

# Separation of Fragrance Materials from Perfumed Consumer Products

By A. Bartsch and F.-J. Hammerschmidt, Dragoco Gerberding & Co. GmbH, Research Department, Holzminden, Germany

A perfumer's work can greatly benefit from investigations of the stabilities and interactions of fragrance materials in consumer products. These investigations (concerning, for example, the shelf life of a cosmetic product or a household product) include isolation of the perfume oil from the finished product. Then individual components of the extracted oil are separated, identified, and finally quantified via high resolution gas chromatography (HRGC) coupled with mass spectrometric detection (MS).

In isolating the fragrance materials, which are usually present only at low levels, the analytical chemist is confronted with a formidable separation challenge, particularly when the sample also contains emulsifiers, surfactants, clarifiers, thickeners, solubilizers, pigments, antioxidants, UV-absorbers, preservatives, solvents, and other materials.

Two of the principal techniques for isolating volatile components like fragrance materials from samples are water or steam hydrodistillation (WSD) and simultaneous hydrodistillation and extraction (SDE). For routine analysis, the most widely used circulatory distillation apparatuses were developed by Sprecher (WSD) and by Likens and Nickerson (SDE).<sup>2,3</sup> The original apparatus design was modified by several other workers to suit specific requirements.<sup>4,6</sup> These techniques have many advantages including relative simplicity and ease of operation as well as supposed high efficiency in isolating volatile materials. However, no single method will provide a fragrance profile truly representative of the sample, particularly when the sample contains ingredients of different chemical classes which cover a very wide range of volatilities, solubilities, and

polarities.<sup>7</sup> The most appropriate technique will be a compromise based on analytical considerations such as time consumption, accuracy, precision and reproducibility, especially when quantitative work is involved.

In our experiments, fragrance materials covering a wide range of physical and chemical properties were recovered from a model cosmetic product in the form of a shower bath, and the rate of recovery was examined to see how it was affected by each of four variables.

- The distillation technique (WSD or SDE)
- The extraction solvent used (in SDE)
- The distillation period
- The salting out effect (in SDE)

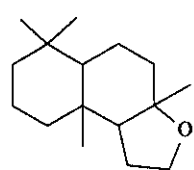
## Experimental Materials

**Model system:** The experimental materials were three samples. The samples were formulated alike to represent a typical cosmetic product (Formula 1). However, each sample

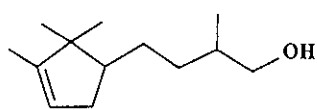
### Formula 1. The shower bath base, used to represent a model cosmetic product

Distilled water	51.1%
Anionic surfactant	42.0
Sodium chloride	2.0
Thickener	1.0
Refatting agent	1.0
Preservative	0.2
Citric acid	0.2
<b>Test perfume oil</b>	<b>1.5</b>
	100.0

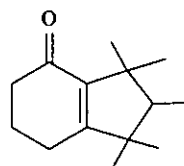
This article is based on a poster presentation at the 12th International Congress of Flavors, Fragrances and Essential Oils, October 4-8, 1992, Vienna, Austria.



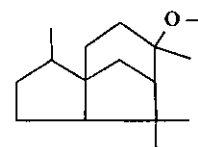
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(Henkel)



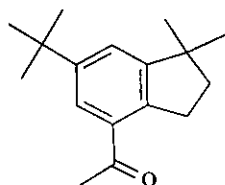
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(DRAGOCO)



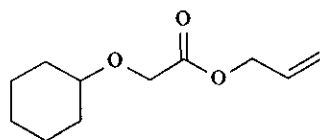
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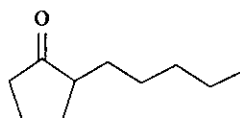
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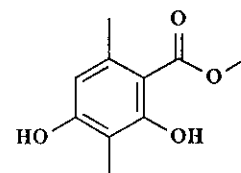
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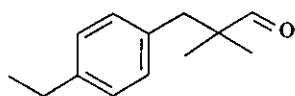
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(DRAGOCO)



