

(Table IX). (e) Exposure of dyed, permethrin-treated wool fabric to light in a Fade-O-Meter causes slow but measurable degradation of permethrin in the wool (Table XI). These results suggest that exposure of permethrin-treated wool to sunlight over long periods will lead to measurable degradation of permethrin. This slow loss is expected to have little practical effect, but deserves further study; cf. Holmstead and Casida (1978).

In conclusion, this study demonstrates that it is possible to impart multibenefit finishing treatments to wool to give products that are moth, flame, and shrink resistant. We believe these multipurpose finishes merit consideration for commercial use.

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Comparison by Twelve Laboratories of the Odor Qualities of Nine Chemicals Sniffed from the Bottle and as Gas-Liquid Chromatography Effluents

Andrew Dravnieks,¹ Harry C. McDaniel,² and John J. Powers*³

Twelve laboratories carried on a collaborative study to compare the odors of nine pure chemicals as sniffed from the bottle and as gas-liquid chromatography (GLC) effluents at three different concentrations. Odor intensities varied according to known psychophysical functions. There was some confusion between odors of sequentially eluted odorants, but it was not great and it could be reduced by improvements in design of GLC splitter ports, suggestions for which are made. Many of the problems of contamination of one odorant with another are the same involving mass spectrometric identification. The recognizability of an odor was not greatly impaired when the odorant was delivered from a GLC effluent port as compared with sniffing the pure compound. Although 126 judges generated 136 descriptor terms for the nine chemicals, the terms could be classified into 22 groups and there was good agreement among the 12 laboratories involved.

Much of the information we have about the odor and taste qualities of chemicals—including several poisonous ones—came to be because early chemists used their senses

of odor and taste as analytical instruments. The practice has fallen into disuse except for two kinds of chemists. The traditional perfumer or flavorist still uses his nose and remarkable memory to catalogue hundreds of compounds and often to identify the source of components of a mixture. Gas chromatographers constitute the other group. They frequently "sniff" compounds as they emerge from a gas-liquid chromatography column to evaluate their odor qualities and sometimes use sensory analysis as an aid in identification. There are questions whether the odor qualities of a compound are the same when sniffed from a bottle or a perfumer's stick and as a hot vapor coming from a GLC instrument. Furthermore, there are questions

Odor Sciences Center, IIT Research Institute, Chicago, Illinois 60616.

¹Present address: 211 Tampa St., Park Forest, IL 60466.

²Present address: Toilet Good Division, Procter & Gamble, Cincinnati, OH 45224.

³Present address: Department of Food Science, University of Georgia, Athens, GA 30602.

Table I. Components and Percent of Each Component in Three Mixtures Formulated

material	percent of component in mixture		
	1	2	3
1. 1-butanol	30	3	0.3
2. pyridine	3	0.3	30
3. cyclohexanol	0.3	30	3
4. <i>p</i> -cresyl methyl ether	30	0.3	3
5. propylene glycol	3	30	0.3
6. acetophenone	0.3	3	30
7. <i>l</i> -carvone	30	0.3	3
8. anethole	3	30	0.3
9. phenylethanol	0.3	3	30
	99.9	99.9	99.9

as to whether descriptions assigned by one laboratory are comparable with those which would be used by another group of investigators.

Subcommittee E-18.04 of Committee E-18 (Sensory Evaluation of Materials and Products) of the American Society for Testing & Materials undertook a cooperative experiment, involving 12 laboratories at home and abroad, to answer some of these questions.

The following criteria were used in the selection of odorant substances. (a) The individual materials must encompass high, medium, and low aroma intensities as well as nondescript and hedonically characteristic, pleasant and unpleasant aroma characters. (b) The individual materials must be commercially available in 99+% GLC purity and lack trace odor contaminants of high odor intensity. The individual materials must also be stable on storage, both individually and in mixtures, so that the criteria of GLC purity is not compromised over the several months of the experiment. (c) The individual materials must be liquid at room temperature and miscible in all proportions with each other so that the materials can be sniffed as is and from the GLC effluent in the same liquid form. (d) The individual components must be easily separable from each other by GLC analysis using readily available column liquid substrates (e.g., Carbowax 20M). (e) Mixtures of individual components must involve widely different concentrations (e.g., 30, 3, 0.3%) so that the effect of concentration of material on aroma intensity and character might be assessed.

EXPERIMENTAL SECTION

Materials. Using the criteria above, nine individual materials and three mixtures with each of these nine materials were selected and are listed in Table I.

GLC Conditions. Carbowax 20M was selected as an appropriate column stationary phase. The various co-operators were instructed to inject whatever amount was needed according to the split ratio they were using so that 1 μ L would go to the sniffing port. Other GLC conditions (e.g., isothermal vs. temperature programming, etc.) were left to the discretion of the individual laboratories. One laboratory (coded no. 9 in this article) used a FFAP column, a modified Carbowax 20M.

Sniff Test Instructions. The laboratories were asked to use both experienced and inexperienced judges and to report their results separately. The GLC effluent was to be sniffed in the manner that was customary and normal for the laboratory and judge. Odor intensity measurements were on a 0-5 scale, with 0 = no odor; 1 = odor barely perceptible; 2 = weak odor; 3 = moderate odor; 4 = strong odor; and 5 = extremely strong.

Each individual judge was permitted to describe the aroma character with a free-choice description. Each laboratory was requested to have some of its judges carry

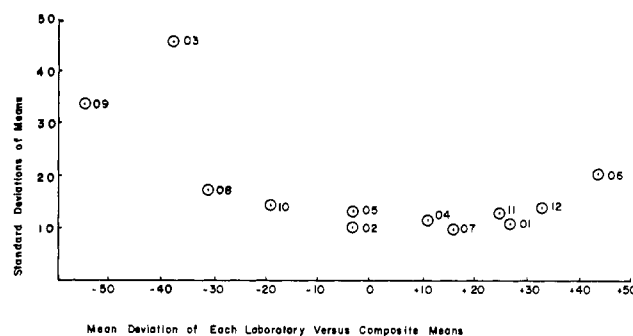


Figure 1. Comparison of mean deviation of each laboratory in determining Kovats Index vs. the composite mean and relation of laboratory standard deviation to departure from composite mean. The numbers are for the various laboratory codes.

on sniffing as is (from a bottle) first and then to do the GLC effluent sniffing, while other judges were to do the two types of sniffing in the reverse order.

Participating Laboratories. The initial group of participating laboratories was drawn from ASTM Committee E-18 membership, but other laboratories became participants and are included in this report. The 12 laboratories were the following: Campbell Soup Co., Food Research Institute (Australia), Food Research Institute (England), Illinois Institute of Technology Research Institute, International Distillers and Vintners (England), McCormick & Co., Mead-Johnson Co., Olin Mathieson Co., Pepsico Co., Procter & Gamble Toilet Goods Division, Swedish Institute for Food Preservation Research (now the Swedish Food Institute), and the University of Georgia. They are listed alphabetically here, but in the tables where laboratories are designated by a number, the order was changed so as not to reveal the identity of the laboratory.

Data Analysis. Data from the participating laboratories were collected and key punched for the various forms of computer analysis, which will be described as the results are presented so as to avoid confusion.

