Odor Measurement Review

Office of Odor, Noise and Vibration
Environmental Management Bureau
Ministry of the Environment, Government of Japan

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Ⅰ Profile

Dr. Yoshiharu Iwasaki

Born on December 19, 1944

Most recently acquired academic qualification:

Tokyo Institute of Technology, (M.Sc.)

Career:

1970 Tokyo Metropolitan Research Institute for Environmental Protection, Researcher

1986 Tokyo Metropolitan Research Institute for Environmental Protection, Chief Researcher

1995 Tokyo Metropolitan Research Institute for Environmental Protection, Director

2002 Tokyo Metropolitan Research Institute for Environmental Protection, Chief Director

Major Committee Membership, etc.:

Japan Society for Atmospheric Environment, Standing Director
Tokyo Metropolitan Institute of Technology, Guest Professor
Tokyo University of Science, Part-time Lecturer

Other Distinctions (Awards, etc.):

Winner of the Tokyo Metropolitan Governor Prize (1984,1997)
Winner of the Japan Society for Atmospheric Environment Prize

Mr. Kenji Kamigawara

Born on July 17, 1960

Most recently acquired academic qualification:

Bachelor, Faculty of Law, Tohoku University, 1984

Career:

Entered to the Environment Agency, Government of Japan, 1984
Seconded to the Global Environment Division, the Ministry of Foreign Affaires,
Seconded to the Japanese Mission in Geneva to the International Organizations,
Appointed to the Secretary to the Minister of the Environment,
Appointed to the Chief of Noise and Odor Division(present)

Major Committee Membership:

Lecturer(part-time), Graduate School on Human-Environmental Study, Kyoto University, FY2002
Lecturer(part-time), Faculty of Law, Waseda University, FY2003

Major Publications:

The Basel Convention and its Conference of the Parties-Beyond Confrontation and Confusion, Social System Study, No5, Faculty of Integrated Human Study, Kyoto University, 2002

Mr. Anton Philip van Harreveld

Most recently acquired academic qualification:

MSc

Career:

Ton van Harreveld is founder and chairman of the OdourNet group. His operational activities include consultancy, international marketing, R&D supervision and product development.

OdourNet specialises in all aspects of environmental odour annoyance and conducts many hundreds of odour related surveys each year. Several thousands of odour samples are collected and analysed in two accredited OdourNet laboratories to provide supporting data. OdourNet employs a staff of about 30 in four European countries.

OdourNet develops and markets instruments and associated software for odour sampling and measurement. Olfaktomat-n olfactometers are installed in laboratories worldwide.

The roots of OdourNet roots were established in 1980, when van Harreveld established Project Research Amsterdam BV, environmental consultants specialising in characterisation and abatement of odours. The company has since developed from a one man start-up into a leading firm of specialist consultants on odour issues, serving industry and regulators alike.

In the process, van Harreveld became closely associated with efforts to develop standardised methods for odour impact assessment. From 1988 onwards he has been an active member of a working group to standardise olfactometry in Holland, under the auspices of the Netherlands Normalisation Institute (NNI). In 1993 he was invited to act as convenor for the working group that is to draft a European CEN standard on determination of odours using dynamic olfactometry CEN/TC264/WG2 ‘Odours’. In 1994 he joined a NNI working group on sampling of odours.

The involvement in standardisation was matched by R&D within the company, that has developed a strong position as supplier of odour sampling equipment and olfactometers with related software.

In 1994 Ton van Harreveld founded a sister company in England, Project Research Environmental Consultants Ltd., renamed in April 2000 to OdourNet UK Ltd. A joint laboratory was founded in Australia at that same time. These were the first steps towards establishing the OdourNet group of companies, that now operate four offices: PRA OdourNet BV (Amsterdam, Holland), OdourNet UK Ltd. (Bradford-on-Avon, UK), Project Research Gent NV (Gent, Belgium) and OdourNet SL (Barcelona, Spain).

As a senior consultant he recently completed major scientific review reports to support policy development for livestock odours by the Irish EPA and general odour guidelines for the Environment Agency (UK). Previously he headed a team to prepare a feasibility study for developing a method for odour annoyance potential measurement for the Dutch government.

Ton van Harreveld took an MSc in environmental engineering in 1979 at the Agricultural University of Wageningen, The Netherlands, with Air Quality and Environmental Health as main subjects. In preparation for his thesis he worked abroad at the Ministry of the Environment in Mexico City and at the Environmental Protection Agency in the United States.

Ton and his family now live in Barcelona, Spain.

Major Committee Memberships:

- Technical Committee on Olfactometry of the Netherlands Calibration Organisation NKO.
- CEN standardisation working group TC264/WG2 ‘Odours’ (convenor)
- Netherlands Normalisation Institute working groups (Olfactometry, Sampling of Odours)
- Netherlands ‘Platform Odour’, a section of the Netherlands Association of Environmental Scientists, where industry, consultants, and regulators discuss

implications and development of odour policies, with a focus on differentiating criteria based on type of odour (vice-chairman)

- US Air and Waste Management Association committee EE-6 on odours (member)
- Source Testing Association (UK, member)
- Verein Deutsche Ingenieure (member)
- IWA International Water Association (member)
- Member of the scientific committee of the IWA/WEF conference on odours in Singapore, September 2003
- Chairman of the planned European conference on Odour Management, organised in Köln, Germany, by Verein Deutsche Ingenieure (VDI), November 2004

Major Publications:

- EN13725: 2003, *Odour concentration measurement by dynamic olfactometry*, April 2003 (contributed as convenor). www.cenorm.be
- Van Harreveld, A. Ph., (2003) *Odour Concentration Decay and Stability in Gas Sampling Bags*, J. Air & Waste Manage. Assoc. vol. 53 (1), 51-61, ISSN 1047-3289, January 2003
- *Assessment of community response to odorous emissions.*, R&D Technical Report P4-095, Environment Agency, WRc Document Distribution Centre, United Kingdom, 2002. www.environment-agency.gov.uk
- Van Doorn, Reind, Ruijten, Marc, Van Harreveld, A.Ph., *Guidance for the Application of Odor in the Derivation of AEGL-1 4*, version 02/06/2001, Public Health Service of Rotterdam, the Netherlands.
- Van Harreveld, AP, Jones, N, Stoaling, M, (2001) *Odour Impacts and Odour Emission Control Measures for Intensive Agriculture. Part A: Odour annoyance assessment and criteria for intensive livestock production in Ireland.*, Environmental Monitoring, R&D Sub–programme of the Environmental Services Operational Programme 1994-1999, Environmental Protection Agency, Report 14, ISBN 1-84095-075-7, 2001. www.epa.ie
- Bongers, M.E, Vossen, FHJ, van Harreveld, AP, *Geurhinderonderzoek stallen intensieve veehouderij (translation: Survey of odour nuisance in the vicinity of intensive livestock housing)*, in Dutch, Ministry of Planning, Public Housing and the Environment, Distribution centre, document number 22317, March 2001.
- Van Harreveld, A.P., *From odorant formation to odour nuisance: new definitions*

for discussing a complex process, Water Science & Technology Vol 44 No 9 pp 9-15, ISSN: 0273-1223, 2001

- *Odour control in wastewater treatment - a technical reference document*, UK Water Industry Research Ltd, London, Technical reference document 01/WW13/01/3,2000 (major contributor) www.ukwir.org
- Van Harreveld, APh, Schakel, A., Valk, CJ, Vreeburg, S, *Feasibility study for the development of a standardised method for assessment of odour nuisance and odour annoyance potential*, (in Dutch) Project PD 207 Action programme Standardisation and Validation of Environmental Assessment Methods, Project Research Amsterdam BV, December 1999.
- Van Harreveld, A. Ph., (1998) *A review of 20 years of standardization of odour concentration measurement by dynamic olfactometery in Europe*, J. Air & Waste Manage. Assoc. 49, 705-715, ISSN 1047-3289, June 1998
- Van Harreveld, A.Ph., *Interlaboratory Comparison of Olfactometry. Validation of draft CEN standard 'Odour concentration measurement using dynamic olfactometry*, CEN TC264/WG2 'Olfactometry' document N220, OdourNet UK Ltd, 1996.
- Van Harreveld, A. Ph., Heeres, P., (1995) *Quality control and optimisation of dynamic olfactometry using n-butanol as a standard reference odorant*, STAUB -Reinhaltung der Luft 55 pp 45-50, Spinger Verlag.
- Van Harreveld, A.P., (1991) *Introduction and Related Practical Aspects of Odour Regulations in the Netherlands*:, in: Derenzo and Gnyp, TR18, Recent Developments and Current Practices in Odour Regulations, Controls and Technology, AWMA, ISBN 0-923204-0, 1991

Presentations:

- Van Harreveld, A. Ph, *Dynamic olfactometry - fundamentals, developments and wastewater applications*, keynote paper, in: Proceedings of the 6th IWA conference on off-flavours in the aquatic environment, International Water Association, Barcelona, October 7-10, 2002
- Van Harreveld, A. Ph, Chemicals as odour predictors: what causes the odour deficit, in: Proceedings of the CIWEM conference 'Odours – what a nuisance', Hatfield, UK, CIWEM, November 7th, 2002.
- Van Harreveld, A. Ph, *Scent - Smell - Stink - Stech: How to draw the nuisance line*, keynote paper, in: Proceedings of Enviro 2002, April 7-12, Melbourne
- Van Harreveld, A. P., *From odorant formation to nuisance: new definitions for*

discussing a complex process, in: Proceedings of the 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques,, University of New South Wales, March 2001.

- Bongers, M.E., Van Harreveld, A.P, Jones, N,, *Recent pig odour policy reviews in the Netherlands and in Ireland*, in: Proceedings of the 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques,, University of New South Wales, March 2001.
- Van Harreveld, A.P.,, *Current status of introduction and implementation of CEN standard EN13725 Odour Concentration Measurement by dynamic olfactometry*, In: Proceedings of the 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques,, University of New South Wales, March 2001.
- Van Harreveld, A. Ph., *Odour measurement as a tool to support industry in the art of being good neighbours and protect long-term interest in the process,* Presented at the Canadian Paper and Pulp Association 1995 Environment Conference, Halifax, Nova Scotia, October 1995
- Van Harreveld, A. Ph., (1995) *Main features of the final draft European Standard 'Measurement of odour concentration using dynamic olfactometry'*, in: Proceedings of the Specialty Conference: Odours: Indoor and Environmental Air, organised by Air and Waste Management Association, Sept.1995
- Schulz T, van Harreveld Aph, Kaye R, *Olfactometry Art to Science*, Centre for Waste Water Treatment, 12th CASANZ Conference, Perth, October 1994.
- Van Harreveld, A. Ph., Klarenbeek, J.V., *On the regulations, measurement and abatement of odours emanating from livestock housing in the Netherlands, in: new knowledge in Livestock Odor*, Proceedings of the International Livestock Odor Conference 1995, pp.16-21, Iowa, USA, Iowa State University, College of Agriculture, Ames
- Van Harreveld, A. Ph., Schulz T, Seminar - *Environmental odours- detection, minimisation and control.*, the Munro centre for civil and environmental engineering. The University of New South Wales, July 1994
- Harreveld, A. van, Clarkson, C. *Techniques and Methodology in Odour Measurement*, paper presented at the First Irish Conference on the Atmospheric Environment, Cork, February 1992

Mr. Thomas Dean Mahin

Most recently acquired academic qualification:

BS Environmental Engineering, University of Texas at Austin

Career:

Thomas Mahin is a section chief with the Massachusetts Department of Environmental Protection (MDEP) in the USA. He is also chair of the MDEP's odor policy workgroup.

He has given presentations at technical conferences in numerous cities in the USA and Canada and was a keynote speaker at the 1st IWA Conference on Odors and VOCs at Sydney, Australia in 2001.

Major Committee Memberships:

He was the chair of the Water Environment Federation's (WEF) Air Quality Committee from 1998 - 2001. He is currently chair of a WEF task force that is rewriting and combining WEF's Odors and Toxic Air Emission's manuals of practice. He was co-chair of the WEF Odors and Toxic Emissions Specialty Conferences in 2000 and 2002.

Other Distinctions (Awards, etc.):

Water Environment Federation Service Award
MDEP Outstanding Performance Award

Major Publications:

- "Comparison of Different Approaches Used to Regulate Odors Around the World", Proceedings of the 1st IWA Conference on Odor and VOCs: Measurement, Regulation and Control Techniques, Sydney, Australia, March 25-28, 2001 & published in *Water Science and Technology,* Vol 44 No 9 pp 87-102.
- "Odor Measurement and Public Perception" (Mahin, T.) included in WEF 2001 publication: *Odor and Corrosion Prediction and Control in Collection Systems and Wastewater Treatment Plants*.
- "When is a Smell a Nuisance? An Overview of Different Approaches Taken Around the World in Setting Odor-Control Regulations" (Mahin, McGinley and Pope). May, 2000 issue of *Water Environment & Technology.*
- "Elements of Successful Odor Laws" (McGinley, Mahin and Pope). Proceedings of the Odor and VOCs Emissions 2000 WEF Specialty Conference, Cincinnati, Ohio April 16-19, 2000.
- "Overview of Different Approaches Used in Odor Regulations and Policies" (Mahin and Pope), Proceedings of the Water Environment Federation (WEF) 1999 Annual Conference (WEFTEC99) New Orleans, La.
- "Odor Dispersion: Models and Methods" (Pope, Mahin and Diosey). Proceedings of the WEF 1999 Annual Conference (WEFTEC99) New Orleans, La., USA.
- "Using Odor Intensity Ratings During Complaint Investigations to Determine Whether Nuisance Conditions Exist" (Mahin, T.). Proceedings of the WEF 1998 Annual Conference (WEFTEC98) Orlando, Fl., USA.
- "Odor Modeling Using D/T Levels" (Mahin, T.). February, 1998 issue of *Water Environment & Technology.*
- Using Dispersion Modeling of Dilutions to Threshold (D/T) Odor Levels to Meet Regulatory Requirements for Composting Facilities"(Mahin, T.). Proceedings of the Air & Waste Management Association's 90th Annual Meeting & Exhibition, June 8-13, 1997, Toronto, Ontario, Canada.
- "Massachusetts Odor Policy and Design and Dispersion Modeling Technical Guidance" (Mahin, T.). Proceedings of WEF Specialty Conference Control of Odors and VOCs Emissions, April 20-23, 1997, Houston, Texas.
- "Comparing a New Odor Policy to Permit Conditions at a Massachusetts Composting Facility" (Mahin, T.). January, 1997 issue of *Biocycle*.
- "Odor Policy for Composting Facilities" (Mahin, T.). December, 1995 issue of *Biocycle.*

Prof. Dr. Sang Jin Park

Born on January 03, 1957 at Yesan in Korea

Most recently acquired academic qualification:

1983	Graduated from Korea University (MS)
1993	Completed the doctor's course from the Korea University(Ph.D)

Career:

1995-present	Professor, Dept of Civil & Environmental Eng. Dean of Student Affairs, Woosong University
1985-1995	Research fellow, Dept. of Environmental Eng., Korea Institute of Construction Technology under Ministry of Construction
1992-1993	Visiting Scholar, Resource Utilization Lab., Tokyo Institute of Technology in Japan
1983-1985	Section chief, Dept. of Water & Wastewater, Korea Engineering Consulting Cooperation
1979-1981	Professor, Dept. of Civil Eng., Korean 3rd Military Academy

Major Committee Memberships:

President, Korean Society of Odor Research & Engineering
Chairman, Korean Research Group of Odor & VOC
Director, Korean Society of Environmental Engineers
Representatives, Korean Society for Atmospheric Environment

Major Publications:

- Sangjin Park, et. al., "Control of Odor and VOCs Emissions Using Biofiltration :

A Case Study of Styrene", Proceeding of 3rd IWA International Conference on Odour and VOCs (2003 Abstract accepted)

- Sangjin Park, et. al., "Removal of Odor Emitted from Composting Facilities Using Ceramic Biofilter", Journal of Water Science and Technology, Vol. 44, No. 9, pp. 301-308 (2001)
- Park S. J. et. al., “Evaluation of Porous Ceramic as Microbial Carrier of Biofilter to Remove Toluene Vapor", Jour. of Environmental Technology, Vol. 22, pp. 47-56 (2001)
- Sangjin Park, et al., “Evaluation of Porous Ceramic As Microbial Carrier of Biofilter to Remove Tolune Vapor”, Proceeding of the International Conference on Water, Environment, Ecology, Socio-ecomomics and Health Engineering, pp. 258-268 (1999.10)

Mr. Sung Bong Yang

Born on May 27, 1953

Most recently acquired academic qualification:

1988 Ph. D. in Chemistry, Department of Chemistry, Korea Advanced Institute of Science and Technology, Korea

Career:

1981 Full-time Assistant at Department of Industrial Chemistry, Ulsan Institute of Technology

1985 Assistant Professor, Department of Chemistry, University of Ulsan

1995 Professor, School of Chemistry and Life Science, University of Ulsan

Major Committee Memberships:

Korean Society of Odor Research and Engineering
Korean Society for Atmospheric Environment
Korean Chemical Society

Major Publications:

- Investigation of Emission Inventory and Strategy of Reduction of Odor at Siwa-Banwol Industrial Zone, National Institute of Environmental Research, 2003, Korea
- Management of Odor and Emission Source in Korea, Ministry of Environment, 2001, Korea
- Measurement of Odorous Compounds at Industrial Area in Korea, National Institute of Environmental Research, 2002, Korea

Mr. Robert William Sneath

Most recently acquired academic qualification:

Post Graduate Diploma Power Engineering 1971

Career:

1990-2003	Head of the Silsoe Research Institute Odour laboratory and a member of the CEN/TC264/wg2/Odours working group that formulated the European Standard EN13725 Air Quality-Determination of odour concentration by dynamic olfactometry. Project Leader in Environment Research Group; Research into Greenhouse Gas emissions from agricultural livestock production. Research into the application and development of electronic nose technology for detection of food contaminants.
1981-1990	Silsoe Research Institute, Senior Research Scientist, Research on odour and emission control from aerobic treatment of wastes: recently Project Leader:- Methane and Nitrous Oxide Emissions from livestock buildings and manure, olfactometry and.)
1977-1981	NIAE (National Institute for Agricultural Engineering), Silsoe Research into the application of preservatives to hay
1974-1977	NIAE Research into aerobic treatment to improve handling and disposal of livestock wastes to reduce water pollution.

Major Committee Memberships:

CEN/TC264/WG 2 Odours
BSi (British Standards Institution) EH/2/1 Stationary Source Emissions

Major Publications:

- Sneath. R.W. & Persaud. K.C. (2003) Chapter 15 Correlating Electronic Nose and Sensory Panel Data, pp 377-397 in "Handbook of Machine Olfaction". Eds Pearce, Schiffman, Nagle, Gardner. Wiley-VCH Verlag GmbH & Co. KgaA, Boschstrase 12, 69469 Weinheim, Germany.
- SNEATH, R.W. European odour Concentration Measurements by Dilution Olfactometry. Invited presentation at ECRO2002, Erlangen, Nurenburg, Germany. 23-27 July 2002
- Robertson, Hoxey, Demmers, Welch, Sneath, Stacey, Fothergill, Filmer, Fisher; (2002) Commercial-scale studies of the effect of broiler-protein intake on aerial pollutant emissions. Biosystems Engineering 82 (2) 217-225
- SNEATH, R. W. (2001). Chapter 7 `Olfactometry and the CEN standard, prEN 13725`, pp 130-154 in "Odours in Wastewater Treatment: Measurement, Modelling and Control". Eds. R Stuetz and F. Frechen. IWA.Publishing, Alliance House, 12 Caxton Street, London SW1H 0QS, UK.
- O. PAHL, A.G. WILLIAMS, R.W. SNEATH (2002) Reduction Of Ammonia and Odour Emissions From Pig Slurry Under Slats Using Oil And Foam Layers. Environmental Technology Vol. 23. Pp395-403
- BROWN, L., SYED, B., JARVIS, S.C., SNEATH, R.W., PHILLIPS, V.R., GOULDING, K.W.T., & LI, C. (2002) Development and application of a mechanistic model to estimate emission of nitrous oxide from UK agriculture. Atmospheric Environment 36 (2002) 917-928
- PHILLIPS, V.R.; LEE, D.S.; SCHOLTENS, R.; GARLAND, J.A.; SNEATH, R.W. (2001) A review of methods for measuring emission rates of ammonia from livestock buildings and slurry or manure stores. Part 2 Assessment of techniques for measuring flux rates, concentrations and air flows. Journal of Agricultural Engineering Research, Vol 78 1, 1-14H
- MARK R. THEOBALD, C. MILFORD, K.J. HARGREAVES, L.J. SHEPPARD, E. NEMITZ, Y.S. TANG, V.R. PHILLIPS, R. SNEATH, L. McCARTNEY, F.J. HARVEY, I.D. LEITH, J.N. CAPE, D. FOWLER, M.A. SUTTON , (2001) Potential For Ammonia Recapture by Farm Woodlands: Design And Application of A New Experimental Facility, In Optimizing Nitrogen Management in Food

and Energy Production and Environmental Protection: Proceedings of the 2nd International Nitrogen Conference and Policy. The Scientific WorldI 1(S2), 791-801

- SNEATH, R. W., PHILLIPS, V. R., DEMMERS, T. G. M., BURGESS, L.R., SHORT, J. L., WELCH, S. K. (1997) Long term measurements of greenhouse gas emissions from UK livestock buildings. 5th International Livestock Environment Symposium, Minneapolis, USA, 29-31 May 1997,
- EVANS, P.; PERSAUD, K.C.; McNEISH, A.S.; SNEATH. R.W.; HOBSON. N.; MAGAN, N. (2000(Evaluation of a radial basis function neural network for the determination of wheat quality from electronic nose data. Sensors and Actuators ‘B’, 69 348-359

Mr. Hiroyuki Ueno

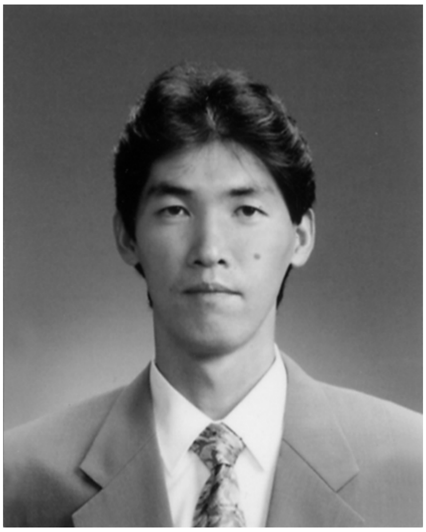
Born on March 29, 1967

Most recently acquired academic qualification:

1991	Tokyo Institute of Technology Interdisciplinary Graduate School of Science and Engineering

Career:

1991-present	Tokyo Metropolitan Government (Research Institute for Environmental Protection, Municipal Waste Management Association of Santama-area, Division of Water Conservation, etc.)



Dr. Takaya Higuchi

Born on March 14, 1969

Most recently acquired academic qualification:

Dr. Eng. in Global Environment Engineering, 1996, Kyoto University

Career:

1999-present	Research Associate Department of Civil Engineering, Yamaguchi University
1997-1999	Research Associate Department of Construction Systems Engineering, Anan College of Technology

Major Publications:

- Takaya Higuchi, Junji Masuda & Akiko Hayano: Establishment of quality control framework for olfactometry in Japan, Proceedings of the WEF's International Specialty Conference, Odors and Toxic Air Emissions 2002, 2002. (on CD-ROM)
- Takaya Higuchi, Ryuji Otomaru, Masahiro Ohsako & Masao Ukita: Laboratory studies on volatilization and diffusion characteristics of odorous compounds for estimating odor emission from wastewater, *Proceedings of 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques*, pp. 339-346, 2001.
- Takaya Higuchi, Masahiro Ohsako & Kumiko Shigeoka: Improvement of triangle odor bag method and triangle odor flask method as sensory measurement methods for odor emitted from wastewater, *Proceedings of the WEF's*

International Specialty Conference, Odors and VOC Emissions 2000, 2000. (on CD-ROM)

- Takaya Higuchi & Konosuke Nishida: Analysis of data measured by the triangular odor bag method, in McGinley, C. M. & Swanson, J. R. (eds.), *ODORS: Indoor and Environmental Air*, Air & Waste Management Association, Pittsburgh, pp. 181-192, 1995.
- Takaya Higuchi, Konosuke Nishida, Takashi Higuchi, Nobumasa Takeuchi & Ryujiro Tsuchihashi, A study on the temporal attribute in sense of smell for estimating Offensive odor, *Journal of Japan Society of Air Pollution*, Vol. 29, No. 6, pp. 313-322, 1994. (in Japanese with English abstract)

Mr. Katsunori Saiki

Born on June 17, 1952

Most recently acquired academic qualification:

1977 Graduate School, Showa University Department of Pharmacy

Career:

1977 Sato Pharmaceutical Co. Ltd
1985 Daiichi Yakuhin Sangyo Co. Ltd

Major Publications:

Olfaction and Taste XI (Springer-Verlag,1993)

Mr. John Kaiyun Jiang

Most recently acquired academic qualification:

Senior Research Fellow/Senior Lecturer

Career:

2003 -	Principal and Director, EnvironOdour Australia Pty Ltd
1995 - 2002	Senior research fellow/senior Lecturer, School of Civil and Environmental Engineering, the University of New South Wales
1990 - 1995	Research assistant, School of Civil and Environmental Engineering, the University of New South Wales

Major Committee Memberships:

- Deputy-Chairman, Organizing Committee, 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques, the University of New South Wales, Sydney, Australia, 25- 28 March 2001
- Organizer, National Workshop on Odour Measurement Standardisation, the University of New South Wales, Sydney, Australia, August 20 - 22 1997.
- Working group member on odour measurement standardization chaired by Standard Australia

Other Distinctions (Awards, etc.):

Principal investigator of three major national projects for poultry, pig and dairy industries to develop odour impact assessment procedures and criteria.

Inventor for a fourth generation dynamic olfactometer (DynaScent) and DynaSampler.

Major Publications:

- Jiang, J. Editor, Water Science & Technology, Vol 44 No 9 pp 17-25 © IWA Publishing 2001
- What are the areas for further research? Water Science & Technology, Vol 44 No 9 pp 17-25 © IWA Publishing 2001
- Jiang, J. Editor, Proceedings of 1st IWA International Conference on Odour and VOCs: measurement, regulation and control techniques, The university of NSW, Sydney, Australia, 25-28 March 2001
- Jiang J. and Kaye R., Chapter 5 Sampling techniques for odour measurement in Odours in Wastewater Treatment: Measurement, Modelling and Control, published by IWA publishing 2001, ISBN: 1 900222 46 9
- Jiang, J. and Kaye, R. Developing odor impact assessment procedures, WEFTEC 2000 Conference Proceedings; October 14-18, 2000, Anaheim, CA
- Jiang, J. Development of odor impact criteria using odor intensity measurement and community survey, Second International Conference on Air Pollution from Agricultural Operations, October 9-11, 2000 Marriott Conference Center Des Moines, Iowa, USA
- Jiang, J. and Kaye, R. A comparison of odor dispersion modelling results using emission rate data from wind tunnel and flux hood, Water Environmental Federation International Specialty Conference: Odors and VOC Emissions 2000, Hyatt Regency Cincinnati, Ohio, USA 16-19 April 2000 (in CD Rom)
- Jiang, J. Odour Impact Assessment for Broiler Farms, Eviron 2000 - odour conference, Sydney, (published in CD Rom).
- Ralph, K. and Jiang, J. Development of odour impact criteria for sewage treatment plants using odour complaint history, Proceeding of Control and Prevention of Odours in the Water Industry, 23-24 September 1999, London, UK, pp65.

Mr. Yoshio Nagata

Born on August 11, 1947

Most recently acquired academic qualification:
Graduate School, Osaka Prefecture University

Career:

1971	Japan Environmental Sanitation Center
Present	Assistant Division Head, Environmental Analysis Division, Environmental Science Department

Major Committee Memberships:
Odor Research and Engineering Association of Japan

Other Distinctions (Awards, etc.)
Scientific Award from the Odor Research and Engineering Association of Japan

Dr. Lawrence Choon Chiaw Koe

Most recently acquired academic qualification:

1983 PhD, University of Queensland, Australia

Career:

2002 Associate Professor at the Nanyang Technological University, Singapore
Director, Environmental Engineering Research Centre, NTU
Director and Chairman of the Board of Directors, Aromatrix Pte Ltd

Major Committee Memberships:

Chairman, Air & Water Management Association, Singapore Chapter
Chairman, IES Technical Committee on Environmental Engineering
Chairman, SAC – Singlas Committee on Environmental Engineering

Other Distinctions (Awards, etc.):

- Recipient of the Michael Flynn Award for presenting the best paper at the 1983 Australian Water and Wastewater Association's Biennal Convention in Sydney.
- Innovation Award 2001 by Singapore's Enterprise Challenge Unit for the trial application of bioscrubber technology to remove sewage odour at Kranji Sewage Treatment Works.
- Enterprise Challenge Award 2003 for most successful innovation

Major Publications:

- Koe, LCC and Y. Wu, Removal of Toluene in waste gases by a biotrickling filter. *Journal of Institution of Engineers*, 41, 3 (2001): 30-35 (Singapore)
- Koe, LCC, A Arellano and J Mc Gregor, Investigating the haze transport from 1997 biomass burning in Southeast Asia: Its impact upon Singapore. *Atmospheric Environment*, 35, no. 15 (2001): 2723-2734. (United States)
- Koe, LCC and F Yang, Evaluation of a pilot-scale bioscrubber for the removal of hydrogen sulphide. *Water and Environment Management*, 14, 6 (Dec 2000): 432-435. (United Kingdom)
- Koe, LCC, J McGregor and A Arellano, Southeast Asia Regional Haze Modelling. *Journal of Institution of Engineers*, 40, 5 (2000): 50-57 (Singapore).
- Koe, LCC and F Yang, A bioscrubber for Hydrogen Sulphide removal. *Water Science and Technology*, 41, 6 (2000): 141-145. (United Kingdom)
- Koe, LCC, Witherspoon, J, Tan, N C, Regan, M, Daigger, G and T Card, Buried treasure. *Water Environment and Technology*, July (2000): 30-35 (USA)

Dr. Masahiro Osako

Born on August 29, 1963

Most recently acquired academic qualification:

Doctor of Engineering, Graduate School, Kyoto University

Career:

1991-1995	Researcher, National Institute of Public Health
1995-2000	Senior Researcher, National Institute of Public Health
2000-	Senior Researcher, National Institute for Environmental Studies

Major Committee Membership, etc.:

Guest Associate Professor, Graduate School, Tokyo Institute of Technology
Guest Researcher, National Institute of Science and Technology Policy

Other Distinctions (Awards, etc.):

1991	Scientific Award from JOREA
2000	Award for Encouragement from the Japan Society of Waste Management Experts

Mr. Jun-ichi Kita

Born on May 09, 1957

Most recently acquired academic qualification:

Graduated from Kyoto University with B.A. at Chemical Engineering in 1981

Career:

Entered Shimadzu Corporation in 1981, studied in IR sensors, X-ray sensors, High temperature superconductor sensors and odor sensors in central research lab. at Shimadzu and then belonged to Analytical Instrument Div. from 1996.

Major Publications:

Aroma science series 21 [6] analysis and evaluation of odor molecules

Prof. Franz Bernd Frechen

Most recently acquired academic qualification:

Ph.D. from RWTH (Technical University) Aachen, Germany, in 1988

Career:

Studied Civil Engineering at the RWTH (Technical University) Aachen, finished 1980.
Received a Ph.D. from RWTH (Technical University) Aachen, Germany, in 1988 concerning Odour emissions of wastewater facilities (magna cum laude)

1988-1993	Employee and shareholder of a mid-sized engineering and consultant company (55 employees)
1994-1996	Foundation of own consultant company for odour and wastewater technology
since 1996	Full Professor and Head of the Dept. of Sanitary and Environmental Engineering, University of Kassel

Major Committee Memberships:

International Water Association:
Chairman of the Specialist Group on Odours and Volatile Emissions
And member of other Specialist Groups
German Association for Water, wastewater and waste:
Member, Main Committee on “Domestic wastewater Treatment”
Chairman, Committee on “Emissions of wastwater facilities”
Member, Committee on “Odour emissions from sewerage systems”
And others

Major Publications (only English, only related to odour):

- Book Editor (together with Richard Stuetz, Cranfiled University): Odours in Wastewater Treatment - Measurement, Modelling and Control, 2001, IWA Publishing, London
- Environmental Pollution by Odorous Emissions of Domestic and Toxic Waste Landfills - Basics, Present Situation and Reduction by Waste Gas Treatment Techniques
- Aktuelle Entwicklungen in der Umweltschutztechnik, Hrsg.: Umweltbundesamt Wien und Internationale Gesellschaft für Umweltschutz, Essen 1989, Westarp Wissenschaften, Essen
- Odour Emissions and Controls, Sanitary Landfilling: Process, Technology and Environmental Impact, Hrsg. Christensen, T. H.; Cossu, R.; Stegmann, R., London 1989, S. 425ff, Academic Press Limited, London
- Odor Emissions of large WWTP's: Source Strength Measurment, Atmospheric Dispersion Calculation, Immission Prognosis, Countermeasures - Case Studies - Water Science and Technology, 1992, Vol 25, Nr. 4-5, S. 375ff
- Effect of aerobic pre-treatment of waste on odour emissions, IV. International Landfill Symposium, Cagliari, 11.-15.10.1993
- Odour Emissions of Wastewater Treatment Plants - recent German Experiences, Wat. Sci. Tech., Vol. 30, No. 4, S. 35 - 46, 1994
- A new model for estimation of odour emissions from landfill and composting facilities , 5th International Landfill Symposium, Cagliari, Sardinia, Proceedings Volume I, S. 815, 1995, CISA
- Odour Emissions and Control, Landfilling of Waste: Biogas, Hrsg. Christensen, T. H.; Cossu, R.; Stegmann, R.
- Effect of Aerobic Treatment on Odour Emissions - Results from Large-Scale Technical Plant Operation, Sixth Intern. Landfill Symposium, Cagliari, Vol. 1, S. 555ff., 1997
- Odour Emission Capacity of Wastewaters - Standardization of Measurement Method and Application, Wat. Sci. Tech., Vol. 38, No. 3, S. 61 - 68, 1998, together with Köster, W.
- Anaerobic treatment of biowaste - odour emissions and control technologies at a full size treatment plant, 7th International Landfill Symposium, Cagliari, Sardinia, Proceedings Volume V, S. 421, 1999, CISA
- Determination of and measures against odour emissions from sewerage systems, 11th European Sewage and Refuse Symposium, Liquid wastes

section, S. 181, GFA, 1999

- Odour measurement and odour policy in Germany, Wat. Sci. Tech., Vol. 41, No. 6, S. 17 - 24, 2000
- Prediction of odorous emissions, in: Odours in Wastewater Treatment - Measurement, Modelling and Control, S. 201, IWA Publishing, London, 2001
- Regulations and Policies, in: Odours in Wastewater Treatment - Measurement, Modelling and Control, S. 16, IWA Publishing, London, 2001

Ⅱ　Odor Measurement Review

1. Policy Frameworks and Role of Measurement

The History of Odor Measurement in Japan and Triangle Odor Bag Method

YOSHIHARU IWASAKI
Tokyo Metropolitan Research Institute for Environmental Protection

Keywords
Odor Measurement, Triangle Odor Bag Method, Panel

Abstract

An outline of the history of odor measurement methods in Japan is mentioned in this paper. The syringe method was widely used about 30 years ago. This method, however, had the several disadvantages.
In order to eliminate these disadvantages, the syringe was replaced by a plastic bag (polyester film) with a 3 liter volume and any preconception of the panel was removed by adopting a triangle test. This new method (Triangle Odor Bag Method) was published for the first 1972 by the author. The outline, procedure and precision of this method were printed in this paper.

1. History of odor measurement in Japan

For odor measurement, there have been instrumental methods which use instruments such as gas chromatograph to determine the odorous gas concentration in ppm and olfactory sensory methods which use the human sense of smell.
The results of the olfactory sensory methods are represented by odor intensity, allowable limit (or acceptability), and odor concentration. Particularly, the last one has widely been used in Japan and foreign countries such as the European countries and the United State of America with high reliability.
Odor concentration is indicated by the dilution ratio, that is, pure air volume required to dilute odorous air to an odor threshold point (odor free point).
In Japan, the A.S.T.M. syringe method1) that was published in 1960 by N.A.Huey2), had been widely used as a means of measuring odor concentrations until about 30 years ago. This method, however, had the following several disadvantages .

(1) Small volume of syringe (100ml).
(2) Adsorption of odor on syringe surface.
(3) Long preparation time of highly diluted sample.
(4) Occurrence of unnatural feeling when sniffing odor from the syringe into nose.
(5) Influence of preconception of panel members.