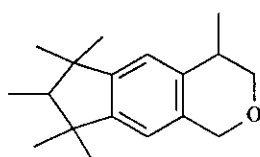
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(Firmenich)



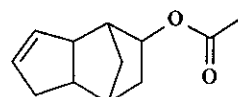
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(RBF)



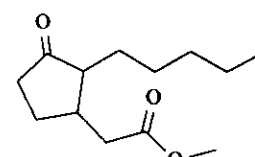
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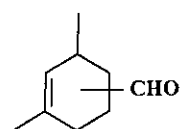
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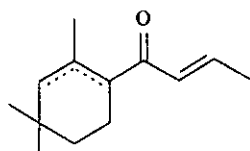
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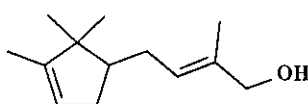
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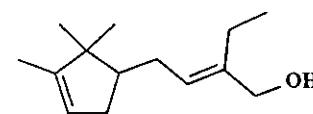
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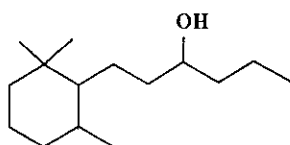
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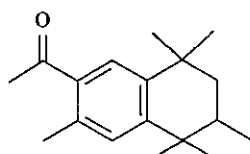
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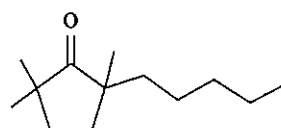
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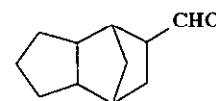
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(DRAGOCO)



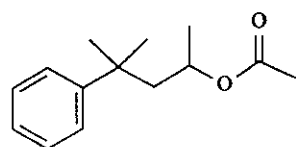
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(PFW)



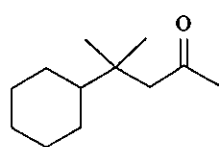
**Veloutone**  
(Firmenich)



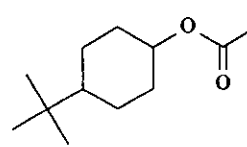
**Vertral**  
(DRAGOCO)



**Veticol acetate**  
(DRAGOCO)



**Vetival**  
(DRAGOCO)



**p - Ylanate**  
(DRAGOCO)

**Figure 1. Molecular structures of some fragrance materials used in the test perfume oils**

had a unique perfume. Each of the three test perfume oils consisted of 23-25 fragrance components (Tables II and III), and was composed so that gas chromatographic baseline separation of the components was possible. The molecular structures of some of the components are illustrated in Figure 1.

**Reagents:** The following six extracting solvents were used: methylene chloride (Merck 6050), n-pentane (Merck 820957), n-hexane (Merck 4367), diethylether (Merck 926), ethyl acetate (Merck 9623), and Freon 113 (Kalichemie AG). Two additional extracting solvents were mixtures of pentane and diethylether in ratios of 1:1 and 7:3 by volume. All solvents were purified by fractional distillation and were routinely checked by gas chromatography prior to the experiments. Sodium sulfate (6404), sodium chloride (6649), and silicone antifoaming agent (7743) were from Merck. 2-Methyl-1-pentanol (21,401-9; purity grade 99%) and mesitylene (M720-0; purity grade 99%) were from Aldrich.

### Experimental Methods

**Instrumentation:** The Likens and Nickerson apparatus modified by Flath and Forrey is described in detail together with a drawing in previous publications.<sup>5,8-10</sup> Detailed information on the hydrodistillation apparatus can be obtained from the DAB.<sup>11</sup> The apparatus was modified to integrate the glass bubble containing the organic extracting solvent into the cooled condenser part.

A Hewlett-Packard 5890 II gas chromatograph equipped with both an evaporation split injector and a hydrogen flame ionization detector (FID) was used. A second corresponding chromatograph was coupled with a Hewlett-Packard 5970 mass spectrometric detector.

