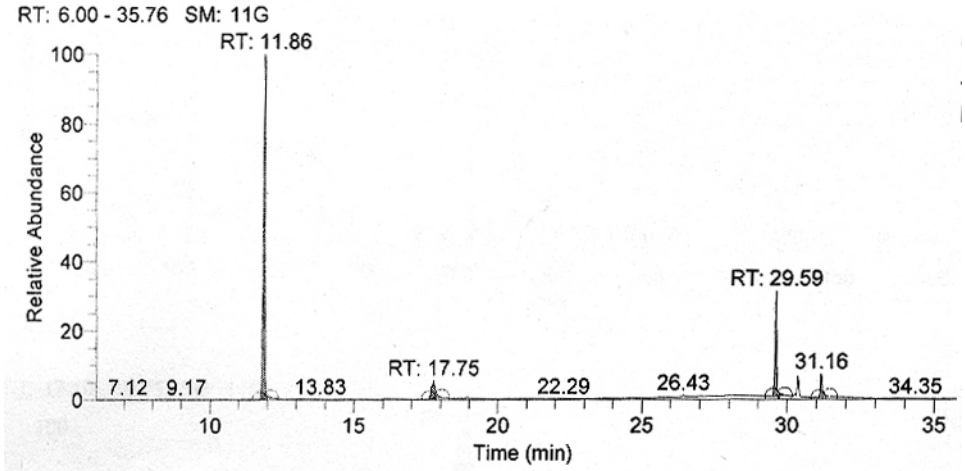
RT	Peak Area	Area %
11.85	367727	74.61
17.85	30749	6.24
29.60	67813	13.76
31.15	26551	5.39

GCMS Spectra; THF as reaction medium with SDOSS; Sample withdrawn after 30 min. (Entry 49, Table 6)



RT	Peak Area	Area %
11.85	232548	77.01
17.91	13165	4.36
29.60	38353	12.70
31.15	17909	5.93

GCMS Spectra; THF as reaction medium with SDOSS; Sample withdrawn after 60 min. (Entry 50, Table 6)



RT	Peak Area	Area %
11.86	814887	66.34
17.75	98408	8.01
29.59	245011	19.95
31.16	70026	5.70

4. Studies on oxygen uptake by various surfactants:

Figure A: Oxygen uptake by different surfactants

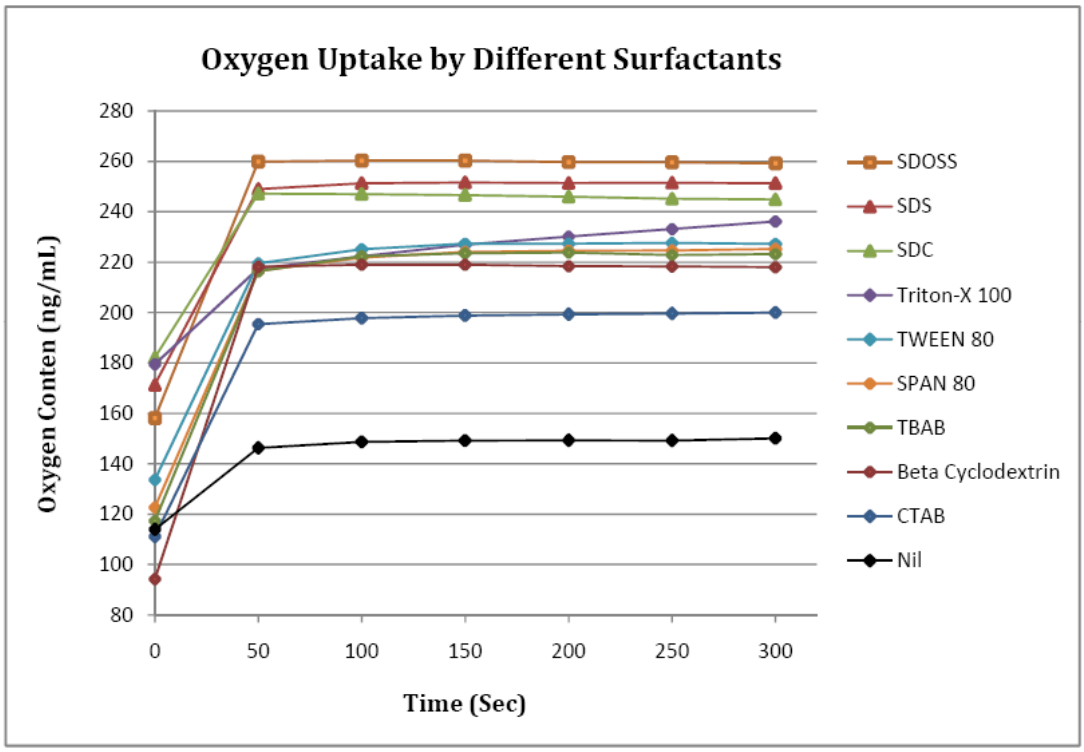


Figure B: Oxygen uptake at different concentration of SDOSS

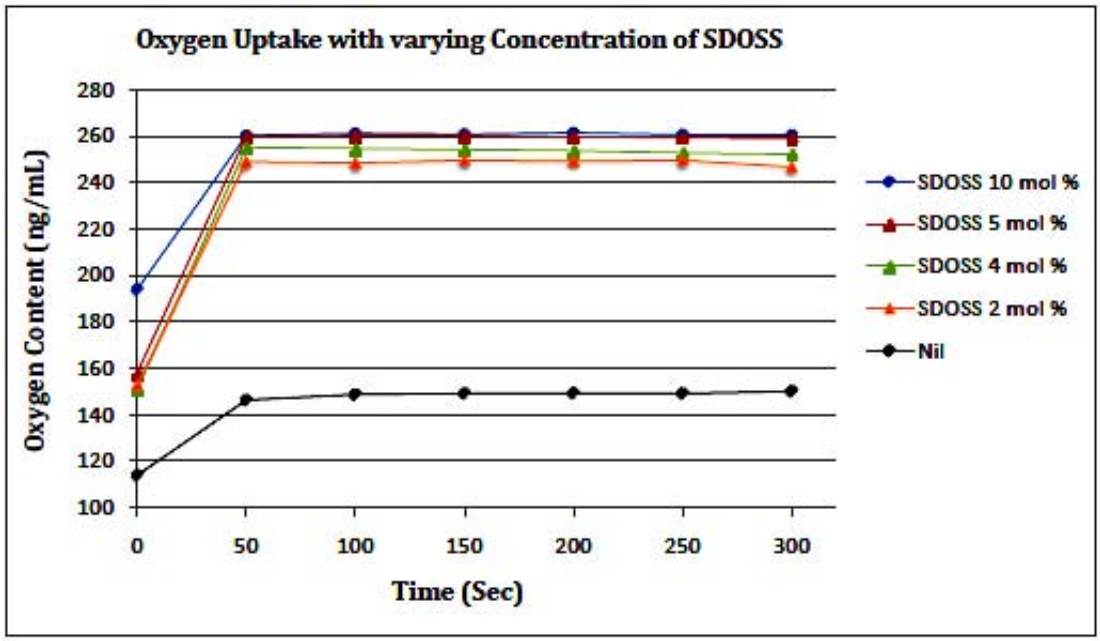
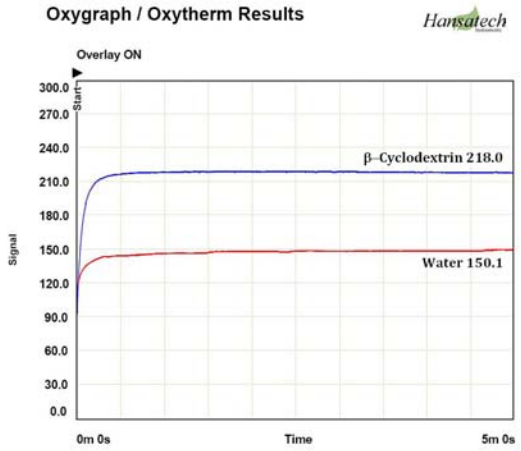
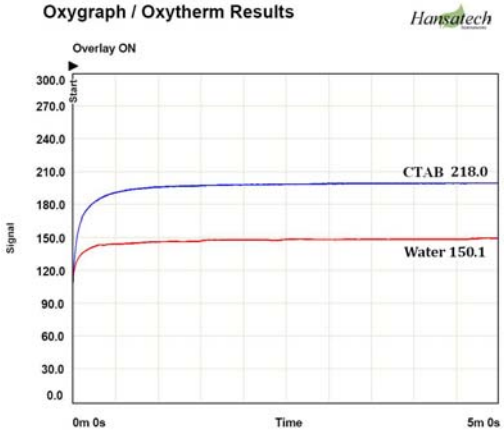


