Survey of chemical compounds in consumer products

Survey no. 8 – 2002

Contents of selected fragrance materials in cleaning products and other consumer products

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Summary

EU Scientific Committee on Cosmetic products and Non-Food Products intended for consumers has identified 24 fragrance chemicals as potential contact allergens: amyl cinnamal, amyl cinnamyl alcohol, benzyl alcohol, benzyl salicylate, cinnamyl alcohol, cinnamal, citral, coumarin, eugenol, geraniol, hydroxycitronellal, Lyral®, isoeugenol, anisyl alcohol, benzyl benzoate, benzyl cinnamate, citronellol, farnesol, hexyl cinnamaldehyde, Lilial®, d-limonene, linalool, methyl heptine carbonate and γ-methylionone.

The content of these fragrance chemicals has been determined in 43 consumer products in the present investigation by the Danish Environmental Protection Agency and the National Environmental Research Institute. The products analysed were mainly dishwash, laundry detergents, and hard and soft surface cleaners.

Of the investigated products, 93% (n=40) were found to contain from to nine of the target fragrance substances, and none of the target fragrances were detected in three products. The concentration of various target fragrances in the products varied from < 0.0001% to 0.7600%.

1 Introduction

Fragrances are commonly used in the formulations of consumer products. The purpose is to give the consumer a feeling of well being. Sometimes, fragrances are also used to mask the odour of other chemical ingredients in a product. About 3000 chemical substances and essential oils are used in the formulation of perfumes. A perfume typically contains 10-300 (or more) fragrance materials.

An increasing frequency of allergic contact dermatitis, associated with the use of perfumed products, in the European population has been reported in the last two decades. The EU Scientific Committee on Cosmetic Products and Non-Food Products intended for Consumers (SCCNFP) has recently reviewed the problem of fragrance allergy in consumers (1). SCCNFP has identified 24 potential fragrance allergens (Table 1). Contact allergy to 13 of these fragrance materials (No.1-13, Table 1) have been reported more frequently than for the remaining eleven fragrance materials. Following the Opinion of SCCNFP (1), the EU Commission has proposed regulation of fragrance chemicals in cosmetic products. However, no initiative has yet been taken to regulate fragrance chemicals in cleaning products and other consumer products. The reason for this may be that there is not sufficient knowledge about exposure estimates. Present study was initiated with the aim to assess the fragrance exposure of general population by the use of cleaning products as well as some other selected consumer products. The contents of SCCNFP identified 24 fragrance allergens (Table 1) have been determined in a selection of products to which Danish population is commonly exposed.

Table 1.1
Fragrance materials analysed

Fragrance No.	Fragrance material	CAS Reg. No.	Chemical name
1	Amyl cinnamal	122-40-7	2-(Phenylmethylene) -1-heptanal
2	Amylcinnamyl alcohol	101-85-9	2-(Phenylmethylene) -1-heptanol
3	Benzyl alcohol	100-51-6	Benzenemethanol
4	Benzyl salicylate	118-58-1	2-Hydroxybenzoic acid, benzyl ester
5	Cinnamyl alcohol	104-54-1	3-Phenyl-2-propen-1-ol
6	Cinnamal	104-55-2	3-Phenyl-2-propen-1-al
7	Citral	5392-40-5	3,7-Dimethyl- 2,6-octadienal
8	Coumarin	91-64-5	2H-1-Benzopyran-2-one
9	Eugenol	97-53-0	2-Methoxy-4-(2-propenyl)phenol
10	Geraniol	106-24-1	3,7-Dimethyl 2,6-octadien-1-ol
11	Hydroxycitronellal	107-75-5	3,7-Dimethyl-7-hydroxy octanal
12	Lyral®	31906-04-4	4-(4-Hydroxy-4-methylpentyl)-3- cyclo-hexene carboxaldehyde
13	Isoeugenol	97-54-1	2-Methoxy-4-(1-propenyl)phenol
14	Anisyl alcohol	105-13-5	4-Methoxybenzyl alcohol
15	Benzyl benzoate	120-51-4	Benzoic acid phenylmethyl ester
16	Benzyl cinnamate	103-41-3	Benzyl 3-phenyl-2-propenoate
17	Citronellol	106-22-9	3,7-Dimethyl-6-octenol
18	Farnesol	4602-84-0	3,7,11-Trimethyldodeca-2,6,10-trienol
19	Hexyl cinnamaldehyde	101-86-0	2-(Phenylmethylene)-1-octanal
20	Lilial®	80-54-6	2-Methyl-3-(4-tert-butylbenzyl) propanal
21	d-Limonene*	5989-27-5	(R)-p-Mentha-1,8-diene
22	Linalool	78-70-6	3,7-Dimethyl-1,6-octadien-3-ol
23	Methyl heptine carbonate	111-12-6	Methyl 2-octynoate
24	γ-Methylionone	127-51-5	3-Methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one

* Content of dl-limonene in the products has been determined in the present investigation

2 Products

The content of target fragrances was analysed in 43 products purchased in the detail outlets, in the period July-September 2001. The products selected were mainly those that are commonly used by the general population: dishwash, fabric detergents and hard and soft surface cleaners. The selection criteria further included 'perfume' labelling on the product, and a possible significant skin exposure by the product. On the basis of these criteria some speciality products were also analysed, although their use might be limited.
The analysed products comprised:
fifteen fabric detergent,
twelve hard and soft surface cleaners,
six dishwash,
two car shampoo,
two panties,
two toilet paper,
one hand cleaner,
one nappy,
one eraser (box with 50 erasers)
and one doll.

Table 2.1
Products analysed.

NERI Reg. Nr.	Product description
01-0828	Panties with strawberry fragrance, white
01-0829	Panties with lemon fragrance, black
01-0830	Nappy ("perfume" not indicated in the ingredient list)
01-0831	Fragrance toilet paper, orange
01-0832	Fragrance toilet paper, yellow
01-0833	Soft soap, coconut
01-0834	Liquid soft soap
01-0835	Liquid soft soap for wood
01-0836	Surface cleaner
01-0837	Surface cleaner for bathrooms
01-0838	Multi-purpose cleaner with flower fragrance
01-0839	Multi-purpose cleaner with orange fragrance
01-0840	Cleaning wipes for hard and soft surface
01-0841	Cleaner for ceramic hotplates
01-0842	Chalk/lime remover (hard surface cleaner)
01-0843	Scrub clean, lemon
01-0844	Dishwash
01-0845	Concentrated dishwash
01-0846	Dishwash with mild fragrance
01-0847	Dishwash
01-0848	Dishwash, yellow
01-0849	Dishwash
01-0850	Fabric conditioner with “spring” fragrance
01-0851	Fabric conditioner, fresh air
01-0852	Fabric conditioner, fresh air
01-0853	Fabric conditioner, apple flowers
01-0854	Fabric conditioner
01-0855	Fabric conditioner, refreshing odour
01-0856	Fabric conditioner, lavendel
01-0857	Detergent for woollen fabric
01-0858	Laundry detergent
01-0859	Laundry detergent for hand laundering
01-0860	Laundry detergent, granular
01-0861	Laundry detergent, granular
01-0862	Laundry detergent, granular
01-0863	Cleaning wipes, lemon
01-0864	Eraser (box with 50 erasers of several figures and colours)
01-0923	Car shampoo
01-0924	Car shampoo
01-0986	Hand cleaner
01-0987	Laundry detergent, liquid
01-0988	Laundry detergent, liquid
01-1310	Doll

3 Experimental

3.1 Chemicals

Eugenol 99%, isoeugenol 98%, geraniol 98%, cinnamic alcohol 98%, α-amyl cinnamaldehyde 97%, α-hexyl cinnamaldehyde (99%), benzyl alcohol (99%), anis alcohol (98%), benzyl cinnamate (99%) citronellol (95%) crystalline coumarin, farnesol (mixture of isomers) 95% and citral (mixture of cis- and trans-isomers) 95% were from Sigma-Aldrich, Germany; cinnamyl aldehyde (trans-) 98% was from Fluka, Switzerland; benzyl salicylate and hydroxycitronellal 95% were from ICN, USA, linalool (94%) and dl-limonene (97%) were from Merck, Germany and γ-methylionone (99%) was from TCI, Japan. Lyral® and Lilial® were obtained through Dragoco, Germany. Silica gel for column chromatography was ICN Active Silica 100-200 mesh from ICN, England/USA. All other chemicals of analytical grade were from E. Merck, Germany. All chemicals were used as obtained.

