# Critical Evaluation of Two Commonly Used Techniques for the Treatment of Data from Extract Dilution Sniffing Analysis

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The data from the extract dilution sniffing analysis of beer samples have been treated by two methods of analysis to give either "charm" or "FD" values. The results obtained from these two methods were compared and demonstrated that the rank order of intensity of the odor-active regions was different for most panelists when the data were presented as charm rather than FD values. Points of uncertainty observed while using this method such as between- and within-individual reproducibility and gaps in the coincident response have also been discussed.

## INTRODUCTION

GC eluate sniffing has been extensively used in aroma research as a means of separating odor-active compounds from those which are volatile but odorless (i.e., not important for the human nose). Traditional methods of sniffing, however, do not allow either the relative intensity or the odor threshold of odor-active compounds to be determined.

This problem was initially addressed by combining sniffing experiments with traditional threshold analysis to give a value called the aroma value (Rothe and Thomas, 1962). This value was defined as the ratio of the concentration of a flavor compound to its odor threshold. Other groups have used this ratio with various methods of threshold determination to give values that include the odor unit number (Guadagni, 1966), the number of odor intensity units (Teranishi et al., 1971), the odor value (Mulders, 1973), the odor intensity index (Barth, 1973), the flavor unit (Meilgaard, 1982), and the threshold odor number (Hill and Barth, 1976).

More recently, a technique of extract dilution sniffing analysis (EDSA) has been developed by two different research groups (Acree et al., 1984; Ullrich and Grosch, 1987) in an effort to simplify the method used for determining a unit of odor intensity. The aim of this technique in food flavor research is to determine the relative odor potency of compounds (odor-active regions) present in an extract. Thus, the method gives the priority order for chemical identification and adds to the understanding of the chemical origins of olfactory differences (Grosch, 1993).

The FD value is simply the last dilution at which an odor-active compound is detected at a certain RI. The results are generally expressed as the logarithm of the factor of dilution (log FD) vs the RI or by listing the factors of dilution. This method is now known as aroma extract dilution analysis (AEDA) (Blank et al., 1989). This method has been used to determine potent odorants in many different types of food products including wheat and rye bread crust (Schieberle and Grosch, 1987), soybean oil (Guth and Grosch, 1990), fresh and stored lemon (Schieberle and Grosch, 1988), wheat bread crumb and crust (Schieberle and Grosch, 1991, 1992), beers (Schieberle, 1991), and orange juice (Marin et al., 1992).

Although sniffing procedures have already been extensively used in flavor science, the concept of the odor unit number as a measure of the relative intensity of odor-active compounds in an extract has been criticized (Frijters, 1978). According to this author, the measures are based on two major assumptions that are contrary to present psychophysical theories of odor perception. The use of the odor unit number assumes that there is a linear relationship between the perceived intensity of a compound and its concentration. This assumption has been proven invalid by both Fechner's and Steven's laws (Sauvageot, 1990), which show that there is a logarithmic or power relationship between these two variables. The second assumption, that the steepness of slopes of perceived intensity vs concentration is equal for all odorants, is also invalid as it has been demonstrated by many authors that the value of this slope differs for different compounds (Cain, 1969; Laffort et al., 1974; Patte et al., 1975; Laing et al., 1978). Due to this phenomenon, the order of the relative intensity of two compounds with the same threshold value does not necessarily correspond to their relative concentration in the same mixture. Care must therefore be taken when results using odor unit numbers and all relevant values are interpreted especially when compounds are ranked in order of intensity.

This paper compares two methods, charm and FD values, of the extract dilution sniffing analysis and emphasizes critical steps in using these methods. The sniffing results from two beer extracts have been used to highlight these problems.

## MATERIALS AND METHODS

**Beer Samples.** Two commercially available beer samples, a lager (A) and a lager (C) with special malt (with peat fire), were purchased from a large retail outlet and kept at 4 °C until use.

