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Direct Sampling Capillary GLC Analysis of Flavor Volatiles from Ovine Fat

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A rapid, direct sampling capillary gas chromatographic procedure for the quantitation of volatiles from lamb fat was developed. Following trapping of volatiles by heating 300 mg of fat containing an internal standard (2-methyl-3-octanone), volatiles were eluted in an external sampling device onto a SE-54 coated capillary column, separated, and identified by mass spectrometry. Fifty-two volatile compounds were quantitated by GC. Accuracy and precision were determined on selected compounds. Average recovery of these compounds was 95.4% and the average relative standard deviation was 6.29%.

Although lamb is potentially an excellent source of meat that can be finished on different forages, it has low acceptance in many countries due to undesirable flavor. Studies by Wong et al. (1975) established that 4-methyloctanoic acid contributed to mutton flavor and suggested that nonacidic fractions might also contribute to mutton odor.

Nixon et al. (1979) published information on 93 nonacidic volatiles from cooked mutton identified by GC/MS. Sample preparation involved refluxing minced meat for 3 h, steam distillation, extraction in a continuous ether extraction apparatus (Likens and Nickerson), and removal of acidic compounds with Na_2CO_3.

A similar study was published by Lorenz et al. (1983) on volatile compounds from sheep liver. These investigators identified 108 compounds from 35 kg of lamb liver. Extensive sample fractionation was necessary prior to identification of volatiles by GC/MS.

Simple quantitative chemical methodologies are essential for further studies of the influence of various factors including diet on the flavor volatiles of lamb and similar meat animals.

Clark and Cronin (1975) and Cronin (1982) described direct sampling procedures for trapping volatiles on charcoal packed into a glass capillary tube and eluting onto a capillary GC column by heating in an injection port at 260 °C.

Galt and MacLeod (1984) trapped volatiles from beef in Tenax contained in a 20 cm long × 4 mm id tube cooled in dry ice, then desorbed heating at 250 °C, and flashed than to a packed column with N_2.

The innovative direct GC method of Dupuy et al. (1976) for the analysis of volatiles related to undesirable oil flavor was an important contribution to methodology needed to relate chemical constituents of food products to acceptability. This procedure was improved for packed column GC by the more versatile procedure of Legendre et al. (1979). Both methods are limited by the inefficiency of the packed column for separating complex mixtures of volatile compounds such as those from animal fat.

Recently developed fused silica capillary columns are much more efficient for separating the complex mixture of hydrocarbons, ketones, alcohols, aldehydes, acids, esters, and lactones found in these samples.

In this communication we report on a new, rapid, quantitative direct sampling procedure for analyzing ovine depot fat for volatiles that can be used to study the influence of different factors on flavor.

EXPERIMENTAL SECTION

Lamb Fat Samples. Two fat samples were analyzed in these experiments; one was taken from the loin of a lamb

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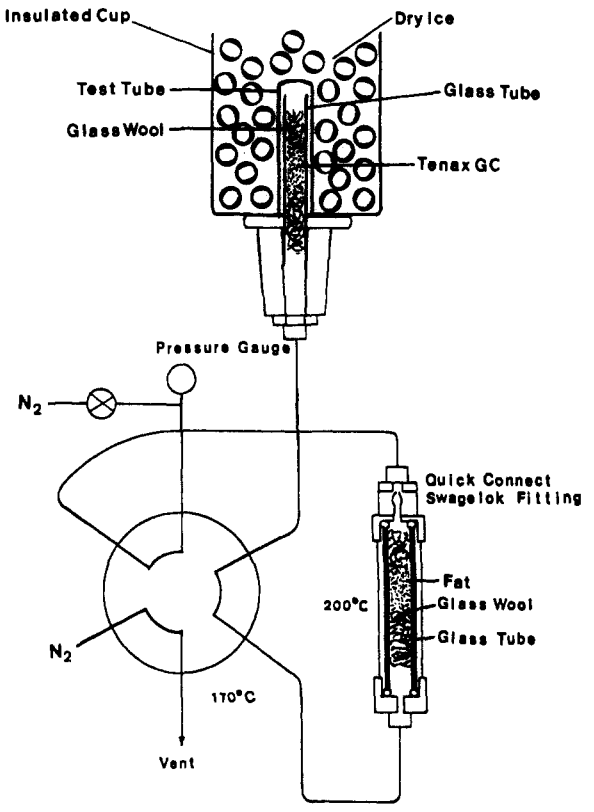

Figure 1. Apparatus used for removing and trapping volatiles from fat.

