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Aqueous Odor Thresholds of Organic Pollutants In Industrial Effluents

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AQUEOUS ODOR THRESHOLDS OF ORGANIC POLLUTANTS IN INDUSTRIAL EFFLUENTS

By

Dorris A. Lillard
John J. Powers
Department of Food Science
University of Georgia
Athens, Georgia 30602

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Project Officer

Ronald G. Webb
Southeast Environmental Research Laboratory
National Environmental Research Center
Athens, Georgia 30601

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ABSTRACT

This investigation was designed to determine the odor thresholds in water of organic pollutants that have been identified in industrial effluents. Seven to fourteen judges were used to determine the odor threshold values of 13 compounds at room temperature and 60°C. Odor threshold values for the compounds in ppm at room temperature are: acenaphthene, 0.08; 2-ethyl-1-hexanol, 1.28; butanol, 2.77; geosmin, 0.13×10^{-3}; 2-methyl naphthalene, 0.01; 1-methyl naphthalene, 0.02; diacetone alcohol, 44.1; dibenzofuran, 0.12; 2-benzothiazole, 0.08; 2-mercaptobenzothiazole, 1.76; 2-ethyl-4-methyl-1,3-dioxolane, 0.38; caprolactam, 59.7; d-camphor, 1.29. Extreme value calculations were made to predict a concentration below which a certain percentage of the population might still be able to detect the compound(s). The threshold values obtained at 60°C in most cases do not differ or are higher than those determined at room temperature.

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SECTION I

CONCLUSIONS

The detectable odor threshold in water of a compound was not the same for all judges. Also, the judges' ability to detect odor varied with the compounds being tested. A judge may be the most sensitive to one compound and the least sensitive to another. Conducting the odor threshold determinations at 60°C offers no advantage over the determinations done at room temperature. Since it is impractical to determine odor thresholds using a large number of people, it is best to use at least seven judges and by using extreme value calculations on their results to determine the probability of people being able to detect the odor at concentrations below the odor threshold value of any compound.

SECTION II

INTRODUCTION

GENERAL

Within the last few years, considerable interest has been placed on the pollution of surface water by organic chemicals. In a recent review, Zoeteman and Piet[1] reported that organic pollutants have been traced to both industrial effluents and microorganism that grow in surface water. Regardless of how the organic chemicals enter the water, their presence can result in complaints about the taste and odor of drinking water, as well as off-flavored fish harvested from polluted streams and reduced aesthetic value of polluted rivers and lakes that are used for recreation. Also, the extreme toxicity of certain chemicals to aquatic species as well as man cannot be overlooked. Fortunately for man, many organic chemicals can be detected by the olfactory system before they reach toxic concentrations.

Advanced analytical techniques using gas chromatography-mass spectrometery have resulted in the identification of several hundred compounds in water[2,1]. The complex nature of odor sensation and the wide variability of people's ability to detect odor has slowed the research effort on determining the odor threshold of organic chemicals in water. Zoeteman and Piet reported that they were able to find threshold concentrations for approximately 400 chemicals. Their search of the literature also illustrated the wide discrepancies among the threshold values for the same compounds as determined by different investigators. This difference in threshold concentration, which varied by a 1,000 fold for some compounds, may be due to the different sensitivity of judges, the procedure used for threshold determination or impurities in the compounds studied.

A number of procedures have been developed to measure the odor threshold of compounds in water[3,4,5,6,7]. Baker[8] evaluated several methods of determining odor measurements and concluded that a triangle test (based on a modification of the ASTM method of test, D1292) was statistically the best procedure and was preferred by the panelists. Rosen[9] preferred the consistent series method since it minimized distractions and odor fatigue and yielded data with economy of time and effort. Since each group has its own preferred method of determining threshold concentration and may be biased in their evaluations, a standard procedure for measuring thresholds should be developed by evaluating several procedures on several compounds. This should be a cooperative study among different laboratories that are conducting odor threshold work. Regardless of the method used for their determination, threshold studies should provide information concerning the distribution of the sensitivity to chemicals in people. Zoeteman and Piet[1] utilized the results of their judges and probability calculations to determine the percentage of observers still able to detect the odor at subthreshold levels. Working with taste thresholds, Powers et al.[10] used extreme value calculation to predict the range within which the threshold of the population might occur. This was accomplished by using a panel of only seven people. The taste thresholds of 63 additional judges were within the predicted range.