3.2 Calibration solutions

10% (w/v) stock solutions of all the fragrances were prepared in ethanol. These solutions were stored at 4°C for maximum three weeks. Calibration standard solutions 0.001%, 0.002%, 0.005%, 0.010% and 0.020% of all of the fragrances were prepared by diluting the stock solutions in ethanol. These solutions were freshly prepared from stock solutions and they were analysed within 24 hours.

3.3 Sample Preparation

3.3.1 Dishwash, surface cleaners, laundry detergents (liquid), car shampoos and handcleaner

Approximately 2 g sample was accurately weighed in a 30 ml dark bottle with screw cap. A small portion of boiling chips and 8 ml methanol was added to the sample and the bottle was capped. The mixture was shaken gently and then heated at 60°C for five minutes to facilitate extraction (heating is not necessary for homogenous liquid products). The solution/homogeneous suspension thus obtained was immediately cooled to room temperature (20°C). The fragrance rich fractions from the solution/suspension were extracted as described below.

A 20 cm x 1.8 cm Ø glass column was packed with wet silica gel (in methanol) to seven cm. The cooled sample solution/suspension was quantitatively transferred into the column and that was allowed to pass through the column. The initial 5 ml of the eluate was discarded. The fragrances, which eluted thereafter, were collected in a 25 ml volumetric flask. The column was further eluted with additional 20 ml methanol and the eluate was collected in the same 25 ml volumetric flask. The flask was filled with methanol up to the mark. The fragrance extract was immediately transferred into autosampler vials and analysed within 24 h. Each sample was analysed in duplicate and each sample extract was analysed twice by GC-MS.

3.3.2 Laundry detergents (granular)

Approximately 2 g sample, accurately weighed, was dissolved in 3 ml distilled water. 8 ml methanol was added to the detergent solution and mixed thoroughly. Fragrance rich fraction from this mixture was extracted by silica gel column chromatography as described above (3.3.1). Each sample was analysed in duplicate and each sample extract was analysed twice by GC-MS.

3.3.3 Toilet paper, nappy and panties

Approximately 2 g sample was weighed accurately in a 60 ml dark bottle. 25 ml methanol (10 ml for nappy) was added into the bottle, the mixture was treated with ultrasound for 30 min and centrifuged thereafter. The clear solution was analysed for the content of target fragrances. Both outer and the inner material of the nappy were analysed.

Each sample was analysed in duplicate and each sample extract was analysed twice by GC-MS.

3.3.4 Doll and eraser

Fragrances from doll and eraser were extracted in various solvents: methanol, ethanol, acetonitrile, cyclohexane, n-hexane, acetone and dichloromethane. Approximately 2 g sample, chopped to tiny pieces, was accurately weighed in a dark bottle with screw cap. 10 ml solvent was added into the bottle and that was capped. The mixture was treated with ultrasound for 60 min, and allowed to stand thereafter at room temperature for 30 min. After centrifugation, the clear extract was analysed by GC-MS. Two erasers (one sample) were analysed for the content of target fragrances.

3.4 Analysis

Qualitative analyses of the target fragrance substances in the sample extracts were performed by GC-MS (3.5). Quantification of the identified fragrances was performed using the intensity of selected ion for each fragrance. Calibration curves of target fragrance substances were prepared by the analysis of calibration solutions under the same conditions as the sample extracts. The fragrance contents in the sample solutions/suspensions were calculated using calibration curves of respective target fragrance substances. All samples were analysed in duplicate.

3.5 GC-MS

Instrument:	Perkin Elmer Turbo Mass, GC Perkin Elmer Auto System XL
GC column:	CP-Sil-5CB, 50 m x 0.32 mm Ø, film thickness 0.12 µm, 2.5m x 0.32mm fused silica retention gap (methyl deactivated)
Oven program:	Initial temperature 40°C, initial hold 0 min, ramp1: 30°C/min to 140°C, hold for 0 min, ramp 2: 4°C/min to 280°C, hold for 3 min, equilibration time 2 min.
Injector:	Split less, temperature 300°C, injection volume 2 µl, injectiontime 1 min
Carrier gas:	He, total flow 35 ml/min, column flow 3 ml/min
MS:	Ionisation at 180 eV, Scan m/z 33 - 400, scan time 0.6 sec
MS Library:	NIST and Wiley

4 Results and Discussion

An earlier described GC-MS method (2), with some modifications, has been employed for the analysis of content of target fragrances in the present investigation. The recoveries of fragrances from cosmetics, domestic products and occupational products by this method have been found to be 72 – 116% (2, 3). Furthermore, the recoveries of various fragrance materials were found to be solely dependent on the product matrix. The relative standard deviation of the method for the determination of various fragrances was within 12%.

Chromatographic separation of standard fragrance chemicals by the present method is shown in Figure 4.1, and chromatograms of some selected samples are shown in Figures 4.2 – 4.9. The fragrances were identified by comparing the GC-retention time (t_R) as well as mass spectrum (m/z 33 – m/z 400) of the sample GC peaks with the t_R and mass spectrum of chemicals in two standard mass spectrum libraries. The detection limits of the target substances were 0.00001 – 0.0002 %.

The contents of all target substances, except amyl cinnamyl alcohol, were determined in the present investigation. Only qualitative analysis of amyl cinnamyl alcohol was performed. The determination was performed using calibration curves of the target fragrances. The correlation coefficient of all calibration curves were > 0.995. The contents of target fragrances in the investigated products are described in Table 4.1.

In the absence of a suitable method for the analysis of fragrance materials in special products (erasers and doll) a screening analysis of extracts of these samples in seven different solvents (polar, non-polar, chlorinated and non-chlorinated) has been performed. None of the target fragrance substances were detected in the two eraser samples. The doll sample was found to contain cinnamyl alcohol and coumarin. The content of coumarin and cinnamyl alcohol in the doll are estimated to be approximately 0.007% and 0.005% respectively. Besides several other fragrance materials, for example, vanillin, ethyl vanillin and piperonal were also identified in the doll sample.

The frequency of the presence of target fragrances and their concentration range in the investigated products are summarised in Table 4. Of the investigated products,
67% contained limonene;
56% lilial;
40% hexylcinnamic aldehyde, linalool and γ-methylionone each;
30% benzyl alcohol and coumarin each;
26% benzyl benzoate and citronellol;
21% benzyl salicylate and geraniol each;
19% eugenol;
and 16% of the products contained citral.
All other target fragrances were present in < 10% of the products. Benzyl cinnamate, farnesol and methyl heptine carbonate were not found in any the investigated products. Among the investigated products, three products (nappy, a soft soap for floor wash and erasers) did not contain any of the target fragrance substances. It was not possible to trace a trend in the use of certain fragrances (and their amounts) in a specific product category. The reason for this may be that the number of products in each category is rather small.

Table 4.1 Contents of target fragrances in the investigated products.