Figure C: Comparision of oxygen uptake by different surfactants with water

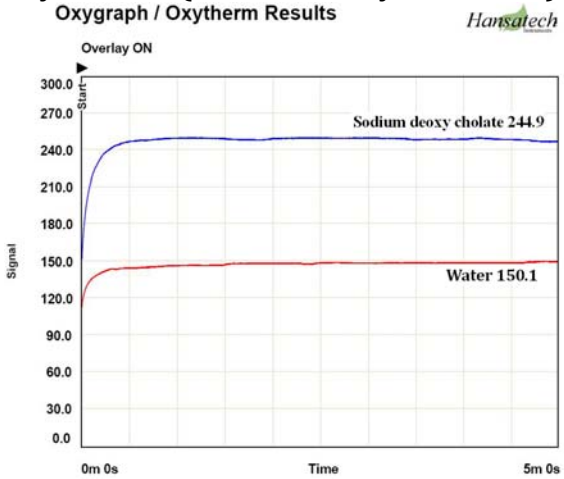
i) β-Cyclodexrin (5 mol%:Entry 2, Table 8)



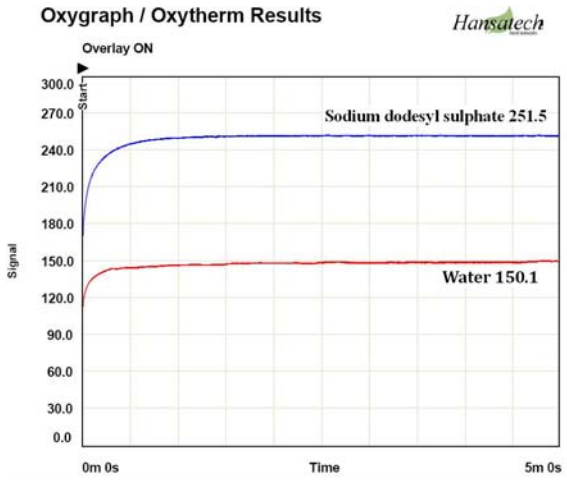
ii) Cetyl tetrabutyl ammonium bromide (5 mol%:Entry 3, Table 8)



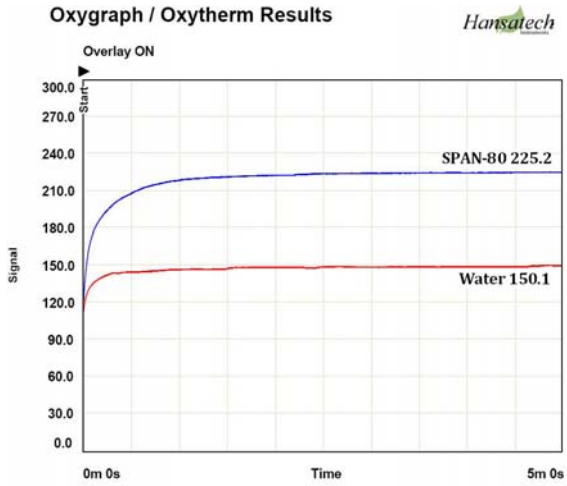
iii) Sodium deoxy cholate (5 mol%:Entry 4, Table 8)



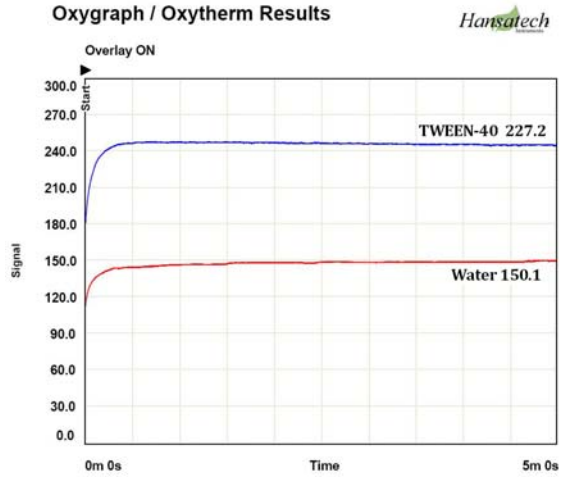
iv) Sodium Dodecyl Sulfate (5 mol%:Entry 5, Table 8)



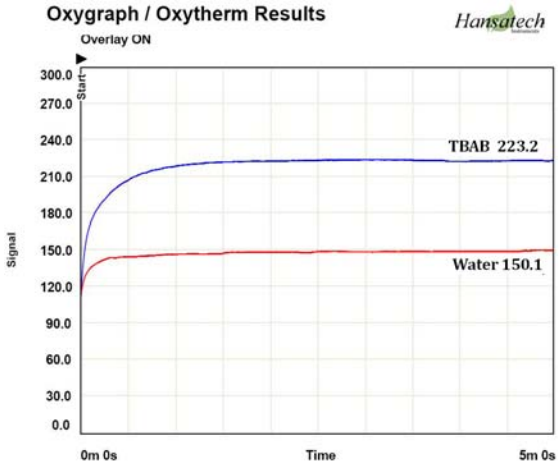
v) SPAN-40 (5 mol%:Entry 6, Table 8)



