Analysis of chlorinated, sulfochlorinated and sulfonamide derivatives of *n*-tetradecane by gas chromatography/mass spectrometry

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Abstract

The photosulfochlorination of *n*-tetradecane by sulfuryl chloride leads to a reaction mixture containing unreacted *n*-tetradecane, chloro *n*-tetradecanes and *n*-tetradecanesulfonyl chlorides. Direct and simultaneous GC analysis of the mixture of the sulfochlorinated and chlorinated isomers is followed by mass spectrometry identification of all the components either by electron impact (EI-MS) and by negative and positive chemical ionisation (NCI-MS and PCI-MS). With the goal of performing an accurate quantitative GC analysis, and as *n*-tetradecanesulfonyl chlorides prone to degrade partially into the corresponding chlorides, the former are converted to *N*,*N*-diethylsufonamides, more stable thermally, and then analysed by GC/EI-MS and GC/PCI-MS. The chloro *n*-tetradecanes, sulfonylchlorides and sulfonamides spectra present strong similarities. However, some differences between terminal and internal isomers are noticed and the peculiar behaviour of sulfonamides is emphasized.

Keywords: Alkanesulfonyl chlorides; Alkyl chlorides; Alkanesulfonamides; Positional isomers; GC/EI-MS; GC/NCI-MS; GC/PCI-MS

1. Introduction

Secondary alkanesulfonates (SASs) are biodegradable surfactants largely used as active matter in detergent formulations (especially liquid dishwashing), as emulsifying agents and even useful in flotation. Their detergent properties compare well with those of linear alkylbenzenesulfonates (LASs), whereas their aqueous solubility and ultimate biodegradation rate are slightly higher [1]. SASs are manufactured by photosulfoxidation or photosulfochlorination using gas mixture [1,2]. Recent works investigated the photosulfochlorination of single-length chain C_7–C_{12} *n*-alkanes using sulfuryl chloride (SO_2Cl_2) instead of SO_2 and Cl_2 gas mixture [3,4]. All positional isomers of chlorides and sulfonyl chlorides (four and six in each family) were analysed simultaneously by GC without derivatisation step, and identified by GC–MS [4,5]. Few detailed works have been reported on mass spectrometry of *n*-alkanesulfonyl chlorides. Indeed, the different studies are related especially to sulfonyl chlorides having alkyls chain R from C_1 to C_4, or R is aromatic (benzene, naphthalene, . . .) [6]. The technique currently used for these sulfonyl chlorides is electron impact mass spectrometry (EI-MS) [6,7]. These compounds were rather analysed by EI-MS after their transformation into sulfonate salts [8–10]. Several theoretical and thermodynamic studies highlight the formation of the SO_2^+ fragment which characterizes the mass spectra of this kind of compounds [11–13], but long-chain sulfonyl chlorides can hardly be analysed directly by GC–MS because of their instability and their low volatility, so that their analysis requires their conversion, for example, into esters or sulfonamides [14–19]. In fact, long-chain *n*-alkanesulfonyl chlorides are rather analysed by LC–MS after their transformation into sulfonates or sulfonamides [20]. In the present work, our previous studies are extended to an in-depth analysis of the *n*-tetradecane derivatives by GC–MS. These derivatives are obtained by photosulfochlorination of *n*-tetradecane at 25 °C using sulfuryl chloride and a catalyst. The reaction mixture obtained contains unreacted *n*-tetradecane, and all the positional isomers of chloro

n-tetradecane and *n*-tetradecanesulfonyl chloride. These components have been analysed by GC, and the isomeric distribution of *n*-tetradecane derivatives have been determined [4].

We present here the direct and simultaneous GC–MS analysis of chloro *n*-tetradecane and *n*-tetradecanesulfonyl chloride isomers. As there are different positional isomers, the identification of isomer one has been achieved by cross injection of samples obtained by organic synthesis and characterised by usual spectroscopic methods. EI–MS was used to identify the *n*-tetradecanesulfonylchloride, chloro *n*-tetradecane, and *N*,*N*-diethyl *n*-tetradecanesulfonamide isomers produced by the photosulfochlorination of *n*-tetradecane. Positive (PCI) and negative (NCI) chemical ionisation techniques were chosen in order to enhance the signal intensity of the molecular ion and to confirm the assignment of molecular weight, difficult to obtain by EI.

2. Experimental

2.1. Chemicals

n-Tetradecane, with a purity of 99%, sulfuryl chloride, 97% pure, pyridine, 99.8% pure, *N*,*N*-diethylamine, 99.5% pure, and diethyl ether, 99.5% pure, were purchased from Fluka (Buchs, Switzerland). 1-Chlorotetradecane (98%), and anhydrous sodium sulfate, extra pure, were purchased from Merck (Darmstadt, Germany).

2.2. Sample preparation

The reaction mixture (containing unreacted *n*-tetradecane, chloro *n*-tetradecane and *n*-tetradecanesulfonyl chlorides) was obtained by photosulfochlorination of *n*-tetradecane with sulfuryl chloride using pyridine as a catalyst [3–5]. The alkanesulfonyl chlorides produced were then derivatised to the corresponding sulfonamides with *N*,*N*-diethylamine using Berthold's method [5–17].

