Standardized Odor Measurement Practices for Air Quality Testing

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ABSTRACT

Ambient air holds a mixture of odorous chemicals from everyday activities of industrial and commercial enterprises. From state to state and in communities across the United States odor issues are addressed by a variety of odor ordinances, regulations and policies. When air quality is compromised with odors, effective study, investigation, and enforcement requires that odors be measured using standardized methods that are dependable, reproducible, objective, and quantitative.

Point, area and volume emission sources can be sampled and tested for odor parameters using standard practices published by the American Society of Testing and Materials (ASTM E679 and E544) and by the European Union (European Normalization Standard, EN 13725). The most common odor parameter determined during odor testing is "odor concentration" (odor strength). This determination is made using an instrument called an "olfactometer." In the United States the standard followed for olfactometry is ASTM Standard of Practice E679-91, "Determination of Odor and Taste Threshold by a Forced-Choice Ascending Concentration Series Method of Limits." In 2002 this ASTM standard is under review to incorporate latest recommendations from the AWMA EE-6 "Odor Committee" and elements from prEN 13725 – "Air Quality – Determination of Odour Concentration by Dynamic Olfactometry". The 18 countries in the European Union are bound by the CEN/CENELEC International Regulations to implement this European Standard. The new European standard has been adopted in Australia, New Zealand, and much of the Pacific Rim. Therefore, "EN 13725" will become the de facto International Standard for odor/odour testing.

Odor can also be measured and quantified directly in the ambient air using one of two standard practices. ASTM E544-99, "Standard Practice for Referencing Suprathreshold Odor Intensity", is used to measure and quantify ambient odor intensity using an "Odor Intensity Referencing Scale (OIRS). An air pollution inspector, plant operator, or community odor monitor can observe the ambient odor and compare it to the OIRS (a series of concentrations of a reference odorant, i.e. n-butanol).

A second standard method for measuring and quantifying odor in the ambient air utilizes a field olfactometer. The standardized method (U.S. Public Health Service Project Grant A-58-541) uses a portable odor detecting and measuring device known as a field olfactometer (e.g. scentometer). The field olfactometer dynamically dilutes the ambient air with carbon-filtered air in distinct dilution ratios known as "Dilution to Threshold" dilution factors (D/T's), i.e. 2, 4, 7, 15, etc.

INTRODUCTION

Community odors remain at the top of air pollution complaints to regulators and government bodies around the U.S. and internationally. Ambient air holds a mixture of odorous chemicals from everyday activities of industrial and commercial enterprises. From state to state and in communities across the United States odor issues are addressed by a variety of odor ordinances, regulations and policies. When odors compromise air quality effective investigation and enforcement requires that odors be measured using standardized methods that are dependable, reproducible, objective, and quantitative.

The quantification of odors is typically required for the following purposes:

- 1. Monitoring for compliance assurance.
- 2. Determination of compliance for permit renewal.
- 3. Determination of baseline status for expansion planning.
- 4. Determination of specific odor sources during complaint investigation.
- 5. Monitoring operations for management performance evaluation.
- 6. Comparison of operating practices when evaluating alternatives.
- 7. Monitoring specific events or episodes for defensible, credible evidence.
- 8. Comparison of odor mitigation measures during tests and trials.
- 9. Determination of an odor control system's performance for warranty testing.
- 10. Verification of estimated odor impacts from dispersion modeling.

Odor is measurable using objective, quantitative, standardized scientific methods in odortesting laboratories. Point, area and volume emission sources can be sampled and tested for odor parameters such as odor concentration, intensity, persistence, and descriptors.

Odor can also be measured and quantified directly in the ambient air using one of two standard practices by trained inspectors. The first method uses a standard "odor intensity referencing scale" (OIRS) made up of the standard odorant, n-butanol, to quantify odor intensity. The second method utilizes a field olfactometer, which dynamically dilutes the ambient air with carbon-filtered air in distinct dilution ratios known as "Dilution-to-Threshold" dilution factors (D/T's).

POINT, AREA & VOLUME ODOR SOURCE MEASUREMENT

An odorous air sample can be collected from a point emission source and from surface and volume emission sources. "Whole-air" samples for laboratory odor testing are typically collected in 10-liter Tedlar gas sample bags for transport to the odor-testing laboratory.