Our data obtained by the A.S.T.M. syringe method at that time was very scattered.
In order to eliminate these disadvantages, the syringe was replaced by a plastic bag (polyester film) with a 3 liter volume and any preconception of the panel was removed by adopting a triangle test.
This new method (Triangle Odor Bag Method) was published for the first time at the

annual meeting of the Japan Society for Atmospheric Environment in 1972 by the author. The details (sampling, procedure, calculation, etc.) of this method will be explained later.

Next, I will explain the odor measurement methods, except for the Triangle Odor Bag Method. From the late 1970s, we imported the Scentometer[3)] from The Barnebey-Cheney Company located in the United States of America and investigated the credibility of this method such as dilution accuracy, repeatability and so on. As a result, the Scentometer was scarcely fit for use.
In the early 1970s, some institutions in Japan made a respective Olfactometer similar to that used in European countries. Some instruments adopted the 2-step dilution method and some equipment adopted a triangle test.
In 1973, a certain Japanese company placed the Olfactometer on the market. However, adsorption loss of odors on the inner surface of the gas line of the olfactometer in those days could not be disregarded. For this reason, the production of the olfactometer was stopped after the several years.
We investigated the accuracy and availability of the various odor measurement method mentioned above from 1970 to 1990.
After 1974, in Japan, many local governments such as Tokyo, Saitama, Niigata, Yamaguchi and so on adopted the Triangle Odor Bag Method[4)5)] for the measurement method for local odor regulations or ordinances. The national government adopted this method for the Odor Control Law in 1997.
Now, the production of the bags has reached one million bags per year in Japan. The sample number measured by the Triangle Odor Bag Method may be over 10,000 samples.

2. Triangle odor bad method

2.1 Introduction

The A.S.T.M. syringe method had widely been used as a means of measuring odor concentration in Japan, as already mentioned. The syringe method was very easy for measurements at the sources of offensive odor, and there were many examples of this type of measurement till 1975 in Japan.
However, because of the unreliability of obtaining reproducible concentration values, it has been considered that the syringe method is useful only for a rough estimation of odor and not for precise administrative regulation. This paper presents a new Triangle Odor Bag Method which is almost completed after many investigations.
In order to eliminate the disadvantages mentioned above, a 3 liter plastic bag is used instead of the 100 ml syringe. The diluted sample is prepared in the bag by filling it with odor free air and injecting a certain amount of odor sample（primary odor）into it. In this way, the use of the odor bag eliminates (1), (2) and (3) described in a previous paper. (4) is eliminated by breathing odor in the bag using a nose cone.
For elimination of (5), we adopted the triangle method, that is , each panel chooses one bag having odor out of 3 bags in order to obtain more objective results.

2.2 Panel

The sensory method is done using the human nose. It is very important to investigate the scattering of the human olfactory sense results. We used the 5 standard dilution liquids that were developed by Takagi et al. in about 1972. These standard odorants are called “the T&T olfactometer”. These standard odorants are now on the market.

We have investigated the data scatters of the olfactory sense for about 1,000 Japanese. The following results were obtained.

(1) The number of dysosmia, who are unsuitable as panel members, was about 5% of the total study subjects and the ratio increased with age.

(2) The mean threshold difference between the sexes depended on the quality of the odor.

(3) It is evident with age that the olfactory sensitivity to isovaleric acid declines at the rate of 30% per 10 years, which corresponds to a 50% loss of sensitivity in 20 years. Based on the condition of health, the slightly poorer group achieved a lower threshold than the ordinary group.

(4) As a result of analyzing the accuracy of the measured values from the olfactory thresholds of normal subjects, the probability between ±10% of the population mean was about 91% in case of 4 panel members.
When the panel members is 6, it was about 95%.

2.3 Panel screening test

The panel screening test is done using standard 5 odorants. The concentration of the odorants is as follows (dilution liquid is odor-free liquid paraffin).

β—phenyl ethyl archole	$10^{-4.0}$ w/w
methyl cyclo pentenorone	$10^{-4.5}$ w/w
isovaleric acid	$10^{-5.0}$ w/w
γ－undecaractone	$10^{-4.5}$ w/w
Skatole	$10^{-5.0}$ w/w

(1) The 5-2 method is adopted as the procedure for the panel screening test.

(2) 5 odor-free papers (size: 14cm × 7mm) are prepared. We soak the top 1 cm of 2 papers in a standard odorant liquid. The remaining 3 papers are soaked in the odor-free liquid paraffin using the same method.
The subjects sniff the 5 papers, and report the 2 papers that contains the odor

(3) Each subject is tested for the 5 standard odorants using the same above mentioned method.

(4) The subject whose answers are all correct for the 5 standard odorants is passed in the panel screening test.

2.4 Instruments

2.4.1 Sampling instruments

(1) For sampling of odorous flue gas

① Pump
Diaphragm pump
Exhaust capacity is greater than 5 liters/min.

② Sampling bags
Polyester bags or polyvinyl fluoride bags
The capacity of this bag is about 15 liters.

(2) For sampling of odorous ambient air

① Pump
Fan-type pump in general. Exhaust capacity is greater than 20 liters/min.

② Sampling bags
Polyester bags or polyvinyl fluoride bags
The capacity of this bag is about 15 liters

2.4.2 Instruments for olfactory sensory test

(1) Pump to send odor-free air
Diaphragm pump or non-oil pump
Exhaust capacity is greater than 30 liters/min.

(2) Syringe
Glass syringe with volume of 5 to 200 ml and PS gas syringe of 1 ml or less in volume

(3) Odor bag
Polyester bag with glass pipe （outer diameter of 12 mm and length of 6 cm）and labeled No.1 to 3

(4) Nose cone
It is made of hard polyvinyl chloride, nose shaped cover and connected to the glass pipe of the odor bag.

(5) Odor-free air supply equipment
Holder with activate carbon

2.5 Sampling

2.5.1 Method for sampling of odorous flue gas

(1) Sampling time is 1-3 minutes.

(2) We must use an odor-free pump such as a diaphragm pump, for the direct sampling method.

(3) Sampling volume is about 10 liters. Material of sampling bag is usually polyester. （refer to Fig.1）

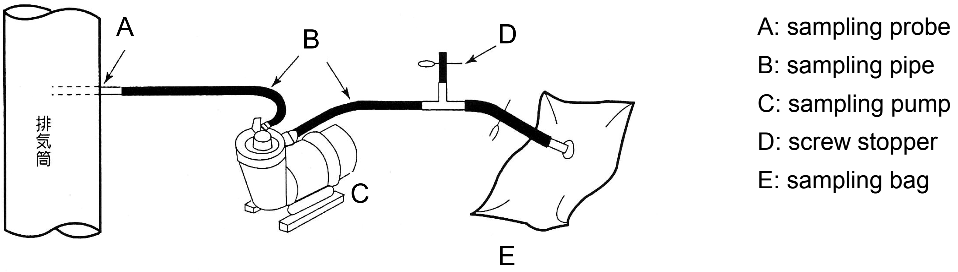


Fig.1 Sampling of odorous flue gas

(4) When moisture of flue gas is at a very high level, it is better to have a drain trap between the sampling probe and sampling bag for the purpose of avoiding condensation in the sampling bag.

- The indirect method has the advantage of avoiding odor contamination in the sampling pump, but sampling is very difficult when the pressure of the inner duct is very low.
- Sampling must be done on the sensory test day or on the previous day.

2.5.2 Method for sampling of odorous ambient air

(1) Sampling time is 6-30 sec.
(2) We must use odor-free pump such as a fan-type pump. The capacity of the sampling pump must be over 30 L/min.
(3) Volume of sampling bag is about 15 liters,
(4) Sampling volume is at least 10 liters.
(5) Material of sampling bag is usually polyethylene terephthalate.
(6) The sampling is carried out when the odor is strong. Method of the sampling is shown in Fig.2. Odor is sampled in a bag after 1 to 2 exchange of the odor between the outside air and inside of the bag.

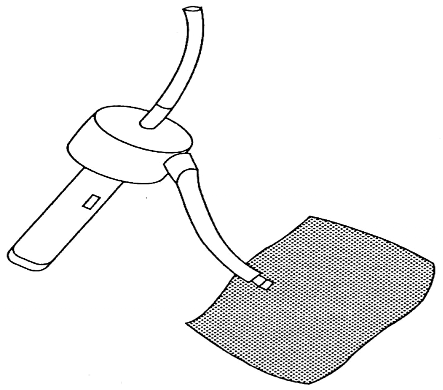


Fig.2 Sampling of odorous ambient air

2.6 Sensory test

2.6.1 Abstract

One bag is the odor bag into which a certain amount of the primary odor is injected and the two other bags are filled with only odor-free air.

Panel tests to determine the odor concentration are carried out by means of dilution, that is, the test is started with a concentration that the panel can easily recognize the odor, and the dilution ratio is successively diluted approximately 3 times in any step of dilution when the answer of the panelist is correct. It is continued until an incorrect answer occurs.

In this way, panels can easily distinguish the odor sample and avoid the risk of coincidence with the concentration ascending method. The dilution of approximately 3 times is adopted because of the distinguishable limit of the human olfactory and convenience in the preparation of diluted sample.

Usually the maximum dilution ratio of the correct answer has been taken for each panel to determine the threshold concentration, but in the present method, the mean maximum dilution ratio coupled with the correct answer and minimum dilution ratio coupled with incorrect answer is used to represent the concentration.

For the final presentation of the result using the present method and not the mean of the threshold concentrations for 6 panels, but the mean of the threshold concentrations for 4 out of 6 panel test results which excludes the maximum and minimum values is taken. This is to avoid the influence of an anomalous threshold value due to panelists who are ill on the test day or make a mistake in writing down the number of dilutions. These mistakes are possible even if they have a normal olfactory.

An odor scale, which is the logarithm of the odor concentration is introduced to indicate similar scale to the human sense. The odor scale corresponds to decibels in sound level.

The screening of a panel is performed based of the conception of J. Amoore, that is, the screening is not for the selection of the ones having an excellent olfactory sense but for ones who have an average olfactory sense, provided that those having an abnormal sense are excluded.

2.6.2 Panel member

A panel consists of over 6 panelists. It is necessary for each panelist to be over 18 years old. All panelist need to pass the panel screening test.

2.6.3 Procedure

(1) Fill 3 odor bags with odor-free air until the bags are almost full and closed with silicone rubber stoppers. Beforehand, completely exchange the remaining air in the bag with odor-free air.
(2) Inject primary odor taken from a sampling bag into one of the odor bags through its label. Its injection volume should meet the required concentration.
(3) The other 2 bags are filled only with odor free air, holes are marked on the labels of the bags by the syringe needle, and the bags are delivered together with an odor-filled bag to a panelist.

(4) The panel sniffs the air in the bags with his or her nose which is connected to glass pipes and lists the number of the bag from which he or she senses an odor.(refer to Fig.3)

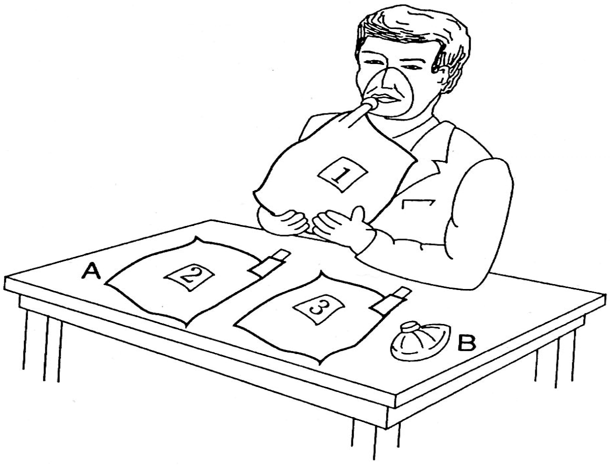


Fig.3 Triangle Odor Bag Method

(5) When his or her answer is correct, the same procedure is carried out at the next stage in which the odor is diluted approximately 3 times. This procedure is continued until the panel gives an incorrect answer and then test by the panel is finished.

2.7 Calculation

2.7.1 For the emission point sample

(1) Threshold values are calculated for each panelist using the following formula (For example, Panelist A).

$Xa = (\log a1 + \log a2) / 2$
Xa : threshold value for panelist A
a1 : correct maximum dilution ratio
a2 : incorrect minimum dilution ratio

(2) Mean of the threshold values calculated for each panelist in (1) excluding minimum and maximum values is taken as the threshold value for a group of all the panelists.

(3) The odor concentration is calculated by converting the threshold value obtained in (2) as follows:

$Y = 10^X$
X : threshold value for a group of all panels
Y : odor concentration

(4) Introduce order index（Z）as follows.

$$Z = 10 \log Y = 10X$$

2.7.2 For an environmental sample (site borderline sample)

As for a sample collected on the site borderline, the selected operations mentioned above are carried out three times for each panel at the dilution rate of 10 as a rule. We calculated the average correct answer rate as follows.

The scores ”correct answer = 1.00”, ”incorrect answer = 0.00” and “unknown = 0.33” to various results, and the average correct answer rate is obtained for all the answers.

When the average correct answer rate falls below 0.58, the sensory test will stop, and the final odor index of the sample is below 10.

When the average correct answer rate becomes greater than 0.58 with the initial dilution rate, the same operation will be repeated at the second operation (dilution rate = 10 × initial dilution rate).

The final odor index of the odor sample is calculated using the following equation.

$$Y = 10 \log \{M \times 10^{(R1-0.58)/(R1-R0)}\}$$

where

Y : odor index

M : the dilution rate 10 at a rule

R1: average correct answer rate obtained from the first operation

R2: average correct answer rate obtained from the second operation (dilution rate = 100 as a rule)

3. Precision

3.1 Volume error of the odor bags

The size of the odor bag（25cm × 25cm） is specified as a volume of approximately 3 liters when filled with the odor-free air. The variation in the volume of the odor bags was measured. Table I shows the variation when ten operators separately prepared 12 bags filled with odor-free air. The measurement is taken as follows. At first the air in the bag is pumped out by a diaphragm pump through a syringe needle which is inserted through a silicone rubber stopper and the air volume in the bags is measured with an authorized wet-type gas meter.

The errors in the volume in comparison with the specified volume of 3 liters are as follows. For 7 operators out of 10, the errors for all of the 12 bags are within ±150 ml (5% of 3,000ml) and for the remaining 3 operators, one of their measurements was more then 150ml, that is, +170ml, +152ml and -219 ml . Regarding the operator to operator variation, the maximum average for one operator is 3,108 ml and the minimum is 2,929 ml. Based on our opinion, the allowable error for this kind olfactory testing is about 10%, so that the above results are within the limit both personally and interpersonally.

Table 1　Fluctuation in the volume of the odor bag

Operator	A	B	C	D	E	F	G	H	I	J
N	12	12	12	12	12	12	12	12	12	12
Min	2966	3067	3038	2875	2870	2897	2954	2924	2978	2981
Max	3170	3152	3145	3003	2983	3030	3056	3048	3117	3017
Ave	3108	3103	3100	2938	2929	2986	2999	2996	3059	2961
S.D.	50	23	31	31	32	43	33	33	41	59

3.2　Diffusion speed of odor in the odor bags

For preparing the odor bag, the primary odor sample is injected through the label as mentioned before. We examined the diffusion speed of the odorants in the bag using the following experiment. The odor bag was filled with odor-free air and then an odorous gas sample of 3 ml was injected into it. 1 ml of the sample at a corner of the bag（2 cm away from the corner）is periodical sampled with a syringe and the concentration of the odorants determined by gas-chromatography.

The final concentrations are 70 ppm for CH4, 0.4 ppm for CH3SSCH3，7 ppm for C6H4CH3，and 4 ppm for CH3COC2H5.

Fig.4 shows the diffusion speed of the odorants versus time.

The vertical axis indicates the ratio of the concentration of the odorants in the air sampled at the corner of the bag to that after they have completely diffused. The figure shows that all the gases completely diffused within one minute.

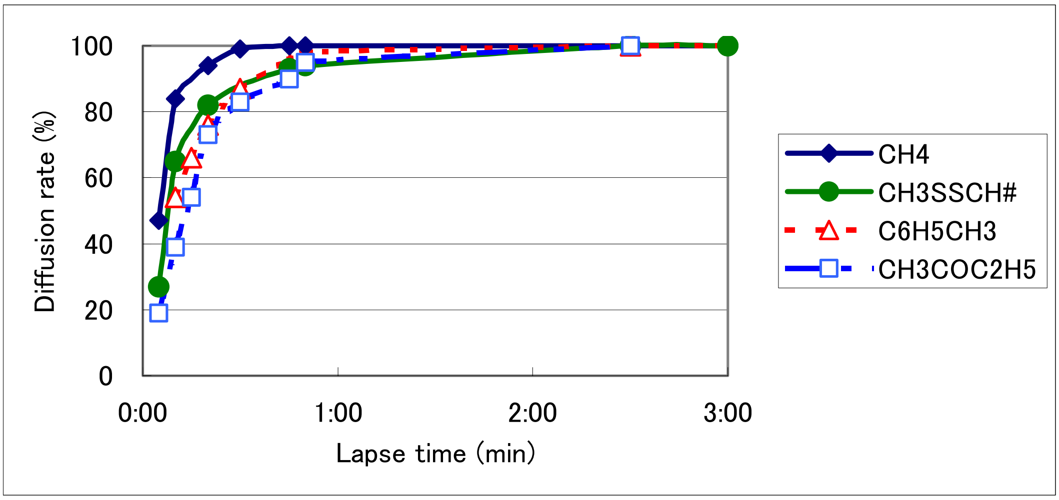


Fig.4　Diffusion rate of odorants in the odor bag

3.3　Stability of sample gases in the sampling bag

To examine the stabilities of odorous sample gases, we used typical offensive odorants and field sample gases. We investigated the relationship between the odor concentration and the time required since the sample gases added to the sampling bag. Six panelists examined the odors on all of the days. The odor samples were dry exhaust odor from a fishmeal plant，indoor air of chocolate manufacturing plant and exhaust gas from incinerator of sewage sludge cake, and drying exhaust air from gravure printing industry. As shown in Fig.5, the measured results remain unchanged for several days after the samplings.

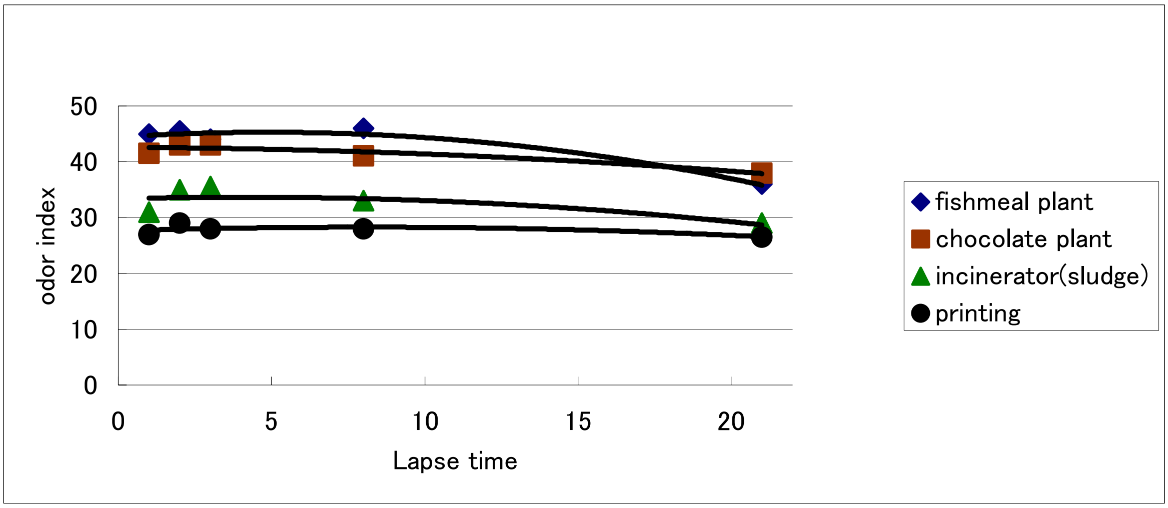


Fig.5 Stability of odors in the sampling bag

3.4 Influence of panel on precision of odor measurement

Panel selection and their number have a strong influence on the precision of the odor measurement. Abnormal olfactory persons are to be excluded by panel screening test, but it is not expected that all of them are excluded through the panel screening test indicated in 2.3). Sometimes it is also probable that some one gets ill on the test day. To exclude these problems, the Triangle Odor Bag Method adopted the mean of calculation method averaging with removal of the maximum data and minimum data.

Thus it might be mentioned that the influence from any olfactory abnormality is almost totally excluded.

It is natural that increasing of the number of panel members would increase the reproducibility of the measurement results.

4. Measurement Results by Triangle Odor Bag Method

The measurement results taken at the odor sources by the Triangle Odor Bag Method are shown in Table 2. The odor concentrations of the exhaust gas from the offset printing were higher than that of the exhaust gas from gravure printing. This result corresponds to actual complaints near the odor sources.

As for metal printing, the odor intensity depends on the quality of the ink used. The measured printing factories had odor control instruments of the activated carbon type or catalytic combustion system. The concentrations at the printing factories were measured upstream of the odor control apparatus.

The results for the dried exhaust air of the fish meal plant showed a high odor concentration of 31,000. This value was reasonable considering the severe trouble caused by this type of industry.

The sludge cake incinerator of the vertical multistage incineration type was exhausted directly without going through a high temperature combustor. For this reason, the odor concentration for this type was at a very high level.

The results for boilers showed that the odor concentration of the exhaust gas depended on the type of fuel used. For city gas or kerosene, the odor concentrations were very low, but the concentration was rather high for heavy oils A and C.

Table 2 Measured results of odor concentrations at the odor source

Type of industry	Sampling point	Odor concentration	Odor index
printing (gravure)	drier outlet	410 ～ 1700	26 ～ 32
〃 (off-set)	〃	9700 ～ 41000	40 ～ 46
〃 (metal)	〃	1700 ～ 31000	32 ～ 45
fish meal	〃	31000	45
Food (chocolate)	roaster outlet	41000	46
〃 (〃)	kneading room	13000	41
〃 (curry)	mixing room	1300	31
〃 (chewing gum)	mixing room	23000	44
Incinerator (refuse)	stack inlet	1300 ～ 1700	31 ～ 32
〃 (sludge cake)	〃	970 ～ 3100	30 ～ 35
boiler (city gas)	〃	97 ～ 310	20 ～ 25
〃 (kerosene)	〃	97 ～ 310	20 ～ 25
〃 (heavy oil A)	〃	550	27
〃 (heavy oil C)	〃	4100	36
〃 (waste wood)	〃	1300	31

Odor index = 10 × log(odor concentration)

References

1) American Society for Testing Materials "Standard method for measurement of odor in the atmosphere (Dilution method) "Designation D1391-57
2) N. A. Huey et al., J. Air Pollut. Control Assoc., 10, 441 (1960)
3) J. S. Nader, Am. Ind. Hyg. Assoc.,19, 1 (1958)
4) Y. Iwasaki et al,. J. Air Pollut. Soc. Japan, 7, 168 (1972)
5) Y・Iwasaki et al, Annual Report of Tokyo Metropolitan Research Institute for Environmental Protecton 4, 78, (1973)

Odor Regulation and Odor Measurement in Japan

Kenji Kamigawara
Noise and Odor Division
The Ministry of the Environment, Japan

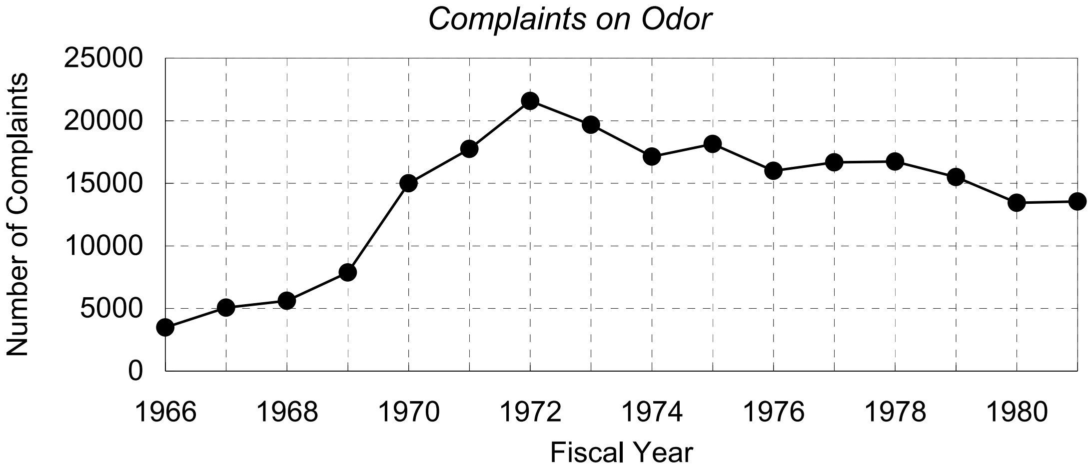
Keywords
Complaints, measurement, local government, regulation

Abstract

The Offensive Odor Control Law, which enacted in 1972, introduced an instrument measurement method on each substance. The number of complaints on offensive odors, especially from factories and livestock farms, has gradually declined. However, the number of complaints on offensive odors of service industries has been increasing. It is difficult to apply the instrumental odor measurement method to odors from those service industries. The 1995 Amendment to the Law introduced olfactory measurement using “triangular odor bag method” and obliged local government to contract with certified operators when they commission olfactory measurement. The Ministry published a manual of quality control on olfactory measurement and a manual of safety assurance of olfactory measurement in 2002. In order to support technical development, the Ministry started technology verification on odor control equipment for small service industries.

1. Establishment of National Policy on Odor Control

During 1960’s, local Governments had received increasing complaints on offensive odors. First, large factories, such as oil refineries and pulp industries, were established in many areas of Japan. Also, urban area spread out and houses were constructed even near livestock farms.


Complaints on Odor

The Ministry of Welfare launched studies on environmental odor control in latter half of 1960s. The most difficult issue was the establishment of odor measurement methods.

The Society for the Study of Offensive Odor was launched in 1969. This was the predecessor of Japan Association on Odor Environment. The Ministry of Welfare contracted with the Society to carry out the research and development of odor measurement methods. The Society recommended the instrumental measurement method in 1970. The main reasons were as follows;

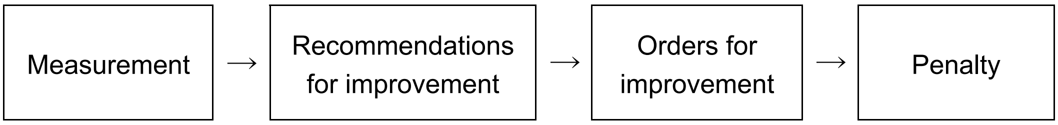
(1) Odorous substances cause social problem above the level of ten times to hundred times higher than detective threshold. Therefore, the instrumental measurement can be already technically applicable.
(2) The instrumental measurement can easily detect emitter of odor.
In addition, the olfactory measurement method was not fully established yet at that time.

In 1971, the Ministry of Welfare sent the Bill on the Offensive Odor Control to the Diet and it was passed in May 1971. The Environment Agency, established in July 1971, succeeded odor control policies from the Ministry of Welfare. Then it prepared the implementing rules of the Offensive Odor Control Law (hereafter called “the Law”), and the Law was enacted in May 1972.

The Law introduced an instrumental odor measurement method on each substance, mainly by utilizing gas chromatography. Now 22 substances are designated under this law (see annex).

The Law stipulates that;

(1) Prefectural governors shall designate regulation areas and shall establish regulatory standards on acceptable concentration of each odorous substances in accordance with national guidelines.
(2) Factories and other business establishments in regulation areas shall comply with regulatory standards.
(3) Mayors may make recommendations or orders to factories and other business establishments in regulation areas to take measures to reduce the emission of odor emission, when they do not comply with regulatory standards and the offensive odor from them is damaging living environment of residents.
(4) If those factories and other business factories do not comply with above-mentioned orders, they may be penalized.



Why are only the regulation areas subject to the regulation? The reason comes from the nature of odor problem. There are a variety of ways how people feel odors, for example, there are some differences between cities and agricultural areas. The typical areas to be regulated are built-up areas and suburban areas with schools and hospitals. The number of municipalities that have regulation areas has increased year by year, and amounts to 1,792 which is 55.2% of the total in the end of FY of 2001. The municipalities that do not have any regulation area are mostly rural villages. In FY2001, 80% of complaints on offensive odor occurred in regulation areas.

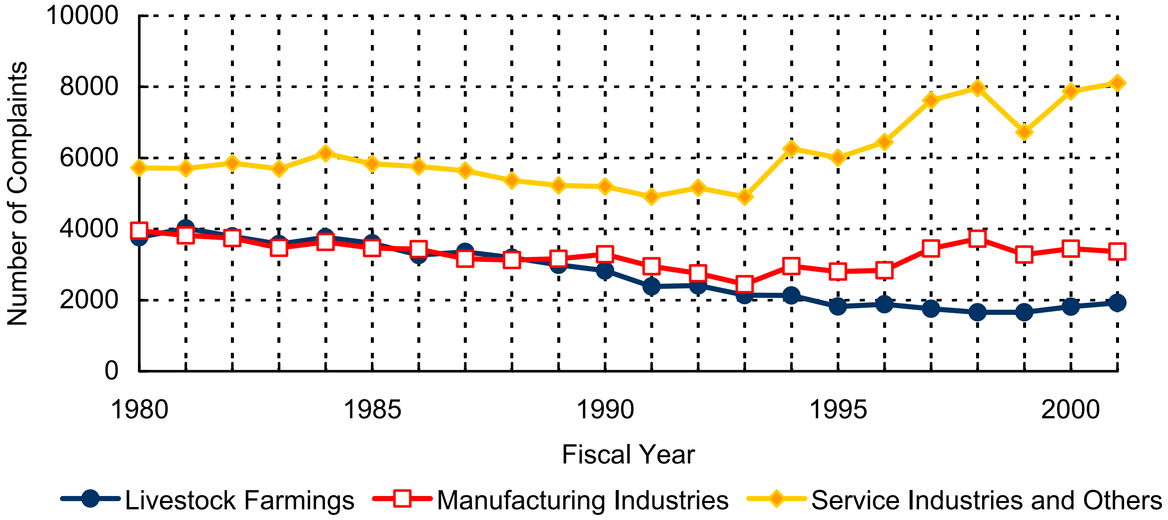
In FY2001, based on the Law, local governments made 6,844 inspections, ordered reports on 772 cases and made recommendations on 7 cases. In FY2001, they also made informal recommendations on 11,376 cases and made measurement on 850 factories and business establishments with 2,821 samples.

2. The Changing Situation

The numbers of complaints on offensive odors from factories and livestock farms, have a gradually declined by mid 1990s. However, the number of complaints on offensive odors of service industries, e.g. restaurants and garages, has been increasing.

It is difficult to apply the instrumental odor measurement methods to odors from those service industries, because they emit many kinds of odor substances. It is estimated that the instrumental measurement methods are only applicable to 30% of factories and business establishments concerning offensive odor.

Number of Complaints on Odor
except Concerning Outdoor Burning



3. 1995 Amendment to the Law

The Environment Agency had started studies on the olfactory measurement methods in the middle of 1970’s. The Tokyo Metropolitan Government had already established the “triangular odor bag method” in 1972. The Environment Agency verified reliability of this method. The report of the study stressed the need of quality assurance for measurement operators. The Environment Agency established the national method for olfactory measurement using “triangular odor bag method” and the national certification system for operators of olfactory measurement in 1992.

At that time, over 30 local governments including 13 prefecture besides the Tokyo Metropolitan Government had already introduced the olfactory measurements to their bylaws or guidelines to control offensive odor.

In this context, the Environment Agency drafted the amendment to the Law in order to introduce the olfactory measurement using “triangular odor bag method”, and the amendment went through the Diet in 1995.

The amendment stipulates that a Prefectural Governor shall choose the regulation using either the instrumental measurement method of the concentration of designated odorous substances or the olfactory measurement method, which is called “odor index regulation”.

The major advantages of the olfactory measurement method are as follows;

(1) It is applicable to any odorous substances.
(2) It can evaluate the additive and multiplicative effects of odorous substances.
(3) The results well meet the residents’ sentiment.
(4) Therefore it is widely used in other developed countries as a reliable method.

4. Operators of Olfactory Measurements

Mayors may make recommendations or orders based on the results of measurements to factories and other business establishments, and these orders based on the Law may lead to penalties. Therefore these measurements requires accuracy. The amended Law obliged local government to contract with certified operators when they commission olfactory measurement.

The Environment Agency contracted with the Association to carry out the certification system of operators of olfactory measurements. If a person wants to have a certification as the operator of olfactory measurements, he/she is required to pass the paper examination carried out by the Association. After passing the paper examination, he/she shall pass olfactory test. The purpose of the test is to eliminate persons who do not have normal ability of olfaction.

Occupational categories of persons certified for Operators as of 1 April 2003

Type of Occupation	Number	%
Measurement and Analysis	921	44.3
Research and Study	338	16.2
Engineering and Planning	255	12.3
Executive and Management	126	6.0
Trade and Sales	127	6.1
Manufacturing and Quality Control	93	4.5
Others	221	10.6
Total	2081	100

At the end of the FY 2002, 2081 persons have the certification. Around 40% of them are working for private laboratories for measurement and analysis.

(The Ministry estimates that about 0.8 million odor-bags were sold in 2002 in Japan.)

5. Introduction of “Odor Index Regulation” to the Local Governments

After the amendment in 1995, the number of local governments that adopt “odor index regulation” increased slowly. One reason was that full set of national guidelines on regulatory standards for odor emission had completed in just 2000. However, Tokyo Metropolitan Government, the biggest prefectural government in Japan, introduced odor index regulation by the Law in July 2002. This accelerated the adoption in other areas. (Tokyo Metropolitan Government had their bylaw with “odor index regulation”.

However this bylaw limited its application to designated factories and business establishments. The Law does not have this kind of limitation, so the Law is applicable much more widely than the bylaw.)
The Ministry of the Environment is promoting this movement by carrying out seminars in many regions and by providing information materials.

6. Quality Control and Safety Assurance of Olfactory Measurement

In order to spread “odor index regulation” to local governments, quality control of olfactory measurement is essential. The Ministry contracted with the Association to make a manual of quality control on olfactory measurement and the Ministry published it in 2002.
Also, in carrying out olfactory measurement, it is important to assure safety of sampler, panelists and operators. The Ministry contracted with the Association to make a manual of safety assurance of olfactory measurement in and published it in 2002.

7. Technology Verification on odor control equipment

As already mentioned, the number of complaints concerning odors from service industries has increased. Those service industries include many kinds of small business. They are not able to install odor control equipment, which is expensive or occupies large space. The makers of equipment are providing information on their goods, however the information is not verified by the third parties.
In order to support technological development, the Ministry of the Environment contracted with the Association in 2002, to carry out technology verification on odor control equipment for small service industries. The Association received application of 51 technologies from 38 companies and it made technology verification for 20 technologies. The report of result has been published in this June.

8. Concluding Remarks

Japan has set up the system for odor control, consisted of the Law and its implementing rules, local bylaws, measurement methods, certification for operators, and quality control process. Corporation among the Ministry, local Governments and experts of the Association has been essential to develop and implement the system.
There remain some issues. First, the Ministry should continue promoting the odor index regulation and the olfactory Measurement to be adopted by further local governments. Secondly, they should keep quality control process. Thirdly, small, cheap and easy-to-maintain equipment is required for small business to control offensive odor.

References

1) Quality of the Environment in Japan 1999, the Environment Agency, pp94-97
2) Higuchi, T. and Nishida, K.1995, Analysis of data measured by the triangular odor bag method, in McGinley, C.M.and Swanson, J.R.(eds.), *ODORS: Indoor and Environmental Air*, Air & Waste Management Association, Pittsburgh, pp.181-192
3) Higuchi, T. et al. 2000, Improvement of triangle odor bag method and triangle odor flask method as sensory measurement methods for odor emitted from wastewater, in *Odors and VOC Emissions 2000*, Water Environment Federation, Alexandria, VA, USA.

4) Higuchi, T. et al. 2002, Establishment of quality control framework for olfactometry in Japan, in *Odors and Toxic Air Emissions 2002*, Water Environment Federation, Alexandria, VA, USA.

Annex

Specified Offensive Odor Substances

Acetaldehyde,
Ammonia,
Butyraldehyde,
Butyric acid,
Dimethyl disulfide,
Dimethyl sulfide,
Ethyl acetate,
Hydrogen sulfide,
Isobutyraldehyde,
Isobutyl alcohol,
Isovaleraldehyde,
Isovaleric acid,
Methyl isobutyl keton,
Methyl mercaptan,
Propion aldehyde,
Propionic acid,
Styrene,
Toluene,
Trimethylamine,
Valeraldehyde,
Valeric acid,
Xylene

Odor Regulation and the History of Odor Measurement in Europe

Anton Ph. Van Harreveld, OdourNet
Crtra. de l'Esglesia 60 B, Barcelona 08017, Spain
tonvanharreveld@odournet.com

Keywords
Olfactometry, standardisation, odour

Abstract

The concentration of people in cities in Europe has led to considerable odour problems, which were addressed by a variety of regulations for many hundreds of years. In the past 30 years there is a trend to move away from using the judgement of an environmental health officer, and to rely on quantitative measurements of odour instead. Pioneered in The Netherlands, the trend is towards quantitative odour management based on measurement of emissions, dispersion modelling to define exposure and criteria derived from dose effect studies to define a level where no 'reasonable cause for annoyance' exists. These criteria may be specific to an industry, depending on the offensiveness of the odour. A reliable method for odour concentration measurement is an indispensable tool required for this approach, and such a method is now available is the European standard EN13725:2003. An initial comparison of results shows a remarkable agreement of results between this method and the Japanese Triangle method. The rigorous selection of assessors for the panel is likely to be the critical operational parameter that contributes to this agreement.

