Qualitative and quantitative analysis of linear Alkyl Benzen compounds in Detergents by Gas-liquid Chromatography technique

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Abstract:

In this research an accurate, precise and sensitive method was developed for the determination of the Linear Alkyl Benzene (LAB) compounds (Decyl, undecyl, Dodecyl and Tridecyl) benzene using (GC Varian 3300, GC-MS Varian 3800 with FID detector). Two packed columns (SE-30 and OV-101) and three capillary columns with different polarity of liquid stationary phases (SPB-1, CB-sill13CB and VF-5ms) have been used. The optimum conditions for analysis of these compounds using the best capillary column (VF-5ms) were: column temperature $180C^0$, detector temperature $275C^0$, injector temperature $250C^0$ sample size 0.25 µl with flow rate of 2.5ml/min for Nitrogen as a carrier gas. GC-MS Varian 3800 was also used for identification of these compounds and was found that their elution sequence was (decyl, undecyl, Dodecyl and tridecyl) benzene respectively. The calibration curve and area normalization methods have been applied for quantitative determination. These two methods give an accuracy wherein the relative error was between (-0.072 to 0.904) while the precision (RSD%)was not more than (0.7) with recovery between 98.59-99.8%. The detection limits were (7.6×10^{-5}, 3.3×10^{-4}, 3.4×10^{-4} and 1.2×10^{-4}) molar for (Decyl, undecyl, Dodecyl and tridecyl) benzene respectively.

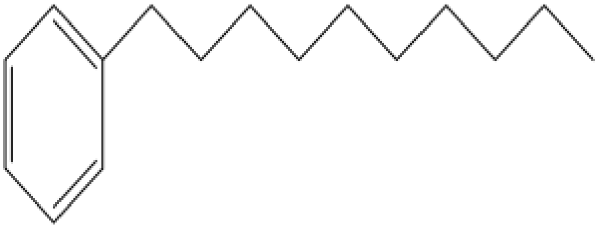
This method was applied on samples :

1- Standard from (ARADET/Baiji/Iraq) contains all isomers for each compound, (relative error between -0.79 to 0.95).
2- Products in (ARADET), (relative error between -1.7 to 1.58).
3- From Syrian united company for Petrochemical industry.

The comparison of the results obtained from this method with the results of Spanish Pernz Company for the same sample has shown relative error in the range of (-0.042 to 1.09). The storage of linear alkyl benzene at different temperatures have no effect on its composition.

Introduction

A mixture of Linear Alkyl Benzene (LAB) compounds with precise known percentages is used to produce the detergents commercially after sulfonation with H_2SO_4 or pure SO_3 then neutralizing with NaOH and the addition of Colors, scents and other additives(1). The most important LAB compounds involved in the detergent industry are(2): Decyl, undecyl, Dodecyl and tridecyl Benzene and their chemical formula are: $C_{16}H_{26}$, $C_{17}H_{28}$, $C_{18}H_{30}$ and $C_{19}H_{32}$ respectively. The chemical structure of one of these compounds (Decyl Benzene) is: $C_{16}H_{26}$



Although there are many methods & studies about the industry of the commercial detergents materials, but, there are few studies on the LAB compounds. Many companies such as Syrian United Company for Petrochemicals relies on (UOP 673-88) technique[3] to calculate the concentrations of LAB isomers using the capillary column (DB-17). On the other hand ARADET and the Joradanian Al-riad company are using (UOP698-696) technique using the capillary column (SPB50) and Gas-Liquid Chromatography (GLC) apparatus[3].

The impurities existed in the LAB have been analyzed by Berna and John[4] using gravimetric analysis and the results were compared with that of HPLC technique.

The isomers of LAB have been determined by different methods: GLC[5],GC-MS[6] using DB5Ms as a capillary column, HPLC[4,7- 9],HPLC-FL[10],LC-ES-MS[11], LC-SPME[12], spectrophotometry[13], flow injection photometric[14], and UOP429-75[15]. David and Fredric[16] reduced Bromine number of the LAB using clays and Zeolite. The aim in this study is to develop a precise and fast method to separate the isomers of the main material which involved in the detergents industry using GC-MS technique and comparison with the methods used in ARADET Company, the Syrian United Company for detergents industry and the Spanish Pernz Company.

Experimental part

1-Apparatus & instruments:

Two sophisticated apparatus have been used in this study:

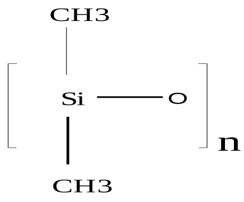
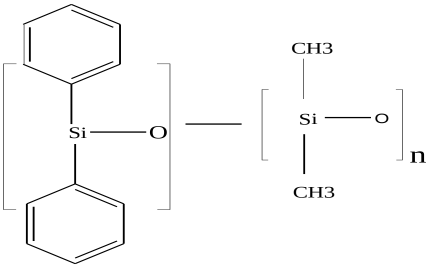
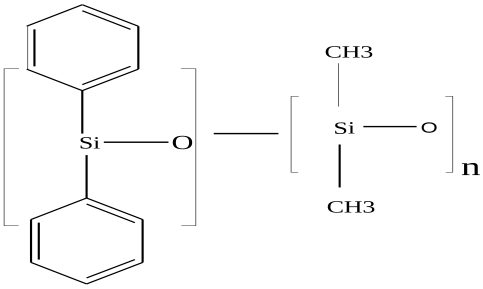
- A-) Gas-Liquid Chromatography (GLC) of type **(Varian 3300)** equipped with flame ionization detector (FID) with N_2 of 99.99% purity as a carrier gas.
- B-) Gas Chromatography-Mass spectroscopy (GC-MS) of type **(Varian 3800)** with He as a carrier gas.