The statistical theory of extreme values has been used in many diverse fields such as meteorological extremes, floods, breaking strength of textiles, span of human life, gust loads experienced by an airplane in flight, and breakdown voltage of capacitors[11]. The work of Powers et al.[10] demonstrated that extreme value statistics could be used on threshold data and give useful information on the distribution of the sensitivity to taste or odor of chemicals in people.

OBJECTIVE

The objectives of this investigation were to determine the odor threshold in water of organic compounds that were identified in industrial effluents and to predict the percentage of the population that might have odor thresholds lower than that of the panel by using extreme value calculations.

SECTION III

MATERIALS AND METHODS

The compounds used for odor thresholds determinations were supplied by the Southeast Environmental Research Laboratory, United States Environmental Protection Agency. The purity of most of the chemicals were determined by gas liquid chromatography and with the exception of 1-methyl naphthalene were at least 98% pure. These compounds were found frequently as pollutants in industrial effluents[2].

The odor thresholds in water were determined using a procedure derived from a modification of a sensory test that was used for taste thresholds in earlier work[10]. Stock solutions of the chemicals were made by dissolving the chemicals in odor free water. Geometric dilutions were made and the sample was evaluated by judges. When the compound was not soluble in water, it was dissolved in 50 ml of propylene glycol. The appropriate dilutions in water were made from this propylene glycol solution solution. The same amount of proplyene glycol that was in the sample dilutions was also dissolved in the water blank to prevent the judges from making their decision by looking at the difference in surface tension of the solutions. The appropriate dilution of the chemical was added to the odor flask, 500 ml glass stoppered (ST32) Erlenmeyer flask, containing enough odor-free water to make the total volume of 200 ml.

THRESHOLD DETERMINATION

Using the triangle procedure the odor thresholds were determined against odor-free water. In each set of three flasks, two contained odor-free water and one the test substance or two test substances and one odor-free water. The judges were asked to determine the different sample in each set of three samples. The evaluations were conducted on ten three-sample sets at each concentration of the test substance. In order for the judge to significantly detect the odor at the 95% confidence level, seven correct responses were required at any concentration. A geometric increase or decrease in concentration was made and the evaluation repeated until the threshold was determined for each judge. Seven to fourteen judges were used to determine the odor threshold of the compounds.

The odor threshold determinations were conducted in a room designed for sensory evaluations. Five three-sample sets were placed in the room and were evaluated by the judges. The judges were instructed to shake the flask, remove the stopper and sniff the vapors and record their response. Each judge evaluated samples twice a day, once at mid-morning and mid-afternoon.

The odor thresholds were determined at room temperature and 60°C. For the 60°C evaluation, the odor flask were placed in a 60 ± 1°C water bath prior to and during the evaluation.

The geometric mean of all judges' thresholds was calculated to indicate the threshold for each substance tested. The individual judge's threshold was used to make extreme value calculations in order to predict the lowest threshold that a given percentage of the population might have[10]. Liebleim's[11] method of extreme value calculation was followed. An example of the calculation is given in Liebleim's[11] report. A computer program was developed and all calculations in this report were done at the University of Georgia Computer Center.

SECTION IV

RESULTS AND DISCUSSION

THRESHOLD VALUE DETERMINATIONS

The judges for the odor threshold determinations were selected from graduate students, faculty and technicians in the Food Science Department. At the start of the study, the judges were asked to evaluate 10 3-sample sets. However, it was soon observed that 10 sets were too many samples for the judges to evaluate at one time because their olfactory system became fatigued. It was found that the judges could easily evaluate 5 3-sample sets without over-working their olfactory system.

