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Aroma of Birch Syrup

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The composition of the aroma extract of Finnish birch syrup was studied by gas chromatography and gas chromatography-*EL*-mass spectrometry. A total of 70 volatile compounds were identified. The major compounds were 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one (11-45 mg/kg), acetic acid (4-7), 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone (furanol) (0.8-2.4), 2,6-dimethylpyrazine (0.8-2.3), 2-oxopropanol (0.7-2.0), 3-methyl-2(5*H*)-furanone (0.6-1.6), 2,3-butanediol (0.8-1.0), 4-aminophenol (0.4-0.9), and 2-hydroxy-3-methyl-2-cyclopenten-1-one (cyclozene) (0.2-0.5). When the syrup was heated, several furan and pyran derivatives together with cyclozene typically increased, whereas 2,6-dimethylpyrazine decreased. The total content of the identified aromatic compounds was low.

When carbohydrate-rich foodstuffs are heated, sugar-derived cyclic compounds are found in most of the products, regardless of the origin of raw materials or the unit operations of the processes.

2,3-Dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one, 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone (furanol), and 2-hydroxy-3-methyl-2-cyclopenten-1-one (cyclozene) are all typical heat-accelerated reaction products of reducing sugars, or the mixture of reducing sugars and amino compounds in aqueous media (Mevissen and Baltes, 1983; Hayase and Kato, 1985; Ledl, 1984; Baltes and Mevissen, 1988). The presence of an amino acid does not greatly affect the formation rate of these compounds (Baltes and Mevissen, 1988). For the reaction rates the moisture-temperature conditions are of primary importance.

Furanol is common in heated carbohydrate-rich foodstuffs such as roasted almonds (Takei and Yamanishi, 1974), soy sauce (Osaki et al., 1985), roasted coffee (Tressl et al., 1983), sukiyaki (Shibamoto et al., 1981), beef broth (Tonsbeek et al., 1968), green tea (Hara and Kubota, 1982), liquid smoke (Fiddler et al., 1970), maple syrup (Kallio, 1988), and tobacco volatiles (Matsukura et al., 1985). Because of its exceptionally low odor threshold value, 0.03 $\mu\text{g/L}$ (Honkanen et al., 1980), furaneol seems to be one of the character impact compounds in many foodstuffs.

Besides its direct influence on the odor and aroma of food components, furaneol is a precursor of other volatiles. In a model experiment, when heating furaneol over various

pH values at 160 °C, Shu et al. (1985, 1986) found several compounds being formed such as 3-hydroxy-2-butanone, 2,3-butanedione, 2,3-pentanedione, and 2,5-dimethyl-3(2*H*)-furanone.

Both 2,3-butanedione and 2,3-pentanedione have been identified in heated foodstuffs: sweet potato (Tiu et al., 1985), textured soy protein (Ames and MacLeod, 1984), winged beans and soybeans (del Rosario et al., 1984), cooked meat (Shibamoto et al., 1981), molasses (Godshall et al., 1980), sour cream butter (Mick et al., 1982), and roasted coffee (Wang et al., 1983). The diones were also found in the aroma fraction of yeast extract (Ames and MacLeod, 1985) and Rooibos tea (Habu et al., 1985) when the Likens and Nickerson steam distillation and extraction method (boiling required) or headspace collection followed by thermal desorption of the volatiles from the Tenax trap was used.

Cyclozene is an aroma factor in many of the protein- and carbohydrate-based foodstuffs consumed daily: fried (Ho et al., 1983) and cooked bacon (Shu and Mokherjee, 1985), sukiyaki (Shibamoto et al., 1981), dried bonito (Yajima et al., 1983), soy sauce (Aishima, 1981), malt extract (Przybylski and Kaminski, 1983), "Wasanbon" sugar (Matsui and Kitaoka, 1981), bread crust (Folkes and Gramshaw, 1981), caramel color (Hardt and Baltes, 1987), roasted coffee (Tressl et al., 1983), and roasted tobacco flavor (Matsukura et al., 1985).

Although, 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one is odorless (Shaw et al., 1971), other compounds such as furaneol, may originate from the ring contraction of this pyranone (Mills, 1978), which indicates the importance of pyranone derivatives as aroma precursors of foodstuffs.