**Analytical Reagents.** The XAD resins were purchased from Fluka AG, Switzerland. Each resin was washed continuously in a Soxhlet apparatus with ether and methanol for 24 h, respectively. The resins were then rinsed before use with water (10 × 50 mL).

All reagents used were of AR grade, and all water was purified by a Milli-Q system (Millipore Corp., France).

**Extraction of Beer Volatiles.** Ethanolic extracts were obtained by liquid–solid extraction of the beers on a mixture of Amberlite resins previously described (Abbott et al., 1993). Great care was taken to check the olfactory representativeness of these extracts compared to the original beers. This was done by performing sensory analysis of the extracts as described by Abbott et al. (1993). Blank extracts were prepared systematically by substituting water (45 mL) for beer.

**Gas Chromatography.** Analysis of the beer extracts and blanks, *n*-paraffin standards, and Grob standard mixture were undertaken with a Hewlett-Packard 5890 gas chromatograph using the following conditions: column, JW DB1701, 15 m, 0.25 mm id, 1-$\mu$m film thickness; carrier gas $H_2$, 50 cm/s; split–splitless

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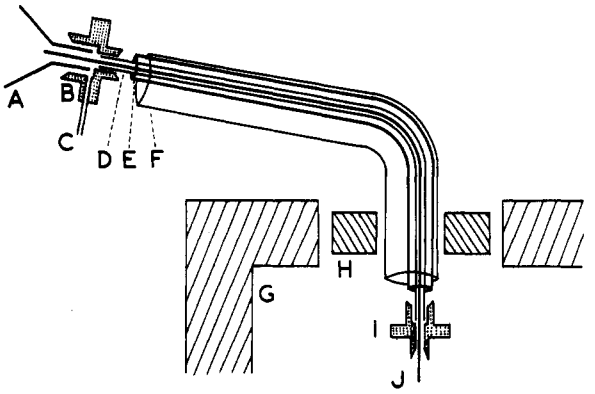


**Figure 1.** Diagram of the sniffing port connected to the Hewlett-Packard 5890 GC: (A) glass funnel, (B) union $^1/_4$, $^1/_8$ in., (C) humidified air 25 mL/min, (D) copper tubing $^1/_8$ in., (E) copper tubing 8 mm (soldered with silver), (F) copper tubing 20 mm, (G) wall of the GC oven, (H) heating block, (I) union $^1/_8$, $^1/_{16}$ in., (J) nonphasic capillary column.

injection; oven temperature, 30–200 °C at 5 °C/min, then 200–220 °C at 10 °C/min.

The beer extracts and the blanks were analyzed with the column connected either directly to the sniffing port (Figure 1) or directly to the FID. The *n*-paraffin standards, $C_7$–$C_{18}$, and Grob standard mixture (Grob et al., 1981) were analyzed with the column connected directly to the FID.

To overcome any possible condensation or adsorption of the compounds in the sniffing port, the last 30 cm of the column was replaced with a heated empty silica tubing.

**Extract Dilution Sniffing Analysis.** The sniffing panel consisted of six people: three from INRA (Laboratoire de Recherches sur les Arômes), all of whom had participated in the triangle and matching tests of the beer extracts as previously described (Abbott et al., 1993); and three from the town of Dijon. This latter series of judges was chosen initially by their response to a questionnaire (Marin et al., 1988) and by their ability to reproducibly detect specific volatile compounds as they eluted from the column.

Sensory data from the GC sniff experiments were recorded by each panel member by pressing the space bar on the computer keyboard when an odor was detected and releasing it the moment the odor was no longer perceived. The yes/no response data (the start time and end time of the odor-active region measured in seconds) were collected directly into a program developed at INRA (Almanza, A). The judges were also asked to give a verbal description of each perceived odor.