1-Tetradecanesulfonyl chloride was obtained from the corresponding chloride by a Grignard reaction [21]. It was further transformed into the corresponding sulfonamide. The 1-tetradecanechloride, 1-tetradecanesulfonyl chloride and 1-tetradecanesulfonamide were analysed by IR, ^{1}H and ^{13}C NMR spectroscopies.

n-$C_{14}H_{29}Cl$: b.p. (°C): 290^{760} [22]; IR (cm^{-1}): 720 (C–Cl); ^{1}H NMR (ppm): $0.8 < \delta < 0.95$ (t, 3H), $1.1 < \delta < 1.5$ (m, 22H), $1.6 < \delta < 1.8$ (q, 2H), $3.4 < \delta < 3.6$ (t, 2H); ^{13}C NMR: 45.15 (C_1), 32.73, 32.00, 28.84, 28.74, 28.56, 28.45, 28.08, 26.98 (C_2–C_{12}), 22.77 (C_{13}), 14.18 (C_{14}).

n-$C_{14}H_{29}SO_2Cl$: m.p. (°C): 48.1; IR (cm^{-1}): 1357, 1160 (–SO_2–); ^{1}H NMR (ppm): $\delta = 0.85$ (t, 3H), $1.1 < \delta < 1.6$ (m,

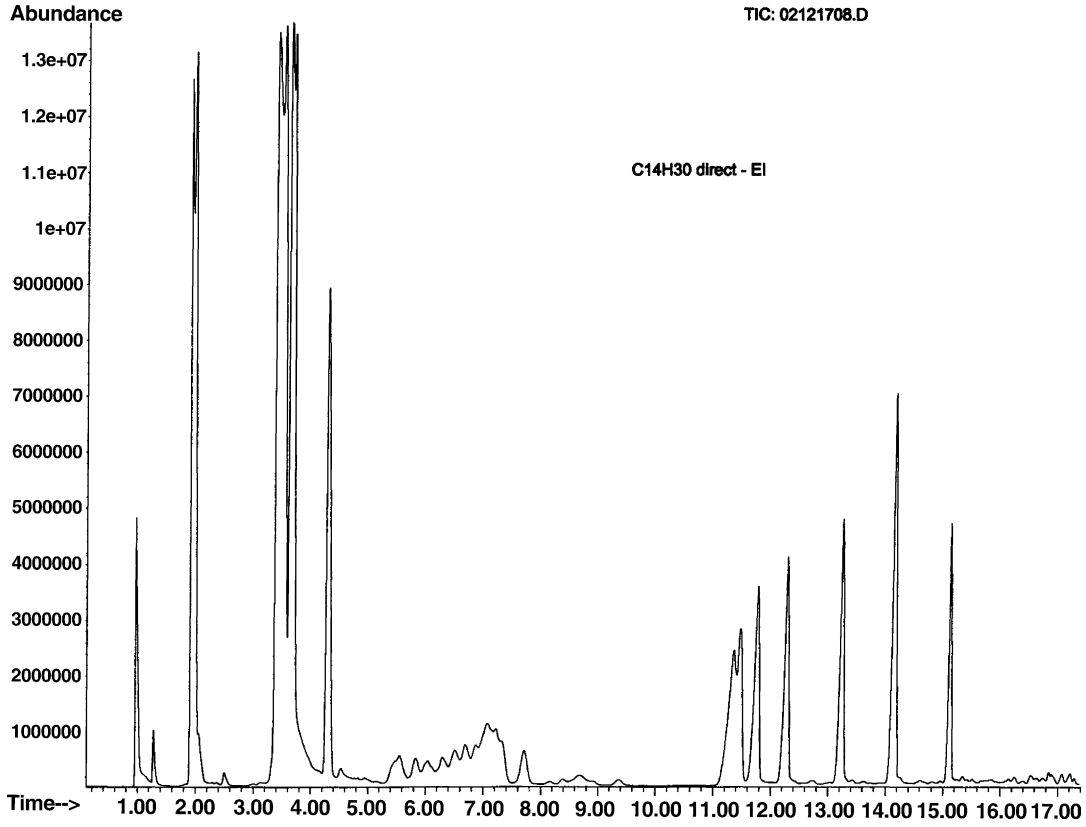


Fig. 1. Chromatogram of a reaction mixture obtained by photosulfochlorination of *n*-tetradecane with sulfuryl chloride at 25 °C.

22H), $\delta = 2.02$ (q, 2H), $\delta = 3.63$ (t, 2H); ^{13}C NMR: 65.54 (C_1) 31.97, 29.69, 29.49, 29.40, 29.22, 28.94, 27.62, 24.33 (C_3–C_{12}), 22.74 (C_{13}), 14.17 (C_{14}).

n-$C_{14}H_{29}SO_2NEt_2$: m.p. (°C): 37; IR (cm^{-1}): 1330, 1140 (C–SO_2–N); ^{1}H NMR (ppm): $\delta = 0.8$ (t, 3H), $1 < \delta < 2.2$ (m, 29H), $\delta = 2.87$ (t, 2H), $3.1 < \delta < 3.4$ (q, 4H); ^{13}C NMR: 52.42 (C_1), 41.53 (C_{16}–C_{17}) 31.97, 29.70, 29.57, 29.56, 29.17, 28.52, 23.51, 22.74 (C_2–C_{12}), 14.54 (C_3) 14.18 (C_{14}).

2.3. Procedures

2.3.1. Gas chromatography GC/FID

GC separations were performed with a Hewlett-Packard Model 5730 A gas chromatograph. An ULTRA 2, a poly (5% phenyl/95% methylsiloxane) capillary column 25 m × 0.20 mm I.D., 0.33 μm film thickness (Hewlett-Packard) was used for the analysis with He carrier gas (0.6 ml/s).

