Sniffer to determine the odor of gas chromatographic effluents

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Performance Test for Direct Reading Balance

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Although our analytical balances are maintained on a regular basis by a service group, it is still the laboratory's responsibility to be sure that the day-to-day measurements are correct. To this end, a fast, simple way to evaluate direct reading balances has been developed. The direct reading balance has obviated the necessity of handling weights and this improvement has unfortunately reduced the operator's awareness of the need for checking calibration. We have over 25 balances in our laboratory areas; and since we operate 15 remotely located service laboratories, our needs to assure uniform quality testing are especially critical. The use of National Bureau of Standards calibrated sets of weights is time consuming and the repetitive handling poses the danger of wear and damage to the calibrated reference weights.

The method devised serves to check the linearity of the balance, provides a direct intercomparison with all balances, and relates all balances to a Bureau of Standards reference. This is accomplished using the regular balance operators and results in a minimum of handling of our National Bureau of Standards reference weights. The entire operation is accomplished in a short period of time and makes no unusual demands on the laboratory staff. Three test objects were prepared by drilling out random amounts of metal from the bottoms of some old weights. These gave us a set of test objects weighing about 14, 29, and 56 g. They were carried to each balance station where an operator who normally used the balance weighed them individually and collectively. The data from each balance were recorded and no calculations made until all balances were checked. When the data for each balance had been compared with the mean, the balance closest to the mean was used to weigh the National Bureau of Standards certified weights and thus by handling these calibrated weights only once, the entire lot of balances was checked.

Table I. Data for 25 Balances

	Av from 25 balances, wt in g	Std dev, g	Rel std dev, %
A. First weight	13.9565	0.000 49	0.003 51
B. Second weight	29.4151	0.000 37	0.001 26
C. Third weight	56.4625	0.000 35	0.000 62
D. 3 Weights simultaneously[a]	99.8343	0.000 54	0.000 54
E. Sum of 3 weights[a]	99.8341		

[a] The difference between D and E provides an estimate of the linearity of the balance.

Table I summarizes the data for 25 balances.

A malfunctioning balance is quickly detected. It is a tribute to contemporary balance design that, out of the 25 balances checked, only 2 were suspect and required attention. Some of the balances have been in use for 15 years. In general, we are quite pleased with their overall performance.

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Sniffer to Determine the Odor of Gas Chromatographic Effluents

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Sniffing the effluent of a gas chromatograph (GC) is the most useful means of determining which components of a complex mixture of volatiles have odor. Although such a bioassay procedure is both qualitative and subjective, it is the only simple procedure for selecting those volatiles to isolate, identify, and evaluate for odor significance (*1*). Unfortunately, sniffing the effluent of a GC is both uncomfortable and inaccurate because of the irritating effects of the hot dry carrier gas and the lingering presence of strong odors in the environment of the GC output. Although the dryness of the carrier gas can be reduced by adding water to it upstream of the GC (*2*), this procedure limits the flexibility of the chromatography. This communication describes a simple inexpensive "sniffer" which improves the comfort and accuracy of sniffing GC effluents. The principle is to mix the GC effluent with a larger volume of rapidly moving humid air.

EXPERIMENTAL

The gas chromatography was done in a 4 m × 2 mm glass column packed with 5% SP 1000 on 100/120 mesh Chromasorb W installed in a Packard model 800 gas chromatograph. The column was operated isothermally at 100 °C and with a helium carrier gas flow of 20 ml/min. A nominal 50 to 1 splitter was installed in the flame ionization detector oven with the larger portion of the effluent going to the sniffer.

Figure 1 shows a diagram of a sniffer constructed from a brass laboratory filter pump (A). The air supply is deoderized by an in-line charcoal (Pittsburgh activated, 12–40 mesh) filter (H) and humidified by passing the air over the surface of distilled water in a half filled

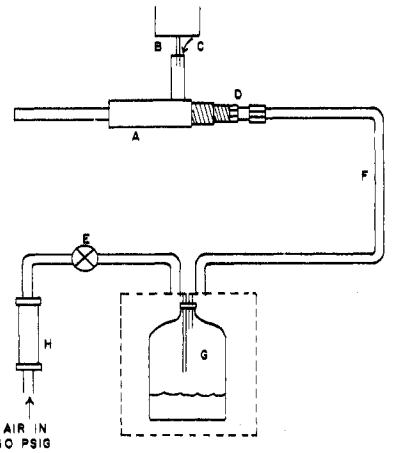

Figure 1. Diagram of the sniffer constructed from a laboratory filter pump

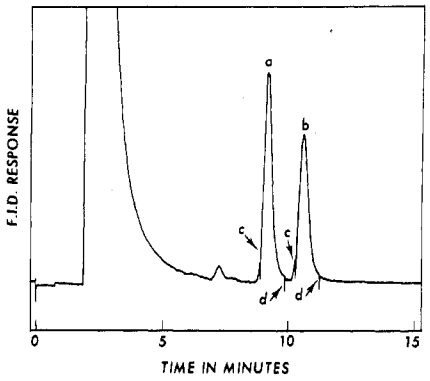

Figure 2. Gas chromatogram of *n*-hexylacetate (*a*) and 2-heptanol (*b*). The event marks (*c*) represent the first detection of odor while the marks (*d*) represent the disappearance of detectable odor