RESULTS AND DISCUSSION

The results will be discussed in two major categories: (a) the rather straightforward odor intensity scores and the Kovats Indices measurements and (b) the odor-description results.

Average Odor Intensity. Table II gives the average odor intensity for each of the nine chemicals as a pure component (100%) and as a GLC effluent from the three mixtures, separately for experienced and novice panelists. Table III lists odor intensity ratings by laboratories.

Kovats Index Values. Table IV summarized the Kovats Indices by laboratories vs. the composite (pooled) data. Laboratories differed systematically in their means from the composite indices and the deviations themselves changed with chemicals more for some laboratories than for the others. Figure 1 shows that laboratories, the data of which exhibited less scatter, reported Kovats Indices closer to the composite values than the other laboratories. Just as each laboratory determined its own GLC operating conditions, the laboratories were allowed to make the Kovats Index calculations either graphically or mathematically according to their usual practice.

Influence of Concentration on Odor Intensity Rating. Figure 2 is a plot of data for experienced judges in terms of odor intensity score vs. the logarithm of the dilution factor. The propylene glycol curve appears to represent only statistical noise. For the others, the GLC-presented stimuli approximately follow the psychophysical relation: intensity score = $\log K + n \log$

Table II. Mean Values of Odor Intensity Ratings for Experienced (E) and New (N) Panelists (in parentheses, number of panelists)

chemical	from bottle concn code: 1000			30% from GLC concn code: 300			3% from GLC concn code: 30			0.3% from GLC concn code: 3		
	E	N	all	E	N	all	E	N	all	E	N	all
1-butanol	3.57 (76)	3.43 (44)	3.52 (120)	3.27 (71)	2.65 (62)	2.98 (133)	2.23 (71)	1.99 (64)	2.12 (135)	1.44 (72)	1.38 (62)	1.41 (134)
pyridine	4.54 (70)	4.78 (35)	4.62 (105)	4.17 (71)	4.41 (62)	4.28 (133)	3.64 (75)	3.28 (68)	3.47 (143)	2.49 (75)	1.95 (68)	2.23 (134)
cyclohexanol	2.76 (75)	3.00 (44)	2.85 (119)	3.04 (71)	2.74 (63)	2.90 (134)	2.32 (71)	2.02 (61)	2.18 (132)	1.39 (70)	1.03 (62)	1.22 (133)
<i>p</i> -cresyl methyl ether	3.68 (76)	3.57 (44)	3.64 (120)	3.92 (71)	3.79 (62)	3.86 (133)	3.29 (72)	2.87 (59)	3.10 (131)	2.55 (71)	2.17 (63)	2.37 (134)
propylene glycol	0.45 (76)	0.39 (43)	0.43 (119)	1.14 (69)	1.50 (63)	1.31 (132)	1.14 (71)	1.12 (63)	1.13 (134)	0.97 (72)	1.14 (61)	1.05 (133)
acetophenone	3.84 (76)	3.40 (44)	3.68 (120)	4.29 (72)	3.96 (61)	4.14 (133)	3.72 (71)	3.51 (63)	3.62 (134)	2.89 (71)	2.46 (61)	2.69 (132)
<i>l</i> -carvone	3.67 (76)	3.64 (41)	3.66 (117)	4.19 (72)	3.97 (61)	4.09 (135)	3.46 (72)	3.00 (60)	3.36 (132)	2.88 (72)	2.62 (62)	2.76 (134)
anethole	3.65 (75)	3.28 (45)	3.51 (120)	4.08 (71)	3.65 (63)	3.88 (134)	3.38 (71)	2.99 (62)	3.20 (133)	2.07 (72)	1.87 (60)	1.98 (132)
phenylethanol	3.04 (75)	2.93 (45)	3.00 (120)	3.42 (71)	3.20 (61)	3.32 (132)	2.71 (72)	2.88 (63)	2.79 (135)	1.86 (71)	2.21 (61)	2.02 (132)

Table III. Odor Intensity Data by Laboratories

chemical	concn code	laboratory code											
		01	02	03	04	05	06	07	08	09	10	11	12
1-butanol	1000	3.20		3.50	3.93		3.67	3.00	2.94	3.80	3.70	3.50	3.40
	300	3.80	3.10	3.50	3.16	1.50	2.25	3.00	3.37	2.30	2.89	3.10	3.20
	30	3.20	2.20	2.00	2.85	0.70	1.50	1.82	2.00	1.60	2.10	2.80	2.50
	3	1.80	1.80	1.10	1.65	0.60	1.50	1.18	1.75	0.70	1.40	1.40	1.60
pyridine	1000	5.00		4.75	4.41		4.29	4.58	4.56		4.60	4.88	4.75
	300	4.20	4.70	2.90	4.80	3.90	3.50	3.82	4.57	3.80	4.60	4.50	4.70
	30	3.60	3.30	3.70	3.58	1.50	4.00	3.55	3.54	3.75	3.90	3.80	3.30
	3	3.20	2.30	2.50	3.30	0.70	2.75	2.36	1.46	1.10	2.50	2.50	2.90
cyclohexanol	1000	2.80		3.00	2.69		3.00	2.82	2.72	2.70	3.00	3.20	2.89
	300	3.40	3.40	3.20	3.90	2.00	3.00	3.18	1.38	2.80	3.60	3.50	2.90
	30	3.00	2.40	2.80	2.10	1.30	2.50	1.82	2.41	1.90	2.40	1.80	2.20
	3	2.67	0.80	1.80	0.90	0.50	1.00	1.00	1.88	1.40	1.30	0.80	0.91
<i>p</i> -cresyl methyl ether	1000	3.60		3.80	3.69		3.92	3.46	3.39	3.80	3.50	3.70	3.60
	300	3.20	3.50	3.60	4.26	3.20	4.25	3.55	4.04	4.00	4.10	4.00	4.00
	30	4.00	2.67	3.20	3.60	2.10	2.75	3.18	3.13	3.30	2.79	3.10	3.00
	3	3.60	2.00	0.90	2.70	1.00	2.50	2.55	3.63	1.80	1.80	2.20	2.40
propylene glycol	1000	1.60		0.00	0.79		0.25	0.46	0.11	0.22	0.70	0.20	0.70
	300	2.25	1.10	0.40	0.45	0.30	1.67	0.46	2.42	2.00	1.50	2.30	1.10
	30	2.80	0.30	1.00	0.16	0.40	1.00	0.82	2.58	1.00	1.00	1.60	0.60
	3	2.00	1.10	0.60	0.40	0.40	1.00	0.36	1.68	1.60	0.90	1.80	1.20
acetophenone	1000	3.40		3.40	4.14		3.67	3.36	3.56	3.60	3.30	3.50	3.80
	300	4.40	4.00	4.50	4.65	3.80	4.00	3.91	4.00	4.20	3.90	3.60	4.50
	30	3.80	3.40	3.40	3.85	3.40	3.50	3.27	3.63	3.70	3.30	3.70	4.30
	3	3.60	2.33	2.80	2.95	1.40	4.00	3.00	2.83	3.00	2.00	2.60	2.50
<i>l</i> -carvone	1000	4.00		4.00	3.93		3.58	3.36	3.50	3.70	3.50	3.60	3.33
	300	4.20	4.00	4.50	4.79	3.40	4.25	4.00	3.79	4.10	3.90	4.20	3.90
	30	3.60	3.70	3.90	3.84	2.50	3.00	3.00	3.13	3.30	3.10	3.30	3.80
	3	3.40	2.50	2.70	3.45	2.30	2.75	2.27	2.83	2.90	2.60	2.60	2.40
anethole	1000	3.60		3.70	3.76		3.58	3.27	3.11	3.70	3.20	3.50	3.60
	300	4.40	4.10	4.30	4.30	3.00	4.25	3.82	3.17	3.90	4.10	3.80	4.50
	30	4.00	3.10	3.90	3.37	1.00	4.00	3.09	3.46	3.10	3.00	3.30	3.40
	3	2.60	2.00	1.70	2.00	0.80	1.75	1.91	2.68	1.90	1.60	2.30	1.90
phenyl-ethanol	1000	3.20		3.10	3.24		3.17	2.36	3.06	2.80	2.80	3.00	3.00
	300	3.40	3.70	4.20	3.32	2.70	3.50	2.64	3.13	3.00	3.00	3.50	4.20
	30	3.20	3.20	3.40	2.05	2.00	2.25	2.55	3.33	2.50	2.90	3.00	3.10
	3	2.00	2.00	1.90	1.74	1.70	1.33	1.55	2.52	1.90	2.44	2.70	1.80