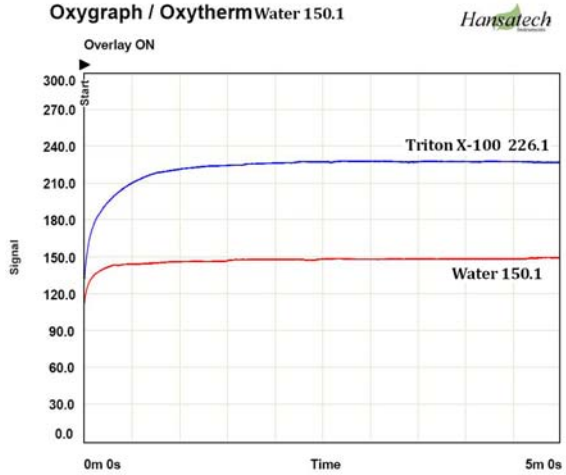
vi) TWEEN-40 (5 mol%:Entry 7, Table 8)



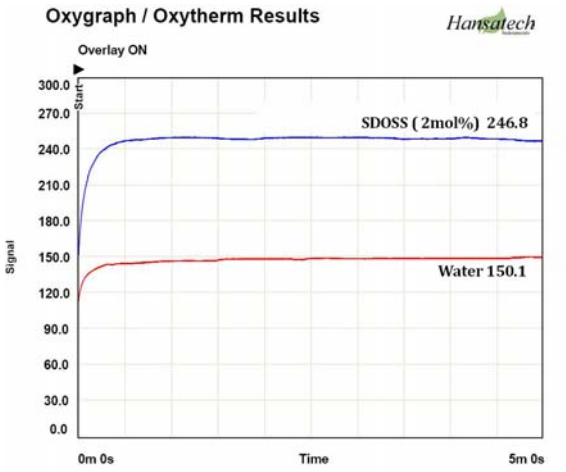
vii) Tetrabutyl ammonium bromide (5 mol%:Entry 8, Table 8)



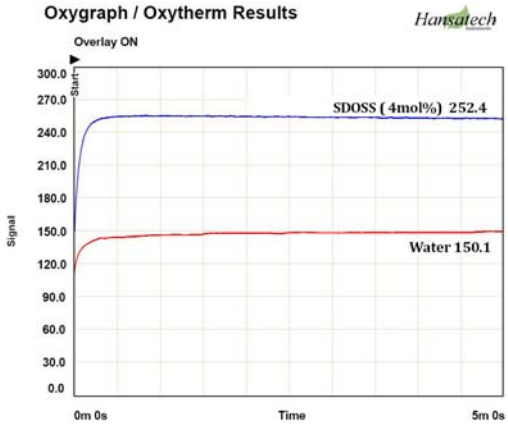
viii) Trioton X-100 (5 mol%:Entry 8, Table 8)



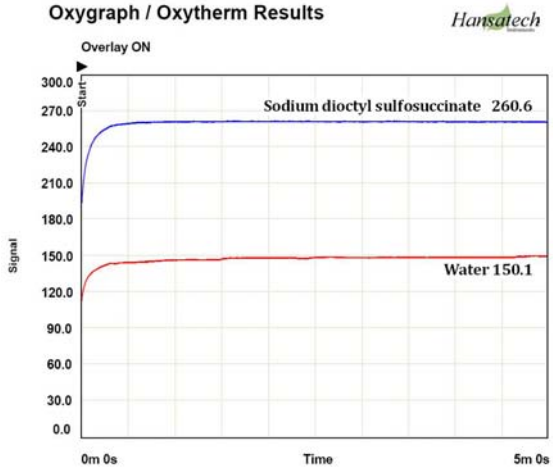
ix) Sodium dioctyl sulfosuccinate (2 mol %:Entry 9, Table 8)



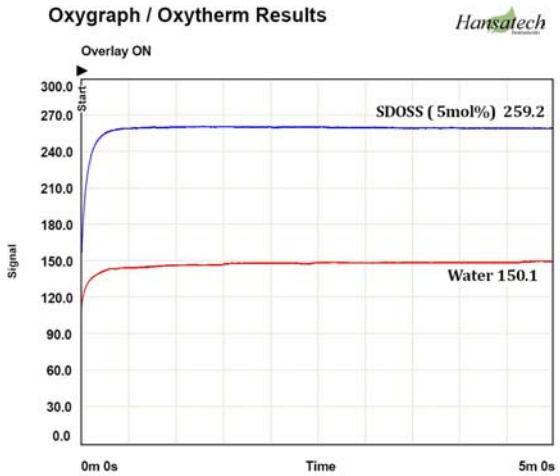
x) Sodium dioctyl sulfosuccinate (5 mol %:Entry 10, Table 8)



xi) Sodium dioctyl sulfosuccinate (10 mol %:Entry 11, Table 8)



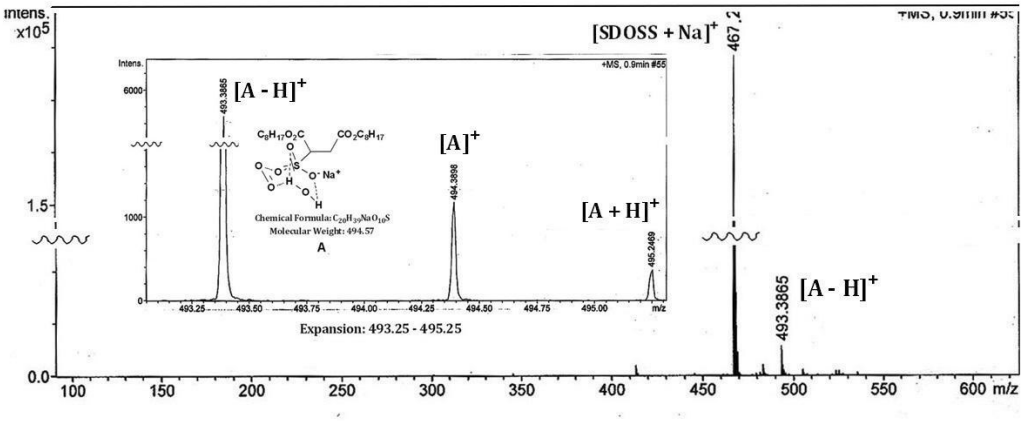
xii) Sodium dioctyl sulfosuccinate (15 mol %:Entry 12, Table 8)



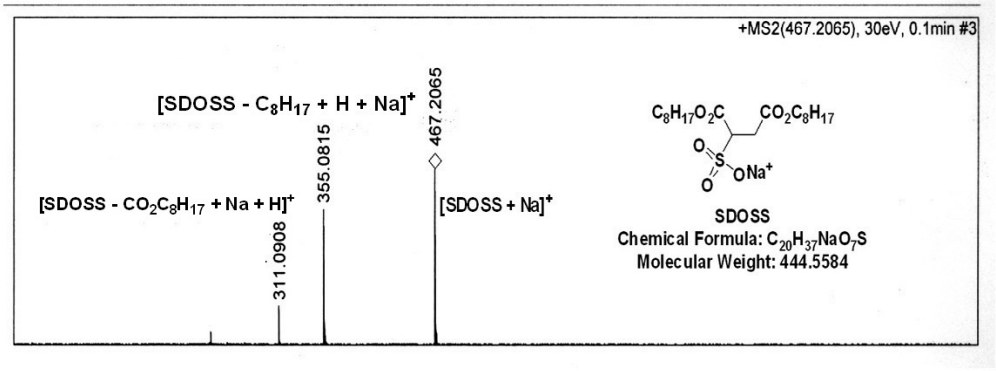
5. Mass Spectrometric studies:

A) MS studies with SDOSS:

I) (+ve) ESI HRMS (Bruker Maxis: Q-TOF) of aliquot of sample from a stock solution of 5 mol % (with respect to aldehyde during reaction) of SDOSS in 5 mL of 1:1 MeCN-water.