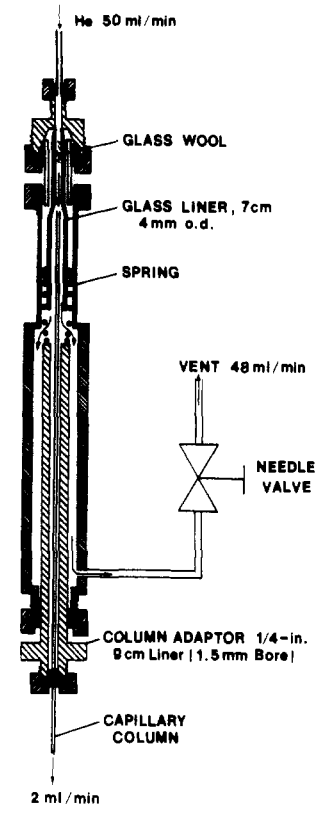

Figure 2. Modified packed column injection port to facilitate direct sampling capillary chromatography.

finished on clover (*Trifolium repens*) at Massey University, Palmerston North, New Zealand, and the other was from the loin of a lamb finished on corn at the University of Missouri, Columbia, Missouri.

Approximately 200 g of loin subcutaneous fat was melted in a 110 °C oven for 2 h as soon as samples were received. The melted fat was immediately transferred to 10 × 130 mm screw cap tubes with teflon liners. The tubes were flushed with nitrogen gas, capped tightly, and stored at −20 °C until GC analysis. The samples were melted in a 60 °C water bath prior to analysis.

Direct Sample Analysis. Volatiles were removed from 300-mg samples of fat by heating in a Pyrex tube (9 × 90 mm) in the apparatus shown in Figure 1. The lower $^2/_3$ of the tube was packed with volatile-free silanized glass wool, and the sample was added and covered with glass wool ($^1/_4$ of tube). Exactly 10 μL of pentane containing 1.40 μg of 2-methyl-3-octanone was injected into the fat sample. The volatiles were removed at 200 °C for 30 min with a nitrogen flow of 50 mL/min and trapped in another tube (9 × 90 mm) on 100 mg of Tenax GC (80/100 mesh) supported on glass wool. The trap was cooled with dry ice.

The cold Tenax trap was immediately transferred to a second direct sampler similar to that described by Legendre et al. (1979). This sampler was connected to a modified injection port (Figure 2) used as a sample splitter which in turn was connected to a capillary column in the gas chromatograph. The cap of the inlet port (Figure 3) was tightened securely with the six-port valve in the "purge" position. The valve was then turned to an intermediate position between "purge" and "run" to prevent loss of volatiles. The quick connect Swagelok fitting was then connected and the valve turned to the "run" position to initiate the GC program.

Quantitative Analysis by GLC. A 50-m fused silica capillary column (0.32 mm id) coated with SE-54 was used to separate volatiles transferred to the column through the splitter injection port (Figure 2). The split ratio was adjusted to 1:24 with carrier gas at 25 psi. The linear flow rate was 41.7 cm/s, the column flow was 2.0 mL/min, and the split purge was 48 mL/min. The injection port temperature was 200 °C. The column temperature was maintained at 35 °C for 5 min and programmed at 8 °C/min to 200 °C and then at 2 °C/min to 250 °C. The gas chromatograph used was a Perkin Elmer Sigma 2 equipped with a FID detector heated to 265 °C.

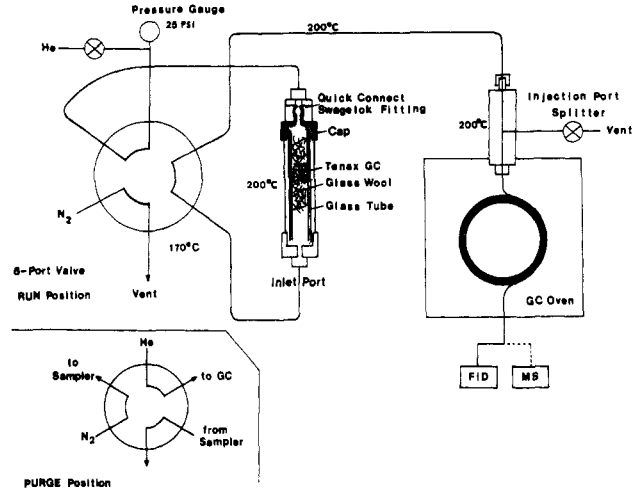

Figure 3. Direct sampling inlet and capillary column for trapped volatile analysis.