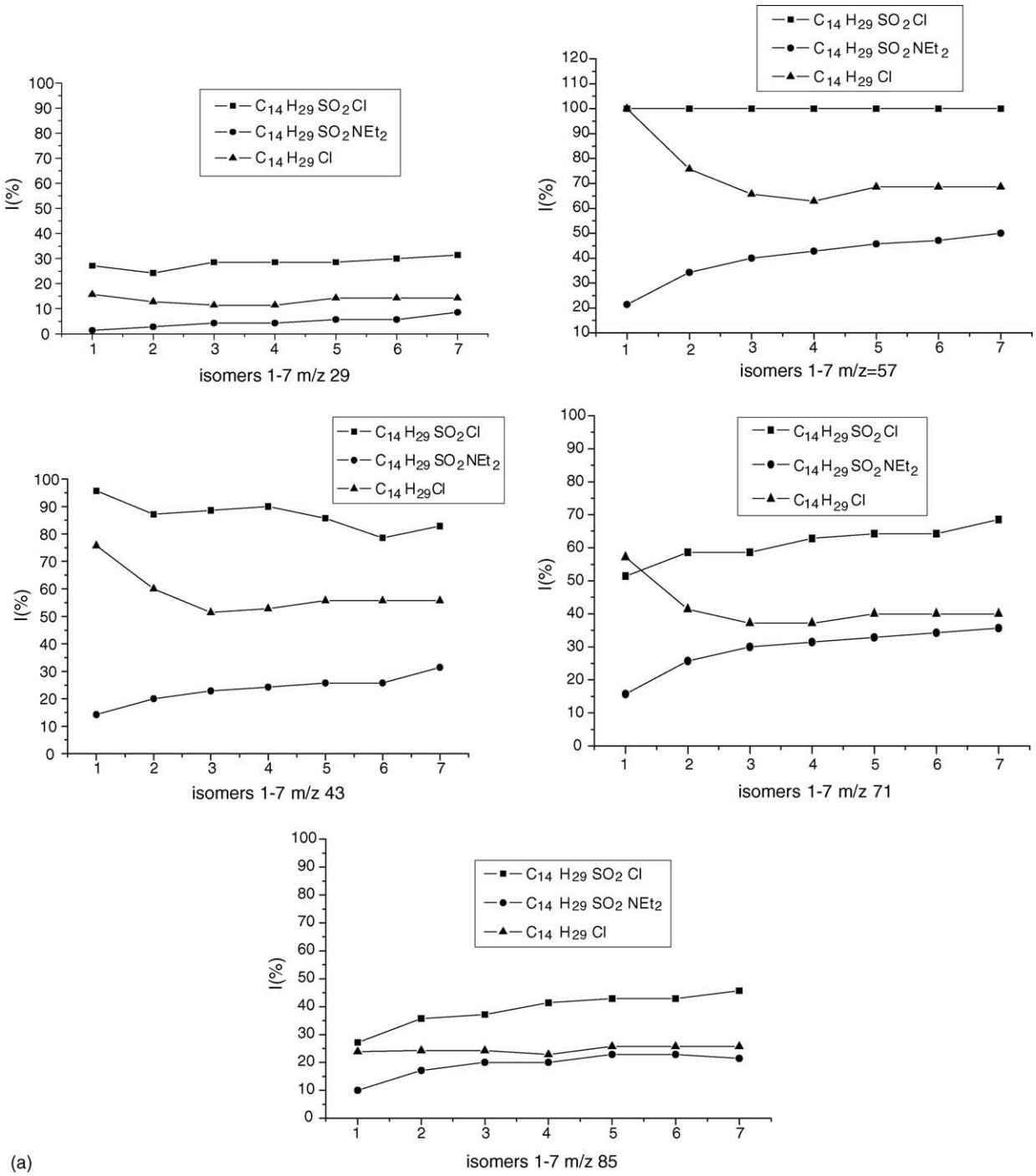
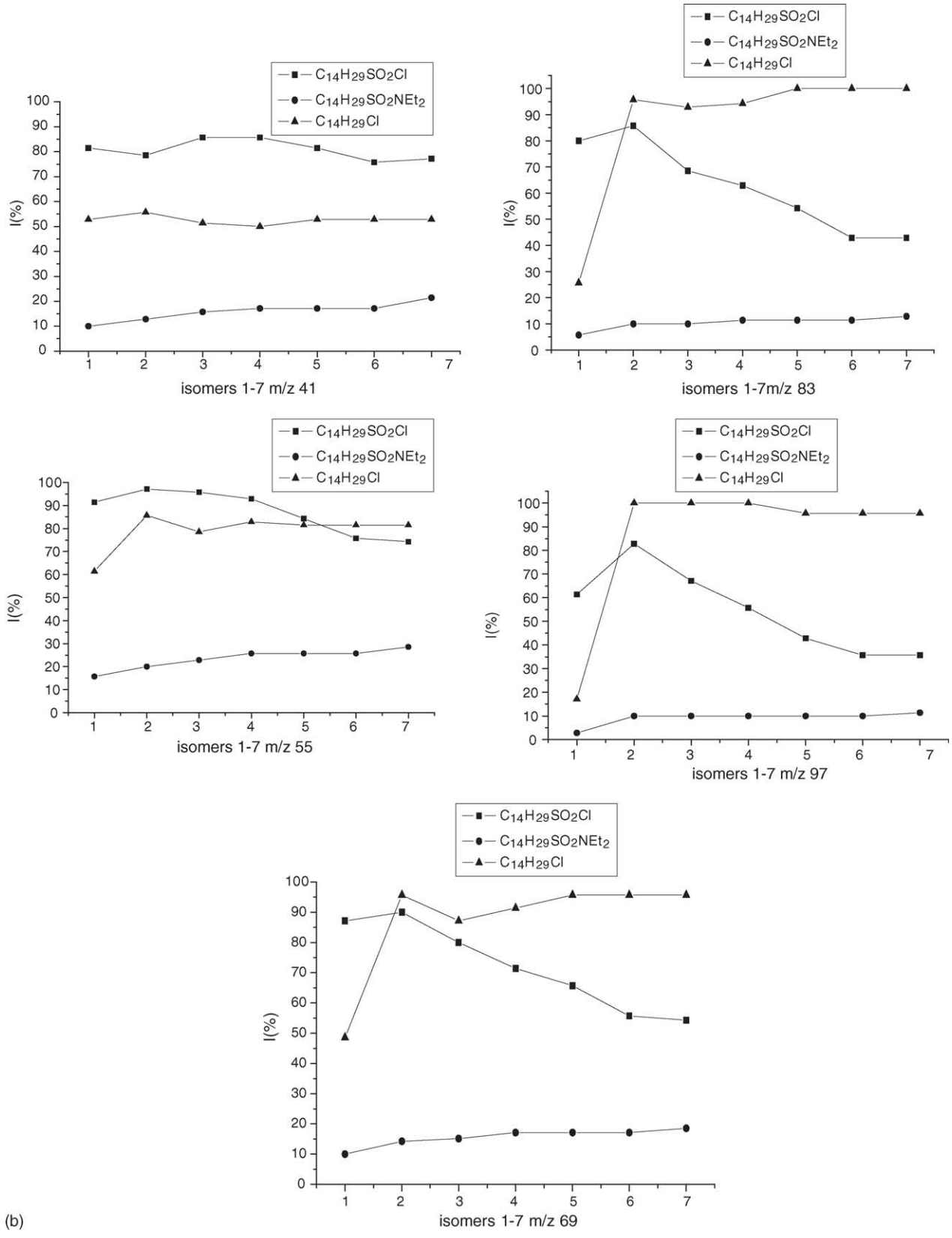