Fragrance material	Content % (m/m)									
	Sample No.	01-0828 Panties A	01-0828 Panties B	01-0829 Panties A	01-0829 Panties B	01-0830 Nappy C	01-0830 Nappy D	01-0831 Toilet paper	01-0832 Toilet paper	01-0833 Surface cleaner	01-0834 Surface cleaner
Amyl cinnamal		-	-	-	-	-	-	-	-	0.0092	-
Amylcinnamyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl alcohol		-	-	-	-	-	-	0.0012	0.0033	0.0075	-
Benzyl salicylate		-	-	-	-	-	-	-	-	-	-
Cinnamyl alcohol		-	-	-	-	-	-	0.0021	-	-	-
Cinnamal		-	-	-	-	-	-	-	-	-	-
Citral		-	-	-	-	-	-	-	+	-	-
Coumarin		-	-	-	-	-	-	-	-	0.0035	-
Eugenol		-	-	-	-	-	-	-	-	-	-
Geraniol		-	-	-	-	-	-	+	-	0.0086	-
Hydroxycitronellal		-	-	-	-	-	-	0.0025	-	-	-
Lyral®		-	-	-	-	-	-	-	-	-	-
Isoeugenol		-	-	-	-	-	-	-	-	0.0085	-
Anisyl alcohol		-	-	-	-	-	-	0.0014	-	-	-
Benzyl benzoate		0.0065	0.0077	0.0095	0.0152	-	-	-	0.0003	0.0069	-
Benzyl cinnamate		-	-	-	-	-	-	-	-	-	-
Citronellol		*	-	-	-	-	-	+	-	-	-
Farnesol		-	-	-	-	-	-	-	-	-	-
Hexyl cinnamaldehyde		-	-	-	-	-	-	0.0005	+	-	-
Lilial®		-	-	-	-	-	-	0.0010	0.0009	-	-
dl-Limonene		0.0041	-	0.0102	0.0174	-	-	-	+	0.0060	-
Linalool		*	-	-	-	-	-	0.00009	0.00002	0.0096	-
Methyl heptine carbonate		-	-	-	-	-	-	-	-	-	-
γ-Methylionone		-	-	-	-	-	-	-	-	0.0134	-
Total content of analysed fragrances		0.0106	0.0077	0.0197	0.0326	-	-	0.00879	0.00452	0.0732	-

A: Fragrance test attached to the fabric B: The fabric it self C: Nappy outer material D: Nappy inner material,
+ fragrance present at detection level
* fragrance present but not determined due to interference

Table 4.1 Continued.

Fragrance material	Content % (m/m)									
	Sample No.	01-0835 Surface cleaner	01-0836 Surface cleaner	01-0837 Surface cleaner	01-0838 Multi-cleaner	01-0839 Multi-cleaner	01-0840 Cleaning wipes	01-0841 Cleaning wipes	01-0842 Surface cleaner	01-0843 Scrub clean	01-0844 Dish-wash
Amyl Cinnamal		-	-	-	-	-	-	-	-	-	-
Amylcinnamyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl alcohol		0.0082	0.0087	-	-	0.2354	0.0118	-	-	-	-
Benzyl salicylate		-	-	-	0.0089	-	0.0587	-	-	-	-
Cinnamyl alcohol		-	-	-	-	-	-	-	-	-	-
Cinnamal		-	-	-	-	-	-	-	-	-	-
Citral		-	0.0092	-	-	+	-	0.0160	-	-	0.0260
Coumarin		0.0151	-	-	0.0270	-	-	-	0.0051	-	-
Eugenol		+	-	-	-	-	-	-	-	-	-
Geraniol		0.0103	-	-	-	-	0.0069	-	-	0.0843	-
Hydroxycitronellal		-	-	-	-	-	-	-	-	-	-
Lyral®		-	-	-	-	-	-	-	-	-	-
Isoeugenol		-	-	-	-	-	-	-	-	-	-
Anisyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl benzoate		0.0062	-	-	0.0062	-	-	0.0071	-	-	-
Benzyl cinnamate		-	-	-	-	-	-	-	-	-	-
Citronellol		-	-	0.0091	-	-	0.0529	-	-	-	-
Farnesol		-	-	-	-	-	-	-	-	-	-
Hexyl cinnamaldehyde		-	-	-	-	-	0.0492	-	-		-
Lilial®		0.0064	-	-	0.0053	-	0.0500	-	0.0077	0.0076	-
dl-Limonene		-	0.1108	0.0044	0.0565	0.7639	0.0061	0.0255	0.0103	0.1949	0.0963
Linalool		0.0147	-	-	-	0.0092	-	-	-	-	-
Methyl heptine carbonate		-	-	-	-	-	-	-	-	-	-
γ-Methylionone		0.0188	-	-	-	-	0.0346	-	0.1586	-	-
Total content of analysed fragrances		0.154	0.128	0.0135	0.104	1.009	0.270	0.0486	0.182	0.287	0.122

+ fragrance present at detection level

Table 4.1 Continued.

Fragrance material	Content % (m/m)										
	Sample No.	01-0845 Dish wash	01-0846 Dish-wash	01-0847 Dish-wash	01-0848 Dish-wash	01-0849 Dish-wash	01-0850 Fabric condi-tioner	01-0851 Fabric condi-tioner	01-0852 Fabric condi-tioner	01-0853 Fabric condi-tioner	01-0854 Fabric cond-tioner
Amyl Cinnamal		-	-	0.0284	-	-	-	-	-	-	-
Amylcinnamyl alcohol		-	-	n.d.	n.d.	-	-	-	-	-	-
Benzyl alcohol		-	-	0.0277	-	-	-	-	-	+	-
Benzyl salicylate		-	-	-	-	-	-	0.0112	0.0072	0.0069	-
Cinnamyl alcohol		-	0.0030	-	-	-	0.0050	-	-	-	-
Cinnamal		-	-	-	-	-	-	-	-	-	0.0061
Citral		0.0501	-	-	-	-	-	-	-	-	-
Coumarin		-	-	-	-	-	0.0054	0.0064	0.0069	-	0.0219
Eugenol		-	-	0.0083	-	-	-	-	-	0.0090	-
Geraniol		-	0.0070	-	0.1454	0.0079	-	-	-	-	-
Hydroxycitronellal		-	-	-	-	-	-	0.0071	-	-	0.0078
Lyral®		-	-	-	-	-	0.0110	-	-	-	0.0072
Isoeugenol		-	-	-	-	-	-	-	-	0.0097	-
Anisyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl benzoate		-	-	-	-	-	-	0.0039	-	-	0.0058
Benzyl cinnamate		-	-	-	-	-	-	-	-	-	-
Citronellol		-	0.0162	0.0763	0.0160	-	0.0157	-	-	-	0.0717
Farnesol		-	-	-	-	-	-	-	-	-	-
Hexyl cinnamaldehyde		-	-	0.0177	-	-	0.0289	0.0301	0.0112	0.0068	0.0284
Lilial®		-	-	0.0074	-	-	0.0049	0.0148	0.0055	0.0182	0.0102
dl-Limonene		0.1183	0.0003	0.0091	0.4164	0.0048	0.0165	0.0481	0.0156	-	-
Linalool		-	0.0023	0.0270	0.0056	0.0060	-	-	0.0070	0.0095	-
Methyl heptine carbonate		-	-	-	-	-	-	-	-	-	-
γ-Methylionone		-	-	-	-	-	0.0175	0.0178	0.0184	0.0463	0.0598
Total content of analysed fragrances		0.168	0.0288	0.202	0.583	0.0187	0.105	0.139	0.0718	0.106	0.219

+ fragrance present at detection level
n.d.: not determined, standard not available.

Table 4.1 Continued.

Fragrance material	Content % (m/m)										
	Sample No.	01-0855 Fabric condi-tioner	01-0856 Fabric condi-tioner	01-0857 Deter-gent Wool	01-0858 Deter-gent laundry	01-0859 Deter-gent laundry	01-0860 Deter-gent laundry	01-0861 Deter-gent laundry	01-0862 Deter-gent laundry	01-0863 Cleaning wipes	01-0864 Eraser
Amyl Cinnamal		-	-	-	-	-	-	-	-	-	-
Amylcinnamyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl alcohol		0.0103	-	-	0.0146	-	-	+	-	-	-
Benzyl salicylate		0.0136	0.0114	0.0142	0.0173	-	-	-	-	-	-
Cinnamyl alcohol		-	-	-	-	-	-	-	-	-	-
Cinnamal		-	-	-	-	-	-	-	-	-	-
Citral		-	-	-	-	-	-	-	-	-	-
Coumarin		0.0098	-	-	0.0027	-	-	-	0.0043	-	-
Eugenol		0.0090	-	-	0.0094	-	-	-	-	-	-
Geraniol		-	0.0018	-	-	-	-	-	-	-	-
Hydroxycitronellal		-	-	-	-	-	-	-	-	-	-
Lyral®		-	-	-	-	-	-	-	-	-	-
Isoeugenol		-	-	0.0048	-	-	-	-	-	-	-
Anisyl alcohol		-	-	-	-	-	-	-	-	-	-
Benzyl benzoate		-	-	-	-	-	-	-	-	-	-
Benzyl cinnamate		-	-	-	-	-	-	-	-	-	-
Citronellol		-	+	-	-	-	-	-	-	-	-
Farnesol		-	-	-	-	-	-	-	-	-	-
Hexyl cinnamaldehyde		0.0203	0.0057	-	0.0317	0.0056	-	0.0096	0.0071	0.0059	-
Lilial®		0.0110	0.0075	0.0075	0.0109	0.0074	0.0071	0.0073	0.0076	0.0052	-
dl-Limonene		0.0083	0.0083	-	0.0105	-	-	+	-	0.0158	-
Linalool		-	-	0.0099	0.0095	-	-	0.0055	-	0.0039	-
Methyl heptine carbonate		-	-	-	-	-	-	-	-	-	-
γ-Methylionone		0.0452	0.0134	0.0185	-	-	0.0073	0.0076	0.0076	-	-
Total content of analysed fragrances		0.128	0.0481	0.0549	0.107	0.0130	0.0144	0.0300	0.0266	0.0308	-