Quantitation was accomplished by using an internal standard (2-methyl-3-octanone) and a Perkin Elmer Laboratory Integrator LCI-100. Response factors relative to the internal standard were determined on authentic standards purchased from Aldrich Chemical Co. (Milwaukee), Alfred Bader (Milwaukee), and K & K Laboratories (New York) or received as a gift from Warner-Jenkins, Inc. (St. Louis). Where authentic standards were not

Table I. Volatile Compounds Found in Lamb Fat by GC/MS Analysis

peak no.	compd	Kovats index[a]	av concn, ppm[b] clover-fed	av concn, ppm[b] corn-fed	ID[c]
		Hydrocarbons			
4	heptane	700	<0.05	<0.05	MS
6	octane	800	<0.05	<0.05	MS
20	dodecane	1200	<0.10	0.12 ± 0.01	MS
24	tridecane	1300	<0.10	0.13 ± 0.02	MS
28	tetradecane	1400	<0.05	<0.10	MS
36	hexadecane	1600	<0.10	0.93 ± 0.08	MS
42	octadecane	1800	0.82 ± 0.24	1.19 ± 0.17	MS
		Terpenoids			
10	α-pinene[d]	890	<0.10	0.00	MS
41	phyt-1-ene	1795	1.27 ± 0.17	0.52 ± 0.09	$MS^{P,1}$
44	phytane	1810	0.33 ± 0.14	<0.10	$MS^{N/E}$
45	neophytadiene[d]	1840	4.97 ± 1.13	0.39 ± 0.25	$MS^{P,2}$
46	phyt-2-ene	1853	8.58 ± 1.88	0.82 ± 0.20	$MS^{P,1}$
47	phytadiene (M_r 278)[d]	1865	0.26 ± 0.04	<0.05	(MS)
51	phytol[d]	2130	0.30 ± 0.09	<0.05	MS
		Aldehydes			
3	pentanal	685	<0.05	0.00	MS
5	hexanal	798	0.63 ± 0.22	0.79 ± 0.21	MS
8	heptanal	905	1.43 ± 0.51	0.26 ± 0.07	MS
9	2,4-hexadienal	910	1.16 ± 0.61	<0.10	$MS^{N/E}$
12	octanal	1010	0.47 ± 0.12	0.15 ± 0.02	MS
17	nonanal	1110	0.39 ± 0.09	0.40 ± 0.06	MS
18	2-nonenal	1160	1.14 ± 0.52	0.32 ± 0.07	MS
21	2-decenal	1260	0.93 ± 0.24	0.76 ± 0.14	MS
25	2,4-decadienal	1325	0.54 ± 0.11	0.66 ± 0.29	MS
26	2-undecenal	1360	1.00 ± 0.22	0.63 ± 0.11	MS
		Ketones			
11	2,3-octanedione	913	107.61 ± 14.61	<0.05	MS
16	3-hydroxyoctan-2-one	1105	14.78 ± 1.78	<0.05	$MS^{P,1}$
23	2-undecanone	1296	0.31 ± 0.10	0.24 ± 0.02	MS
30	2-tridecanone	1503	0.73 ± 0.23	1.20 ± 0.11	MS
38	2-pentadecanone	1700	3.65 ± 0.44	3.81 ± 0.32	(MS),(Rt)
43	2-hexadecanone	1804	0.40 ± 0.09	0.67 ± 0.07	(MS),(Rt)
48	2-heptadecanone	1910	6.27 ± 1.70	6.20 ± 0.63	(MS),(Rt)
		Acids			
1	acetic acid	660	1.60[e]	0.41[e]	MS
2	propionic acid	680	<0.05[e]	0.00	MS
7	pentanoic acid	900	0.55[e]	0.00	MS
12	hexanoic acid	1000	1.33[e]	0.20[e]	MS
14	heptanoic acid	1070	0.60[e]	<0.10[e]	MS
19	octanoic acid	1170	0.60[e]	0.30[e]	MS
22	nonanoic acid	1267	1.17[e]	0.30[e]	MS
27	decanoic acid	1400	2.48[e]	1.46[e]	MS
35	undecanoic acid	1560	0.70[e]	0.73[e]	MS
40	dodecanoic acid	1755	0.63[e]	0.29[e]	MS
		Lactones			
31	δ-decalactone	1530	0.23 ± 0.05	0.32 ± 0.13	MS
37	γ-dodecalactone	1695	<0.10	2.07 ± 0.22	MS
39	δ-dodecalactone	1720	0.23 ± 0.05	0.61 ± 0.07	MS
49	δ-tetradecalactone	1970	1.49 ± 0.65	1.77 ± 0.37	MS
50	δ-pentadecalactone	2050	0.25 ± 0.09	0.18 ± 0.02	(MS),(Rt)
52	δ-hexadecalactone	2190	1.19 ± 0.44	1.27 ± 0.22	$MS^{P,1}$
		Others			
15	unknown 1	1090	0.64 ± 0.04	<0.05	
29	unknown 2	1455	0.52 ± 0.10	0.26 ± 0.04	
32	BHT	1534	0.32 ± 0.05	1.07 ± 0.05	MS
33	unknown 3	1540	0.24 ± 0.03	<0.05	
34	unknown 4	1550	0.57 ± 0.12	0.00	