The initial concentration of a compound to be evaluated was one that all of the judges was expected to detect. This familiarized the judges with the odor of the compound and also assured that only decreasing dilutions were needed in future evaluations on that compound. In some instances, however, some judges could not detect the initial concentration and samples with a higher concentration had to be made for them.

If a judge made seven or more correct decisions out of ten evaluations, he was asked to evaluate the samples at the next lower dilution. A judge stopped evaluating samples when he gave fewer than seven correct responses. Table 1 illustrates the type of data obtained for the threshold determination of acenaphthene. If a judge obtained more than 7 correct answers at one concentration and less than 7 on the next dilution, the log of percent positive answers was plotted against concentration and his threshold was obtained from the 70% positive point on the graph.

Tables 2 and 3 list the threshold values in water of the 13 compounds used in this study. The odor threshold of n-butanol was determined in order to compare values obtained with our procedure to values determined by other workers. Reported odor threshold of n-butanol in water range from 1 to 2.5 ppm[12,13]. Our odor threshold values for n-butanol were 2.77 at room temperature and 2.88 at 60°C. These values compare very favorably with reported data. The range of odor threshold for n-butanol as obtained by our group of judges was also very narrow (1.66 - 5.00 ppm at room temperature and 2.14 - 4.04 ppm at 60°C). This would tend to indicate that the variation in sensitivity to n-butanol among people is not too great.

Table 1. JUDGES RESPONSE FOR THE ODOR THRESHOLD OF ACENAPHTHENE AT ROOM TEMPERATURE

Judge	Concentration (ppm)						Threshold (ppm)
	0.500	0.250	0.125	0.063	0.031	0.015	
1	10/10[a]	10/10	10/10	9/10	7/10	3/10	0.031
2	10/10	10/10	8/10	6/10			0.097
3	10/10	10/10	6/10				0.162
4	10/10	9/10	8/10	3/10			0.114
5	10/10	7/10	10/10	5/10			0.092
6	10/10	9/10	10/10	7/10	4/10		0.063
7	10/10	10/10	10/10	9/10	9/10	6/10	0.021
8	10/10	10/10	8/10	7/10	9/10	4/10	0.025
9	10/10	10/10	9/10	9/10	4/10		0.053
10	10/10	10/10	8/10	5/10			0.108
11	10/10	9/10	9/10	4/10			0.105
12	10/10	9/10	5/10				0.196
13	10/10	10/10	9/10	4/10			0.103
14	10/10	8/10	4/10				0.226

[a] Correct responses/number evaluated

Table 2. ODOR THRESHOLD CONCENTRATIONS IN WATER OF CHEMICALS AT ROOM TEMPERATURE (ppm)

Compound	Number of Judges	Room Temperature	
		Threshold	Range
Acenaphthene	14	0.08	0.02 - 0.22
2-Ethyl-1-Hexanol	13	1.28	0.58 - 2.08
Butanol	8	2.77	1.66 - 5.00
Geosmin	9	0.13×10^{-3}	$(0.03 - 0.50) \times 10^{-3}$
2-Methyl Naphthalene	10	0.01	0.003 - 0.04
1-Methyl Naphthalene[a]	10	0.02	2.52×10^{-3} - 0.17
Diacetone Alcohol	9	44.12	5.63 - 269
Dibenzofuran	10	0.12	0.04 - 0.51
2-Benzothiazole	8	0.08	0.01 - 0.98
2-Mercaptobenzothiazole	7	1.76	0.40 - 10.9
2-Ethyl-4-Methyl-1,3-Dioxolane	8	0.38	0.14 - 1.39
Caprolactam	8	59.7	36.0 - 100.0
d-Camphor	8	1.29	0.25 - 3.83