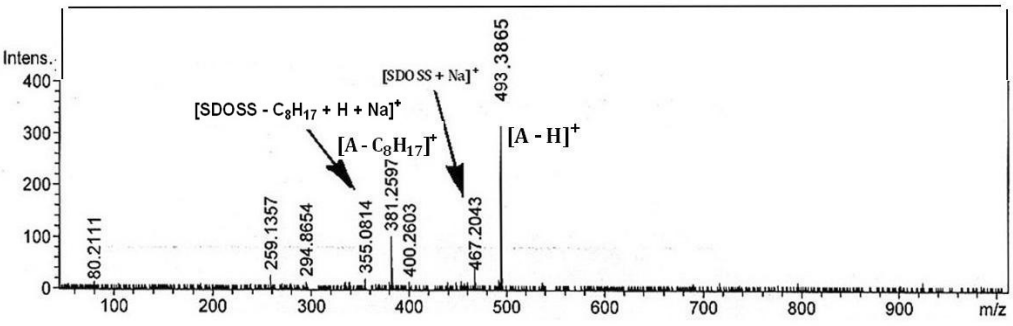


(a) TIC



Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
467.2065	1	C 20 H 37 Na 2 O 7 S	100.00	467.2050	-3.2	-3.2	122.0	1.5	even	ok