concentration. The bottle stimulus because of its different mode of delivery does not fit into the same relation. Most bottle stimuli were judged weaker than the strongest concentration of the same chemical delivered as a GLC effluent pulse. In other words, an odorant in a GLC effluent may be perceived to have a higher odor intensity than when smelled in undiluted form from a bottle.

For each chemical, a least-squares straight-line fit was calculated for a plot of odor intensity scores vs. log of the concentration in the injected samples. Table V lists the resulting parameters.

It is of interest to compare the slopes in Table V with slope obtained for the same odorants in other work. This is possible for 1-butanol for which the slope of log (perceived intensity) vs. log (concentration) was found to be 0.66 by Moskowitz et al. (1974) who used a constant-concentration dynamic-stimuli method (ASTM, 1975).

Dravnieks (1972) estimated that on a 0-5 odor-intensity scale the perceived magnitude of the intensity increases by a factor of 3.6 per category unit. From Table V, the odor intensity of 1-butanol increased by 0.79 category unit when the concentration of 1-butanol in the sample was

Table IV. Kovats Index Data

lab code	deviations of Kovats Indices by laboratory vs. composite values										mean difference from composites	SD of mean
	1-bu-tanol	pyri-dine	cyclo-hexanol	<i>p</i> -cresyl methyl ester	propylene glycol	aceto-phenone	carvone	anethole	phenyl-ethanol			
01	+50	+26	+34	+11	+32	+22	+27	+15	+27	+27	11	
02	+10	+6	+2	+11	-10	-6	-5	-15	-22	-3	11	
03	-158	-42	-28	-5	-11	-26	-22	-18	-34	-38	46	
04	+5	+29	+15	-11	+2	+17	+19	+8	+14	+11	12	
05	+21	-28	-8	-10	+12	-2	-4	0	-7	-3	14	
06	+18	+71	+42	+41	+26	+64	+62	+56	+20	+44	20	
07	+1	+25	+20	+28	+13	+27	+14	+5	+12	+16	10	
08	-10	-67	-50	-25	-17	-33	-31	-26	-21	-31	18	
09	-8	-42	-67	-82	-108	-38	-60	-30	-12	-55	34	
10	-8	0	-13	-2	-26	-18	-24	-43	-35	-19	15	
11	+47		+22	+15	+39	+14	+13	+22	+31	+25	13	
12	+34	+58	+30	+30	+55	+26	+20	+17	+26	+33	14	
					Composite Values							
	1098	1212	1398	1455	1562	1676	1762	1838	1904			
					Standard Deviations							
	53	44	34	32	42	38	32	27	24			

Table V. Slope and Straight-Line Fit Parameters for Intensity vs. Log (Concentration) Relationship for GLC-Effluent Stimuli

chemical	slope of intensity score vs. log concn			correlation coeff.		
	exper. judges	new judges	all judges	exper. judges	new judges	all judges
1-butanol	0.91	0.64	0.79	0.997	1.000	0.998
pyridine	0.84	1.23	1.03	0.978	0.999	0.993
cyclohexanol	0.83	0.86	0.84	0.997	0.996	0.997
<i>p</i> -cresyl methyl ether	0.69	0.81	0.75	0.999	0.997	0.998
propylene glycol	0.09	0.18	0.13	0.870	0.840	0.980
acetophenone	0.70	0.75	0.73	0.994	0.974	0.987
<i>l</i> -carvone	0.66	0.68	0.67	0.998	0.999	0.998
anethole	1.01	0.89	0.95	0.985	0.989	0.987
phenylethanol	2.78	0.45	2.65	0.999	0.980	0.994

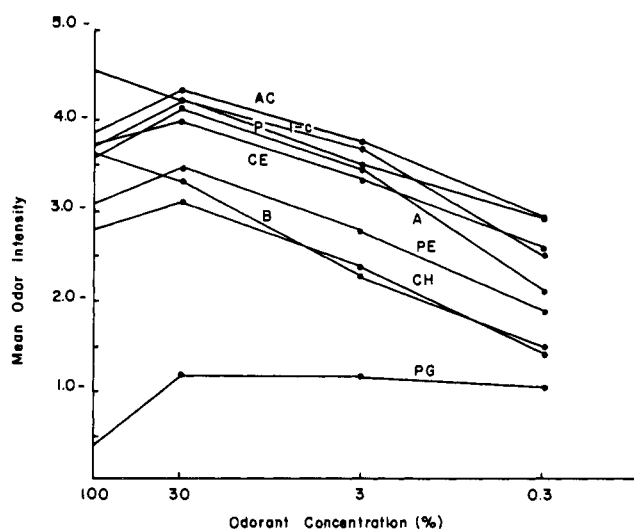


Figure 2. Relation of odor intensity scores to concentration of odorant on semilogarithmic scale. AC, acetophenone; *l*-C, *l*-carvone; P, pyridine; CE, cresyl methyl ether; A, anethole; B, 1-butanol; CH, cyclohexanol; PG, propylene glycol.

increased tenfold. This would correspond to $0.79 \times \log 3.6 = 0.44$, which is the logarithmic increment of the perceived magnitude of the odor intensity per log unit (or tenfold) change in the 1-butanol concentration in the sample.

This is considerably less than was found for 1-butanol by the constant stimulus method. Two explanations are

possible. Because of GLC peak broadening, the concentration at any instant in the GLC effluent during the elution of a peak may not increase in proportion to its concentration in the injected sample (area of peak does increase proportionally). Also, it is known that lower slopes result when odorants are delivered at slower rates, as in static sniffing from flasks.

Influence of Judge Experience on Intensity Rating.