+ fragrance present at detection level

Table 4.1 Continued.

Fragrance material	Content % (m/m)						
	Sample No.	01-0923 Car shampoo	01-0924 Car shampoo	01-0986 Hand cleaner	01-0987 Deter-gent laundry	01-0988 Deter-gent laundry	01-1310 Doll
Amyl Cinnamal		-	-	-	-	-	-
Amylcinnamyl alcohol		-	-	-	-	-	-
Benzyl alcohol		-	-	-	0.0071	-	-
Benzyl salicylate		-	-	-	-	-	-
Cinnamyl alcohol		-	-	-	-	-	0,0071[a]
Cinnamal		-	-	-	-	-	-
Citral		-	+	-	-	-	-
Coumarin		0.0032	-	-	-	-	0,0044[a]
Eugenol		-	-	-	-	0.0119	-
Geraniol		-	-	-	-	-	-
Hydroxycitronellal		-	-	-	-	-	-
Lyral®		-	-	-	0.0053	-	-
Isoeugenol		-	-	-	-	-	-
Anisyl alcohol		-	-	-	-	-	-
Benzyl benzoate		-	0.0042	-	-	-	-
Benzyl cinnamate		-	-	-	-	-	-
Citronellol		-	-	-	0.0301	-	-
Farnesol		-	-	-	-	-	-
Hexyl cinnamaldehyde		-	-	-	0.0109	-	-
Lilial®		-	-	-	0.0132	0.0112	-
dl-Limonene		0.0077	0.0099	0.0255	-	-	-
Linalool		-	-	-	0.0144	-	-
Methyl heptine carbonate		-	-	-	-	-	-
γ-Methylionone		-	-	-	0.0228	0.0239	-
Total content of analysed fragrances		0.0109	0.0141	0.0255	0.104	0.0470	0.0115

+ fragrance present at detection level

[a] Approximate content

Table 4.2
Summary of contents of target fragrances in the investigated products.

Fragrance material	Present in		Content % (m/m)
	No. of products	% Products	Range
d-Limonene*	29	67	+ - 0.7693
Lilial®	24	56	0.0009 - 0.0500
Hexyl cinnamaldehyde	17	40	+ - 0.0492
Linalool	17	40	0.00002 - 0.0270
γ-Methylionone	17	40	0.0073 - 0.1586
Benzyl alcohol	13	30	+ - 0.2354
Coumarin	13	30	0.0027 - 0.0270
Benzyl benzoate	11	26	0.0030 - 0.0152
Citronellol	11	26	+ - 0.0763
Benzyl salicylate	9	21	0.0069 - 0.0587
Geraniol	9	21	+ - 0.1454
Eugenol	8	19	+ - 0.0119
Citral	7	16	+ - 0.0501
Cinnamyl alcohol	4	9	0.0021 - 0.0071
Hydroxycitronellal	3	7	0.0025, 0.0071, 0.0078
Lyral®	3	7	0.0053, 0.0072, 0.0110
Isoeugenol	3	7	0.0048, 0.0085, 0.0097
Amyl cinnamal	2	5	0.0092, 0.0284
Amylcinnamyl alcohol	2	5	n.d.
Cinnamal	1	2	0.0061
Anisyl alcohol	1	2	0.0014
Benzyl cinnamate	-	-	-
Farnesol	-	-	-
Methyl heptine carbonate	-	-	-

+ fragrance present at detection level
n.d.: not determined, identification only

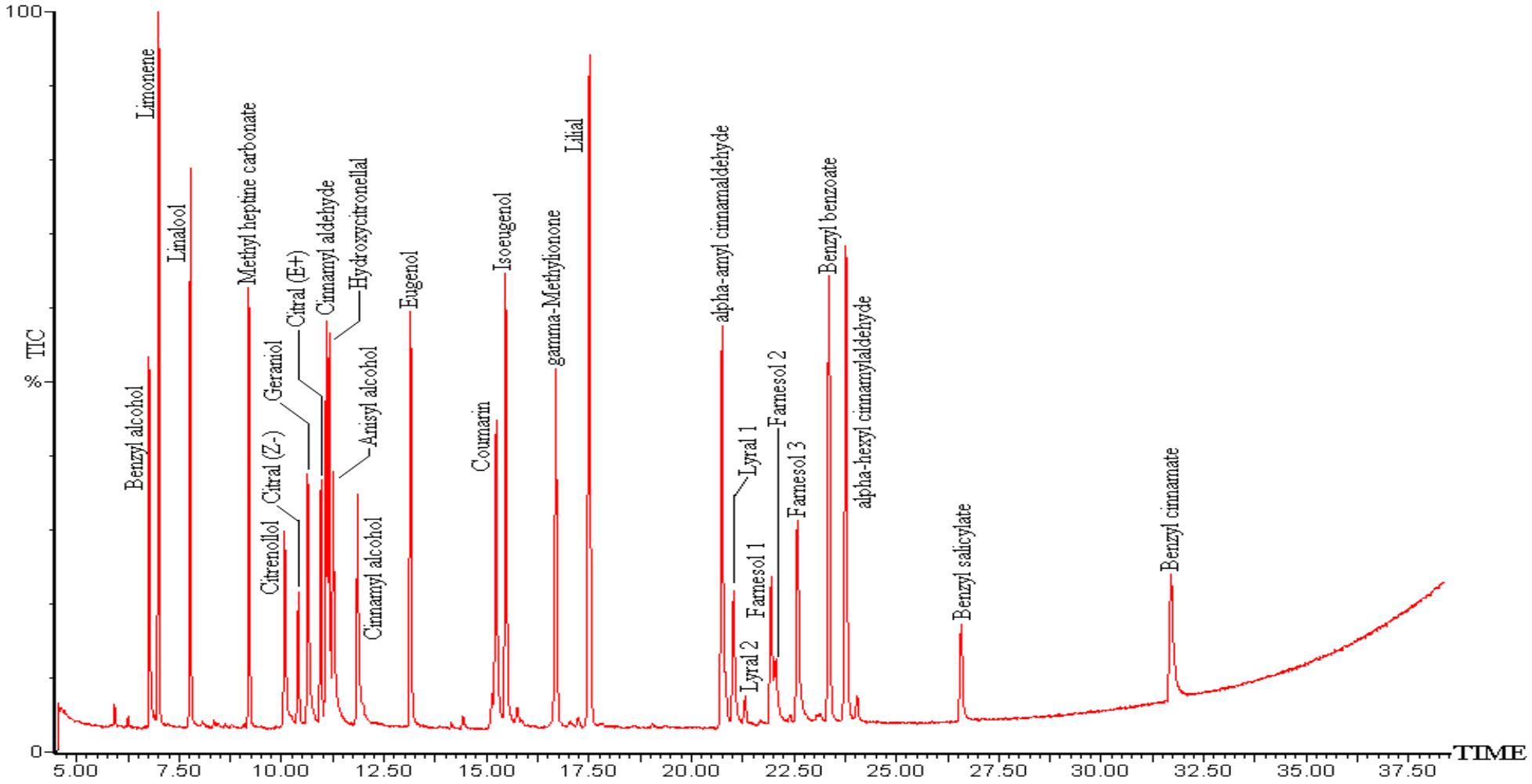

Figure 4.1
GC-MS chromatogram of target fragrances. Concentration of each fragrance : approximately 0.01 %.