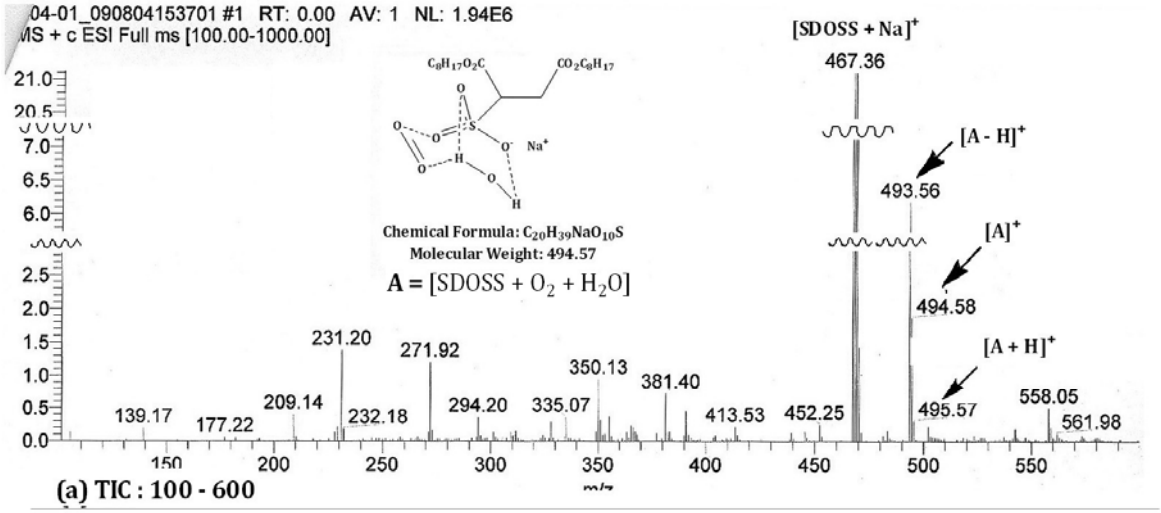
(b) MS^2 of m/z 467.2065

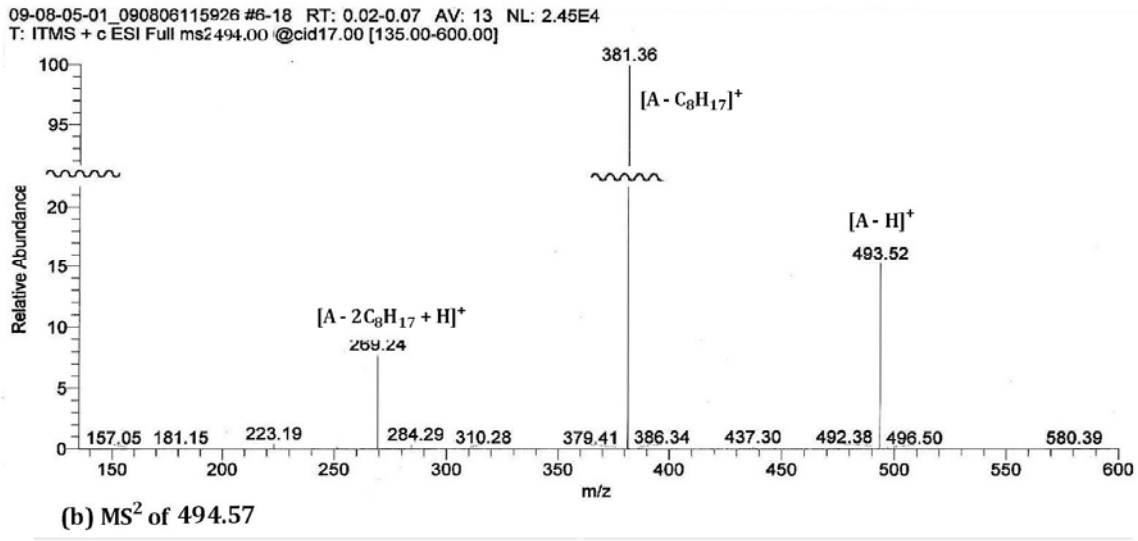


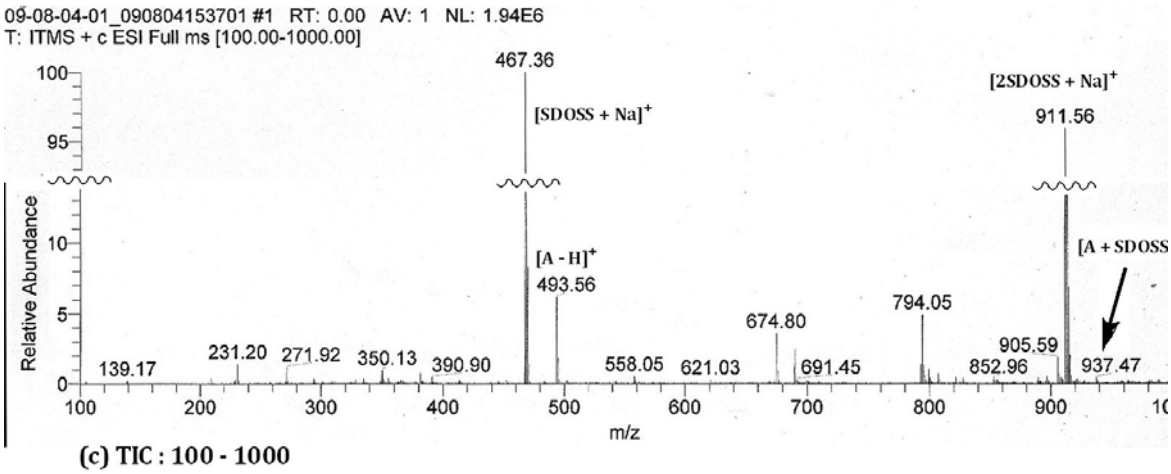
Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
467.2044	1	C 20 H 37 Na 2 O 7 S	100.00	467.2050	1.2	1.1	122.0	1.5	even	ok

(c) MS^2 of m/z 493.3898

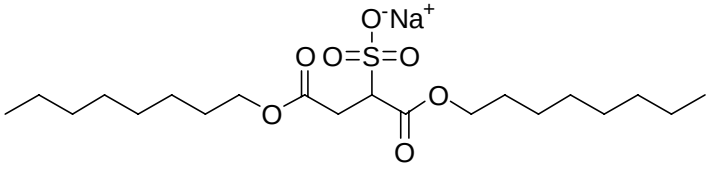
II) (+ve) ESI MS (Thermo Scientific LTQ-XL: Linear ion trap) of aliquot of sample from a stock solution of 5 mol % (with respect to aldehyde during reaction) of SDOSS in 5 mL of 1:1 MeCN-water.



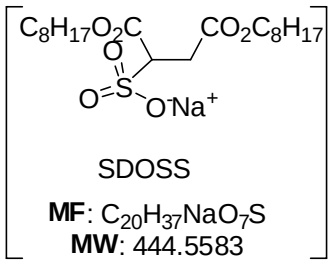




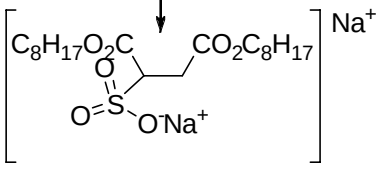
III) Structure, Molecular Formula (MF) and Molecular Weight (MW) (Calculated using Chem Draw) of the different species/ions observed in the (+ve) ESI HRMS/(+ve) ESI MS of aliquot of sample from a stock solution of 5 mol % (with respect to aldehyde during reaction) of SDOSS in 5 mL of 1:1 MeCN-water.



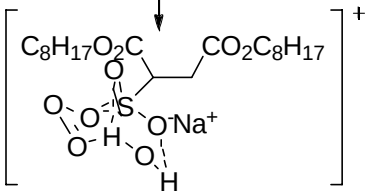
Sodium Dioctyl Sulfosuccinate (SDOSS)
MF: $C_{20}H_{37}NaO_7S$; **MW**: 444.5583



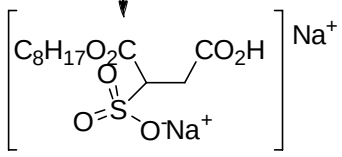
SDOSS
MF: $C_{20}H_{37}NaO_7S$
MW: 444.5583



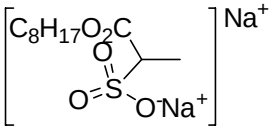
$[SDOSS + Na]^+$
MF: $C_{20}H_{37}Na_2O_7S$
MW: 467.5476



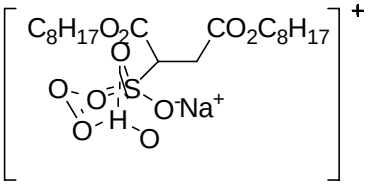
[A: $SDOSS+O_2+H_2O]$
MF: $C_{20}H_{39}NaO_{10}S$
MW: 494.5724



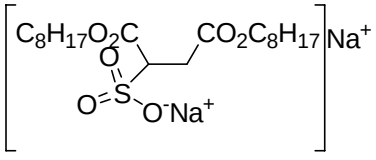
$[SDOSS + Na - C_8H_{17} + H]^+$
MF: $C_{12}H_{20}Na_2O_7S$
MW: 355.3349



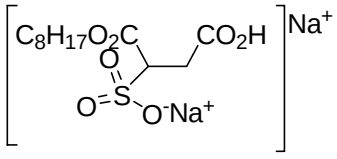
$[SDOSS + Na - CO_2C_8H_{17} + H]^+$
MF: $C_{11}H_{21}Na_2O_5S$
MW: 311.3254



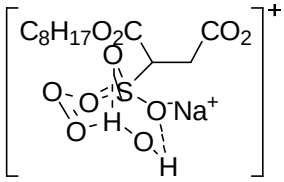
$[\mathbf{A} - H]^+$
MF: $C_{20}H_{38}NaO_{10}S$
MW: 493.5645



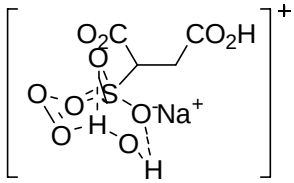
$[SDOSS + Na]^+$
MF: $C_{20}H_{37}Na_2O_7S$
MW: 467.5476



$[SDOSS + Na - C_8H_{17}^+ H]^+$
MF: $C_{12}H_{21}Na_2O_7S$
MW: 355.3349



$[\mathbf{A} - C_8H_{17}]^+$
MF: $C_{12}H_{22}NaO_{10}S$
MW: 381.3518



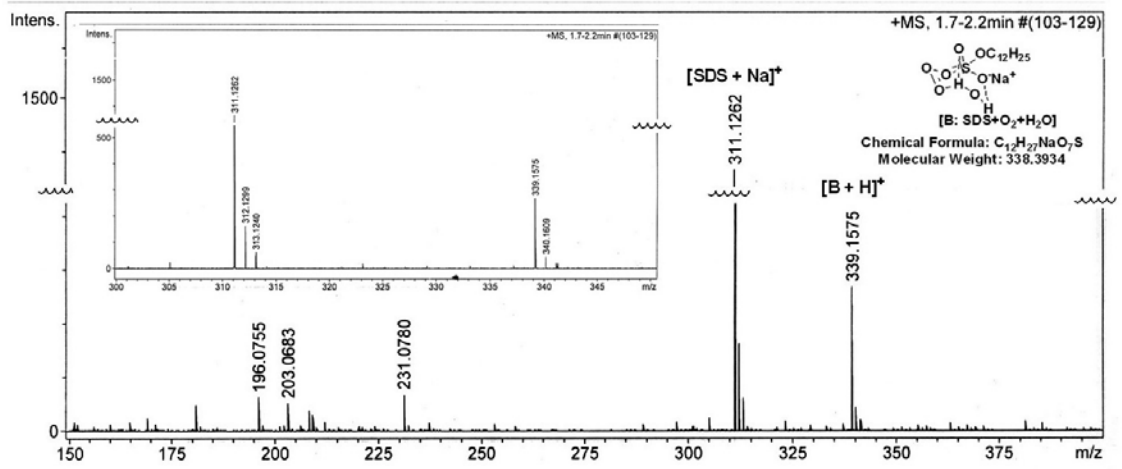
$[\mathbf{A} - 2C_8H_{17} + H]^+$
MF: $C_4H_6NaO_{10}S$
MW: 269.1387

IV) Various ions observed for sample from a stock solution of 5 mol % (with respect to aldehyde during reaction) of SDOSS in 5 mL of 1:1 MeCN-water.