- C-) Flow meter has been used to measure the gas velocity in the column especially in GC-Varian 3300.
- D-) Integrator Computer of type (Varian 4900) with capacity of 1ml/volt was connected with a device to detect and treat the signals using (Work station system) as a soft ware.

2-Chromatographic Columns

In addition to the packed columns SE-30 and OV-101, three types of capillary columns of different length, diameter and polarities have been used as shown in table(1):

Table(1): Types of capillary columns.

Column	Polarity	Scientific name	Chemical structure of Stationary phase
SPB1	Non polar	100% Dimethyl Siloxane	
VF-5ms	Semi polar	5% Phenyl 95% methyl Siloxane	
CP-Sil 13CB	Polar	14%Phenyl 86% methyl Siloxane	

3- Reagents

1- Standard solutions of (Decyl, undecyl, Dodecyl and Tridecyl) Benzene with purity of 99.5% were supplied from Japanese TCI Company .

2-Mixture (A) of the above standard solutions has been prepared with same percentage of detergent raw material industry (table2).

Table(2) :The standard solutions of the mixture A

Wt%	Components
8.343	Decyl benzene
45.777	Undecyl benzene
33.400	Dodecyl benzene
12.480	Tridecyl benzene

3-To construct the calibration graph for each compound individually, seven solutions of each standard solution with selected percentage have been prepared.

4- 0.25µl from each solution prepared previously have been injected individually at different temperature conditions to find the elution consequences .

5-This developed method was applied to the LAB produced in ARADET and samples produced in the Syrian united company for petrochemicals industry which are analyzed previously in the Spanish Petrisa company.

6-To study the effect of storage, the LAB product from ARADET in Baiji has been stored in sealed containers for four months at different temperatures.

4-Column selection

The analysis has been carried out for one sample on the packed columns SE-30 , OV-101 and on the capillary columns of the different polarities (CP-Sili 13CB, VF-5ms and SPB-1) in different temperature conditions to choose the best column for the study.

Results and discussion

Optimum conditions

In order to find out the optimum conditions by using GLC in terms of the best column, detector and injector temperature, flow rate of the carrier gas and the optimum volume for the sample injection, many chromatographic columns have been examined including the packed columns SE-30 and OV-101. The separation efficiency using SE-30 and OV-101 columns was not sufficient, thus capillary columns of different polarities (SPB-1, VF-5ms and CP-sill 13CB) were investigated.

To find out the retention time for each isomer and its elution consequence, the four isomers were injected individually as a standard active component and as a mixture in different temperatures in the range (150-250C^0).The results in table(3) show that the best column is (VF-5ms) of good seperation efficiency with less retention time and best weight percentage recovery. Therefore, the affecting factors on the separation process such as the optimum temperature, the flow rate of the carrier gas, the optimum volume of the injected sample and the separation coefficient (α) for mixture of isomers in the LAB have been carried out on (VF-5ms) column. Table(4) show the changes for isomers mixture in different temperatures on the (VF-5ms) column while table (5) shows the separation coefficient (α) at different temperatures .

Table(3): Results of mixture (A) on three capillary columns at optimum conditions

Isomer	Theoretical weight%	SPB1 Column		VF-5ms Column		CP-Sill 13 CB Column	
		Retention time(min)	Practical (Wt%)	Retention time (min)	Practical (Wt%)	Retention time (min)	Practical (Wt%)
Decyl Benzene	8.343	19.38	8.024	13.99	8.489	12.71	8.396
Undecyl Benzene	45.777	25.99	43.243	18.63	45.726	18.11	45.570
Dodecyl Benzene	33.400	35.11	34.134	23.94	33.879	25.62	33.288
Tridecyl Benzene	12.480	48.09	11.192	30.28	12.444	36.25	12.367

Table(4): The changes for isomers mixture at different temperatures on the (VF-5ms) column.

Column temperature (C^0)	Retention time of Decyl benzene(min)	Decyl benzene (wt%)	Retention time of undecyl benzene(min)	Undecyl benzene (wt%)	Retention time of Dodecyl benzene (min)	Dodecyl benzene (wt%)	Retention time of Tridecyl benzene (min)	Tridecyl benzene (wt%)
150	15.400	8.473	23.870	45.836	36.170	33.130	54.600	12.232
180	13.990	8.469	18.630	45.726	23.940	33.079	30.280	12.444
190	6.800	8.420	9.200	45.711	12.470	33.233	17.050	12.407
200	5.610	8.465	7.240	45.684	9.440	33.179	12.460	12.324

Table(5): Seperation coefficient (α) for mixture of LAB isomers using (VF-5ms) at different column temperatures.

Mixture	Seperation coefficient / Column temperature (Cº)			
	150	180	190	200
Decyl-Undecyl Benzene	1.550	1.332	1.353	1.291
Undecyl-Dodecyl Benzene	1.515	1.285	1.355	1.304
Dodecyl-Tridecyl	1.510	1.265	1.367	1.320

Despite the fact that seperation coefficient (α) at 150C^0 is the best, but , seperation coefficient (α) at 180C^0 was chosen because the less retention time of the isomers and good percentage recovery. Flow rate for the carrier gas of 2.5ml/min was chosen since separated and sharp peaks of less retention time (table 6) and good separation coefficient (table 7) were obtained. The optimum sample volume was 0.25 µl (table 8). At this injected volume, the recovery and E$_{rel}$% are calculated (table 9). Fig.(1), shows the chromatogram of the best separation obtained for the isomers on the (VF-5ms) column.