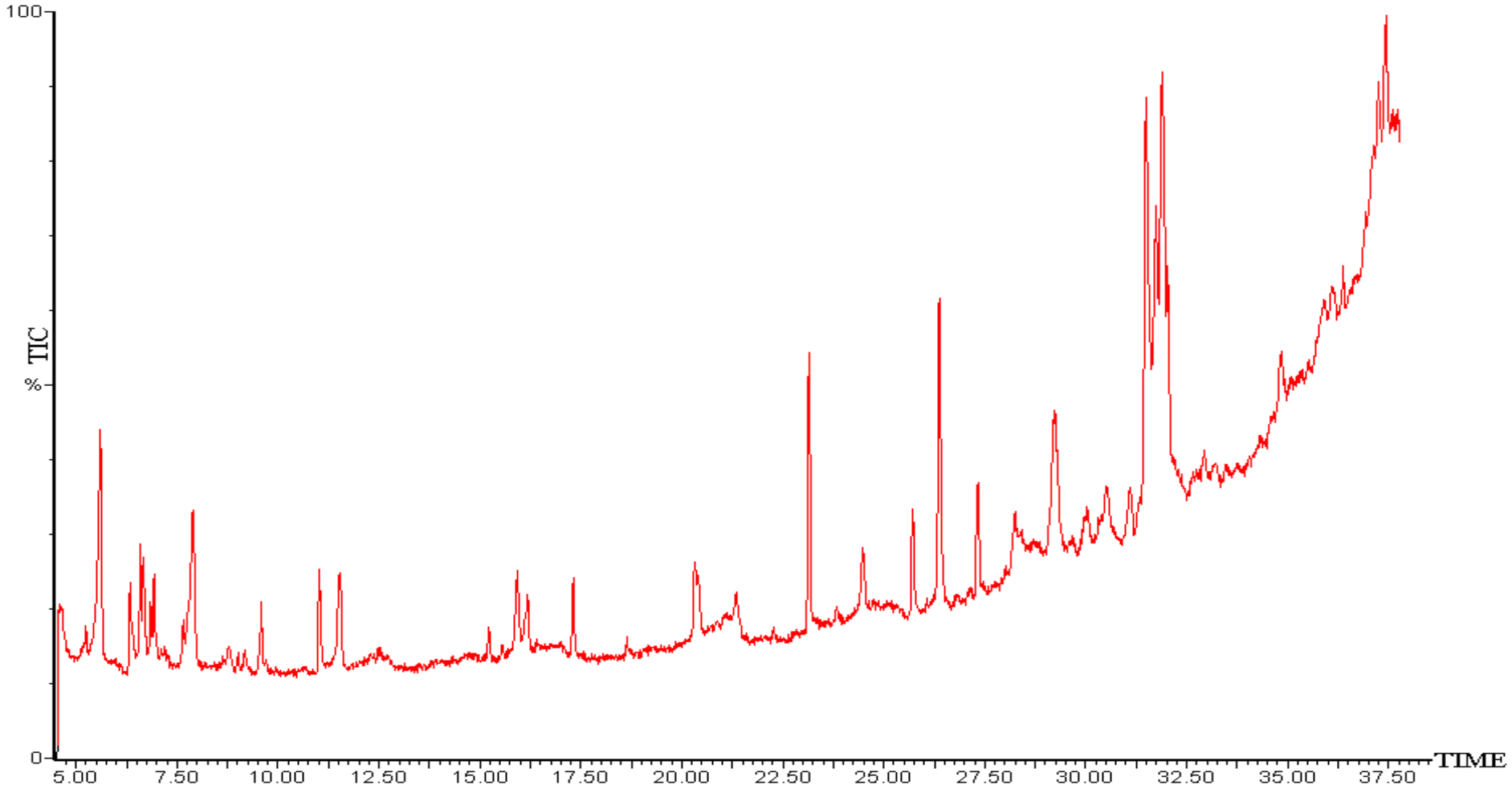

Figure 4.2
GC-MS chromatogram of fragrance toilet paper 01-0831.

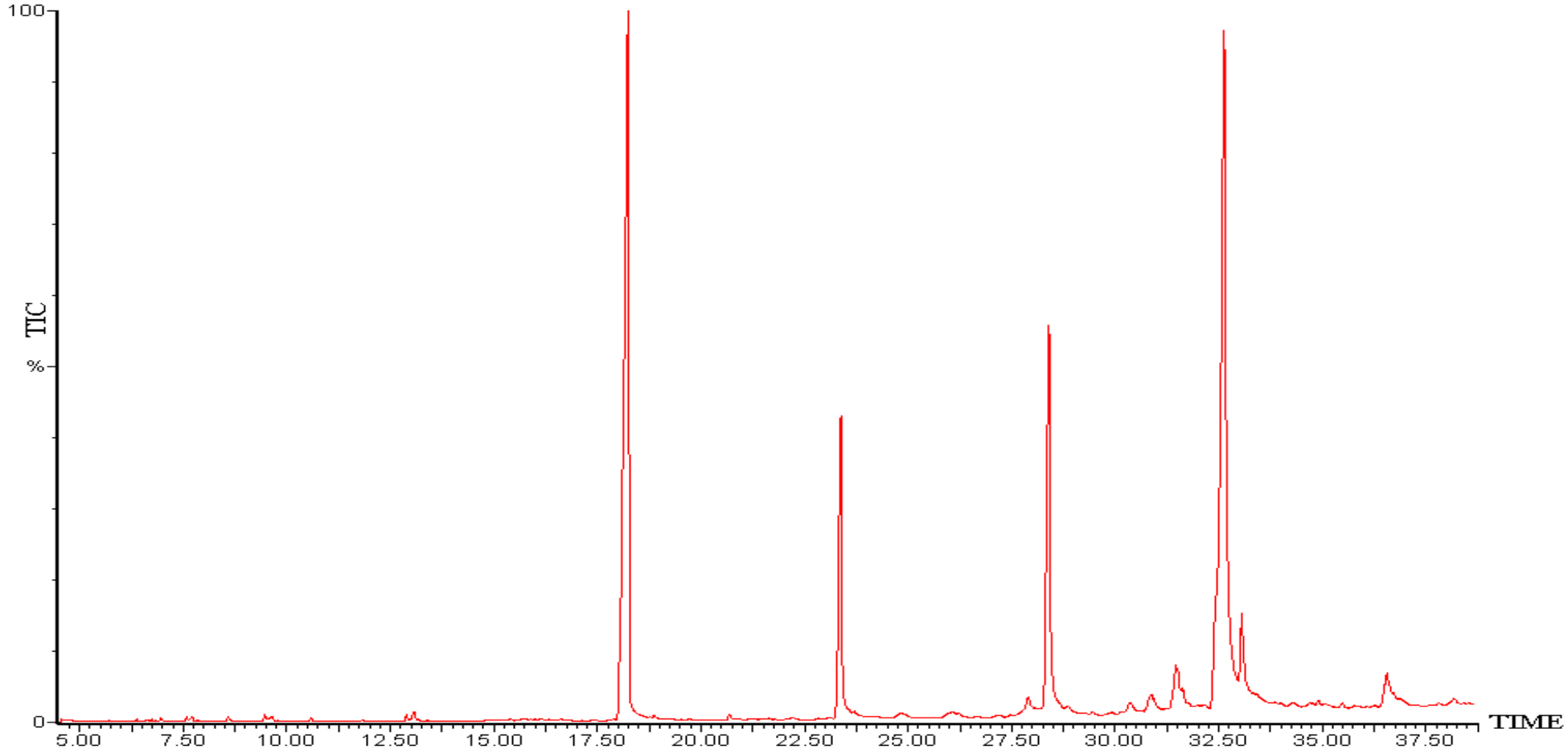

Figure 4.3
GC-MS chromatogram of the soft soap 01-0833.

