# Olfactometry Flow Rate Criteria A Multiple Laboratory Study Part II


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**ABSTRACT**

The "odor presentation flow rate" or the "olfactometer flow rate" is the rate of airflow that is presented to the odor panelists from an olfactometer. The airflow rate from an olfactometer to the odor panelists contains the diluted odor sample or the blank air sample during presentation.

In the U.S. the American Society for Testing and Materials (ASTM) Standard Practice E679-91, Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series of Limits, does not specify the "olfactometer flow rate". The 1999 draft "European Standard", prEN 13725, Determination of Odour Concentration by Dynamic Olfactometry, specifies an "odour presentation flow rate" of 20 liters per minute (lpm). The 1995 revision of the draft guidelines of the Air & Waste Management Association (AWMA) EE-6 Odor Committee, "Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry", recommends an "odor presentation flow rate" of between 5 and 10-lpm.

A "Multiple Laboratory Study - Part I" was presented in 1997 at the Water Environment Federation (WEF) Odor & VOC Specialty Conference (Houston) [McGinley, et al, 1997]. In addition to presenting the background information of the subject, the Part I paper presented the testing protocol and the decision criteria for accepting an "odor presentation flow rate" standard. Specialists, experts, and practitioners in the field of olfactometry have commented and contributed to the study since 1997.

Part II of the "multiple laboratory study" involved an analysis of the proposed "European Standard" and a rigorous effort to standardize laboratory practices of the participating odor laboratories, i.e. common panelist training. This paper presents some of the results of the flow rate study to date and offers recommendations for continuing interlaboratory testing.

**Keywords:** odor, odour, olfactometry, olfactometer, flow rate, odor panel, standard.

## INTRODUCTION

The olfactometry methods used by laboratories across the USA and by the international community generally follow the basics of the American Society for Testing and Materials (ASTM) Standard Practice E679-91, Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series of Limits. This standard, however, does not specify the "odor presentation flow rate" to the sensory panel members.

The "odor presentation flow rate" or the "olfactometer flow rate" is the rate of airflow that is presented to the odor panelists from an olfactometer. The airflow rate from an olfactometer to the odor panelists contains the diluted odor sample or the blank air sample during presentation.

In the U.S. ASTM Standard Practice E679-91, Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series of Limits, does not specify the "olfactometer flow rate". The 1999 draft "European Standard", prEN 13725, Determination of Odour Concentration by Dynamic Olfactometry, specifies an "odour presentation flow rate" of 20 liters per minute (lpm). The 1995 revision of the draft guidelines of the Air & Waste Management Association (AWMA) EE-6 Odor Committee, "Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry", recommends an "odor presentation flow rate" of between 5 and 10-lpm.

Previous published papers, specifically, "The Flow Rate of Test Odor Spouting into the Mask in the Dynamic Odor Test", Nishida, K. Japan, 1989, and "Effects of Sample Flow Rate in the Determination of Odor Thresholds", O'Brien, USA, 1995, have presented their laboratories' testing and results. These papers further recommended additional studies that may lead to a "standard" odor presentation flow rate for olfactometry.

A "Multiple Laboratory Study - Part I" was presented in 1997 at the WEF Odor & VOC Specialty Conference (Houston) [McGinley, et al, 1997]. In addition to presenting the background information of the subject, the Part I paper presented the testing protocol and the decision criteria for accepting an "odor presentation flow rate" standard. Specialists, experts, and practitioners in the field of olfactometry have commented and contributed to the study since 1997.

During the development of this study and through its implementation to date, the following laboratories participated directly with one of the study's olfactometers:

Australian Water Technologies, Sydney, NSW
Alberta Research Council in cooperation with the University of Alberta
Iowa State University, Ames, IW
Metropolitan Council Environmental Services, St.Paul, MN
Purdue University, West Lafayette, IN
St.Croix Sensory, Stillwater, MN
University of Minnesota, St.Paul, MN

Part II of this "multiple laboratory study" involved an analysis of the proposed "European Standard" and a rigorous effort to standardize laboratory practices of the participating odor laboratories, i.e. common panelist training. This paper presents some of the results of the flow rate study to date and offers ideas for continuing interlaboratory comparisons.