Table(6): The changes for LAB isomers mixture using (VF-5ms) at different flow rates of the carrier gas.

Flow rate (ml/min)	Retention time of Decyl benzene(min)	Decyl benzene (wt%)	Retention time of undecyl benzene(min)	Undecyl benzene (wt%)	Retention time of Dodecyl benzene (min)	Dodecyl benzene (wt%)	Retention time of Tridecyl benzene (min)	Tridecyl benzene (wt%)
1.5	22.98	8.267	29.87	44.696	38.55	32.412	47.88	11.996
2.0	19.21	7.589	25.06	42.140	32.07	31.382	41.61	11.171
2.5	13.22	8.337	17.73	45.773	22.89	33.376	30.31	12.471
3.0	11.23	8.434	15.56	45.651	20.07	33.175	25.51	12.306
3.5	10.15	8.397	14.25	44.430	18.67	31.844	23.72	11.831

Table(7):Seperation coefficient (α) for mixture of LAB isomers using (VF-5ms) at different flow rates of the carrier gas.

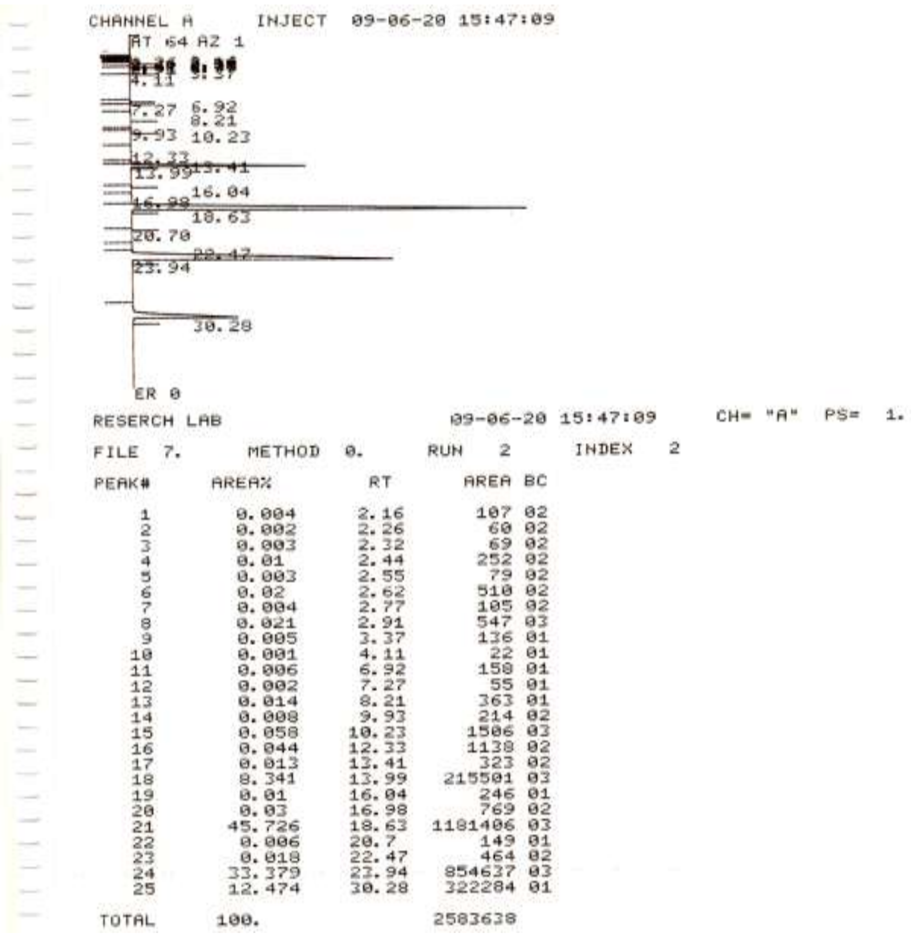
Compound	Flow rate of the carrier gas (ml/min)				
	1.5	2.0	2.5	3.0	3.5
Decyl-Undecyl Benzene	1.300	1.305	1.341	1.386	1.404
Undecyl-Dodecyl Benzene	1.291	1.280	1.291	1.282	1.310
Dodecyl-Tridecyl Benzene	1.424	1.297	1.324	1.271	1.270

Table(8):The changes for isomers mixture using (VF-5ms) at different injected sample volumes .

Volume sample µL	**Retention time of Decyl benzene(min)**	**Decyl benzene (wt%)**	**Retention time of undecyl benzene(min)**	**Undecyl benzene (wt%)**	**Retention time of Dodecyl benzene (min)**	**Dodecyl benzene (wt%)**	**Retention time of Tridecyl benzene (min)**	**Tridecyl benzene (wt%)**
0.10	14.340	8.360	18.940	45.460	24.290	33.416	31.030	12.324
0.25	13.990	8.341	18.630	45.726	23.940	33.379	30.280	12.474
0.30	14.730	8.430	19.580	45.648	25.130	33.208	31.780	12.350

Table(9):The results obtained for the isomers mixture at 0.25 µl injected sample volume on VF-5ms column.