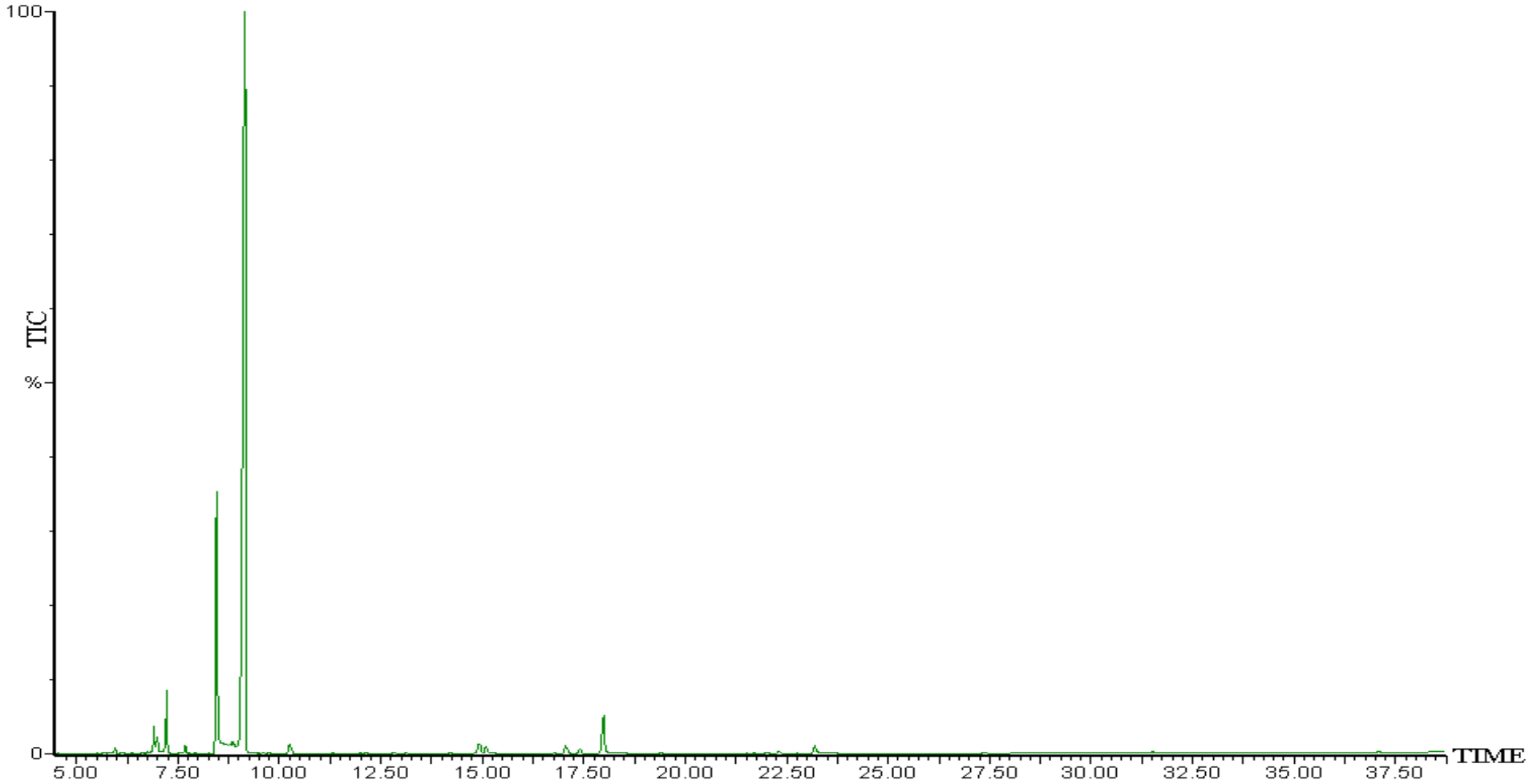

Figure 4.4
GC-MS chromatogram of the multipurpose cleaner 01-0838.

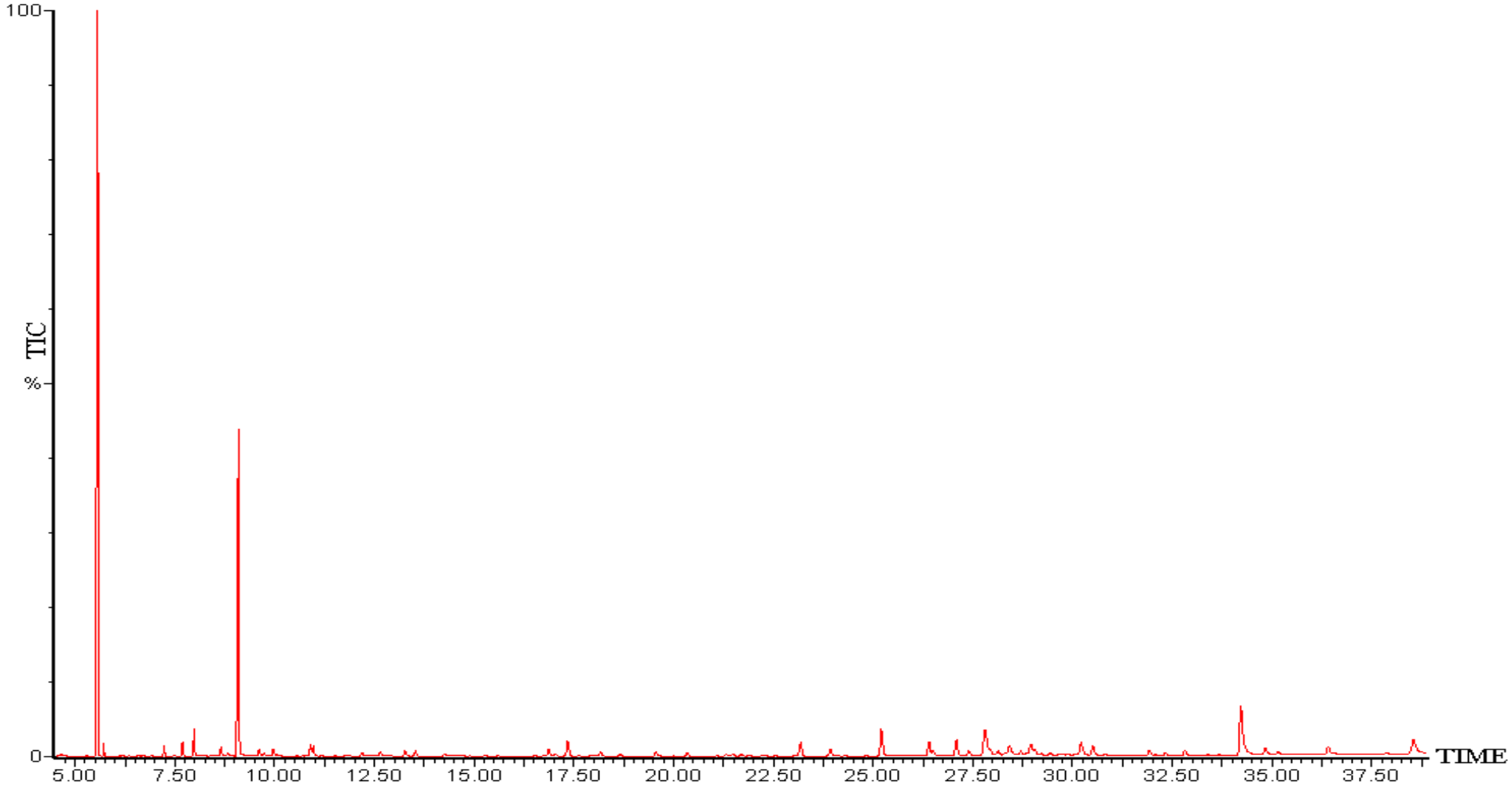

Figure 4.5
GC-MS chromatogram of the cleaning wipes 01-0840.