In the early years of odor testing a syringe dilution technique measured odors in the laboratory from samples collected at the odor source¹. That standard was ASTM D1391, "Standard Test Method for Measurement of Odor in Atmospheres (dilution method)". The syringe dilution technique used a series of dilutions known as "dilution ratios". In

1979, ASTM E679-79, "Standard Practice for Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series Method of Limits" was published². The current edition of this odor-testing standard was approved on August 15, 1991, and published in October 1991, as ASTM E679-91. This standard defines a method of dynamically diluting the odor sample with an instrument called an olfactometer.

When practicing ASTM E679-91 during an odor test, the odor panelist (assessor) sniffs a dilute sample of the odor as it is discharged from the olfactometer as one of three sample presentations (one presentation with the dilute odor and two with odor free air). The assessor sniffs all three of the presentations and must select the one of the three that is different from the other two, even if they must guess. This statistical approach is called "triangular forced-choice." The assessor declares to the test administrator if the selection is a "guess", a "detection" (the selection is different from the other two), or a "recognition" (the selection smells like something) as defined by ASTM E679-91.

The assessor is then presented with the next set of three presentation choices, one of which 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 assessor continues to additional levels of higher concentration (lower dilution) presentations following the "triangular forced-choice" procedure and the required designation of "guess", "detect", or "recognition". This statistical approach of increasing levels of sample presentation is called "ascending concentration series."

European Odor Testing Standard

In the 1980's countries in Europe began developing standards of olfactometry. Some of these standards developed and published include:

France AFNOR X-43-101, Method of the Measurement of the Odor of a Gaseous Effluent, Bureau de Normalisation, Paris, France (drafted in 1981 & revised in 1986)

Germany VDI 3881, Parts 1-4, Richtlinien, Olfactometry, Odour Threshold Determination, Fundamentals. Verein Deutsche Ingenieure Verlag, Dusseldorf, Germany (drafted in 1980 & revised in 1989)

Netherlands NVN 2820, Provisional Standard: Air Quality. Sensory Odour Measurement using an Olfactometer. Netherlands Normalization Institute, The Netherlands (drafted in 1987 & issued in 1995)

Various inter-laboratory studies as well as collaborative projects involving multiple odor testing laboratories in the 80's showed that laboratory results still differed significantly even with these standards in practice³.

The development of a draft odor-testing standard in the Netherlands led to an Inter-Laboratory Comparison study organized in 1989⁴. N-butanol and hydrogen sulfide were used as standard odorants for the study. Through 1990 to 1992 the results of this Dutch Inter-Laboratory study led to the development of strict assessor performance criteria. During the first year, the inter-laboratory repeatability was in the range of factors from 3 to 20. An analysis of the data from this first year showed the majority of variability was between assessors. Individual assessors were repeatable within a factor 3 to 5. The researchers found that the only way to meet agreed upon repeatability criteria was to control the instrument sensor, the human assessors, by selecting assessors who were all similar in sensitivity.

Standards were set for assessor performance to the standard odorant n-butanol. Only assessors who met predetermined repeatability and accuracy criteria were allowed to continue as assessors. Over the next two years, these new criteria were implemented within each of the labs involved in the study.

The work of this inter-laboratory study led to the final Dutch standard released in 1995 and set the foundation for the development of the new European odor testing standard.

A working group was formed within the Committee European de Normalisation (CEN) Technical Committee 264 – "Air Quality" to develop a unified olfactometry standard. This working group saw a need to help industry and regulators develop a consistent basis for monitoring and testing odors, and, thus help determine the potential for odor nuisance. This was to be accomplished by developing a method that:

- 1. Improved consistency within each laboratory (repeatability);
- 2. Achieved comparable results among laboratories (reproducibility); and
- 3. Connect the results to a traceable reference material, e.g. n-butanol (accuracy)

In order to achieve these goals, the committee focused on the following issues: sampling procedures, sample containers, olfactometer construction and operation, the olfactometer and assessor interface, the odor testing room, methods of data processing, and assessor selection, training, and performance

The first complete draft of the European olfactometry standard was released in 1995. Then in the spring and summer of 1996, nineteen laboratories from five countries participated in an Inter-laboratory Comparison of Olfactometry (IOC) study. The purpose of this study was to validate the requirements, methods, and procedures outlined in the draft. The conclusions of this study were⁵:

- 1. All quality requirements and performance criteria were attainable for all testing methods studied (Forced-Choice and Yes/No); and
- 2. Those following the standard for the longest period of time performed the best with regards to accuracy and repeatability.