Isomer	**Theoritical (wt%)**	**Practical (wt%)**	**Recovery %**	**Relative error percentage (E_{rel} %)**
Decyl Benzene	8.341	8.341	99.98	-0.00024
Undecyl Benzene	45.777	45.726	99.89	-0.00111
Dodecyl Benzene	33.400	33.379	99.94	-0.00063
Tridecyl Benzene	12.480	12.474	99.96	-0.00048



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    2         0.002       2.26          60 02
    3         0.003       2.32          69 02
    4         0.01        2.44         252 02
    5         0.003       2.55          79 02
    6         0.02        2.62         510 02
    7         0.004       2.77         105 02
    8         0.021       2.91         547 03
    9         0.005       3.37         136 01
   10         0.001       4.11          22 01
   11         0.006       6.92         158 01
   12         0.002       7.27          55 01
   13         0.014       8.21         363 01
   14         0.008       9.93         214 02
   15         0.058      10.23        1506 03
   16         0.044      12.33        1138 02
   17         0.013      13.41         323 02
   18         8.341      13.99      215501 03
   19         0.01       16.04         246 01
   20         0.03       16.98         769 02
   21        45.726      18.63     1181406 03
   22         0.006      20.7          149 01
   23         0.018      22.47         464 02
   24        33.379      23.94      854637 03
   25        12.474      30.28      322284 01

 TOTAL      100.                   2583638
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Fig.(1): The Chromatogram of the best separation for the isomers on the (VF-5ms) column.

Quantitative determination of LAB isomers.

After fixing the optimum conditions for the separation of LAB isomers on the VF-5ms column (table 10), standard solutions with different and known weight percentages have been prepared to construct the calibration graph for each isomer. Fig.(2) shows the calibration graph of decyl Benzene isomer alone whereas fig.(3) shows the calibration graph of decyl benzene in the mixture.

Table(10): The optimum conditions of GLC apparatus and the separation using VF-5ms column.

Sample volume (µl)	Flow rate (ml/min)	Sensitivity	Column temperature (C^0)	Detector temperature (C^0)	injection port temperature (C^0)
0.25	2.5	11	180	275	250

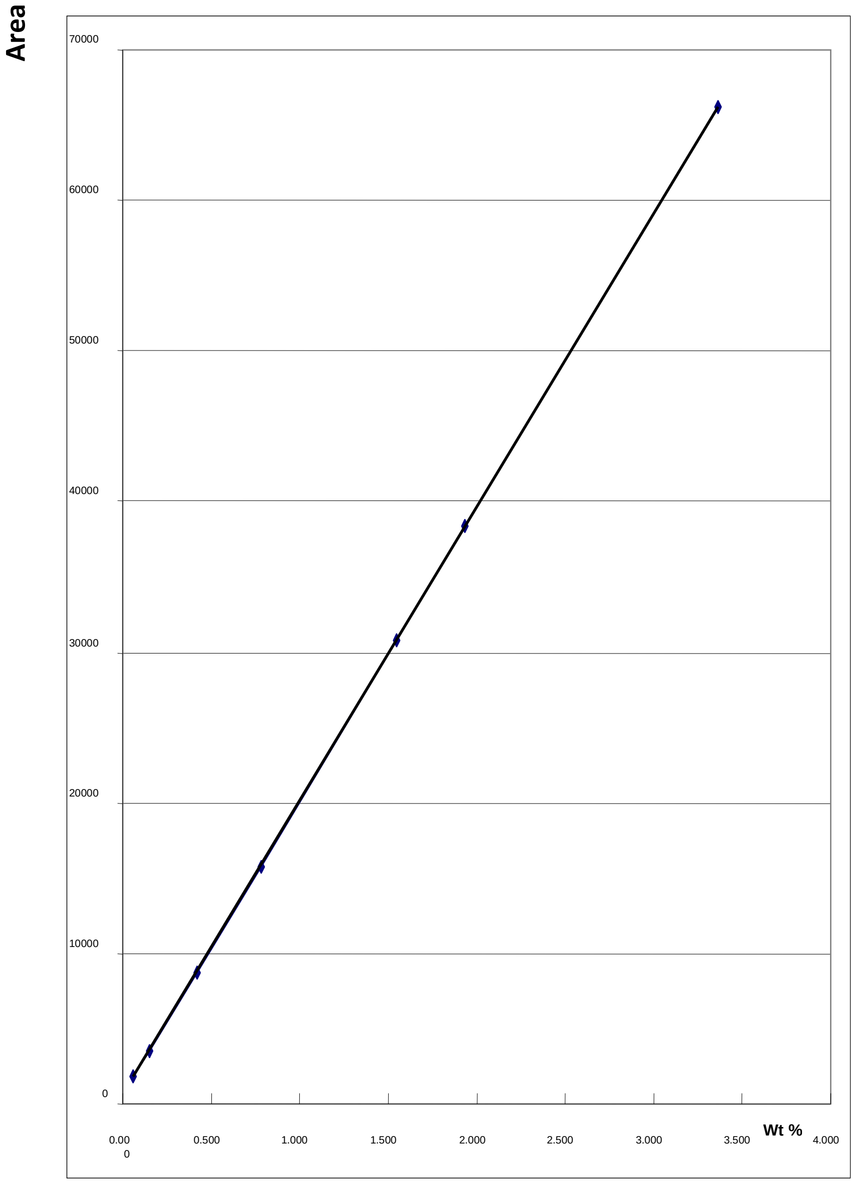

Fig.(2): The calibration graph of Decyl Benzene individually.