## BACKGROUND

ASTM Standard Practice E679-91, Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series Method of Limits, was originally published as E679-79 in 1979 and the current edition was approved on August 15, 1991, and published in October 1991, as ASTM E679-91. This standard presents two of the basic statistical concepts that have been incorporated into the olfactometry practices of today and are accepted internationally. These statistical concepts are known as the "forced-choice method" and the "ascending concentration series method". These methods are used for delivering a dilute odor sample to odor panelists for determining threshold. The device used to deliver the dilute odor sample to odor panelists is called an "olfactometer".

The process of evaluating an odor using an olfactometer and an odor panel involves connecting the odor sample to the olfactometer. The olfactometer takes the odor sample, dilutes the odor sample and presents the diluted odor to the odor panelist. The presentation method, as described by ASTM E679-91, is called the "3-alternative forced choice presentation" method. The method is commonly called the "triangular forced-choice" method. Each odor panelist performs the odor evaluation task by sniffing the diluted odor from the olfactometer. The panelist sniffs three sample presentations, one of which contains the odor while the other two are "blanks," and must select one of the three that is "different" from the other two. This statistical approach is called "triangular forced-choice". The panelist is required to declare to the panel leader, either verbally or by pushing a button, if the selection was a "guess", "detection" or "recognition", as defined by ASTM E679-91.

ASTM E679-91 defines the panelist judgement procedure (Paragraph 7.2). From the "triangular forced-choice" presentation, the panelist is required to indicate if the selected sample is **different** from the two other samples (*detection threshold*) or if the selected sample exhibits a **recognizable** odor (*recognition threshold*). If the panelist cannot discriminate between the three samples, the ASTM procedure requires a **guess** to be made.

The panelist is then presented with the next set of three sample choices, one of which again contains the diluted odor sample. However, this next set of three samples presents the odor at a higher concentration (e.g. two times higher). The panelist continues to additional higher levels of sample presentation following the "triangular forced-choice" procedure and the required designation of "guess", "detection" or "recognition". This statistical approach of increasing levels of sample presentation is called "ascending concentration series".

The odor evaluation procedure requires the first sample presentation to be presented to the odor panelist at a dilution that will be below the detection threshold (sub-threshold). Therefore, the first several presentations to the odor panelist require the panelist to select one of the three by guessing (or subconsciously guessing correctly).

The present convention of calculating "dilution factors" (dilution of the odor sample) for olfactometers is based on the ratio of "Total Flow" divided by "Sample Flow" [Dravnieks, 1980 and 1986], [ASTM E679-91], [AWMA EE-6 DRAFT Guidelines, 1995], and [prEN 13725, 1999].

$$\text{Dilution Factor} = \frac{\text{Dilution Volume} + \text{Odorous Sample Volume}}{\text{Odorous Sample Volume}} = \textbf{'Z'}$$

"The dilution factor, **'Z'**, is used in modest honor of H. Zwaardemaker, a Dutch scientist and early investigator in olfactometry. Alternative terminology in use (1991): Dilution-to-Threshold Ratio (D/T or D-T); Odor Unit (OU); Effective Dose (ED)" [ASTM E679-91, Appendix].

However, in laboratory olfactometry the "dilution factor" (**Z**) is not the value directly reported for the odor sample concentration. Laboratory olfactometry uses a group of assessors called "panelists"[ASTM E679-91]. Each panelist observes an odor sample in the ascending concentration series (increasing concentration). If a panelist does not detect an odor at **Z** = 1000 but does detect an odor at **Z** = 500, then the panelist's individual "detection threshold" is calculated as the geometric mean between 1000 and 500, which is 707. The statistical method is called the "best-estimate criterion" (ASTM E679-91, Paragraph 3.1.7).

$$(\log 1000 + \log 500)/2 = (3.0 + 2.7)/2 = 2.85$$

$$\{10^{2.85} = 707\}$$

Then the group threshold of all the panelists is calculated as an average from the logarithm values (i.e. 2.85,…) of each individual panelist.