The CEN olfactometry standard was released to the public at the end of 1999 through the standard organizations of each participating country. The standard was released as

Proposed CEN standard #13725 (prEN 13725) "Air Quality – Determination of odour concentration by dynamic olfactometry." ⁶ A public comment period closed at the end of January 2000. Comments were submitted to each country's standardization body separately. These comments were reviewed in early 2000. The working group met in 2000 to review all comments and issue a final revision of the standard. The final revision was sent to the CEN organization in 2001 for official acknowledgement and approval.

The final CEN standard approval will obligate all countries of the European Union to adopt the standard and withdraw any conflicting or redundant national standards. These countries include: Austria, Belgium, Denmark, Finland, France, Greece, Germany, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

The final standard will be published in three official languages: English, French, and German. The standards are distributed through the individual country standardization boards. For example, an English language copy can be obtained from the British Standards Institute (BSI).

The new European standard has been adopted in Australia, New Zealand, and much of the Pacific Rim. Therefore, "EN 13725" will become the de facto International Standard for odor/odour testing.

AWMA Guidelines and ASTM E679 Standard

The proliferation of large scale animal confinement facilities (i.e. feedlots) throughout the United States, as well as the general trend of urban sprawl moving people closer to odorous industrial facilities, wastewater treatment facilities, and agricultural facilities has created a resurgence in funding for odor related research. In 1995, universities and research institutions in the United States and Canada began to develop and expand odor testing laboratories for conducting odor research in agricultural facilities, municipal solid waste facilities, and other odorous industries. Odor research laboratories founded or expanded in the past five years include:

- Agriculture Canada Charlottetown, Prince Edward Island
- Alberta Research Council/University of Alberta Edmonton, Alberta
- Duke University Durham, North Carolina
- Iowa State University Ames, Iowa
- Los Angeles County Sanitation Districts Los Angeles, California
- Metropolitan Council, Environmental Services St. Paul, Minnesota
- Purdue University West Lafayette, Indiana
- University of Illinois Urbana, Illinois
- University of Manitoba Winnipeg, Manitoba
- University of Minnesota St. Paul, Minnesota
- West Texas A&M University Amarillo, Texas

In 1995 the AWMA EE-6 Odor Committee formed a subcommittee on the "Standardization of Odor Measurement". The EE-6 Subcommittee developed "Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry"⁷ and submitted them for review on August 23, 2002, to the ASTM International E18 Sensory Committee.

In October, 2002, the EE-18 Sensory Committee of ASTM International initiated a comprehensive review of ASTM E679-91 and elements of the AWMA guidelines and the EN13725 Standard.

EN13725 standardizes the following elements of odor testing (olfactometry) and exceeds the existing ASTM standard:

- 1. Ascending concentration series sample presentation (same as ASTM E679-91)
- 2. Binary or triangular forced-choice selection (ASTM requires triangular)
- 3. Dilution ratio of "2" for the presentation series (ASTM allows 3)
- 4. "Odor free" dilution and blank air (same as ASTM)
- 5. Olfactometer materials of Teflon, stainless steel, or glass (same as ASTM)
- 6. Presentation flow rate of 20-liters per minute (ASTM does not specify)
- 7. ISO 5725 Accuracy of Measurement and Results (exceeds ASTM)
- 8. Requires periodic olfactometer calibration (exceeds ASTM)
- 9. Requires assessor certification (exceeds ASTM)
- 10. Uses the traceable reference odorant n-butanol (exceeds ASTM)

The objective of the ASTM review is to achieve repeatability, reproducibility, and accuracy in odor testing that was achieve by EN13725:

- > Repeatability within a laboratory
- Reproducibility between laboratories
- Traceability to n-butanol standard

AMBIENT ODOR MEASUREMENTS

Odor can also be measured and quantified directly in the ambient air using one of two standard practices by trained inspectors. The first method uses a standard "odor intensity referencing scale" (OIRS) made up of the standard odorant, n-butanol, to quantify odor intensity. The second method utilizes a field olfactometer, which dynamically dilutes the ambient air with carbon-filtered air in distinct dilution ratios known as "Dilution-to-Threshold" dilution factors (D/T's).