A comparison of the data in Table II indicates that experienced judges tended to give higher odor intensity ratings than novice judges did when the chemical was smelled from a bottle. Pyridine and cyclohexanol are exceptions, perhaps because of their pungency. However, the number of comparisons (nine stimuli) is too small to draw significant conclusions.

When the stimuli were delivered via the GLC mode, the relation is clearer. If only real odors (excluding propylene glycol) are considered, in 22 out of 24 types of stimuli the experienced judges gave a higher odor-intensity rating. This is significant at $p = 0.01$, by the sign count test.

Correlation between Lab and Composite Intensities.

Correlation coefficients between the odor-intensity data averaged by laboratories (Table III) and the composite values (Table II) are listed in Table VI.

The absolute numerical values can be compared only for the odors smelled from bottles since here the vapor concentrations presented to different laboratories were the same, within limits of possible temperature differences. For the bottle stimuli (code 1000), correlations between laboratories and the composite were calculated using a least-squares regression-fit equation. The correlation

Table VI. Odor Intensity Data: Correlations of Data from Individual Laboratories vs. Composite Data (nine stimuli)

lab code	bottle linear regression		rank correlations for GLC stimuli at concentrations		
	r	slope	30%	3%	0.3%
1	0.932	0.74	0.76 x ^a	0.72 x	0.77 x
2			0.91 xx	0.90 xx	0.93 xx
3	0.990	1.17	0.54	0.79 xx	0.67 x
4	0.966	0.89	0.93 xx	0.85 xx	0.93 xx
5			0.97 xx	0.77 xx	0.85 xx
6	0.995	1.09	0.71 x	0.89 xx	0.88 xx
7	0.981	0.95	0.85 xx	0.93 xx	0.88 xx
8	0.988	1.03	0.63 x	0.85 xx	0.66 x
9	0.992	1.10	0.80 xx	0.95 xx	0.63 x
10	0.983	0.89	0.84 xx	0.97 xx	0.87 xx
11	0.991	1.07	0.69 x	0.96 xx	0.71 x
12	0.990	0.93	0.87 xx	0.92 xx	0.84 xx

^a x = significant at $p < 0.05$; xx = significant at < 0.01 ; all correlation coefficients for bottle stimuli, significant at < 0.001 .

Table VII. Total Odor-Profile Word-Description Count Results^a for Nine Aromatic Chemicals from Experienced Judges

chemical	total odor-profile word-description count for chemical at concn code of			
	1000	300	30	3
1-butanol	79 (0)	70 (7)	71 (7)	66 (12)
pyridine	72 (0)	70 (7)	75 (6)	68 (9)
cyclohexanol	76 (1)	70 (8)	68 (10)	59 (19)
<i>p</i> -cresyl methyl ether	78 (0)	71 (6)	71 (7)	69 (9)
propylene glycol	56 (22)	51 (27)	51 (26)	55 (23)
acetophenone	78 (0)	72 (6)	70 (8)	71 (6)
<i>l</i> -carvone	77 (0)	72 (6)	72 (6)	70 (9)
anethole	77 (0)	71 (7)	71 (6)	69 (9)
phenylethanol	77 (0)	71 (6)	70 (9)	65 (12)

^a Number count of word descriptions and in parentheses number of missing descriptions.

coefficients are all high ($p < 0.001$). The slopes of the regression lines fluctuated around unity but showed considerable differences between the usage of the category scale between laboratories, although the relative positions of nine odors were closely correlated from the laboratory and the composite values. The mean slope over all laboratories is close to unity.

For stimuli presented as GLC effluents, the absolute numerical values of the intensities might be expected to vary from laboratory to laboratory since GLC parameters, split ratio detector/sniffing port, were not standardized except for the general instruction to deliver 1 μ L to the sniffing port; otherwise each laboratory followed its own practice. Here the comparison of the laboratories to the composite was conducted using Spearman's rank corre-

lation test. The values in the table indicate only one case where the correlation did not reach $p = 0.05$.

Correlations were best at the intermediate concentration (3%, code 30) of the chemicals. Apparently, there is more disagreement in rating the weak and strong stimuli. Correlations indicate that the intensity ratings of GLC effluent odors are relatively stable among the laboratories.

Use of Descriptors. There is a need to standardize terminology as to various odors or at least know what one industry means when it uses certain words as compared with another industry. This is the reason laboratories from different areas (food, chemicals, toilet goods, research institutes) and from different countries were included as collaborators. This is also the reason free choice of descriptors was permitted, so as to ascertain the range of descriptive terms which might be used. A third reason is that sniffing of gas chromatographic effluents is becoming more common as a means of training individuals to detect and describe odors. It is thus important to know whether the use of descriptive terms varies in comparison with those resulting from more traditional means of odor presentation.

Odor Descriptor Profiles. In total, 158 different terms (after combining different spellings or word forms of the same term) were utilized by the participants to characterize the odors of the nine chemicals. In average, 1.25 terms per stimulus per judge were used; some used consistently only one term at a time, others used several terms for the same stimulus.

Table VII gives the odor-profile word-description count for the same chemicals/concentrations for experienced judges. Each individual word description was treated distinctly according to the first eight letters of the word description so that SPEARMIN covers "spearmint", "spearmint-peppermint", etc., but not "chewing gum-spearmint, peppermint-spearmint", etc.

Table VIII gives the highest odor-profile word percentage for the same chemicals/concentrations. The percentages in Table VIII do not include the "number of missing descriptions", and the selection of the highest percentage is done alphabetically if more than one odor-profile word description has the same "highest" percentage.

Computer analysis was initially used to sort out the descriptions, but manual classification was used at the end because judgement had to be exercised when the same terms were used but in a different order (see example above of spearmint-peppermint vs. peppermint-spearmint).

Twenty-two clusters resulted, based on the frequencies-of-use of descriptors, common knowledge of odor similarities, reference to the Givauden Index (1961) and the books of Moncrieff (1967) and Harper et al. (1968). The clusters are listed in Table IX. Raw data were scanned word-by-word for every stimulus and every pa-

Table VIII. Highest Odor Profile Word Percentage Results for Nine Aromatic Chemicals from Experienced Judges

chemical	highest odor profile word percentage for chemical at concn code of			
	1000	300	30	3
1-butanol	11.4% butanol	10.0% butanol	9.9% ester	9.1% butanol
pyridine	65.3% pyridine	64.3% pyridine	54.7% pyridine	32.4% pyridine
cyclohexanol	14.5% medicinal	5.7% medicinal	4.4% floral	10.1% sweet
<i>p</i> -cresyl methyl ether	5.1% aromatic	2.8% benzaldehyde	5.6% aromatic	5.8% medicinal
propylene glycol	17.9% "no odor"	11.8% "none"	7.8% "none"	10.9% "none"
acetophenone	9.0% carvone	8.3% benzaldehyde	5.7% benzaldehyde	8.5% floral
<i>l</i> -carvone	46.8% spearmint	48.6% spearmint	47.2% spearmint	47.1% spearmint
anethole	27.3% aniseed	28.2% aniseed	22.5% aniseed	18.8% aniseed
phenylethanol	7.8% floral	14.1% floral	12.1% floral	15.4% floral