The detection threshold [ASTM E679-91, Paragraph 3.1.5 and 7.2] and recognition threshold [ASTM E679-91, Paragraph 3.1.6 and 7.2] of an odor sample are derived using "dilution ratios" and the "best-estimate criteria" and, therefore, are dimensionless. However, the pseudo-dimensions of "Odor Units" (O.U.) or "Odor Units per Unit Volume" is commonly applied. For example: "Odor Units per Cubic Meter". The abbreviations for "detection threshold" (DT) and "recognition threshold" (RT) are sometimes used in order to clarify which 'Z' value is being reported by the odor laboratory.

## EUROPEAN AND USA PRACTICES

With the global increase of environmental regulations in the 1970's, European countries, Australia, and the United States began to develop odor regulations. These regulations created the need to standardize the methods of odor measurement. Some examples of these standards include: US - ASTM D-1391 (1978) and ASTM E679-91(1991), Germany - VDI 3881 (1980), France - AFNOR - X-43-101 (1986), and the Netherlands - NVN2820 (1996).

In 1990 the European Committee for Standardization (CEN: Comite Europeen de Normalisation) formed a technical committee (TC264) and in 1999 released a "for comment" draft European standard for odor concentration measurement [van Harreveld, 1999].

In 2000 the European Union (European Community) may follow the new standard, prEN 13725, "Air Quality - Determination of Odour Concentration by Dynamic Olfactometry" (prEN refers to a proposed European Normalization Standard - the "pr" will be removed if the draft is accepted after review and incorporation of comments). The following countries are bound by the CEN/CENELEC (European Committee for Standardization: "Comite Europeen de Normalisation") International Regulations to implement European Standards: Austria, Belgium, Denmark, Finland, France, Greece, Iceland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom. "prEN 13725" may be adopted or adapted in Australia, Canada, New Zealand, and much of the Pacific Rim.

In 1990 the Air & Waste Management Association (AWMA) EE-6 Odor Committee formed a subcommittee on the Standardization of Odor Measurement and developed a set of draft "Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry" [AWMA EE-6, Second Revision, November 1995].

**Presentation Statistics:** The CEN proposed standard (prEN 13725) and the EE-6 subcommittee guidelines agree that test odors should be presented to human assessors (panelists) utilizing dynamic dilution olfactometry following a "Forced Choice" ascending concentration series method. There are two minor differences between the CEN proposed standard and the EE-6 proposed guidelines regarding presentation statistics. The first difference is the EE-6 subcommittee stands behind the original ASTM E679-91 method, which requires two blank presentations per diluted odor presentation. This approach is called a triangular presentation. The CEN proposed standard agrees with the triangular approach, but also allows only one blank per diluted odor presentation (binary presentation). The second difference is in the dilution increase between dilution presentation levels (ascending concentration series). While the EE-6 subcommittee recommends a constant increase of a factor of two, the CEN proposed standard is slightly more lenient, specifying an increase factor between 1.4 and 2.4.

The CEN prEN 13725 also allows the Yes/No Presentation Method and the Forced-Choice Probability Method. The Yes/No method is similar to audiometry protocol where dilute odors and blanks are randomly presented and the assessor identifies when they detect the odor. The Forced-Choice Method is a complex Forced-Choice Probability Method derived from the French standard AFNOR NF 43-101.

**Olfactometer Design:** All standards developed to date have specified "odor free" dilution air [ASTM E679-91]. Further, the most recent drafts released by the EE-6 subcommittee and the CEN have specified the olfactometer must be constructed of components made of glass, stainless steel, or polytetrafluoroethylene (PTFE). These proposed standards and guidelines also come relatively close to agreeing on an acceptable range of dilutions. The CEN (prEN 13725) has specified a minimum upper limit of $2^{14}$ and a maximum lower limit of $2^{7}$. The EE-6 subcommittee recommends a minimum upper limit of 10,000 ($\sim 2^{13}$) and a maximum lower limit of 10 ($\sim 2^{3}$).

**Olfactometer Performance:** The CEN proposed standards and EE-6 guidelines discuss olfactometer performance criteria and instrument calibration. The EE-6 guidelines only outline these topics generally, leaving specifics to the laboratory. The CEN proposed standard goes one step further by creating the groundwork for a laboratory quality assurance plan. The CEN proposed standard specifies laboratory accuracy and repeatability performance criteria (including instructions on how to test the criteria) for the olfactometer following the international standard ISO5725 *Accuracy (Trueness and Precision) of Measurement Methods and Results Parts 1-4*.