[a] Contains 28% 2-Methyl Naphthalene

Table 3. ODOR THRESHOLD CONCENTRATIONS IN WATER OF CHEMICALS AT 60°C (ppm)

Compound	Number of Judges	60°C	
		Threshold	Range
Acenaphthene	14	0.08	0.0019 - 0.33
2-Ethyl-1-Hexanol	13	0.78	0.58 - 1.24
Butanol	8	2.88	2.14 - 4.04
Geosmin	9	$.18 \times 10^{-3}$	$(0.0078 - 1.54) \times 10^{-3}$
2-Methyl Naphthalene	10	0.02	0.003 - 0.17
1-Methyl Naphthalene[a]	10	0.05	$0.97 \times 10^{-3} - 0.4$
Diacetone Alcohol	9	54.9	7.90 - 90.0
Dibenzofuran	10	0.25	0.05 - 0.51
2-Benzothiazole	8	0.45	0.024 - 0.96
2-Mercaptobenzothiazole	7	1.20	0.28 - 2.80
2-Ethyl-4-Methyl-1,3-Dioxolane	8	0.36	0.07 - 0.81
Caprolactam	8	208.7	10.7 - 1482.0
d-Camphor	8	0.28	0.18 - 0.44

[a] Contains 28% 2-Methyl Naphthalene

Gas chromatography analysis of 1-methyl napthalene revealed that it contained 28% 2-methyl naphthalene. Therefore, the 1-methyl naphthalene odor threshold values listed in Tables 2 and 3 are for this mixture. Since the odor thresholds for these two compounds are similar and if there is no additive or synergistic effect between the odors of 1-methyl naphthalene and 2-methyl napthalene, the threshold values for pure 1-methyl naphthlene would not change significantly from the values reported in Tables 2 and 3.

The threshold values for all the compounds at 60°C were close to or higher than the threshold values at room temperature. Since more molecules would be in the vapor phase at 60°C than at room temperature, one would expect the threshold value to be lower at the higher temperature. Perhaps the threshold values at 60°C were influenced by the increased water vapor which saturated the olfactory system and made the judges less sensitive to the compounds. This increased water vapor did not affect all judges in the same way. Some judges were more sensitive at 60°C than at room temperature while for other judges the reverse was the case. This phenomenon also varied from compound to compound. In one instance a judge may be the most sensitive to one compound and the least sensitive to the next.

As indicated in Tables 2 and 3 the range of threshold values for the limited number of judges used in this study is large. Many of the threshold values differed by a factor of 100 and for 1-methyl naphthalene at 60°C the difference between the highest and lowest threshold values was over 1,000. Because of this wide range in threshold values, the geometric average threshold value for a compound is not too helpful in providing information to the people in charge of controlling the odor of the water supply. Information concerning the concentration of a compound that a given percentage of the people cannot detect would be more useful to them. Extreme value calculation is one method that can provide this information.

EXTREME VALUE CALCULATIONS

Each judge's threshold value was used in the extreme value calculations. Figures 1 and 2 illustrate extreme value plots for the threshold values for two of the compounds. In these figures the center line (-□-□-) is the regression line as calculated from the experimentally determined threshold values. The upper line (-○-○-) and the lower line (-△-△-) are the upper and lower 0.95 confidence limits. The points on the graphs were taken from the computer calculations and were used only to draw the line on the figure.

Using the lower .95 confidence level for geosomin (Figure 1), 90% of the population would have a threshold of 4.4×10^{-6} ppm (log = -5.39) or higher. The other 10% of the population would be able to detect