Odor nuisance is a matter between neighbours. It occurs whenever people concentrate their lives, homes and activities in cities and towns, as has been happening in Europe in the past 3-4 centuries. It is not surprising, therefore, that even the earliest written legislation in European countries addresses nuisance, including that caused by smells

Early European legislation on a local level, regulated smelly activities such as slaughtering and tanning of hides, typically by deciding that this should be done outside of the town, or downstream on the river. Europe was nevertheless a very smelly place, until quite recently. Imagine the smell of the first cities to house large numbers of people, such as Paris and London, well before sewers and sanitation became commonplace in the second half of the 19th century. London had over 100,000 inhabitants in 1600 and the second census of 1811 put the population of London at over 1 million for the first time. There was no sanitation to speak of, and the waste of all those people was discarded in the same river that provided most of them with drinking water, resulting in outbreaks of disease, such as cholera, claiming many lives. It was, after all, not until the mid 19th century that the link between water and disease was made by Dr Snow in London (1854) and Louis Pasteur in Paris. Until then bad smells and 'vapours' were associated with disease.

It was actually not the disease, but rather the *smell* of the polluted Thames River, that caused the UK Parliament to decide, after the ‘big stink’ of the summer of 1858, to allow the construction of the main London sewers, creating a bypass along the Thames to sea. In those days the curtains and drapes of the Houses of Parliament, were treated with ‘chloride of lime’ to combat the odours. In spite of these attempts Parliament was closed in 1858 because of the unbearable smells from the river. For a vivid description of the smells of Paris in the pre-sanitation times I can suggest reading the book of historian Alain Corbin[1] that inspired the even more fascinating novel The Perfume, by Patrick Süskind[2].

The issue of nuisance caused by smells was traditionally regulated by common sense regulations. Very smelly processes were to be located away from where people lived. If conflicts arose, the situation was assessed by the relevant authority. More general principles were included in Nuisance Law, which was established in many countries in the late 19th century, when industrialisation led to larger scale processes and increasing urbanisation, and hence more residents affected. The details of these legal developments and the differences between countries are beyond the scope of this paper.

The principles of Nuisance Law are used until today, especially in countries with a legal system based on Common Law. However, society increasingly demands transparent and uniform environmental regulations, with the aim to achieve a uniform level of risk and protection for all citizens. Also, industry requires a predictable and clear set of performance criteria, to be able to plan their investments in environmental management. Recently, as a result of the common market in the European Union, there is a movement to achieve convergence of environmental protection, with the economic objective of ensuring uniform regulatory pressure, and hence uniform competitive conditions throughout the EU. These developments have led to a gradual introduction of regulations and guidelines that increasingly depended on quantification of impacts and criteria for ‘acceptable exposure’ to odors[3].

The first sector to be regulated on a national level specifically for odour impacts was the intensive livestock sector. In the Netherlands, with a very large pig production sector, a practical guideline was imposed in 1971 on new and existing livestock operations, which determined the minimum distance between residential housing and livestock housing facilities, depending on the capacity as counted in number of pigs[4].

This regulation was initially based on experience of public health inspectors. However, it led to research into methods to quantify odour emissions. In the Netherlands, with its high population density, industrial and agricultural activity and high economic level, the need for managing odour impacts was felt. In 1984 a quantitative air quality guideline for odours from industrial sources was introduced[3].

The guideline was based on measurement of odour emissions using olfactometry, followed by dispersion modelling to predict frequency of exposure to hourly average concentrations in excess of a certain limit value. In 1984 the Ministry of Public Planning, Housing and the Environment in the Netherlands set two exposure criteria, a more lenient one for existing facilities and a tougher limit for new installations:

- The odour concentration at the location of 'sensitive objects', such as residential housing, shall not be higher than 1 ge/m^3 as a 99.5 percentile of hourly average concentration, new industrial facilities ($C_{99.5,\ 1\text{-hour}} < 0.5\ ou_E/m^3$)
- The odour concentration at the location of 'sensitive objects' shall not be higher than 1 ge/m^3 as a 98.0 percentile of hourly average concentration, for existing industrial facilities ($C_{98.0,\ 1\text{-hour}} < 0.5\ ou_E/m^3$)

These regulations were applied between 1984 and 1995, and were found effective in reducing annoyance among the population leaving nearby regulated industries. However, a number of problems were identified in the years after introduction:

- The regulations did not take into account differences in offensiveness, and regulated a bread factory in the same way as a rendering factory
- The regulations were perceived to be too protective, too strict, and too rigid in their approach
- The measurement techniques available were not capable of providing sufficiently accurate data for enforcement

These issues have been addressed in the following years, and a more flexible approach has been introduced in 1995, and now formalised in the Netherlands Emission Guidelines of 2000[5].

Of course it was absolutely essential that the methods of measurements were improved so that they could be used in court. This requirement was recognised and led to development of standards, which will be described in more detail below.

The approach in the Netherlands was typical for a trend in other Northern European countries, such as Germany and Denmark. More recently, Belgium has started a systematic programme to develop a regulatory framework for managing its environmental odours, and the Irish Environmental Protection Agency has moved to define criteria for specific sectors, e.g. the livestock production sector (pigs)[6] and the mushroom growing substrate composting sector. A description of the history of odour related regulations in different European and non-European countries can be found in a recent research document that was prepared for the UK Environment Agency [7].

Recent regulatory developments in the United Kingdom are perhaps the most interesting to consider in some more detail. Until recently the regulation of odour emissions and nuisance was based on Local Authority Air Pollution Control legislation and the chapter on statutory nuisance in section 79 of the Environmental Protection Act 1990, stating: *"A Statutory nuisance includes any dust, steam, smell or effluvia arising on industrial, trade or business premises which are prejudicial to health or a* *nuisance"*. Ultimately, these regulations relied on Environmental Health officers to judge specific situations to decide if a statutory nuisance was present, in which case they could invoke measures to remove the causes. In practice, this led to wide variations in assessment. This is expected to change in the near future after publication of *Technical Guidance Note H4, Integrated Pollution Prevention and Control (IPPC), Horizontal Guidance for Odour* by the Environment Agency, in January 2003[8,9]

The H4 guidance provides a framework of assessment and regulation for processes as provided in the Integrated Pollution Prevention and Control directive of the European Union[10]. Specific processes that fall under IPPC are required to determine their impact on 11 criteria, one of which is odour impact. The sectors of industry involved are encouraged to define 'Best Available Technique' (BAT) on a European level to achieve greater efficiency in environmental management of these facilities.
With the H4 guidance the UK Environment Agency has provided a well defined framework for implementation of the IPPC directive for odours in the UK.It is to be expected that production facilities that fall under other regulatory frameworks, such as the local authority, will be assessed with the H4 guidance in mind. Planning procedures are similarly likely to consider principles laid out in the H4 guidance.

The H4 guidance provides a considerable degree of flexibility. It does, however, require a quantitative approach, based on quantification of emissions and dispersion modelling to determine if *'reasonable cause for annoyance'* exists at the location of residential property and other sensitive localities. It also recognises that differences in offensiveness, or annoyance potential[11], that can lead to differentiated exposure standards for specific sectors of industry. In Appendix 6 of Part 1 of the H4 guidance[8] an indication of possible criteria is provided, which range from $C_{98.0,\ 1\text{-hour}} = 1.5\ ou_E/m^3$ for more offensive odours to $C_{98.0,\ 1\text{-hour}} = 6\ ou_E/m^3$ for odours with a low annoyance potential. These indicative criteria are derived from a dose effect study conducted in the Netherlands for the livestock production sector[12], that were also used as a starting point to derive air quality criteria for exposure to livestock odours for the Irish EPA[6].

The H4 guidance explicitly states that sectors of industry can develop specific exposure criteria defining the level of exposure associated with *'reasonable cause for annoyance'*, by carrying out dose effect studies. In such a study, the effect is measured using a Standardised Telephone Questionnaire technique, where 100-250 people living in a delimited area are asked a number of questions, two or three of which are relevant to odour annoyance. They are not made aware that odour annoyance is the objective of the survey. Based on the answers given, each respondent is classified 'annoyed' or 'not annoyed. In this way a prevalence of 'odour annoyed' is obtained, for people exposed to a certain level of odours. A minimum of five exposure levels is surveyed in this way, and from the responses a correlation cure between the effect (annoyed) and the dose (odour exposure, $C_{98,\ 1\ \text{hour}}$) is obtained[9, 12, 13]

The uncertainty of the method is typically 3 percentage points, while in control areas a ‘background’ of 2-3% annoyed is observed. Therefore, at the background level plus two times the uncertainty, or approx. 10% annoyance, the annoyance effect is likely to be detected with sufficient statistical confidence.

The dose is measured by determining the emission of odours, using olfactometry, followed by dispersion modelling. This leads us to the central and crucial requirement for any quantitative method to reliably manage odour impacts: sufficiently accurate emission measurement. From the historical perspective of this paper it is notable that one of the earliest legal texts, he Magna Charta granted by King John of England and Ireland on June 5th 1215 recognised the need to use the same units for mass and length throughout the Kingdom:

There shall be one measure of wine throughout all our kingdom, and one measure of ale, and one measure of corn, namely the quarter of London; and one breadth of dyed cloth, and of russets, and of halberjects, namely, two ells within the lists. Also it shall be the same with weights as with measures.

The same basic requirements of reproducibility apply to odour measurement today, especially when its results are to be used in a legal context of licensing and enforcement. Olfactometry, the measurement of odour concentration using human subjects, has been practised for over a century now. The first reported odour thresholds are from 1848[14] with comprehensive studies appearing in the 1890’s[15]. The early olfactometers were built by pioneers such as professor. Zwaardemaker, of the University of Utrecht, the Netherlands, as shown in figure 1. A more recent, but less portable model is shown in Figure 2.

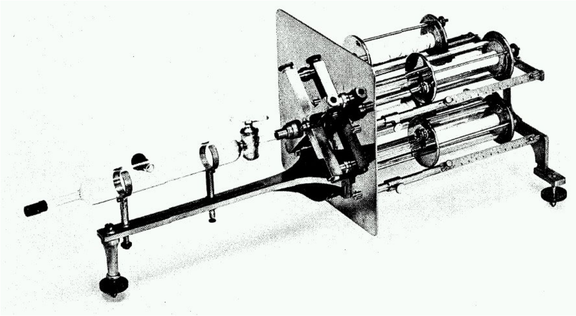


Fig 1: Zwaardemaker olfactometer, The Netherlands,1886

Fig.2 Modern olfaktomat olfactometer, OdourNet, UK, 2000

A remarkable volume of published data from the early years of the 20th century exists on odour detection thresholds for compounds. Unfortunately, the differences in results of odour thresholds in literature are very considerable. Compilations that have been published[16, 17] typically show a range of several orders of magnitude. When olfactometry was taken out of academic research, and increasingly drawn into the arena of environmental management of odours, standardisation was a logical step. A number of standards appeared such as VDI3881 (Germany)[18]. However, these failed to address the *'significant operational variables'* that had been accurately identified by Dravnieks in 1980[19]. The main issue was that no reference odours were defined, and no 'agreed reference values' for these odours agreed that could be used to 'calibrate' panels through selection of assessors for a specific olfactory acuity. As we now know, the variability of olfactory acuity between individuals is too large to accurately form a representative sample of the population, at practical panel sizes (e.g. n<10). Other issues were of a more technical nature, such as instrumental calibration, materials of construction and ensuring sufficient flow of odorous stimulus presented to avoid dilution with ambient air during inhalation (>20 l/min).

The significant operational variables, as identified by Dravnieks, were addressed in standards that included a form of assessor selection using reference odours such as AFNOR[20]. The Dutch NVN2820:1990 standard, in addition to panel selection, set a reference level of 20 ppb/v n-butanol for 1 'Dutch odour unit', or ge/m^3, and added statistical QA/QC procedures[21, 22].

These national standards of EU countries will now be replaced by the EN13725:2003 standard, that has been introduced in April 2003, after close to 10 years of preparation[23]. This standard defines the EROM, or a mass that is just detectable when evaporated into 1 m^3 of neutral gas, as equivalent to 123 μg n-butanol. In other words: 1 $ou_E/m^3 \equiv$ 40 ppm/v. Strict panel selection procedures, using n-butanol as a reference odour, are used as a form of 'span adjustment'. Statistical QA/QC procedures are integrated in the measurement protocol. These measures have resulted in a marked improvement in the performance of olfactometry, which has been verified in an increasing number of blind interlaboratory tests. These developments, which were driven by a regulatory demand, have been described in more detail in a paper published in the AWMA journal[24]. It is satisfying that Australian Standards have published a standard AS/NZ4323.3 that closely resembles EN13725.

Table 1		**Odor detection thresholds in ppm**		
Compound	Odor quality	NL	Japan	Factor Japan/NL
Acetone	Sweet/fruity	28.0		
Benzene	Aromatic/sweet	1.7		
n-Butylacetate	Sweet/banana	0.076		
n-Butanol	Sweet/alcohol	0.040	0.038	0.95
Ethyl Alcohol	Sweet/alcohol	0.370		
Hydrogen Sulfide	Rotten eggs	0.0005	0.000495	0.99
Isobutyl Alcohol	Sweet/musty		0.012	
Methyl Ethyl Ketone	Sweet/sharp	3.1		
Methyl Mercaptan	Rotten cabbage		0.000102	
Styrene	Sharp/sweet	0.025	0.033	1.32
Toluene	Sour/burnt	1.6	0.9	0.58

It is very interesting to note that the key 'significant operational parameter' of panel selection is so elaborately addressed in the Japanese Triangle Method. Maybe that explains why, in spite of a very different technical approach on the instrumental level, the results obtained in Japan appear to be very close to those obtained using the NVN2820 method, that is compatible with the European EN13725 standard. The odour thresholds (or EROM's) for a limited number of compounds, that could be found in available papers, are compiled in table 1. The agreement between the methods is quite good, with differences of less than 50%.

With this promising indicative review in mind, it will be very interesting to learn of the results of more elaborate comparisons of the Triangle method and the EN13725 method.

The aim, after all, is that odour measurements all over the world can be compared and used to add to our combined knowledge on how odour emissions can be characterised, with the ultimate purpose of managing odorous impacts and avoiding detrimental impacts of offensive odours on the enjoyment of life.

Conclusions

- In Europe there is a trend towards quantitative air quality criteria for odours, using dose-effect studies to determine a level where 'no justified cause for annoyance' exists
- A precondition for this approach is the availability of odour measurement techniques with a known uncertainty, that is sufficiently small for use in a legal framework
- Selection of assessors appears to have been the main 'critical operational parameter' causing the lack of reproducibility in olfactometry
- The detection thresholds obtained using the Japanese Triangle Method appear to be in close agreement to those obtained using EN13725.

References

1) Corbin, Alain, *The Foul and the Fragrant: Odor and the French Social Imagination*, Harvard University Press, 1988, ISBN 0674311760

2) Suskind, Patrick, *Perfume*, Penguin Books,ISBN 0140120831, 1989

3) Van Harreveld, A.P., (1991) *Introduction and Related Practical Aspects of Odour Regulations in the Netherlands*:, in: Derenzo and Gnyp, TR18, Recent Developments and Current Practices in Odour Regulations, Controls and Technology, AWMA, ISBN 0-923204-0, 1991

4) Van Harreveld, A. Ph. , Klarenbeek, J.V., *On the regulations, measurement and abatement of odours emanating from livestock housing in the Netherlands*, in Proceedings: New knowledge in Livestock Odor, Proceedings of the International Livestock Odor Conference 1995, pp.16-21, Iowa, USA, Iowa State University, College of Agriculture,Ames, 1995

5) Infomil, (2000) *NeR Nederlandse Emissie Richtlijn. (Netherlands Emissions Guideline)*, ISBN 90 76323 01 1, 2000 (English version available via www.infomil.nl)

6) Van Harreveld, A.P., Jones, N., Stoaling, M., *Odour Impacts and Odour Emission Control Measures for Intensive Agriculture. Part A: Odour annoyance assessment and criteria for intensive livestock production in Ireland*., Environmental Monitoring, R&D Sub programme of the Environmental Services Operational Programme 1994-1999, Environmental Protection Agency,

Ireland, ISBN 1-84095-075-7, 2001

7) Anon., *Assessment of community response to odorous emissions*., R&D Technical Report P4-095, Environment Agency, WRc Document Distribution Centre, United Kingdom, 2002. available in hardcopy from www.environment-agency.gov.uk
8) *Technical Guidance Note H4, Integrated Pollution Prevention and Control (IPPC), Horizontal Guidance for Odour, Part 1 Regulation and Permitting*, Environment Agency, UK, 2002. Available on www.environment-agency.gov.uk
9) *Technical Guidance Note H4, Integrated Pollution Prevention and Control (IPPC), Horizontal Guidance for Odour, Part 2 Assessment and Control*, Environment Agency, UK, 2002. Available on www.environment-agency.gov.uk
10) EU Council Directive 96/61/EC of 24 September 1996 concerning integrated pollution prevention and control, *Official Journal L 257 , 10/10/1996 P. 0026-0040*
11) Van Harreveld, A.P., *From odorant formation to odour nuisance: new definitions for discussing a complex process*, Water Science & Technology Vol 44 No 9 pp 9-15, ISSN: 0273-1223, 2001
12) Bongers, M.E., Van Harreveld, A.P., Jones, N., *Recent pig odour policy reviews in the Netherlands and in Ireland*., in: Proceedings of the 1st IWA International Conference on Odour and VOCs: Measurement, Regulation and Control Techniques,, University of New South Wales, March 2001.
13) Miedema, H.M.E., Walpot, J.I., Steunenberg, C.F. (2000), *Exposure-annoyance relationships for odours from stationary sources*, Atmospheric Environment,34.
14) Valentin, G., *Lehrbuch der Physiologie des Menschen*, 2 ,2 , 279-283, Friedrich Bieweg und Sohn, Braunschweig, 1848
15) Passy, ., *Sur quelques minimums perceptibles d'odeurs*, C.R. Hebd. Seances Acad. Sci, 114, 786-788, 1892
16) Van Gemert, *Compilations of odour threshold values in air and water*, Boelens Aroma Chemical Information Service, Huizen, The Netherlands, 1999
17) Devos, M., Patte, F., Rouault, J., Laffort, P., van Gemert, L.J., *Standardised human olfactory thresholds*, IRL Press, Oxford, 1990
18) VDI 3881:1986 Part 1, *Olfaktometrie; Geruchsschwellenbestimmung (Olfactometry – determination of odour thresholds)*, Beuth Verlag, Germany, 1986
19) Dravnieks, A., Jarke, F., *Odor Threshold Measurement by Dynamic Olfactometry: Significant Operational Variables*, Journal of the Air Pollution Control Association, Pittsburg, USA, December 1980
20) Norme Francaise NF X 43-101, Determination of the dilution factor at the perception threshold, AFNOR, December 1986.
21) NVN2820:1990 Luchtkwaliteit. Sensorische geurmetingen met olfactometer (Air Quality – Sensory measurement of odour using an olfactometer), NEN Netherlands Normalisation Institute, Delft, 1990. www.nen.nl
22) NVN2820:1996 annex 1 Luchtkwaliteit. Sensorische geurmetingen met olfactometer (Air Quality – Sensory measurement of odour using an olfactometer), NEN Netherlands Normalisation Institute, Delft, 1996. www.nen.nl
23) EN13725:2003, *Air Quality Determination of Odor Concentration by Dynamic Olfactometry,* CEN, Brussels, April 2003. www.cenorm.be
24) Van Harreveld, A. Ph., (1998) *A review of 20 years of standardization of odour concentration measurement by dynamic olfactometery in Europe,* J. Air & Waste Manage. Assoc. 49, 705-715, ISSN 1047-3289, June 1998

Measurement and Regulation of Odors in the USA

Thomas Mahin
Massachusetts Department of Environmental Protection
205a Lowell Street
Wilmington, Massachusetts 01887 USA

Keywords
Regulations, United States, measurement

Abstract

This paper will present highlights of the current approaches used in the USA relative to odor regulations and guidelines. The issue of odor standardization has progressed significantly during the last few years. In the USA, the Air & Waste Management Association's EE-6 Odor Committee has forwarded its guidelines to the American Society of Testing Materials (ASTM) as a suggested replacement for ASTM Method E679-91. Among other things, the guidelines recommend a minimum flow rate of 3 liters per minute (lpm) for olfactometers. However a large number of odor laboratories in the USA have adopted the European Standard approach of a 20 lpm flow rate. The author asks whether current olfactometry based odor regulatory standards in the USA standards will now be inconsistent with the higher D/T (OU) levels that may be associated with the higher flow rates used as part of the European Standard approach?

1. Introduction

Odors are increasingly the cause of complaints to environmental regulatory agencies in the USA. One reason for this increase is the fact that more homes are being built near waste processing facilities such as wastewater treatment plants and landfills due to a lack of buildable land. Also as home prices have risen significantly in recent years, many residents have become less tolerant to even occasional odors or other nuisance conditions that are perceived to have an impact on property values. In addition, in agricultural areas of the USA there has been a dramatic increase in corporate large-scale confined animal feeding operations. Because most of these animal facilities do not really have significant odor treatment systems in place, there has been a significant increase in complaints and regulations relative to animal feeding operations in the USA.

2. Types of Odor Regulatory Approaches Used in the USA

There are generally a number of different approaches that are commonly used in the USA to regulate odors.

(1) The use of ambient air limits for individual compounds such as hydrogen sulfide as used in the state of Minnesota (see Table 1 below). The existence of so many different odorous compounds associated with WWTPs and particularly most

livestock operations creates serious potential problems when using individual compounds as the basis for assessing odors. In addition, detection and odor annoyance thresholds cited in the literature and in regulations vary widely for compounds such as hydrogen sulfide.

Table 1. Examples of Ambient Standards for Odor Causing Compounds
(all agencies listed are state agencies unless otherwise noted) *from Mahin, 2001 (1)*

Location	Compound	Ambient Odor Standard
California	Hydrogen sulfide	30 ppbv* (1-hour average)
Connecticut	Hydrogen sulfide Methyl mercaptan	6.3 ug/m^3 2.2 ug/m^3
Idaho	Hydrogen sulfide	10 ppbv (24 hour average) 30 ppbv (30 min. average)
Minnesota	Hydrogen sulfide	30 ppbv (30 minute average)** 50 ppbv (30 minute average)***
Nebraska	Total reduced sulfur	100 ppb (30 minute average)
New Mexico	Hydrogen sulfide	10 ppbv (1 hour avg.) or 30 - 100 ppbv (30 minute avg.)
New York State	Hydrogen sulfide	10 ppbv (14 ug/m^3) 1-hour average
New York City	Hydrogen sulfide	1 ppbv (for wastewater plants)
North Dakota	Hydrogen sulfide	50 ppbv (instantaneous, two readings 15 min. apart)
Pennsylvania	Hydrogen sulfide	100 ppbv (1 hour average) 5 ppbv (24 hour average)
Texas	Hydrogen sulfide	80 ppbv (30 minute avg.) - residential/commercial & 120 ppbv - industrial, vacant or range lands

* - parts per billion by volume
** - not to be exceeded more than 2 days in a 5-day period
*** - not to be exceeded more than 2 times per year

(2) General regulatory language that prohibits off-site nuisance or annoyance conditions as determined by field inspectors in response to complaints from the public. Some agencies have implemented procedures whereby inspectors rate the intensity of the odor in the field, based on an intensity scale. Six point scales are sometimes used with 1 = very weak, 2 = weak, 3 = distinct, 4 = strong, 5 = very strong and 6 = extremely strong. The advantage to this approach is its simplicity and the fact that it is not a theoretical value predicted by a model. One disadvantage for both this approach and the hydrogen sulfide hand-held meter approach is that odor nuisance conditions occur much more frequently in the evening and early morning when regulatory staff are usually not working.

(3) Off-site limits based on levels predicted by dispersion modeling and using the dynamic olfactometry approach with the criteria reported as odor units (OU),

OU/m^3 or dilutions/threshold (D/T). The terms D/T, OU/m^3 and OU will be used interchangeably in this paper since they all represent the same concept (see Table 2 below).

(4) Best available control technology (BACT) or similar approaches that specify required levels of odor treatment controls for new or upgraded large facilities.

(5) The American Society of Agricultural Engineering (ASAE) document Engineering Practice 379.1 "Control of Manure Odors" recommends setbacks from livestock facilities of 0.4 to 0.8 km for neighboring residences and 1.6 km to residential development (2).

Table 2 Examples of OU/m3 (D/T) Limits Used *from Mahin (1)*

Location	Off-site standard or guideline	Averaging times
Allegheny County Wastewater Treatment Plant (WWTP)	4 D/T (design goal)	2-minutes
San Francisco Bay Area Air Quality District	5 D/T	Applied after at least 10 complaints within 90-days
State of Colorado	7 D/T (Scentometer)	
State of Connecticut	7 D/T	
State of Massachusetts	5 D/T*	
State of New Jersey	5 D/T **	5-minutes or less
State of North Dakota	2 D/T (Scentometer)	
State of Oregon	1 to 2 D/T	15-minutes
City of Oakland, CA	50 D/T	3-minute
City of San Diego WWTP	5 D/T	5-minutes
City of Seattle WWTP	5 D/T	5-minutes

* draft policy and guidance for composting facilities

** for biosolids/sludge handling and treatment facilities

The European Committee for Standardization or CEN has developed a standard method for odor laboratory measurement using olfactometry. The standard, which is to be called "Air Quality – Determination of Odour Concentration by Dynamic Olfactometry" will be referred to in this paper as the "European Standard" (3). In the USA, several universities and WWTP districts follow the European standard's basic tenets including: Duke University, Iowa State University, the University of Minnesota, Purdue University, the Los Angeles County Sanitation District and the Minnesota Metropolitan Council (4).

A study conducted for the California Air Resources Board (USA) included the review of six published studies that related to recognizability, unpleasantness and annoyance associated with a variety of unpleasant odors. The analysis concluded that for unpleasant odors the threshold of annoyance is at approximately five times the threshold of detection (5). California's South Coast Air Quality Management District's states that at 5 D/T (OU/m^3) people become consciously aware of the presence of an odor and that at 5 to 10 D/T odors are strong enough to evoke registered complaints (6)(7). It should be pointed out that there are questions as to whether these assumptions are still valid given the apparent increased sensitivity of the European Standard laboratory methods compared to ASTM Method E 679-91 (8). Given the background OU/m^3 levels commonly reported and because of the residual odor associated with Tedlar and similar bags, the olfactometric approach should not be used for ambient air odor analysis but rather for impact predictions using dispersion modeling.

3. Air & Waste Management Association Guidelines for Odor Sampling and Measurement

A subcommittee of the EE-6 Odor Committee of the Air and Waste Management Association (A&WMA) was formed to develop a set of guidelines or recommended practices for the standardization of odor sampling procedures and odor measurement techniques by dynamic dilution olfactometry. The A&WMA EE-6 Subcommittee on the Standardization of Odor Measurement prepared a document titled Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry August 23, 2002 (9). The EE-6 Odor Committee has submitted the Guidelines to the ASTM as a more detailed odor testing replacement method for the current ASTM method E679-91 (Standard Practice for Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series Methods of Limits) (8).

The method accepts the use of forced choice or non-forced choice sample presentation method in an ascending concentration triangular method (one diluted odor sample and two blanks per presentation) or a binary method (one diluted odor sample and one blank per presentation). To reduce the variability obtained, the guidelines recommend that panelists also indicate their basis for the choice: pure guess, possible difference or recognize the presence of an odor.

The guidelines recommend that the flow rates of the olfactometer should be calibrated regularly using a primary volume-measuring device (i.e. soap bubble flow meter). To obtain consistent and accurate values, the flow rates of both the dilution (odor-free) air and the sample flows should be measured at all delivery settings several times and averaged to ensure stability.

The guidelines state that screening for detection of n-butanol and at least one other odorant should be conducted using aqueous solutions. Initially, a sub-threshold concentration of the selected odorant in distilled water is compared to two bottles containing only distilled, odor-free (triangular presentation) water. The candidate is asked to pick the bottle containing the odorant. A series of similar triangular presentations are made in an ascending series with the odorant concentrations doubling at each step.

The second screening procedure involves familiarization of the potential candidates with the olfactometric procedures and determines each individual's detection threshold for: a standardized concentration on n- butanol and an odor sample or prepared standard representative of the specific project.

The screening samples should be run in triplicate. To be accepted as a panelist, the geometric mean of the individual detection thresholds should be within 0.5 and 2 times the accepted reference value for the reference material used. After all panelists have evaluated a series of dilutions for the test sample, individual panelists' best estimate thresholds (BET) are determined. The BET for a panelist is the geometric mean of that dilution level (or equivalent concentration) at which the first point (highest dilution level) of a consistently correct series of (+) responses (with some degree of certainty) and the dilution level prior to this point. All responses indicated by the panelists as being guesses are disregarded.

3.1 Olfactometer Flow Rates

The guidelines state that the airflow rate from the olfactometer sniff ports must be regulated at a **minimum** of 3 liters per minute (lpm) to account for the variability of individual breathing/sniffing volumes and techniques during olfactory evaluations. The resultant face velocity at the cup face should be between 1 -10 cm/sec.

In the effort to reach international consensus on the standardization of odor measurement techniques, flow rate has probably been the most controversial issues (10). An earlier draft version of the EE-6 Odor Committee guidelines recommended a flow rate of 8 lpm (11). The final version includes a minimum flow rate but no maximum so that the 20 lpm flow rate used in the European Standard approach would still be consistent with the guidelines.

The guidelines also state that smelling chambers should be a cylindrical shape or an ergonomically shaped nasal mask and must be made out of a non-reactive, odor-free material (glass or Teflon). The cup design must allow for an even flow profile at the face of the cup. The diameter of the chambers must be between 5 and 10 cm to allow full insertion of the panelists' nose into the chamber and result in a face velocity that is barely perceptible by the panelists. Note: high flow rates and high face velocities may result in notable discomfort of the panelists.

3.2 Odor Sample Collection

The guidelines state that odor samples should be collected using a sampling line made of an odor-free, chemically inert and non-reactive material (i.e. Teflon or similar). The samples should be collected into gas sampling bags made of Tedlar. This material has been specified because it is the best at maintaining sample integrity and has the lowest background odor. New bags should be purged with odor-free air prior to use to ensure that there is no contamination due to manufacturing “bag” odor. This is especially critical with the collection of low level or ambient odor samples.

Re-use of sampling bags may be possible with low odor (i.e. less than 50 D/T) samples. Pre-used bags should be purged continuously with odor-free air for a minimum of 24 hours and tested to ensure that they are acceptable prior to re-use.

The sample bag must be half filled at least once and emptied prior to collecting the final sample in order to precondition the sampling line and the interior walls of the sampling bag. The guidelines state that if pre-dilution of the sample is necessary due to an excessively high odor level, high temperature, or high humidity of the sample gas, pre-conditioning of the sample bag with the diluted sample is also required.

The sampling train should allow for transfer of the gas through the sampling line directly into the sample bag without going through any potential sources of contamination such as rotameters, pumps etc. The recommended method for sample collection is the “evacuated drum” or “sampling lung” where the sample bag is placed within a rigid, leak-proof container. The air inside the container is evacuated using a pump, which causes the bag to fill with sample at a rate equal to the container evacuation rate. Pre-dilution of the sample may be required to prevent condensation in the bag if the sample gas contains a significant amount of moisture

4. Conclusions

- The issue of odor standardization has progressed significant during the last few years. The CEN European Standard has become the official olfactometry odor analysis approach for a number of countries. In the USA, the A&WMA EE-6 Odor Committee has forwarded its guidelines to the American Society of Testing Materials (ASTM) as a suggested replacement for ASTM Method E679-91. In addition, an interlaboratory comparison of seven olfactometry laboratories was conducted in Japan in late 2000 (12).
- The A&WMA guidelines are similar to the European Standard but they do allow quite a bit of flexibility in what olfactometer flow rates cab be used. This could potentially be a problem when attempting to compare data and results from different olfactometry laboratories.
- With the A&WMA guidelines now final, an important issue needs to be analyzed in the future. Current OU/m^3 (D/T) odor regulatory standards in the USA have traditionally been based on lower olfactometry flow rates used in the past. Will these regulatory standards now be inconsistent with what are believed by some to be the higher D/T (OU) levels associated with the higher olfactometric flow rates associated with the European Standard? There appears to be a need for studies in the future that would compare results from analysis of odor samples using varying olfactometry flow rates.

References

1) Mahin, T.D., “Comparison of Different Approaches Used to Regulate Odors Around the World”, *Water Science and Technology,* Vol 44 No 9 pp 87-102

2) Heber, A.J. (1997) Setbacks for sufficient swine odor dispersion and dilution. Livestock Symposium, http://pasture.ecn.purdue.edu/~heber/setba.htm

3) CEN, prEN 13725 Air Quality – Determination of Odour Concentration by Dynamic Olfactometry

4) Mahin, T.D., Pope, R. and McGinley, M., When is a smell a nuisance? (2000) *Water Environment & Technology,* **12** (5) pp. 49-53.

5) Amoore, J.E., A matching-standards method for evaluating odor annoyance. (1991).In:

Proceedings of *Recent Developments and Current Practices in Odor Regulations, Controls and Technology.* Air& Waste Management Association Specialty Conference.

6) Amoore, J.E., The Perception of Hydrogen Sulfide odor in Relation to Setting an Ambient Standard, (1985), Prepared for the California Air Resources Board
7) South Coast Air Quality Management District (1993). California Environmental Quality Act (CEQA) Air Quality Handbook. .
8) ASTM, Standard Practice for Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series Method of Limits, E 679-91. Annual Book of Standards, American Society for Testing and Materials, Philadelphia, PA.
9) AWMA EE-6 Subcommittee on the Standardization of Odor Measurement, Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry, August 23, 2002
10) O'Brien, M.A., Duffee, R., and Ostojic, N., Effect of Sample Flow Rate in the Determination of Odor Thresholds, (1995), Proceedings of the Indoor and Environmental Air Specialty Conference, Bloomington, MN, Air and Waste Management Association.
11) O'Brien, M.A., Standardization of Odor Measurement Techniques, (1994), Proceedings of the 87th Annual Meeting of the Air and Waste Management Association.
12) Higuchi, T., Masuda, J. and Hayano, A., Establishment of Quality Control Framework for Olfactometry in Japan (2002), Proceedings of the WEF Odors and Toxic Emissions 2002 Specialty Conference.

The Regulation and Measurement of Odor in Korea

Sang Jin Park
Dept. of Civil & Environmental Engineering Woosong University,
Jayang-dong Dong-gu Daejeon Korea 300-718

Keywords
Odor measurement, Odor policy, Odor prevention law

Abstract

Recently, the number of civil petitions on odor has soared, and a great deal of attention has been paid to odor in Korea. This paper reviews the status and management of odor in Korea and introduces the Direct Sensory Method, Air Dilution Sensory Method, and the Instrument Analysis Method as measurements applied in Korea. Secondly, this paper introduces regulation criteria on the two sensory methods and permission level of odor release of eight malodorous chemical compounds and lastly, odor prevention laws to be passed in the near future.

1. Odor status in Korea

In addition to noise through the human olfactory system, odor has been a major pollution element with many civil petitions filed as an environmental pollution index. Every year the number of civil petition cases related to odor in Korea increases (see Figure 1). It is reported that more than 2,760 civil petition cases among 1,626 manufacturing plants have been filed. The major cause of civil petition is that residential and manufacturing areas are in close vicinity, and consequently it has caused large manufacturing plants to restructure their policies(1).

The Ministry of Environment inspected approximately 45,805 order-emitting companies in 1999, prosecuted 790 companies, and imposed administrative measures such as facility renovation, suspension of businesses, and fines on law violation(2). The Ministry of Environment conducts inspection of odor-emitting companies annually, and 526 companies under stricter government-monitoring at least three times a year. The Environmental Protection Agency takes action and reduces odor through technical support for small and medium-sized companies and making a database of odor-emitting facilities, deodorization fuel, and odor-victimized areas.