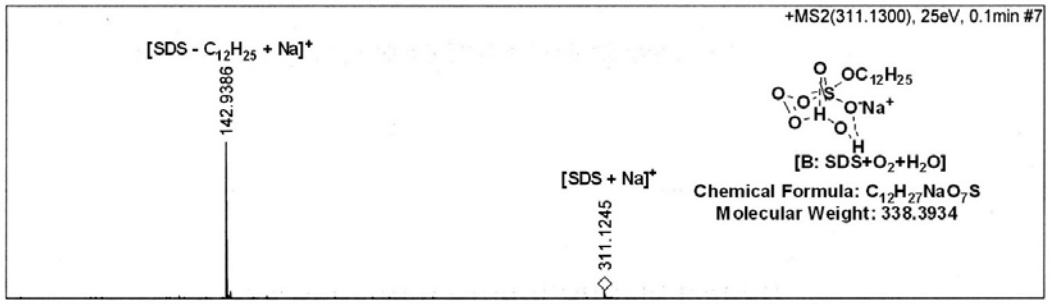
Entry	Species/Ions	Calc Mass (Chem Draw)	+ve ESI HRMS Observed (*m/z*)	+ve ESI MS Observed (*m/z*)	Spectra Type
1	$[SDOSS + Na]^+$	467.5476	467.2043	467.36	TIC
2	$[SDOSS + O_2 + H_2O - H]^+$	493.5645	493.3865	493.56	TIC
3	$[SDOSS + O_2 + H_2O]^+$	494.5724	494.3898	494.58	TIC
4	$[SDOSS + O_2 + H_2O + H]^+$	495.5804	495.2469	495.57	TIC
5	$[SDOSS + Na]^+$	467.5476	467.2065	--	MS^2 of 467
6	$[SDOSS + Na - C_8H_{17} + H]^+$	355.3355	355.0815	--	MS^2 of 467
7	$[SDOSS + Na - CO_2C_8H_{17} + H]^+$	311.3260	311.0908	--	MS^2 of 467
8	$[SDOSS + O_2 + H_2O - H]^+$	493.5645	493.3865	493.52	MS^2 of 493
9	$[SDOSS + Na]^+$	467.5476	467.2043	--	MS^2 of 493
10	$[SDOSS + O_2 + H_2O - C_8H_{17}]^+$	381.3518	381.2597	381.36	MS^2 of 493
11	$[SDOSS + Na - C_8H_{17} + H]^+$	355.3355	355.0814	--	MS^2 of 493
12	$[SDOSS + O_2 + H_2O - 2C_8H_{17} + H]^+$	269.1393	--	269.24	MS^2 of 493

B) MS studies with SDS:

I) (+ve) ESI HRMS (Bruker Maxis: Q-TOF) of aliquot of sample from a stock solution of 5 mol % (with respect to aldehyde during reaction) of SDS in 5 mL of 1:1 MeCN-water.

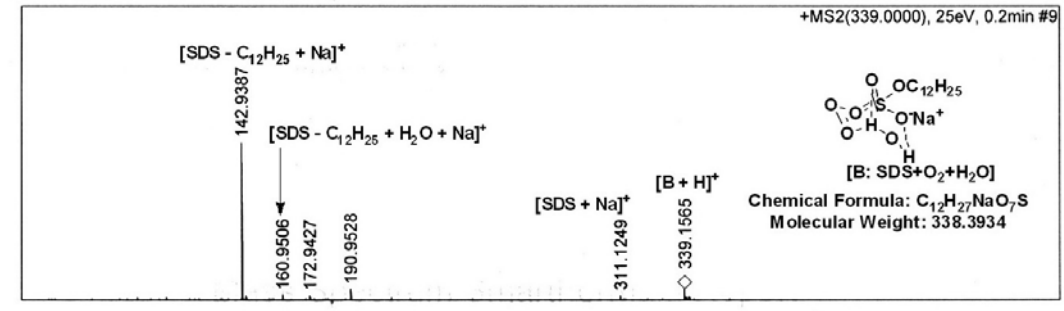


(a) TIC



Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSig ma	rdb	e⁻ Conf	N-R ule
311.1245	1	C 12 H 25 Na 2 O 4 S	100.00	311.1263	5.9	5.7	75.7	-0.5	even	ok