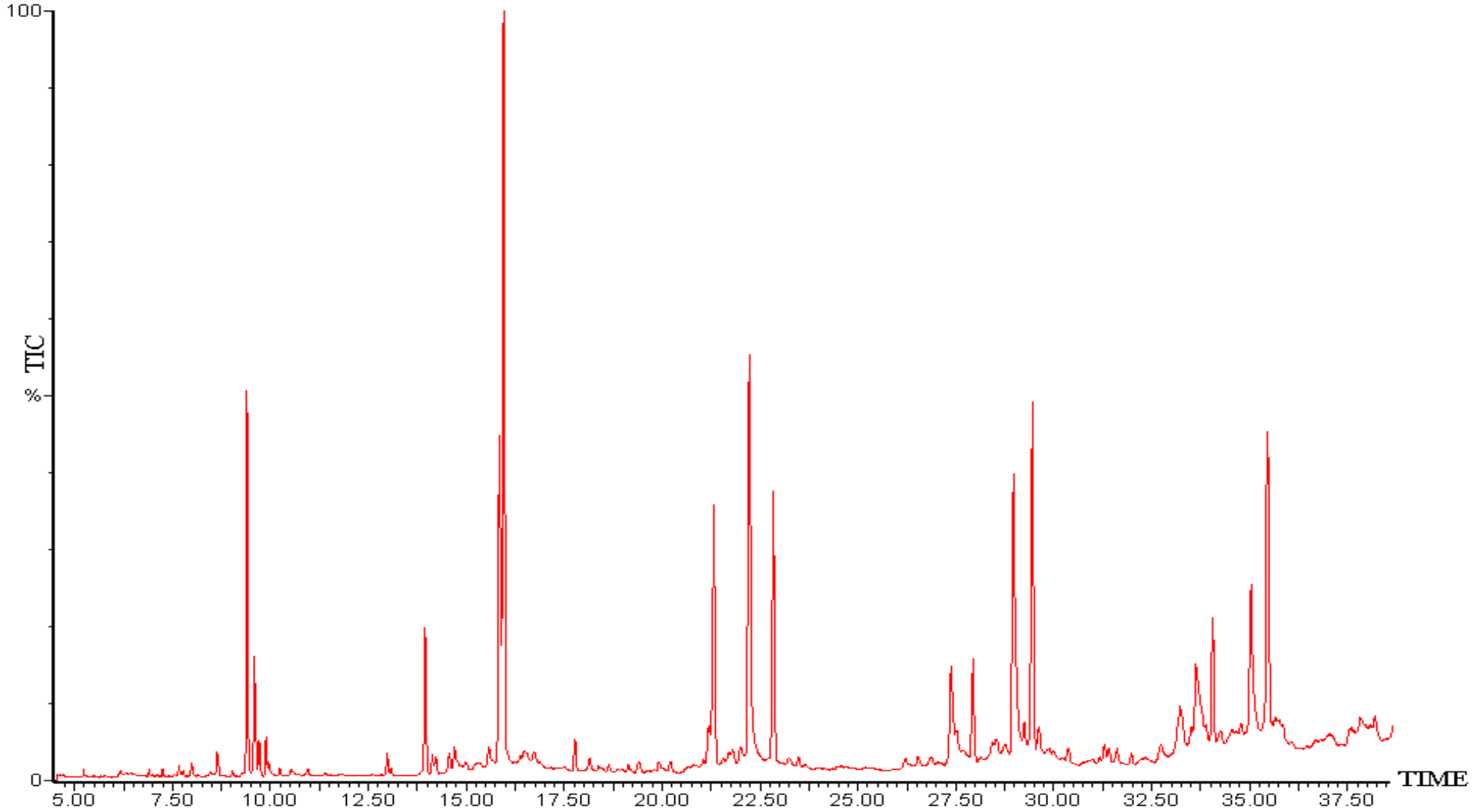

Figure 4.6
GC-MS chromatogram of dishwash product 01-0846.

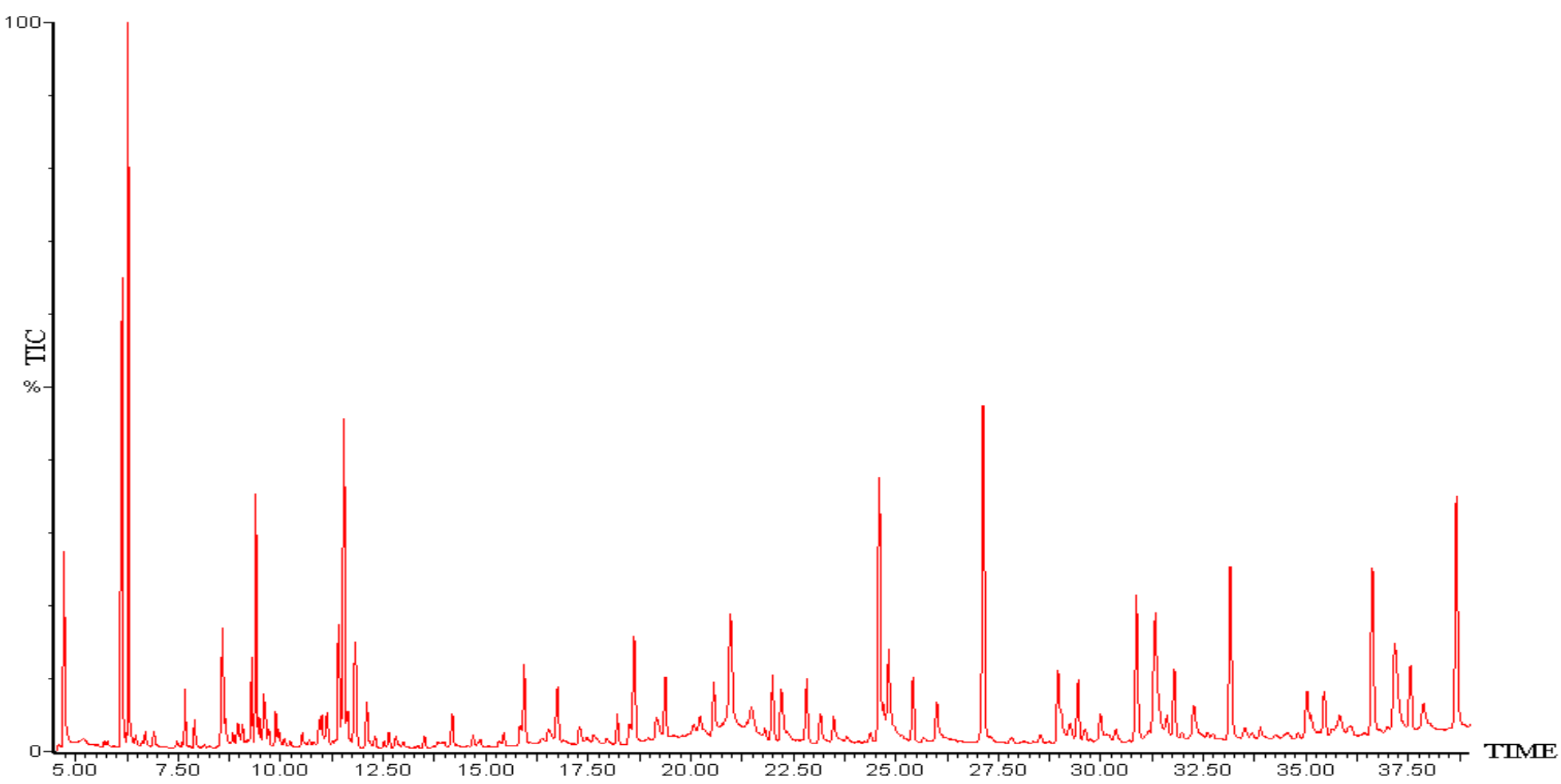

Figure 4.7
GC-MS chromatogram of the dishwash product 01-0847.