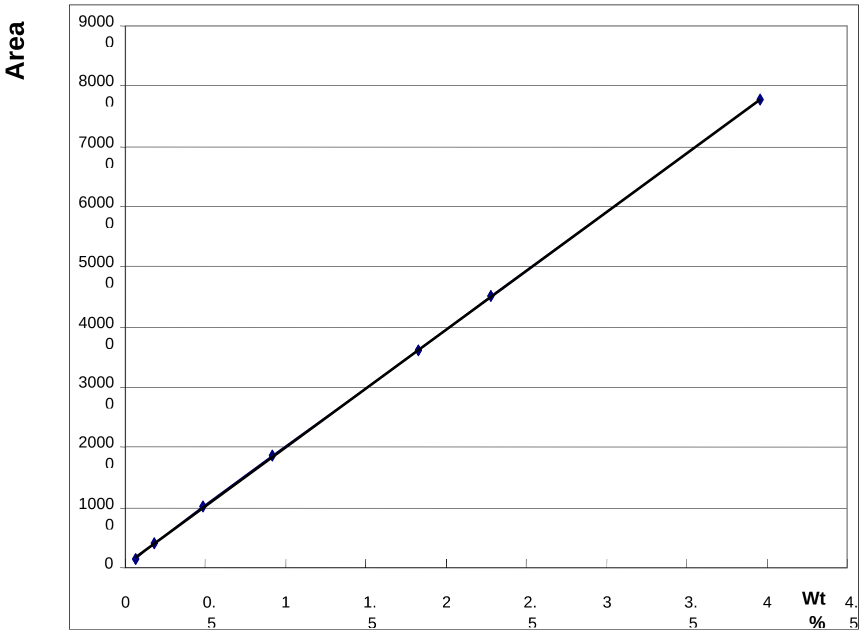

Fig.(3): The calibration graph of Decyl Benzene in the mixture.

Precision and accuracy for the determination of LAB isomers

The percentages of theoretical and calculated wt% for the standard solutions (Japanese TCI company) of purity 99.5% are shown in table (11) .

Table(11): The results of LAB isomers as standard material using VF-5ms at the optimum conditions.

Isomer	Theoretical percentage (Wt%)	Calculated percentage (Wt%)
Decyl Benzene	99.500	99.429
Undecyl Benzene	99.500	99.510
Dodecyl Benzene	99.500	99.446
Tridecyl Benzene	99.500	99.443

The calculation of recovery and relative error E_{rel}% show that the method is of good accuracy and recovery between (98-99) % and relative error percentage between (-0.072 to +0.904) % and (-0.098 to +0.826) % for the individual and mixture of isomers respectively. The calculation of RSD% shows value of not more than 0.617%. Table (12) and (13) shows the results of accuracy and precision. The results of detection limits are shown in table (14).

Table(12): Recovery%, relative error%, RSD% for samples of LAB isomers as standard material using VF-5ms column.

Isomer	Solution number	Wt% (gm)	Theoretical percentage (wt%)	Practical percentage (wt%)*	Recovery(%)*	Relative error (E_{rel}%)	Relative standard deviation (RSD%)
Decyl Benzene	1	0.4532	3.021	3.015	99.801	-0.199	0.597
	2	0.3330	2.22	2.216	99.820	-0.180	0.226
	3	0.0776	0.517	0.511	98.820	-1.161	0.587
Undecyl Benzene	1	2.7035	18.023	18.087	100.355	0.355	0.088
	2	1.5711	10.474	10.427	99.551	-0.449	0.480
	3	0.4976	3.317	3.347	100.904	0.904	0.508
Dodecyl Benzene	1	2.2946	15.297	15.308	100.072	0.072	0.059
	2	1.2222	8.148	8.103	99.448	-0.552	0.074
	3	0.3422	2.281	2.249	98.597	-1.403	0.578
Tridecyl Benzene	1	0.7497	4.998	5.008	100.200	0.200	0.100
	2	0.5625	3.750	3.774	100.640	0.640	0.159
	3	0.0987	0.66	0.648	98.480	-1.818	0.617

*Average of four determinations

Table(13): Recovery%, relative error%, RSD% for mixture samples of LAB isomers as standard material using VF-5ms column.

Isomer	Solution number	Wt% (gm)	Theoretical percentage (wt%)	Practical percentage (wt% *)	Recovery (%)*	Relative error (E_{rel}%)	Relative standard deviation (RSD%)
Decyl Benzene	1	0.3192	2.128	2.141	100.611	0.611	0.186
	2	0.3986	2.657	2.642	99.435	-0.565	0.189
	3	0.5311	3.541	3.561	100.565	0.565	0.421
Undecyl Benzene	1	0.6807	4.538	4.563	100.551	0.551	0.219
	2	0.7653	5.102	5.097	99.902	-0.098	0.137
	3	1.0575	7.05	7.089	100.553	0.553	0.367
Dodecyl Benzene	1	0.6018	4.012	3.989	99.427	-0.573	0.401
	2	0.7943	5.295	5.311	100.302	0.302	0.190
	3	1.0698	7.132	7.185	100.743	0.743	0.237
Tridecyl Benzene	1	0.2996	1.998	2.005	100.350	0.350	0.249
	2	0.3811	2.541	2.562	100.826	0.826	0.351
	3	0.5327	3.55	3.573	100.647	0.611	0.447

* Average of four determinations.