Serial dilutions (1:3) of the beer extracts were analyzed until odor-active regions were no longer detected. Due to the presence of sodium chloride, the extracts were initially diluted with methanol (dilutions 1 and 2) and then with dichloromethane. Two replicates of each dilution were assessed.

For each sniffing session, the panelist sniffed the effluent of two randomized dilutions for not more than 30 min with a break of at least 15 min between each injection. The sniffing analysis of the extracts was conducted over two periods of 2 months each in an isolated room kept at approximately 22 °C.

**Reproducibility.** A series of dilutions of a standard solution was assessed in duplicate by each panel member. Furthermore, to determine the reproducibility of the detection of odor-active compounds in a complex solution, one beer extract (dilution 1, beer A) was assessed five times by three panel members.

## RESULTS

**GC-FID Analysis.** The FID chromatograms presented in Figure 2 demonstrate that the compositions of beer extracts A and C were different. Furthermore, GC sniffing demonstrated that the odor of the two beers could be characterized by the description of the odor-active regions (Table I). The odor-active regions in the extract of beer A were described mainly in terms of floral and caramel aromas compared with that of beer C, which had more coffee, yeast, and rubber aromas. Many of the compounds characterized by sniffing could not be, or were barely detected, by FID.

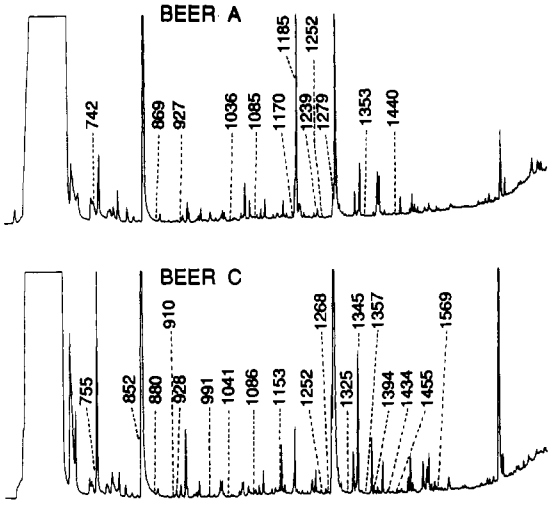


**Figure 2.** FID chromatograms for beer extracts A and C. The retention times indicated on these chromatograms correspond to the odor-active areas described in Table I.

**Table I. Description of Odor-Active Regions Detected in Beer Extracts A and C for the Series of Dilutions and for All Panelists**

| extract of beer A | | extract of beer C | |
|---|---|---|---|
| KI[a] | description | KI | description |
| 742 | caramel, chocolate | 755 | coffee |
| 869 | plastic, rubber | 852 | coffee, plastic |
| 927 | vitamin, rubber | 880 | fruity, coffee |
| 1036 | grilled, burnt | 910 | uncharacterized |
| 1085 | cooked potatoes, vegetable broth | 928 | beer, rubber, coffee |
| | | 991 | bread, yeast, vanilla, coffee |
| 1170 | floral | 1041 | cooked potatoes |
| 1185 | grassy, rose | 1086 | tropical fruit, terpene |
| 1239 | caramel | 1153 | uncharacterized |
| 1252 | dry grass, caramel | 1252 | uncharacterized |
| 1279 | burnt caramel | 1268 | coffee, caramel |
| 1353 | licorice | 1325 | grassy, floral |
| 1440 | beer | 1345 | burnt |
| | | 1357 | coffee, rubber, caramel |
| | | 1394 | coffee, walnuts |
| | | 1434 | licorice, rubber |
| | | 1455 | toast, roasted coffee |
| | | 1569 | cloves |

[a] KI, retention index.

Although the compositions of the two beers are obviously different, EDSA must be performed on the extracts to determine which compounds or odor-active regions contribute significantly to the odor of the extracts and to the differences between the two extracts. As the odors of the extracts have been shown to be representative of the beer itself (Abbott et al., 1993), valid conclusions can be drawn from the results of the EDSA about the odor of the beer samples.