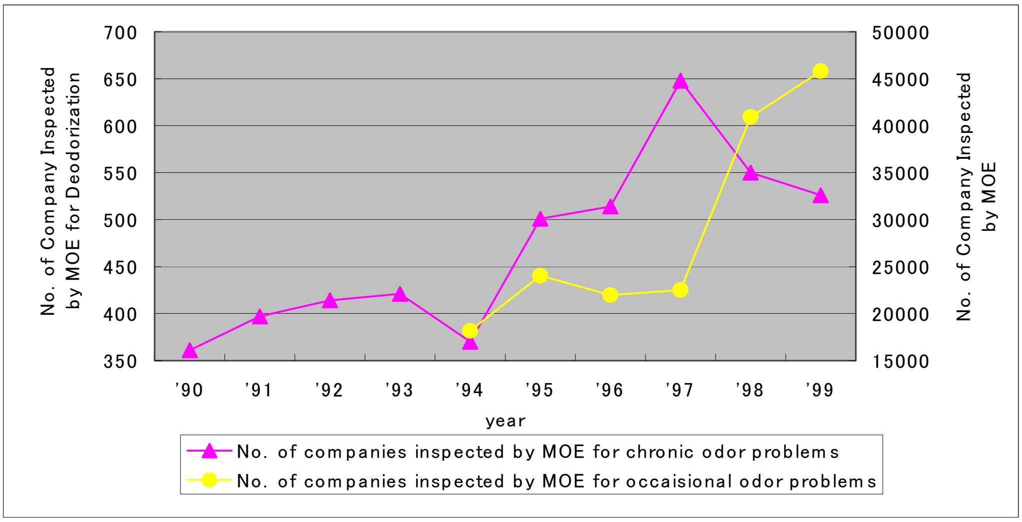


Figure 1. No. of odor emitting companies sighted by the MOE for odor prevention

2. Odor measurement in Korea

There are three major methods of odor analysis: direct sensory test, air dilution method, and chemical compound of analysis as shown in Tables 1 and 3. The actual spot odor analysis should use the direct sensory test or the air dilution method at the boundary of companies including the enclosures. It is regulated that the researcher could use chemical compound analysis using GC or UV as well as the two former tests, in case the eight odorous substances are contained among odor samples as defined in the atmospheric and environmental protection law(3).

The direct sensory test has been a major measurement. However, the sensory test using air dilution method has been welcomed recently by researchers as a measurement to identify the cause of odor more precisely. The direct sensory test was not effective in analyzing the cause of odor in wide-open spaces of industrial complexes and outlets, which cause highly-intensified odor.

The measurement and regulation of odor in Korea has been conducted in two places: outlets including stack and boundaries of companies including enclosures. (1) It can be conducted within outlets including stack and boundaries of companies including enclosures if the stack height is over 5m. There are other causes of odor besides outlet including stack in the industrial area if the emitted odorous substances are ammonia, hydrogen sulfide, and trimethyl amine. (2) It can be conducted only in outlets including stack if there are no other causes of odor. (3) It can be conducted only in boundaries of companies including enclosures besides the two cases mentioned above.

Table 1. Direct sensory test method

Intensity (degree)	Status
0	None
1	Threshold
2	Moderate
3	Strong
4	Very strong
5	Excessively strong

Table 2. Procedure for air dilution sensory test method in Korea

- ▸ Sampling
  - 3 ~ 20Liter/ less than 5minutes,   - use the teflon sampling bag
- ▸ Prepare odor free air
- ▸ Panelist screening test
  - test the olfactory sensibility using 4 standard odors
  - more than five persons
  - wait for the test for 10 minutes
  - stop the negative effective activity of the test
- ▸ Perform the sensory test
  - prepare diluted odor samples using odor free air (dilution ratio 3, 10, 30, 100, 300 times, etc …) by the method of descending series
  - prepare odor samples which consist of 2 bags filled with odor-free air and 1 odor-injected bag and keep the break period to maintain the olfactory sensibility after the first phase of the test.
- ▸ Calculation of odor using the sensory test results
  - calculate the odor concentration using the statistics equation, and disregard the extraneous data.

Table 3 Odor-containing compounds analysis using instruments

Compounds	Instruments
Ammonia	UV-Spectrophotometry
Hydrogen sulfides, Methyl mercaptan Dimethyl sulfide, Dimethyl disulfide	GC-FPD
Trimethyl amine	GC-FID
Acetaldehyde	GC-FID
Styrene	GC-FID

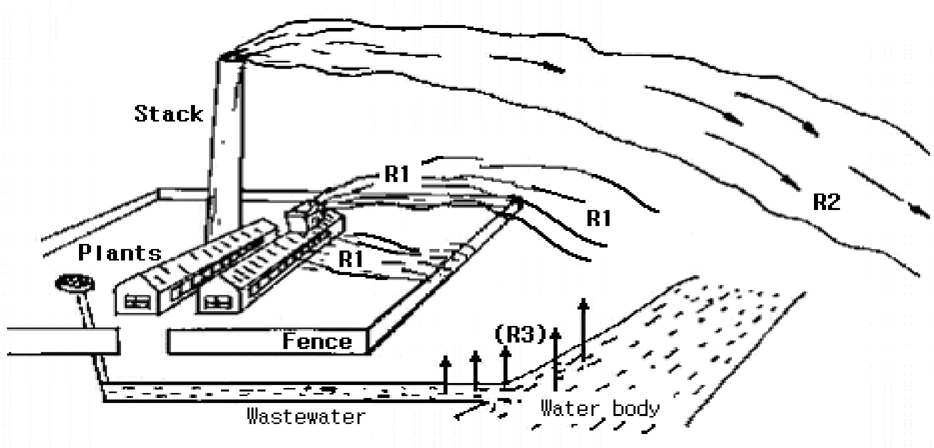


Figure 2. Schematic diagram of odor control strategy in Korea

3. Odor policy in Korea

3.1 Status of odor policy

As seen in Table 4, atmospheric and environmental protection laws (Chap. 3 Sec.30) classified eight odorous compound substances (e.g., hydrogen sulfide) in Korea according to the place of measurement.

Facilities for regulations are classified as outlet facilities of atmospheric pollutants (Preservation Law of Air Environment Article 2) and as facilities for control of odor in residential areas (Preservation Law of Air Environment Article 30). Outlet facilities of atmospheric pollutants consist of rubber and plastic product manufacturing plants, leather product manufacturing plants, industrial waste incinerators, painting mills, and petrochemical refinery plants. Facilities for the control of odor in residential areas constitute agricultural product wholesale markets, joint markets, butchery treatment areas, excretion treatment facilities, livestock farming waste treatment facilities and cleaning facilities. It is designated to other facilities, excluding the facilities mentioned above. This law and act prevent the establishment of waste incinerators and outdoor waste incinerators able to burn rubber, leather and synthetic resin in order to reduce the amount of odor (Preservation Law of Air Environment Article 29).

Table 4. Analysis and permission level of odor in Korea

Methods of odor analysis	Criteria for permission level of odor release		
Direct sensory test	Odor Intensity : less than 2 degree		
Air dilution sensory test	A. Outlets including stack (a) Companies in industrial area: less than 1000 OC (b) Companies in other area : less than 500 OC B. Boundaries of companies including enclosures (a) companies in industrial areas : less than 20 OC (b) companies in other areas : less than 15 OC		
Chemical compound analysis using GC or UV	Compounds	in industrial areas	in other areas
	Ammonia	less than 2 ppm	less than 1 ppm
	Methane ethiol	less than 0.004 ppm	less than 0.002 ppm
	Hydrogen sulfide	less than 0.06 ppm	less than 0.02 ppm
	Dimethyl sulfide	less than 0.05 ppm	less than 0.01 ppm
	Dimethyl disulfide	less than 0.03 ppm	less than 0.009 ppm
	Trimethyl amine	less than 0.02 ppm	less than 0.005 ppm
	Acetaldehyde	less than 0.1 ppm	less than 0.05 ppm
	Stylene	less than 0.8 ppm	less than 0.4 ppm

3.2 Legislation of odor prevention law (Draft)

As mentioned above, odor is a part of atmospheric pollution. Although atmospheric pollutants occur widely and consistently, there is a big difference as compared to other pollutions because it occurs in small areas and disappears instantly. There are many instances where various types of odorous substances occur, caused by the outlet facilities of atmospheric pollutant including various manufacturing processes. As a result, current atmospheric and environmental protection laws are highly limited to control over regulation of odor emission. Accordingly, odor prevention was separated from atmospheric and environmental protection on July 1, 2002. Odor prevention bills will be enacted and are as follows(4):

(1) Local communities in charge of odor management on behalf of the government: Since the scope of odor influence is not nationwide but regional, local communities should take charge of odor management and consider the needs of each area.
(2) Designation and management of problematic areas: The law designates the scope of problematic facilities or areas and effectively regulates odor emission by gradually expanding the designated odor management areas to industrial complexes and facilities making frequent civil petitions.
(3) Establishment and management of the permission level of odor release according to the characteristics of areas: Intensifying the criteria by the rules of city and province in cases where odor problems do not settle down on the permission

standard of odor release is in accordance with the Ministry of Environment (MOE)

(4) Application of regulation standards of odor emitting facilities: Regulating odor emitted from the processing and storing of products as well as odor emission facilities, excluding small businesses.

(5) Utilization of preliminary prevention measures for odor emitting facilities: Reducing odor emission by making designated odor emission facilities report odor prevention plans. The designated odor emitting facilities are decided by the MOE or by the rules of the city and province.

(6) Establishment of odor inspection institutions for the reliability and objectivity of measurements: Setting up odor inspection institutions for the development of measurement techniques, the improvement of inspection accuracy, and ruling out the application of permission level of odor release.

(7) Management of odor by the inspection of odor status on a regular basis: Having governors of local communities periodically investigate odors occurring in concentrations of designated odor compounds, status of civil petitions filed against odor problems, and report to the minister of MOE. Contents of odor inspection are to be decided by the ordinance of MOE.

References

1) Ministry of Environment, Environmental White Paper, 2000
2) Ministry of Environment, Investigation study to improve the management system of odor emission sources, 2001, p138
3) Ministry of Environment, Preservation Law on Air Environment Article 12 Annexed Chart 8 , 1999
4) Ministry of Environment, Major Contents of Odor Prevention Law (Draft), 2003

2. Application and Quality Control of Odor Measurement

A Comparative Study on Odor Regulation in Japan and Korea

Sung Bong Yang, School of Chemistry and Biological Sciences
University of Ulsan, Ulsan, 680-749, Korea

Keywords
Offensive Odor Control Law, Korean, Japanese

Abstract

Odor control policy in Japan has been based on the Offensive Odor Control Law, which regulates the emission of offensive odors generated from business to prevent a nuisance occurring. In Korea, odor control has been under the Air Conservation Law at present. But the Korean Offensive Odor Control Law, newly prepared to be enacted this year, strengthening the regulation of emission of odor, have common aspects of the Japanese law. Both laws describe the standards of maximum permissible concentration of specified offensive odor substances and a multiple of dilution of odor at border of business or emission source in regulation area. The Japanese rule has been promulgated since early 1970's and developed to be more ideal but complicated. In other hand, the current Korean rule is very simple, but new regulation is expected to be similar as that of Japan.

1. Introduction

In Korea, the offensive odor is defined as the odor of such as hydrogen sulfide, mercaptans, amines and other irritant gas that cause unpleasantness and repugnance, and it has been treated as the sensory pollution like noise and vibration and categorized in air pollution (1). The early industrialized nation, Japan, coped with this problem and promulgated Offensive Odor Control Law in 1973 to abate odor nuisance. Later than Japan, Korea had been industrialized in 1990's and public complaints like offensive odor have become a social problem in heavily industrialized area, such as Ulsan and Yeocheon located in the southern coast of the Korean peninsula. In 1993, Korea enacted the limit of concentrations of the offensive odor substances at the boundary of the site, that was very similar to the Japanese Offensive Odor Control Law amended in 1978, and the limit of dilution factor of the offensive odor at the point of emission from facilities, similar to the state or city ordinance for the prevention of offensive odor of many countries. Measurement and regulation of odor in Korea and in Japan have very similarity but differences in many points. In here, difference and similarity of odor policy in two countries will be introduced.

2. Measurement method and regulation

6 stages of odor strength are the standard criteria for regulation of odor at the boundary of factories or other places of business. Table 1 shows the current Korean law and the Japanese amended in 1976 for offensive odor. Both countries use the sensory method for offensive odor and instrumental analysis for odorous compounds to estimate the odor strength. In Korea, direct sensory measurement at the boundary

of business has been used for judgment of illegality of odor emission till now. Therefore a person establishing a place of business shall observe the permissible limit of odor, meaning maximum emission of odor or odorous substances from the boundary of business or point source. In Japan, direct sensory method is not used for regulation but the primary investigation of offensive odor.

Table 1 Comparison of the current Korean permissible limit for offensive odor and the Japanese offensive odor control law promulgated in 1976

Measurement Method			Korean permissible limit for odor		Japanese offensive odor law in 1976	
Direct Sensory Method			Less than 2(odor strength)		None	
Sensory Method	Gas releasing port	Industrial	Maximum dilution factor	1000	Range of Concentration	500~1800
		Residential		500		300~500
	Border line	Industrial		20		20~30
		Residential		15		10~15
Instrumental Method	Specified offensive odor substance (ppm)		Residential Area	Industrial Area	Residential Area	Industrial Area
	Ammonia		2	1	2~5	1~2
	Methyl mercaptan		0.004	0.002	0.004~0.01	0.002~0.004
	Hydrogen Sulfide		0.06	0.02	0.06~0.2	0.02~0.06
	Methyl Sulfide		0.05	0.01	0.05~0.2	0.01~0.05
	Dimethyl Sulfide		0.03	0.009	0.03~0.1	0.009~0.03
	Trimethyl amine		0.02	0.005	0.02~0.07	0.005~0.02
	Acetaldehyde		0.1	0.05	0.1~0.5	0.05~0.1
	Styrene		0.8	0.4	0.8~2	0.4~0.8

A multiple of dilution (called as "odor concentration" in Japan and "dilution factor" in Korea), where the gas has been diluted until an offensive odor is no longer detectable to the human sense of smell, is described as the standards for odor regulation of the boundary and the emission point. Both countries differentiate the level of regulation according to the population and usage of land. In Korea, 1,000, the allowable highest dilution factor at gas releasing port of the factory established in industrial area is the substantial regulation of offensive odor. In Japan, each prefecture has his regulation standards for each regulation area within the range stipulated by the government. 8 specified offensive odor substances declared in the permissible odor emission standard enacted in the Korean air conservation law in 1993 were equal to those of the Japanese Offensive Control Law amended in 1978. The current Japanese Offensive Control Law (2) describes 22 specified substances for the regulation at boundary of a business. Although the Japanese law states the range of concentrations of specified odor substances, each prefecture applies the most severe standard to each regulation area. Regulation standards at the border of business in industrial area are equal to 3.5 in 6 stages of odor strength and in residential area 2.5 in 6 stages.

3. Standard odors for panelist and manipulation of data

Table 2 shows the standard odor liquid for selection of the panel recognized to have normal smell function. Chemicals used in Korea are easily obtainable but the Japanese are used for otolaryngology.

Table 2 The standard odors of sensory test for panelist in Korea and Japan

In Korea		In Japan	
The Standard odors	Concentration(wt%)	The Standard odors	Concentration(ppm)
Acetic acid	1.0	i-Valeric acid	30
		Skatol	10
Trimethyl amine	1.0	Methyl cyclopentenolone	30
		β-Phenyl ethyl alcohol	100
Phenol	0.1	γ-Undecalactone	30

Calculation method of a multiple of dilution called as the odor concentration in Japan and also the dilution factor in Korea differentiates the Offensive Odor Control Law of Japan and the permissible odor limit of Korea. Table 3 shows each calculation method of a dilution multiple for the odor sample obtained from an emission point. In Japan, at least 6 members of panelists are needed for judgment of odor concentration but in Korea at minimum 5 panelists are needed. In both countries, data of maximum and minimum must be excluded from calculation. Judgment of existence of smell by each panel is done with triangle comparison method of odor bag in both countries. The odor concentration calculated by the Japanese method is 500 but the dilution factor by the Korean is 448 in this table.

Table 3 Example of sensory test on odor sample at gas releasing port

Multiple of dilution		30	100	300	1000	3000	10000	Threshold value of each panel		Exception of Max. and Min.
Algebraic value		1.48	2.00	2.48	3.00	3.48	4.00	Japan	Korea	
Panel	A	/	○	X				2.24	100	Exception
	B	/	○	○	X			2.74	300	
	C	/	○	○	○	○	○	3.74	10000	Exception
	D	/	○	○	X			2.74	300	
	E	/	○	X				2.24	100	(Exception)
	F	/	○	○	○	X		3.24	1000	

(Data of A and C were excluded from the Japanese calculation and data A, C and E from the Korean.)

- Permissible dilution factor of Korea : $\sqrt[3]{300\times300\times1000} = 448$
- Odor concentration of Japan : $10^{2.74} = 550$

448 is the geometric average of each threshold value and 2.74 is the arithmetic mean of threshold values of each panel.

4. Offensive Odor Control Law in Japan and Korea

Current Japanese regulation on odor consists of maximum permissible concentration of specified offensive odor substances and odor concentration. Table 4 shows the 22 specified substances regulated as odorous compounds at the border of a business defined in the Japanese law. Among them, 13 substances at a gas releasing port have their permissible concentrations and also maximum allowable concentrations of 4 substances in water are defined therein. 13 substances discharged from smoke stacks or other gas emission facilities are shown in table 5. This rule means higher stack can emit more odor in Japan. But in Korea, odor from all stacks in industrial area must be under 1000 of dilution factor. The Japanese odor policy has been developed since 1973, to be very complicated now. In Korea, the concept of offensive odor has not been prevailing and common and it makes the regulation not to be complicated.

Table 4 Specified odor substances regulated in the Japanese Offensive Odor Control Law

Subastance	Boundary	Gas emission	Water	Substance	Boundary	Gas emission	Water
Ammonia	○	○		i-Valeraldehyde	○	○	
Methyl mercaptan	○		○	i-Buthanol	○	○	
Hydrogen sulfide	○	○	○	Ethyl Acetate	○	○	
Methyl sulfide	○		○	MIBK	○	○	
Dimethyl sulfide	○		○	Toluene	○	○	
Trimethyl amine	○	○		Styrene	○		
Acetaldehyde	○			Xylene	○	○	
Propionaldehyde	○	○		Propionic acid	○		
n-Butyl aldehyde	○	○		n-Butyric acid	○		
i-Butyl aldehyde	○	○		n-Valeric acid	○		
n-Valeraldehyde	○	○		i-Valeric acid	○		

Table 5 Regulation standard for the flow rate or concentration of the specified odor substances at the point of emission from facilities at Chiba prefecture in Japan

Odor substance	Maximum flow rate at point source(N m^3/h)	Odor substance	Maximum flow rate at point source(N m^3/h)
Ammonia	$0.108 \times 1 \times He^2$	i-Valeraldehyde	$0.108 \times 0.003 \times He^2$
Hydrogen sulfide	$0.108 \times 0.02 \times He^2$	i-Butanol	$0.108 \times 0.9 \times He^2$
Trimethyl amine	$0.108 \times 0.05 \times He^2$	Ethyl Acetate	$0.108 \times 3 \times He^2$
Propion aldehyde	$0.108 \times 0.05 \times He^2$	MIBK	$0.108 \times 1 \times He^2$
n-Butyl aldehyde	$0.108 \times 0.009 \times He^2$	Toluene	$0.108 \times 10 \times He^2$
i-Butyl aldehyde	$0.108 \times 0.02 \times He^2$	×ylene	$0.108 \times 1 \times He^2$
n-Valeraldehyde	$0.108 \times 0.009 \times He^2$		

(He: Corrected height of the gas emission point)

Recently, the Ministry of Environment of Korea is planning the new policy for offensive odor and e×pected to enact the Korean Offensive Odor Control Law, involving more kinds of specified odor compounds at border and severe regulation of odor emission from point sources.

5. Conclusion

The Japanese Offensive Odor Control Law has been enacted in 1973 and developed to be ideal but complicated today. In late of 1900's Korea, an odor nuisance has increased drastically to be a social problem. Recently, Korean government have been considering a new policy for odor, and the Korean Offensive Odor Control Law, planned to be promulgated in this summer, will reduce the odor nuisance of Korea. Various odor control policies of industrialized countries (especially, the Offensive Odor

Control Law of Japan) have been under the investigation for establishment of the new Korean odor law.

References

1) The Korean Law of Air Reservation, Chapter 2, section 7, amendment, April 15, 1999
2) Offensive Odor Control Law of Japan (English version), http://www.env.go.jp/en/lar/olaw/index.html
3) Concepts of the standardization of 2nd regulation for offensive odor at Tokyo – new regulation standard for odor concentration at the point of emission from facilities –, Iwazaki, Y. et al, J. Odor Research and Eng. 2000, Vol. 33, pp.76 – 85(Japanese)
4) S. Yang, et al, “Study on investigation and control of the emission sources of odor substances”, Ministry of Environment, May 2001(Korean)

Quality Control of Olfactometry at SRI and in Europe

Robert Sneath,
Silsoe Research Institute,
Wrest Park,
Silsoe,
Bedfordshire
MK43 9JY

Keywords
Accreditation, repeatability, interlaboratory comparison, BSEN13725

Abstract

Quality of odour measurements is assured in the UK laboratories by following the European standard for “Determination of odour concentration using dynamic olfactometry”. The EN13725 is now the standard followed by all European countries for odour concentration measurements. The accuracy and repeatability of the measurements is assured by conforming to the quality criteria for accurate dilution equipment, compliance to the panel selection criteria and standardised data collection and data analysis methods.

1. Introduction

Until 1997 when a draft of the European standard for olfactometry was issued to the working group there was no single method for objectively measuring odour concentration. After an inter-laboratory comparison, committee members accepted that the draft method was suitable and most European laboratories adopted the use of the prEN13725 (now BSEN 13725 in the UK)(1). The European standard, EN13725, was ratified in late 2002 and Silsoe Research Institute Odour Laboratory has obtained Laboratory Accreditation to ISO17025 for “Determination of odour concentration using dynamic olfactometry” following the BSEN13725 (2).

In this paper the quality controls required are described and data from the measurement of environmental odours illustrate the benefits of the method in producing good repeatability.

2. Principle of measurement

The odour concentration of a gaseous sample of odorants is determined by presenting a panel of selected and screened human subjects with that sample, changing the concentration by diluting with neutral (odourless) gas, in order to determine the dilution factor at the 50% detection threshold ($Z_{50} \equiv \overline{Z}_{\text{ITE,pan}}$).
At that dilution factor the odour concentration is 1 ou_E/m^3 by definition. The odour concentration of the examined sample is then expressed as a multiple (equal to the dilution factor at Z_{50}) of one European Odour Unit per cubic metre [ou_E/m^3] at standard conditions for olfactometry (20C, 1013mbar).

2.1 The forced choice mode

The Silsoe Research Institute laboratory operate a forced choice dynamic olfactometer, (model OdourNet olf-n6) Fig. 1, it has two outlet ports from one of which the diluted odour flows and clean odour-free air (neutral gas) flows from the other(s).
The other choice mode allowed under the Standard is "Yes/No" mode where panel members respond "yes" to and odour and "no" when the odour is not detected in the air stream flowing from a single port. Odours of random dilution are presented interspersed with blanks.

In the forced choice mode, measurement starts with a dilution of the sample large enough to make the odour concentration below the panel members' thresholds, the concentration is increased by an equal factor in each successive presentation, and factor at SRI is between 1.7 and 1.8. The port carrying the odorous flow is chosen randomly by the control sequence on each presentation. The assessors sniff and indicate from which of the ports the diluted odour sample is flowing using a personal keypad. They also indicate whether their choice was a guess, whether they had an "inkling" or whether they were certain they chose the correct port. Only when the correct port is chosen and the panel member is certain that the choice was correct is it taken as a TRUE response. At least two consecutive TRUE responses must be obtained for each panel member. The geometric mean of the dilution factors of the last FALSE and the first of at least two consecutive TRUE presentations determines the individual threshold estimate (ITE) for a panel member. The odour concentration for a sample is calculated from the geometric mean of at least two ITEs for each panel member.

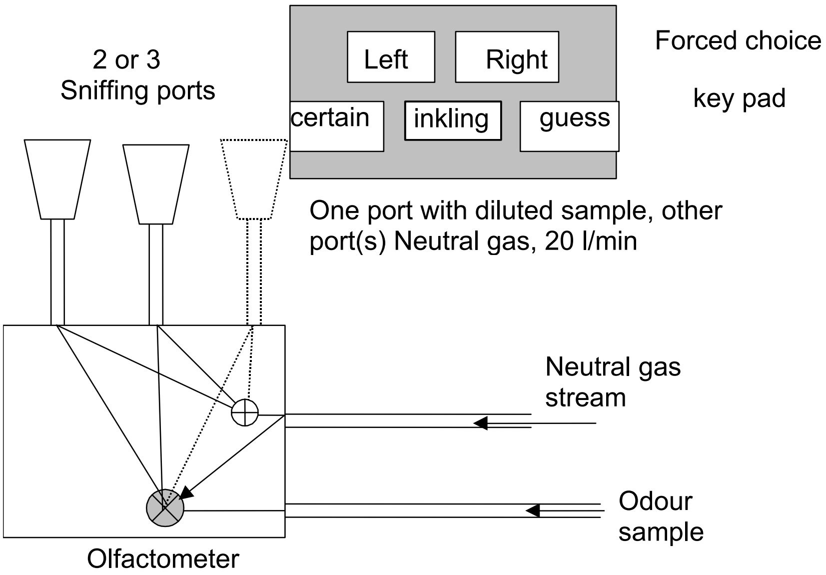


Fig 1 Schematic diagram of a forced choice olfactometer

For measurements on reference odorants this value can be converted to an individual threshold estimate expressed as a mass concentration using the known concentration of the reference gas divided by the ITE.

3. Laboratory Quality Control

3.1 Instrument quality criteria

The accuracy and repeatability of the dilution apparatus, the olfactometer, is the first step in quality control of olfactometry. The first important criterion is the accuracy of the dilution equipment. The criteria set out in the standard BSEN13725 are that the accuracy A_d of the dilution setting should deviate less than 20% from the required setting and the repeatability on setting that dilution ratio must be better than 5%. The ratio or step factor between the set points must be between 1.4 and 2.4. At SRI it is between 1.7 and 1.8

The Standard stipulates that a calibration should be done at least annually, however as a precaution the SRI quality assurance system ensures that a check on the calibration settings is carried out monthly. This is done using a Brηel & Kjaer 1302 gas analyser and sulphur hexaflouride as the tracer gas. Values recorded in these checks are compared with the values on the calibration certificate, if the values are outside the standard's criteria adjustments are made and previous results are checked and recalculated if necessary.

3.2 Assessor selection

The second key part of accurate odour measurement is the selection of the odour assessors who make up the panel. In order to select odour assessors *n*-butanol (butan-1-ol) has been chosen as the reference material. (It is recognised that a single component reference gas is not the ideal but no representative odorant mixture has yet been formulated.) Only people with a mean ITE for n-butanol in neutral gas of between 20 ppb and 80 ppb and a log standard deviation of less than 2.3, are acceptable, calculated from the previous 10 to 20 ITEs. These assessors are checked after no more than 12 regular odour measurements, the equivalent to calibrating a gas analyser, for their detection threshold and have to remain within these limits to be a panel member.

These selection criteria used at the Silsoe Research Institute laboratory leads to us rejecting about 43% of those tested because they are not sensitive enough and about 12% because they are too sensitive.
Selection of the panel members using the above method leads to the accuracy and precision to enable the laboratory to comply with the criteria set in the standard (EN 13725)

3.3 Laboratory conditions

The third feature that ensures quality measurement is the laboratory conditions and assessors behaviour. For laboratories to conform to the required standard, they must be guaranteed to be free from odour, at Silsoe we have an air-conditioned laboratory with activated charcoal filtration to ensure an odour free atmosphere. We also have a source of odour free air, neutral gas, again cleaned with an activated charcoal filter, with which to dilute the odour sample. The olfactometer, which is a dilution device, is made entirely from approved materials, glass, FEP, or stainless steel. Samples are processed the next day, within 30 hours of collection as dictated by the BSEN13725.

3.4 Quality criteria

The Standard is based on the following accepted reference value and shall be used when assessing trueness and precision:

$1\ \mathrm{ou_E} \equiv 1\ \mathrm{EROM} = 123\ \mu\mathrm{g}\ n\text{-butanol}$

When 123 μg *n*-butanol is evaporated in one m³ of neutral gas at standard conditions for olfactometry the concentration is 0,040 μmol/mol (40 ppb or a $\log_{10}$ value of 1.6)

Two quality criteria below are specified to measure the performance of the laboratory in terms of the standard accuracy and precision, respectively.

Accuracy reflects the trueness or closeness to the correct value, in this case the true value for the reference material is 40 ppb and the precision is the random error. The standard specifies how these two quality criteria are calculated.

The criterion for accuracy A_{od} (closeness to the accepted reference value) is

$\mathrm{A_{od}} = \leq 0{,}217$, at Silsoe this statistic is currently $A_{od} = 0.152$

In addition to the overall accuracy criterion, the precision, expressed as repeatability, *r*, shall comply with

$\mathrm{r} \leq 0.477$, currently $r = 0.283$

This criterion for repeatability can also be expressed as:

$10^r \leq 3.0$, currently $10^r = 1.92$

This laboratory repeatability implies that the factor that expresses the difference between two consecutive single measurements, performed on the same testing material in the laboratory will not be larger than 1.92 in 95% of cases.

3.5 Laboratory History

Data for establishing the above criteria are collected at least once per day of measurement; the laboratory history is displayed in Fig 2. The mean and standard deviation are plotted and all lie within the criteria. Minor variations can be seen these are caused by variations in the make up of the panel as well as day to day variations in individual's sensitivity. These records provide us with a continuous record of the quality status of our measurements.

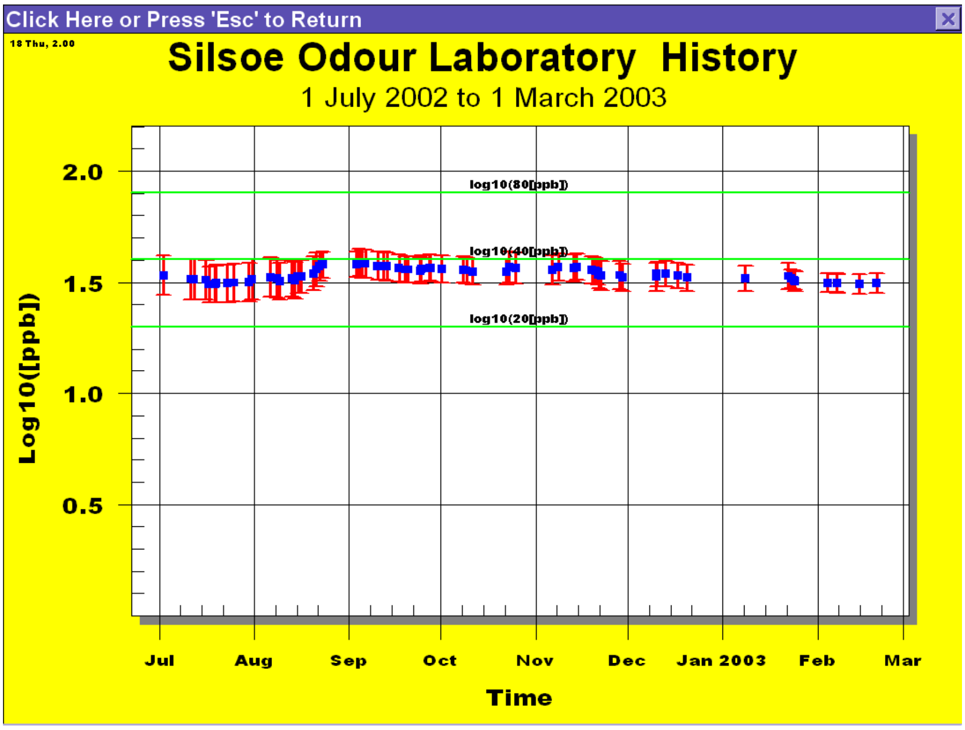


Fig 2 Record of the laboratory accuracy and precision with n-butanol.

4. Quality of measurements of environmental odours when the EN quality criteria are met

SRI laboratory repeatability calculated in 3.4 above implies that the difference between two consecutive single measurements, performed on the same testing material in the laboratory will not be larger than a factor 1.92 in 95% of cases.

However there are always questions about the applicability of this single reference odorant when the primary use of olfactometry is measurement of environmental odours that contain scores of odorants. Early this year we briefly investigated the repeatability by replicating measurements on samples reaching the lab; these are shown in Table 1. Although there may be questions about the transferability of assessor’s perception of *n*-butanol to their perception of real odours the results do show that the repeatability of measurements on real odours is better than predicted from the n-butanol data.

In order to comply with all the requirements of ISO17025 an accredited laboratory is required to participate in a “Round Robin” interlaboratory comparison, we have arranged for this to take place in spring 2003. The samples used in the comparison will be both the reference material, *n*-butanol, and environmental odour samples.

Table 1 Comparison of range of results from real odours and n-butanol.

Odour source	Measured values, $ou_E\ m^{-3}$	Mean, $ou_E\ m^{-3}$	sd	Maximum ratio between duplicate measurements
n-Butanol			(n=20)	1.92 (95%ile)
Pig feed 1	11250			
	12304			
	16633	13124	1.22	1.47
Pig feed 2	6988			
	6637			
	7659	7082	1.07	1.15
Restaurant	751			
	997	865		1.32
Restaurant	789			
	806	797		1.02
Restaurant	997			
	1324	1149		1.32

5. Conclusions

(1) The European Standard BSEN13725 provides a basis for quality assurance for measurement of odour concentration.

(2) Key quality criteria are

- Accurate dilution apparatus
- Rigorous selection of the odour assessors
- Carefully controlled laboratory conditions

(3) Measurement results on real odours show the level of repeatability to be better than with the *n*-butanol reference material

References

1) BSEN 13725 Air quality – Determination of odour concentration by dynamic olfactometry 2002 BSI, 389 Chiswick High Road, London, W4 4AL, +44 (0) 20 8996 9001, www.bsi.org.uk

2) ISO17025 (1999) General requirements for the competence of testing and calibration laboratories.

A Comparative Study of Japanese and European Olfactometry Standards

Hiroyuki Ueno, Masato Higuchi, Sukehisa Tatsuichi and Yoshiharu Iwasaki
Tokyo Metropolitan Research Institute for Environmental Protection
1-7-5 Shinsuna, Koto-Ku, Tokyo, 1360075, Japan

Keywords
Olfactometry, Triangle odor bag method, Dynamic olfactometer

Abstract

This report provides a comparative study between the odor measurement method by olfaction, which has been adopted as the Offensive Odor Control Law in Japan and the dynamic olfactometry, which has been standardized in Europe. Dilution accuracy, panel selection and odor measurements have been compared in this study. As a result of the dilution test with three standard odorants, a 46% decrease in the concentration of hydrogen sulfide at a high dilution ratio has been observed using the olfactometer, while the Japanese method has shown good performance. Twenty people have been given both screening tests. Eighteen people have passed the Japanese test, while only seven people have passed the European test. In the odor measurement of three standard odors and six actual source samples, if the panel is the same, the results of both methods have corresponded well.

1. Introduction

The method of measuring odor concentration by sniffing samples diluted with odor-free air is common worldwide. The triangle odor bag method is adopted as an olfactometry standard in the Offensive Odor Control Law in Japan. However, the dynamic olfactometry1) has been standardized in Europe and there is a possibility it might become the international standard in the future. Therefore, it is necessary to conduct a proper comparative study of both methods in view of such progress. The following differences exist between the Japanese and the European methods, although the aim of both methods is the same, which is to determine the olfactory threshold by sniffing diluted odor samples.

Dilution Method: Although odor samples are diluted with odor-free air using bags and syringes in the triangle odor bag method, a dynamic olfactometer continuously dilutes samples using a compressor and flow controllers, etc.

Presentation of Samples to Assessors: A diluted series is presented in descending order of stimuli in the triangle odor bag method and the step factor is 3. In the European method, the ascending method is used and the step factor is 2. The sniffing conditions are different; one sniffs the air in the bag and the other sniffs the air that emanates from a port.

The Panel Screening Test: In the Japanese method, the test is performed with five standard odorants to exclude dysosmias. In the European method, panel members who have sensitivity to n-butanol within a certain range are selected.

In this report, we describe the results of comparative experiments on these points.

2. Comparison of both methods

2.1 Dilution accuracy

The European Method recommends CO as a tracer gas for calibrating the diluting apparatus. However, some actual odorants tend to be adsorbed on the surface of certain materials, therefore the dilution accuracy might not be the same as that of CO.

Three odorants, m-xylene, 100ppm; n-butanol, 100ppm; and hydrogen sulfide, 10ppm, were then diluted to concentrated levels of the olfactory thresholds by each method and the concentrations of the odorants in the diluted gases were analyzed.

The dilution system of the olfactometer depends on the device. The olfactometer used in this study (Olfactomat-n2, Project Research Amsterdam B.V Netherlands) dilutes the sample gas with odor-free air by controlling the gas flow with mass flow controllers and fixed valves. The diluted sample gas emanates at 20L per minute from the sniffing port.