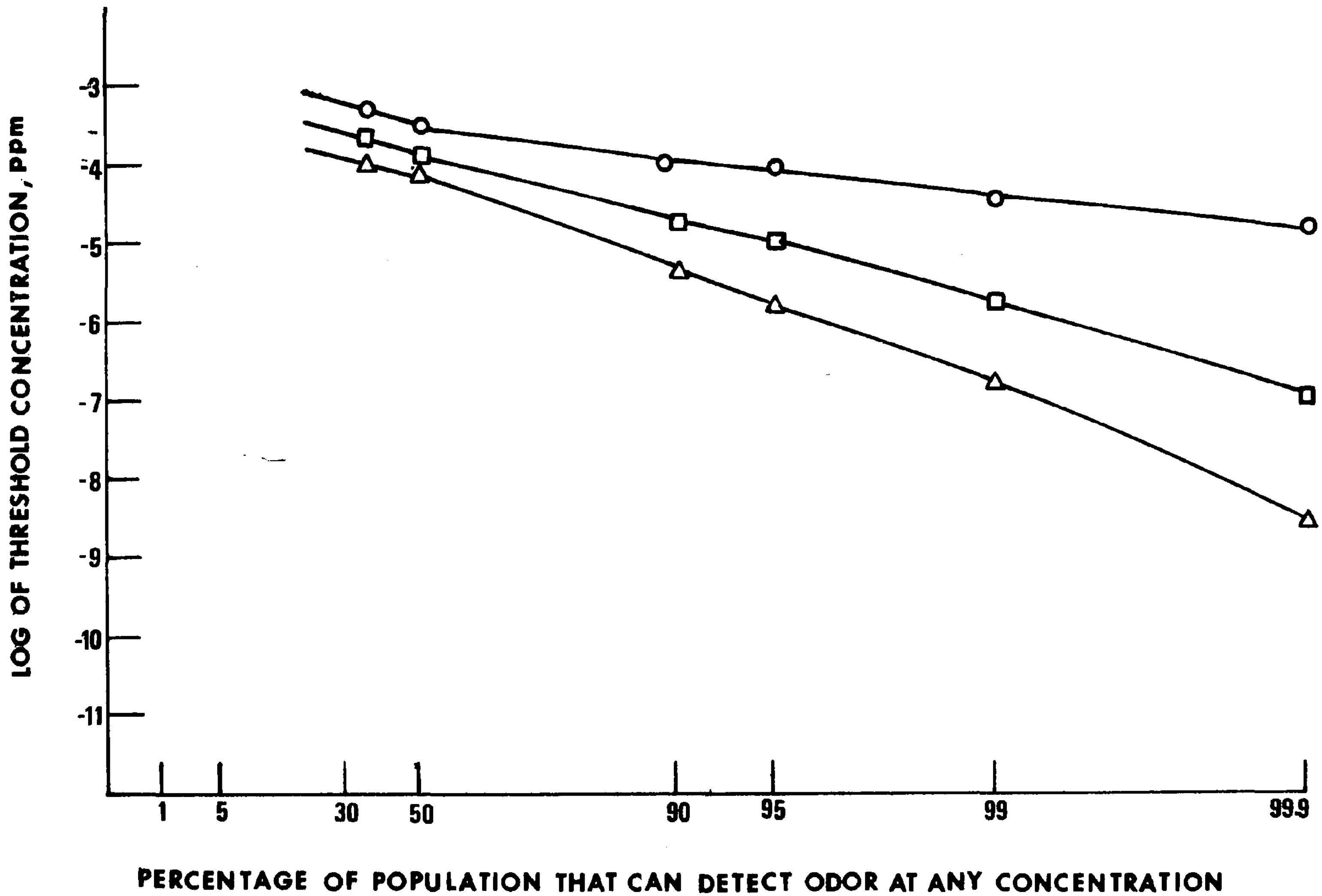

FIGURE 1. EXTREME VALUE PLOT FOR ODOR THRESHOLD LEVELS FOR GEOSMIN

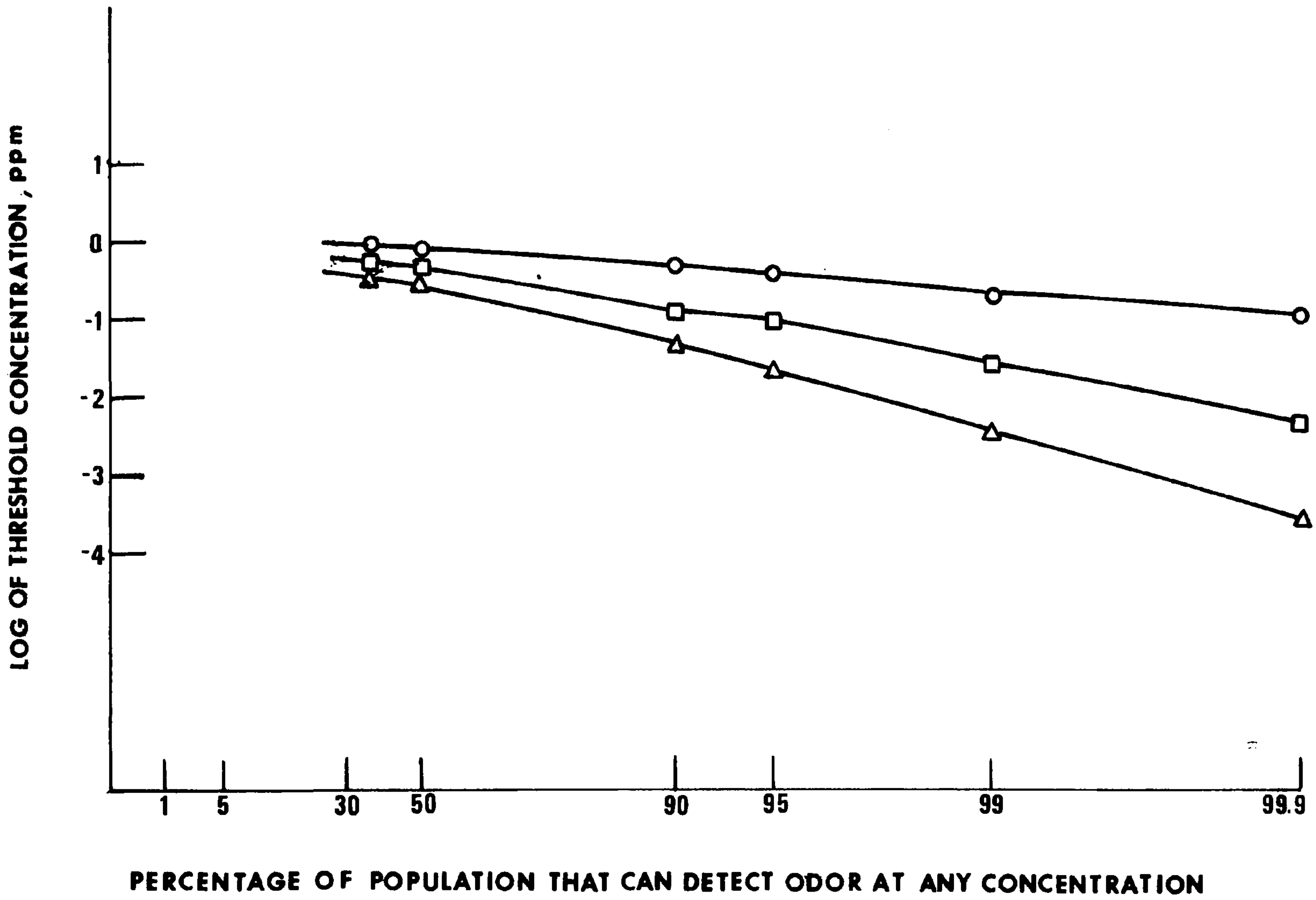

FIGURE 2. EXTREME VALUE PLOT FOR ODOR THRESHOLD LEVELS FOR 2-ETHYL-4-METHYL-1,3-DIOXOLANE

Table 4. CONCENTRATIONS OF CHEMICALS THAT MAY BE DETECTED BY VARIOUS SEGMENTS OF THE POPULATION AS PREDICTED BY EXTREME VALUE CALCULATIONS (ppm)