To construct an aromagram from a series of dilutions, it is imperative that the retention index of each odor-active region detected by the panelist can be reproducibly determined. This was achieved by regularly injecting a series of *n*-paraffin standards whose retention times were then used to convert the sensory data to retention indices, thus taking into account any modification of the column and subsequent changes in the retention times of the compounds.

**GC-EDSA.** Data from the EDSA for two panel members and two beer extracts are presented in Figure 3. The

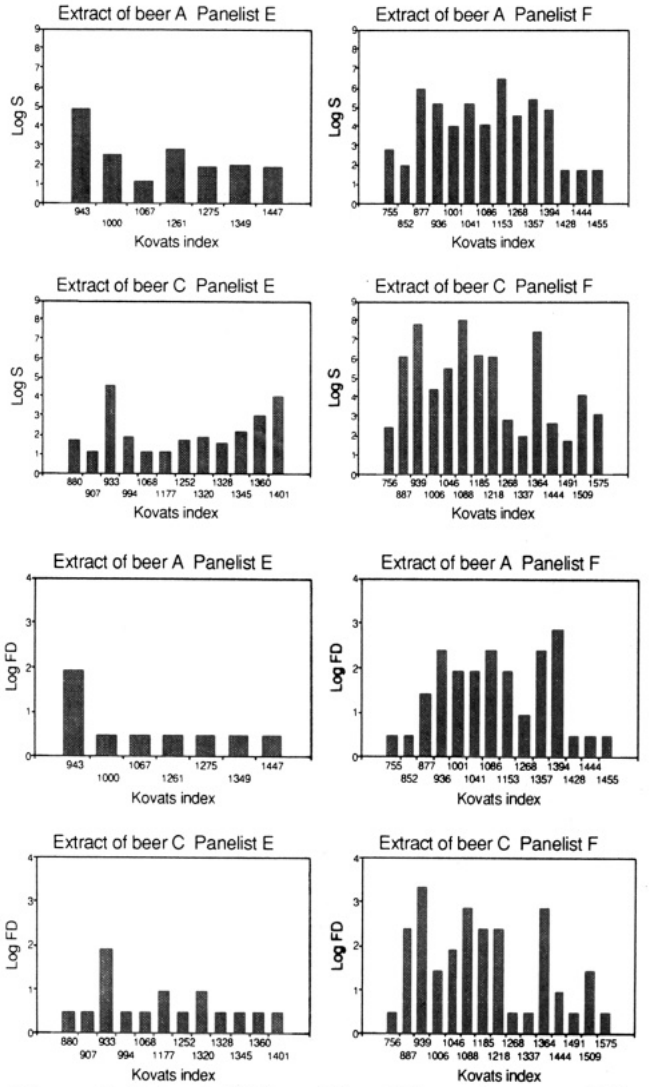


**Figure 3.** (a, top half) Logarithm of the surface area of the odor-active regions vs their retention indices for beer extracts A and C and panel members E and F. (b, bottom half) Logarithm of the factor of dilution of the odor-active regions vs their retention indices for beer extracts A and C and panel members E and F.

logarithm of the surface area of the odor-active regions vs the RI is presented in Figure 3a, whereas Figure 3b represents the logarithm of the last dilution at which an odor-active region was detected. Both values were calculated from the number of coincident (positive) responses across all dilutions. These results are representative of the range of data obtained across all panel members, and so all 12 aromagrams have not been presented.

The differences in the odors of the two beers are exemplified by the aromagrams presented in Figure 3, both in the number of peaks detected and in the intensity of the odor-active regions. In general, beer C was found to have a greater number of odor-active regions than beer A. Furthermore, the sum of log $s$ and log FD for beer C was also greater than that for beer A, suggesting that the former beer has a greater intensity of odor. One cannot conclude from these data, however, that the concentration of volatile compounds is greater for beer C than for beer A; rather, beer C contains odor-active compounds that are more potent than those of beer A.