In the dilution procedure of the triangle odor bag method, first, odor-free air is filled in a 3L odor bag. A certain amount of an original sample is then injected into the bag with a glass syringe.

0.5-2L of the diluted gas is concentrated with liquid oxygen and then injected into a gas chromatograph (HP5890). The coefficient of variation of the analysis ranged from 1 to 4%, when 0.5L of the three standard gases was concentrated and analyzed five times.

The error (%) shown in Table 1 indicates the bias of the actual dilution factor relative to the theoretical dilution factor. In the case of the olfactometer, it was not more than 13% for m-xylene. However it was -23% after being diluted 3,543 times for n-butanol, and -46% after being diluted 10,467 times for hydrogen sulfide. The error for hydrogen sulfide tends to increase as the dilution factor becomes higher. It was also observed that the actual concentration of diluted gas tends to be lower during the first dilution operation for n-butanol and hydrogen sulfide. The results indicate a possibility of the actual concentration being lower than the setting value at a higher dilution factor for some odorants. On the other hand, the error for the same three odorants was 12% less in the odor bag method. Incidentally, the odor bags used were manufactured larger than the original size; therefore some checks are necessary on the product of each manufacture.

Table 1 Dilution test results (n=3)

	Olfactometer				Odor bag		
m-Xylene							
Dilution factor	3543	1672	870	492	3430	1140	343
Theoretical concentration (ppb)	30.8	65.2	125	222	31.8	95.3	318
Actual concentration (ppb)	33.8	67.8	142	232	29.4	87.1	306
Error (%)	10	4.0	13	4.5	-7.6	-8.7	-3.6
n-Butanol							
Dilution factor	3543	1672	870	492	3430	1140	343
Theoretical concentration (ppb)	16.5	34.9	67.1	119	17.0	51.1	170
Actual concentration ppb)	12.6	32.4	67.8	125	15.3	47.8	151
Error (%)	-23	-7	1	5	-10	-6	-12
Hydrogen sulfide							
Dilution factor	10467	6494	3543	1672	11400	3430	1140
Theoretical concentration (ppb)	0.955	1.54	2.82	5.98	0.875	2.92	8.75
Actual concentration (ppb)	0.513	1.06	2.29	5.57	0.899	2.91	8.34
Error (%)	-46	-31	-19	-6.9	2.8	-0.4	-5

Note: Volume of odor bag used was 3.43L.

Error (%) = (Actual concentration–Theoretical concentration)/ Theoretical concentration・100

2.2 Panel Screening Test

The same assessors were examined by each panel screening test on the same day, and the results were compared.

The outline of each screening procedure is as follows:

The Japanese method: Five standard odor solutions, which are prepared by dissolving β-phenylethyl alcohol, methyl cyclopentenolone, isovaleric acid, γ-undecalactone, and Scatorl in odor-free liquid paraffin, are used for the screening. The test is carried out using odor-free paper by a 5-2 method. Assessors who can distinguish two of the papers which were soaked in the standard solution form the other three papers soaked in the odor-free solution for all of the five odorants can be a panel member. The concentrations of the standard solutions are set at the point of 1.5 times the standard deviation from the mean value based on the olfactory threshold distribution of Japanese people. In this study, the individual threshold values for the five odorants were measured using lower concentration solutions.

The European method: Assessor selection is based on their individual sensitivities and variability for n-butanol. At least ten individual threshold values for each assessor are measured in at least three sessions on separate days with a pause of at least one day between sessions. The antilog of the standard deviation expressed as log (ppb) should be less than 2.3, while the geometric mean should be between 20 and 80 (ppb). In this study, measurement of the threshold by the olfactometer has been carried out in conformity with the European method. Assessors are presented with two ports and choose which of the ports with stimulus, and indicate their certainty: certain, guess, or inkling. Presentations are done in ascending order and continued until at least two consecutive TRUE responses (correct and certain) are collected. The individual threshold is determined by the geometric mean of the dilutions at which odor is detected and the preceding higher dilution.

There were twenty assessors between the ages of 18 and 63 years old examined in this experiment. Each assessor participated in the tests for three non-consecutive days. The individual threshold measurement for each assessor was carried out twice for five Japanese standard odorants and six to eight times for n-butanol by the olfactometer on each day. The number of individual data for the threshold is six for five Japanese standard odorants, and about twenty for n-butanol.

Table 2 shows the result of the panel screening tests. In the Japanese method, two people did not pass the β-phenylethyl alcohol test. In the European method, ten people did not meet the sensitivity criterion and four people did not meet the variability criterion. In total, fourteen people did not pass the test. The selection criteria in the European method are considerably stricter than those in the Japanese method.

Table 2 Results of Panel screening tests

Assessor	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
Japanese method	○	○	○	○	○	○	○	×	○	○	○	○	○	○	○	○	○	○	○	×
European method Sensitivity	×	×	×	○	×	○	○	×	○	×	×	×	○	○	○	×	○	×	○	○
Variability	○	○	×	○	○	○	○	○	×	○	○	○	○	×	○	○	○	○	○	×

Note: Odorant for which assessors did not meet the criteria in the Japanese method was β-phenylethyl alcohol

Assuming that the distribution of the logarithm of the individual threshold becomes a normal distribution, the distance of each selection criterion of sensitivities from the mean value were calculated. In addition, the ratio of the group, which does not meet the criterion, was obtained from the normal distribution table. The results are shown in Table 3.

Table 3 Comparison of criteria selection in view of the distribution of the individual Threshold

	β-phenylethyl alcohol	methyl cyclopentenolone	isovaleric acid	γ-undecalactone	scatorl	n-butanol	
Mean (m)	5.37	5.97	6.14	5.60	6.99	1.91	
Standard Deviation(s)	0.98	0.33	0.28	0.50	0.40	0.38	
Selection Criteria	4 m-1.4s	4.5 m-4.5s	5 m-4.1s	4.5 m-2.2s	5 m-5.0s	1.3 m-1.6s	1.9 m+0.04s
Ratio of disqualification (%)	8.1	0.1<	0.1<	1.4	0.1<	5.5	48

Note: The values for five Japanese standard odorants are n of concentration 10^{-n} (w/w). For n-butanol, the values are the logarithm of the concentration (ppb). Therefore, in the case of five odorants, if the individual threshold is smaller than the value of the criterion, it does not meet the criteria as dysosmia. In the case of n-butanol, if the individual threshold is smaller than 1.3, it means the assessor has a super-nose, and if it is larger than 1.9, the assessor's sensitivity is weak.

The selection criteria for the five standard odorants became at the point of 1.4 - 5 times the standard deviation from the mean value. Ratios of disqualification were 1.4% or less except for β-phenylethyl alcohol, the ratio of which was 8.1%.

In the case of n-butanol, the criterion to exclude the super-nose was at the point of 1.6 times the standard deviation from the mean value. The criterion concerning weak sensitivity was at the point of 0.04 times the standard deviation. The latter value is almost the same as the mean value. The ratio of disqualification as a super-nose was 5.5%, while that due to weak sensitivity was 48%. In total, more than half of the people might not qualify to be a panel member.

Whether a panel member who passed the screening test using one standard odorant has adequate sensitivity for any actual odor is a difficult question to answer, because individual sensitivity might vary significantly depending on the odor substances. The results in table 2 show that one out of two assessors who did not have sufficient sensitivity for β-phenylethyl alcohol met the sensitivity criteria for n-butanol. Although this result suggests that some mixture is needed as a standard odor, it might be realistic to exclude the outlier by discarding the data after measurement.

2.3 Result of olfactory measurement

To grasp the difference in the odor concentration values determined by both methods, various odor samples were measured.

Twelve people who passed the Japanese screening test were selected as panel members. They were divided into 2 groups of 6 people. Group A consisted of 6 people who passed the European screening test, in contrast to group B which consisted of the other 6 people who did not pass the European test. In each group, the same samples were measured by the both methods on the same day.

Three standard odorants were measured as samples, m-xylene, 35.6ppm; n-butanol, 31.9ppm and hydrogen sulfide, 0.299ppm, and then six actual source samples were also measured. All samples were prepared in 50L polyester bags.

In the case of source samples, the original gas samples were left untouched for two weeks after sampling to stabilize their odor concentration. They were then diluted and the odor was measured. There was a one-day gap between measuring group A and group B.

An individual threshold was measured five or more times for standard odor samples and three times for source samples. The first measurement data taken by the olfactometer were discarded in conformity with the European method.

The measurement results for the standard odor samples are shown in Table 4. The mean values in this table are indicated in the logarithm of the olfactory threshold. The measured results of both methods in each group were generally the same. A difference outcome was expected from the dilution accuracy test for hydrogen sulfide, however none was apparent. That is, there was a possibility that the logarithm of the threshold determined by the olfactometer would raise, due to a decrease of the diluted gas concentration. The corresponding data for m-xylene, which is diluted very accurately, indicate that a difference in the methods such as descending or ascending was not apparent.

Table 4 Olfactory measurement results for standard odor samples (threshold logarithm)

		Group A		Group B	
		Triangle odor bag method	Dynamic olfactometry	Triangle odor bag method	Dynamic olfactometry
m-xylene	Mean （ppb）	2.0 (98)	2.0 (90)	2.2 (166)	2.3 (215)
	Standard deviation	0.37	0.08	0.11	0.04
	n	7	7	8	7
n-butanol	Mean （ppb）	1.3 (20)	1.6 (40)	1.4 (23)	1.4 (24)
	Standard deviation	0.17	0.06	0.15	0.10
	n	6	5	7	6
hydrogen sulfide	Mean （ppb）	2.7 (523)	2.8 (661)	2.8 (591)	2.7 (497)
	Standard deviation	0.20	0.08	0.19	0.15
	n	7	5	7	5

Note: Threshold of m-xylene, n-butanol are indicated in ppb, hydrogen sulfide in ppb.

The panel-screening test using n-butanol was held six months before this odor measurement. The results showed the threshold values of group A members were approximately 40ppb while most of the members from group B were valued at 100-200 ppb. However, a significant difference was not observed in this measurement. This suggests that the sensitivity of some individuals may largely vary over a period of years. Provided that this is true, selecting panel members by the European method has to be performed very carefully.

The measurement results for the source samples are shown in Table 5. The value is indicated in the odor index. (odor index = 10 log [odor concentration]) The results of both methods in each group corresponded very well as well as for standard odors, though a difference of 4 was observed for the excrement odor for group B. It seems that the odor index in group A tends to be higher than that for group B. However, it cannot be concluded that the reason is whether there is a difference of sensitivity between members of each panel or differences of sample concentrations.

Table 5 Olfactory measurement result for source sample (odor index)

	Group A		Group B	
	Triangle odor bag method	Dynamic olfactometry	Triangle odor bag method	Dynamic olfactometry
Spray painting	27	25	22	22
Baking finish	28	26	26	23
Offset printing	29	29	27	26
Sewage	28	30	24	25
Excrement	32	31	28	24
Rendering	29	30	30	29

3. Conclusion

Provided that the same panel is used, the triangle odor bag method and dynamic olfactometry agree in results in spite of such differences as descending or ascending, sniffing conditions, etc. However, the selection criteria for the panel screening show a large difference. Although this may influence odor measurement results, the effect could not be observed in this present study because of variation in sensitivity. It will be necessary to acquire further data on variability over the long term, and to study the relation between measurement results and the performance of panel.

References

1) European Committee for Standardization, DRAFT prEN13725 Air quality-Determination of odor concentration by dynamic olfactometry (1999)

Quality Control of Olfactometry in Japan

Takaya Higuchi
Department of Civil Engineering, Yamaguchi University
2-16-1, Tokiwadai, Ube,
Yamaguchi, 755-8611 Japan
E-mail: takaya@yamaguchi-u.ac.jp

Keywords
Olfactometry, quality control, triangle odor bag method

Abstract

In Japan, the necessity of developing quality control system for olfactometry and standardization of measurement procedure for the promotion of nationwide spread of olfactometry has been recognized especially in recent years. In this paper, the establishment of quality control framework for olfactometry in Japan, including selection of a reference odor, development of reference odor preparation technique, interlaboratory comparison of olfactometry, and fundamental constitution of quality control manual for laboratory use, was discussed. Ethyl acetate was selected as a reference odor for olfactometry, and four preparation methods (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were verified and confirmed to be applicable to quality control processes. In late 2000, interlaboratory comparison of olfactometry was carried out in order to collect basic data for the establishment of quality control procedure and the determination of quality criteria. Mean values, repeatability standard deviation, reproducibility standard deviation, and standard deviations under intermediate conditions of detection threshold were calculated from the measurement results. On the bases of these discussions, quality control framework for olfactometry was proposed. In early 2003, the quality control manual for laboratory use was published.

1. INTRODUCTION

In Japan, regulatory policy of offensive odor based on olfactometry (i.e., triangle odor bag method and triangle odor flask method) was first introduced into the Offensive Odor Control Law in 1995.(1) However, the necessity of developing quality control system for olfactometry and standardization of measurement procedure for the promotion of nationwide spread of olfactometry in municipalities has been recognized especially in recent years.

This paper presents about the establishment of quality control framework for olfactometry in Japan, including selection of a reference odor, development of reference odor preparation technique, interlaboratory comparison of olfactometry, and proposal of fundamental constitution of quality control manual for laboratory use.

2. FUNDAMENTALS OF TRIANGLE ODOR BAG METHOD

The triangle odor bag method, the most popular olfactory sensory test in Japan, was first developed by Tokyo metropolitan government in 1972.[2)3)] This is an air dilution method in which "odor concentration" or "odor index" is measured. Odor concentration is the dilution ratio when odorous air is diluted by odorless air until the odor becomes unperceivable. Odor index is the logarithm of odor concentration, multiplied by ten. The olfactory measurement method of offensive odor was notified in 1995 (Notification No. 63 of the Japan Environment Agency, 1995). This method consists of the following parts:

- Panel selection
- Apparatus
- Sampling
- Testing procedure

Individual panelist is required to have passed the screening test of using five odorous compounds (i.e., phenethyl alcohol, methylcyclopentenolone, isovaleric acid, γ-undecanolactone, and skatole). Measurements for samples taken at odor emission sources are made in three-fold dilution descending series. In this method, three odor bags are prepared and filled with odorless air passed through the activated carbon column. Odorous sample is injected into one of these three odor bags. Each panelist sniffs these odor bags and chooses one odor bag that is likely to contain odorous air. The test is continued until all panelists make incorrect replies. Then odor concentration or odor index is calculated.[1)]

3. REFERENCE ODOR FOR OLFACTOMETRY

3.1 Selection of reference odor

Reference odor is necessary in order to conduct interlaboratory comparison of olfactometry as well as routine verification of measurement results in each olfactometry laboratory. Four odorous compounds (i.e., *n*-butanol, ethyl acetate, *m*-xylene, and dimethyl sulfide) were proposed for reference odor. In Europe, *n*-butanol is defined to be a reference odor in CEN draft standard prEN 13725.[4)] Ethyl acetate is one of "specific offensive odor substances" designated in the Offensive Odor Control Law in Japan, and *m*-xylene and dimethyl sulfide are compounds used as reference odors in previous interlaboratory comparison in Japan. Reference odor for olfactometry should fulfill the following requirements:

- Odor sample should be prepared easily and accurately.
- Odor sample should remain stable for a period of the measurement.
- Odor threshold values of panelists should not vary widely.
- Odor quality should be easily recognized.
- Low health and psychological effect on operators and panelists should be ensured.

Considering all these things, ethyl acetate was selected as a reference odor for olfactometry in Japan. Although *n*-butanol is designated as a reference odor in CEN prEN 13725, it was not selected because there is less measurement data for *n*-butanol in Japan and ethyl acetate has the advantages in sample preparation and data accumulation.

3.2 Preparation of reference odor

Easy-to-operate and cost-effective technique for reference odor preparation is necessary to be employed in nationwide municipalities and olfactometry laboratories. On the assumption that reference odor sample with odor concentration of two to three thousand is appropriate to be used in quality control process, the concentration of ethyl acetate is calculated to be around 2000 ppm in consideration of odor threshold of 0.87 ppm.[5)]

Four preparation methods for reference odor (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were proposed. Steel cylinder containing ethyl acetate of 2010 ppm was specially ordered. In odor bag/vacuum bottle method, an odor bag or a glass vacuum bottle is employed to vaporize ethyl acetate reagent. These four preparation methods were verified at three olfactometry laboratories and confirmed to be applicable to quality control processes.

4. INTERLABORATORY COMPARISON OF OLFACTOMETRY

4.1 Method

In late 2000, interlaboratory comparison of olfactometry was carried out in order to collect basic data for the establishment of quality control procedure and the determination of quality criteria. A total of seven olfactometry laboratories in Japan participated in the test. A three-liter-capacity sampling bag filled with ethyl acetate of around 2000 ppm was delivered to each laboratory four times. Odor index and odor concentration of each sample were measured according to the official procedure of the triangle odor bag method. The tests were conducted six times over four days (i.e., three times for the second sample and only once for other three samples). Steel cylinder method was used to prepare reference odor, i.e., ethyl acetate of 2010 ppm. Gas concentration of each sample was analyzed with GC-FID just before the delivery.

4.2 Results

Figure 1 shows detection thresholds of ethyl acetate calculated from odor concentration and gas concentration obtained from the tests.

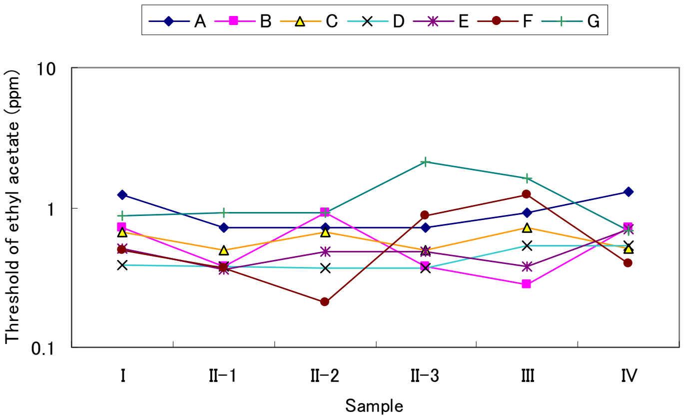


Figure 1. Detection thresholds of ethyl acetate measured in seven laboratories (A-G). The measurement results of II-1, 2, and 3 represent the repeated data of the same sample within one day.

The mean value, repeatability standard deviation, and reproducibility standard deviation of detection threshold were calculated from the results of II-1, 2, and 3 according to JIS Z 8402-2 6), which is Japanese version of ISO 5725-2 (1994). On the other hand, the results of I, II-2, III, and IV were used for the calculation of the mean value and standard deviations of detection threshold under intermediate conditions.7) **Table 1** shows these evaluation results. In practice, the logarithm of detection threshold was used for the calculation of these values.

Table 1. Mean values (m), repeatability standard deviation (s_r), reproducibility standard deviation (s_R), and standard deviation under intermediate conditions (s_i) of the logarithm of detection thresholds.

Repeatability and reproducibility conditions	Measurement results	*m*	Antilog of *m* (ppm)	s_r	s_R
	II-1, 2, 3	-0.26	0.56	0.17	0.22
Intermediate conditions	Measurement results	*m*	Antilog of *m* (ppm)	s_i	s_R
	I, II-2, III, IV	-0.20	0.63	0.18	0.21

5. FRAMEWORK OF QUALITY CONTROL MANUAL

On the bases of foregoing discussions about reference odor and interlaboratory comparison, quality control manual for laboratory use was published in early 2003. **Figure 2** shows quality control framework for olfactometry in a laboratory. The fundamental topics in the manual are as follows:

- Establishment of quality control system and organization in a laboratory
- Education and training of staff concerned
- Documentation of measurement processes
- Preparation of standard operating procedures (SOPs)
- Evaluation and report of measurement results
- Regular internal quality checks using reference odor
- Occasional proficiency tests using certified reference odor

Quality assessment process is depicted in **Figure 3**. On the basis of collaborative assessment experiment, accepted reference value, repeatability, and reproducibility of reference odor are obtained. Then individual olfactometry laboratory is able to carry out regular quality checks and compare the results with these values. In this research, mean value, repeatability, and reproducibility of reference odor threshold were calculated from the results of interlaboratory comparison. However, further investigation will be necessary to obtain the accepted values. Odor bag/vacuum bottle method and handy gas cylinder method seem to be appropriate for the preparation of reference odor for internal use because of the easiness of preparation technique and cost-effectiveness. Certified reference odor (CRO) is necessary for the proficiency tests. Therefore, CRO supply system should be developed in the framework of traceability concept. In this case, steel cylinder method and handy gas cylinder method have advantages.

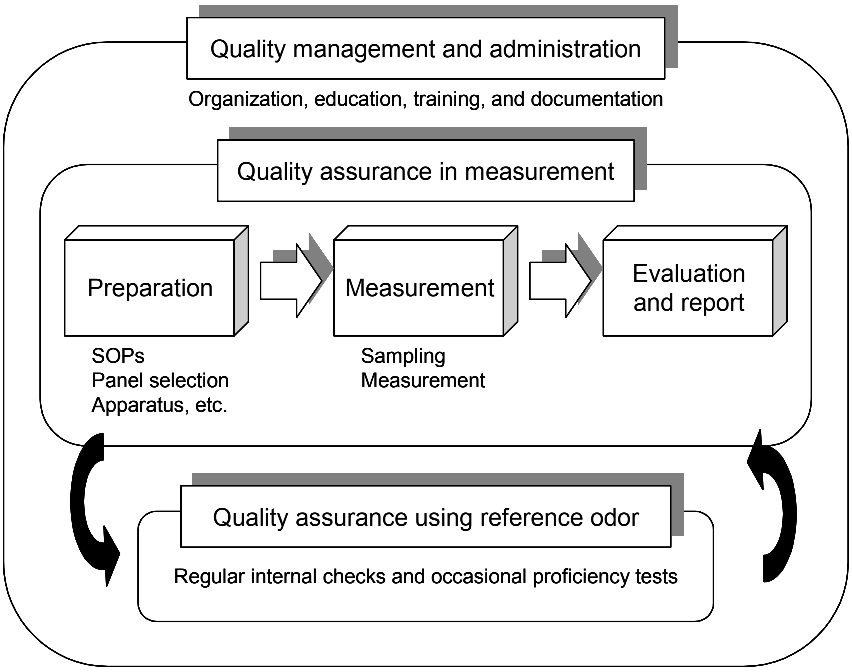


Figure 2. Quality control framework for olfactometry in a laboratory.

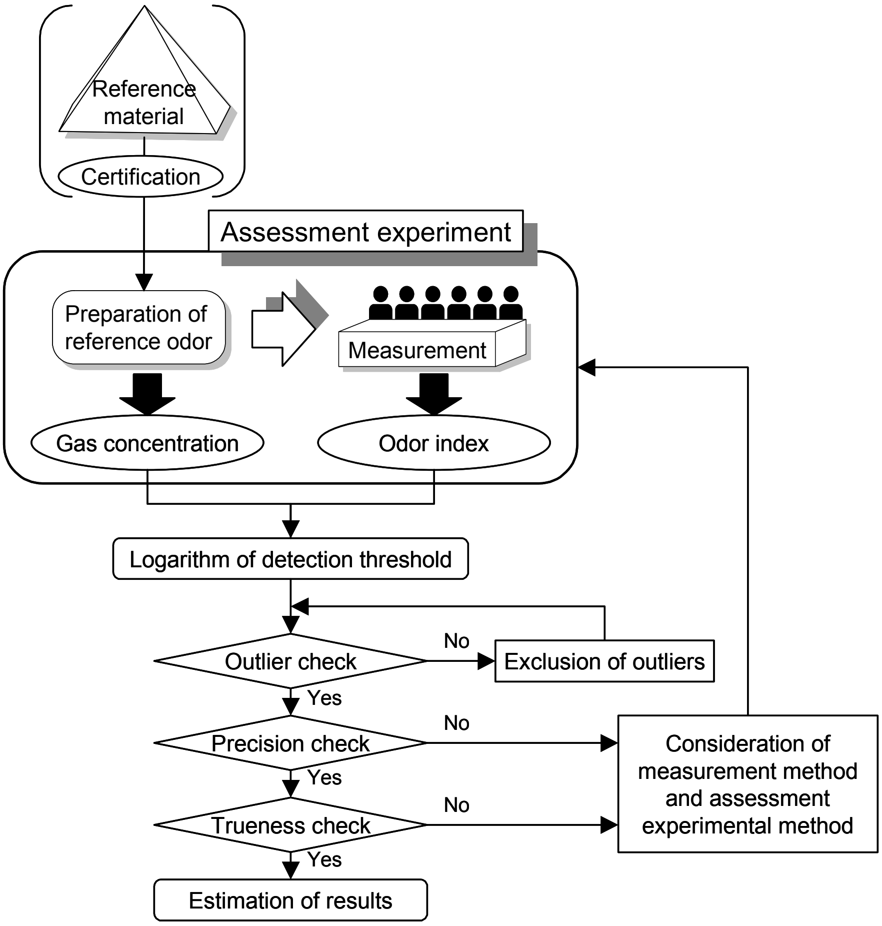


Figure 3. Outline of quality assessment using reference odor.

5. CONCLUSIONS

In this paper, the establishment of quality control framework for olfactometry in Japan was discussed. Ethyl acetate was selected as a reference odor for olfactometry, and four preparation methods (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were verified and confirmed to be applicable to quality control processes. In late 2000, interlaboratory comparison of olfactometry was carried out and mean values, repeatability standard deviation, reproducibility standard deviation, and standard deviations under intermediate conditions of detection threshold were calculated from the results. On the bases of these discussions, quality control framework for olfactometry was proposed. In early 2003, the quality control manual for laboratory use was published.

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References

1) Higuchi, T. and Nishida, K. (1995) Analysis of data measured by the triangular odor bag method, in McGinley, C. M. and Swanson, J. R. (eds.), *ODORS: Indoor and Environmental Air*, Air & Waste Management Association, Pittsburgh, pp. 181-192.

2) Iwasaki, Y., Ishiguro, T., Koyama, I., Fukushima, H., Kobayashi, A. and Ohira, T. (1972) On the new method of determination of odor unit, in *Proceedings of the 13th Annual Meeting of the Japan Society of Air Pollution*, p. 168. (in Japanese)

3) Iwasaki, Y., Fukushima, H., Nakaura, H., Yajima, T. and Ishiguro, T. (1978) A new method for measuring odors by triangle odor bag method (I) — Measurement at the source, *Journal of Japan Society of Air Pollution*, Vol. 13, No. 6, pp. 246-251. (in Japanese)

4) CEN prEN 13725 (1999) Air quality — Determination of odour concentration by dynamic olfactometry.

5) Nagata, Y. and Takeuchi, N. (1990) Determination of odor threshold value by triangle odor bag method, *Bulletin of Japan Environmental Sanitation Center*, No. 17, pp. 77-89. (in Japanese)

6) JIS Z 8402-2 (1999) Accuracy (trueness and precision) of measurement methods and results —

Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method. (Japanese version of ISO 5725-2, 1994)

7) JIS Z 8402-3 (1999) Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method. (Japanese version of ISO 5725-3, 1994)

Standard Odors for Selection of Panel Members

Katsunori Saiki
Manager of Planning & Development Dept.
Daiichi Yakuhin Sangyo Co.,Ltd.
14-4, Nihonbashi 2-Chome, Chuo-ku, Tokyo 103-0027, Japan

Keywords
Standard Odors, sense of smell, selecting panel

“The Olfactory Measurement Method" is the method of evaluating the stink by sense of smell. The members to do this measurement are called Panel members.
It is essential to have panel members with an accurate sense of smell to check odors, particularly stink smells. However, up to now, no method existed to confirm whether panel members had an accurate sense of smell or not.
In order to confirm this, it is necessary to have standard odors, and the T & T Olfactometer (Standard Odors for Measuring Sense of Smell) was developed for this purpose. It is used in Japan with approval of the Ministry of Health and Welfare to diagnose symptoms of abnormality in sense of smell.
The standard odors for selecting panel members have been developed based on the T & T Olfactometer, and a lot of data has been collected on Environment Pollution of Offensive Odors by authorities and large organizations. These standard odors are used for selecting panel members by the "5-2 method".

The Offensive Odor Control Law in Japan was revised on April 1, 1996. A national qualification of 'Olfactory Measurement Operator' came to be given to the person who passed the national examination which Ministry of the Environment provided by this revision. The Standard Odors for Selection of Panel Members is used in this national examination. Moreover, The Offensive Odor Control Law in Japan was partly revised on April 1, 2001. In this revision, The Olfactory Measurement Method was added. This product is similarly adopted for selection of panel by this method.

1. Chemical Components and their Odor Quality

A ---------- β-Phenylethyl Alcohol
  * Flower odor
  * Smell of rose petals
B ---------- Methyl Cyclopentenolone
  * Sweet burning smell
  * Smell of caramel in custard pudding
C ---------- Isovaleric acid
  * Smell of sweat
  * Smell of stinking socks

D ---------- γ -Undecalactone
* Smell of ripe fruit
* Smell of canned peaches

E ---------- Skatole
* Musty smell
* Smell also found in excrement

2. Content of the Standard Odors

The densities of the standard odors are all w/w.
The control liquid is odorless liquid.
There are 500 olfaction test papers in one packet.

3. Five standard odors for selecting panel members

This set consists of 5 standard odors A, B, C, D, and E. The middle density is set at the "standard density for selection" as stated in the report by the Environment Agency.

3.1 Standard odors for measuring sense of smell:

A ($10^{-3.0}$,$10^{-3.5}$,$\underline{10^{-4.0}}$,$10^{-4.5}$,$10^{-5.0}$)
B ($10^{-3.5}$,$10^{-4.0}$,$\underline{10^{-4.5}}$,$10^{-5.0}$,$10^{-5.5}$)
C ($10^{-4.0}$,$10^{-4.5}$,$\underline{10^{-5.0}}$,$10^{-5.5}$,$10^{-6.0}$)
D ($10^{-3.5}$,$10^{-4.0}$,$\underline{10^{-4.5}}$,$10^{-5.0}$,$10^{-5.5}$)
E ($10^{-4.0}$,$10^{-4.5}$,$\underline{10^{-5.0}}$,$10^{-5.5}$,$10^{-6.0}$)

	5 ml bottle each
Control liquid	50ml 3 bottles
Olfaction test papers	4,000

(note. ﹏ underlining indicates the standard odor for selection)

3.2 Application

This is used to test whether the examinees have a normal sense of smell. At the same time, it is possible to check the degree of ability to sense smells of candidates with a normal sense of smell, by checking whether they can discern densities lower than the standard density for selection.

For examinees with an abnormal sense of smell, it is possible to check how close to normal it is.

4. Preparation

Note. The operator (examiner at the sense of smell test) and the collector of test materials should have a normal sense of smell. This should be confirmed before carrying out the tests on panel candidates. Also the hands of the operator and examinees should be checked to ensure they do not carry any odor. If they do, the hands should be washed well with an odorless, liquid soap.

5. How to Use

The test is carried out using either the 3 standard odors or the 5 standard odors, by the

"5 - 2 method". In this method, the operator selects at random 2 olfaction test papers out of 5, and dips them up to 1 cm from the edge into one standard odor liquid. (Each paper has the 1 cm level marked on it). The remaining 3 olfaction test papers are then dipped into the control liquid. (Fig.1)

The examinees are given 5 test papers one by one, and sniff the tip of each one by bringing the test paper close to, but not actually touching the nose. (Fig.2)

After sniffing all 5 papers, the examinees should select 2 test papers which they think carry the odor, and answer by giving their numbers (both should be correct).(Fig.3)

The examinee passes the test by giving the correct answers for all the standard density odors.

Fig.1

Fig.2

Fig.3

Five standard odors for selecting panel members

6. Points on Usage

(1) How to sniff

Examinees should sniff lightly and briefly while concentrating. They may sniff a second time if they cannot discern the odor the first time. There should be a slight time interval before sniffing again.

(2) Using and Discarding the Olfaction Test Papers

Always wipe the test papers against the inside neck of the bottle after dipping them into the standard odor liquids, to prevent them from dripping.

Do not use olfaction test papers again once they have been dipped into the standard odor liquid. They should be discarded after each test. New olfaction test papers must be used for each examinee, dipping them into the standard odor liquid for each sense of smell test.

Since the discarded test papers would leave some odor in the examination room, they should be thrown away in a waste basket with a lid, or placed in a plastic bag and closed with a rubber band before discarding into a waste basket.

(3) Answering the Test

Answers should be written on the answer sheet provided by the operator.

(4) Letting the Examinee Check his Own Odor Sensitivity

The examination should be carried out using the "5-2 method" for each standard odor liquid.

7. Preliminary Practice to Test for Selection of Panel Members

The operator should give each examinee a preliminary practice. This is to allow the examinee to get used to the test, and to free him of anxiety. The practice should be given once or twice using a density one step higher than that used for the standard density for selection.

8. Supplementary Test for the Selection of Panel Members

If an incorrect answer is given to only one of the standard density odor liquids, the examinee may be given a supplementary test depending on the wishes of both parties.

9. Retesting

It is generally considered that the sense of smell for people who have passed the test for the selection of panel members, remains stable for a period of 5 years. Therefore panel members should be retested every 5 years. It is wiser to retest panel members over 40 years of age, since their sense of smell ability tends to deteriorate.

However, the above does not apply to people who were found to have an abnormality in their sense of smell due to disease, traffic accidents, and so on.

10. Validity Period of Standard Odors for Measuring Sense of Smell

The standard odors remain valid for one year after opening, or two years from the date of manufacture for unopened bottles. Do not use standard odors which validity period was over.

11. Storage

Please note that the standard odors for measuring sense of smell should be stored in a cool, dark place.

3. Researches and Developments on Odor Measurement

Development of the Next Generation Dynamic Olfactometer – Dynascent

John Jiang
EnvironOdour Australia Pty Ltd, Sydney, Australia
j.jiang@environodour.com.au

Keywords
Odour, dynamic olfactometer, calibration

Abstract

The introduction of an international standard on odour measurement presents a new challenge for olfactometry laboratories to meet tough new instrumental performance and panellist performance criteria. The paper reviews a number of olfactometers used in the last ten years both in Australia and overseas. It has found that the back pressure could contribute a significant error in the delivery of the required dilution ratio and the contamination of the olfactometer was the source of large variation in olfactometry results. The paper discusses the instrumental performance of the newly developed olfactometer. DynaScent is a fourth generation olfactometer which uses no flow measurement device (flowmeter or mass flow controller). The dilution ratios of the DynaScent were calibrated using CO gas and auto calibration feature. The effects of the CO sampling point locations (within the system and above the sniffing cup) could have significant impacts on the dilution ratio. Practically, the manual calibration is both time consuming and labour intensive. Furthermore, it was found that the accuracy and instability results were sensitive to the selection of the CO gas range. It is concluded that the calibration of a dynamic olfactometer should be carried out more frequently than once a year. The DynaScent olfactometer was able to achieve averaged instability of 1.7% and accuracy of 9% over the dilution range of 2 - 65000.

1. INTRODUCTION

Uncertainty in odour measurement is a major concern to environmental regulators, researchers, and stakeholders[1]. The large variations in odour concentrations have limited the use of dynamic olfactometry results in the regulations in the United States and European countries. In some studies, the olfactometry method was considered to be "comparable" within the studies and was not compatible to other studies due to different olfactometry standards being used. The reliability of odour measurements has often been debated in environmental courts. Odour is the most contentious issue in environmental regulations.

Berglund *et al*[2] suggested that "*A substantial proportion of the large variation attributed to the observers in odor studies, originates from olfactometric malperformance*". A more recent study at the University of Minnesota indicated that "*Sample and supplemental airflow rates were significantly different at the beginning and end of a typical session*" with their mass flow based olfactometer[3]. Jiang also confirmed that the ability of dynamic in delivering the required dilution radio over many sessions of odour testing is one of the two critical factors and suggested that the instrumental calibration should be carried out in more frequent than once per year[4].

The importance of instrument calibration to determine the dilution ratio and panel management to monitor individual panellist performance over standard odorant (such as n-butanol) is also disputed among the professionals. Most commercial olfactometers did not produce the instrumental accuracy and instability from the tracer gas calibration results over the entire dilution range as required in the standards. van Harreveld et al. reviewed 20 years of olfactometry development in Europe and concluded that panel selections and span adjustment were the only two “crucial” steps[5]. Span adjustment meant that “laboratories could adjust their method to achieve the same value for the odour unit: 1 ou/m^3 = 40 ppbv n-butanol.” Jiang noted that meeting the instrumental performance and panellist performance criteria was a major challenge for olfactometry laboratories in the implementation of the Australian standard[1].

The calibration of any analytical instrument is the first step toward producing reliable testing results. Odour measurement is no exception and instrumental calibration is the most important step in controlling uncertainty during the measurement to ensure the delivery of the required dilution ratio. There are two assessment criteria. The accuracy of the dilution apparatus ensures that the dilution ratio between five series is delivered within 20% of the set dilution ratio. The instability of the dilution apparatus ensures the same dilution ratio is given to all the panellists within 5% of the expected values in the same dilution step[6, 7].