[a] Kovats indices were determined by using a series of hydrocarbons on the fused silica column (SE-54) described under the Experimental Section. [b] Average of six determinations. [c] MS (complete spectrum) and RT data were consistent with that of authentic compounds unless specified as follows: MS^P = MS data were consistent with published spectra, namely, (1) Nixon et al. (1979) and (2) Urbach and Stark (1975). $MS^{N/E}$ = tentatively identified from NIH/EPA data. (MS) = tentatively identified from only interpretation of MS data. (Rt) = RT is consistent with retention predicted from homologous compounds. [d] First time identified in lamb. [e] Estimated by triangulation.

available, compounds of similar functional group and molecular weight were used for determining the relative weight response (RWR) compared to the internal standard. A value of 1 was used as RWR of unidentified volatile compounds. The recovery of selected compounds was accomplished by dissolving samples in Crisco oil and analyzing by direct sampling.

Qualitative Analysis by GLC/MS. The sampling procedure used for identifying volatiles by GLC/MS was the same as that described above except that the gas

Table II. Mass Spectra of Unidentified Compounds

peak no.	mass spectrum, m/e
15	43 (100), 87 (27), 41 (18), 99 (18), 55 (17), 71 (16), 57 (15), 142 (15), 73 (10), 100 (8), 113 (8)
29	57 (100), 43 (82), 71 (82), 41 (40), 85 (33), 55 (27), 56 (26), 70 (20), 42 (13), 113 (9), 99 (8), 141 (8), 212 (1)
33	83 (100), 55 (75), 111 (42), 43 (37), 112 (25), 41 (20), 39 (18), 53 (16), 69 (8), 182 (7)
34	55 (100), 124 (57), 137 (57), 54 (33), 41 (24), 39 (24), 81 (23), 53 (20), 96 (13), 82 (12), 69 (11)

Table III. Standard Deviation and Recovery of Selected Compounds[a]

compd	I_K[b] (SE-54)	added, μg	found, μg	SD,[c] μg	RSD,[d] %	recovery, %
2,3-hexanedione	790	1.400	1.438	0.074	5.15	102.71
2-nonanone	1095	1.731	1.603	0.054	3.37	92.56
2-nonenal	1160	1.471	1.379	0.047	3.41	93.77
2-undecanone	1296	1.404	1.448	0.089	6.15	103.13
2,4-decadienal	1325	1.604	1.563	0.086	5.50	97.44
pristane[e]	1705	1.318	1.215	0.091	7.51	92.19
octadecane	1800	1.405	1.208	0.156	12.92	86.00
			av		6.29	95.40
δ-tetradecalactone	1970	1.755	0.320	0.103	32.16	18.23

[a] Average of six determinations of 200 mg of Crisco oil containing standards. [b] I_K (Kovats index). [c] SD (standard deviation). [d] RSD% (relative standard deviation). [e] Pristane (2,6,10,14-tetramethylheptadecane).

chromatograph used was a Carlo Erba 41-60 and the sample split ratio was 1:10 through the conventional splitter. The SE-54 capillary column was connected to the mass spectrometer through a jet (Ryhage) separator. The double-focussing mass spectrometer (Kratos MS-25) was equipped with a Kratos DS-55 data system with a NIH/EPA data base (NIH/EPA Chemical Information System, 1978) for library search. The ionization voltage was set at 70 eV, ion source temperature at 250 °C, and the resolution was 600 (10% valley).