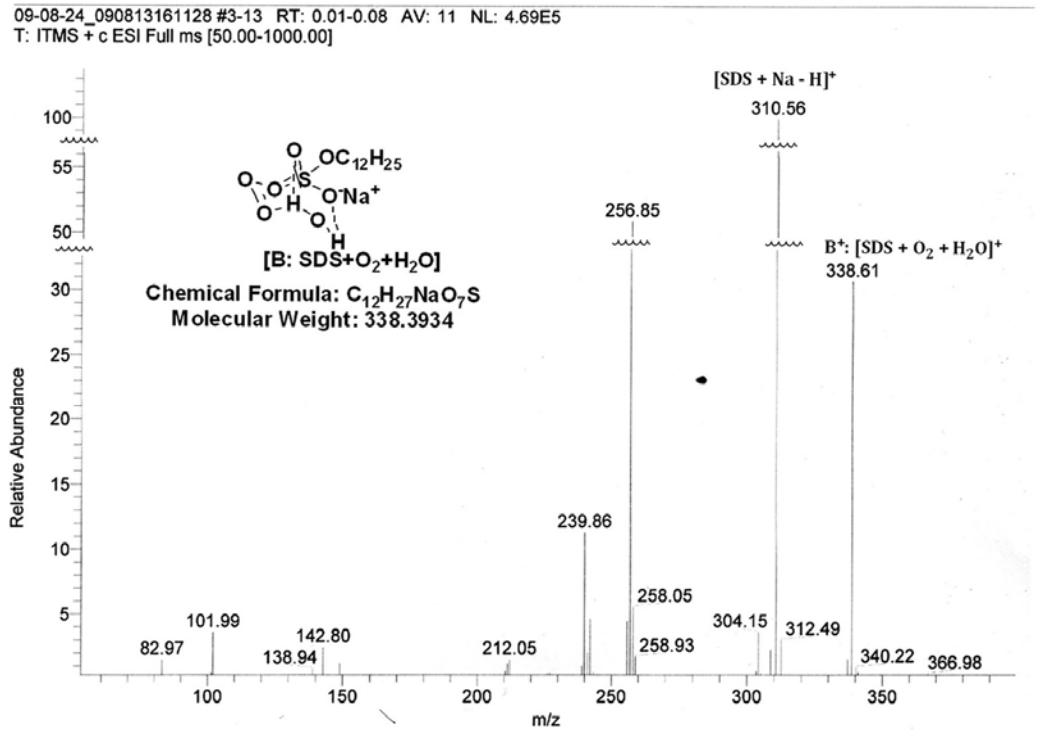
(b) MS^2 of m/z 311



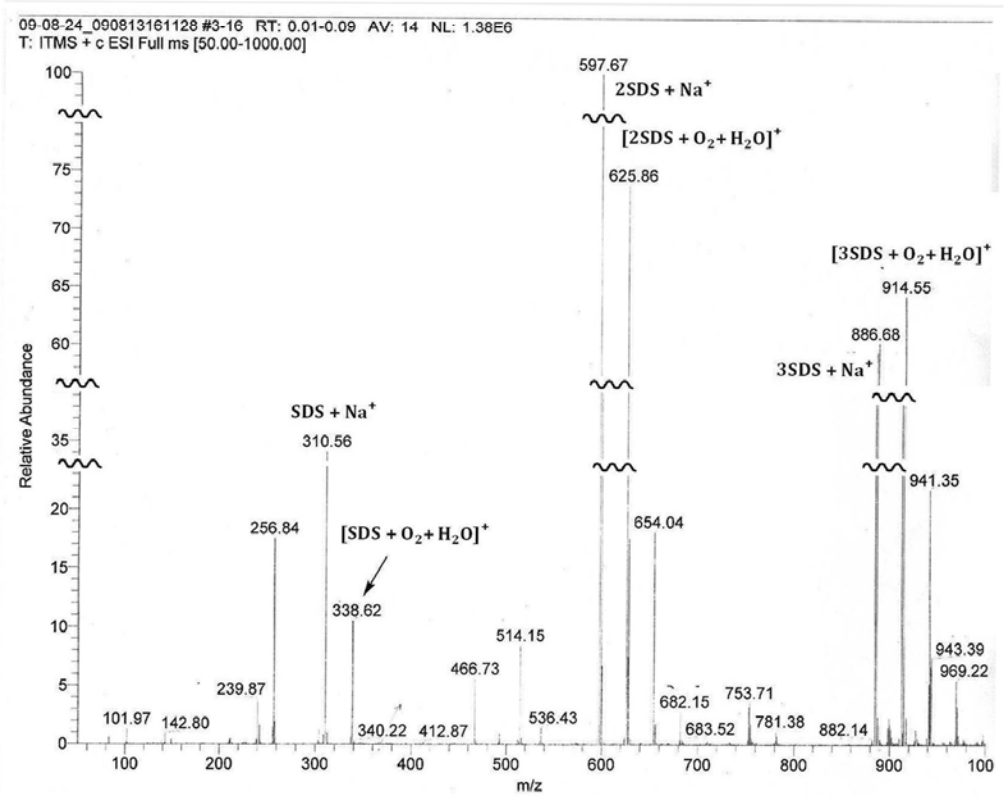
Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSig ma	rdb	e⁻ Conf	N-R ule
311.1249	1	C 12 H 25 Na 2 O 4 S	100.00	311.1263	4.6	4.3	75.7	-0.5	even	ok

(c) MS^2 of m/z 339

II) (+ve) ESI MS (Thermo Scientific LTQ-XL: Linear ion trap) of sample from stock solution of 5 mol % (with respect to aldehyde during reaction) of SDS in 5 mL of 1:1 MeCN-water.

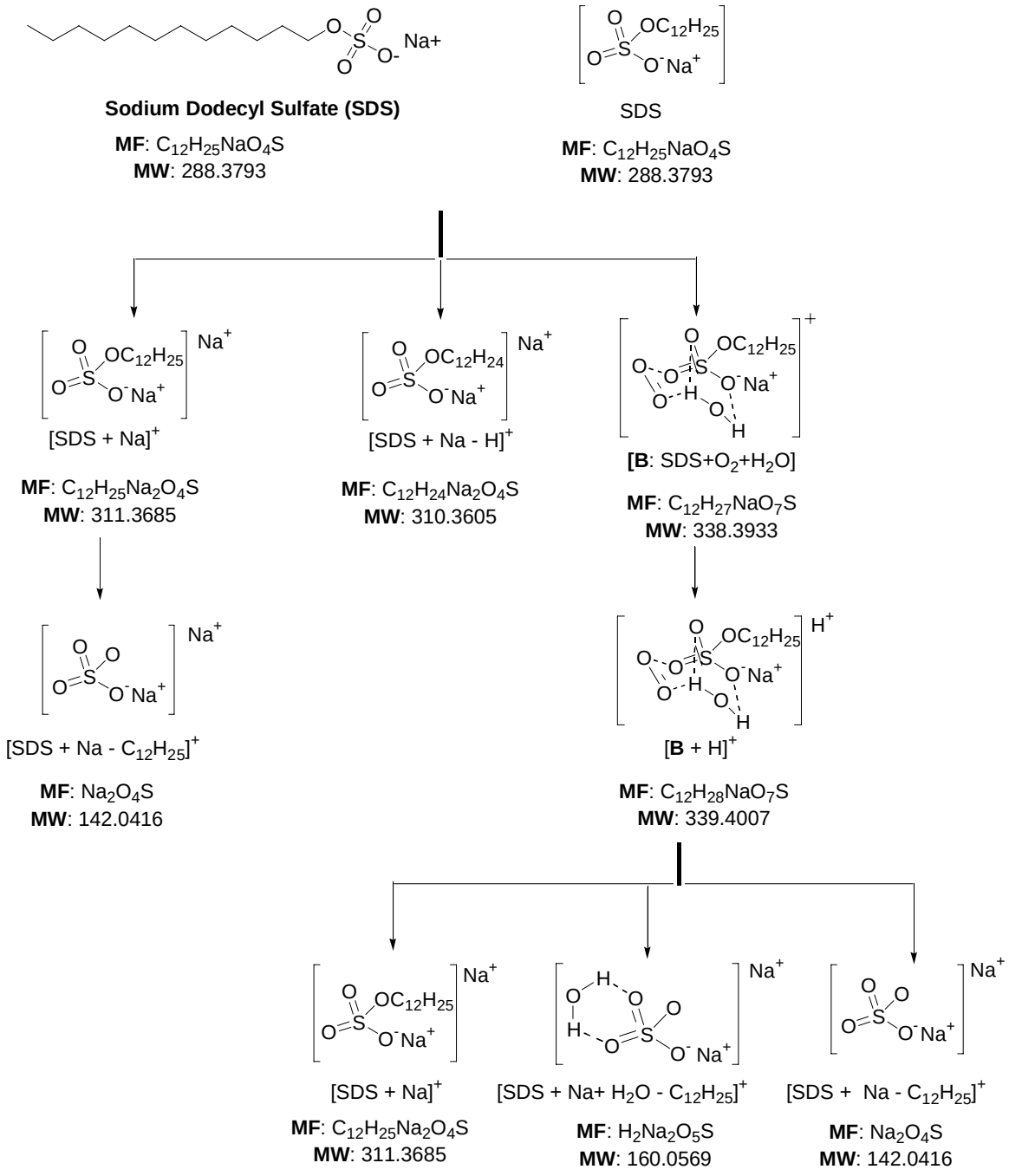


(a) TIC : 50 - 400



(b) TIC : 50 - 1000

III) Structure, Molecular Formula (MF) and Molecular Weight (MW) (calculated using Chem Draw) of different species/ion observed in the (+ve) ESI HRMS/(+ve) ESI MS of sample from stock solution of 5 mol % (with respect to aldehyde during reaction) of SDS in 5 mL of 1:1 MeCN-water.