Unfortunately, some olfactometers are not so easy to calibrate. For example, those olfactometers that use sniffing masks have some difficulties in confirming the dilution ratio at the time when the panellist is sniffing. Consequently, an uncalibrated olfactometer undoubtedly affects the panel selection and results in the panellists' making inappropriate assessment of the odour. As a result, the odour concentrations measured using the uncalibrated olfactometer might suffer from poor accuracy and instability.

During the 1980s, considerable effort was made in developing olfactometric odour measurement techniques in the Netherlands and elsewhere in Europe[8, 9]. Initially, the application of these olfactometric results was limited mainly to comparing odour emissions from various manure treatment systems in intensive animal production. In 1985, the Victoria EPA in Australia first introduced legislation based on olfactometer results and air dispersion model (Ausplume). In North America, despite earlier interest in olfactometric measurement techniques during the 1970s, it was not until the mid 1990s that North American universities set up olfactometry laboratories to investigate odour from animal production.

The development of olfactometric measurement techniques continued in Europe and resulted in the introduction of the first draft European Standard for odour measurement by dynamic olfactometer. In Australia, a national workshop on odour measurement standardization was held in 1997 and consensus was reached to adopt the draft European standard. In 2001, Australia published the first official standard ahead of European countries. European countries have officially agreed to adopt the CEN standard in early 2003.

In summary, most olfactometers currently used around the world can be categorised in three groups on the basis how the dilution is achieved:

- static method (syringe method in USA, triangle bags in Japan);
- rotameter/fixed orifice based olfactometers (VIC. EPA B2 in Australia, TO7 in Germany, IITRI in USA) and
- Mass Flow Controller (MFC) based olfactometers (Ac'scent olfactometer in USA, Olfaktomat as used in the Netherlands).

Static method utilises the syringe to make the necessary dilution. The error in reproducing the necessary dilution ratio is so large. The flow rates at the sniffing ports are so low. The manual operation can no long meet the requirements specified in Australia and European standards.

Rotameter based olfactometers are currently used in many laboratories in Australia and elsewhere. The rotameters are extremely sensitive to downstream pressure variations that could result in errors in rotameter readings of up to 25%. Such pressure variations may be occurred during the mixing of clean air and odorous air to create the required dilution radio or subsequently during the sample presentation of the diluted sample. The latter may be accentuated by the use of an enclosed sniffing mask, adversely affecting overall performance of the olfactometer. The manual mode of operation for rotameter based olfactometers makes it impossible to meet stringent instrumental performance criteria, particularly at the high dilution ratio end of the range. Furthermore, high labour costs when using manual data input for monitoring panellist performance and in the data processing used for retrospective screening may also make compliance with the proposed standard excessively expensive.

In contrast, automated, MFC based olfactometers have demonstrated an inherent ability to comply with both instrumental and panellist performance criteria. But the MFC based olfactometers are also sensitive to the downstream pressure of the flow measurement devices. The backpressure occurring during mixing can be compensated for by instrumental calibration. However, pressure variations occurring during the sample presentation stage cannot be predicted and therefore cannot be compensated for by calibration. Backpressures can vary from panellist to panellist. In practice, the reduced flow arising from the specific personal characteristics of a panellist will be sensed by the mass flow meter resulting in the valve being further opened. However, the presentation time for each panellist is long (10 – 30 seconds) in comparison with the response time of the mass flow controller to change the valves (several seconds). These unstable conditions will be repeated many times during the session. As a result, the actual dilutions of odour samples at the sniffing ports can be highly variable.

Furthermore, the MFCs are susceptible to contamination buildup that can alter the calibration and result in the reduced performance. The tiny space between the temperature elements inside the mass flow meter can be easily contaminated or blocked. The MFC is really designed for single component gas and better suited to a clean and non-sticky gas. In particular, the odour samples can sometime be very sticky and dusty. Therefore, MFC based olfactometers could easily suffer from the poor performance of the MFCs during the operation. This has proved to be a major limitation in the use of MFC based olfactometers. Flushing the MFCs may take hours and is not effective at all. Over a period of usage, dust and residuals eventually become irreversibly adhered to the surfaces of the temperature elements and the MFC must be replaced. The performance of MFC based olfactometer in delivering the required dilution radio can not guaranteed.

2. METHODOLOGY

The DynaScent olfactometer is a fourth generation dynamic olfactometer which is fully computer controlled and uses no flow measurement devices (The DynaScent Olfactometer, EnvironOdour Australia Pty Ltd, 2003). The sample is mixed with odourfree air within a custom designed venturi gas jet. The dilution ratios are adjusted by a series of needle valves controlled by the digital precision motion controller with ±0.0001 revolution accuracy. The repeatability of the dilution is purely based on the mechanical repeatability which is capable to reproduce highly repeatable dilution ratio. The precision needle valves can be dismantled and cleaned to minimise the effects of the contamination. The variation in the dilution ratios during the sample mixture and during the panellist sniffing is minimised by the use of the critical nozzle and the improved sniffing cups.

Carbon monoxide was chosen as a tracer because CO is a non-reactive gas and because of the reliability of CO gas monitors. Due to the limited detection range in the gas monitor, a series of CO gases were used so that the final gas concentrations at the sniffing cup were within the detection range of the gas analyzer. The CO gas was loaded as a normal sample and placed within the sampling drum. A Monitor Labs 9830 CO analyzer (range from 0 to 200 ppm), calibrated by an accredited laboratory, was used to determine the CO concentration at the sniffing cup.

The following table lists the range of CO gases used in the calibration.

Table 1 CO concentrations at source and cup

Dilution step	Dilution range	CO conc. at source, ppm	Expected CO conc. at cup, ppm
1 – 4	2 – 16	162	81 – 10
5 – 8	32 – 256	4,840	150 – 20
9 – 12	512 – 8,192	100,000	195 – 24
13 – 19	16,384 – 524,288	1,000,000	122 – 2

The signal output of the CO analyzer was connected into the DynaScent olfactometer for direct display of CO readings and data logging. The Dynascent automatically starts a dilution step, takes 10 CO readings over 100 seconds (one reading every 10 seconds), then moves to the next dilution step and takes 10 CO readings, until all the steps are completed. This process is then repeated five times. The CO results are used to calculate the accuracy and instability of the olfactometer as per the Australian and New Zealand standard[6].

Figure 1 Calibration setup

Figure 1 illustrates the calibration setup. A second CO monitor was used to monitor the CO concentration in the room to keep it below 15 ppm at all times for safety reasons. A flow meter with a range of 4 – 40 Liters Per Minute (LPM) was used to calibrate the flow rate at the sniffing ports. Before the calibration of the dilution ratio, the flow rates were checked to be 20 LPM at both sniffing ports before and after the completion of the dilution adjustment.

3. RESULTS AND DISCUSSION

Instrumental calibration of an olfactometer is time consuming and labour intensive. Calibration of the olfactometer involves hundreds of measurements (950 single measurements comprising 50 measurements for each dilution step for a 19-step olfactometer). The process could take up to five days if adjustment of the instrument setting is required for a MFC based olfactometer. Consequently, the inclusion of an automated calibration feature in the olfactometer is necessary.

The location of the sampling tubing could have an effect on the CO readings. As shown in Figure 2, CO concentrations at the sniffing cups (Figure 2, right) were 40% smaller than those collected in the tubing (Figure 2, left) which was below the sniffing cup. Initially, it was suspected that extra gas went into the gas analyzer to cause the large bias. A bulb flow meter was used to measure the sampling rate of the CO analyzer. It was found that there was no difference between these two arrangements. The only possibility was that the open space in the cup might dilute the CO concentrations. In this study, the sampling at the sniffing cup was used.

Figure 2 Sampling arrangement

Figure 3 shows the consecutive CO concentrations for dilution steps 5 – 8 over 45 minutes. The results are distributed over a narrow range which depends on the expected concentrations. The results show a higher CO variation (130 – 162 ppm) at the higher expected CO concentration (150 ppm) and a lower CO variation (19 – 22 ppm) at the lower expected CO concentration (20 ppm). These variations were likely caused by the CO gas analyser.

The accuracy (expressed as a bar chart) and instability (expressed as a single line chart) of the dynamic olfactometer are shown in Figure 4. The results show an excellent instability of 1.7% for the dilution step of 1 - 16 and an increased instability for the dilution step of 17 - 19. This was caused by the small CO concentrations, less than 10 ppm. The averaged accuracy of the olfactometer is 9% over the range of 2 - 65000. The accuracy of the instrument seems to be more sensitive to the absolute CO concentration levels at the sniffing cup.

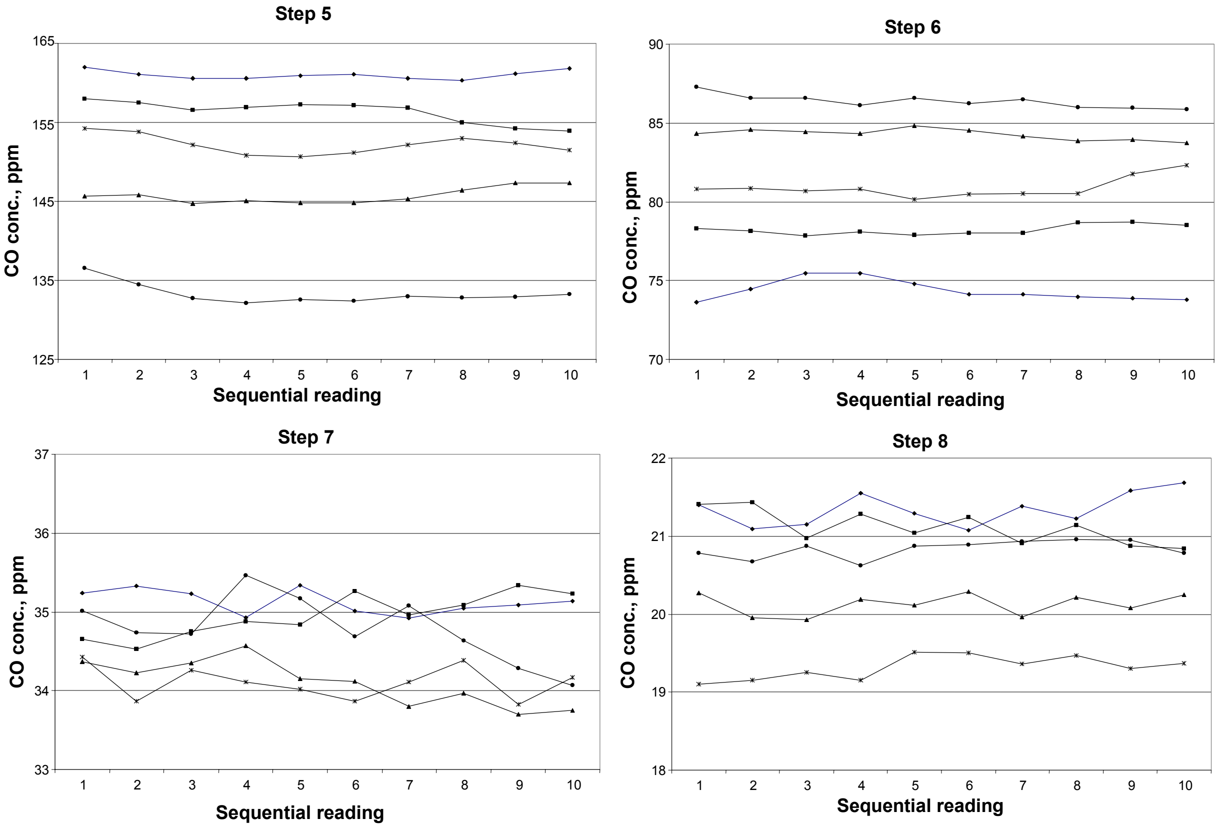

Figure 3 CO concentrations at dilution steps 5 – 8

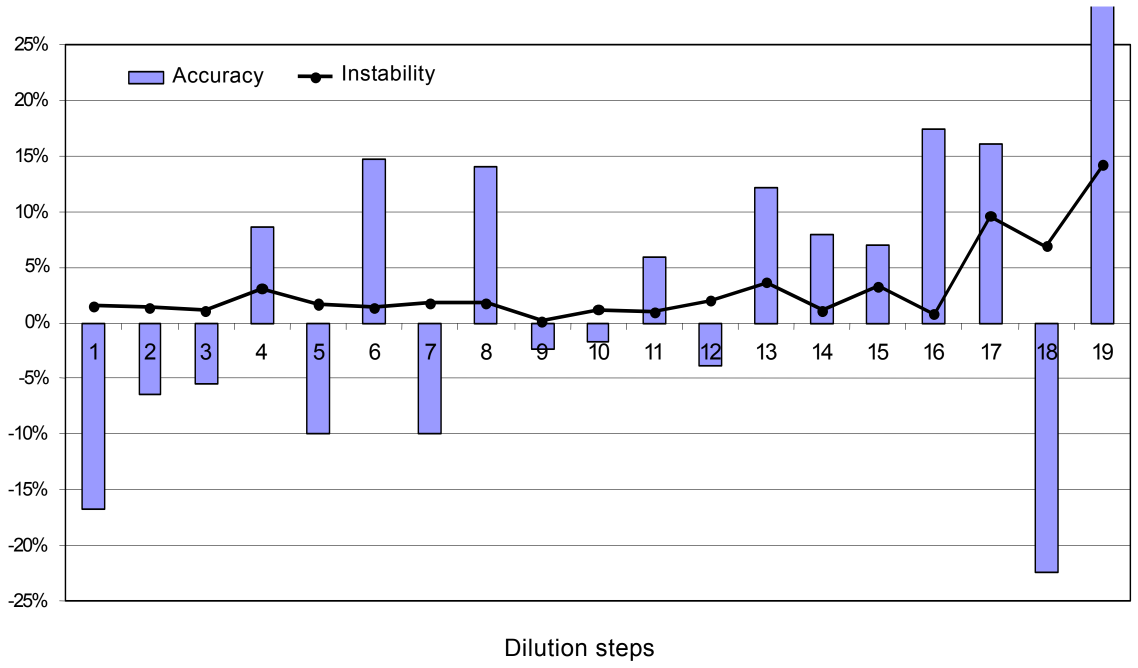

Figure 4 Accuracy and instability of the dynamic olfactometer

4. CONCLUSIONS

Instrumental calibration is a fundamental issue for dynamic olfactometers in addressing uncertainty. Unfortunately, the calibration of such instruments is both time consuming and labour intensive. It is understood that most laboratories around the world do not perform the calibrations of dilution ratios. Some olfactometers do not have a feature to allow the end-users to adjust the olfactometer.

The performance of the flow meter and MFC based olfactometers was found to degrade over time. This could be caused by the accumulation of dirt on the contact surfaces and the change of operating conditions. The calibration of a dynamic olfactometer should be carried out more frequently than once a year as suggested in the standard. A procedure should be in place to check the accuracy of the olfactometer frequently so that the performance of the olfactometer can be monitored. A full instrumental calibration should be carried out if necessary.

The study discussed several important aspects of instrumental calibration. The selection of sampling points during the calibration might produce different calibration results. Sampling within the sniffing cup is recommended since the arrangement is similar to the nose's position during the sniffing. The selection of CO gas concentrations can also affect the measured accuracy and instability. The fact that the calibration results are subject to the selection of the CO concentrations suggested that the use of CO to calibrate the olfactometer may not be the best technique. A more sensitive analytical instrument with sensitivity to a low ppb level should be used.

The instrumental performance of the DynaScent olfactometer has been demonstrated by CO gas calibration. Overall, the DynaScent olfactometer could achieve an averaged instability of 1.7% and an averaged accuracy of 9% over the dilution range of 2 – 65000 (step 1 – step 16).

References

1) Jiang, J., What are the areas for further research? Water Science & Technology, Vol 44 No 9 pp 17-25 © IWA Publishing 2001
2) Berglund, B., Berglund, U. and Lindvall, T. (1987). Quality assurance in olfactometry, in the proceedings of "*Volatile emissions from livestock farming and sewage operations*" edited by Nielsen V.C., Voorburg J. H. and L'Hermite P.; Uppsala, Sweden, 10-12 June 1987, 12-25
3) Department of Biosystems and Agricultural Engineering, the University of Minnesota, Last updated 12/13/3902, http://www.bae.umn.edu/annrpt/1998/ research/lvstk2.html
4) Jiang, J., Towards odour measurement standardisation on dynamic olfactometer, Proceedings of the National Workshop on Odour Measurement Standardization, August 20-22 1997, Sydney, Australia, pp 33-41
5) van Harreveld, A. P., Heeres, P. and Harssema, H. (1999), A Review of 20 Years of Standardization of Odor Concentration Measurement by Dynamic Olfactometry in Europe, J. Air & Waste Management. Assoc. Vol. 49, pp. 705-715
6) Australian and New Zealand Standard, AS 4323.3, 2001, Stationary source emissions: Part 3: Determination of odour concentration by dynamic olfactometry

7) CEN (Comitté Européen de Normalisation), 2001, Standard prEN 13725, "Air Quality – Determination of odour concentration by dynamic olfactometry"
8) Hangartner, H., Hartung, J. and Voorbury, J. H. (1985). Recommendations of olfactometric measurements. *Environmental Technology Letters*, Vol. 6, 415-420
9) Hangartner, M., Hartung, J., Paduch, M., Pain, B. F. and Voorbury, J. H. (1989). Improved recommendations of olfactometric measurements, *Environmental Technology Letters*, 10, 231-236

Measurement of Odor Threshold by Triangle Odor Bag Method

Yoshio Nagata
（Japan Environmental Sanitation Center）
10-6 yotuyakamichou kawasaki-ku kawasaki-si kanagawa-ken
E-mail : nagata-y@ jesc.or.jp

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Odor threshold , Triangle odor bag method , Reproducibility

Abstract

The detection thresholds of odor substances analyzed in field investigations were measured by the triangle odor bag method(1). The number of substances used for the experiment is 223. The experiment was carried out from 1976 to 1988.
As the results of the experiments, the odor thresholds were distributed over the concentration of large range depending on the odor substances. Isoamyl mercaptane exhibited the lowest threshold (0.77ppt), and propane exhibited the highest threshold (1500ppm). The distribution of thresholds expresses the normal distribution. Sulfur compounds with the exception of sulfur dioxide and carbon disulfide have the comparatively low threshold. It is showed the tendency that threshold becomes low as the increase of molecular weight in a certain range of molecular weight.
When the dispersion of odor thresholds for the same substance was shown at the ratio of the highest to the lowest odor threshold tested, the dispersion of odor thresholds was about 5 at the maximum. The thresholds of 223 substances measured by our laboratory were considered to be the average values with small bias comparatively.

1. Introduction

The thresholds were needed also in the evaluation based on instrumental measuring method, and also in the evaluation based on olfactory measuring method in odor studies. On that occasion, the data of the threshold by the foreign researcher, for example, Leonardos et al. (53 substances)(2) or Hellman et al. (101substances)(3), has greatly been made reference in Japan. But, the thresholds of substances that aren't reported to these literatures are also needed. And, a threshold may vary considerably in the difference of measuring method to the same material. Therefore, the need to measure thresholds individually is arising. The detection thresholds of 223 substances detected in various odor sources were measured in our laboratory by the triangle odor bag method(4).

2. Odorants and experimental method

2.1 Preparation of primary odor sample

The standard gas such as the sulfurous acid gas taken from the standard gas bomb was injected in polyester bag filled with nitrogen gas using gastightsyringe. In case the reagent was liquid, the primary odor sample was prepared by vaporizing, after it was injected in polyester bag filled with nitrogen gas with microsyringe. And in case the reagent was a solid like Skatole, the sublimation gas was collected in the bag. The odor samples were left for 2 hours or more in order to stabilize their gas concentration.

2.2 Concentration measurement of primary odor sample

Ammonia was measured by indophenol method, diosmin, skatole, indole were measured by gas chromatography-mass spectrometry. Other odorants were measured by gas chromatography (FID, FPD, FTD). In case of the standard gas such as sulfur dioxide, the concentration displayed on the bomb were used.

2.3 Measurement of odor concentration , and odor panel

The odor concentration was measured by the triangle odor bag method. In the triangle odor bag method, the threshold is obtained by detecting the difference from odor-free background. Therefore, the odor thresholds reported are nearly equal to the detection threshold. The measurement of the threshold was carried out in 12 years from 1976 to 1988 (Figure 1). An odor panel consists of 6 panelists. All panelists have passed the panel screening test by T&T olfactometer. Their ages are 50-year-old from 20-year-old. Some panelists changed in these 12 years. However, four persons (woman) among 6 panelists are the panelists from the first time. All panelists are trained.

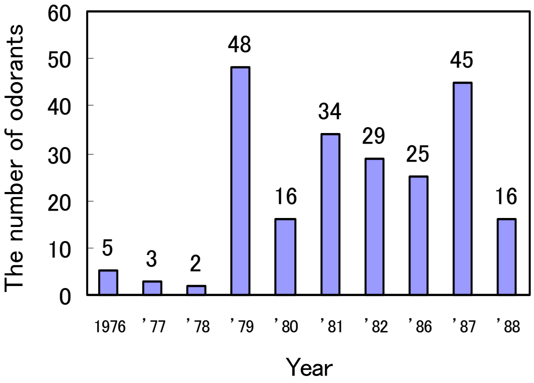


Figure 1 Carried-out year and the measured number of substances

2.4 Calculation of threshold value

In this examination, the value which divided the concentration of the primary odor sample by the odor concentration as a principle was determined as the detection threshold (ppm,v/v).

detection threshold (ppm,v/v) =
the concentration of primary odor sample/ odor concentration

Table1 Dilution error of the odor bag

Substance	Primary odor (ppm)	Dilution multiple	Recovery rate %
Hydrogen sulfide	20	10 ～ 300	102
Methyl mercaptane	10	10 ～ 300	93
Dimethyl sulfide	80	10 ～ 300	100
n -Hexane	600	10 ～ 1000	98
Toluene	900	10 ～ 1000	94
n-Nonane	800	10 ～ 1000	93
o,m,p - Xylene	23	10 ～ 1000	99
Styrene	22	10 ～ 1000	105
Ammonia	1100	10 ～ 1000	95
Trimethylamine	5.0	10 ～ 3000	50
〃	5.0	30 ～ 3000	93*
〃	0.02	30	13
Propionaldehyde	6.7	100 ～ 300	82
Isobutylaldehyde	6.6	100 ～ 300	82
n-Valeraldehyde	4.6	100 ～ 300	83
n-Butyric acid	0.3	10 ～ 30	40
Isobutyric acid	83	1000 ～ 3000	35
Isovaleric acid	0.5	10 ～ 30	39
Indole	1.8	30 ～ 100	6.5
Skatole	1.7	30 ～ 3000	13

* The injector made from a plastic was used.
The glass injector was used in the result of others.

As shown in Table 1, about the odorants such as amines, fatty acids, skatole and indole, since the dilution error was large compared with other substances, their thresholds were corrected by their recovery rate. About the odorants of which the thresholds were measured repeatedly, the geometric mean of each observed value was taken as the threshold of the odorant.

3. Result of threshold measurement

The thresholds of 223 odorants measured in the experiment are shown in the Table 2. The thresholds in the wide range of about 2 billion times to 1500ppm (propane) from 0.77ppt (Isoamyl mercaptane) were observed.

3.1 Comparison with the measurement results of odor intensity by the odorless chamber method

About 53 offensive odor substances, the relation between odor intensity (6-points scale) and the concentration of odor substance was observed in our laboratory[5)]. The odorless chamber of 4 m^3 was used for the experiment. As for 51 of 53 substances, the threshold of each substance was determined also by the triangle odor bag method. Then, the threshold determined by the triangle odor bag method was substituted for the relational expression between the concentration of odorant and odor intensity, and the threshold was converted into odor intensity. As the calculated results, the average value of the odor intensity equivalent of each substance was almost scale 1 of odor intensity. Scale 1 of odor intensity corresponded to the detection threshold. Both the measuring methods are based on the air dilution method, and the thresholds observed by both methods agreed in many substances approximately.

3.2 Distribution of thresholds for chemical compounds

The histogram of Figure 2 shows the distribution of the thresholds of compounds, such as sulfur compounds and oxygenated compounds, etc. The distribution of thresholds expresses the normal distribution. As shown in this figure, the thresholds are distributed in a wide range of concentration depending on the odor substances and compounds. The top of the distribution of the threshold was 10ppt～1ppb as for the sulfur compounds, 1ppb～10ppb as for the oxygenated compounds, 10ppb ～ 100ppb as for the nitrogen compounds, 100ppb～1ppm as for the hydrocarbon and 1ppm～10ppm as for the chlorine compounds. Sulfur compounds with the exception of sulfur dioxide and carbon disulfide have the comparatively low threshold.

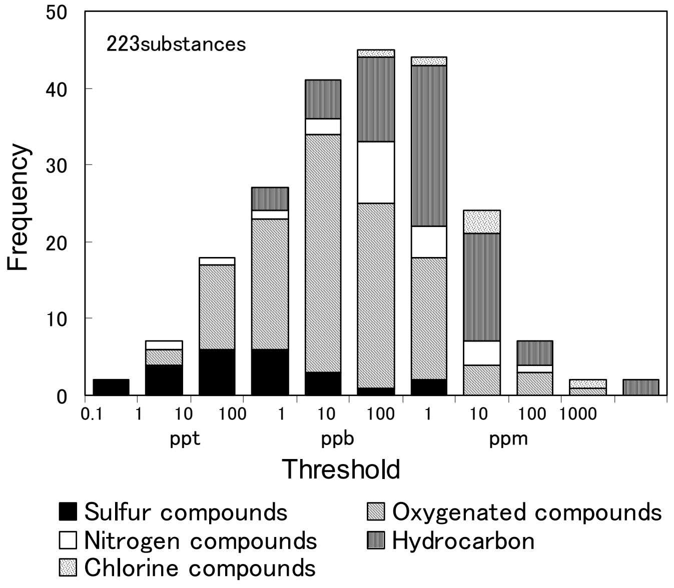


Figure 2 Distribution of thresholds for compounds

3.3 Relation between threshold and Molecular Weight

Although a clear tendency is not recognized on the whole, there is the tendency that the threshold decreases as the increase of molecular weight in the range to 120-130 as molecular weight (Figure 3).

Further that tendency becomes clear when it is observed in the homologous series.

In most case of homologous series in the chemical compounds such as alcohol (Figure 4), aldehyde, mercaptan, ketone and hydrocarbon, it is showed the tendency that threshold becomes low as the increase of molecular weight in a certain range of molecular weight.

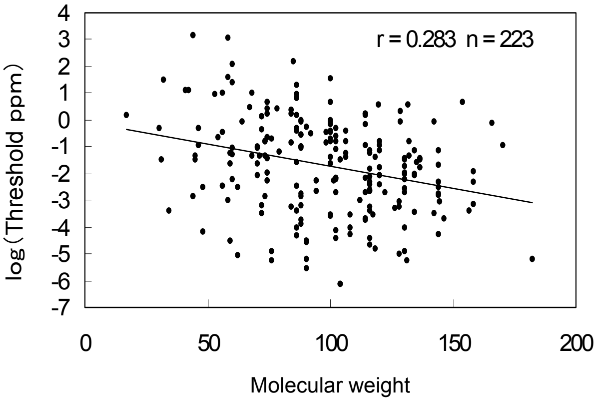


Figure 3 Relation between threshold and molecular weight

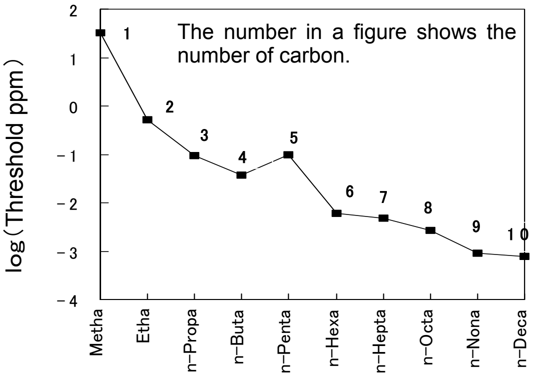


Figure 4 Thresholds of Aliphatic alcohols (Homologous series)

3.4 Difference of the threshold between isomers

It is further found that a great difference in the thresholds between isomers. When the functional group is different such as aldehyde and ketone, fatty acid and ester, it is not rare that the thresholds are different about 10000 times between isomers. Moreover, the thresholds may be different even between position isomerism more than 100 times (Figure 5).

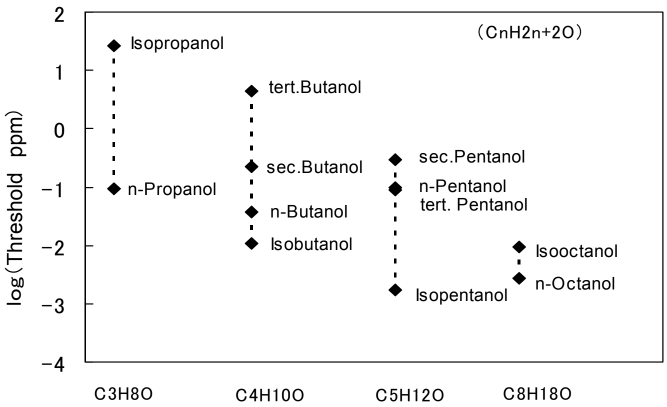


Figure 5 Thresholds of Aliphatic alcohols (Between isomers)

Table 2 Odor thresholds measured by the triangle odor bag method (ppm,v/v)

Substance	Odor Threshold	Substance	Odor Threshold
Formaldehyde	0.50	Hydrogen sulfide	0.00041
Acetaldehyde	0.0015	Dimethyl sulfide	0.0030
Propionaldehyde	0.0010	Methyl allyl sulfide	0.00014
n-Butylaldehyde	0.00067	Diethyl sulfide	0.000033
Isobutylaldehyde	0.00035	Allyl sulfide	0.00022
n-Valeraldehyde	0.00041	Carbon disulfide	0.21
I sovaleraldehyde	0.00010	Dimethyl disulfide	0.0022
n-Hexylaldehyde	0.00028	Diethyl disulfide	0 0020
n-Heptylaldehyde	0.00018	Diallyl disulfide	0.00022
n-Octylaldehyde	0.000010	Methyl mercaptane	0.000070
n-Nonylaldehyde	0.00034	Ethyl mercaptane	0.0000087
n-Decylaldehyde	0.00040	n-Propyl mercaptane	0.000013
Acrolein	0.0036	Isopropyl mercaptane	0.0000060
Methacrolein	0.0085	n-Butyl mercaptane	0.0000028
Crotonaldehyde	0.023	Isobutyl mercaptane	0.0000068
Methanol	33	sec. Butyl mercaptane	0.000030
Ethanol	0.52	tert. Butyl mercaptane	0.000029
n-Propanol	0.094	n-Amyl mercaptane	0.00000078
I sopropanol	26	Isoamyl mercaptane	0.00000077
n-Butanol	0.038	n-Hexyl mercaptane	0.000015
I sobutanol	0.011	Thiophene	0.00056
sec.Butanol	0.22	Tetrahydrothiophene	0.00062
tert.Butanol	4.5	Nitrogen dioxide	0.12
n-Pentanol	0.10	Ammonia	1.5
Isopentanol	0.0017	Methylamine	0.035
sec.Pentanol	0.29	Ethylamine	0.046
tert. Pentanol	0.088	n-Propylamine	0.061
n-Hexanol	0.0060	Isopropylamine	0.025
n-Heptanol	0.0048	n-Butylamine	0.17
n-Octanol	0.0027	Isobutylamine	0.0015
Isooctanol	0.0093	sec. Butylamine	0.17
n-Nonanol	0.00090	tert. Butylamine	0.17
n-Decanol	0.00077	Dimethylamine	0.033
2-Ethoxyethanol	0.58	Diethylamine	0.048
2-n-Buthoxyethanol	0.043	Trimethylamine	0.000032
1-Butoxy-2-propanol	0.16	Triethylamine	0.0054
Phenol	0.0056	Acetonitrile	13
o-Cresol	0.00028	Acrylonitrile	8.8
m-Cresol	0.00010	Methacrylonitrile	3.0
p-Cresol	0.000054	Pyridine	0.063
Geosmin	0.0000065	Indole	0.00030
Acetic acid	0.0060	Skatole	0.0000056
Propionic acid	0.0057	Ethyl-o-toluidine	0.026
n-Butyric acid	0.00019	Propane	1500
Isobutyric acid	0.0015	n-Butane	1200
n-Valeric acid	0.000037	n-Pentane	1.4
Isovaleric acid	0.000078	Isopentane	1.3
n-Hexanoic acid	0.00060	n -Hexane	1.5
Isohexanoic acid	0.00040	2-Methylpentane	7.0
Sulfur dioxide	0.87	3-Methylpentane	8.9
Carbonyl sulfide	0.055	2, 2-Dimethylbutane	20

Table 2 Odor thresholds measured by the triangle odor bag method (ppm,v/v) (continued)

Substance	Odor Threshold	Substance	Odor Threshold
2, 3-Dimethylbutane	0.42	Ethyl acetate	0.87
n-Heptane	0.67	n-Propyl acetate	0.24
2-Methylhexane	0.42	Isopropyl acetate	0.16
3-Methylhexane	0.84	n-Butyl acetate	0.016
3-Ethylpentane	0.37	Isobutyl acetate	0.0080
2, 2-Dimethylpentane	38	sec.Butyl acetate	0.0024
2, 3-Dimethylpentane	4.5	tert.Butyl acetate	0.071
2, 4-Dimethylpentane	0.94	n-Hexyl acetate	0.0018
n-Octane	1.7	Methyl propionate	0.098
2-Methylheptane	0.11	Ethyl propionate	0.0070
3-Methylheptane	1.5	n-Propyl propionate	0.058
4-Methylheptane	1.7	Isopropyl propionate	0.0041
2, 2, 4-Trimethylpentane	0.67	n-Butyl propionate	0.036
n-Nonane	2.2	Isobutyl propionate	0.020
2, 2, 5-Trimethylhexane	0.90	Methyl n-butyrate	0.0071
n-Undecane	0.87	Methyl isobutyrate	0.0019
n-Decane	0.62	Ethyl n-butyrate	0.000040
n-Dodecane	0.11	Ethyl isobutyrate	0.000022
Propylene	13	n-Propy n-butyrate	0.011
1-Butene	0.36	Isopropyl n-butyrate	0.0062
Isobutene	10	n-propyl isobutyrate	0.0020
1-Pentene	0.10	Isopropyl isobutyrate	0.035
1-Hexene	0.14	n-Butyl n-butyrate	0.0048
1-Heptene	0.37	Isobutyl n-butyrate	0.0016
1-Octene	0.0010	n-Butyl isobutyrate	0.022
1-Nonene	0.00054	Isobutyl isobutyrate	0.075
1,3-Butadiene	0.23	Methyl n-valerate	0.0022
Isoprene	0.048	Methyl isovalerate	0.0022
Benzene	2.7	Ethyl n-valerate	0.00011
Toluene	0.33	Ethyl isovalerate	0.000013
Styrene	0.035	n-Propyl n-valerate	0.0033
Ethylbenzene	0.17	n-Propyl isovalerate	0.000056
o-Xylene	0.38	n-Butyl isovalerate	0.012
m-Xylene	0.041	Isobutyl isovalerate	0.0052
p-Xylene	0.058	Methyl acryrate	0.0035
n-Propylbenzene	0.0038	Ethyl acryrate	0.00026
Isopropylbenzene	0.0084	n-Butyl acryrate	0.00055
1, 2, 4-Trimethylbenzen	0.12	Isobutyl acryrate	0.00090
1, 3, 5-Trimethylbenzen	0.17	Methyl methacryrate	0.21
o-Ethyltoluene	0.074	2-Ethoxyethyl acetate	0.049
m-Ethyltoluene	0.018	Acetone	42
p-Ethyltoluene	0.0083	Methyl ethyl ketone	0.44
o-Diethylbenzene	0.0094	Methyl n-propyl ketone	0.028
m-Diethylbenzene	0.070	Methyl isopropyl ketone	0.50
p-Diethylbenzene	0.00039	Methyl n-butyl ketone	0.024
n-Butylbenzene	0.0085	Methyl isobutyl ketone	0.17
1, 2, 3, 4-Tetramethylbenzen	0.011	Methyl sec.butyl ketone	0.024
1, 2, 3, 4-Tetrahydronaphthalene	0.0093	Methyl tert.butyl ketone	0.043
α-Pinene	0.018	Methyl n-amyl ketone	0.0068
β-Pinene	0.033	Methyl isoamyl ketone	0.0021
Limonene	0.038	Diacetyl	0.000050
Methylcyclopentane	1.7	Ozone	0.0032
Cyclohexane	2.5	Furane	9.9
Methylcyclohexane	0.15	2, 5-Dihydrofurane	0.093
Methyl formate	130	Chlorine	0.049
Ethyl formate	2.7	Dichloromethane	160
n-Propyl formate	0.96	Chloroform	3.8
Isopropyl formate	0.29	Trichloroethylene	3.9
n-Butyl formate	0.087	Carbon tetrachloride	4.6
Isobutyl formate	0.49	Tetrachloroethylene	0.77
Mthyl acetate	1.7		

4. Precision and accuracy of the measurement results of the threshold

4.1 Reproducibility-within-laboratory (the result measured by our laboratory)

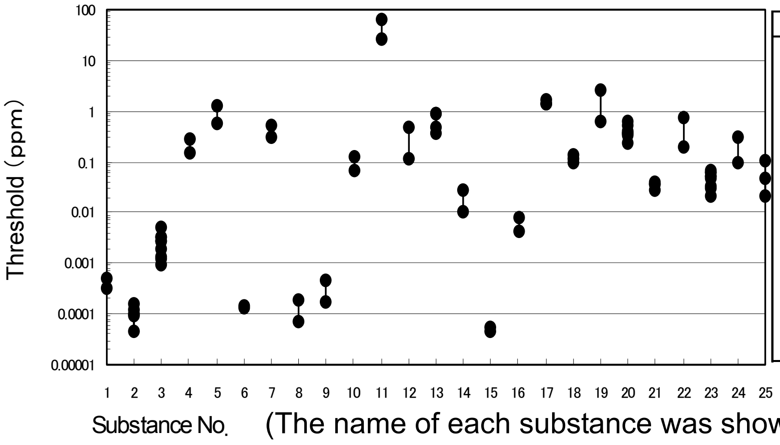
It was thought that the odor thresholds would vary because of the difference in the measuring method and the attribute of odor panel, etc.