2,3-Octanedione Synthesis and Characterization. 2,3-Octanedione was synthesized from 2-octene by using the method described by Sharpless et al. (1971). The resulting yellow oily mixture was purified by preparative TLC on silica gel with $CHCl_3$ as solvent. The infrared spectrum of thin liquid film was recorded with a Perkin-Elmer 710 spectrophotometer and the retention index and mass spectral data were obtained by the method described previously. ^1H NMR spectrum was obtained on a 300-MHz Nicolet NT-300 NMR spectrometer. Sample was run in $CDCl_3$ with $(CH_3)_4Si$ as an internal reference standard. Spectral data of the isolated compound are as follows: Kovats retention index $K_{I(SE-54)}$ 913; IR 2975, 2945, 2890, 1715, 1355, 915, 735 cm^{-1}; MS, m/z (relative intensity) 142 (M^+, 1), 99 (20), 71 (18), 55 (15), 43 (100), 42 (9), 41 (27), 39 (15); ^1H NMR ($CDCl_3$, 300 MHz) δ 2.73 (2 H, t, J = 7 Hz), 2.34 (3 H, s), 1.2–1.7 (6 H, m), 0.89 (3 H, t, J = 7 Hz).

RESULTS AND DISCUSSION

Representative chromatograms from analysis of the two lamb samples are given in Figure 4. Peak numbers refer to the compounds listed in Table I which has the identities of the volatile constituents analyzed by GC/MS and their concentrations. Identification of unknown compounds was achieved by comparing MS and retention time (RT) data with that of authentic compounds. Where the authentic compounds were not available, unknowns were tentatively identified from MS data of published reference spectra and/or NIH/EPA reference spectra (NIH/EPA Chemical Information system, 1978). Many of these compounds are the same as those identified by Nixon et al. (1979) from cooked mutton mince following extraction of the volatiles in a Likens–Nickerson apparatus for 4 h.

Fifty-two of these compounds were quantitated for fat samples from lambs finished on corn and clover. The outstanding difference between the volatiles from fat of the animal finished on clover compared to those of the

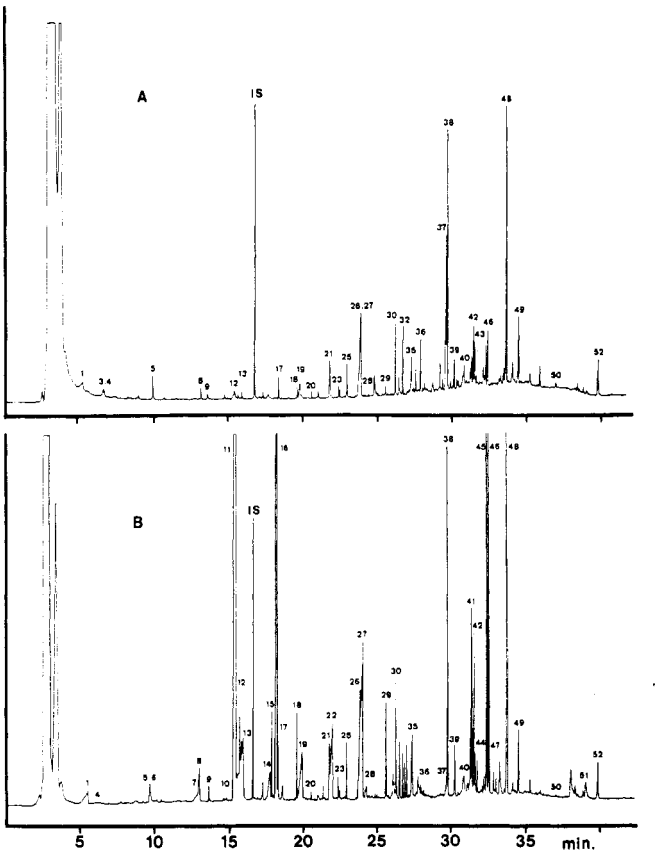

Figure 4. Chromatograms of volatiles from fat of corn-fed (A) and clover-fed (B) lamb.

animal finished on corn was the very high contents of 2,3-octanedione (108 ppm) and 3-hydroxyoctan-2-one (15 ppm) found in the former samples. These compounds were first identified in cooked mutton mince by Nixon et al. (1979).