Compound	Threshold Value	Percent of Population Still Able to Detect Odor			
		20	10	1	0.1
Acenapthene	8.0×10^{-2}	2.6×10^{-2}	1.4×10^{-2}	1.9×10^{-3}	2.1×10^{-4}
2-Ethyl-1-Hexanol	1.3	6.1×10^{-1}	4.2×10^{-1}	1.2×10^{-1}	3.5×10^{-2}
Butanol	3.8	1.5	1.2	4.4×10^{-1}	1.6×10^{-1}
Geosmin	1.3×10^{-4}	1.3×10^{-5}	4.1×10^{-6}	9.7×10^{-8}	2.3×10^{-9}
2-Methyl Naphthalene	1.3×10^{-2}	2.0×10^{-3}	7.9×10^{-4}	3.9×10^{-5}	1.9×10^{-6}
1-Methyl Naphthalene[a]	2.3×10^{-2}	2.1×10^{-3}	7.5×10^{-4}	1.8×10^{-5}	4.5×10^{-7}
Diacetone Alcohol	44.1	4.6	1.4	3.2×10^{-2}	7.6×10^{-4}
Dibenzofuran	1.2×10^{-1}	1.9×10^{-2}	6.1×10^{-3}	1.7×10^{-4}	4.9×10^{-6}
2-Benzothiazole	8.8×10^{-2}	1.8×10^{-3}	2.6×10^{-4}	4.1×10^{-7}	6.8×10^{-10}
2-Mercaptobenzothiazole	1.8×10^{-1}	7.9×10^{-2}	1.6×10^{-2}	8.8×10^{-5}	5.1×10^{-7}
2-Ethyl-4-Methyl-1,3-Dioxolane	3.8×10^{-1}	8.8×10^{-2}	3.9×10^{-2}	2.9×10^{-3}	2.2×10^{-4}
Caprolactam	59.6	25	16	3.8	9.2×10^{-1}
d-Camphor	1.3	1.3×10^{-1}	4.1×10^{-2}	9.2×10^{-2}	2.1×10^{-5}

[a] Contains 28% 2-Methyl Naphthalene

geosmin at concentration lower than 4.4×10^{-6} ppm. The minimal concentration that any desired percentage of the population could detect can be obtained from the extreme value regression plot of the experimentally determined threshold values (lower confidence line in Figure 1).

Table 4 lists the odor threshold values determined at room temperature for the 13 compounds. Also included in Table 4 are the concentrations which a given percentage of the people are still able to detect. The minimal detectable concentration does not differ from the threshold value by the same magnitude for all of the compounds. If we consider the concentrations that one percent of the observers can still detect, we find that the detectable concentrations for butanol, acenaphthene and caprolactan differ from the threshold concentrations by a factor of approximately 10 while the magnitude of the difference for 2-mercaptobenzothiazole, d-camphor and geosmin is approximately 10,000. Also, one tenth of the observers can still detect 2-benzothiozole at a level which is 1/100,000th of the threshold concentration.

It appears that the distribution of the sensitivity to odors in man differs from compound to compound and it is impossible to predict the concentration of a compound that a given population can detect from the information obtained from another compound. Each compound that is found to be an odor pollutant should be evaluated by a small group of judges and the odor threshold determined. Calculations such as extreme value analysis could be done to predict the concentration that a given percentage of people could still detect. This would provide a guideline which could be used by those in charge of removing the pollutant from the water if complete removal of the pollutant is impossible.

ODOR THRESHOLD CONCENTRATIONS REPORTED BY OTHER WORKERS

The odor threshold in water of 56 of the compounds listed by Webb[2] were reported by other workers. These odor thresholds are listed in Table 5. Many of these were reported in the reviews made by Zoeteman[12] and Stahl[13]. Some of these compounds have several reported thresholds with large differences among them. This difference in threshold concentrations may be due to the procedures used for the odor threshold determinations and sensitivities of the judges used to detect the odor. Although the odor threshold concentration is known for these compounds, information concerning the distribution of the observers sensitivities to these compounds would be of greater value to the workers in charge of removing pollutants from the water supply.