Table(14): Detection limits .

Isomer	**Minimum molar conc. (Theoritical)**	**Area(%)**	**Molar* conc. (Practical)**	**Relative standard deviation (RSD%)**	**Relative error (E_{rel}%)**	**Recovery (%)***	**Detection limit (molar)**
Decyl Benzene	0.0058	1831.2	0.00578	0.436	- 0.309	99.691	7.5×10^{-5}
Undecyl Benzene	0.0297	10305	0.02982	0.380	0.389	100.389	3.3×10^{-4}
Dodecyl Benzene	0.0204	9755.5	0.02041	0.557	0.031	100.031	3.4×10^{-4}
Tridecyl Benzene	0.0072	2574.5	0.00723	0.553	0.461	100.461	1.2×10^{-4}

*** Average of six determinations.**

Storage effect on LAB isomers at different temperatures.

LAB isomers have been stored for more than four months inside laboratory at 25C^0 outside laboratory in the range of temperature between (10-45)C^0 and in the refrigerator in the range between (-5 to -7) C^0. The comparison of results have shown that there is no effect on the LAB isomers . In addition, another sample from the LAB tank (05V28B) of the Arabic Company of detergents in Beiji in the range between (25-52)C^0 has been analyzed before and after four months of sample storage. The results obtained from storage effect study are shown in table(15) .

Table(15): Analysis results of (LAB) sample stored at different temperature conditions: 1- Inside laboratory 2- Outside laboratory 3- In the refrigerator 4- In the factory tanks.

Isomer name	Wt% before storage	Analysis results for the stored sample at different temperature conditions			
		1-) Wt%	2-) Wt%	3-)Wt%	4-)Wt%
5-Phenyl Decan	1.527	1.541	1.563	1.528	1.516
4-Phenyl Decan	1.745	1.748	1.756	1.73	1.732
3-Phenyl Decan	2.088	2.104	2.079	2.088	2.083
2-Phenyl Decan	2.225	2.238	2.17	2.25	2.267
Phenyl Decan	7.585	7.631	7.568	7.596	7.598
6-Phenyl undecan	6.987	7.102	6.917	6.947	6.945
5-Phenyl undecan	12.706	12.448	12.897	12.648	12.832
4-Phenyl undecan	9.634	9.72	9.673	9.582	9.522
3-Phenyl undecan	7.925	7.972	7.945	7.897	7.884
2-Phenyl undecan	7.586	7.559	7.594	7.598	7.591
Phenyl undecan	44.837	44.801	45.026	44.672	44.774
6-Phenyl Dodecan	8.530	8.507	8.512	8.533	8.546
5-Phenyl Dodecan	7.850	7.856	7.836	7.863	7.809
4-Phenyl Dodecan	5.696	5.628	5.672	5.768	5.709
3-Phenyl Dodecan	4.788	4.758	4.799	4.821	4.815
2-Phenyl Dodecan	4.671	4.665	4.66	4.672	4.728
Phenyl Dodecan	31.535	31.414	31.479	31.657	31.607
6-Phenyl Tridecan	4.699	4.726	4.658	4.718	4.692
5-Phenyl Tridecan	2.843	2.862	2.828	2.847	2.834
4-Phenyl Tridecan	2.014	2.022	2.008	2.02	2.005
3-Phenyl Tridecan	1.632	1.647	1.633	1.616	1.63
2-Phenyl Tridecan	1.445	1.466	1.428	1.449	1.435
Phenyl Tridecan	12.631	12.723	12.555	12.65	12.596
Total LAB	96.588	96.569	96.628	96.575	96.575

Qualitative identification for LAB Isomers using GC- MS

GC-MS apparatus is used to identify (LAB) isomers, wherein a mixture of LAB isomers with known weight percent has been injected in VF-5ms column. Fig. (4) shows the chromatogram of LAB isomers in the mixture. Table (16), shows the retention time for each isomer and their elution consequence. Fig. (5) shows the identification of each isomer in the mixture through the comparison of each isomer spectrum with those spectrum which are known and existed in (NIST) library.