Ambient Odor Intensity

Odor intensity of the ambient air can be measured objectively using an "Odor Intensity Referencing Scale" (OIRS) [ASTM E54475, 88, "Standard Practice for Suprathreshold Intensity Measurement"]⁸. Odor intensity referencing compares the odor in the ambient

air to the odor intensity of a series of concentrations of a reference odorant. A common reference odorant is n-butanol. Sec-butanol is an alternative to n-butanol for a standard referencing odorant. The air pollution inspector, plant operator, or community odor monitor observes the odor in the ambient air and compares it to the OIRS. The person making the observation must use a carbon-filtering mask to "refresh" the olfactory sense between observations (sniffing)⁹. Without the use of a carbon-filtering mask, the observer's olfactory sense would become adapted to the surrounding ambient air or become fatigued from any odor in the surrounding air. The adaptation of an observer's olfactory sense is a common phenomenon when attempting to evaluate ambient odors, i.e. a wastewater treatment plant operator monitoring treatment plant odors "off-site".

ASTM E544-75, 88, "Standard Practice for Referencing Suprathreshold Odor Intensity", presents two methods for referencing the intensity of ambient odors: Procedure A - Dynamic-Scale Method and Procedure B - Static-Scale Method. Field inspectors commonly use the Static-Scale Method and it has become incorporated as a standard of practice by a number of odor laboratories, because of its low cost of set-up compared to a dynamic-scale olfactometer device (Procedure A).

Using the OIRS, the intensity of the observed ambient air is expressed in "parts per million" (PPM) of n-butanol (or sec-butanol). A larger value of butanol means a stronger odor. An important aspect of using a butanol intensity referencing scale is knowing that a variety of scales are available. Common butanol static-scales include:

- ✓ 12-point static-scale starting at 10-ppm butanol with a geometric progression of two;
- ✓ 10-point static-scale starting at 12-ppm butanol with a geometric progression of two;
- ✓ 5-point static-scale starting at 25-ppm butanol with a geometric progression of three;

The OIRS serves as a standard practice to quantify the odor intensity of the ambient air objectively. To allow comparison of results from different data sources and to maintain a reproducible method, the equivalent butanol concentration is reported or the number on the OIRS is reported with the scale range and starting point.

An example 5-point OIRS with a geometric progression of three is:

<u>Reference Leve</u> l	<u>n-Butanol PPM in Air</u>
0	0
1	25
2	75
3	225
4	675
5	2025

Field air pollution inspectors (field odor inspectors), using a standard odor intensity referencing scale (OIRS), can provide measured, dependable, and repeatable observations of ambient odor intensity.

Ambient Odor Concentration

In 1958, 1959, and 1960 the U.S. Public Health Service sponsored the development of an instrument and procedure for field (ambient) odor measurement (olfactometry) through Project Grants A-58-541; A-59-541; and A-60-541.¹⁰ The original field olfactometer was manufactured by Barnebey-Cheney Company and subsequently manufactured by Barnebey Sutcliffe Corporation.

The U.S. Public Health Service method defined the <u>dilution factor</u> as "Dilution to Threshold", D/T. The method of producing "Dilution to Threshold" (D/T) with the field olfactometer consists of mixing two "volumes" of carbon-filtered air with specific "volumes" of odorous ambient air. The method of calculating "Dilution to Threshold" (D/T) for the field olfactometer is:

D/T = Volume of Carbon Filtered Air / Volume of Odorous Air

The field olfactometer instrument, the "Dilution to Threshold" (D/T) terminology, and the method of calculating the "D/T" are referenced in a number of existing agencies' odor regulations and permits. Therefore, a field olfactometer instrument, in the hands of trained air pollution investigators or monitors, is a realistic and proven method for quantifying ambient odors.

Common "Dilution-to-Threshold" (D/T) ratios used to set ambient odor guidelines are: D/T's of 2, 4, and 7. Field olfactometers typically have additional D/T's (dilution ratios) such as 15, 30, 60 and higher dilution ratios.