Birch syrup is a novel syrup innovation, the commercial production of which started in Finland in spring 1988. Its composition (Kallio and Ahtonen, 1987a,b), production

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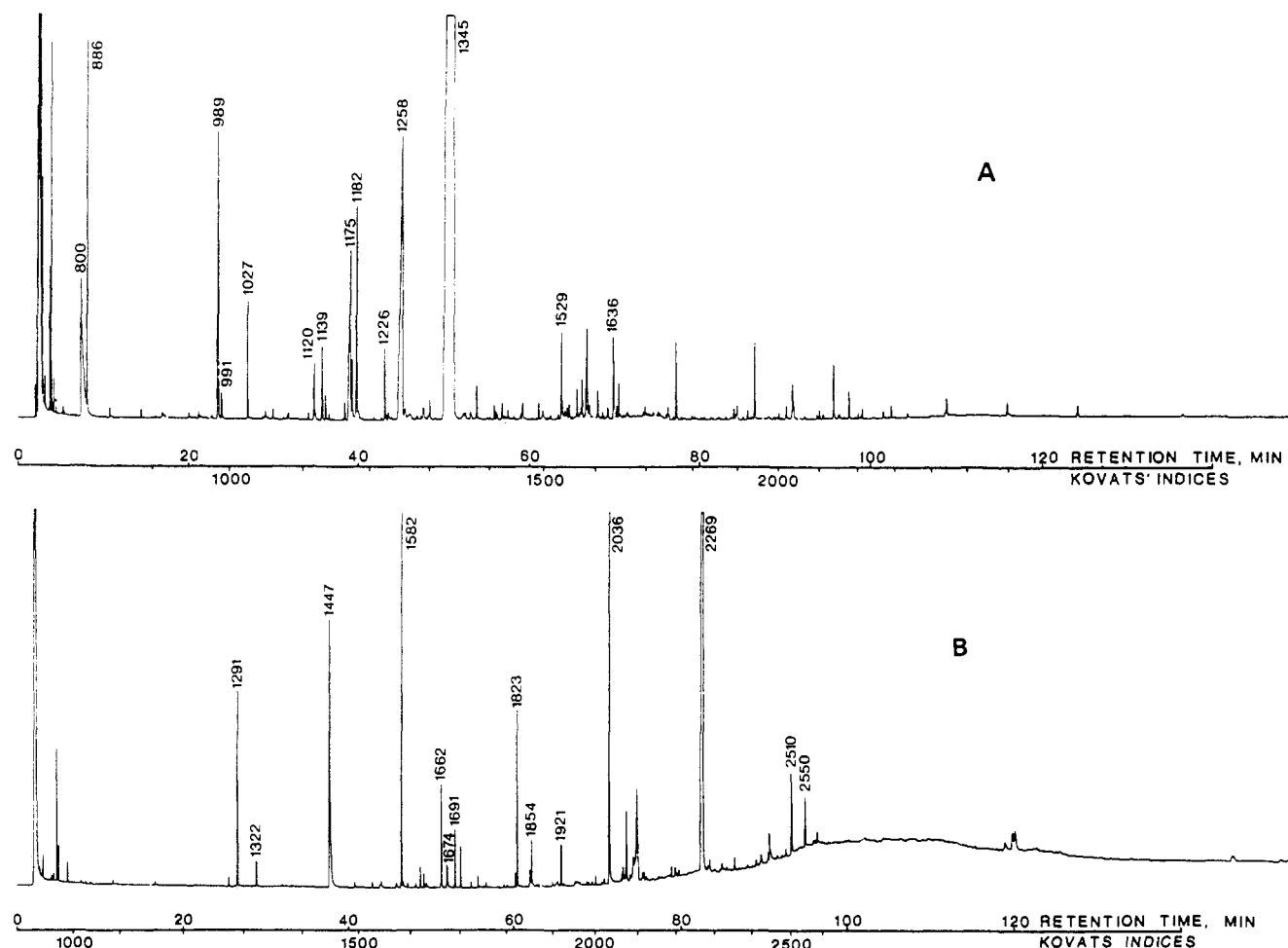


Figure 1. Chromatograms (qualitative analysis, split injection, 5-min heat treatment) of pentane-diethyl ether extract of birch syrup (*B. pubescens*): A, DB-1701; B, DB-WAX. I_K values of some of the major compounds shown on the chromatograms ($I_K(\text{DB-1701})/I_K(\text{DB-WAX})$): acetic acid, 800/1447; 2-oxopropanol, 886/1291; 2,3-butanediol, 989/1582; 2,6-dimethylpyrazine, 991/1322; 2-furanmethanol, 1027/1662; 2,3-butanediol diacetate, 1120/1921; 5-methyl-2(5*H*)-furanone, 1139/1674; 3-methyl-2(5*H*)-furanone, 1175/1691; 2-hydroxy-3-methyl-2-cyclopenten-1-one, 1182/1823; 2,3-butanediol acetate, 1226/1854; 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone, 1258/2036; 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one, 1345/2269; 4-aminophenol, 1529/2510; 4-hydroxy-3-methoxybenzaldehyde, 1636/2550.

(Kallio et al., 1985), and properties (Kallio et al., 1987, 1989) have been studied and discussed earlier in detail. The aim of this study was to investigate the chemical composition of the aroma of Finnish Birch syrup with optimized quality.

MATERIALS AND METHODS

Birch Syrup and Isolation of Its Aroma. Birch syrup (75° Brix, $\text{IU}_{560} = 2\text{--}3 \text{ cm}^{-1} \cdot \text{Brix}^{-1}$) was prepared from the sap of *Betula pubescens* Ehrh. by reverse osmosis and evaporation under reduced pressure with extra heat treatment lasting 0, 5, or 10 min (Kallio et al., 1989).

The aroma fraction was isolated from 60-mL samples at 40 °C by shaking with 5 × 30 mL pentane-diethyl ether (1:2), 5 min each time. The combined extract was dried by passing the solution through a column filled with MgSO_4 . The dried extract was concentrated with a Widmer column and dry nitrogen to a final volume of 500 μL . All isolations were made in duplicate.

Gas Chromatographic Analysis. A qualitative GC analysis was performed on a Varian Aerograph 3400 chromatograph equipped with an FI detector (Walnut Creek, CA) interfaced to a Shimadzu C-R3A integrator (Kyoto, Japan). Two 60-m fused silica capillary columns (J&W Scientific, Folsom, CA) were used: DB-1701, 0.315-mm i.d., d_f 0.25 μm ; DB-WAX 0.256-mm i.d., d_f 0.25 μm . The flow rate of the H_2 carrier gas was 46 cm/s ($t_{R(\text{CH}_4)} = 130.4 \text{ s}$) at a temperature of 32 °C in the DB-1701 column and 48 cm/s in the DB-WAX column. The split ratio of the carrier in the injector was 1:28 for both columns between the column and the exit. The temperature of the DB-1701 column was pro-

grammed from 30 °C after a 10-min hold to 230 °C at the rate of 2 °C/min and that of the DB-WAX column from 40 °C after a 10-min hold to 210 °C at a rate of 2 °C/min. The temperatures of the injector and the detector were the same, 250 °C with the DB-1701 column and 230 °C with the DB-WAX column. The Kovats indices (I_K) were determined by coinjecting the sample with a mixture of saturated *n*-hydrocarbons.

The semiquantitative analyses were performed by on-column injection (J&W Scientific). The aroma concentrate was supplemented with *n*-nonane, *n*-undecane, and *n*-dodecane (1.9 μg each/g syrup), and 0.2 μL of the solution was introduced into a column equipped with a cold trap. The results were not corrected according to the varying detector responses or losses when isolating the aroma because of a deficiency in many of the standard compounds.

Some only partially separated GC fractions of the aroma concentrate were collected on the analytical DB-1701 column. The outlet of the column was connected, via a low dead volume union, to two identical 3-m lengths of another DB-1701 column (0.326-mm i.d., d_f 0.25 μm) with a two-hole graphite ferrule. One of the column ends was connected into the FID, and the other was led through a glass liner out of the oven top. A spout located close to the outlet liner was filled with liquid N_2 . Just prior to elution of the fraction to be collected, 30 cm of the trapping column was pulled out of the oven and placed in liquid N_2 . When the collection was completed, 5 cm more of the trap was pulled out of the oven and cut at the present oven outlet point. A 4-cm length of the front part of the trap was cut to remove the possibly condensed impurities, both ends were then sealed with a rubber

Table I. Aroma Compounds of Birch Syrup (5-min Heat Treatment, *B. pubescens*) Identified with Both DB-1701 and DB-WAX Columns

compound	content, ^a mg/kg	compound	content, ^a mg/kg
Alcohols			
ethanol		2-pentanol	
2-oxopropanol	0.7–2.0 ^b	2,3-butanediol	0.8–1.0
Acids			
acetic acid	4.0–7.0	<i>n</i> -hexanoic acid	
Esters			
ethyl acetate		2,3-butanediol acetate	0.1–0.2
2-oxopropanol acetate		2,3-butanediol diacetate	0.1–0.2
Aldehydes			
2-methylpropanal		1-hexanal	
1-butanal		1-octanal	
2-methylbutanal		1-nonanal	
3-methylbutanal		1-decanal	
1-pentanal		1-undecanal	
Ketones			
acetone		6-methyl-5-hepten-2-one	
3-buten-2-one		2,3-butanedione	
3-hydroxy-2-butanone		2,3-pentanedione	
3-penten-2-one		3-hydroxy-2,4,5-hexanetrione	
Furan Derivatives			
furan		3-phenylfuran	
2-methylfuran		3-methyl-2(5 <i>H</i>)-furanone	0.6–1.6 ^b
2,5-dimethylfuran		5-methyl-2(5 <i>H</i>)-furanone	0.1–0.2
2-furanmethanol	0.2–0.3 ^b	5-methyldihydro-3(2 <i>H</i>)-furanone	
2-furancarboxaldehyde		2,5-dimethyl-3(2 <i>H</i>)-furanone	
2-furancarboxylic acid		2,5-dimethyl-4-hydroxy-3(2 <i>H</i>)-furanone (furanol)	0.8–2.4 ^b
3-furancarboxylic acid	tr–0.1 ^b	4-hydroxy-2-(hydroxymethyl)-5-methyl-3(2 <i>H</i>)-furanone	
5-hydroxymethyl-2-furancarboxaldehyde			
2-furancarboxylic acid methyl ester			
Pyran Derivatives			
2,3-dihydropyran		3,5-dihydroxy-2-methylpyran-4 <i>H</i> -one	
3,4-dihydro-2 <i>H</i> -pyran		2,3-dihydro-3,5-dihydroxy-6-methylpyran-4 <i>H</i> -one	11–45 ^b
3-hydroxy-2-methylpyran-4 <i>H</i> -one (maltol)			
Aromatics			
benzene		4-hydroxy-3-methoxybenzaldehyde	0.1–0.3
phenol		4-hydroxy-3,5-dimethoxybenzaldehyde	
benzaldehyde		1-(4-hydroxy-3,5-dimethoxyphenyl)-ethanone	
benzoic acid		2-aminophenol	
4-hydroxybenzaldehyde	0.1–0.2	4-aminophenol	0.4–0.9
benzeneacetaldehyde			
Others			
diethyl ether		dimethyl trisulfide	
limonene		3-(methylthio)propanal	
pyrid-4(1 <i>H</i>)-one		2-oxohexamethylene imine	
1 <i>H</i> -imidazole-4-carboxylic acid	0.0–0.1	2-hydroxy-3-methyl-2-cyclopenten-1-one (cyclotene)	0.2–0.5 ^b
2,6-dimethylpyrazine	0.8–2.3 ^c		
dimethyl disulfide			

^a Minimum content because of incomplete yield in extraction. ^b Increasing with increasing heat treatment. ^c Decreasing with increasing heat treatment.

septum, and the trap containing the fraction was stored in liquid N₂ for further analyses. Gathering of the other required fractions was performed in the same way. The shortening of the trapping column was taken into account when retention times of the eluting fractions were determined. The fractions were analyzed by connecting a trap at the front part of the DB-WAX column with a low dead volume union after cutting off 1-cm lengths at both ends of the trap (Kallio and Shibamoto, 1988) and analyzed as previously stated.

Mass Spectrometric Analysis. A VG ZAB-2F-HS mass spectrometer (VG Analytical, Wythenshawe, Manchester, U.K.) in combination with a VG 11/250 data system was employed. The conditions of the Hewlett-Packard 5790 gas chromatograph (Palo Alto, CA) connected to the mass spectrometer (EI mode) were the same as in the FID analyses, with one exception. The carrier gas was He flowing at a rate of 35 cm/s. The ionization energy was 70 eV, and the temperature of the ion source 210 °C. The concentrates were analyzed with both DB-1701 and DB-WAX columns and some of the isolated fractions with DB-WAX column.

RESULTS AND DISCUSSION

The birch syrup analyzed was a 75° Brix (calibrated on sucrose) all-natural product made from birch sap by removing most of the water. The syrup contained 60–65% (w/w) sugar, 3% ash, 1% proteins and free amino acids, and 27% water. About 90% of the sugar consisted of glucose and fructose in equimolar ratio (invert sugar) (Kallio et al., 1989).

The reducing sugars and the high content of free amino acids (0.4–0.5%, w/w) make the product extremely susceptible to darkening when heated, and also when stored for longer periods at room temperature. This prevents the possibility of evaporating water at atmospheric pressure, as is used in maple syrup production. During the optimized birch syrup process, a light yellow color and a mild, sweet aroma are, however, obtained by controlled gentle heating.

Table I summarizes 70 volatiles identified by the capillary GC-MS analyses using both DB-1701 and DB-WAX columns. The minimum contents of the major compounds are also listed in Table I. The furan and pyran derivatives, 2-hydroxy-3-methyl-2-cyclopenten-1-one (cyclotene), and the diones are process-borne aroma compounds worthy of further discussion. Figure 1 shows the chromatograms of birch syrup aroma isolate, the major compounds being marked with Kovats indices.

2,3-Dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one and 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone (furanol) were the two main volatiles isolated from birch syrup. The contents of the pyranone derivative and furaneol increased substantially when birch syrup was heated (Table I) and were typically higher than in maple syrup (Kallio, 1988), which contains mainly sucrose instead of invert sugar.

The contents of all the pyran derivatives other than 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one, e.g. 3-hydroxy-2-methylpyran-4*H*-one (maltol) were scanty. This might be due to the low process temperatures (Kallio et al., 1989) because the direct formation of maltol from 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one is a reaction requiring effective heating (Ledl, 1984). On the whole, the formation of maltol and isomaltol (the latter not detected in birch syrup) has been shown to be low in monosaccharide systems (Ledl, 1984).

Because 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one is odorless, the major character-impact compounds have to be detected among the furan derivatives and 2-hydroxy-3-methyl-2-cyclopenten-1-one. Furanol has been found to be a very interesting natural and artificial aroma compound with a sweet, caramel-like tone. In addition to being formed when foodstuffs are heated, it is a natural, biological secondary metabolite in pineapple (Rodin et al., 1965), strawberry (Pickenhagen et al., 1981), arctic bramble (Kallio, 1976), raspberry (Honkanen et al., 1980), mango (Pickenhagen et al., 1981), labrusca grape (Acree, 1981), guava (Idstein and Schreier, 1985), fresh and canned grapefruit juices (Lee and Nagy, 1987), and cheese made from *Lactobacillus helveticus* (Kowalewska et al., 1985).

A total of 16 furan derivatives were identified in the syrup. The contents of furaneol (0.8–2.4 mg/kg), 2-furanmethanol (0.2–0.3), 3-furancarboxylic acid (tr–0.1), and 3-methyl-2(5*H*)-furanone (0.6–1.6) clearly increased when the syrup was heated (Table I).

2,3-Butanedione and 2,3-pentanedione, which might be derived from furaneol (Shu et al., 1985), are evidently not character-impact compounds of the syrup flavor; at most they give some buttery or creamy tone to the product (Tiu et al., 1985).

Cyclotene gave positive aroma to the product, although its content was not higher than 0.2–0.5 mg/kg (Table I). It clearly increased when the syrup was heated, and it probably derived from the reducing sugar, as the conditions for its formation looked to have been favorable. The compound is known to have been formed in various reactions such as in glucose-phenylalanine (Meivissen and Balthes, 1983), glucose-L-cysteine (Sakaguchi, 1984), glucose-glutamic acid (Berry and Gramshaw, 1986), and starch-glycine model systems (Umano and Shibamoto, 1984) and in cellulose pyrolysis (Sakuma et al., 1981).

The compounds discussed above contribute to the typical syrup flavor together with the other volatiles listed in Table I, and with numerous trace components not identified at all. It is of primary importance to control the heat when the syrup is processed since overheating produces bitter taste and off-flavor. The burned aroma of the overheated syrup parallels the content of dimethyl disulfide

(Kallio et al., 1987). It is also possible that the increasing contents of other furan derivatives such as 2-furanmethanol, 3-methyl-2(5*H*)-furanone, and 5-methyl-2(5*H*)-furanone (Table I) may contribute to the burned flavor when the syrup is heated.

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Registry No. Ethanol, 64-17-5; 2-oxopropanol, 116-09-6; 2-pentanol, 6032-29-7; 2,3-butanediol, 513-85-9; acetic acid, 64-19-7; *n*-hexanoic acid, 142-62-1; ethyl acetate, 141-78-6; 2-oxopropanol acetate, 592-20-1; 2,3-butanediol acetate, 56255-48-2; 2,3-butanediol diacetate, 1114-92-7; 2-methylpropanol, 78-84-2; 1-butanol, 123-72-8; 2-methylbutanol, 96-17-3; 3-methylbutanol, 590-86-3; 1-pentanol, 110-62-3; 1-hexanol, 66-25-1; 1-octanol, 124-13-0; 1-nonanol, 124-19-6; 1-decanol, 112-31-2; 1-undecanol, 112-44-7; acetone, 67-64-1; 3-buten-2-one, 78-94-4; 3-hydroxy-2-butanone, 513-86-0; 3-penten-2-one, 625-33-2; 6-methyl-5-hepten-2-one, 110-93-0; 2,3-butanedione, 431-03-8; 2,3-pentanedione, 600-14-6; 3-hydroxy-2,4,5-hexanetrione, 4384-08-1; furan, 110-00-9; 2-methylfuran, 534-22-5; 2,5-dimethylfuran, 625-86-5; 2-furanmethanol, 98-00-0; 2-furancarboxaldehyde, 98-01-1; 2-furancarboxylic acid, 88-14-2; 3-furancarboxylic acid, 488-93-7; 5-(hydroxymethyl)-2-furancarboxaldehyde, 67-47-0; 2-furancarboxylic acid methyl ester, 611-13-2; 3-phenylfuran, 13679-41-9; 3-methyl-2(5*H*)-furanone, 22122-36-7; 5-methyl-2(5*H*)-furanone, 591-11-7; 5-methyldihydro-3(2*H*)-furanone, 34003-72-0; 2,5-dimethyl-3(2*H*)-furanone, 14400-67-0; furaneol, 3658-77-3; 4-hydroxy-2-(hydroxymethyl)-5-methyl-3(2*H*)-furanone, 17678-20-5; 2,3-dihydropyran, 3174-74-1; 3,4-dihydro-2*H*-pyran, 110-87-2; maltol, 118-71-8; 3,5-dihydroxy-2-methylpyran-4*H*-one, 1073-96-7; 2,3-dihydro-3,5-dihydroxy-6-methylpyran-4*H*-one, 28564-83-2; benzene, 71-43-2; phenol, 108-95-2; benzaldehyde, 100-52-7; benzoic acid, 65-85-0; 4-hydroxybenzaldehyde, 123-08-0; benzeneacet-aldehyde, 122-78-1; 4-hydroxy-3-methoxybenzaldehyde, 121-33-5; 4-hydroxy-3,5-dimethoxybenzaldehyde, 134-96-3; 1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone, 2478-38-8; 2-aminophenol, 95-55-6; 4-aminophenol, 123-30-8; diethyl ether, 60-29-7; limonene, 138-86-3; pyrid-4(1*H*)-one, 108-96-3; 1*H*-imidazole-4-carboxylic acid, 1072-84-0; 2,6-dimethylpyrazine, 108-50-9; dimethyl disulfide, 624-92-0; dimethyl trisulfide, 3658-80-8; 3-(methylthio)propanal, 3268-49-3; 2-oxohexamethylene imine, 105-60-2; cyclotene, 80-71-7.

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