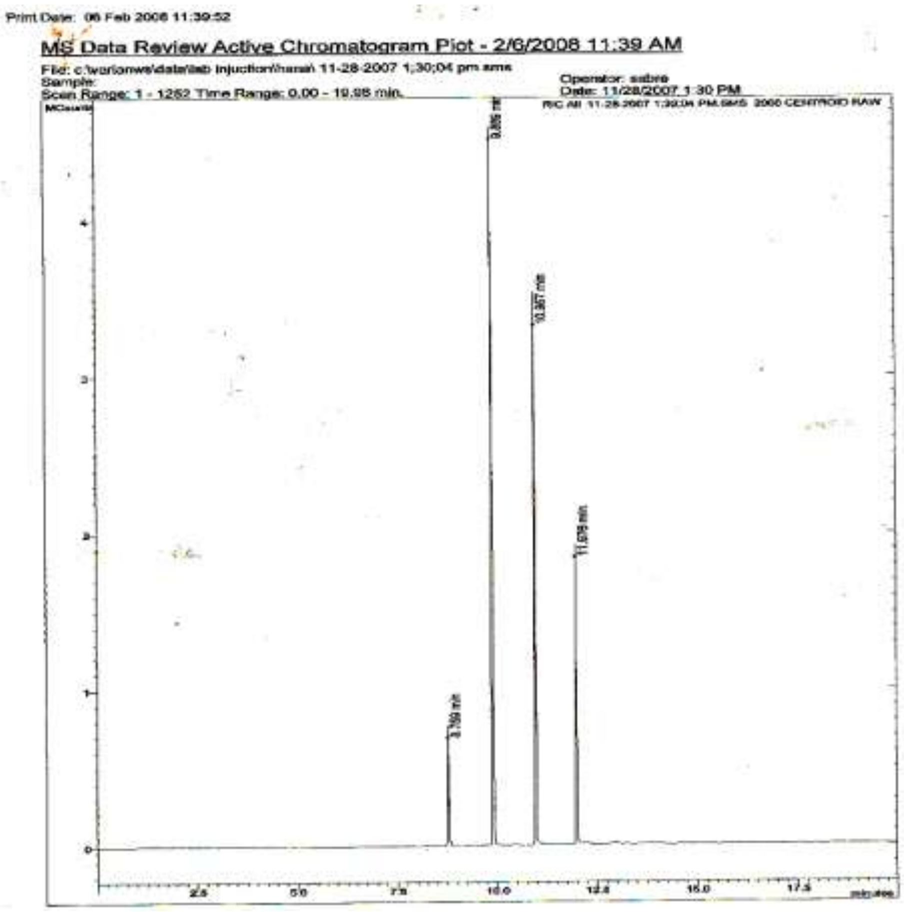

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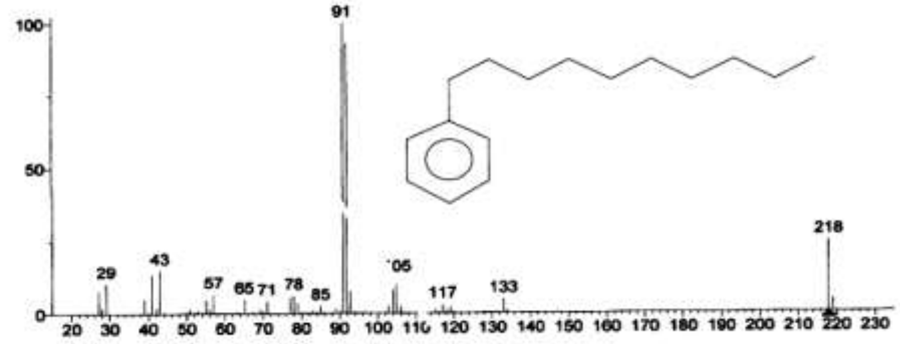
Fig.(4):GC-MS Chromatogram of the mixture of LAB isomers using VF-5ms.

Table(16): Retention times and elution consequences for each isomer in the mixture using GC-MS.

sequences	Isomer	Retention time (min)
1	Decyl Benzene	8.759
2	Undecyl Benzene	9.889
3	Dodecyl Benzene	10.957
4	Tridecyl Benzene	11.976