**Gaps in the Coincident Responses.** Throughout the course of this study it has been observed that for a series of dilutions an individual may not detect an odor at a certain RI in, for example, dilution 2, but then detect an odor at this same RI at higher dilutions. These "gaps" in the coincident responses were observed for four of the six panel members for both beer samples A and C.

An example of the coincident responses for one panelist has been given in Table II. For this panel member the factor $n$, i.e., the number of positive responses, for many of these peaks can be expressed according to one of several options. For example, the odor-active region at RI 1088 for beer A demonstrates the possible range of values for $n$, depending upon the criteria chosen. In this case $n$ is calculated as the number of coincident responses, $n = 6$, but could be regarded as the last dilution where an odor was detected, $n = 9$; the number of the highest dilution before a gap in the coincident responses occurred, $n = 2$; or the geometric mean between dilutions 2 and 9.

**Within-Individual Reproducibility.** The reproducibility of the results from an individual was determined by injecting one beer extract at one dilution (dilution 2) five times on successive days for three panel members. The results demonstrate that while the panelists could reproducibly detect the start of an odor, they found that it was more difficult to determine the end of the same odor (Table III). In most cases, the end of the peaks became even harder to detect toward the end of the chromatogram.

Within-individual variation was also noted in the results from the replicate series of dilutions, with all panelists detecting a greater number of odor-active regions in the first series of dilutions than when the same series of dilutions was repeated.

**Between-Individual Reproducibility.** Duplicate analysis of the standard solution by sniffing demonstrated that although the repeatability within a subject was good, there was a larger difference in response between subjects. A difference between individuals was also noted for the analysis of the beer samples, for both the number and intensity of the odor-active regions detected (Figure 3; Table IV).

The last dilution at which each individual detected an odor-active region was also found to vary by up to four successive dilutions for the same retention index, i.e., from a FD of 3 to 243.

**Data Treatment.** The aromagrams presented in Figure 3 indicate clearly that the method by which the data are treated can lead to quite different results. The rank order by intensity of the odor-active regions was found to be different for most panelists when the data were presented as log $s$ rather than log FD.

## DISCUSSION

An explanation for the above observations may be found among the physiological studies concerning olfactory performance of groups and individuals. During the sniffing period the panelists must decide when they perceive an odor and give a simple yes/no response according to this decision. According to the signal detection theory (Engen, 1971; Köster, 1975) the signal due to a stimulus is always assessed against the effect of noise and background factors mainly due, in this case, to the chromatograph. The subject must therefore decide if a perceived signal is above the background noise before making this yes/no decision; i.e., the subject sets a personal response criteria. Different people are known to set either more or less conservative response criteria (O'Mahoney, 1991) which may explain the large differences in the number of odor-active peaks detected by the six panelists.

Threshold limits are known to vary greatly among individuals and as such may contribute to the differences observed in the aromagrams presented here (Figure 3).