Fig. 2. (a) EI-MS analysis of chloro *n*-tetradecane, *n*-tetradecanesulfonyl chloride and *n*-tetradecanesulfonamide isomers: intensities of C_nH_{2n+1} fragments. (b) EI-MS analysis: intensities of the C_nH_{2n-1} fragments.



(b)

Fig. 2. (*Continued*.)

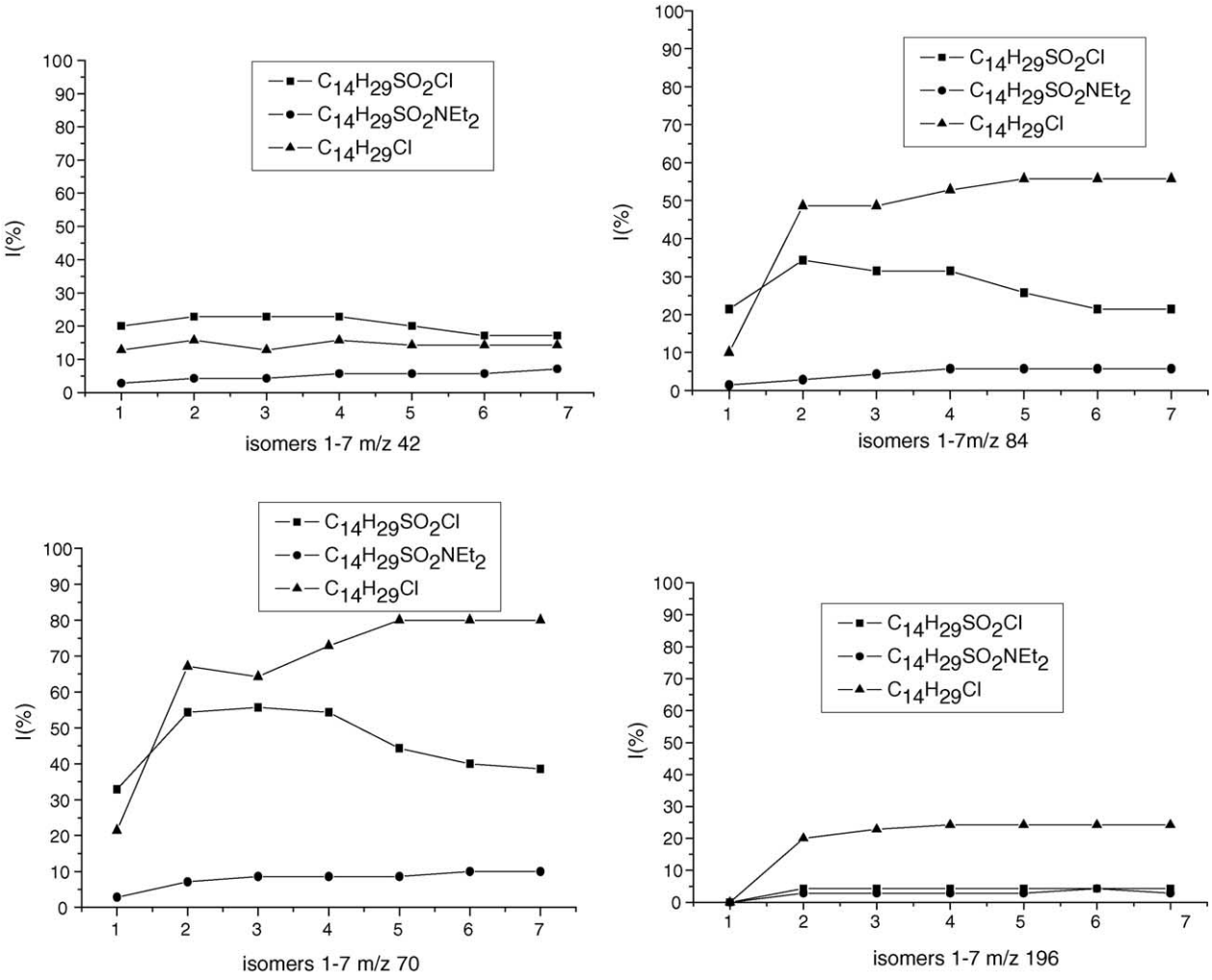


Fig. 3. EI-MS analysis: intensities of the C_nH_{2n} fragments.

Direct analysis of the reaction mixture was carried out with the following temperature program: initial temperature 180 °C for chlorides, and the temperature was increased at a rate of 8°/min up to 250 °C for sulfonyl chlorides. The GC analysis of derivatised reaction mixtures started at an initial temperature of 180 °C for the chlorides, the temperature was increased up to 270 °C at a rate of 8 °C/min, then held at 270 °C for all the sulfonamide isomers. Injector and FID temperatures were 300 °C. One microliter samples were injected into the column.

As the analysis of *n*-tetradecanesulfonyl chlorides was done at high temperature, it was necessary to verify the stability of these compounds during the analysis. For this purpose, the analysis 1-tetradecanesulfonyl chloride was carried out by gas chromatography. The two peaks obtained were analysed by GC/EI-MS. They correspond to 1-tetradecanesulfonyl chloride and 1-chlorotetradecane. So, in order to perform a quantitative analysis, it was necessary to convert *n*-tetradecanesulfonyl chlorides into thermally more stable sulfonamides.