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(replib) Benzene, decyl-

Name: Benzene, decyl-
Formula: C16H26
MW: 218 CAS#: 104-72-3 NIST#: 27553 ID#: 11485 DB: replib
Other DBs: None
Contributor: CONTINENTAL OIL CO., PONCA CITY, OKLA, USA
10 largest peaks:
  91 999 |   92 929 |  218 243 |   43 148 |   41 133 |  105 120 | 29 103 |   27  79 |  104  79 |   93  75 |
Synonyms:
1.Decane, 1-phenyl-
2.n-Decylbenzene
3.Decylbenzene
4.1-Phenyldecane
5.n-Decyl benzene
```

Fig.(5):MS-spectrum for decyl Bemzene isomer.

It is worth noting that the injection of each isomer individually gave similar retention time to that of the mixture and they were in agreement with that in (NIST) library.

Application:

The method has been applied on standard sample from (ARADET) containing all the LAB isomers with weight percentage similar to those produced in (ARADET) company and on the LAB produced in the (United Syrian Company for petrochemicals) and the results are shown in table (17). In addition, one of the samples produced in (ARADET) using our method has been sent to the (Spanish Tepreza Company) wherein the results were correspondent to those obtained by this method using VF-5ms column as shown in table(18).

Table(17): The results for LAB sample from the (United Syrian Company for petrochemicals) using VF-5ms column.

Isomer	Syrian company analysis*	New method using VF-5ms column**	Recovery %	Relative error ($E_{rel\%}$)
5-Phenyl Decan	5.232	5.112	97.706	-2.294
4-Phenyl Decan	4.158	4.013	96.513	-3.487
3-Phenyl Decan	3.799	3.691	97.157	-2.843
2-Phenyl Decan	3.912	3.860	98.671	-1.329
Phenyl Decan	17.101	16.676		
6-Phenyl undecan	6.111	6.052	99.035	-0.965
5-Phenyl undecan	9.389	9.285	98.892	-1.108
4-Phenyl undecan	7.878	7.744	98.299	-1.701
3-Phenyl undecan	6.712	6.693	99.717	-0.283
2-Phenyl undecan	6.803	6.706	98.574	-1.426
Phenyl undecan	36.893	36.480		
6-Phenyl Dodecan	7.209	7.248	100.541	0.541
5-Phenyl Dodecan	6.603	6.713	101.666	1.666
4-Phenyl Dodecan	4.981	5.069	101.767	1.767
3-Phenyl Dodecan	4.263	4.363	102.346	2.346
2-Phenyl Dodecan	4.509	4.461	98.935	-1.065
Phenyl Dodecan	27.565	27.854		
6-Phenyl Tridecan	4.733	4.606	97.317	-2.683
5-Phenyl Tridecan	2.759	2.817	102.102	2.102
4-Phenyl Tridecan	2.032	2.077	102.215	2.215
3-Phenyl Tridecan	1.698	1.720	101.296	1.296
2-Phenyl Tridecan	1.711	1.742	101.812	1.812
Phenyl Tridecan	12.933	12.962		

*The analysis of Syrian company is considered as the theoretical percentage %.

** The average of three determinations using VF-5ms column (present method).

Table(18): The comparison of the LAB analysis using VF-5ms column with the analysis of the Spanish Petriza Company.

Isomer name	Petriza analysis *(wt%)	New method using VF-5ms column# (wt%)	Recovery %	Relative error ($E_{rel\%}$)
Decyl Benzen	11.1	11.221	101.090	1.090
Undecyl Benzen	40.2	40.632	101.075	1.075
Dodecyl Benzen	30.6	30.587	99.958	-0.042
Tridecyl Benzen	13.4	13.51	100.821	0.821

*The analysis of Petriza company is considered as the theoretical percentage.

is the average of three determination using VF-5ms (new method).

Results evaluation

The values of of recovery%, RSD% and the relative error percentage% shows that the method is of high accuracy with recovery between (98-99%) for the individual isomers and in the mixture. The relative error was not more than +0.904. The RSD% was not more than 0.5 to 0.7% for the individual isomer and in the mixture respectively. The application of the method on standard sample from the united Syrian company for Petrochemicals has been shown a recovery% between (97-101)% and relative error between (-0.283,+2.2). The comparison of the results with ASTM-D4337 for Petriza Company have shown that the results are of good similarity. The low detection limit indicates the high sensitivity of the method.

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Conclusion

The gas chromatographic method proposed here for the separation of LAB isomers using VF-5ms column shows an excellent efficiency, recovery and accuracy. The results indicate that this method can be adopted for routine analysis in the commercial companies instead of standard methods. This method is successfully applied for the determination of LAB isomers produced in different companies.

التحليل النوعي والكمي لمركبات ألكيلات البنزين المستقيمة في تركيبة المنظفات بتقنية كروماتوغرافيا الغاز – سائل

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الملخص

يتضمن هذا البحث تطوير طريقة تحليل دقيقة ومضبوطة وحساسة للتقدير الكمي لمركبات اللاب (LAB) الداخلة في تركيبة المنظفات (ديسايل، أنديسايل،دوديسايل،تراي ديسايل) بنزين بتقنية كروماتوغرافيا الغاز –سائل بإستخدام جهاز GC Varian3300 المزود بكاشف شعلة التأين باللهب FID بإستخدام عمودين معبأين (OV-101 و SE-30) مع أعمدة كروماتوغرافية شعرية مختلفة القطبية (CP-Sil 13CB و VF-5ms وSPB-1) . ومن خلال هذه الدراسة تم التوصل إلى أن العمود الشعري VF-5ms هو أفضل عمود لفصل مركبات اللاب وبظروف مثالية من درجة حرارة (العمود180مْ والكاشف 275مْ وغرفة الحقن 250مْ) وحجم النموذج المحقون 0.25 مايكروليتر وبسرعة جريان لغاز النتروجين 2.5 مل/دقيقة. و تم تشخيص مركبات اللاب نوعياً بإستخدام جهاز GC-MS(Varian 3800) وكان تسلسل خروجها (ديسايل،أنديسايل،دوديسايل،تراي ديسايل) بنزين على التوالي.

قدرت هذه المركبات كمياً بإستخدام طريقة منحني المعايرة وطريقة النسبة المئوية للمساحة تحت الحزمة Area%. وأعطت هاتين الطريقتين مدى الدقة لمركبات اللاب حيث كان مدى الخطأ النسبي يتراوح بين (0.072- إلى 0.904+%) بينما أعطى مدى التوافق من خلال حساب RSD% والتي لا تزيد عن (0.7) وبنسبة إسترجاع تتراوح بين 98.59-99.80%. كما تم حساب قيمة حد الكشف للمركبات (ديسايل، أنديسايل،دوديسايل وتراي ديسايل) بنزين وكانت 7.6×10^{-5} و 3.3×10^{-4} و 3.4×10^{-4} و 1.2×10^{-4} مولاري على التوالي.

تم تطبيق الطريقة على نماذج قياسية من الشركة العربية لكيمياويات المنظفات (ARADET) الحاوية على جميع الآيزومرات وكان مدى الخطأ النسبي ما بين (0.79- و 0.95) بينما كان الخطأ النسبي للنماذج المنتجة في هذه الشركة هو (1.7- و 1.58) . كما طبقت هذه الطريقة على النماذج المنتجة من الشركة المتحدة لصناعة البتروكيماويات السورية. وعند مقارنة نتائج هذه الطريقة مع نتائج تحاليل شركة بتريز الإسبانية لنفس النموذج، أعطت النتائج دقة تتراوح بين (0.042 و1.09−). كما تم دراسة مدى تأثير درجة الحرارة على ظروف خزن المنتوج حيث تبين بأن الحرارة ليس لها أي تأثير على مكونات اللاب.