**Distillation:** About 20 g to 30 g of the sample, accurately weighed, and 500 mL of either bidistilled water or saturated sodium chloride solution were suspended in a 1-L round-bottom flask. This solution was then neutralized (pH = 7) and 0.5 mL of antifoaming agent was added. Distillation and extraction were carried out at atmospheric pressure for 2, 4 and 8 hrs, with 100 mL of organic solvent in the case of SDE and about 10 mL in the case of WSD. The important distillation parameters are summarized in Table I.

Vigorous boiling of both flask fillings and adequate stirring of the aqueous sample solution are essential for optimum efficiency. Stirring can also avoid local overheating of the sample solution. After the required time had elapsed, the boiling of the water was stopped but the reflux of the organic solvent was continued for 15 additional minutes.<sup>12</sup> The extracts obtained were dried over anhydrous sodium sulfate. The solution was then filtered into a small flask and the residual sodium sulfate was rinsed three times with small portions of extracting solvent. After concentration of the filtrate to 1 mL on a Vigreux column (10 cm x 0.8 cm, 40-80°C, dependent on the solvent) and finally to about 100  $\mu$ L by carefully blowing off the solvent under a moderate jet of an inert gas at room temperature, the extract was spiked with 50  $\mu$ L of an internal standard (IS)

solution containing 120 mg/mL each of mesitylene and 2-methyl-1-pentanol. The concentrations of the two internal standards should be matched to the expected levels of the extracted fragrance materials.

**Capillary gas chromatography:** The concentrated extracts were separated into their constituents by HRGC. A 60 m x 0.25 mm i.d. fused silica capillary column with 0.25  $\mu$ m thickness of crosslinked Carbowax 20M (J&W Scientific) was employed. A nitrogen carrier gas with a flow rate of 2 mL/min was passed through the column. Both the injector and detector were maintained at 250°C. The column temperature was programmed from 60°C to 240°C at 4°C/min and finally was held at 240°C for 30 min. The inlet splitter was operated at a split ratio of 1:50 and the extract volume injected into the GC system was about 1  $\mu$ L with split on, applying the hot empty needle sampling technique.<sup>12,13</sup>

The HRGC/FID was used for quantitative analysis. The components and the artifacts were identified via mass spectrometric detection in the EI mode. For better detection of the Evernyl component, a 50 m x 0.2 mm i.d. column with a DB-1 stationary phase (J&W Scientific), 0.33  $\mu$ m thick, was installed. Column temperature was programmed from 60°C to 300°C at 4°C/min; injector and FID temperatures were 240°C and 300°C, respectively.