Field olfactometry with a calibrated field olfactometer is a cost effective means to quantify odor strength. Facility operators, community inspectors, and neighborhood citizens can confidently monitor odor strength at specific locations around a facility's property line and within the community when using a calibrated field olfactometer.

The following "protocols" are presented in brief exemplary form as an application guide for field olfactometry:

- (1) On-Site Monitoring Operators have the unique ability to monitor odors throughout the day with field olfactometry. Operator monitoring can include odor observations of arriving materials, outdoor process activities, and fugitive air emissions. Monitoring with a field olfactometer on-site may include odor observations at predetermined locations, i.e. open doorways, driveways, storage areas, and fence lines.
- (2) **Random Monitoring** A frequently used method for ambient odor monitoring is the "random inspection" approach. Random monitoring leads to a compilation of data that can be correlated with meteorological information and on-site activities. Managers and regulators alike find that random odor monitoring with a field olfactometer is a cost effective protocol.

- (3) **Scheduled Monitoring** Well-planned scheduled monitoring can be limited to a daily "walk-about" or "drive around", or structured with several visits to predetermined monitoring locations. Data from a field olfactometer can be used to correlate the many parameters that influence odor episodes, including meteorological conditions and on-site operating activities.
- (4) Intensive Odor Survey An in-depth evaluation of on-site odor generation and off-site odor impact may be needed for permit renewal or facility expansion. Extensive data collection with a field olfactometer will identify which sources or operations cause odor and which ones do not cause odor off-site. All potential odor sources and operations could be ranked and their relative contributions determined. Short-term trials or tests of odor mitigation measures, e.g. odor counteractants, would also require an intensive period of data collection using a field olfactometer.
- (5) **Citizen Monitoring** The implementation of citizen odor monitoring with field olfactometers can be part of an interactive community outreach program. The primary function of citizen odor monitoring is to collect information, through accurate record keeping, which represents real conditions in the community. Citizens recruited and trained to measure odors using field olfactometers would also report odor descriptors. Citizen odor monitoring will assist in determining prevalent times and prevalent weather conditions of odor episodes. Citizen odor monitoring with field olfactometers will also help in understanding the odor strength at which an odor first becomes a nuisance.
- (6) Complaint Response The use of "Odor Compliant Hot Lines" is a common method used by facilities and communities to respond to odor episodes. A complaint response plan, with designated "on-call" responders, creates opportunities for verifying odor episodes, tracking odor sources, and quantifying odor strength with a field olfactometer.
- (7) Plume Profiling Standard and specialized air dispersion modeling predicts the transport and dilution of odors by the wind. A protocol, known as plume profiling, supplements and "calibrates" air dispersion modeling. Several inspectors with field olfactometers, spaced cross wind and down wind from an odor source, would measure and record the odor strength as "D/T" values. The odor plume profile would then be documented and overlaid on the local terrain map. Therefore, the air dispersion modeling and the local topography would be integrated with actual odor measurements from a field olfactometer.

These "protocols" are presented in brief exemplary form as guide and are <u>not</u> mutually exclusive, often being combines into a comprehensive odor management program.

CONCLUSIONS

Odor is measurable using objective, quantitative, standardized scientific methods in odortesting laboratories and out in the ambient air by trained inspectors. Point, area and volume emission sources can be sampled and tested for odor parameters such as odor concentration, intensity, persistence, and descriptors. Odor can also be measured and quantified directly in the ambient air using one of two standard practices by trained inspectors. The first method uses a standard "odor intensity referencing scale" (OIRS) made up of the standard odorant, n-butanol, to quantify odor intensity. The second method utilizes a calibrated field olfactometer, which dynamically dilutes the ambient air with carbon-filtered air in distinct dilution ratios known as "Dilution-to-Threshold" dilution factors (D/T's).

The stakeholders for standardized odor measurement are:

- ➢ Regulators
- > Researchers
- > Industry
- Manufacturers
- ➢ Consultants
- ➤ Citizens

With the knowledge of fundamental odor testing an objective approach can be taken to addressing community nuisance odors and problematic odorous emissions. The subjectivity of nuisance odors can be replaced with objective, scientific methods of odor measurement with laboratory olfactometry and field olfactometry.

ACKNOWLEDGMENTS

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dilution-to-threshold dilution ratio odor concentration odor intensity odor olfactometer olfactometry