The measurement of the threshold of each odor substance was carried out on separate days. The measuring instruments used on each test were the same. 4 persons in panel member of 6 persons are same during the measurement period. About some substances, the measurements of the threshold have carried out after ten years or more have passed since the first measurement. Though the measurements for many of prepared substances were carried out only once. But the measurements were carried out twice or more per substance about 25 substances of 223 substances.

Figure 6 shows that variation of odor thresholds for repeated tests on the same substances. The sensory tests were carried out on separate days. And, the dispersion of odor thresholds for the same substance was shown at the ratio of the highest to the lowest odor threshold tested, and it was shown in Table 3. Though the number of repetitions is different with substance from 2 times to 9 times, the dispersion of odor thresholds was about 5 at the maximum.

Table 3 Variation of thresholds on the same substances

The number of times of measurement	The number of substances	Ratio of the highest to the lowest threshold
2	１６	１．２～４．０
3	３	１．２～５．０
4	２	１．５，２．４
5	１	２．６
7	１	３．０
9	２	３．０，５．２



No.	Substance	No.	Substance
1	Hydrogen sulfide	14	n-Butyl acetate
2	Methyl mercaptane	15	Diacetyl
3	Dimethyl sulfide	16	Acetic acid
4	Carbon disulfide	17	Ammonia
5	Sulfur dioxide	18	Nitrogen dioxide
6	Methyl allyl sulfide	19	Isopentane
7	Formaldehyde	20	Toluene
8	I sovaleraldehyde	21	Styrene
9	n-Hexylaldehyde	22	o-Xylene
10	n-Propanol	23	m-Xylene
11	Isopropanol	24	Ethylbenzene
12	sec.Butanol	25	Chlorine
13	Ethyl acetate		

(The name of each substance was shown in the right table.)

Figure 6 Result of repeated tests on the same substances by trained panel.

4.2 Reproducibility-within-laboratory (the results of the practices in the Environment training center where these are carried out once a year)

We have held the training session of the sensory test method for inexperienced person once a year since 1983. The thresholds of hydrogen sulfide, m-xylene and ethyl acetate were measured during the practical training. The measurements were carried out in the same place every year. The measuring instruments used on each test were also the same. Operators and panel members are untrained persons and are changed every year. The results are shown in Table 4 and Figure 7.

When the results by the untrained panel were compared with the results by the trained panel, the significant difference was not recognized on mean value and dispersion of the thresholds[6)]. The untrained panel members are considered to have got used to the sensory test through the panel screening test and the preliminary practice of the triangle odor bag method before the measurement of the thresholds.

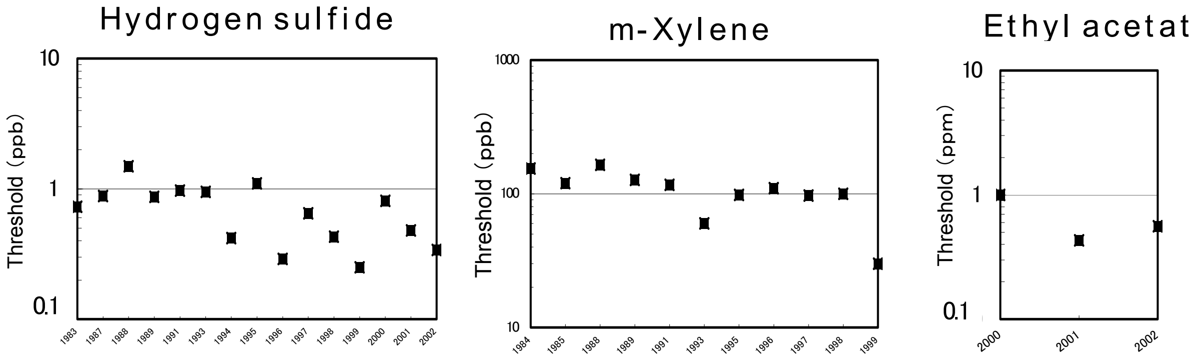


Figure 7 Result of odor thresholds on the same substances (Untrained persons carried out the measurements once per year.)

Table 4 Variation of odor thresholds on the same substances (from Figure 7)

Substance	carried-out year	The number of panelist	The number of times of measurement	Ratio of the highest to the lowest threshold	Geometric mean
Hydrogen sulfide	1983 ～ 2002	6 ～ 16	15	6.0	0.63 ppb
m-Xylene	1984 ～ 1999	6 ～ 16	11	5.5	99 ppb
Ethyl acetate	2000 ～ 2002	11 ～ 12	3	3.2	0.62 ppm

4.3 Reproducibility by inter-laboratory test

In 1985, inter-laboratory comparison test by the triangle odor bag method was carried out. 5 odor laboratories including our laboratory participated in the test. The results are shown in Figure 8 and Table 5. m-Xylene and dimethyl sulfide were chosen as the reference materials for sensory test. The sample no.1,2,3,4 are m-xylene of which the concentration differs, and the sample no.5,6,7 are dimethyl sulfide of which the concentration differs.

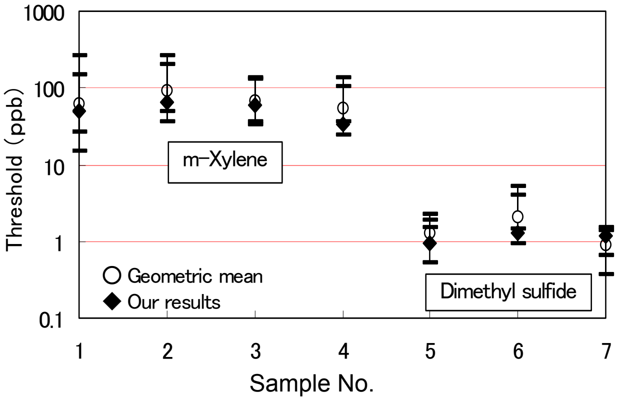


Figure 8 Results of inter-laboratory test by 5 laboratories

The dispersion of the measurement results was shown the ratio of highest to lowest odor threshold measured by each laboratory. The dispersion of the thresholds between 5 laboratories was as large as 18 in the sample no.1 that was measured first. And, the dispersion of other 6 samples was less than 8. When the measurement results of 2 laboratories which have a few measurement experience are removed, the dispersions are less than 5 every sample.

Table 5 Dispersion of thresholds measured by 5 laboratories on the same substances (from Figure 8)

Sample	Substance	Ratio of the highest to the lowest thre shold	Geometoric mean	
			Every sample	Every substance
1	m-Xylene	18	61 ppb	67 ppb
2		7.4	92 ppb	
3		4.2	67 ppb	
4		5.6	53 ppb	
5	Dimethyl sulfide	4.3	1.3 ppb	1.3 ppb
6		5.6	2.0 ppb	
7		4.2	0.9 ppb	

4.4 Accuracy of the thresholds measured by our laboratory

(1) In 2002, the inter-laboratory test was carried out in order to raise the accuracy of the triangle odor bag method. A total of 137 odor laboratories in Japan participated in the test. In the test, the threshold of ethyl acetate was measured7). As the result measured by 137 laboratories, the mean value of the threshold of ethyl acetate was 0.89 ppm. The threshold of ethyl acetate measured by our laboratory－0.87 ppm (the measured value in 1979) is almost the same as this value.

(2) As shown in Figure 8, in the inter-laboratory test by 5 laboratories, the threshold measured by our laboratory is 0.6 times to 1.3 times of the geometric mean, almost near the average value.

(3) In Europe, the dynamic olfactometry has been standardized as the measuring method of odor concentration, and it has been reported that the threshold of n-butanol measured by this method was approximately 40 ppb8). We had reported that the threshold of n-butanol measured by the triangle odor bag method was 38 ppb (the measured value in 1980). Although measuring method is different, both of results are almost the same.

From these results, the thresholds of 223 substances measured by our laboratory are considered to be the average values with small bias comparatively.

5. Conclusion

Although the threshold values shown in this report were reported 15 years ago, but the remarkable differences from the reported values are not seen in the latest remeasurement results. So, I was sure of the practicality of the triangle odor bag method anew.

References

1) Iwasaki,Y. and Ishiguro,T. : Measurement of odor by triangle odor bag method (I) : Japan Society Atmospheric Environment ,(1978),13(6),pp.34-39

2) Leonardos,G.,D.,Kendall and N.Barnard : Odor threshold determination of 53 odorant chemicals,J.of APCA.,(1969),19(2),pp.91-95

3) Hellman,T.M.and F.H.Small : Characterization of the odor properties of 101 petrochemicals

using sensory method,J.of APCA., (1974), 24 (10), pp.979-982

4) Nagata,Y. and Takeuchi,N. : Measurement of odor threshold by triangle odor bag method, Bulletin of Japan Environmental Sanitation Center, (1990), 17, pp. 77-89

5) Nagata,Y. and Takeuchi,N. : Relationship between concentration of odorants and odor intensity , Bulletin of Japan Environmental Sanitation Center, (1980), 7, pp. 75-86

6) Nagata,Y. and Takeuchi,N. : A report on sensory measurement of odor in exercise at National Environmental Training Institute , Bulletin of Japan Environmental Sanitation Center,(1996),23,pp.67-79

7) Fukuyama,Jyoji : Evaluation of a cross-check examination result, Odor Research and Engineering Association of Japan, (2003), pp.37-49

8) Ishikawa,Y. and Nishida, K. (translation) : A Review of 20 years of standardization of odor concentration measurement by dynamic olfactometry in europe, Journal of odor research and engineering, (2000) ,31(3),pp.6-13

Development of the Odormat for Sewage Odour Measurement

Lawrence C C KOE
Director, Environmental Engineering Research Centre
School of Civil & Environmental Engineering
Nanyang Technological University
Block N1, #B3b-18
50, Nayang Avenue
Singapore 639798

Jurn Wei CHAI
Aromatrix Pte Ltd
1 Bukit Batok Street 22, #03-02
GRP Industrial Building
Singapore 659592

Keywords
Olfactometer, odour, odor measurement

Abstract

With today's increasing levels of development, residential areas are inevitably built closer to odour generating facilities, such as sewage treatment and chemical industrial plants. Odour measurements, which provide important information in the planning of the plants and odour treatment facilities, are needed to underpin the numerous decisions that will have to be made to reduce odour nuisance. The use of dynamic olfactometry is widely regarded as a favoured sensory technique for quantifying odour. In recent years, emphasis has been directed towards standardizing equipment and techniques of odour sampling and measurement so that results can be compared between laboratories and between different olfactometers with some degree of reliability. This paper describes the development of a computerized olfactometer for sewage odour measurement in Singapore.

1. INTRODUCTION

The increasing standard of living in Singapore over the last 30 years has resulted in an increasing demand for a cleaner and healthier living and working environment. Due to the scarcity of land resources, parts of the residential and commercial areas are located in close proximity of the odour generating zones, such as the sewage treatment plants. Dynamic olfactometry has played a key role in helping environmental engineers to understand odorous emission characteristics of the plants so that proper odour treatment systems can be designed to contain malodours within the boundaries of the plants. Odour inventory study using dynamic olfactometry has become a common and regular practice in the maintenance and evaluation of odour treatment facilities.

The strength or concentration of an odour sample is measured by the number of dilutions with odour-free air required to render it barely detectable by an odour observer. Odour concentration is typically expressed in terms of odour units per cubic metre of odour-free air^2 (ou/m^3) (1). This is the amount of odour necessary to contaminate 1 m^3 of clean, odour-free air to the threshold level of the observer (2). The number of dilutions to threshold, otherwise known as the dilution factor, Z, is computed as:

$$Z = \frac{(F1 + F2)}{F2} \tag{1}$$

where F1 = flow of odour-free air
F2 = flow of odorous air.

Both F1 and F2 are expressed in units of volume or flowrates. Hence Z is a dimensionless ratio (3). However, Z is numerically equal to the term “ou/m^3” since the number of dilutions to threshold level is a measure of the odour concentration. The device used to deliver the diluted odour sample to the odour observers is known as an olfactometer.

2. METHODOLOGY

In 1984, a manual dynamic olfactometer was developed under the supervision of Professor Lawrence Koe at the National University of Singapore. It works on the principle of dynamic dilution by continuously increasing the flowrates of an odorous sample with odour-free air until the mixture reaches its threshold odour level and becomes detectable by an odour observer. The first dynamic olfactometer is largely made up of manual components as shown in Figure 1, such as stainless steel rotameters and air valves, which require the operator to adjust and monitor the gas flowrates during odour measurement. With its unique dilution technique, the manual dynamic olfactometer was built to deliver high dilution rates to measure the high odour concentrations of foul gases typically found in sewage treatment plants and food and chemical industries in the excess of thousands of odour units.

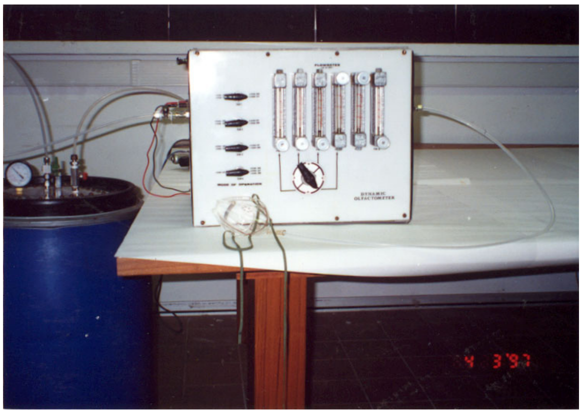


Figure 1. Manual dynamic olfactometer

During odour measurement, odorous air is first collected and contained in chemically inert sampling bags with about 10- to 40- litre capacity. The actual odour contents in the bag are preserved by subjecting the sampling bag to pressure in a special vessel, resulting in direct delivery of the sample to the olfactometer without passing through any air pumps. Odorous air sample is dynamically diluted with continuous odour-free air and the mixture is presented to the odour observer at a constant flowrates of 20 litres per minute. The concentration of the odorous air is gradually increased until the odour threshold becomes detectable by an odour observer. The odour concentration of the odorous sample is expressed as the number of dilutions with odour-free air required to render it detectable by the odour observers.

Over the years, the performance and features of the first manual dynamic olfactometer has become inadequate in meeting the more stringent needs for greater accuracy and reliability in odour measurement. In addition, emphasis has been directed towards standardizing equipment and techniques of odour sampling and measurement so that results can be compared between laboratories and between different olfactometers with some degree of reliability. Harreveld (4) and O'Brien et al. (5) highlighted the performance of olfactometers as one of the factors of olfactometry that is required to be standardized. In 1990, the European Committee for Standardization (CEN) formed a technical committee (TC264), which developed a draft standard for olfactometery.

In 1997, the manual dynamic olfactometer was upgraded as shown in Figure 2. The version II manual dynamic olfactometer was developed with more accurate rotameters to enable odour concentrations of air samples to be measured in a high quality controlled procedure such that results are reliable and accurate.

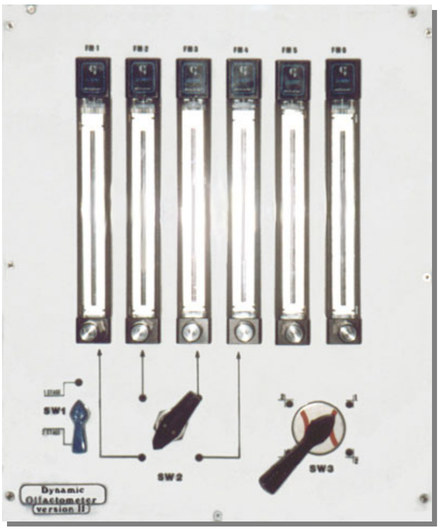


Figure 2. An upgraded version II dynamic olfactometer

Following the successful development of the version II dynamic olfactometer, a computerised dynamic olfactometer, named the Odormat was designed and built to ease the process of odour evaluation with a fully integrated automation to minimize human errors. The Odormat was developed using the unique technology of the first manual dynamic olfactometer as shown in Figure 3 and it conformed to the CEN standard. It was designed with large dilution range of 2^2 – 2^{18}, which allowed odours that are encountered in most odours generating industries, such as sewerage treatment plants, food and chemical processing factories to be accurately measured without the need to pre-dilute the odorous air sample.

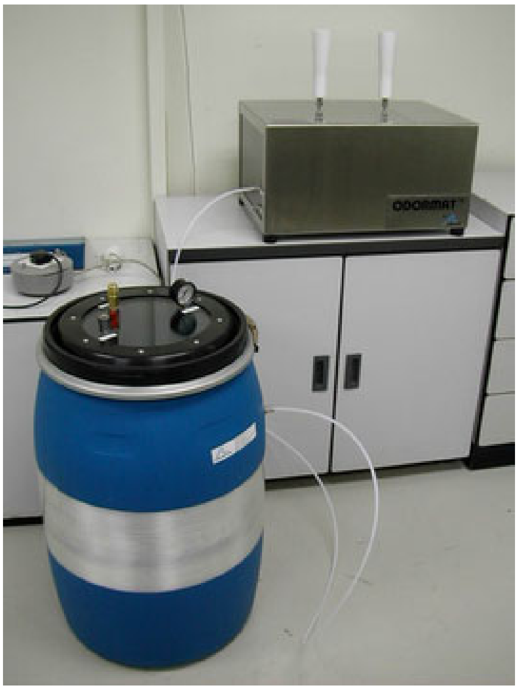


Figure 3. Computerised dynamic olfactometer (Odormat)

The Odormat was constructed of components entirely made of stainless steel and polytetrafluoroethylene (PTFE). It is capable of delivering dilutions in the range of 2^2 to 2^{18}. The CEN has specified a minimum upper limit of 2^{14} and a maximum lower limit of 2^7 (4). Odours are presented to human observers utilizing dynamic olfactometry following a “force-choice” ascending concentration series method. In this method, the odour observers are presented with a diluted odour sample and one blank sample of odour-free air. The observers must choose which sample contains the odour, even if they must guess. This approach is called the “forced-choice” presentation method. After the observers make a selection, they are presented with the next set of odour sample and blank. However, this next odour sample is at a higher concentration. The observers continue with additional higher levels of sample presentation following these methods. This statistical approach of increasing levels of sample presentation is called “ascending concentration series”.

The most significant variable in the delivery of the odour stimulus to the nose is the flow rate from the olfactometer and the face velocity of the air. The Odormat operates in accordance to the CEN standard at a presentation flow rate of 20 litres/min with a face velocity of 0.5 m/s. The odour observers are selected strictly based on the observers’ sensitivity towards a standard reference odorant as specified in the CEN standard. Each potential observer 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. The antilog of the standard deviation must be less than 2.3 (4).

A set of stringent laboratory accuracy and repeatability performance criteria was specified in CEN standard. Harreveld (4) has proposed a set of criteria for the Accuracy (A) and Repeatability (r) in the CEN standard for olfactometry:

- $A < 0.217$
- $r < 0.477$

To verify the accuracy and repeatability of the odour measurement conducted with the three dynamic olfactometer, a series of odour evaluation using standard reference gas, n-butanol was carried out. A panel of qualified odour observers, in the age group of 18 – 26, was selected for the tests. Standard 18.8 ppm n-butanol was used as reference gas in the test. Ten odour measurements were conducted using the standard n-butanol gas over a period of one month.

3. RESULTS

Tables 1, 2 and 3 summarize the results of the odour performance evaluation using the standard 18.8 ppm n-butanol gas. The odour thresholds of standard 18.8 ppm n-butanol gas measured with the first dynamic olfactometer, version II dynamic olfactometer and the Odormat were found to be 471 ppb, 114 ppb and 46.6 ppb respectively.

Table 1. Summary of the performance evaluation of the first dynamic olfactometer

Reference gas	n-butanol
Mean Group threshold (ppb)	471
Log (threshold)	2.6939
Standard Deviation of Log (threshold)	0.0414
CEN threshold for n-butanol, ppb (4)	40
Repeatability Criteria: r < 0.477 ?	**0.107** **yes**
Accuracy, A Criteria: A < 0.217 ?	**1.116** **no**

Table 2. Summary of the performance evaluation of the version II dynamic olfactometer

Reference gas	n-butanol
Mean Group threshold (ppb)	114
Log (threshold)	2.0569
Standard Deviation of Log (threshold)	0.0747
CEN threshold for n-butanol, ppb (4)	40
Repeatability Criteria: r < 0.477 ?	**0.238** **yes**
Accuracy, A Criteria: A < 0.217 ?	**0.508** **no**

Table 3. Summary of the performance evaluation of the Odormat

Reference gas	n-butanol
Mean Group threshold (ppb)	46.6
Log (threshold)	1.6683
Standard Deviation of Log (threshold)	0.0867
CEN threshold for n-butanol, ppb (4)	40
Repeatability Criteria: r < 0.477 ?	**0.276** **yes**
Accuracy, A Criteria: A < 0.217 ?	**0.128** **yes**

The continuous research and development of the Odormat technology over the last 20 years has resulted in significant improvement on the performance and quality of odour measurement. The results of the repeatability and accuracy of odour measurement using the Odormat were found to be 0.276 and 0.128 respectively, which were well within the CEN requirement ($r < 0.477$ and $A < 0.217$).

4. CONCLUSIONS

The research and development of dynamic olfactometry in Singapore over the last 20 years has successfully resulted in developing a new generation of computerised dynamic olfactometer that measures odour concentration with improved accuracy and repeatability, which were tested to satisfy the stringent requirements in the CEN standard.

References

1) Koe, L.C.C.; Brady, D.K. J. Env. Eng., 1986, 112(2), 311-327
2) Arthur, C.S. Air Pollution: Measuring, Monitoring, and Surveillance of Air Pollution, Vol. 3, 3rd ed., pp. 335-336, Academic Press, 1976
3) Manual of Practice No. 22, Odour Control for Wastewater Facilities; Water Pollution Control Federation, Washington, D.C., 1979, pp 3-34
4) Harreveld, A.P., European standardisation of olfactometry, Project Research Amsterdam B.V., UK, May 1993
5) O'Brien, M.A, Guidelines for Odour Sampling and Measurement by Dynamic Dilution Olfactometry, Draft, AWMA EE-6 Subcommittee on the Standardisation of Odour Measurement, revised May 1993

Prospects for Development of Simplified Evaluation Methods used in Odor Management

Masahiro Osako
National Institute for Environmental Studies
16-2 Onogawa, Tsukuba, Ibaraki Pref. 305-8506, Japan
Tel +81-298-50-2835 Fax +81-298-50-2830 E-mail mosako@nies.go.jp

1. Introduction

As the functional requirements for odor evaluation methods differ according to the application purpose, not only is the selection of an adequate evaluation method important but so is its application to the corresponding purpose to appropriately conduct odor management. In this paper, odor evaluation methods were first divided and categorized according to the technical functions and application purposes. Emphasis was placed on the importance of “simplification” of the evaluation method to efficiently carry out odor management. Finally, it was proposed that an application concept using simplified odor evaluation methods, such as odor sensor systems including electric noses, detection tubes and basic olfactory measurements, should be designed to cost-effectively control odor problems.

2. Categories of odor evaluation methods

Odor evaluation methods could be categorized from various standpoints. In Japan, a typical category consists of instrumental and sensory methods, as shown in Fig. 1. The category for instrumental (machinery) methods includes gas chromatography (GC), gas chromatography/mass spectrometer (GC/MS), detection tube, devices for monitoring a specified constituent, and odor sensors. The latter three instrumental methods are regarded as simplified methods.

In the sensory, or olfactory, methods there is the triangular odor bag method as a legally designated method for regulation, the dynamic olfactometer as a standardized method in Europe and the subjectively direct indication for rating odor intensity or hedonic tone.

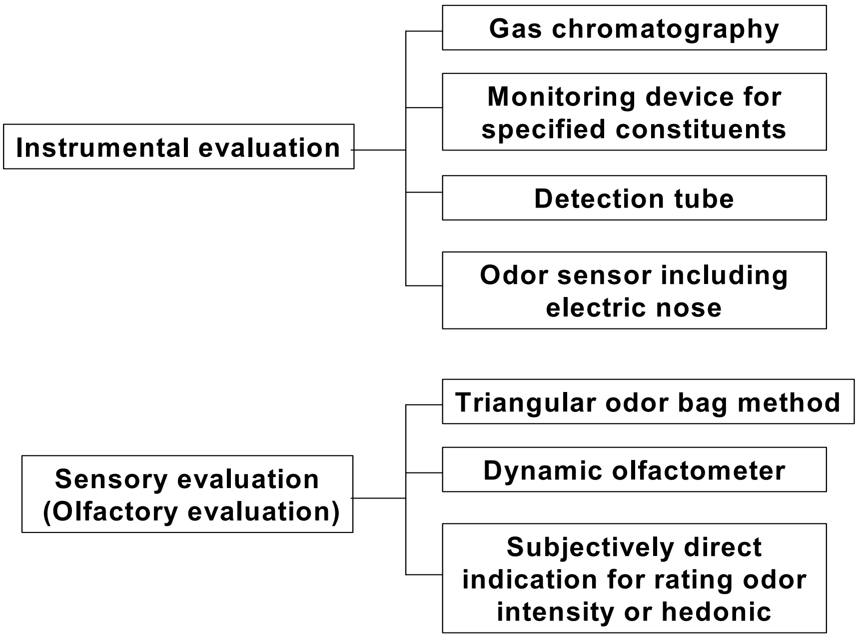


Figure 1 General categorization for odor evaluation methods

The sense of odor involves three principal factors: concentration of odorous substances, sensory odor intensity and odor quality including hedonic tone. Thus, the odor evaluation method can be categorized from these three aspects as shown in Table 1.

Table 1 Categorization of odor evaluation methods from the viewpoint of indicator objectives

Objective of indicator	Higher accuracy ⇔ Lower accuracy	
	Legally obligatory method	Simplified evaluation method
Concentration of individual constituent	Gas chromatography	Detection tube Monitoring device for specified constituents
Odor index, odor unit and odor intensity	Triangular odor bag method	Simplified olfactory methods Odor sensor including electric nose Sensory direct indication for odor intensity
Odor quality including hedonic tone		Sensory direct indication for odor hedonic tone Electric nose

On the other hand, it is possible to divide odor evaluation methods into three categories, as shown in Table 2, from the viewpoint of application purpose. According to Table 1, the three categories are legal evaluation for compliance, evaluation for voluntary management and evaluation for characterization of odor emission source. It is understandable that in each category, the requirements associated with accuracy and cost are different depending upon the application purpose. Therefore, a unified method that does not relate to the purpose should not be applied. We should have the rationality to select/utilize an evaluation method suitable to the purpose.

Table 2 Categorization of odor evaluation methods from the viewpoint of application purposes

Category	Outline	Examples of the evaluation method
Evaluation for compliance	Obligatory method based on the law. Necessary to ensure sufficient accuracy for judgment in legal action. Generally expensive method.	Triangular odor bag method, dynamic olfactometer, gas chromatography, etc.
Evaluation for voluntary management	Not obligatory. Use in voluntary management. Easier and cheaper methods are better. Highly accurate methods are not always necessary. Highly frequent monitoring is possible.	Detection tube, odor sensor, subjectively direct rating for intensity or hedonic tone, etc.
Evaluation for characterization of odor emission source	Characterization of time-dependent change of odor emission from the odor source, detailed composition of odorous constituents and contribution of constituents to sensory magnitude of odor.	Continuous monitoring using odor sensor, GC-olfactometer, GC/MS, electric nose

3. International movement on odor evaluation methods

In Japan, the instrumental method using GC was legally adopted due to concentration standards for regulation against individual constituents in complex odors, based on the Offensive Odor Control Law. It was confirmed, however, that the impact of complex odors could not be controlled using the individual constituent concentration as an indicator to reproduce the actual phenomena. Therefore, in 1995 additional regulation standards based on an “odor index” evaluated by olfactory measurement called “triangular odor bag method” was set up. In addition, the Ministry of the Environment recently carried out offensive odor controls emphasizing the odor index regulation.

On the other hand, from the beginning, European countries have conversely taken greater account of odor evaluation by the olfactory method using the dynamic olfactometer, and have adopted the “odor unit” as a standard unit corresponding to the so-called “odor concentration” in Japan. Standardization of the dynamic olfactometer method was recently accomplished in CEN.

The olfactory method would have a higher cost to ensure higher accuracy whereas it is the better method for reproducing the actual impact caused by complex odors.

Therefore, while the olfactory method is a basis of odor evaluation, odor emission control using odorous constituents as “reference odors” that correlate to the odor unit specified at each emission source, is performed for easy and cost-efficient management. For instance, at the sewage treatment plant hydrogen sulfide (H_2S) has been applied as a reference odor in order to control the emission. In addition, the feasibility of applying a kind of simplified monitoring system using plural sensors called “electric noses” has been studied in the field of odor control and so on.

In summary, simplification is progressing in order to attain a low-cost method, whereas olfactory evaluation has become a basis in the strategy for odor control, according to recent international trends.

4. Simplified evaluation method as a tool for odor control

In the future field of odor control, various evaluation methods from highly accurate to simplified measurements should be developed and applied according to the respective objectives. Administrative or academic authorities should propose not only a framework for the evaluation strategy but also details of the application program, including how to use various methods concretely. Figure 2 illustrates an example of a program for monitoring the odor emission source, consisting of the odor sensor method as a simplified evaluation and the olfactory method as an evaluation for compliance. This efficient program allows the highest level of control to be accomplished.

In 2000, the Japanese Odor Research and Engineering Association (JOREA) established the “Research Group for Standardization of Simplified Odor Evaluation Technologies”. The objective of the research activities is to standardize the required functions of simplified evaluation technology, the functional items to be indicated, the testing methods for them and the application program. An application manual based on the research results will be published in 2003.

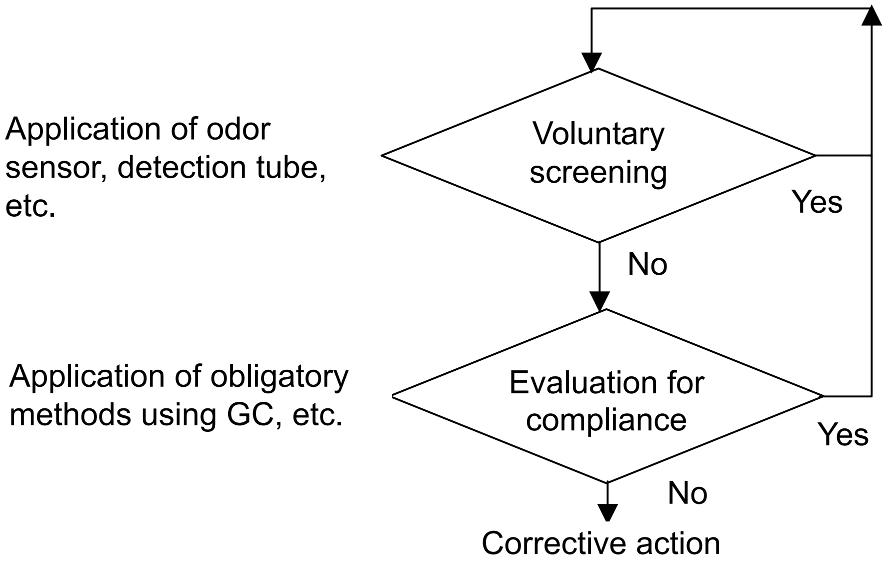


Figure 2 An example of a program for monitoring odor emission source

5. Manual to promote the application of simplified odor evaluation technologies

JOREA is producing a manual to promote the application of simplified odor evaluation technologies. One of the most important items included in the manual contents will be the standardization of testing methods for the basic functions of evaluation technologies in addition to the determination of functional items to be commonly indicated on the products. Another item emphasized in the manual will be the set-up of criteria used to judge the validity of their use in actual application.

According to the tentative manual, the functional items to be commonly indicated on the product in the case of the odor sensor, including the odor recognition device (the so-called electric nose), are shown in Tables 3 and 4. Furthermore, standardization of an appropriate method to measure each item is ongoing.

Table 3 Examples of functional items to be indicated in odor sensors

Indicating item	Detailed item	
Functional indicators	Minimum detectable limit (sensitivity)	
	Maximum measurable concentration	
	Measurable range	
	Response characteristics	Response rate
		Recovery rate
		Standard time required to reach stable state
Confident indicators (accuracy)	Reproducibility	Simultaneously repeated reproducibility
		Reproducibility within a day
		Reproducibility between different days
	Dependence on temperature/humidity	Temperature
		Humidity
	Interfering gaseous substances	Positive interference
		Negative interference
	Influence of gas pressure	
Durable indicators	Range of measurable temperature	
	Range of measurable humidity	
	Influence of corrosive gas	
	Influence of gas causing other deterioration	
	Lifetime of sensor	
	Durable time for continuous use	

Table 4 Examples of functional items to be indicated in odor recognition device (electric nose)

Functional, confident and durable indicators
Range of measurable concentration, Minimum detectable limit, Recognition ability, Measurement time, Interval time between measurements, Reproducibility within a day, Influence of humidity and temperature, etc.

On the other hand, the criteria used to judge whether or not the use of the evaluation method in actual application is valid should be set up to ensure nationwide use. According to the tentative manual, the following criteria are proposed for application of the odor sensor to prediction of the odor index.

> Standard error of predicted value, based on the correlation equation (calibration equation) between indicated values by the sensor and measured odor index values for the odor index should be within ±5 in unit scale of the odor index.

6. Simplification of olfactory measurement method

In the triangular odor bag method formally adopted in Japan, not only are more than six subjects needed for a panel, but more or less odor bags are also consumed, and the expense becomes higher. However, application of such an accurate and expensive method is not always necessary for voluntary monitoring and thus reduction of the cost by simplification of the triangular odor bag method should be accomplished, even if the accuracy somewhat deteriorates.

JOREA has been developing simplified olfactory methods capable of estimating the odor index. Presently, two types of methods using odor bags are tentatively proposed in the manual. They are the 6-4 selection and the 2-1 selection method. Both methods allow remarkably reduced expense. An outline of the procedures are described below.

(1) 6-4 selection method

In this method, two subjects are used. In the first step, six odor bags comprising two controls (non-odorous) and four steps of dilution ratio, are provided together for each subject. The four steps consist of the four odor bags prepared by gradually diluting the original sample odor by the three-times series (for instance, dilution ratios of 100, 300, 1,000 and 3,000). Thereafter, the two subjects select four bags with odor quality of the sample from the six bags provided. If the odor bag with the lowest dilution ratio is selected in addition to one or more wrong bags, the test is finished and then the threshold for the subject can be calculated. In the case of selecting all correct bags, the next four higher steps of dilution ratio are prepared, and in the case of not selecting the lowest dilution ratio (starting dilution ratio), the reverse four lower steps are prepared. For further clarification, an example associated with the results obtained from a subject's selection and the subject's threshold calculated as a logarithmic mean value is shown in Table 5.

Table 5 An example of the results obtained by the 6-4 selection method

No. of odor bag	1	2	3	4	5	6
Dilution ratio	100	No odor	1000	300	No odor	3000
Selection (answer)	○			○	○	○
Calculation of Individual threshold	(log300 + log1000)/2 = 2.74					

(2) 2-1 selection method

In this method, two subjects are needed and one odorous bag from a pair of prepared bags is chosen by each subject. The dilution is performed by the three-times series. Namely, the procedure is similar to the triangular odor bag method with the exception of the number of odor bags and the subject.

The research group on simplified odor evaluation in JOREA has investigated availability of these two methods through application to sample odors taken at actual odor sources. The results show the correlation between the triangular odor bag test and the above two simplified methods, as illustrated in Figure 3. There are good correlations between both. Results obtained from these two simplified methods seem somewhat higher than the triangular bag method. Incidental correct answers are assumed to affect this tendency.