Table I. Distillation parameters

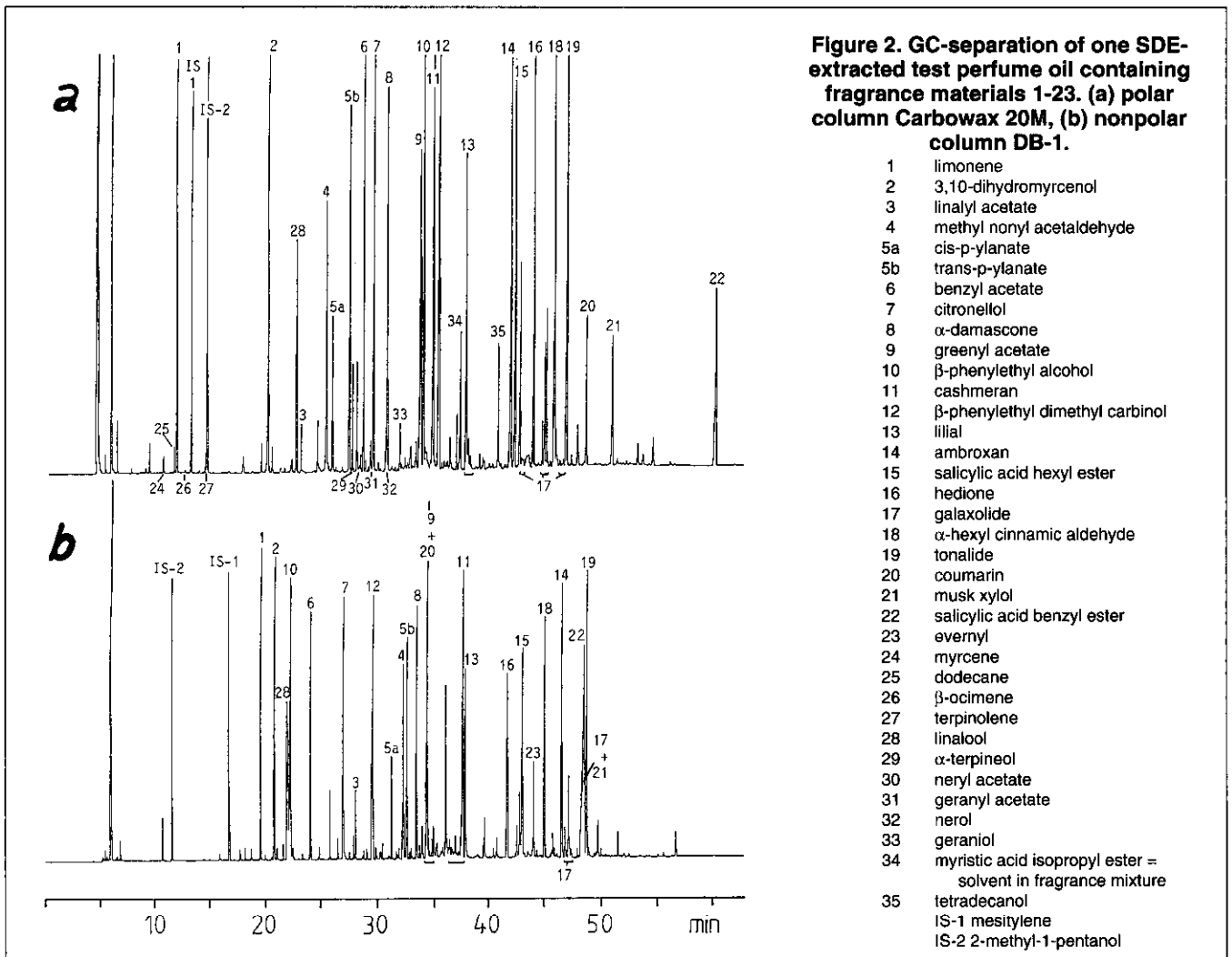
Method No.	Technique	Extracting solvent(s)	Distillation period [hrs]	Salting-out exploitation
1	WSD	hexane	4	-
2	WSD	diethylether	2	-
3	WSD	diethylether	4	-
4	WSD	pentane/diethylether (1:1 vol)	2	-
5	WSD	pentane/diethylether (1:1 vol)	4	-
6	SDE	hexane	4	-
7	SDE	freon 113	4	-
8	SDE	methylene chloride	4	-
9	SDE	methylene chloride	4	Yes
10	SDE	pentane/diethylether (1:1 vol)	2	-
11	SDE	pentane/diethylether (1:1 vol)	4	-
12	SDE	pentane/diethylether (1:1 vol)	4	Yes
13	SDE	pentane/diethylether (1:1 vol)	8	Yes
14	SDE	pentane/diethylether (7:3 vol)	4	-
15	SDE	pentane/diethylether (7:3 vol)	4	Yes

Recovery rates were determined by the internal standard method after the instrument was calibrated with response factors using a standardized solution containing the fragrance materials to be tested and, in addition, the internal standard materials.

## Results and Discussion

The best recoveries were found to be obtained with the simultaneous distillation and extraction method using a pentane/diethylether mixture or methylene chloride as extraction solvent. When a salting-out effect was exploited, recovery rates generally increased. For routine work the simultaneous distillation and extraction seems to be a feasible method for isolating volatile components from perfumed consumer products.

**Solvents:** Eight solvents or solvent mixtures were examined for their extracting



## SEPARATION OF FRAGRANCE MATERIALS

efficiencies in various WSD/SDE distillation methods (Table I).