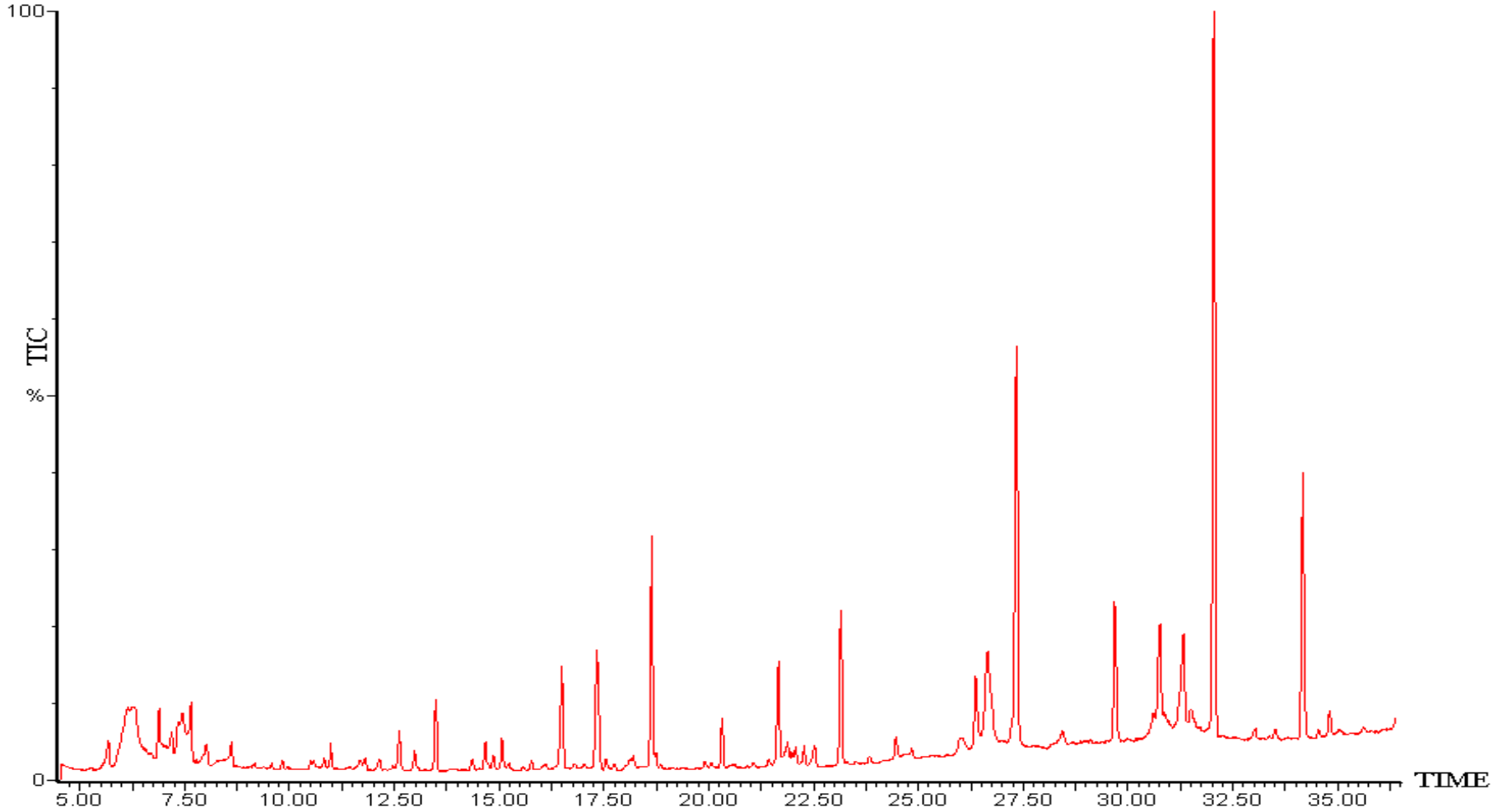

Figure 4.8
GC-MS chromatogram of the fabric conditioner 01-0855.

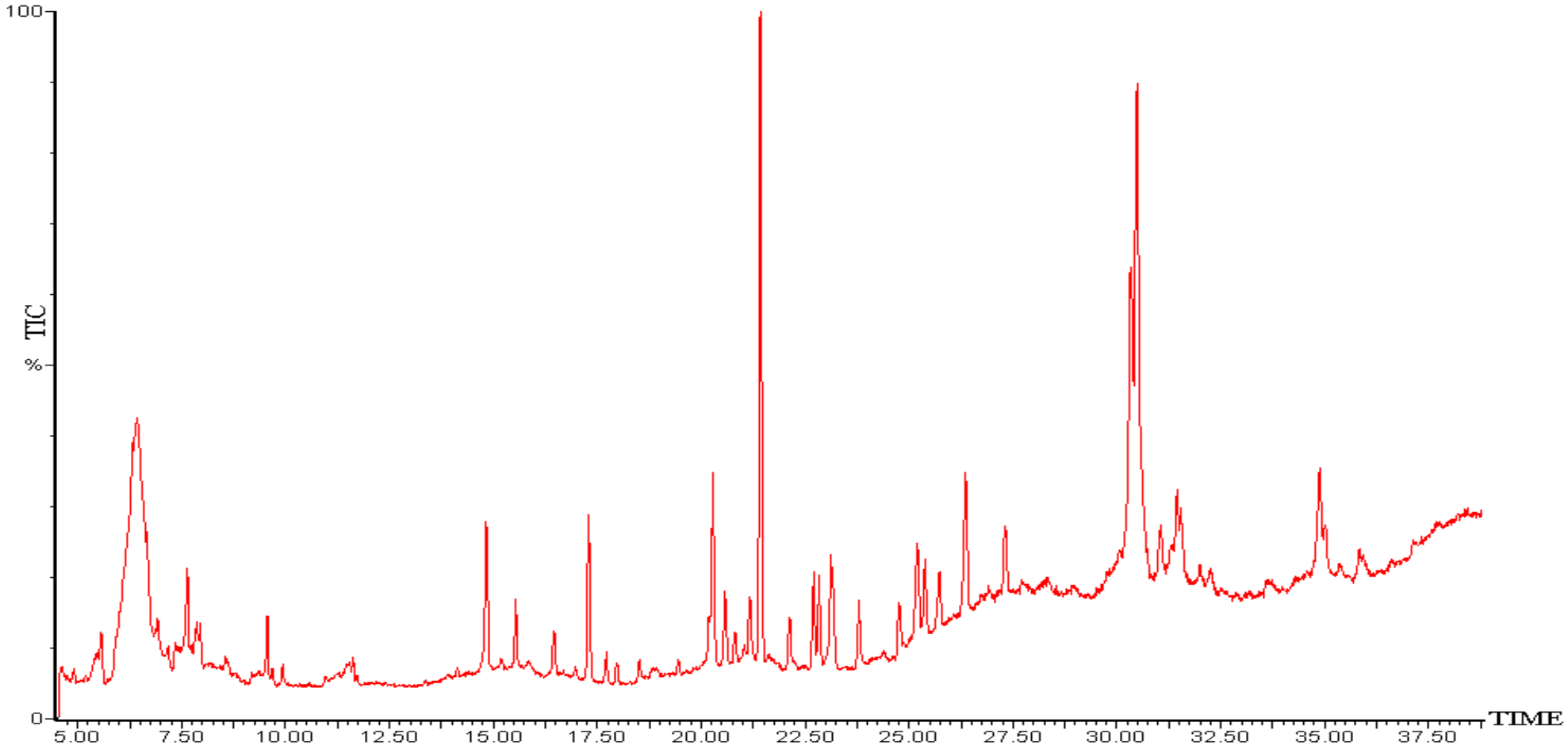

Figure 4.9
GC-MS chromatogram of the laundry detergent 01-0987.

References

1. EU Scientific Committee on Cosmetic Products and Non-Food Products. Fragrance Allergy in Consumers. A review of the problem. Brussels EU Commission, DG SANCO, SCCNFP/0017/98 final, 8th December 1999. http://europa.eu.int/comm/food/fs/sc/sccp/out98_en.pdf

2. Rastogi S.C. (1995): Analysis of fragrances in cosmetics by gas chromatography -mass spectrometry. J. High Resolution Chromatogr. 18: 653-658.

3. Rastogi S.C., Heydron S., Johansen J.D. and Basketter D.A. (2001): Fragrance chemicals in domestic and occupational products. Contact Dermatitis 45: 221-225.