**Assessor (Panelist) Qualifications:** The CEN proposed standard lists strict criteria for qualifying an assessor for olfactometry panels. The EE-6 guidelines state that individuals "representing normal sensitivity are selected to serve as panelists" [ASTM EE-6, 1995]. The EE-6 guidelines do not go any further to define "normal sensitivity." Since odor sensitivity in the general population is normally distributed, it is implied that a laboratory should aim for assessors who lie near the center of the bell curve (i.e. within 1 to 2 standard deviations of the mean).

The proposed European standard takes a different approach altogether. The prEN 13725 gives very strict criteria for assessor selection based on accuracy and repeatability to a standard, reference odorant (n-butanol). The standard states that "assessors with a specific sensitivity to the reference odorant n-butanol are selected to be panel members." Each potential assessor must be tested to n-butanol on the olfactometer a minimum of 10 times. The individual's average threshold measurement of n-butanol must be in the range of 20 - 80 ppb. Further, the antilog of the standard deviation must be less than 2.3. Once the assessor is accepted as a panelist, they must be continually checked to this n-butanol reference with a rolling average of 20 measurements compared to the above criteria.

Therefore, the European proposed standard is different from the EE-6 guidelines in two respects. First, the criterion for selecting an assessor is based on the assessor's accurate and repeatable to one specific odorant (n-butanol). ). It is assumed that each assessor's accuracy and repeatability will be the same for all odors measured in the lab (livestock, wastewater, compost, MSW landfill, etc.). Second, the CEN criterion does not necessarily select assessors who are "normal" in the population. The prEN 13725 assessor selection criterion explicitly abandoned the notion that the assessor panel is representative of the general population [van Herreveld, 1999]. It is not known if these assessors actually lie above, below, or on the average in the general population.

**Odor Presentation Flow Rate:** The proposed European standard (prEN 13725) specifies that the olfactometer must operate at a presentation flow rate of 20-lpm. The draft guideline of the EE-6 subcommittee recommends an odor presentation flow rate of between 5 and 10 lpm.

**Presentation Face Velocity:** The presentation face velocity at the olfactometer-to-assessor interface (mask or port) also continues to be a discrepancy between the European and EE-6 drafts. The EE-6 subcommittee recommends a face velocity of between 0.02 - 0.05 meters per second, m/s (6-10cm mask @ 8-lpm). The CEN prEN13725 draft specifies a face velocity greater than 0.2 m/s and recommends that it be set less than 0.5 m/s (3-5cm mask @ 20-lpm).

## FLOW RATE VARIABLES

The "odor presentation flow rate" of the olfactometer has an influence on the detectability of an odor, as stated in the "Introduction" of ASTM E679-91 and reported by other researchers [Dravnieks, 1980], [Laing, 1986], [Nishida, 1989], and [O'Brien, 1995].

All the researchers generally agree that the presentation interface (sniffing port, cone, or mask) must minimize the opportunity for room air to be inhaled (sniffed) by the panelist during the panelist's "observation" (sniffing) of the diluted odor from the olfactometer. The presentation interface must further minimize forcing of the air (odor) in to the panelist's nostrils.

During previous studies by researchers [Dravnieks, 1980] [O'Brien, 1995], other concerns were expressed regarding olfactometer presentation flow rates. For example, as the olfactometer flow rate is increased the required odor sample size is increased and the required odor laboratory exhaust ventilation is increased. However, these concerns where based on the assumption that the odor sample would be continuously consumed by the olfactometer and continuously released by the olfactometer into the odor laboratory.

The researchers in this "multiple laboratory study" revisited these concerns and the underlying assumptions. The testing protocol first used a presentation device (mask) common to the medical community's practices in inhalation therapy. The selected mask, known as an "adult high concentration, non-rebreathing" mask, is used for supplying a high oxygen concentration airflow to the user without rebreathing exhaled air. The soft-vinyl mask, fitted with low resistance check valves to prevent rebreathing, allows excess air to leave the mask. This mask design achieves the two primary objectives: preventing ambient air inhalation and preventing the forcing of air into the panelist's nostrils. However, the commercially available masks of this design where made of vinyl and retained a characteristic plastic odor. Treatments to completely eliminate the residual vinyl odor from the masks were unsatisfactory.