It must be realized that of these eight solvents, ethyl acetate and a mixture of methylene chloride and pentane (1:1 by volume) are not suitable as SDE-extracting solvents (although each would seem to be an excellent extracting agent in liquid/liquid extraction).<sup>14</sup> Use of ethyl acetate can cause some degree of emulsification, slowing the formation of the two phases in the separation area of the SDE apparatus. The methylene chloride/pentane mixture, after coming into contact with the water steam, was observed to separate during the condensation period into two phases, one heavier than water and one lighter than water.

The GC separations of one test perfume oil's extract, using a suitable solvent, on a polar and a nonpolar capillary column are illustrated in Figure 2. On the polar column the peaks are baseline separated to the greatest possible extent. The chosen internal standards are well separated from all the other sample ingredients extracted. Some components cause more than one peak since they are mixtures of several

isomers; their quantification was carried out by peak grouping. Evernyl does not elute from the polar column because of its relatively high polarity; for its quantification the GC runs were repeated on a nonpolar column, although generally this column does not separate as well as the polar column.

All extractions were performed three times and each extract was separated by GC twice for each of the two internal standards, thus each reported value of "percentage recovery" is the average value of twelve individual observations.

Tables II and III give recovery rates of the extracted components from the shower bath, depending on the method and the choice of extracting solvent.

When a single solvent was used as the extractant, methylene chloride gave the best overall recovery with values usually in excess of 75%. In contrast, diethylether exhibited the poorest recovery in this study. Methylene chloride is also reported by several other authors to be a good general-purpose extractant with a low boiling point, higher polarity and a wider range of selectivity, more suitable for a one-step

**Table II. Recovery rates of fragrance materials extracted by both WSD and SDE**  
(See Table I for method parameters and Formula 1 for the shower bath base)

Component	Recovery rate (%)												
	WSD Methods					SDE Methods							
	1	2	3	4	5	6	7	8	10	11	12	13	15
Ambroxan®	56	51	57	64	78	60	85	85	72	95	100	91	81
Benzyl acetate	59	49	48	56	68	55	80	79	70	91	90	85	83
Cashmeran®	60	51	57	70	77	63	87	86	74	100	104	105	83
Citronellol	58	48	52	58	71	58	79	82	71	94	92	87	85
Coumarin	1	2	3	2	5	17	22	32	8	26	63	59	32
α-Damascone	60	49	52	52	73	61	83	83	71	94	98	97	78
3,10-Dihydromyrcenol	60	51	48	58	71	58	94	82	73	93	94	96	90
Evernyl®	74	48	50	46	66	61	83	88	67	71	91	104	83
Galaxolide®	53	45	53	49	64	52	80	85	63	90	95	89	79
Greenyl acetate	58	50	53	60	75	60	87	78	56	89	100	102	91
Hedione®	46	39	52	46	61	54	89	90	64	91	101	107	95
α-Hexyl cinnamic aldehyde	57	51	57	59	75	57	86	88	72	97	96	92	81
Lilial®	52	47	52	54	66	50	73	80	66	78	75	67	62
Limonene	50	40	32	43	51	44	61	60	56	65	62	46	53
Linalyl acetate	13	7	13	11	15	8	12	19	15	15	21	14	8
Methyl nonyl acetaldehyde	48	39	43	48	58	47	71	71	55	70	66	55	57
Musk xylol	43	22	87	78	93	21	76	49	41	88	92	87	66
β-Phenylethyl alcohol	7	14	17	9	15	29	57	77	55	84	96	96	86
β-Phenylethyl dimethyl carbinol	44	39	50	40	56	54	63	70	67	77	73	79	64
Salicylic acid benzyl ester	40	34	43	39	55	34	64	67	47	74	77	93	68
Salicylic acid hexyl ester	56	49	53	61	73	59	84	82	70	94	94	95	78
Tonalide®	58	52	55	62	76	52	89	86	82	99	95	99	85
p-Ylanate	63	51	53	62	75	70	84	81	73	91	96	97	91