**Table II. Coincident Response Data for Panelist F**

| | extract of beer A | | | | | | | | | | | extract of beer C | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | dilution no. | | | | | | | | | | | dilution no. | | | | | | | | | |
| KI[a] | 1 | 2 | 3 | 4 | 5 | 7 | 8 | 9 | 10 | 11 | KI | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| 887 | X | X | X | X | – | – | – | X | – | – | 755 | X | – | – | – | – | – | – | – | – | – |
| 939 | X | X | X | – | X | – | X | X | X | – | 852 | X | – | – | – | – | – | – | – | – | – |
| 1006 | X | – | – | – | X | X | – | – | – | – | 877 | X | X | X | – | – | – | – | – | – | – |
| 1046 | X | X | X | – | – | – | X | – | – | – | 936 | X | X | X | X | – | X | – | – | – | – |
| 1088 | X | X | – | – | X | X | – | X | X | – | 1001 | X | X | – | – | X | X | – | – | – | – |
| 1185 | X | – | X | X | – | X | X | – | – | – | 1041 | X | X | X | X | – | – | – | – | – | – |
| 1218 | X | – | X | – | X | – | X | X | – | – | 1086 | X | X | X | – | – | X | X | – | – | – |
| 1268 | X | – | – | – | – | – | – | – | – | – | 1153 | X | X | X | – | X | – | – | – | – | – |
| 1337 | X | – | – | – | – | – | – | – | – | – | 1268 | X | X | – | – | – | – | – | – | – | – |
| 1364 | X | X | X | – | X | X | – | X | – | – | 1357 | X | X | X | – | – | X | X | – | – | – |
| 1444 | X | X | – | – | – | – | – | – | – | – | 1394 | X | X | X | – | – | X | X | X | – | – |
| 1491 | X | – | – | – | – | – | – | – | – | – | 1428 | X | – | – | – | – | – | – | – | – | – |
| | | | | | | | | | | | 1444 | X | – | – | – | – | – | – | – | – | – |
| | | | | | | | | | | | 1456 | X | | – | – | – | – | – | – | – | – |

[a] KI, Kovats index.

**Table III. Reproducibility of the Detection of the Start and End of Odor-Active Regions in Beer Extract A (Dilution 1), $n = 5$**

| panelist D | | | | panelist E | | | | panelist F | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| start | | end | | start | | end | | start | | end | |
| KI[a] | SD[b] | KI | SD | KI | SD | KI | SD | KI | SD | KI | SD |
| 871 | 3 | 895 | 5 | 870 | 0 | 877 | 1 | 743 | 1 | 757 | 4 |
| 928 | 2 | 951 | 9 | 930 | 1 | 935 | 1 | 869 | 0 | 891 | 9 |
| 1043 | 3 | 1053 | 4 | 1037 | 2 | 1045 | 3 | 926 | 1 | 942 | 4 |
| 1173 | 1 | 1187 | 2 | 1177 | 1 | 1181 | 1 | 1031 | 3 | 1058 | 4 |
| 1236 | 4 | 1249 | 6 | 1278 | 2 | 1287 | 4 | 1082 | 1 | 1091 | 2 |
| 1245 | 1 | 1263 | 6 | | | | | 1171 | 2 | 1185 | 3 |
| 1270 | 4 | 1288 | 19 | | | | | | | | |
| 1285 | 4 | 1317 | 11 | | | | | | | | |

[a] KI, Kovats index. [b] SD, standard deviation.

**Table IV. Number of Odor-Active Regions Detected by Each Panel Member**

| panelist | beer A | beer C | panelist | beer A | beer C |
|---|---|---|---|---|---|
| A | 7 | 15 | D | 13 | 16 |
| B | 13 | 17 | E | 7 | 12 |
| C | 5 | 5 | F | 14 | 14 |

Using a model system, Marin et al. (1988) have demonstrated how the dilution sniffing chromatograms reflect the individual sensitivity. With groups of 26–44 subjects, the standard deviation for individual thresholds has been shown to range from 0.78 to 5.31 binary dilution steps (Punter, 1983). Furthermore, a wider distribution has been noted for some compounds, eg., isobutyraldehyde, which has a threshold range of 20 binary dilution steps (Amoore et al., 1976).

The observation of gaps in the coincident responses of a subject has not been discussed by any previous author, although we are certain that our observations are not an isolated case. Such observations, however, can readily be explained as threshold concentration is not a specific point below which stimuli cannot be detected and above which stimuli are always detected but is a region where the stimulus effect varies. It is also possible that at one dilution, dilution 1, a compound (**x**) is not detected as a consequence of cross-adaptation (Engen, 1971) due to the presence of another prior-eluting compound (**y**), yet at dilution 2 cross-adaptation no longer has an effect and compound **x** can be detected. However, this phenomenon has propably not occurred as all odor-active regions were detected at the higher concentration.