As for the accuracy and time consumption of these two methods, the simplified methods could shorten the measurement time to half of that needed for the triangular odor bag method. However, according to a report by JOREA (2002), their accuracy was somewhat lower than the triangular method in a comparative examination using ethyl acetate as a standard substance.

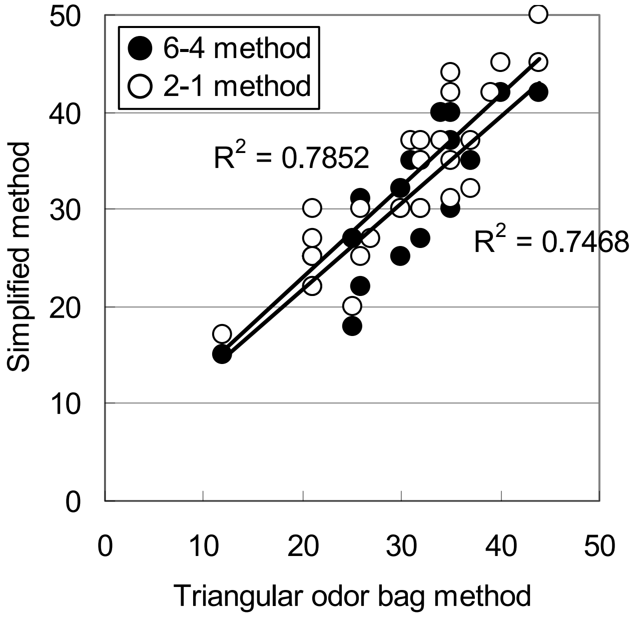


Figure 3 Correlation in the odor index between the triangular odor bag and the simplified method.

7. Concluding remarks

Offensive odor causes a sensory nuisance that results in resident's complaints, albeit without physical damage in most cases. In that sense, offensive odor control to prevent complaints is the most substantial purpose given to our task. In this case, highly frequent monitoring using a simplified and rapid evaluation method with lower expense has more advantages for reliable control than the legally specified method with higher expense.

JOREA is presently carrying out work to complete their manual as soon as possible. Through the manual, it is expected that the proper method for simplified odor evaluation would be widely used.

I would like to express my great respect for the research activities of the Research Group for Standardization of Simplified Odor Evaluation Technologies in JOREA.

References

1) Research Group for Standardization of Simplified Odor Evaluation Technologies in JOREA: A tentative manual
2) JOREA: Final report of Investigation on quality control and safety management in olfactory measurement methods (2002)

Attempts at Simplified Measurement of Odors in Japan using Odor Sensors

Junichi Kita
Analytical & Measuring Instruments Division, SHIMADZU Corporation
1,Nishinokyo-Kuwabaracho Nakagyo-ku,Kyoto 604-8511
Tel +81-75-823-1961 Fax +81-75-811-8187 E-mail j-kita@shimadzu.co.jp

Keywords
Odor sensor, electronic nose, simplified odor measurement

Abstract

The simplified odor measurement in Japan is conducted using simplified versions of the conventional sensory tests or by methods using a detection tube. In addition, research has recently been undertaken to create practical odor sensors with a single sensor element and electronic noses with multiple sensor elements.
The odor sensor with a single sensor element, already used for more than 10 years, features the ability to provide measurement results immediately on-site and to conduct continuous monitoring. However, to match the sensitivity of the nose, the odor sensor requires the creation of calibration curve for each odor element. Also care must be taken about interfering gases when using the odor sensor.
To solve these problems, research has been conducted in recent years into electronic noses that incorporate multiple odor sensor elements. Attempts are being made to create electronic noses that automatically evaluate odor category, evaluate the intensity of odor regardless of its category and output odor category information for the identification of odor sources. Future investigation is required to determine if the electronic nose incorporating multiple sensors can act as a replacement for the human sense of odor, as well as to determine the practical limits to its application.

1. Introduction

The Japanese Offensive Odor Control Law prescribes olfactory and instrumental methods to measure odors. While these methods are effective in providing accurate measurement results, they involve complex procedures. These methods also require the preparation of a human panel, as well as specialist operators.

In recent years, however, there are increasing demands for simplified methods to evaluate the strength and category of odors in applications such as the daily performance evaluation of deodorizing units, self-regulation of odors emitted from workplaces, and screening before conducting official measurement methods. Furthermore, in addition to the conventional demand for the measurement of atmospheric odors, there are increasing demands for the evaluation of odors from automobiles, household appliances and fabrics.
To respond to these demands, research has been made into the practical application

of odor sensors for the simplified measurement of odor intensity and of electronic noses to numerically determine both odor intensity and category.

Table 1 shows the current status of these instruments. Thanks to the development of hand-held devices, the odor sensors offer the advantage of conducting odor measurements on-site. Electronic noses feature the ability to determine the category of odor, and to predict the intensity of odor regardless of its odor category. Odor sensors have been in practical use for about ten years. However, the practical application of electronic noses in the evaluation of offensive odors has just started to be suggested. The determination of the effectiveness of electronic noses must wait for further investigation.

Table 1 Odor Evaluation Methods in Japan

			Advantages	Disadvantages
Official methods	Olfactory method	Triangular odor bag method	Official method	Requires a panel of at least 6 members. Complex procedures.
	Instrumental method	GC GC-MS Absorptiometry	Official method	Instrument preparation required. May not reflect actual odor intensity.
Simplified methods	Instrumental method	Odor sensor	Permits on-site measurements.	Requires calibration for odor element.
		Electronic noses	Can evaluate odor category. Can identify odors and determine the intensity.	Expensive.
		Detector tube	Permits on-site measurements.	Low sensitivity for some odors.
		Monitoring specific components	Permits on-site measurements.	Cannot measure some odors.
	Olfactory method	Comparison using 2 odor bags 6-4 selection method	Simpler than official methods.	Reduced accuracy.

2. Odor Sensors

2.1 Outline

Odor sensors incorporate a sensor element that reacts to odors. The signal from the sensor element is processed to display the odor intensity as a numeric value. The instrument incorporates an internal micro air pump for odor intake. Metal Oxide Semiconductor sensor elements are used to achieve sensitivity and stability. Different types of odor sensor instrument are available: instruments with one sensor element that display the odor intensity only, and instruments with two sensor elements that display simple odor category information in addition to the intensity.

2.2 Sensors with One Sensor Element (Fig. 1)

These instruments achieve good correlation with the odor index if they have high sensitivity for the odor components that make a large contribution to the odor index. However, care is required, as the correlation with the odor index may not be obtained if sensitivity is low for these components or if substances are present that interfere with the sensor.

Fig. 1 Odor Sensor with One Sensor Element

The odor index is determined from a correlation equation (calibration equation) between the odor sensor indicated values and odor indices determined by an official method. This relationship is determined by measuring several different odor concentrations and determining the values of a and b from the following regression equation:

$$SR = a \times OI - b$$

(where, SR is the odor sensor value and OI is the odor index.)

The optimal odor element can be selected to suit the application; elements are available for various fragrance and odor components, hydrogen sulfide, and ammonia.

2.3 Sensors with Two Sensor Elements (Fig. 2)

Fig.2 Odor Sensor with Two Sensor Elements

Apart from the number of sensors, the configuration of these instruments is basically identical to instruments with a single sensor element. The instrument incorporates two sensors: sensor A and sensor B. According to the manufacturer, sensor B reacts sensitively to light odorants, while sensor A is more sensitive to the heavier odorants. "Light" odorants are defined as volatile alcohols, as well as hydrogen sulfide and ammonia with relatively low molecular weights; while 'heavy" odorants are unsaturated aromatic hydrocarbons with relatively large molecular weights, such as toluene, xylene, and methyl mercaptan. Acetic acid and aldehydes are intermediate substances that cause almost identical reactions in both sensors. The sensors are also influenced by the functional groups.

The measurement results are displayed on a two-dimensional Cartesian plane, with the sensor A output along the X axis and sensor B along the Y axis, as shown in Fig. 3. An odor vector is defined as the vector linking the zero-point and A and B Cartesian coordinates. The vector length defines the odor intensity, which is calculated as the square root of the sum of the squares of A and B. The odor quality is defined as the angle (gradient) between the vector and the X axis, which is displayed as an angle from 0° to 90°.

Although this method permits an approximate identification by evaluating the similarity of the measured odor to several pre-measured odors, the identification of complex compound odors is fundamentally difficult. However, the vector does indicate whether a complex compound odor mainly includes heavy or light odor components.

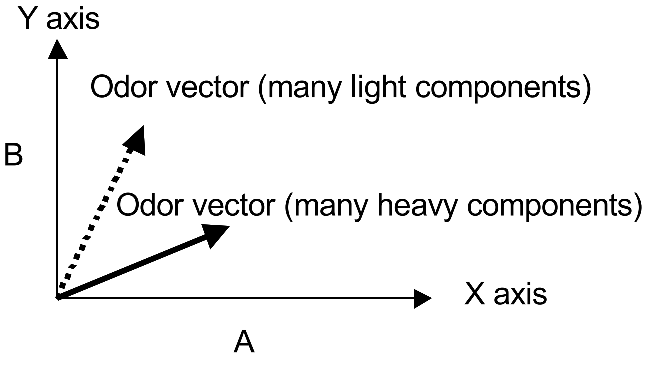


Fig. 3 Odor Sensor Outputs with Two Sensor Elements

3. Electronic Noses

3.1 Outline

An electronic nose is an instrument employing multiple sensor elements that can be operated to offer information on the category, as well as the intensity of odor. In this sense, sensors with two sensor elements described in section 2.3 also belong to electronic noses. Due to the increased number of sensor elements, these instruments are desktop, rather than portable type in many cases. In the subsequent sections, possible applications and additional functions for these instruments will be discussed.

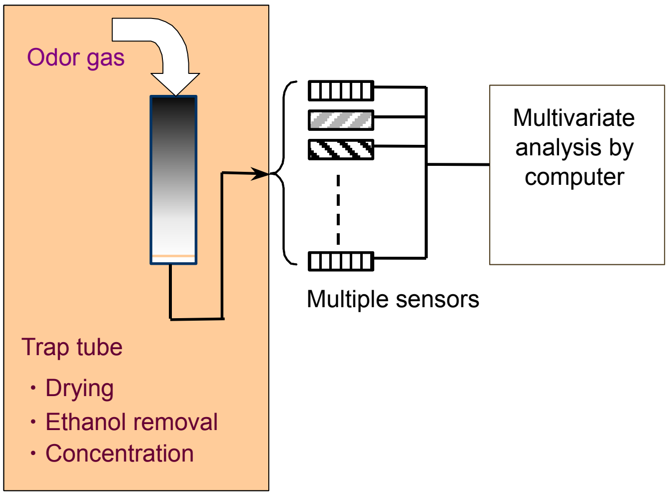


Fig. 4 Electronic Nose Configuration

3.2 Operating Principle

As shown in Fig. 4, the electronic nose resembles a human nose, with the receptor proteins replaced by multiple sensor elements having different properties. The signals generated by these sensor elements in response to an odor are sent as vectors to a computer, which plays the role of the human brain. The computer conducts identification and quantitation of the odors through multivariate analysis or other data processing. (1)

In practice, every sensor responds to different odor substances with different sensitivity, rather than to a specific odor substance. The differences in response between the sensors are subjected to multivariate analysis and other statistical processing, and the disparities between the odor types are displayed graphically for identification and quantitation. (2)
For example, an increase in output intensity with no change in the signal ratio from each sensor is evaluated as a change in odor intensity with no change in odor category. A change in the signal ratio between the sensors is evaluated as a change in odor category.

Fig. 5 Appearance of the Electronic Nose

3.3 Odor Categories using Principal Component Analysis

Principal component analysis is a type of multivariate analysis that can roughly categorize odors.
Fig. 6 shows measurements categorizing normal odors in the environment. Group 1 encompasses odors from printing and painting; Group 2 the odors of exhaust gases (classified by location); Group 3 the sulfur-based odors from piggeries and pulp; and Group 4 the sweet odors of roses and chewing gum.

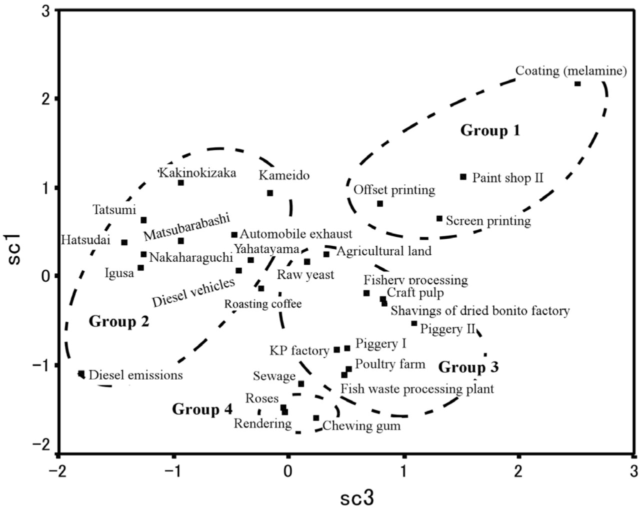


Fig. 6 Principal Component Analysis Results for Environmental Odors

Principal component analysis finds the major direction in which the data group spreads in the odor space formed by multiple sensors. This direction is taken as the principal component axis No. 1 (SC1) and the next major direction of data spreading is taken as the principal component axis No. 2 (SC2). This process is repeated to determine the No. 3 and No. 4 axes. However, for display purposes, two or three of the principal component axes are selected and displayed in a 2- or 3-dimensional representation. (3)

3.4 Odor Intensity Measurements by Multiple Regression Analysis

Multiple regression analysis and the partial least squares (PLS) method determine the correlation between multiple samples of known intensity (odor index) and the output sensor values. The odor intensity (odor index) of unknown samples can then be estimated from this correlation. Fig. 7 shows the multiple regression analysis data from samples taken at three locations with different odor qualities: a printing works, a paint shop and a bone processing plant. The horizontal axis represents the odor index obtained by an olfactory method and the vertical axis represents the odor index estimated from the sensor data using multiple regression analysis. The diagram shows that the method accurately determines the odor index for different odor categories.

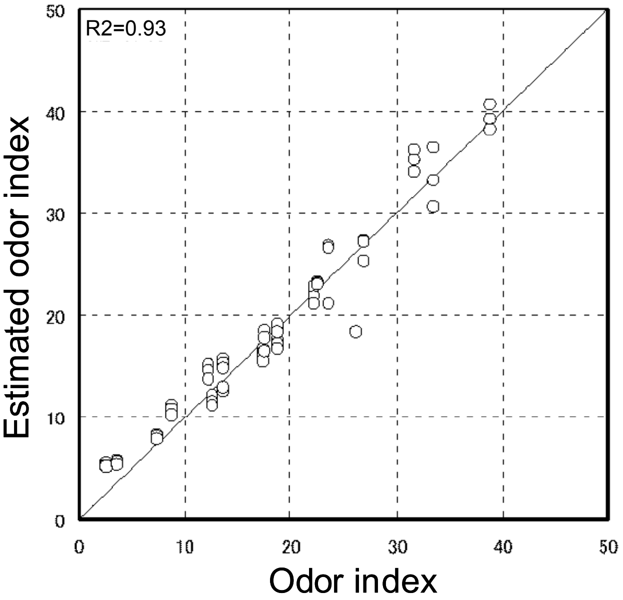


Fig. 7 Odor Intensity Prediction using Multiple Regression Analysis

3.5 New Analysis Methods with the Electronic Nose

Multivariate analysis, such as principal component analysis, is based on the comparison between samples. It obtains no absolute values, making evaluation of the results difficult in the absence of large amounts of comparison data.

Multiple regression analysis offers quantitative information based on large volumes of background data. Therefore, the practical application of this method is difficult, as any change in sensor properties or measurement environment requires a revision of the database.

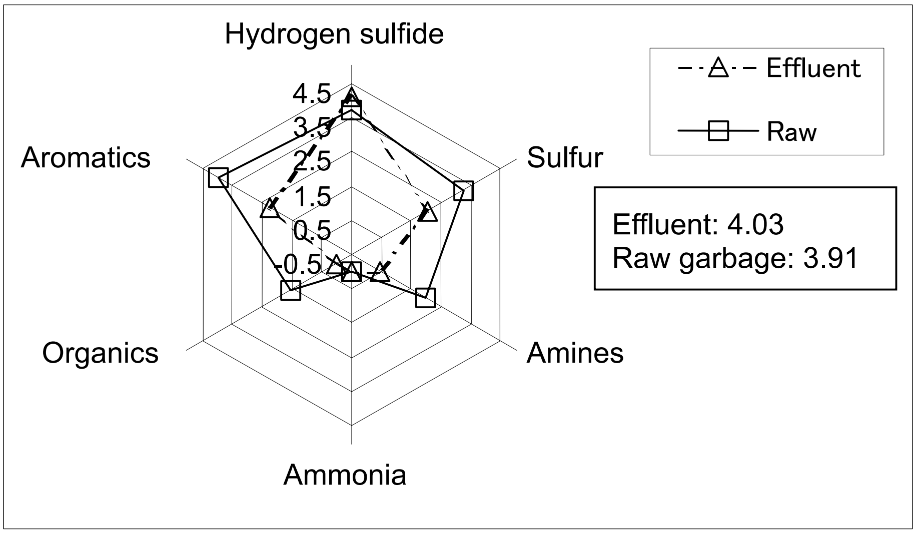
As a means to resolve these problems, we recently proposed absolute value representation software. This method involves the measurement of multiple category gases and evaluating unidentified samples based on these category gases.

In practice, the odor spectrum is determined for several category gases in the odor space formed by multiple output signals, and the sample gas is expressed as the sum of the contributions to each category gas.

If the expression of the odor quality perfectly matches the direction of a category gas, then the sample gas can be evaluated as a gas in the same category as the category gas. The similarity of the sample gas to the category gas decreases as it deviates from the direction of the category gas. The quality of the sample gas odor can be evaluated by numerically representing its contributions to the category gases.

Fig. 8 shows examples of offensive odor measurements using this method. The odor qualities are roughly expressed as a radar chart to provide a visual representation. In addition, totaling the contributions to each category odor gives a 5-stage representation of the overall odor intensity. This odor intensity compensates for the differences in sensitivity between the sensors and the nose, permitting a determination of the odor intensity that is independent of the odor quality.

We anticipate that further developments of this method in the future will permit the evaluation of the quality and intensity of any odor type with sensitivity equivalent to that of the nose.



Effluent: 4.03
Raw garbage: 3.91

Fig. 8 Offensive Odor Analysis using Absolute Value Representation Software

References

1) K.C.Prsaud,G.H.Dodd: Analysis of discrimination mechanisms in the mammalian olfactory system using a model nose, Nature,299,352 (1982)

2) J.W.Gardner, P.N.Bartlett: Electronic noses, Oxford university press, 98 (1999)

3) J.Kita, Y.Aoyama, M.Kinoshita, H.Nakano etal :Quantification of the MOS sensor based Electronic nose utilizing trap tube, Technical Digest of the 17th Sensor Symposium,301 (2000)

State of the Art of Odour Measurement

Prof. Dr.-Ing. Franz-Bernd Frechen,
Head, Dept. Of Sanitary and Environmental Engineering,
University of Kassel, Kurt-Wolters-Str. 3,
34125 Kassel, Germany,
frechen@uni-kassel.de

Keywords
Olfactometry, Sampling, Odour Emission Capacity OEC of liquids

Abstract

As odour is increasingly annoying closed-by residential areas, many approaches were made in the past year, developments achieved and research work done in order to better understand the problem and especially in order to better protect people from severe annoyance. A main aim within this progress – and a basic need – is the development of reliable, repeatable and recognized odour measurement method and procedures. Major parts herein are sampling of odour emissions and the procedure of measurement. These have to serve for the assessment of impact and subsequent definition of odour impact prevention policies. In addition, the characterisation of odour containing liquids by means of the odour emission capacity OEC is explained, and recent developments and results of research work are presented.

1. Introduction

Odour has many facets and can affect man lightly up to seriously, resulting in strong annoyance or even severe health problems. Usually the classical “odour” discussion does not deal with the health aspect rather than with the annoyance aspect of odorants present in the ambient air.

In order to establish a policy and a system to protect man from illegal annoyance caused by odour impact, it is essential to a) measure the odour and b) measure the impact. This paper deals with the odour measurement part, and does not deal with the impact of offensive odours and its resulting annoyance. Annoyance-related work has been carried out to a remarkable extent in Germany in connection with the development of legal standards and governmental policy concerning combat of offensive odours. Winneke has contributed a lot to this, and the reference Winneke et al. (7) may just serve as a younger example of this work. These activities have led to a governmental guideline inside Germany called the “Directive on Odour in Ambient Air”, discussed since the late 80ties and issued as a regulation in the majority of states inside Germany in 1998, as explained by Frechen (3). The “Directive on Odour in Ambient air”, see Both (2), draws the line between nuisance that has to be accepted and annoyance that is not acceptable and thus illegal.

Odour measurement has to help gaining the basic values and numbers that are neeeded for assessment of the community problem that is associated with odour impact.

With regulations existing in several countries of the EU, the European Committee for Standardization CEN decided to issue an European Standard on determination of odour concentration which was finally approved by CEN in December 2002 (1). This is the important key document when discussing odour measurement in the EU, influenced by and vice versa influencing research work conducted in Europe in this field. Thus, frequent reference to the EN 13725 will be found in this paper.

At this time, several aspects of odour measurement are under intense discussion regarding the state of the art in odour measurement. Main topics to be discussed in the presentation more in detail are

- Sampling.
- Olfactometry.
- Odour Emission Capacity of liquids.

2. Sampling

As with all other measurements, accuracy of sampling is a cornerstone for correct results. In addition, it has to be accepted that odours are gaseous emissions, thus sampling in general has to meet all the requirements that are applicable for any analytical measurement of gaseous compounds. The result of all sampling and measurement effort must be the odour emission rate of the respective source, given in odour units per time, e.g. given in o.u./s or Mo.u./h.

According to EN 13725 (1), "Sampling aspects are included in the structure of this Standard, although further research is necessary to complete this issue". Considering the configuration of the odour source, it can be distinguished between sources with a measurable outward airflow (active sources) and sources that do emit odours but have no measurable outward airflow (passive sources).

At active sources, sampling is relatively easy. Outward airflow has to be measured by standard procedures or determined by other means (operator documentation, process control system outputs) and sampling must assure a representative mixture of the emitted air in order to characterize the behaviour of the source. This can be achieved by applying a total cover to the source in order to prevent the sample from being influenced from the ambient air during sampling, see Fig. 1.

Fig. 1 Sampling at an active area source - biofilter

If the source, instead of a stack or a small area source, is a big area, e.g. a surface of a big biofilter or aerated tank, which cannot be covered totally, it is necessary to perform incremental sampling by covering several parts of the active source without changing the pressure conditions (and other, e.g. temperature) and perform sampling here. This may be done with measurement (biofilter) or without measurement (aeration tank) of the outward airflow in the respective separated, covered part of the source. The actual discussion mainly deals with minimum requirements for this type of sampling in terms of percentage of the area that must be covered.

A major problem is encountered when the source obviously emits odour but is a surface without measurable outward airflow. This source type, called “passive source”, is frequently found on wastewater (sedimentation tanks, thickeners etc.) and waste (landfill surfaces etc.) facilities, agriculture (manuring etc.) and others.

However, even in this case it is necessary to measure the odour emission rate originating from the respective passive source. Thus, the sampling method must be appropriate. Different sampling methods were tested throughout the last decades, and the main representatives are

- Indirect measurement: micrometeorological methods using different atmospheric dispersion models.
- Direct measurement: hood methods, commonly divided into static flux chambers, dynamic flux chambers and wind tunnels.

A very good presentation and comparison of these two different types is given by Stuetz (5).

Indirect measurement is not very common and is not used in Germany, except by means of direct field inspections with a trained test person panel using the plume measurement method as described in the VDI-guideline 3940 (6).

Concerning direct measurement, it can be stated that static flux chambers, operating near equilibrium state, are not suitable for producing relevant information on the odour emission rate.

Dynamic flux chambers, usually round with a radial air inlet, also play a minor role today, as the shape of the chamber as well as the radial air inlet inhibit a good control over the flow pattern inside the chamber. However, by using the area-related sweep rate, expressed in sweep air volume per time and per area covered by this hood, and the emission concentration it is possible to calculate the emission rate of the surface where the sampling is done. In general, looking at the different types used, a common feature is a very small airflow, resulting in a very low area-related sweep rate.

Wind tunnels usually cover a rectangular area with a length-to-width-ratio that should be above 2:1, and as researches show, an increasing ratio gives better results, as a more longitudinal stretched tunnel can be better controlled and shows more stable flux conditions. Different types of tunnels, mainly differing in size and shape of inlet and outlet, are used today. Fig. 2 shows an example of a

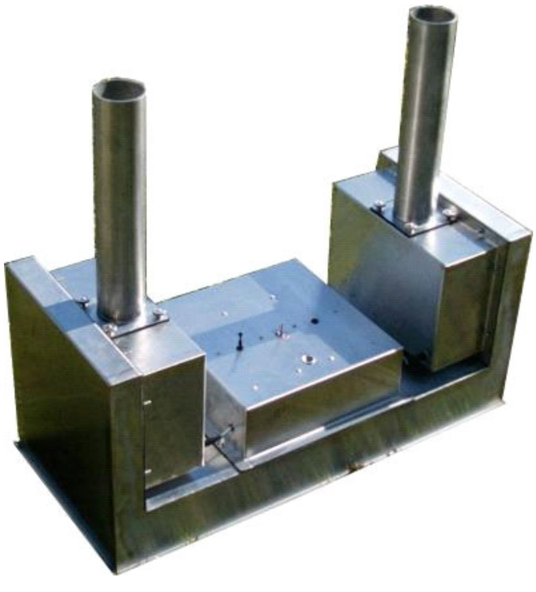


Fig. 2 Compact wind tunnel

wind tunnel with inlet and outlet duct connected to the housing for the fans, one each at inlet and outlet. In the middle, atop the tunnel itself, batteries and other electrical equipment is placed.

Due to the shape of the wind tunnels and due to the fact that odourless air is fed into one of the two narrow sides of the tunnel and extracted at the opposite side, a directed flow is achieved and thus, besides the area-related sweep rate, the sweep flow velocity is an important feature. With most wind tunnels, area-related sweep rates are about 400 $m^3\ m^{-2}\ h^{-1}$ and horizontal velocities are in the range of 0.1 – 0.5 $m\ s^{-1}$. Of course, the sweep flow velocity is influenced by the tunnel height. Thus, with the same area-related sweep rate it is possible to increase the sweep flow velocity by reducing the tunnel's height. Besides this, low tunnels are advantageous due to their better behaviour concerning flow pattern and vertical homogenity.

The sampling box we have been using for many years includes a combination of the advantageous properties of the dynamic flux chamber and the conventional wind tunnel. This system or sampling box may be called a "low speed wind tunnel". Stuetz (5) calls it a dynamic flux chamber which is incorrect due to the flow pattern in the tunnel.

As the description implies, the shape is rectangular with different sizes of between 0.60 m and 1.0 m in length and 0.20 m and 0.33 m in width. Different from other wind tunnel systems, a sweep airflow of about 30 $L\ min^{-1}$ is used, resulting in area-related sweep rates between 5 $m^3\ m^{-2}\ h^{-1}$ and 18 $m^3\ m^{-2}\ h^{-1}$, depending upon the size of the respective sampling box. Using heights around 0.10 m, the velocity inside the tunnel is in a range of 0.01 $m\ s^{-1}$ and 0.03 $m\ s^{-1}$ which is significantly lower than it is found in conventional wind tunnels.

A comparison based upon typical properties of the different types of sampling devices is given in Table1.

Table 1 Comparison of different sampling devices

	dynamic flux chamber	**low speed wind tunnel**	**conventional wind tunnel**
shape	round	rectangular	rectangular
flow pattern	mixed	laminar	laminar/turbulent
height	medium	low	medium/large
area related sweep rate	low < 40 $m^3\ m^{-2}\ h^{-1}$	low < 40 $m^3\ m^{-2}\ h^{-1}$	high 400 $m^3\ m^{-2}\ h^{-1}$
airflow speed (horizontal)	./.	low 0.01 – 0.03 $m\ s^{-1}$	high 0.1 – 0.5 $m\ s^{-1}$
problem with low emission sources	no	no	yes

Due to boundary layer theory, it is likely that low wind speeds inside a sampling system will result in a slight underestimation of the real emission process. However, as olfactometric measurement has a lower limit of reliability and accuracy, severe problems arise when samples from a low emission source are collected via a conventional wind tunnel system, resulting in very low odour concentrations which will make it very likely that odour measurement problems will arise. The choice of the respective sampling system must take these circumstances into consideration.

Another important point is that due to meteorological laws problems in ambient air are always most likely with very low wind speeds or even calm situations. In those situations, with a stable atmospheric condition, transmission is most critical as dilution during transmission is very poor. Thus, measurement should take this into account by using low wind speed in the tunnel system.

At this moment, research work is done at the Kassel University to describe the behaviour of low wind speed tunnels more in detail. Results will be published soon.

Concerning the size of the sampling equipment, a compromise between representativeness and practicability will be necessary. Heights of more than 0.15 m should be avoided. Sizes of area covered by the unit range between 0.1 m^2 and 1 m^2 which may define the lower and the upper limit in the above mentioned sense.

As the EN 13725 explicitly does only include “sampling aspects” and indicates the need of further research and standardization, in the German Engineers Association VDI we just now started a new expert commission on the topic of sampling. Results should be available in 2004.

3. Olfactometry

Concerning olfactometry itself, the two main movements today are standardization of the measurement method, as was done by the EN 13725 standard but also is a hot topic at CASANZ for their area, and development of new olfactometric devices.

Standardization was finished at the end of 2002, as far as the EU is concerned. The final issue of the long discussed EU standard at this time has come to an end concerning the official work but of course will steadily develop further, so it can be expected that within the next 5 to 8 years the standard will be revised according to the experience collected in the meantime.

Looking at the devices, it is sure that standards do have an impact on their design. This is due to the fact that different presentation and evaluation methods – yes/no-method or forced-choice-method – are possible. This is also due to the fact that the EU 13725 requires valid answers from at least 4 panelists after evaluation. Due to the evaluation method it is possible that a panelist may be out of the tolerable range on that specific measurement day, thus it might be advantageous to operate with 5 panelists.

However, most olfactometers today have 1, 4 or 8 sniffing places. A “sniffing place” is formed by one sniffing port per panelist (yes/no-method) or by two sniffing ports per panelist (forced-choice-method). Fig. 3 shows an example of such a

Fig. 3 Example of a 1-sniffing-place forced choice olfactometer (with two sniffing ports)

one-sniffing-place two-sniffing-port olfactometer designed for the forced choice method. Flow control in the past was done by needle valves which were to be operated by the test leader. Mass flow controllers involving a control loop for the airflow showed to be too sensitive and thus too inaccurate concerning the preparation of a defined mixture of the two airflows – sample and odourless air – and thus are not widely used today.

Fig. 4 Example of a four-sniffing-place yes/no olfactometer (in total 4 sniffing ports)

Today the use of saphir orifices is possible to achieve a very constant and stable, defined airflow which is essential for a precise measurement. One of the advantages is that with this type of flow controlling it is possible to let the measurement be done automatically with an appropriate PC-based measurement process controller. However, PC's today are needed in any case for the data management and calculations according to the standards so a PC will always be present. Thus, there is no problem to use the PC for measurement process control plus data acquisition.
Fig. 4 shows a TO 8 device made by ECOMA suited for four panelists doing yes/no measurement. The new series can contain up to 8 sniffing ports allowing for a simultaneous 8-panelist yes/no-measurement or a simultaneous 4-panelist forced-choice-measurement. According to the manufacturer, more than 200 olfactometers are sold worldwide.

4. Odour Emission Capacity (OEC) of liquids

Although olfactometry always deals with (foul) air samples, a characterisation of liquids concerning their content of odorants is urgently needed. By stripping the odorants from a liquid sample, taking samples at suitable times after beginning of the aeration with odourless air and, after the test itself, calculating the integral of the odour units that were stripped from the sample, one can calculate the amount of odour units that can be stripped from one cubic meter of liquid, called the Odour Emission Capacity OEC, as introduced by Frechen and Köster (4). Thus, the OEC is given in o.u. m^{-3}_{Liquid} and characterises the relevance of the respective liquid with respect to odour. The OEC is very important in identification of the main liquid odour sources and is also decisive

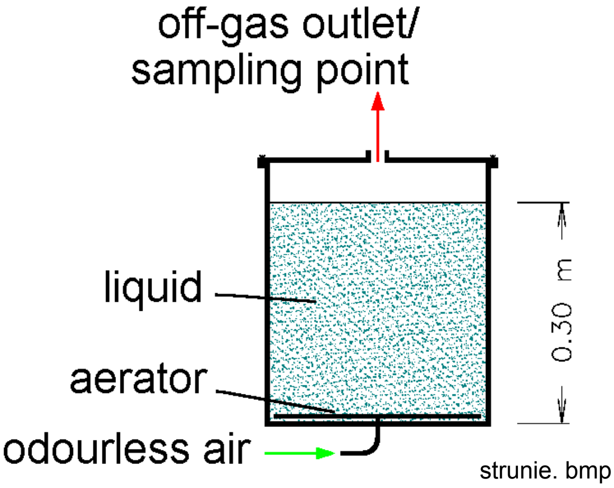


Fig. 5 sketch of the OEC test reactor

concerning the selection of possible measures against odour nuisance resulting from liquid streams.

Fig. 5 shows a sketch of the reactor wherein the OEC test is performed. Of course, besides olfactometric measurement the samples collected from the off-gas outlet should also be analysed concerning analytical measurable compounds such as ammonia, hydrogen sulphide etc., resulting in emission capacities EC for the respective compound and expressed in mg $m^{-3}{}_{Liquid}$. The use of this measurement is just beginning to be recognized, and in the presentation some of the advantages will be presented and values will be given.

Fig. 6 shows the results of a test for foul sewage from a long sewer system. It is evident that we see a H_2S-problem in this case.

In Germany, it is discussed whether to introduce this EC measurement as a way to set standards for indirect discharges of industrial facilities into the publicly owned sewer system in order to minimise odour load.

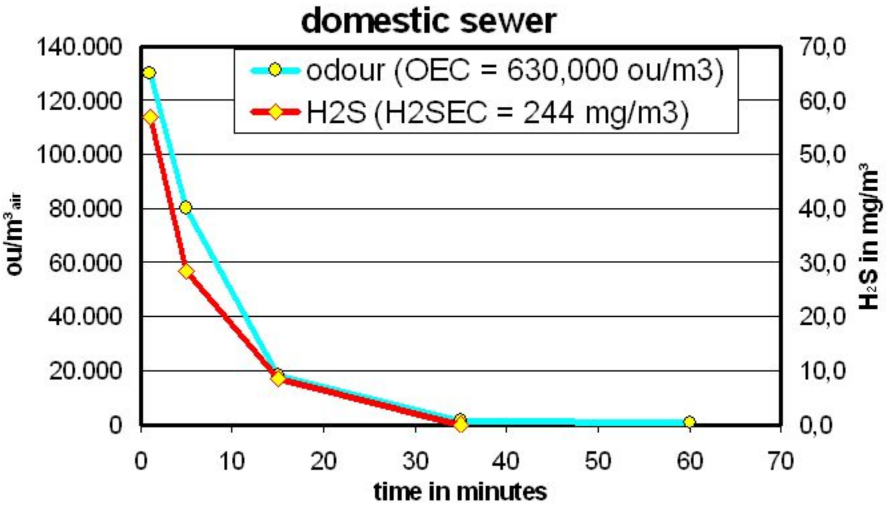


Fig. 6 sample run of OEC test

References

1) AA: Air quality — Determination of odour concentration by dynamic olfactometry, EN 13725:2003(E), CEN, Brussels, March 2003
2) Both, R.: Odor regulations in Germany – A New Directive on Odor in Ambient Air, Proceedings of an International Specialty Conference of AWMA, Bloomington, MM, September 13-15, 1995
3) Frechen, F.-B.: Regulations and policies, 2001, in: Odours in wastewater Treatment, pp. 16-30, edited by R. Stuetz and F.-B. Frechen, IWAP, London
4) Frechen, F.-B. and Köster, W.: Odour Emission Capacity of Wastewaters – Standardization of Measurement Method and Application. Wat. Sci. Tech. Vol. 38, No. 3, S. 61-68
5) Stuetz, R.: Review of Odour Sampling an measurement, Cranfield University, 2002
6) VDI: Determination of Odorants in Ambient Air by Field Measurements, VDI Handbook on Clean Air, Volume 1, Beuth Verlag, Berlin, October 1993
7) Winneke, G.; Frechen, F.-B.; Both, R.; Steinheider, B.: Psychology of odour annoyance: linear and non-linear dose-effect-relationships, memory effects and psychosomatics, (in German) 1995