® = registered trademark

**Table III. Recovery rates of additional fragrance materials extracted by SDE**  
(See Table I for method parameters and Formula 1 for the shower bath base)

Component	Recovery rate (%)					
	6	8	9	11	12	14
Ambrettolide®	78	74	65	90	95	90
p-Anis aldehyde	69	84	83	81	81	73
Brahmanol®	87	72	76	90	93	94
Caproic acid allyl ester	56	57	65	78	79	75
Cedramber®	84	89	87	79	98	83
Celestolide®	93	78	78	85	88	93
Cyclamen aldehyde®	88	73	78	94	89	93
Cyclogalbanate®	80	81	85	81	90	83
Cyclohexylpropionic acid allyl ester	82	84	84	87	96	86
Cyclopentadecanolide	73	89	85	63	93	83
9-Decenol	79	70	75	91	92	89
Delphone®	72	64	72	87	95	85
Dimethyl benzyl carbinyl acetate	82	84	83	82	92	86
Diphenyl ether	81	82	82	84	94	84
γ-Dodecalactone	58	74	81	68	69	62
Dodecanal	78	80	80	76	89	82
Ethylene brassylate (Musk T®)	24	33	39	34	33	33
Ethyl vanillin	<1	4	9	3	1	2
Eugenol	83	72	75	92	93	92
Florazon®	80	66	70	94	97	93
Geranyl nitrile	74	64	73	88	96	85
Helional	58	70	80	68	63	52
Heliotropine	51	82	89	71	67	52
cis-3-Hexenol	20	47	54	63	68	58
cis-3-Hexenyl acetate	43	53	63	69	73	67
Hivertal®	57	58	68	75	82	73
α-Ionone	92	90	89	91	100	93
Isobornyl acetate	73	64	73	86	96	86
Isodamascone®	81	63	72	90	95	87
Madrol®	85	90	87	87	95	88
Methyl-β-naphthyl ketone	57	66	70	79	74	75
Musk ketone	35	45	47	42	49	48
γ-Nonalactone	70	72	73	84	86	80
Phenoxyacetic acid allyl ester	50	52	64	51	56	50
Phenylacetaldehyde dimethyl acetal	70	78	77	76	86	79
α-Pinene	35	30	36	37	47	52
Rose oxide	68	72	72	71	82	79
Salicylic acid cis-3-hexenyl ester	83	89	86	85	95	86
Sandranol®	87	91	88	85	94	92
Tetrahydro linalool	72	75	75	78	89	84
Timberol®	71	65	70	68	80	77
10-Undecenal	75	80	81	80	88	81
Vanillin	<1	2	3	<1	<1	1
Veloutone®	81	83	81	86	92	87
Vertral®	75	64	71	88	96	85
Veticol acetate®	94	80	85	98	94	91
Vetival®	78	66	73	89	98	89
o-Ylanate	81	82	81	89	92	86

® = registered tradename

isolation of several volatile components belonging to different chemical classes. The binary system of pentane and diethylether, which gave all in all better recoveries for most of the components than methylene chloride (methods 8 and 10, 9 and 11), was also found to be a good extractant. The ratio of the mixture seemed to be important. The change from a 1:1 ratio to a 7:3 ratio (pentane to diethylether) reduced the extraction efficiency recognizably (methods 12 and 15).

**Distillation technique:** When comparing SDE and WSD, the two distillation techniques, it has to be emphasized that SDE gives better recoveries than WSD, all other factors being constant (methods 1 and 6, 4 and 10, 5 and 11). This is particularly true with the more polar and higher boiling components like, for example, β-phenylethyl alcohol and coumarin. The advantage of SDE is the continuous refreshing of the extractant by keeping it in a distillation/condensation cycle leaving the extracted components in the organic solvent flask of the apparatus. Therefore, low boiling, water-immiscible solvents such as methylene chloride (b.p. 40°C) or pentane/diethylether (1:1; b.p. about 34°C) are more suitable than the other organic solvents. These solvents also simplify the concentration of the extracts.