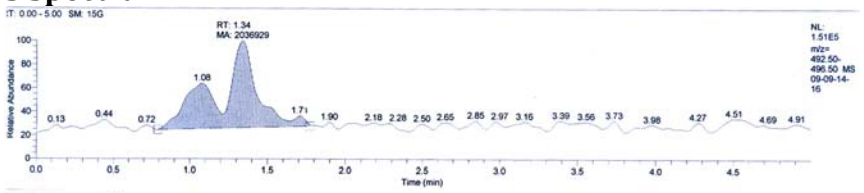


IV) Various ions observed for aliquot of sample from stock solution of 5 mol % (with respect to aldehyde during reaction) of SDS in 5 mL of 1:1 MeCN-water.

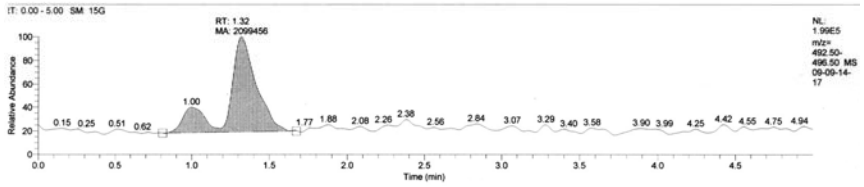
Entry	Species	Calcd. Mass (Chem Draw)	+ve ESI HRMS Observed (*m*/*z*)	+ve ESI-MS Observed (*m*/*z*)	Spectra Type
1	$[SDS + Na - H]^+$	310.3606	--	310.56	TIC
2	$[SDS + Na]^+$	311.3690	311.1262	--	TIC
3	$[SDS + O_2 + H_2O]^+$	338.3934	--	338.61	TIC
4	$[SDS + O_2 + H_2O + H]^+$	339.1453	339.1575	--	TIC
5	$[SDS + Na]^+$	311.3690	311.1245	--	MS^2 of 311
6	$[SDS + Na - C_{12}H_{25}]^+$	142.0421	142.9386	--	MS^2 of 311
7	$[SDS + O_2 + H_2O + H]^+$	339.1453	339.1565	--	MS^2 of 339
8	$[SDS + Na]^+$	311.3690	311.1249	--	MS^2 of 339
9	$[SDS + Na - C_{12}H_{25} + H_20]^+$	160.0574	160.9506	--	MS^2 of 339
10	$[SDS+ Na - C_{12}H_{25}]^+$	142.0421	142.9387	--	MS^2 of 339

6. Ion Current at m/z 492.5-496.5 in (+ve) ESI MS (Thermo Scientific LTQ-XL: Linear ion trap) of different amounts of SDOSS.

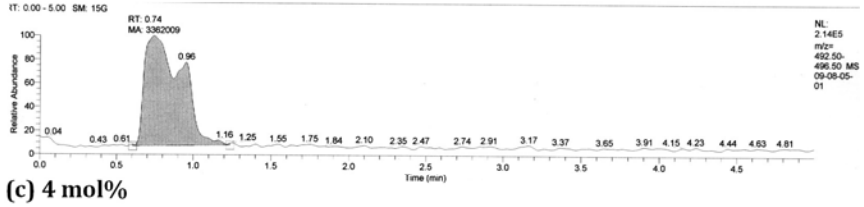
A) MS Spectra



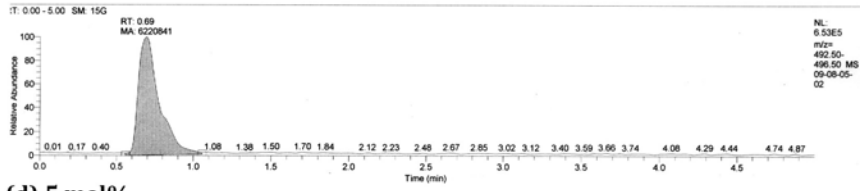
(a) 1 mol%



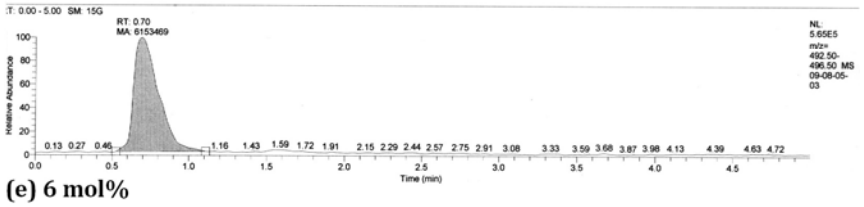
(b) 2 mol%



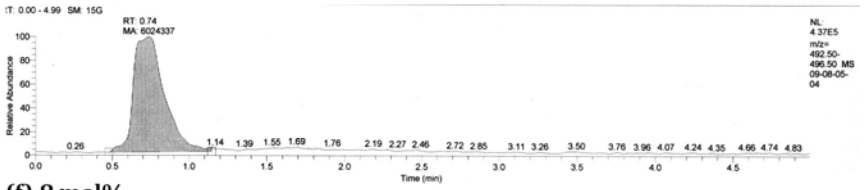
(c) 4 mol%



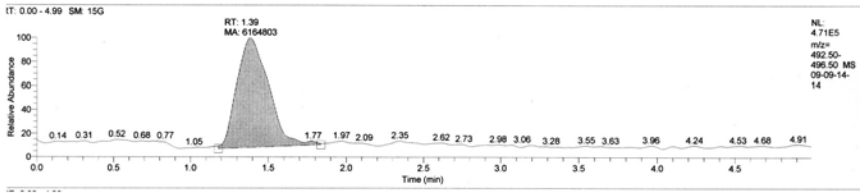
(d) 5 mol%



(e) 6 mol%



(f) 8 mol%



(g) 10 mol%

B) Ion Current of the species at 492.5 – 496.5 using different amount of SDOSS:

Cat Mol%	AUC (492.5-496.5)
1	2036929
2	2099456
4	3362009
5	6220841
6	6153469
8	6024337
10	6164803

C) Supplementary figure 4: Area of the species at m/z 492.5-496.5 using different amount of SDOSS: