

# Chemical and Sensory Properties of Liquid Beet Sugar

Per Pihlsgård,<sup>\*,†</sup> Mats Larsson,<sup>‡</sup> Anders Leufvén,<sup>†</sup> and Hans Lingnert<sup>†</sup>

SIK, Swedish Institute for Food and Biotechnology, P.O. Box 5401, SE-402 29 Göteborg, Sweden, and Danisco Sugar AB, SE-205 04 Malmö, Sweden

Sensory analysis and headspace gas chromatography–flame ionization detection (GC–FID) analysis of liquid sugars (sucrose) designated for food–industrial use were performed in order to elucidate which compounds are responsible for perceived off-odors and off-flavors in liquid sugar. The liquid sugar samples investigated had various kinds of off-odors and off-flavors that might influence the quality of any food product in which they might be used, such as a jam or a soft drink. The sensory scores for the different attributes and the levels of different volatile compounds as measured by GC were analyzed for correlation using partial least-squares (PLS) analysis. A link between sensory analysis and GC analysis was thereby established, and compounds critical for the odor and flavor were identified using MS (mass spectrometry). Approximately 10 compounds were found to be correlated with the defined sensory attributes. Among these compounds associated with the sensory attributes were dimethyl disulfide, 2,6-dimethylpyrazine, 4-methoxyphenol, and 2,5-dimethylfuran.

**Keywords:** Beet sugar; sensory analysis; GC–MS; flavor; chemometrics; PLS

## INTRODUCTION

The use of liquid sugar instead of crystal sugar has proved successful in many industrial applications as this product is easier to handle and it is easier to maintain its quality. Liquid sugar can be manufactured by dissolving white crystal sugar in water or by using process syrups from the refinery. In the latter process, less total crystallization is needed to obtain the final product. The use of this approach makes the overall process of sugar manufacturing more efficient since the total loss of sugar during processing is reduced. However, different food applications have different purity requirements, and since the odor and flavor intensity of the less crystallized sugar is higher, unwanted odors and flavors may be experienced in some applications. It is thus very important to be able to maintain the odor and flavor at levels adjusted to the different requirements of different food applications.

There are many different causes for the off-odors and off-flavors in different production streams in a sugar refinery. The components found in beet sugar products can, according to Godshall et al. (1995), be divided into three main groups: metabolites of the beet itself, compounds formed in the sugar beet as a consequence of microbial activity in the soil and of compounds that are sorbed by the beet during growth, and finally, compounds formed during storage or in the process of sugar manufacturing.

Little, if anything has been published on odors and flavors of liquid sugar; however, research on crystal sugar, molasses, and beets has been carried out. Godshall (1986) studied flavors from beet and cane sugar products using gas chromatography–mass spectrometry (GC–MS), varying the extraction methods prior to the

GC–MS analysis; for example, liquid–liquid extraction with fractionation (into basic, neutral, acidic, and phenolic fractions) and continuous liquid–liquid extraction. When the samples were fractionated, the neutral and basic fractions were shown to contain most of the beet character compounds. Headspace volatiles were also studied, and different compounds, including aldehydes, carboxylic acids, pyrazines, and sulfur compounds, were identified. In addition, the efficiency of different adsorbents in removing odors was investigated. Godshall concluded that an important factor in reducing beet-derived odors appeared to be adjustment of the pH. Lowering the pH to 6.5 or below markedly suppressed the odors.

In a more recent study, Godshall et al. (1995) examined white beet sugars of low odor quality using GC–MS and GC–O (–olfactometry). The compounds identified included diacetyl; carboxylic acids such as propionic, butyric, and isovaleric acids; octanal, nonanal, and decanal; and furfural, benzaldehyde, and 5-(hydroxymethyl)-2-furfural (5-HMF). All the compounds listed had odors, and the volatile acids were considered to be the most important. Marsili et al. (1994) identified and quantitated compounds responsible for the off-odor of beet sugar. Of the identified compounds, geosmin (*trans*-1,10-dimethyl-*trans*-9-decalol), 2,5-dimethylpyrazine, furfural, butyric acid, and isovaleric acid were considered likely to be responsible for the characteristic off-odor of beet sugar. It was further discovered that mixtures of volatile acids in combination with geosmin produced an aroma identical to the typical odor defect of beet sugar.

Colonna et al. (1996) used several different approaches to investigate the characteristic odors of beet sugar. Their aim was to identify sugar odorants, to determine ways to prevent their formation, and to develop processes to remove odorants from beet sugar. Some 40 odorants were identified in sugar, syrups, and wash waters. These included carboxylic acids, hexanal, vanillin,  $\alpha$ -terpineol, and several pyrazines. It was

\* Corresponding author (telephone +46 31 335 56 00; fax +46 31 83 37 82; e-mail per.pihlsgard@sik.se).

<sup>†</sup> Swedish Institute for Food and Biotechnology.

<sup>‡</sup> Danisco Sugar AB, SE-205 04 Malmö, Sweden.

concluded that incomplete washing or washing of the crystals with water containing these odorants could negatively affect the sugar quality. Failure to adequately condition the sugar may also play a contributing role.

In an earlier study, Pihlsgård et al. (1998) used GC–O and GC–MS to determine the odors and identities of volatiles from liquid sugars of different purities. Aldehydes, ketones, pyrazines, and furans, together with diacetyl and dimethyl disulfide, among other compounds, were found, and their odors and odor intensities were evaluated.

Products other than sugar, molasses, and wash waters have also been analyzed for volatile compounds. Parliament et al. (1977) studied cooked beets and found that 4-methylpyridine and pyridine constituted about 60% of the total volatiles. Other components present at substantially lower concentration levels were dimethyl sulfide, isovaleraldehyde, isopentanol, and furfural. The occurrence of geosmin and 2-methoxy-3-sec-butylpyrazine was confirmed. Because of the low flavor and odor thresholds of these chemicals and because of their musty or earthy notes, they were considered good candidates for study with respect to the beet sugar odor problem (Marsili et al., 1994).

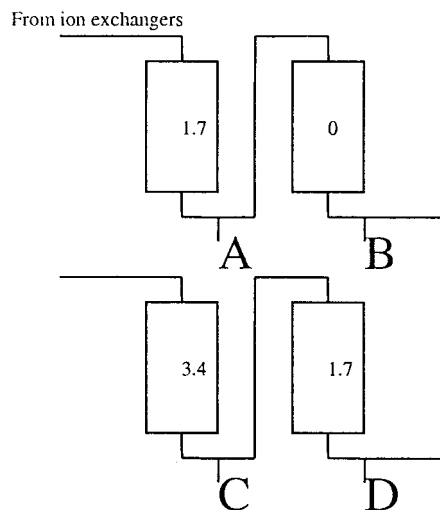
Kaipainen (1990) reported several compounds responsible for odors in granulated sugars. These included *p*- and *m*-methoxybenzaldehyde, *m*-methylanisole, *n*-decanal, myristaldehyde (tetradecanal), geraniol (3,7-dimethylocta-2(*E*),6-dien-1-ol), and citronellyl acetate (3,7-dimethylocta-6-en-1-yl acetate).

In the previously mentioned studies, compounds giving rise to odors and flavors in beet sugar have been identified and quantitated; however, the results from GC analysis have not been correlated to sensory analysis, today's most-used tool for quality control of odors and flavors. Such a link between the instrumental and the sensory analyses is needed to identify the chemical origin of odors or flavors in sugar. In the present study, volatile components in liquid sugars were identified using headspace GC–MS and quantitated using GC–FID. Descriptive sensory analysis of the odors and flavors was carried out, and the FID responses for different compounds and the results from the sensory analysis were analyzed for correlation with a partial least-squares analysis (PLS).

## MATERIALS AND METHODS

**Samples.** Liquid sugar samples were provided by Danisco Sugar AB from its refinery in Arlöv, Sweden. Samples with different odor and flavor profiles were needed, and the sampling model described in Figure 1 was adopted to allow samples with different profiles to be produced.

The liquid sugar passes through a series of purification steps on its way to the finished product. These steps include a mechanical press filter, a polystyrene adsorbent, a cation exchanger, and an anion exchanger. In our experiment, liquid sugar samples taken after the ion exchangers were further treated in an active carbon column system, according to Figure 1. Samples were taken at points when a fixed amount of liquid sugar had passed the columns. In the first series, four different samples, A, B, C, and D, were taken. The sampling conditions are shown in Figure 1. Sample A was taken directly after an active carbon column when 1.7 L sugar solution/g carbon had passed. Sample B was taken at the same time after a second column placed in series with the first one, where the carbon had just been replaced with fresh carbon. Sample C was taken after the first column when 3.4 L solution/g carbon had passed, and finally, sample D was taken directly after the second



**Figure 1.** Sampling procedure. The boxes represent the active carbon columns. Sample A was taken between the columns when 1.7 L of solution/g of carbon had passed the first column and sample B was taken after the second column, just after the carbon had been replaced with new carbon. Samples C and D were taken when another 1.7 L of solution/g of carbon had passed the respective columns.

**Table 1. Sugar Analyses**

	RDS, %	color/ ICU	pH	ash content, %	invert sugar/%
sample A	62.8	9	5.8	0.003	0.335
sample B	62.8	7	7.5	0.006	0.372
sample C	63.0	8	5.5	0.002	0.428
sample D	62.2	12	8.0	0.002	0.412
sample E	64.0	27	4.1	0.001	2.02
sample F	62.9	32	7.1	0.003	1.98
sample G	63.9	18	4.2	0.001	2.45
sample H	63.7	19	4.0	0.001	2.76

column when 1.7 L solution/g carbon had passed. The sampling procedure was repeated about one month later (series 2) and samples E, F, G, and H were taken, where E is equivalent to A in the sampling model, F to B, and so forth. The total number of samples analyzed in this investigation was, hence, eight.

Prior to instrumental and sensory analysis, the samples were diluted to a sugar concentration of 40% with double-distilled water (the original concentration is shown in Table 1) to conform to earlier sensory analyses of liquid sugars performed on site at the sugar refinery.

**Sugar Analyses.** The liquid sugars were analyzed according to International Commission for the Uniform Methods of Sugar Analysis (ICUMSA) methods. These were analysis of RDS% (concentration of sucrose), color, pH, ash content, and invert sugar.

**Instrumental Analysis.** Volatile compounds from the sugar samples were adsorbed on Tenax (60–80 mesh) by the method described by Hall et al. (1985). Each sample (100 g in a 500-mL flask) was allowed to equilibrate for 30 min at 40 °C. Then, 5 L of helium gas was led through the flask and allowed to pass the adsorbent material at a rate of 40 mL/min. Thermal desorption of the adsorbed volatiles was carried out prior to injection on the GC by means of an automatic injector, ATD 400 (Perkin-Elmer, Norwalk, CT). The desorption temperature was 250 °C; the desorption time, 5 min; and the split-ratio between the column and the atmosphere was 1:5. Compounds were analyzed by GC using a Hewlett-Packard 5890 Series II instrument (Hewlett-Packard, Palo Alto, CA) equipped with a 60 m × 0.32 mm capillary column with a 1.0 μm-thick film of DB-1 (J&W Scientific Inc., Folsom, CA). The initial temperature of the chromatographic oven was 35 °C. The temperature was raised to 220 °C at a rate of 4 °C/min and held at that temperature for 20 min. Helium carrier gas

**Table 2. Sensory Scores for the Different Sensory Attributes<sup>a</sup> (Mean Values)**

	A	B	C	D
total odor	53.0 <sup>C,E,G,H</sup>	38.5 <sup>F</sup>	54.3 <sup>A,E,G,H</sup>	45.4 <sup>E,H</sup>
light syrup odor	12.4 <sup>C,D,E,G,H</sup>	20.4 <sup>D,E,F,H</sup>	8.9 <sup>A,G,H</sup>	17.5 <sup>A,B,E,G,H</sup>
dark syrup odor	24.1 <sup>C,E,G,H</sup>	5.18 <sup>D,F</sup>	26.0 <sup>A,E,G,H</sup>	13.3 <sup>B</sup>
bread crust odor	13.6 <sup>B,C,D,E,F,G,H</sup>	10.3 <sup>A,C,D,E,F,G,H</sup>	13.7 <sup>A,B,D,E,F,G,H</sup>	11.3 <sup>A,B,C,E,F,G,H</sup>
rubber odor	24.4 <sup>C,G,H</sup>	9.19 <sup>D,E,F,H</sup>	25.3 <sup>A,G</sup>	13.6 <sup>B,E,H</sup>
chlorine odor	16.0 <sup>C,D,E,H</sup>	7.08 <sup>D,E,F,H</sup>	20.4 <sup>A,H</sup>	8.89 <sup>A,B,E,F,H</sup>
total flavor	53.6 <sup>C,D,E,G,H</sup>	44.7 <sup>F</sup>	54.8 <sup>A,D,E,G,H</sup>	51.0 <sup>A,C,E,H</sup>
light syrup flavor	14.5 <sup>C,D,E,G,H</sup>	25.4 <sup>D,F</sup>	10.7 <sup>A,E,G,H</sup>	21.4 <sup>A,B,E,G,H</sup>
dark syrup flavor	24.0 <sup>C,E,G,H</sup>	4.78 <sup>F</sup>	25.9 <sup>A,E,G,H</sup>	13.6
sweet taste	50.8 <sup>B,C,D,E,F,G,H</sup>	47.6 <sup>A,C,D,F</sup>	50.3 <sup>A,B,D,E,F,G,H</sup>	49.7 <sup>A,B,C,E,F,G,H</sup>
chlorine flavor	17.3 <sup>C,H</sup>	2.90 <sup>D,E,F</sup>	21.3 <sup>A,G</sup>	7.39 <sup>B,E,F,H</sup>
aftertaste	45.2 <sup>C,E,H</sup>	36.4 <sup>D,F</sup>	45.9 <sup>A,E,H</sup>	40.2 <sup>B,E,H</sup>
	E	F	G	H
total odor	50.7 <sup>A,C,D,G,H</sup>	37.4 <sup>B</sup>	56.7 <sup>A,C,E</sup>	49.1 <sup>A,C,D,E</sup>
light syrup odor	15.9 <sup>A,B,D,G,H</sup>	25.6 <sup>B</sup>	13.6 <sup>A,C,D,E,H</sup>	14.1 <sup>A,B,C,D,E,G</sup>
dark syrup odor	24.2 <sup>A,C,G,H</sup>	1.93 <sup>B</sup>	23.1 <sup>A,C,E,H</sup>	24.4 <sup>A,C,E,G</sup>
bread crust odor	14.3 <sup>A,B,C,D,F,G,H</sup>	11.4 <sup>A,B,C,D,E,G,H</sup>	10.9 <sup>A,B,C,D,E,F,H</sup>	14.4 <sup>A,B,C,D,E,F,G</sup>
rubber odor	13.9 <sup>B,D,H</sup>	6.01 <sup>B</sup>	25.6 <sup>A,C</sup>	15.9 <sup>A,B,D,E</sup>
chlorine odor	12.1 <sup>A,B,D,H</sup>	3.14 <sup>B,D</sup>	29.0	14.4 <sup>A,B,C,D,E</sup>
total flavor	51.4 <sup>A,C,D,H</sup>	45.1 <sup>B</sup>	56.9 <sup>A,C,H</sup>	54.0 <sup>A,C,D,E,G</sup>
light syrup flavor	17.1 <sup>A,C,D,G,H</sup>	30.8 <sup>B</sup>	15.7 <sup>A,C,D,E,H</sup>	16.1 <sup>A,C,D,E,G</sup>
dark syrup flavor	24.4 <sup>A,C,G,H</sup>	2.72 <sup>B</sup>	24.3 <sup>A,C,E,H</sup>	23.7 <sup>A,C,E,G</sup>
sweet taste	51.8 <sup>A,C,D,F,G,H</sup>	50.1 <sup>A,B,C,D,E,G,H</sup>	51.6 <sup>A,C,D,E,F,H</sup>	53.1 <sup>A,C,D,E,F,G</sup>
chlorine flavor	8.03 <sup>B,D,F,H</sup>	2.07 <sup>B,D,E</sup>	26.6 <sup>C</sup>	13.6 <sup>A,D,E</sup>
aftertaste	42.5 <sup>A,C,D,H</sup>	36.2 <sup>B</sup>	49.8	43.6 <sup>A,C,D,E</sup>

<sup>a</sup> Superscripts to the sensory scores denote samples that are *not* significantly different from the respective samples ( $P \leq 0.05$ ).

was used at a flow rate of 4.0 mL/min. The effluent from the capillary column was split 1:3, with one-fourth of the effluent routed to an Inco 50 mass spectrometer (Finnigan, San José, CA) and three-fourths directed to a flame ionization detector. Every sample was analyzed three times. A Hewlett-Packard 3550 laboratory data system was used to collect data and to calculate peak areas for the corresponding volatiles from the FID responses. Identification was made from mass spectral data using the software Data Master (Finnigan). In addition, for most of the compounds found to be correlated to sensory data (Table 2), the identifications were verified by injection of authentic reference compounds.

**Sensory Analysis.** Sensory analysis was performed by a panel of eight assessors. During training sessions, the panel was presented with samples with odors and flavors that were typical of those found in the sugar solutions, together with the actual samples that were subsequently evaluated. Both sniffing and tasting were performed in order to allow evaluation of both odors and flavors. Suitable attributes were suggested during the training sessions, and the assessors reached consensus on a final list of flavor and odor attributes to use in the analysis. These attributes were "total odor" and "total flavor", "dark syrup odor" and "dark syrup flavor", "light syrup odor" and "light syrup flavor", "bread crust odor", "rubber odor", "chlorine odor" and "chlorine flavor", "sweet taste", and "aftertaste". Dark syrup and light syrup are commercially available products from Danisco Sugar AB, Sweden, and have licorice-like and caramel-like flavors, respectively. A standard procedure for test design and sample presentation was used (Meilgaard et al., 1991). The intensity of the odors and flavors of the samples with respect to the 12 attributes were then evaluated on a 100 mm scale, where 10 mm corresponds to no odor/flavor and 90 mm to a very pronounced odor/flavor. The samples were served in a fully randomized and balanced order in three replicates. The accuracy and repeatability of the assessors were tested before data processing (Lea et al., 1995). Analysis of variance (ANOVA) was performed, and the Tukey HSD (honestly significant difference) test was used to determine whether the samples were significantly different on the basis of each attribute (O'Mahoney, 1986). The statistical evaluation of the sensory data was carried out using the computer software Systat (SPSS, Chicago, IL).

A principal component analysis (PCA) was performed on the sensory data to elucidate whether the various sensory at-

tributes differed from each other. For this analysis, the software package Guideline (Camo AS) was used. For a more thorough description of the different multivariate techniques, PCA and PLS, the reader is referred to Esbensen et al. (1994).

**Analysis for Correlation between Instrumental Analysis and Sensory Analysis.** The correlation between instrumental data and sensory data was evaluated by multivariate analysis. That is, the FID responses for the different compounds (measured as areas) were analyzed for correlation to the sensory attributes in order to estimate the effect the variations in the peak areas have on the sensory evaluations (scores) of each attribute. Before the actual analysis, the variation (expressed as the variance) between samples was compared with the variation between replicates of the same sample in order to find and discard chromatographic peaks and/or sensory attributes for which the repeatability was insufficient. The multivariate technique used was a partial least-squares analysis (PLS) that was carried out on a dataset containing the remaining gas chromatographic and sensory data. The results were displayed as loading plots. For this analysis, the software package Guideline (Camo AS) was used.

To evaluate the odor of some specific compounds found to be of interest from the correlation analysis, the pure compounds were individually added, to a water solution of granular sugar at the most typical sugar concentration of the investigated liquid sugar samples. The compounds were added gradually until an odor was perceived. The odor was described by two assessors.

## RESULTS AND DISCUSSION

**Sugar Analyses.** The results from the sugar analyses are shown in Table 1. From the color and invert sugar values, it is concluded that the syrup fed to the carbon column system in series 1 (samples A–D) was different from the syrup fed in series 2 (samples E–H), giving final samples with a wide variation in properties. This was in accordance with the aim of the sampling procedure. The higher level of invert sugar (glucose and fructose, both being reducing sugars) in series 2 may, in the presence of amino groups, increase the formation of Maillard reaction products. This could be the reason for the higher color values, and Maillard reaction products might also influence the flavor.

**Table 3. Compounds Associated with "Total Odor" and "Total Flavor"<sup>a</sup>**

butanone		4
3-methyltetrahydrofuran	MS only	5
3-hydroxypentan-2-one	MS only	9
heptan-2-one		14
2,6-dimethylpyrazine		18
3,4-dimethylhexan-2-ol		20
2,5-dimethylhexan-3-ol		21
6-methylhept-5-ene-2-one		24
2-ethyl-5-methylpyrazine		26
2,2-dimethyldecane	MS only	27

<sup>a</sup> Numbers in Tables 3–6 are the same as in the loading plot (Figure 4). MS only denotes that mass spectrometry was the only means of identification.

**Sensory Analysis.** Table 2 shows the mean values for the different sensory attributes. If a comparison between sample A and its counterpart in series 2, sample E, is made, it is seen that only in the case of "rubber odor" and "chlorine flavor" were the sensory evaluations statistically significantly different. Samples B and F were not significantly different for any attribute, whereas samples C and G were significantly different for the attributes "chlorine flavor" and "aftertaste". Samples D and H were only significantly different for "dark syrup odor" and "dark syrup flavor". It is obvious that the samples with a similar history of carbon treatment had similar sensory profiles, even when the material prior to carbon treatment was not the same. As with samples B and F, samples A and C and samples A and H did not exhibit any significant differences for any attribute. In the case of samples A and C, this lack of difference could be explained if the active carbon in the column (A) was already saturated after 1.7 L of sugar solution per gram of active carbon had passed through the column. However, the counterparts of samples A and C in the second series, samples E and G, were significantly different for the attributes "rubber odor", "chlorine odor", "chlorine flavor", "total flavor", and "aftertaste" and were not significantly different for the other attributes.

Sample C and sample G exhibited the highest sensory scores for the respective series, except for the attributes "light syrup odor", "light syrup flavor", and "sweet taste". Sample B exhibited the lowest sensory score in the first series for all attributes, except for "light syrup odor" and "light syrup flavor", which were the highest for this sample. Sample F exhibited the lowest sensory score in the second series for all attributes, except "light syrup odor" and "light syrup flavor", for which sample F exhibited the highest score, and "bread crust odor", which was not significantly different for any sample. An explanation for the significantly higher levels of the sensory scores for "light syrup odor" and "light syrup flavor" in samples B and F could be that this odor and its corresponding flavor were masked by compounds giving rise to strong odors in the other samples and therefore seemed more pronounced in samples B and F.

For the attributes that were evaluated both by sniffing (odor) and tasting (flavor), the intensities did not seem to depend markedly on which of the two methods was used. This is why the odor attributes are presented together with their corresponding flavor attributes.

**Instrumental Analysis.** Figure 2 shows chromatograms (FID) from runs of the eight samples A–H. The chromatogram of every sample exhibits a unique pattern in relation to the other samples. It can be seen that

**Table 4. Compounds Associated with "Dark Syrup Odor" and "Dark Syrup Flavor"**

3-hydroxypentan-2-one	MS only	9
2,6-dimethylpyrazine		18
2,5-dimethylhexan-3-ol		21
6-methylhept-5-ene-2-one		24
2-ethyl-5-methylpyrazine		26
2,2-dimethyldecane	MS only	27

**Table 5. Compounds Associated with "Chlorine Odor" and "Chlorine Flavor"**

2,5-dimethylfuran		6
2,6-dimethylpyrazine		18
3,4-dimethylhexan-2-ol		20
2,5-dimethylhexan-3-ol		21
6-methylhept-5-ene-2-one		24
2-ethyl-5-methylpyrazine		26
2,2-dimethyldecane	MS only	27

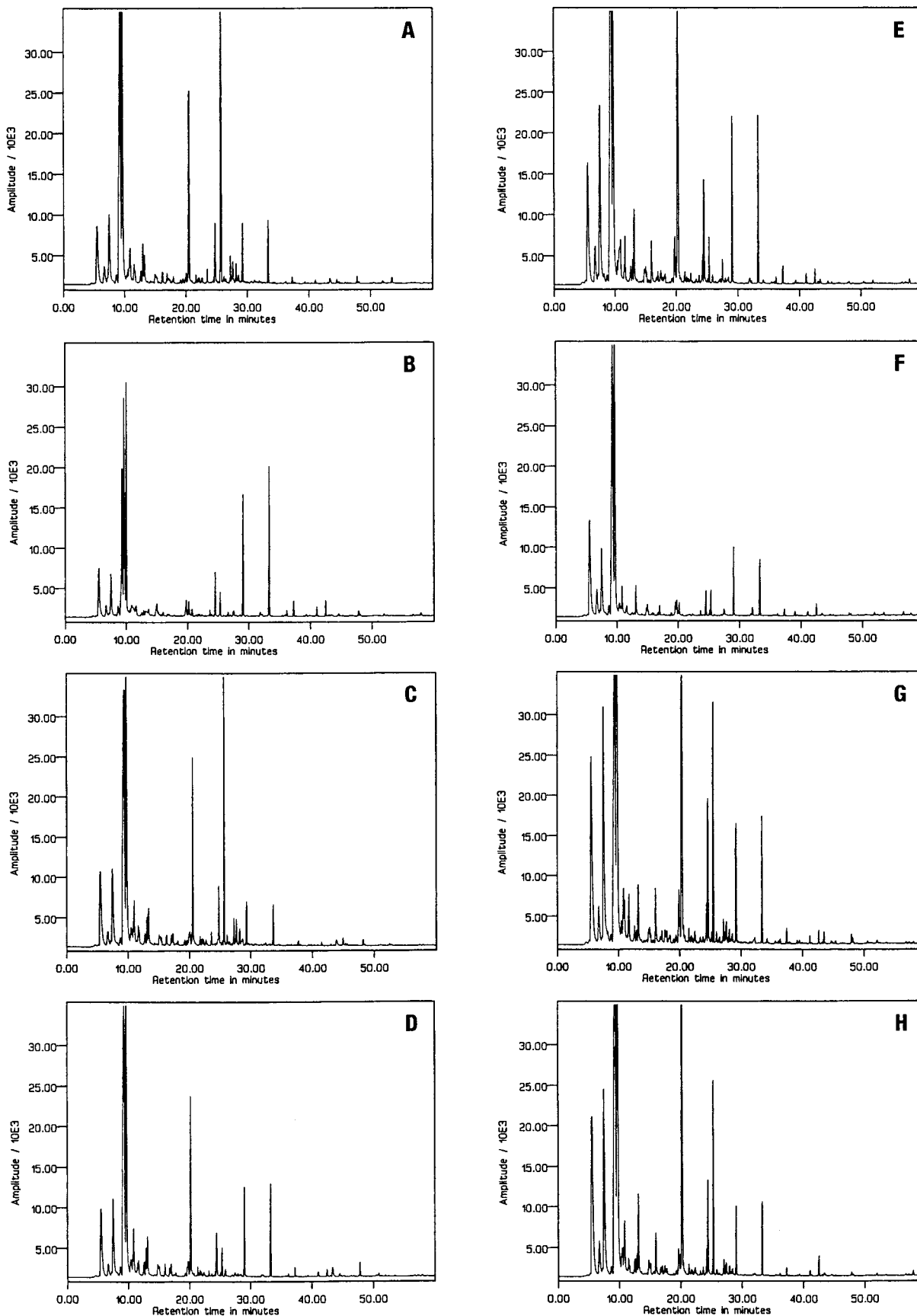
**Table 6. Compounds Associated with "Rubber Odor"**

dimethyl disulfide		7
3-hydroxypentan-2-one	MS only	9
heptan-2-one		15
2,5-dimethylhexan-3-ol		21
4-methoxyphenol	MS only	23
2,2-dimethyldecane	MS only	27
sat.hydrocarbon	MS only	28
3-ethyl-2,5-dimethylpyrazine	MS only	29

for all samples in series 2, except possibly for sample F, the complexity is higher than for the corresponding samples in series 1, which is in accordance with the higher color values and invert contents shown in the ICUMSA analyses. Within each series, the third sample (C and G) was the most complex, whereas the second sample (B and F) was the least complex. This is in accordance with the sampling conditions. Sixty-six different GC peaks were chosen for further analysis on the basis of a preset threshold used by the peak integration algorithm (automatic mode).

**Analysis for Correlation between Instrumental Analysis and Sensory Analysis.** ANOVA was used to discard the sensory attributes and chromatographic peaks for which the observed variation was not statistically significant ( $p < 0.05$ ). For this reason, several sensory attributes were discarded, and the attributes used for further data treatment were "total odor" and "total flavor," "dark syrup odor" and "dark syrup flavor", "chlorine odor" and "chlorine flavor", and "rubber odor". The distribution of the odor attributes is shown in Figure 3. Of the 66 GC peaks, 34 were used in the analysis. PLS analysis was carried out, and each of the sensory attributes was analyzed for correlation to GC peaks. As an example, Figure 4 shows the loading plot that was used to determine which compounds affected the scores of the sensory attribute "rubber odor". The numbers denote the GC peaks (three replicates) and "rubod" symbolizes the three replicates of the sensory analysis with respect to the attribute "rubber odor". The compounds that were found to correlate with the respective odors and flavors, with their numbers in the plot, are listed in Tables 3–6.

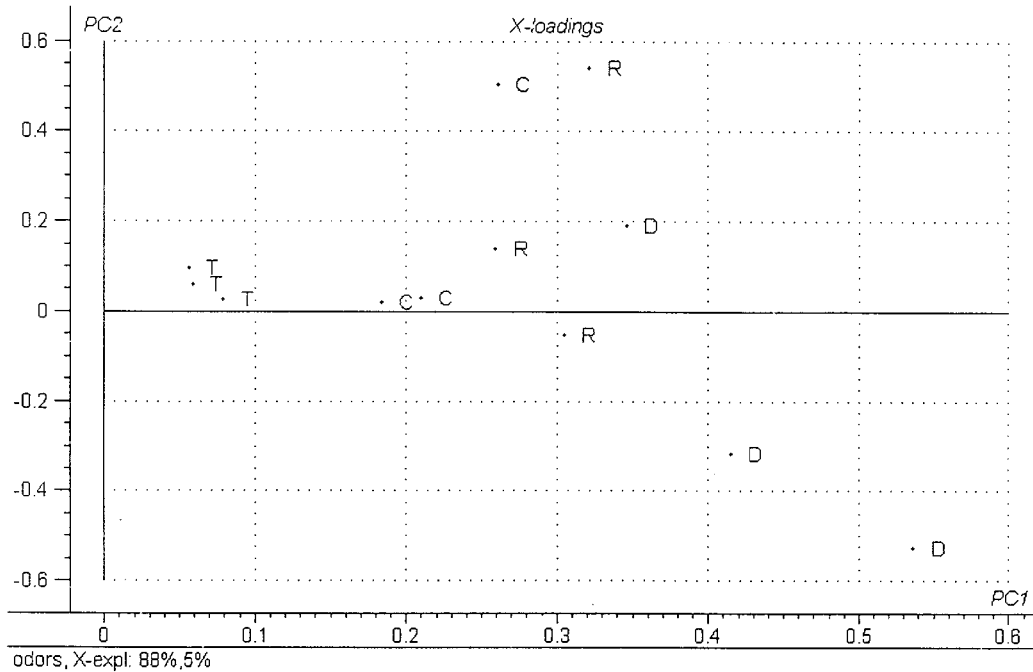
To verify the odor characteristics in liquid sugar matrix, typical odor-active compounds, of different chemical classes, found to be correlated with sensory attributes were individually added. Butanone was found to have a caramel-like odor, dimethyl disulfide an onion-like odor, and 2,6-dimethylpyrazine a roasted, burnt odor. 4-Methoxyphenol had an odor resembling that of raw sausage and 2-heptanone had a sweet, fruity odor. 2,5-Dimethylfuran gave gasoline- and solvent-like notes.



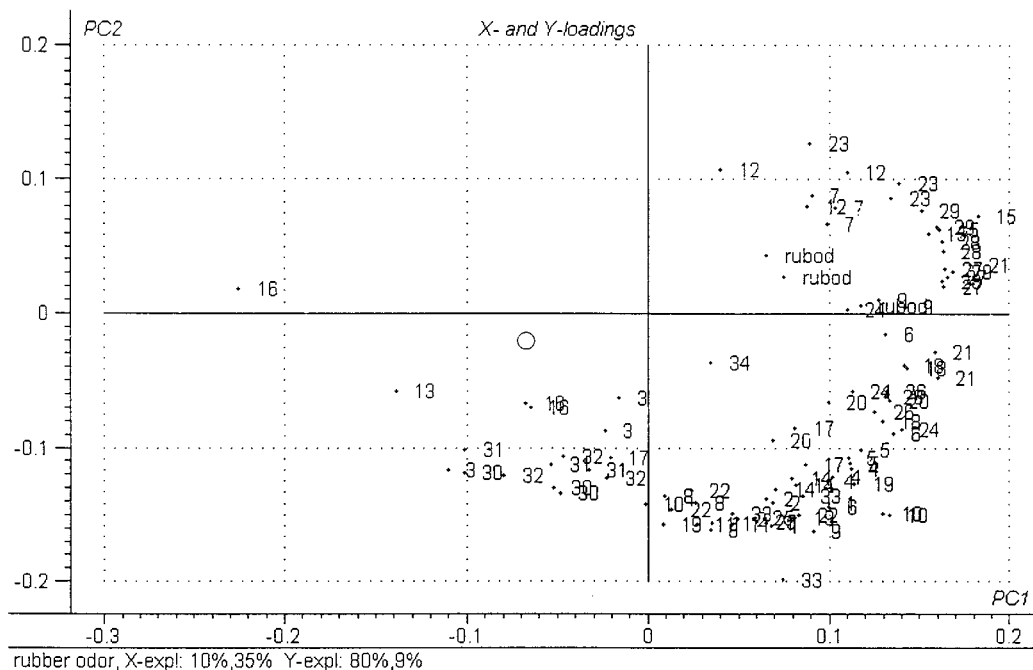
**Figure 2.** Chromatograms of the samples A through H.

Other authors have reported other odors for some of these compounds (Arctander, 1969; Furia and Bellanca, 1975). Descriptions such as "chlorine" or "rubber" are not found in the literature, but it is important to bear

in mind that an individual compound may be identified in the literature as having a particular odor because of the particular matrix in which it was isolated or dissolved. Thus, the combination of odors in this par-



**Figure 3.** Loading plot showing the distribution of the different sensory attributes. T = total odor, D = dark syrup odor, C = chlorine odor, R = rubber odor



**Figure 4.** Loading plot for GC data and sensory data for the attribute "rubber odor".

ticular matrix may give rise to the particular odors detected in this sensory analysis. Furthermore, most odorous compounds exhibit different odors at different concentrations.

Some compounds appear in more than one table and are thus correlated to more than one sensory attribute. For most samples, the attributes chosen for further analysis differentiate well between the samples, according to Table 2.

Most, but not all, of the compounds found associated with odors and flavors in the sugar solutions have previously been identified in beet sugar or beet sugar molasses. Godshall (1986) identified dimethyl disulfide, 2,5-dimethylpyrazine, and butanone in beet molasses.

Marsili et al. (1994) identified 2,5-dimethylpyrazine in malodorous beet sugar, and Colonna et al. (1996) identified 2,5-dimethylpyrazine, 3-ethyl-2,5-dimethylpyrazine, and 2,5-dimethylfuran in beet sugar factory process streams. In an earlier study, where the same type of liquid sugars as in the present study were used, Pihlsgård et al. (1998) identified butanone, dimethyl disulfide, 2-heptanone, 4-methoxyphenol, 2,2-dimethyldecane, and 3-ethyl-2,5-dimethylpyrazine. However, volatile fatty acids, often claimed to be responsible for off-odors or off-flavors in beet sugar (Godshall et al., 1995), have not, in this study of liquid sugars, been found to be among the identified compounds, not even among those identified that were not found to be

correlated with the sensory attributes. The headspace method used is not very sensitive for volatile fatty acids, but none of these acids were found in diethyl ether extracts from corresponding samples. We believe that this is due to the fact that the analyzed product has passed many industrial purifying steps which have removed the volatile fatty acids originally present. The earthy, musty, and acidic beet-derived odors reported by other authors (Colonna et al., 1996; Godshall et al., 1995; Marsili et al., 1994) seem, in these purified samples, to be overshadowed by odors coming from Maillard reaction products and caramelization products, as a result of the high temperature in the process.

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#### LITERATURE CITED

- Arctander, S. *Perfume and Flavor Chemicals*; Montclair, NJ, 1969.
- Colonna, W. J.; McGillivray, T.; Samaraweera, U.; Torgeson, T. Odor in Beet Sugar: Some Causative Agents and Preventative Measures. *Proceedings 1996 Sugar Processing Research Conference*; Sugar Processing Research Institute Inc.: New Orleans, LA, 1996; pp 198–220.
- Esbensen, K.; Schönkopf, S.; Midtgaard, T. *Multivariate Analysis in Practice*; Camo AS: Trondheim, Norway, 1994.
- Furia, T. E., Bellanca, N., Eds. *Fenaroli's Handbook of Flavor Ingredients. Volume 2*, 2nd ed.; CRC Press Inc.: Cleveland, OH, 1975.
- Godshall, M. A. Flavors from Beet and Cane Sugar Products. *Proceedings 1986 Sugar Processing Research Conference*; Sugar Processing Research Institute Inc.: New Orleans, LA, 1986; pp 210–228.
- Godshall, M. A.; Grimm, C.; Clarke, M. A. Sensory Properties of White Beet Sugars. *Int. Sugar J.* **1995**, *97*, 296–343.
- Hall, G.; Andersson, J.; Lingnert, H.; Olofsson, B. Flavour Changes in Whole Milk Powder during Storage III. *J. Food Qual.* **1985**, *7*, 153–190.
- Kaipainen, A. A Study of the Aroma Compounds Arising in Sugar Processing Using Three-channel HRGC and GC/MS. *Chromatography Conference*, Elsevier: Amsterdam, Netherlands, 1990.
- Lea, P.; Rødbotten, M.; Næs, T. Measuring Validity in Sensory Analysis. *Food Qual. Preference* **1995**, *6*, 321–326.
- Marsili, R. T.; Miller, N.; Kilmer, G. J.; Simmons, R. E. Identification and Quantitation of the Primary Chemicals Responsible for the Characteristic Malodor of Beet Sugar by Purge and Trap GC-MS-OD Techniques. *J. Chromatogr. Sci.* **1994**, *32*, 165–171.
- Meilgaard, M.; Civille, G. V.; Carr, B. T. *Sensory Evaluation Techniques*; CRC Press: Boca Raton, FL, 1991.
- O'Mahoney, M. *Sensory Evaluation of Food: Statistical Methods and Procedures*; Marcel Dekker Inc.: New York, 1986.
- Parliment, T. H.; Kolor, M. G.; Maing, I. Y. Identification of the Major Volatile Components of Cooked Beets. *J. Food Sci.* **1977**, *42*, 1592–1593.
- Pihlsgård, P.; Lingnert, H.; Leufvén, A.; Larsson, M. Aroma Analysis of Liquid Sugars. *Int. Sugar J.* **1998**, *100*, 431–436.

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