gallon jug (G). A piece of 1/16-in. stainless steel tubing from a nominal 50 to 1 splitter in the flame ionization detector oven extends 1 cm into the room and 2 mm into the vacuum port (B) of the filter pump. A 1/4-inch male Swagelok fitting was threaded into the filter pump at D to facilitate plumbing, and the ball check valve in the vacuum port was removed. Nylon tubing (1/4-in. o.d.) (F) was used to connect the components and a Whitey B-1RS4 (Whitey Co., Oakland, Calif.) valve was installed at E to control the air flow.

RESULTS AND DISCUSSION

Good results were achieved with a 12 l./min air flow through the filter pump and this caused 250 ml/min room air to flow into the vacuum port at C. Although this represented a 500-fold dilution of the gas chromatographic effluent, it did not appear to result in a severe reduction in odor intensity as compared to directly sniffing the effluent. Without the sniffer, the low velocity of the effluent carrier gas was subjected to room drafts which complicated the detection of peak odors. Figure 2 shows a gas chromatogram of *n*-hexylacetate (*a*) and 2-heptanol (*b*) which were simultaneously sniffed blindly. Each peak represented approximately 5 μg of material and the event marks (*c*) represent the first detection of odor and (*d*) the last detectable odor. This sniffer results in a marked improvement in the sensory resolution of gas chromatographic peaks compared with sniffing the effluent directly. Table I shows a comparison between the sensitivity of the sniffer and sniffing the GC output directly. Both methods yielded an apparent threshold of approximately 10 ng for both *n*-hexylacetate and 2-heptanol with less than a factor of 10 difference between them.

Table I. Comparison between the Detection of Odors Using the Sniffer and by Directly Sniffing the GC Output[a]

Amount injected, ng	Method	*n*-Hexylacetate	2-Heptanol
100	Sniffer	++	++
	Direct	++	++
10	Sniffer	0	0
	Direct	+	0
1	Sniffer	0	0
	Direct	0	0

[a] ++ indicates the compound was clearly detected; +, only faintly detected; and 0, no detectable odor.

When certain odorous compounds were present in high concentrations in the gas chromatographic effluent (e.g., phenyl ethyl alcohol in grape essence) or had an extremely low odor threshold (e.g., geosmin in beets), the sniffer became contaminated and it was necessary to clean the filter pump. Washing with water followed by isopropyl alcohol and then 1,1,2-dichloro-1,2,2,-difluoroethane (i.e., Freon 113) worked well. Contamination of the sniffer was also minimized by removing the sniffer from over the gas chromatographic effluent when strongly absorbant peaks emerged or by replacing it with a clean duplicate.

The sniffer worked equally well when attached to the effluent from a Llewellyn (*3*) type helium separator on a gas chromatograph–mass spectrometer interface.

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Modification of Graphite Furnace Power Supply to Allow Interruption of Analytical Cycle

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The graphite furnace is now well established as a useful atom generator for the analysis of cations by atomic absorption spectrometry (*1–3*). Some of the variety of experimental furnaces described in the literature (*4–17*) have been developed and marketed by manufacturers of atomic absorption spectrometers either as an integral part of the instrument or, more commonly, as an "add on" accessory. These usually operate by automatic sequential switching of the furnace power in accordance with a preset program, generally in three or more steps with increasing current loadings for specific time intervals. For adequate control of the analytical process, it is desirable that time, temperature, and rise rates be reproducible.

A common operating sequence is dry–ash–atomize wherein the sample (e.g., 5 μl of solution) is applied to the furnace, dried at low temperature, ashed at a higher temperature, and finally atomized. It seemed that the graphite furnace had considerable unexploited potential as a chemical reaction vessel. This was realized by placing switches to interrupt the analytical cycle at the end of the drying and ashing cycles. One of these switches (Sx) at the end of the drying cycle would allow for multiple sample application to the furnace as well as the application of reagents to the dried specimen, while a further switch (Sy) to interrupt at the end of the ash cycle would allow for the addition of a variety of reagents to render ash constituents more or less volatile, or to modify the influence of matrix cations. Thus, in operation the sample would be placed in the furnace, the selected cycle interrupt switch closed, and the cycle "start" button pressed. The analytical cycle would then proceed automatically to the interrupt stage