Another important difference in the volatiles from the two types of samples was the relatively high concentrations of diterpenoids present in the samples of animals finished on clover. Phyt-1-ene (3,7,11,15-tetramethyl-1-hexadecene) and phyt-2-ene (3,7,11,15-tetramethyl-2-hexadecene) were first identified in mutton by Nixon et al. (1979), and phytane (2,6,10,14-tetramethylhexadecane) was reported by Lorenz et al. (1983). Urbach and Stark (1975) found diterpenoids including the above terpenoids and neo-

phytadiene (7,11,15-trimethyl-3-methylene-1-hexadecene) in butter fat. However, the presence of neophytadiene in lamb fat has not been reported previously. These compounds are apparently formed from the fermentation of phytol of chlorophyll by rumen microorganisms (Body, 1977).

Heptanal, octanal, and 2-nonenal were also higher in the sample from clover-fed lamb.

Table II is a list of the mass spectral data of unidentified compounds which were present in relatively high concentrations in fat from a lamb finished on clover. Peak no. 15 is probably a tautomer of 2,3-octanedione and its molecular weight was 142. Peak no. 29 was probably a C-15 branched hydrocarbon (M_r 212).

2,3-Octanedione was the most prominant volatile identified in fat from clover-fed lamb. This compound appears to be a good marker compound for forage-fed lamb, although the odor of the diketone is "fruity" and not "grassy".

Precision and accuracy were determined by adding standards to Crisco oil and analyzing 200-mg samples by this method. Results of the recovery for selected compounds are summariezed in Table III. The average recovery of the seven compounds was 95.4% and the relative standard deviation (RSD) was 6.29%. The method was accurate and precise for these compounds. Recovery for octadecane was relatively low (86%) and the RSD (13%) was relatively high. However, this type of precision would be acceptable for the analysis of low volatile compounds by this method. δ-Tetradecalactone eluted following 2-pentadecanone was not quantitated by this method. This was probably due to the low volatility of the compound and the inlet temperature of the direct sampler (200 °C) was insufficiently high to volatilize the compound from the sample. Furthermore, slight temperature changes in the inlet port caused variation in amount of the compound transferred from the sample resulting in poor reproducibility. The method is limited in that it may not be quantitative for high molecular weight–low volatile compounds such as long chain lactones.

This procedure is presently being used to analyze large numbers of samples of fat from lambs and cattle finished on different types of forages and grain.

The direct sampling capillary GLC method described above for the quantitative analysis of volatiles from animal fat should fill an important need in flavor analysis of foods. The sample preparation is simple and rapid and no extraction, distillation, or cleanup procedures are required.

Registry No. Heptane, 142-82-5; octane, 111-65-9; dodecane, 112-40-3; tridecane, 629-50-5; tetradecane, 629-59-4; hexadecane, 544-76-3; octadecane, 593-45-3; α-pinene, 80-56-8; phyt-1-ene, 69382-63-4; phytane, 638-36-8; neophytadiene, 504-96-1; phyt-2-ene, 56554-34-8; phytadiene, 2437-92-5; phytol, 150-86-7; pentanal, 110-62-3; hexanal, 66-25-1; heptanal, 111-71-7; 2,4-hexadienal, 80466-34-8; octanal, 124-13-0; nonenal, 124-19-6; 2-nonenal, 2463-53-8; 2-decenal, 3913-71-1; 2,4-decadienal, 2363-88-4; 2-undecenal, 2463-77-6; 2,3-octanedione, 585-25-1; 3-hydroxyoctan-2-one, 37160-77-3; 2-undecanone, 112-12-9; 2-tridecanone, 593-08-8; 2-pentadecanone, 2345-28-0; 2-hexadecanone, 18787-63-8; 2-heptadecanone, 2922-51-2; acetic acid, 64-19-7; propionic acid, 79-09-4; pentanoic acid, 109-52-4; hexanoic acid, 142-62-1; heptanoic acid, 111-14-8; octanoic acid, 124-07-2; nonanoic acid, 112-05-0; decanoic acid, 334-48-5; undecanoic acid, 112-37-8; dodecanoic acid, 143-07-7; δ-decalactone, 705-86-2; γ-dodecalactone, 2305-05-7; δ-dodecalactone, 713-95-1; δ-tetradecalactone, 2721-22-4; δ-pentadecalactone, 7370-38-9; δ-hexadecalactone, 7370-44-7; BHT, 128-37-0; 2,3-hexanedione, 3848-24-6; 2-nonanone, 821-55-6; pristane, 1921-70-6.

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