Table IX. Twenty-Two Descriptive Clusters Formed from 136 Terms

minty scan	minty, peppermint, spearmint, menthol, carvone, dentine, chewing gum, green mint, caraway seed	solvent scan	solvent, benzene, acetone, ketone, ketonic, hydrocarbon, nail polish remover, cleaning fluid, dry cleaner, chloroform, acetylene, kerosene
almond scan	almond, benzaldehyde, nitrobenzene, marachino, cherry, acetophenone, marzipan, shoe polish, boot polish	fishy scan	fishy, amine, organic base, rotten fish, seafood
anise scan	anise, licorice, anethol, root beer, aniseed, anisaldehyde, black jelly beans, cough sweets, paregoric, fennel, cough lozenges, pepsin medication, saffras	green scan	green, cut grass, aldehyde, herbaceous, grassy, weeds, cucumber
rose scan	roses, rosey, etc. phenylethylalcohol, phenylethylaldehyde, rose petals	camphoraceous scan	camphor, etc., cedarwood, eucalyptic, ^a chest-of-drawers, mothballs, naphthalene
floral scan	does not include those under Rose Scan) floral, flower, flowery, names of various flowers except roses, ionone, gerandol, phenyl ethyl ether	rancid scan	rancid, sweaty, cheesy, sweaty socks, sweaty feet, bed bugs, ^a old butter
alcoholic scan	butanol, butylalcohol, alcoholic, various aliphatic alcohols named, fusel	spicy scan	spicy, spice etc., cinnamon, cinnamaldehyde, eugenol
medicinal scan	medicinal, antiseptic, phenol, cresol, p-cresylmethylester, clinical, surgical, hospital, antibacterial solution, salve for sore muscles, lysol, library paste, cyclohexanol, wintergreen, eucalyptus ^a	pungent scan	pungent, sharp, harsh, sour, acid, acrid, formic acid, formaldehyde, bed bugs, ^a acrolein, mustard, iodine, tangy, astringent, stings, prickles nose, sense in throat, penetrating, suffocating, irritating
pyridine scan	pyridine, methylated spirits, Karl-Fisher reagent	resinous scan	resinous, resin, balsamic, woody, piney, pine, terpene, turpentine, evergreen furniture
fruity scan	fruity, fruit, etc., banana, apple grape, estery, names of berries, candy, sweet wine, chemical names of various aliphatic esters	chemical scan	chemical
sweet scan	sweet	oily scan	oily, varnish, candle, paint
		garlic scan	garlic, onion, skunky, sulfury, sulfuretted, disulfide, H ₂ S, methylmercaptan, cabbage, natural gas, onion grass, sweet onion
		fragrant scan	fragrant, aromatic, perfume
		left outside of clusters (non-classified)	ethereal, musty, earthy, stale, floor polish, waxy, milk, faint, pleasant, unpleasant, strong, weak, something, some unidentifiable words, also descriptor "nonidentifiable"

^a The descriptors, eucalyptus and "bed bug", both with low frequency of use were classified into two clusters.

nelist. Thus, each stimulus became represented by counts of usage in each of 22 clusters. The resulting 22-cluster profiles describing the 36 stimuli are shown in Table X. These will be termed *composite* profiles, pooled from the 12 laboratories.

Included in Table X are also counts of leftover descriptors, not fitting well into the selected clusters, or very little used, unintelligible, or noninformative as to the quality, e.g., "faint". Also shown are counts of "no-odor" reports.

Table XI contains usage frequencies of various clusters by laboratories and some simple statistical parameters derived from these frequencies.

The central theme of the discussion of odor descriptors is not a comparison of responses of individual judges but a comparison of relations within the composite data and of data pooled over specific laboratories vs. the composite data.

There were 126 judges in total among the 12 laboratories and they generated 136 responses for 36 stimuli. Laboratory no. 4 used ten panelists, but duplicated all data, resulting in 20 responses per stimulus; for simplicity, its data are considered to result from 20 panelists.

The principal questions are the following: (1) to what extent does composite data reflect expected relations between various stimuli, (2) what is the effect of GLC mode of presentation as compared with direct sniffing of vapors of the same substance from bottle, and (3) to what extent do data of separate laboratories correlate to composite data, which are likened to "panelist population" response?

Odor Descriptor Data. The data in Table XI indicate that the percentage of descriptors fitting into clusters

ranged from 70 to 91, with a mean value of 81%. Thus, there was relatively little difference between laboratories in producing descriptors though several of the compounds were unfamiliar to most of the inexperienced panelists.

The average number of descriptors (clustered plus unclustered) per laboratory ranged from 1 to 1.9. Ranks for both parameters are shown for possible comparisons in various types of responses of different laboratories. In this case, there is no relation between the tendency to use clusterable descriptors and the usage of more words per stimulus.

Odor Profile Comparisons. Table X characterizes each of 22 clusters by a specific count of descriptor usage for all descriptors assigned to that cluster. Two stimuli at a time can be compared on the basis of these counts using straight-line best-fit plots.

In working with counts, it is frequently advantageous to reduce the curvature of the plot by first transforming each count by a square root operation; thus, (transformed count) = $\sqrt{\text{count} + 0.5}$. This procedure was used here. For 22 data points, the correlation coefficient must exceed 0.50 to reach the $p < 0.02$ statistical significant level.

Same Odorant, Different Modes of Presentation. Table XII gives sets of r values for each odorant separately for each pair of its different modes of presentation. All values are high exceeding $p = 0.0001$, with the exception of cyclohexanol, acetophenone, and *p*-cresyl methyl ether in some modes of presentation.

Here two comments apply. First, in the 0.3% GLC presentation mode, the cyclohexanol odor was weak (cf. Table II); it is the weakest of all GLC stimuli with the