2.3.2. Gas chromatography/mass spectrometry

The gas chromatograph, a Hewlett-Packard Model 6890 was coupled to a Hewlett-Packard Model 5973 mass spectrometer. The column used was HP5 MS, a poly (5% diphenyl/95% dimethylsiloxane) capillary column 30 m × 0.25 mm I.D., 0.25 μm film thickness (Hewlett-Packard), similar to ULTRA 2 phase. The *n*-tetradecane derivatives can be detected either by EI (70 eV) or by chemical ionization (CI) techniques. The ion source was run under

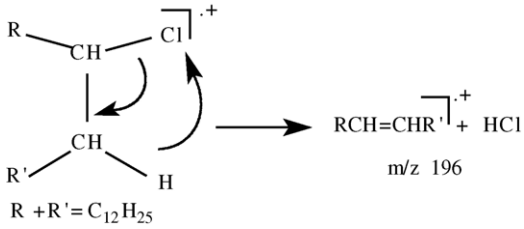


Fig. 4. 1,4 rearrangement of chloro *n*-tetradecanes.

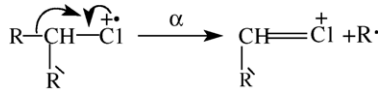


Fig. 5. α-Cleavage of C–Cl bound in chloro *n*-tetradecanes.

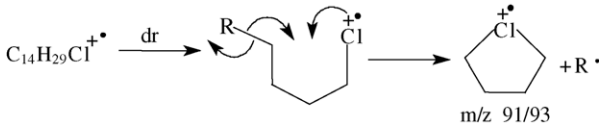


Fig. 6. Displacement reaction of chloro *n*-tetradecane.

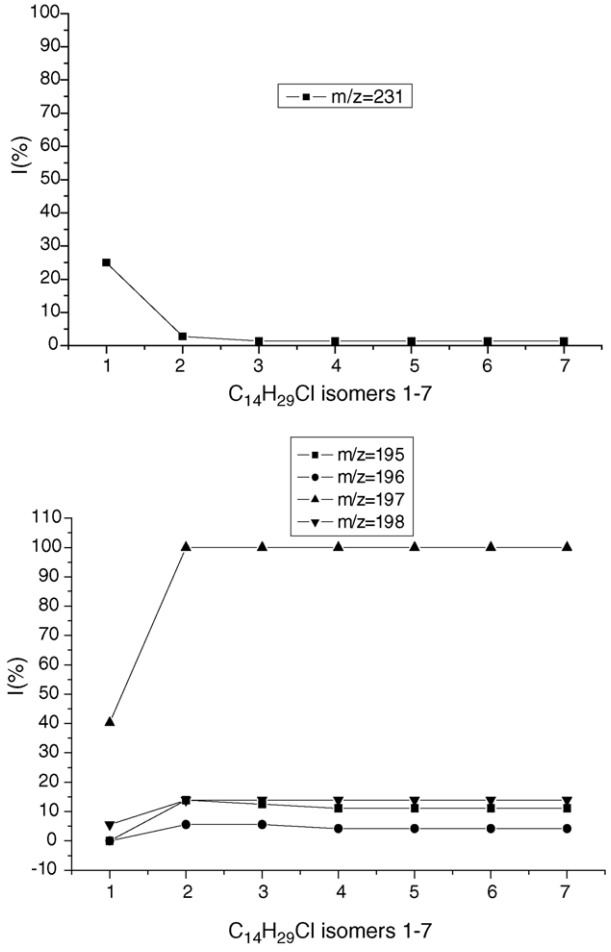


Fig. 7. GC/PCI-MS analysis: intensities of fragments at *m*/z 213, 195, 196, 197, and 198.

positive (PCI) or negative (NCI) conditions using methane as the reagent gas.

3. Results and discussion

3.1. Direct GC analysis of the reaction mixture

The direct GC analysis of the reaction mixture of the photosulfochlorination of *n*-tetradecane leads to the chromatogram presented in Fig. 1. It reveals the presence of

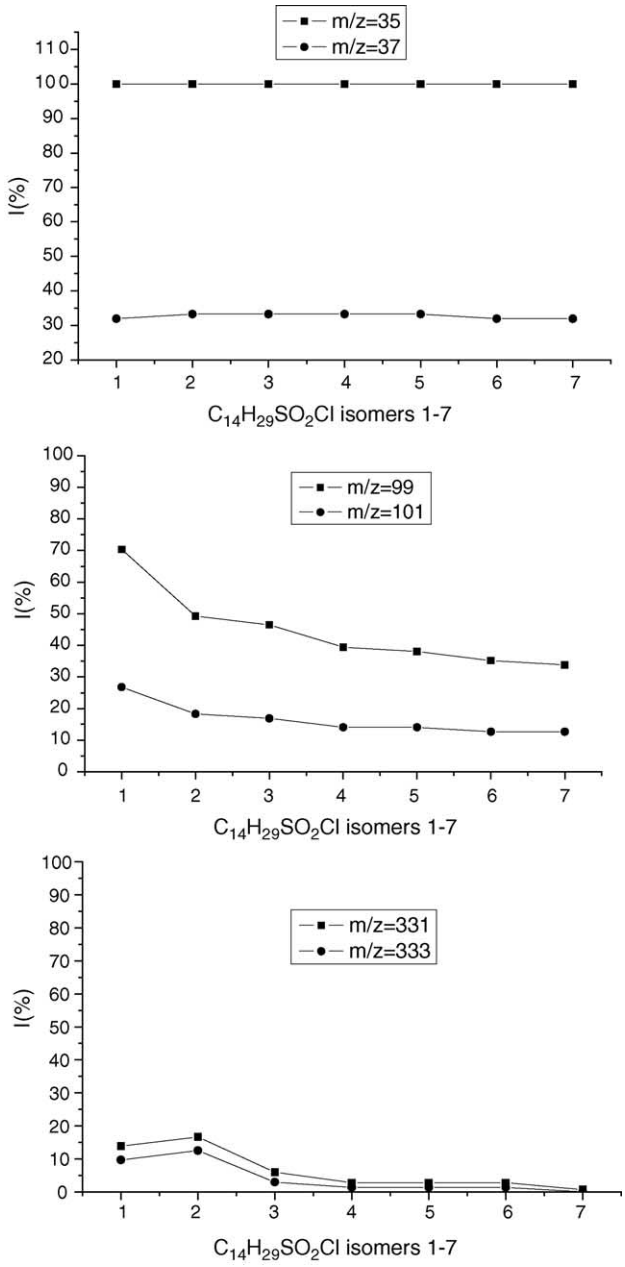


Fig. 8. GC/NCI-MS analysis of *n*-tetradecanesulphonyl chloride isomers: intensities of the peaks of Cl^-, SO_2Cl^- and $C_{14}H_{29}SO_2Cl^+Cl^-$.