Our results have demonstrated that it is more difficult for a panelist to detect the end than the beginning of an odor-active region. This may be caused by the physical properties of the compounds, fluctuations in adaptation from one trial to another one, or physiological reasons not yet understood.

Several authors have demonstrated that the olfactory sensitivity of an individual changes both throughout the day as well as over longer time periods (Köster, 1965, 1968; Good et al., 1976). As the assessment of a series of dilutions by GC sniffing was conducted over a number of weeks, the variation in sensitivity and threshold level for each individual may contribute to the lack of reproducibility between complete charm aromagrams and the gaps in the coincident responses. This lack of repeatability for an individual for replicated chromatograms has also been noted by McDaniel et al. (1990) during quadruplicate analysis of a Pinot noir extract. Darriet et al. (1991), however, have reported a coefficient of variation of 3% for the detection of an odor in one region of the chromatogram and for one sniffer. This result may be better than that normally observed as the results were presented for one area of the chromatogram and for the detection of only one peak, thereby removing the possibility of fatigue caused by the length of the analysis. Grosch (personnal communication) indicates that, according to his experience, the sniffing of an extract could be performed by one panelist within 2 days to avoid the appearance of gaps.

Although both research groups have used the same method of EDSA, differing only in the dilution factor chosen, the data have been treated differently, yielding either charm values (Acree et al., 1984) or FD values (factor of dilution) (Gasser and Grosch, 1988). The charm value ($c$) is calculated from computer-recorded response data according to the formula $c = d^{n-1}$, where $n$ is the number of coincident responses and $d$ is the dilution factor. The results are generally presented as the charm index, or logarithm of the surface area vs retention index (RI). The charm value is equal to the ratio $a_1/a_n$, where $a_1$ is the amount of odor-active compound eluting from the most concentrated sample and $a_n$ is that amount eluting from the most dilute sample producing odor response; this is equivalent to the OUV value (Acree et al., 1984).

The different results obtained from each method of treating the same data suggest that either (a) the differences observed were due to large errors in the charm calculation caused by the nonreproducibility of the start and end of the odor-active regions or (b) both results are valid but are not measures of relative intensity and correspond to two different indices.

## CONCLUSION

Analysis of the beer extracts by EDSA demonstrated that both extracts contained many odor-active regions, the number of which depended upon the individual assessor. Beer C was generally characterized by a greater number of odor-active regions, as well as a greater intensity of odor than beer A. Description of these regions demonstrated that beer A had more confectionary-type aromas compared with beer C, which was described as being more coffee-like.

The contribution to the odor of a product by an individual compound may be better determined by charm analysis than by the FD as the surface area of the peak; i.e., the length of time the odor is perceived is taken into account rather than just the final dilution at which a compound was detected. Our results have indicated that it is more difficult for a panelist to detect the end of an odor-active region than the beginning. The period over which a compound is detected will therefore vary, influencing the surface area of each odor-active region. The error involved when the results are presented as the logarithm of the surface area of the odor-active regions will therefore be greater than if the results are expressed as the dilution factor vs Kovats index.

At present it is difficult to state which method gives the most valid results, and as such we feel that it is best to treat the data obtained by both methods and compare the results obtained. If there is a large discrepancy between the two sets of results, then the data for each individual panelist must be closely scrutinized. To ensure that the results are valid, all variables must be closely monitored, including the temperature of the room, the physiological and psychological health of the panelists, and the state of the extracts.

Furthermore, due to the problems of gaps in the coincident responses as described above, it is extremely important to state in detail how the data have been processed to determine the value of $n$. It must also be stated if the sniffing was conducted beyond the dilution at which the first negative response appeared for a certain retention time.

Further work needs to be conducted with a standard solution to determine if the problem associated with the detection of the end of an odor is linked to the type of compound, or to fatigue of the sniffer, or to design of the sniff port.

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