Table X. Composite Profiles of Odor Stimuli

chemical	concn code	min-ty	al-mond	an-ise	rose	flo-ral	alco-hol	me-dic-i-nal	pyr-i-dine	names of clusters											frag-rant	not class	no odor			
										fruity	sweet	sol-vent	fishy	green	cam-phor	ran-cid	spi-cy	pun-gent	resi-nous	chee-sy				oily	gar-lic	
butanol	1000						71	4	2	12	6	25	1	7	2	6	13	1	3	2	2	25		7		
	300					4	46	3	2	19	9	21		6	1	5	10	1	2	4	4	4	24		8	
	30	3	1	2		7	35	2		12	21	7		10	1	10	13	3	2	5	5	5	41		34	
pyridine	3	3			1	5	32	1	2	7	11	4		2		5	4	3	1			5	41		1	
	1000						4	1	79	1	1	4	13	1	1	1	13	1	3	1	1	7	1	30		1
	300						2	1	85	1	3	13	10	2	4	4	12	2	2	1	1	6	6	28		1
cyclo-hexanol	30	2	2	1		3	5		73	3	3	10	2	2		1	10	2	2	1	6	6	29		7	
	3	3	1	2	1	6	3		38	4	9	5	11	2	17	1	5	6	3	4	2	2	40		15	
	1000					2	2	61		4	6	6	1	2	9	3	6	4	2	11	2	5	33		5	
<i>p</i> -cresyl-methyl-ester	300	4	2	3		9	10	47		8	11	10	2	3	3	3	3	4	1	12	1	6	47		14	
	30	5	2	2		10	14	16	1	7	18		1	3	2	1	2	2	4	1	12	6	47		37	
	3	2	5	6		9	4	5		8	7	18		8	2	1	2	1	4	5	5	6	37		1	
propyl-ene	1000	9	10	11		6	1	19		11	14	21		2	18	8	6	7	3	8	7	12	44		1	
	300	7	10	12		7	4	18		5	6	13		1	18	3	8	6	7	3	14	15	43		2	
	30	6	11	22		8	5	10		11	16	16	1	1	9	4	5	5	4	7	7	10	34		9	
aceto-phenone	3	7	7	16		13	4	19		4	15	8		3	9	4	4	2	5	2	2	5	38		9	
	1000					3	1	1		2	9	1	1	1	1	1	1	1	1	1	1	1	27		93	
	300	7	4	1		13	4	4		8	10	3		1	1	4	2	2	2	2	2	4	46		50	
glycol	30	8	2	5		13	5	2		10	13	5		2	1	1	1	2	2	2	2	2	31		61	
	3	3	3	4		14	3	3		4	10	9		3	1	3	6	7	1	4	4	3	38		56	
	1000	4	45			11	3	4		4	6	22	1	3	9	8	6	7	4	12	10	8	46		2	
<i>l</i> -carvone	300	3	43			14	3	4		15	11	16		2	12	5	8	2	1	10	4	11	46		2	
	30	2	35			33	3	10		11	17	11	1	6	7	7	7	2	3	4	4	31		9		
	3	2	26			35	2	3		9	16	8		3	7	8	8	7	2	4	4	11	38		5	
aneth-ole	1000	113				3	2	2		2	4	4		4	4	1	1	2	3	1	1	3	11		1	
	300	120				1	1	3		2	4	2		3	1	2	2	2	3	1	1	4	15		1	
	30	111	4	7		7	1	2		2	6	6		2	6	3	2	2	2	2	2	3	12		2	
phenyl-ethanol	3	100	1	7		11	2	2		4	9	3		4	4	1	1	1	1	1	1	2	15		1	
	1000	9				1	2	6		2	4	1		2	2	1	1	1	1	1	1	1	12		1	
	300	10				5	1	2		3	6	2		1	1	3	3	3	1	3	3	2	12		2	
phenyl-ethanol	30	18				3	1	1		3	7			3	1	3	2	2	1	3	1	3	18		14	
	3	14				11	1	1		5	7			3	1	1	2	2	1	3	1	3	18		14	
	1000	3	2	2		65	1	1		2	15	4		7	1	3	3	3	1	3	1	9	32		1	
ethanol	300	7	1	1		32	75	1		2	14	4		4	3	1	3	1	1	1	1	9	34		1	
	30	6		13		29	60			2	18	2		4	1	1	4	2	2	1	1	8	28		2	
	3	14	1	2		17	53	1	2	8	9	4	1	4	2	3	4	4	4	1	1	8	37		8	

Table XI. Usage of Clusters as Number of Counts by Code for Cooperating Laboratories

cluster	1	2	3	4	5	6	7	8	9	10	11	12	total
MINT	19	42	37	74	27	49	42	142	43	39	47	51	612
ALMO	9	4	8	45	12	21	23	37	16	26	17	13	231
ANIS	20	41	41	82	39	40	47	45	39	46	42	55	537
ROSE	3	22	1	12	11	36	6	10	5	14	6	2	128
FLOR	15	24	35	64	41	14	25	135	33	19	46	59	510
ALCO	15	22	10	39	10	23	26	55	26	14	32	11	283
MEDI	12	7	15	43	20	21	22	55	19	11	12	30	249
PYRI	18	3	26	51	12	19	29	48	11	21	25	29	292
FRUI	0	18	27	43	23	9	18	29	14	8	23	1	213
SWEE	10	7	16	39	23	12	18	83	25	30	30	64	357
SOLV	13	23	8	66	12	24	20	30	12	24	21	18	271
FISH	0	9	0	9	7	8	3	1	8	1	3	4	53
GREE	1	2	10	17	5	1	7	11	3	13	4	29	103
CAMP	0	1	5	30	13	2	14	19	27	5	13	8	137
RANC	0	7	1	10	3	0	1	8	7	0	0	1	37
SPIC	7	3	10	9	3	4	6	12	9	7	3	8	81
PUNG	15	7	2	46	23	1	14	14	5	16	5	49	197
RESI	0	6	15	4	8	0	5	10	2	10	3	23	86
CHEM	0	2	3	3	15	1	10	11	1	9	0	12	67
OILY	1	14	15	21	14	15	0	11	1	1	3	14	110
GARL	0	7	2	3	0	0	3	1	2	3	8	1	30
FRAG	20	5	4	20	16	0	39	35	11	13	2	22	187
total	178	276	291	730	337	300	378	802	319	330	345	504	4771 = 81%
NCL	42	83	29	122	106	54	95	181	79	139	53	124	1107
% clust	(81)	(77)	(91)	(86)	(76)	(85)	(80)	(82)	(80)	(70)	(87)	(80)	$\mu = 81.3$
rank	7	3	12	10	2	9	5	8	5	1	11	5	
"no odor"	(7)	(26)	(35)	(76)	(63)	(45)	(30)	(49)	(27)	(22)	(21)	(26)	
odor resp.	173	334	325	644	297	387	366	599	333	338	339	334	
descr./	1.3	1.1	1.0	1.3	1.5	(0.9)	1.3	1.6	1.2	1.4	1.2	1.9	$\mu = 1.35$
resp.													(without 6)
rank	7	3	2	7	10	1	7	11	4.5	9	4.5	12	$\mu = 0.25$

Table XII. Correlation Coefficients on $\sqrt{(\text{count}) + 0.5}$ for Comparisons of Composite Profiles of the Same Chemical at Different Modes of Stimulus Presentation

odorant	bottle vs. GC			30% GC vs. GC		3% GC
	30%	3%	0.3%	3%	0.3%	vs. 0.3% GC
1-butanol	0.95	0.78	0.79	0.85	0.82	0.87
pyridine	0.97	0.95	0.86	0.95	0.85	0.92
cyclohexanol	0.89	0.67	(0.26)	0.81	0.42	0.58
cresyl methyl ether	0.91	0.88	0.82	0.84	0.75	0.85
propylene glycol	(0.56)	(0.67)	(0.71)	(0.83)	(0.82)	(0.81)
acetophenone	0.91	0.80	0.75	0.88	0.83	0.93
carvone	0.98	0.97	0.96	0.95	0.93	0.98
anethole	0.98	0.96	0.90	0.97	0.93	0.98
phenylethanol	0.95	0.93	0.90	0.94	0.93	0.89