A nasal mask was developed for this "multiple laboratory study" from basic mask designs used in dentistry anesthesiology. The nasal mask was designed with slots at the periphery to allow excess sample air to release away from the panelist's face. The mask dimensions are 2.3-inches (5.8cm) from nose bridge to upper lip and 2.2-inches (5.6cm) across the nose. The nasal mask designed for the study provided for a comfortable olfactometer-to-panelist interface and was odor-free because of a "Teflon-like" finish. A nasal mask was provided for each panelist in order to maintain appropriate hygiene.

The "multiple laboratory study" used an olfactometer developed expressly for testing different "odor presentation flow rates". The concerns for odor sample size and odor laboratory exhaust ventilation needs were addressed with a flow control scheme on the odor sample flow path. The olfactometer for the study delivered odor only "on demand", during the sniffing time. Further, the olfactometer utilized only one "sniffing port", fitted with the panelist nasal mask, for presenting the three alternative forced-choices ("triangular forced-choice"). A rotary knob was devised to give the panelists the freedom to select and observe any one of the three choices (odor, blank, or blank: each randomly located on the rotary knob). Together with the flow control scheme, the rotary knob provided an olfactometer-to-panelist interface with out physical bias, without excess odor sample consumption, and without excess odor in the odor laboratory space.

## FLOW RATE COMPARISON

The flow rate comparison of foremost interest to the participating odor laboratories of this study is the 10-LPM flow rate compared to the 20-LPM flow rate. Figure 1, Detection Threshold verses Odor Presentation Flow Rate, presents a summary of 69 data points from measuring one of the standard odorants in the study (hydrogen sulfide at a nominal concentration of 1part per million, 1-PPM). All of the odor panel evaluations where conducted with 8 assessors (odor panelists).

Before showing the specific calculations, it is necessary to discuss the importance of the logarithm base 10 transformation that is used during odor testing and associated statistical calculations. The transformation is used to make the non-linear dilution ratio scale (i.e. 125, 250, 500, 1000, 2000, 4000, etc.; x 2 series) a linear scale in logarithm base 10 (2.097, 2.398, 2.699, 3.000, 3.301, 3.602, etc.; + 0.301 series). More, specifically, the transformation is performed in order to stabilize (make uniform) the variance. With the uniform variance, the linear transformed data will show symmetry around the group average (panel average result in log base 10). However, the data will be asymmetrical around the reported "Odor Unit" value of detection threshold (and recognition threshold). All statistical calculations which are based on a normal distribution must, therefore, be conducted with the transformed values, in the case of odor testing, the logarithm base 10 values [McGinley, M.A., 2000].

The panel average standard deviation for all of the testing in the 10-LPM and 20-LPM flow rates did not exceed 0.3 (logarithm base 10). The average detection thresholds for the 10-LPM and 20-LPM flow rates were 300 (log 300 = 2.48) and 950 (log 950 = 2.98), respectively. The null hypothesis that 950 (20-LPM) **is statistically the same** as 300 (10-LPM) is tested with a student **t** test. The test statistic (t) is computed from:

$$\mathbf{t} = (x - u) / s / \mathrm{SQRT}(n-1) = (2.98 - 2.48) / 0.30 / \mathrm{SQRT}(8-1) = 0.63$$

where:
- x = 2.98 (log 950 for the 20-LPM flow rate)
- u = 2.48 (log 300 for th 10-LPM flow rate)
- s = 0.30 standard deviation in logarithm base 10
- n = 8 the number of panelists (degrees of freedom)

The **t** value (0.63) is compared with the **t** value for a two tailed test at 95% confidence (alpha = 0.05) which is +/- 2.365. Since **0.63** ($p > 0.2$) is not larger than **2.365** ($p > 0.05$), the null hypothesis cannot be rejected and in this case 950 (20-LPM flow rate) is statistically **NOT** significantly different from 350 (10-LPM flow rate).