The reproducibility of these methods (including WSD/SDE and HRGC) is indicated in percent relative standard deviations of the calculated percent recovery values which vary from 3% to 20% with the exception of data calculated for musk xylol which can have a wider range of values as high as 40%. This is probably due to its higher GC/FID-detection limit, which results in a more difficult evaluation of the chromatograms.

**Salting out:** An enormous salting-out effect on the recovery could be obtained by adding inorganic salts, such as sodium chloride, to saturate the aqueous solution. When this procedure was used, especially polar components like β-phenylethyl alcohol,

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coumarin, or evernyl showed less affinity for the water phase; therefore improved extraction was observed (methods 11 and 12).<sup>9</sup> Most of the investigated components could then be recovered with recovery rates greater than 90%.

**Distillation period:** Increasing the extraction period improved the efficiency (methods 2 and 3, 4 and 5, 10 and 11, 12 and 13). Particularly, the recoveries of oxygenated, polar and higher boiling components were positively influenced.<sup>13,15,16</sup> However, excess extraction time could result in slight loss of components with a lower boiling point; an example is limonene or pinene. Some comparable findings are presented by other research groups.<sup>3,4,9</sup>

Increased extraction time boosted thermal degradations or interactions of components with a consequent increase in artifact formation. This could be observed with the thermally unstable linalyl acetate for which the recovery rate was only about 20%. This is in good agreement with other published results.<sup>17</sup> The main artifacts formed from linalyl acetate were linalool and  $\alpha$ -terpineol.

The chromatograms in Figure 2 give an overview of both the artifacts and the main co-extracted sample matrix components. Detailed information on the mechanisms of hydration and rearrangement reactions of linalyl acetate, with a list of artifacts which can be formed, are given for example by Morin and Schmaus.<sup>17,18</sup>

Dodecane and tetradecanol were the main co-extracted components which originated from the model sample matrix used in this experiment.

Determination of vanillin and ethyl vanillin failed using these distillation methods because these fragrance materials do not seem to be water/steam-evaporatable. This results in poor recoveries.

## Conclusion

The data presented demonstrate the usefulness of the SDE-method for isolation of fragrance materials from cosmetic products. These volatile components can be isolated with very acceptable recovery rates. Obviously, this will be generally the same for the isolation of fragrance materials from household products; however, in this case more precautions must be taken because of the reactive or aggressive ingredients that are sometimes present in household products. These ingredients can provoke and support artifact formation.

Normal WSD gave recovery rates not nearly as good as the SDE. All in all, SDE has been proved to be a good method, suitable as a routine method, although a single set of operating conditions was not found for optimum recovery of all components.<sup>10</sup> As previously mentioned, a compromise is needed in order to isolate complex mixtures like perfume oils, particularly when a one-step distillation technique is used.

The limitations of SDE are, for example, demonstrated by the problems in distilling linalyl acetate, vanillin and ethyl vanillin. Further improvements of the SDE method to overcome some of these problems can be achieved by

carrying out SDE under vacuum at about 37°C in the sample flask.<sup>6</sup> Results indicate that linalyl acetate is not modified using vacuum SDE.

Other precautions can be taken by using antioxidants, by purging the SDE apparatus with an inert gas prior to distillation to suppress oxidation reactions, or by additional buffering of the neutralized sample solution/dispersion.<sup>4,6</sup>

Another aspect should be mentioned. Most of the cosmetic or household products tend to foam up during distillation, necessitating an efficient antifoaming agent/system. Unfortunately, the agent, such as the silicone antifoaming agent used in this experiment, may contain recoverable quantities of volatile ingredients that sometimes results in overlapping peaks that spoil the chromatograms. Perhaps this agent can be quantity-reduced or substituted by other materials such as magnesium stearate, for example. Other test runs were carried out using a high-grade steel grid between the sample flask and the SDE apparatus to break the foam.<sup>19</sup> Systematic investigations of further improvements of this method are not yet finished; results will be presented at a later date.

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Address correspondence to Dr. A. Bartsch, Dragoco Gerberding & Co GmbH, Research Department, D-W-3450 Holzminden, Germany.

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