Table XIII. Correlation Coefficients for Odor Descriptors of Individual Chemicals within Labs for 100% (from bottle) vs. 3% (from GLC effluent)

lab code (IC)	1-butanol Y1 vs. Y3	pyridine Y5 vs. Y7	cyclohexanol Y9 vs. Y11	<i>p</i> -cresyl methyl ether Y13 vs. Y15	propylene glycol Y17 vs. Y19	aceto phenone Y21 vs. Y23	<i>l</i> -carvone Y25 vs. Y27	anethol Y29 vs. Y31	phenyl ethyl alcohol Y33 vs. Y35
1	0.86	0.95	0.57	0.74	-0.09	1.00	1.00	0.93	0.94
2	0.58	0.52	0.78	0.37	-0.10	-0.02	0.93	0.95	0.98
3	0.39	0.94	0.52	0.44		0.61	1.00	0.97	0.81
4	0.80	0.70	0.23	0.73	0.76	0.99	0.97	0.91	0.96
5	-0.08	0.70	0.15	0.17		0.17	0.77	0.23	0.99
6	0.36	0.56	0.38	0.55		0.14	0.99	1.00	0.78
7	0.52	0.82	0.52	0.69	0.84	0.66	0.89	0.97	0.65
8	0.66	0.80	0.37	0.44		0.56	0.61	0.68	0.20
9	0.66	0.65	0.35	0.45		0.26	0.91	0.93	0.31
10	0.41	0.78	0.67	0.62	-0.12	0.63	0.87	0.92	0.86
11	0.90	0.90	0.20	0.42	0.64	0.68	0.97	1.00	0.96
12	0.46	0.89	0.38	0.37	0.30	0.49	0.89	0.96	0.80

exception of the propylene glycol, which when pure is virtually odorless. Also, there were 37 "no-odor" responses to this lowest cyclohexanol stimulus and 37 more responses

out of the 136 available responses that did not fit into clusters, indicating difficulty in the recognition of odor quality.

Table XIV. Correlations^a of Composite Profiles (Square Root Transformations According to Concentration Levels)

	bottle	30% GC	3% GC	0.3% GC	similarity/ dissimilarity trials ^b
ethanol					
pyridine	0.10	0.07	-0.10	0.13	
cyclohexanol	0.29	0.32	(0.63)	0.26	3.59
cresyl methyl ether	0.06	0.13	0.26	0.08	
acetophenone	0.04	0.28	0.20	0.12	
carvone	-0.19	-0.18	0.04	0.06	
anethole	-0.14	-0.11	0.05	-0.07	
phenethanol	-0.15	-0.04	0.10	0.22	
pyridine					
cyclohexanol	-0.25	-0.22	-0.17	-0.03	
cresyl methyl ether	-0.41	-0.40	-0.26	-0.24	
acetophenone	-0.29	-0.28	-0.24	-0.13	
carvone	-0.21	-0.21	-0.13	-0.01	
anethole	-0.21	-0.20	-0.17	-0.02	
phenylethanol	-0.29	-0.29	-0.22	-0.05	
cyclohexanol					
cresyl methyl ether	(0.58)	(0.73)	(0.61)	(0.66)	× 3.02
acetophenone	0.12	0.34	(0.53)	(0.61)	3.50
carvone	0.12	0.08	0.16	0.19	
anethole	0.09	0.05	0.03	0.35	
phenylethanol	-0.16	-0.02	0.08	0.27	
cresyl methyl ether					
acetophenone	(0.58)	(0.61)	(0.63)	(0.56)	× 2.49
carvone	0.18	0.19	0.24	0.35	× 3.01
anethole	0.26	0.27	(0.51)	(0.51)	× 2.52
phenylethanol	0.03	-0.02	0.17	0.30	
acetophenone					
carvone	0.04	-0.06	0.08	0.13	
anethole	-0.27	-0.17	0.00	0.16	
phenylethanol	0.16	0.19	0.36	(0.59)	× 3.14
carvone					
anethole	0.23	0.32	0.45	0.49	× 2.59
phenylethanol	0.08	0.07	0.26	(0.50)	4.48
anethole					
phenylethanol	-0.09	-0.04	0.35	0.31	
Overall Correlations					
bottle					
30%		0.96			
3%		0.87	0.90		
0.3%		0.81	0.81	0.85	

^a When $r = 0.50$ or greater, $p < 0.02$. ^b Sensory distance from direct similarity/dissimilarity comparisons; × = six closest (least different) odor pairs.

Thus, on the basis of the composite profiles, the same odorant yields vary highly correlated profiles regardless of its mode of presentation unless the concentration is so low that a substantial fraction of individuals, here 25%, cannot discern any odor.

Table XIII lists the correlations coefficients for odor descriptions of individual chemicals within laboratories for the concentrations of 100% (from bottle) vs. 3% (from GLC effluent). This comparison can be used as one basis for testing the "importance" of standardized GLC sniffing procedures. The results are mixed in terms of drawing any final conclusions regarding the "importance" of standardized GLC sniffing procedures. All laboratories, for example, did reasonably well for *l*-carvone with no correlation coefficient falling below 0.61. Most laboratories did not do so well for cyclohexanol with only two correlation coefficients out of 12 falling above 0.65 and none above 0.80. The reader can see for himself/herself intermediate degrees of matching.

In net, a high correlation coefficient for one odorant does not necessarily mean a high correlation coefficient for other odorants.

Different Odorants. Table XIV lists correlations, obtained by the same method as above, between clustered profiles of different odorants for the four modes of presentations.

Those with correlations greater than $r = 0.50$ are significant at the $p = 0.02$ level.

Cyclohexanol, *p*-cresyl methyl ether, and acetophenone were easier to confuse with the other two in one or more modes of presentation. This was found to be also borne out by independent measurements: the right-hand column of Table XIV shows values obtained by direct similarity/dissimilarity judgements of several odorants by 35 different panelists from three laboratories in another ASTM E18.04 collaborative, round-robin test (Dravnieks et al., 1978). The evaluation was on a 0-7 scale (0 = no difference). The values marked by asterisks are the smallest six distances found in the comparison of 45 pairs of odorants. Thus, the ease of confusion evidenced by significant values of r in Table XIV relates to the actual relatively high similarity of odors of these odorants.

Note that $p = 0.02$ signifies that the value $r = 0.50$ may still be exceeded in one case out of 50 merely by chance. With 112 values of r in Table XIV it may happen twice and would tend to occur more likely so with pairs where the components smell somewhat alike.

The bottom part of Table XIV shows comparisons over all 28 pairs between different modes of presentation. Correlations are high (these four values are based on the tabular values directly without the square root transform), indicating that the relative odor qualities of these eight

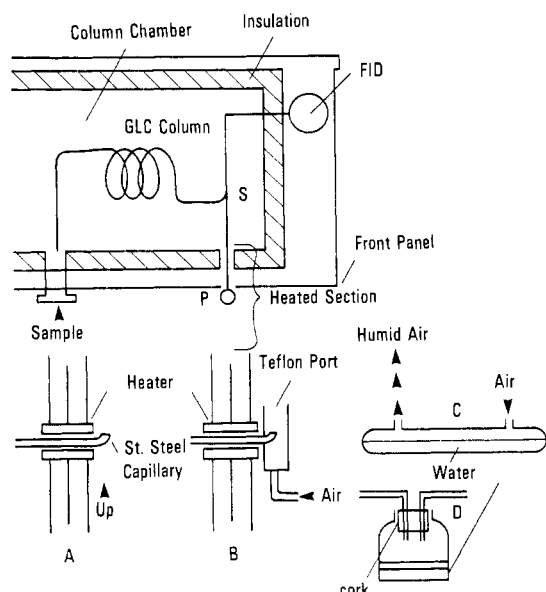


Figure 3. Diagram of sniffing port arrangement, details of exit port, and means of humidifying the effluent.

odorants remain much the same, regardless of the mode of presentation. Only a slight weakening of correlations occurs at lower GLC stimulus concentration, but even there the correlations are quite high.