This analysis means that there is a 5% chance that the two detection thresholds (10-LPM and 20-LPM flow rates) are different, but we treat them as the same. (i.e. we did not reject the "null hypothesis" that they were the same detection thresholds).

The statistical significance of an "odor presentation flow rate" must be based on a criteria universally accepted. Statistical significance is one approach to comparing the results of two "odor presentation flow rates". The researchers recognize the importance to consider "clinical significance" between "odor presentation flow rates".

If the 20-LPM olfactometer flow rate consistently produces odor detection thresholds greater than the 10-LPM olfactometer flow rate the differences may appear to be significant (i.e. statistically), but they may have little or no clinical importance.

For example, odor evaluations that are conducted at a given flow rate may have been conducted to rank eight odor emission sources, compare the inlet and outlet of an odor control system, or compare the results of a trial with previous trials. The decisions made in the ranking or performance comparisons would not be affected if the odor evaluations were conducted at a higher odor presentation flow rate, i.e. 20-LPM verses 10-LPM. In this example of odor evaluation data used for decision making, the statistical significance of a higher flow rate may not be relevant to the clinical use of the data.

The researchers of this study revisited the assumption that laboratory olfactometry, as practiced using ASTM E679-91, was intended to produce a detection threshold that represented odor perception in the ambient air [AWMA EE-6, 1995]. The fundamental problem with the assumption is the distinct difference between the laboratory assessor's observation practice (identifying the odor sample from two blanks) compared to the citizen's observation of the ambient air when surrounded by the "odor".

## CONCLUSIONS

The basic tenets of the EE-6 draft guidelines agree with the European prEN 13725 draft, except for the recommended "air (odor) presentation flow rate" to the panelist (assessors).

The EE-6 draft guidelines recommend a maximum flow rate of 10-liters per minute and the prEN 13725 recommends a flow rate of 20-liters per minute.

The EE-6 draft guidelines are based on the premise that the recommended flow rates will produce repeatable measurements most characteristic of odor perception in the ambient air [AWMA EE-6, 1995]. The European draft standard, prEN 13725, is based on the premise that the assessors do not necessarily represent the general population [van Herreveld, 1999].

To date the "multiple laboratory study" finds no statistical nor clinical difference between data produced from an olfactometer operating with an "odor presentation flow rate" of 10-LPM or 20-LPM.

The participants of this "multiple laboratory study" and their colleagues have taken a pragmatic approach. Each participating odor laboratory has on-going research that is funded through revenues and grants from a variety of sources. They all share in the recommendation to continue interlaboratory studies.

The olfactometer that each participating odor laboratory is now using can operate with an "odor presentation flow rate" of 10-lpm or 20-lpm. Therefore, the laboratories are without bias or prejudices regarding the "European verses the US" draft standards and guidelines. Nevertheless, all of the participating odor laboratories in this study have elected to operate their olfactometers at an "odor presentation flow rate" of 20 liters per minute (LPM), following the tenets of the draft European standard, prEN13725.

The European community's adoption of a standard "odour" measurement method (prEN 17325), including a standard 20-lpm presentation flow rate, predicts a trend in the international community. The proposed "European Standard", prEN 13725 may become the de facto international standard for odor/odour testing. The Air & Waste Management Association, EE-6 Committee's work on developing guidelines for standard odor measurements will certainly consider the implications of the European prEN13725.

A standardized olfactometer flow rate may lead to concise standards of performance for odor control systems and the promulgation of odor regulations with limits for odorous emissions. A move to standardize in the United States may lead to national "voluntary" standards of performance by the odor control industry and, further, lead to local odor ordinances.

The stakes are high to agree on a standard "odor presentation flow rate" for olfactometers.
Stakeholders include:

Citizens,
Local authorities,
State regulators,
Researchers,
Industry representatives,
Facility owners and operators,
Odor control system manufacturers,
Consulting engineers,
Environmental planners, and
Environmental laboratories.

The odor laboratories participating in this "multiple laboratory study" plan to continue interlaboratory comparative testing with different flow rates, various standard odorants, and various "real-life" odor samples.

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**Figure 1 Detection Threshold vs Odor Presentation Flow Rate**