two groups of peaks after that of the solvent and that of unreacted *n*-tetradecane at 2 min. The first group with retention time 3.1–4.4 min corresponds to chlorinated isomers. The second one at 11–15 min is composed of seven well-separated peaks corresponding to the seven *n*-tetradecanesulfonyl chloride positional isomers.

To complete this GC analysis, GC–MS analysis have been achieved with different techniques (EI, PCI, NCI). They were carried out in order to identify the various isomers of sulfochlorinated and chlorinated tetradecane. The mass spectrum of unreacted *n*-tetradecane was identified by GC/EI-MS and compared well with that obtained in the literature [23,24].

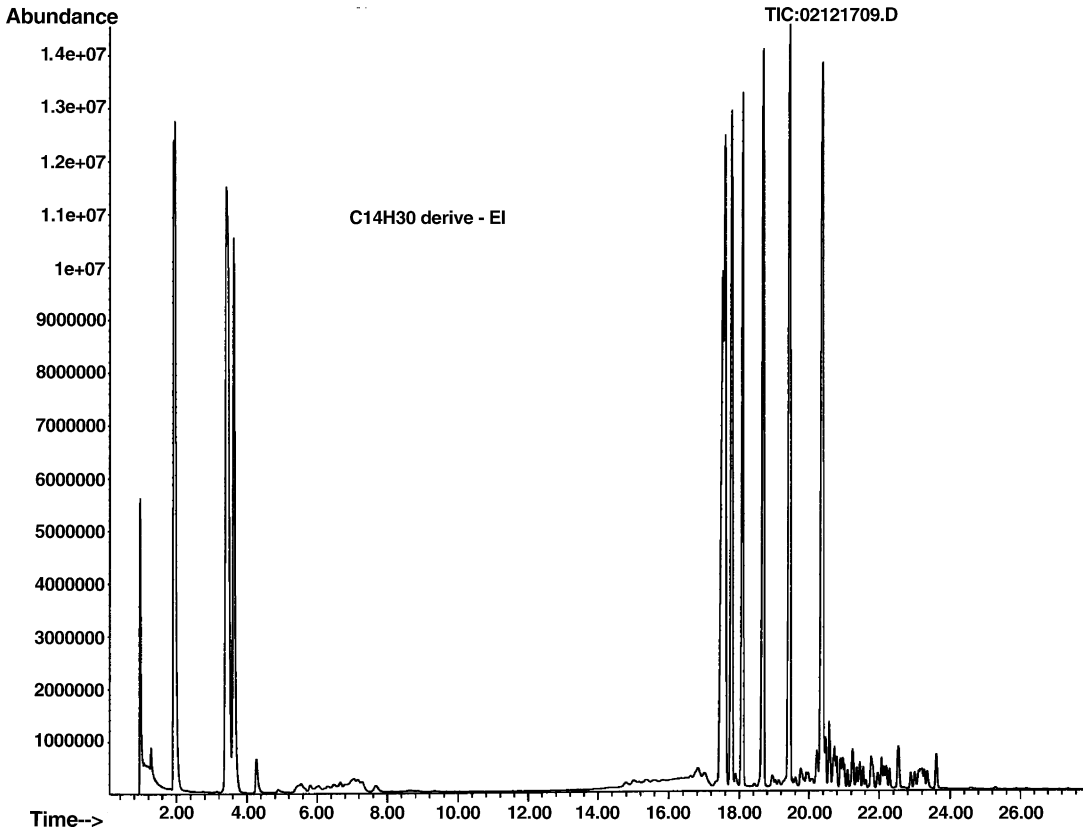


Fig. 9. Chromatogram of a reaction mixture obtained from photosulfochlorination of *n*-tetradecane with sulfuryl chloride at 25 °C after the transformation of sulfonyl chlorides to sulfonamides using *N*,*N*-diethylamine.

3.2. GC/EI-MS and GC/PCI-MS analysis of chloro n-tetradecanes

The GC analysis of chloro *n*-tetradecanes gives five instead of seven peaks, as the isomers five, six, and seven are not well separated. The last one at 4.2 min is that of the primary isomer, which is well separated. However, we have recorded the mass spectra corresponding to the successive poorly resolved peaks in the region 3.1–4 min, which give similar spectra, with fragmentations comparing well with those of the primary isomer and with those found in the literature. As expected, the molecular ion peak is absent for all the isomers. However, we note some differences between the spectra of the secondary isomers and that of the primary one.

The fragments characterizing the linear alkyl chain, as those at *m*/*z* 29, 41, 43, 55, 57, 69, 71, and 85 are present for all isomers with approximately the same intensities, respectively. For the primary isomer, the relative intensities are always quite different from that of the secondary isomers (Fig. 2). Besides, the mass spectrum of 1-chloro *n*-tetradecane compares well with that reported in the literature [25,26].

We notice that the fragment at *m*/*z* 196 is present for all secondary isomers (Fig. 3). This fragment results from 1,4 rearrangement [26] with loss of HCl as presented in Fig. 4.