Profiles of Individual Laboratories vs. Composite Profile. Descriptor profiles for each of 36 stimuli were compared with the composite profiles for the corresponding stimuli. As before, the counts were transformed by the square-root procedure described above. The analysis produced t values from which the probability that the degree of disagreement between a laboratory and the composite profile could have occurred by chance only might be calculated. Out of 432 comparisons (nine compounds, four concentrations, twelve laboratories), only 24 differed at the 0.05 probability level. Actually, one should expect 21.6 values to occur by chance at the 0.05 level out of 432 comparisons. Most of the significant values occurred in comparisons for laboratories no.'s 4, 6, and 8. Since the disagreement may also be observed in the profiles of odors of the bottled chemicals, its origin appears to be in the use of descriptor words rather than the gas chromatographic factors. Data in Figure 1 which show differences between the laboratories and the composite Kovats Indices indicate that these laboratories were not exceptions in their GLC performance.

Application. Based upon the findings above and experience with various forms of splitter assembly, the remainder of this article deals with a discussion of means whereby variation induced by the effluent-sampling procedure might be minimized. Figure 3 shows one such assembly. The splitter delivers a part, usually one-half of the effluent, to a hydrogen-flame ionization detector (FID). The other part is delivered to a sniffing port P outside of the chromatograph. Typically, at least 20 mL/min must be supplied to the FID so that the port also receives the effluent at 20 mL/min carrier gas flow rate.

To prevent condensation or strong adsorption of odorants in the flow to the port, the entire length of the branch leading to the port must be hot. Thus, this branch is heated beginning from the point where the tubing leaves the GLC column chamber, entering the wall of the chamber, and up to the exit where the effluent is sniffed.

Supplementary heating of the port branch in the GLC chamber is undesirable since this interferes with the chamber-temperature control and produces significant

differences in the temperature of the two branches of the splitter, influencing the split ratio. The best arrangement is to have sufficiently long splitter branches in the chamber where they can equally follow the column chamber temperature. Typically, the branches of the splitter may be 60 to 80 cm long, with the section of the port branch in the wall and up to the exit heated to 200–250 °C. Typical tubing for splitters is 1.6 mm i.d. stainless steel tubing, 0.25 mm, or 0.5 mm i.d.; Teflon tubing is not suitable because of large dimensional and flow rate changes with temperature and diffusion of substances through its wall at higher temperatures.

Heating of the port branch in the wall and outside is accomplished by wrapping it with a heating tape, an insulated electrical resistance wire, or placing it in a copper tubing which is heated by an electrical cartridge heater in a bore of a copper rod silver-brazed to the copper tubing.

The sniffing port may be designed in various ways, each of which has advantages as well as disadvantages.

The simplest form is an upward bent in the exit tubing, A, in Figure 3 with the vertical part kept short enough (a few millimeters) to maintain it hot up to the point of emission into air. Form A assures the fastest response of the sniff effluent to GLC column effluent and no opportunity to linger around the exit, with reduction in odor "transfer" to the next peak. This is important with those GLC systems where peaks are narrow and sharp, as in the case of fast programming (e.g., 8 °C/min) of support-coated open tubular columns.

There are several objections to the form A. Hot effluent may irritate the nose, it may smell differently from the same vapor at room temperature, and the hot end may cause a burn if accidentally touched.

Therefore, design B, Figure 3, is frequently used. A Teflon port, e.g., 12 mm i.d. and 20–30 mm long, receives the hot effluent but is also supplied with dilution vapor at 20–80 mL/min. Air is preferable. It may either be room air supplied by an air pump (such as a nonodorous small aquarium pump) or taken from a cylinder and humidified, as in C or D, Figure 3.

In its use, method B is less tiring and more convenient to the sniffer-evaluator. However, with narrow, rapidly sequencing GLC peaks, it allows more chance for odorous vapors to linger in the port, with odors then being assigned to some adjoining peaks.

Freeman and Withycombe (1977) state that there are significantly better materials available for the construction of splitters than those indicated in Figure 3. Specifically, 16 mm o.d. glass-lined stainless steel tubing and the commercially available G.L.T. (SGE, Inc.) configurations are recommended.

Similarly, heating of the port branch is best accomplished by an insulated high-mass, aluminum heating block to alleviate temperature fluctuations. Heating tape and insulated electrical resistance wire tend to permit cold spots and not provide for either a constant or continuous temperature—they cool down, for example, when the oven is cooled.

The effluent tip may be covered with a small piece of Teflon tubing to reduce sample contamination and the risk of burning one's nose while sniffing.

An odor note that may be basically totally inappropriate to an odorant may be assigned to a GLC effluent peak for a variety of causes. First, it may be a genuine impression of the particular odor judge. Second, an odor of the strongly odorous peak-to-come may be building up well in advance of a visible detector response and be assigned to the preceding peak. Third, the concentration of the

odorant in the effluent, even after the detector response appears to have subsided, may still be high enough to exhibit an odor, although a weaker one; here either the column is still feeding the odorant into the splitter ("tailing") or else the odorant is lingering in the splitter or the port, because of an improperly low temperature or adsorption-desorption in the port. Fourth, high-boiling components may even linger into the analysis of the next sample and be assigned to the earlier peaks there. An inspection of Table X hints at marginal possible occurrences of all these effects in the cooperative exercise, of infiltration of pyridine-like notes into 1-butanol descriptors (preceding peak), almond note into 1-carvone (next peak), and anise and rose note into 1-butanol (next analysis). The best solution to these problems is to follow the intensity of the odor as the detector response builds up to the peak and then subsides. The post-peak effects may be reduced by a proper sniffing port design.

In still another approach, the effluent odorants are captured into a small tube or filter paper and evaluated separately, away from GLC. With this method, the intensity ratings are probably less dependable; and weak odors are difficult to evaluate since losses occur in such storage procedure.

Long GLC sniffing sessions are tiresome, may be boring, and yield poorer results than short sessions. Some consider that a single session—one GLC analysis—should be not much longer than 20 min.

Many of the problems which make odor evaluation difficult apply to physical-chemical identification methods too. An odorous compound may coelute with another or a nonodorous compound may coelute and reduce the odor intensity (Powers, 1970, 1978). Trailing or preceding peaks may cause contamination of the peak under study. These difficulties apply to mass spectrometry too, for example, and thus make identification difficult (Dravnieks and O'Donnell, 1971). Williams and Turknott (1977) postulated that a relatively high concentration of a compound with good solubility properties, but very little odor itself, may carry trace amounts of compounds which, under

normal circumstances, would elute at other retention times; hence odor descriptions characteristic of the product itself may be attached to the solubilizing compound though actually the compound is not an important character-imparting component.

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