The absence of this fragment in the spectrum of the primary isomer is probably due to the fact that the primary carbenium ion is less stable than the secondary one, for which the abundance is almost the same (Fig. 3). It can also be due to the competition of the loss of HCl by 1,4 rearrangement with the tendency to form cyclic divalent chloronium ions as shown later.

Several peaks observed in the spectra of chloro *n*-tetradecanes, such as the fragments at *m*/*z* 42, 56, 70, 84, 98, 112, 126, 140, 154, and 168 correspond to the $C_nH_{2n}^{\bullet+}$ formula. They can be produced from the fragment of *m*/*z* 196 (Fig. 3). We also found other fragments characterizing chloro *n*-tetradecanes, as the peaks at *m*/*z* 49/51, 63/65, 77/79, 91/93, 105/107, 119/121, 133/135, 147/149, and 161/163. All these fragments result from the α-cleavage [26,27] as presented in Fig. 5.

The peak at *m*/*z* 49/51 is present only for the primary isomer, with a low intensity: it thus corresponds to $n = 0$ ($R' = H$). In the same context, the peaks at *m*/*z* 91/93 and 105/107 are very intense and provide a strong identification of the primary isomer, as already reported in the literature [12,15,16,20,22]. It can be explained by the fact that, in this isomer, these fragments correspond to five- and six-membered cyclic ions formed by a displacement reaction giving divalent chloronium ions (Fig. 6).

Since by EI the parent peaks do not appear, positive chemical ionization was used to enhance its signal intensity. This technique leads to spectra containing the peak at *m*/*z* 231,

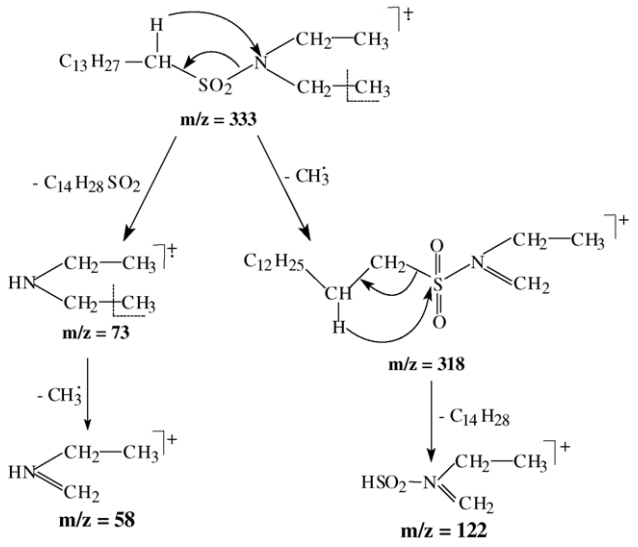


Fig. 10. EI fragmentation of primary *N*,*N*-diethyl *n*-tetradecanesulfonamide.

corresponding to $[M-H]^+$, the quasi-molecular ion for all isomers, even if its abundance is very low for the secondary isomers (Fig. 7). Thus, this allows to confirm the molecular weight.

The peaks at *m*/*z* 195, 196, 197, and 198, corresponding respectively to the $[M-H_2Cl]^+$, $[M-HCl]^+$, $[M-Cl]^+$ and $[M-Cl+H]^+$ fragments, are present for all isomers, but with greater relative intensities for the secondary isomers, and characterize probably the fragments $[C_{14}H_{28}]^+$, $[C_{14}H_{29}]^+$ and $[C_{14}H_{30}]^+$ as shown in Fig. 7.

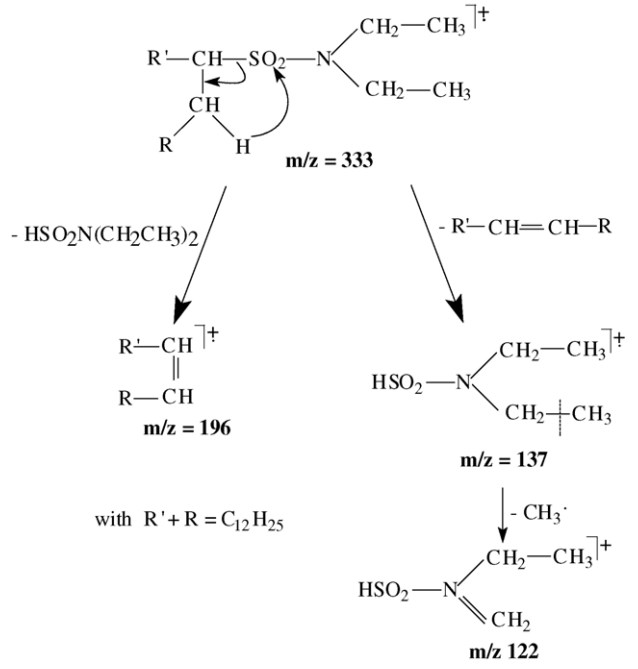


Fig. 11. EI fragmentation of secondary *N*,*N*-diethyl *n*-tetradecanesulfonamides.

3.3. GC/EI-MS, GC/PCI-MS and GC/NCI-MS analysis of n-tetradecanesulfonyl chlorides

As the *n*-tetradecanesulfonyl chlorides are well separated by GC, the analysis is easier than that of the corresponding chlorides. The EI spectra of *n*-tetradecanesulfonyl chlorides show peaks at *m*/*z* 29, 43, 57, ... which characterize the linear hydrocarbon chain, but their intensities are higher in comparison with those of *n*-tetradecane chlorides (Fig. 2). Other fragments characteristic of the presence of a function in the chain are also noticed, as *m*/*z* 196, which correponds to the 1,4 rearrangement of *n*-tetradecanesulfonyl chlorides with loss of HSO_2Cl, in the same way as HCl in Fig. 4. But the most characteristic peaks in the spectra of these compounds although of low intensity, are those at *m*/*z* 64 and 65 present in the spectra of all isomers, assigned to the fragments $[SO_2]^+$ and $[SO_2H]^+$, common with sulfonyl chlorides [6]. As in chloro *n*-tetradecane spectra, peaks at *m*/*z* 42, 56, 70, 84, and 98, first indicating the loss of HX, then the production of $C_nH_{2n}^{\bullet+}$ fragments are seen, but they are less abundant than those of chloro *n*-tetradecanes except for *m*/*z* 42 (Fig. 3).

To complete the EI analysis, PCI allows to observe the peak of the quasi-molecular ion at *m*/*z* 297 corresponding to the $[M+H]^+$ adduct, but only for the primary isomer. Other characteristic peaks, at *m*/*z* 65 and 229, correspond to the $[SO_2H]^+$ and $[C_{14}H_{29}S]^+$ fragments, respectively.

Finally, NCI was found necessary to complete previous analysis. It gives very simple spectra with fewer fragmentations and peaks with higher abundances. The most intense peaks in the NCI-MS spectra of *n*-tetradecanesulfonyl chlorides are at *m*/*z* 35/37 corresponding to the Cl^- ion, *m*/*z* 64 corresponding to the $SO_2{}^-$ fragment, *m*/*z* 99/101 corresponding to the $[SO_2Cl]^-$ fragment and *m*/*z* 331/333 corresponding to the $M+Cl^-$ adduct, the quasi-molecular ion. These fragments, present for all isomers (Fig. 8), clearly indicate the presence of the chlorosulfonyl group and the predictable, above mentioned adduct confirms the assignment of the molecular weight.

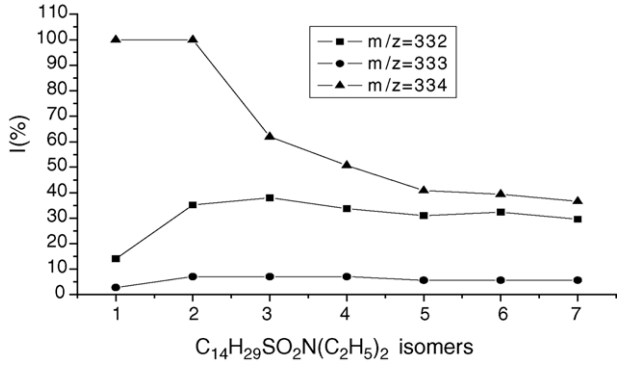


Fig. 12. GC/PCI-MS analysis of *n*-tetradecanesulphonamide isomers: intensity of the $[M-H]^+$, $[M]^+$ and $[M+H]^+$ peaks.

3.4. GC/EI-MS and GC/PCI-MS analysis of N,N-diethyl n-tetradecanesulfonamides

Like sulfonyl chlorides, the corresponding sulfonamides give seven well resolved peaks (Fig. 9). Their analysis by EI gives spectra with peaks which reveal the linear nature of the alkyl chain (*m*/*z* 29, 43, 57, ...) for all isomers, as observed for chlorides and sulfonylchlorides but with lower intensities (Fig. 2). Therefore, the nature of the functional group affects the abundance of the fragments, in agreement with McLafferty's results on *n*-dodecane derivatives [26].

As expected, the molecular ion is not observed, and the peak at *m*/*z* 318 representing the $[M-CH_3]^+$ fragment, is present only for the primary isomer (Fig. 10). This is probably due to the fact that secondary isomers undergo 1,4 rearrangement (Fig. 11). As with chlorides and sulfonyl chlorides, the peak at *m*/*z* 196 corresponds to $[M-HSO_2NEt_2]^+$ with loss of HX and is present for all the secondary isomers, with very low intensity (Fig. 3). Some other peaks (at *m*/*z* 43, 58, 73, 122, and 137) characterize the sulfonamide group specifically (Figs. 10 and 11). Once more, peaks at *m*/*z* 42, 70, 84, and 98 corresponding to $C_nH_{2n}{}^{\bullet+}$ are present, but their intensities are less important when compared to those of chlorides and sulfonyl chlorides derivatives (Fig. 3).

By PCI analysis, the molecular ion (*m*/*z* 333) appears for all the isomers but with very low abundance. The peaks at *m*/*z* 332 and 334 corresponding to $[M-H]^+$ and $[M+H]^+$ adducts, present for all the isomers, are more intense than often reported in the literature for PCI spectra [28] (Fig. 12). Let us underline here the big difference in the behaviour of 1- and secondary-isomers as regards the abundance of the $[M+H]^+$ adduct, which is probably related to the external position of the functional group. This is consistent with the plot of *m*/*z* 196 (Fig. 3). Indeed, the primary isomer does not undergo the 1,4 rearrangement with loss of HX; so, in most case, it leads preferably to the molecular ion or one of its adducts, whereas all the secondary isomers, leading to the $[M-HX]^+$ fragment (*m*/*z* 196), show less tendency to form a molecular ion.

The spectra also reveal the presence of the $[SO_2H]^+$ and $[M-CH_3]^+$ fragments (*m*/*z* 65 and 318, respectively). Some other peaks, like those at *m*/*z* 72, and 120, can be assigned to adducts formed from the sulfonamide function $[N(CH_2CH_3)_2]^+$ and $[SO_2N(CH_3)(CH_2=CH_2)]^+$.

4. Conclusion

The complete identification of some *n*-tetradecane derivatives (chlorides, sulfonyl chlorides and *N*,*N*-diethylsulfonamides) has been achieved by direct GC/EI-MS, GC/PCI-MS and GC/NCI-MS analysis. EI and PCI analysis were useful to identify the *n*-tetradecanesulfonamides and chloro *n*-tetradecanes completely. Negative ionisation was necessary to complete the results related to *n*-tetradecanesulfonyl chlorides. In general, the mass spectra of *n*-tetradecane derivatives exhibit a great similarity in EI-MS, except for a few fragments typical of sulfonamides. The parent peak does not appear in EI spectra, but all the secondary isomers exhibit a peak at *m*/*z* 196 corresponding to the $[M-HX]^+$ fragment obtained probably by 1,4 rearrangement. The nature of the functional group has a significant influence on the abundance of the common fragments, the lowest intensities being observed for sulfonamides.

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