Table 5. ODOR THRESHOLD CONCENTRATIONS OF CHEMICALS IN WATER

Compound	Threshold (ppm)	Source
Acetophenone	6.8×10^{1}	13
	1.7×10^{-1}	12
	6.5×10^{-2}	12
	3.9×10^{-3} - 2.02	7
Acrylonitrite	3.9×10^{-3}	13
	2.02	13
	1.86	13
	2.9×10^{1}	12
	19	15
Aldrin	1.70×10^{-2}	13
	2.0×10^{-3}	12
Arachidic acid	2.0×10^{1}	13
Benzaldehyde	1.8×10^{-4}	13
	4.29×10^{-3}	13
	4.0×10^{-3}	13
	4.4×10^{-4}	13
	3.0×10^{-3}	13
	4.36×10^{-4}	13
	3.0×10^{-3}	
1-Butanol	2.5	13
t-Butylisothiocyante	1.67×10^{-3}	13
Chlordane	2.5×10^{-3}	13
	5.0×10^{-4}	12
o-Cresol	9.0×10^{-2}	13
	6.5×10^{-1}	13
	2.6×10^{-1}	9
m-Cresol	6.8×10^{-1}	13
	2.5×10^{-1}	9
p-Cresol	5.5×10^{-2}	9
Cumene	1.0×10^{-1}	12
Cyclohexanol	3.5	12

Table 5. (Continued) ODOR THRESHOLD CONCENTRATIONS OF CHEMICALS IN WATER

Compound	Threshold (ppm)	Source
p-Cymene	1.0×10^{-1}	12
n-Decane	1.0×10^{1}	12
2,6-Dinitrotoluene	1.0×10^{-1}	12
Diphenylether	1.5×10^{-2}	12
Dodecane	1.0×10^{2}	12
Endrin	4.1×10^{-2}	13
	1.8×10^{-2}	13
2-Ethyl-1-hexanol	2.7×10^{-1}	12
Ethyl Phenylacetate	6.5×10^{-1}	12
Fufural	6.0×10^{-1}	13
	1.0×10^{0}	12
Guaiacol	2.1×10^{-2}	13
	1.3×10^{-2}	12
Heptachlor	2.0×10^{-2}	13
Hexachlorobenzene	3.0×10^{0}	12
Hexachlorocyclopentadiene	1.0×10^{-3}	12
Hexachlorobutadiene	6.0×10^{-3}	13
1-Hexanol	5	13
	5×10^{-1}	12
Indene	1.0×10^{-3}	12
Isopentyl Alcohol	4.0×10^{0}	12
Limonene	1×10^{-3}	13
alpha-Methyl Benzyl Alcohol	1.5×10^{3}	12
o-Methylstyrene	1.0×10^{-1}	12

Table 5. (Continued) ODOR THRESHOLD CONCENTRATIONS OF CHEMICALS IN WATER

Compound	Threshold (ppm)	Source
Myristic Acid	10×10^{-3}	13
Naphthalene	6.8×10^{-2}	13
Nitrobenzene	2.0×10^{-1}	12
	3×10^{-2}	14
o-Nitrophenol	1.0×10^{1}	12
1-Octanol	1.3×10^{-1}	13
Palmitic Acid	1×10^{1}	13
Pentachlorophenol	3.0×10^{-1}	12
Pentadecanoic Acid	1.0×10^{1}	12
Phenathrene	1.0×10^{0}	12
Phenol	5.9	13
	7.5	13
	1.0×10^{0}	12
	4.2	9
beta-Pinene	1.4×10^{-1}	13
Quinoline	7.1×10^{1}	13
	$1.6 \times 10^{-2} - 4.3$	7
Stearic acid	2.1×10^{1}	13
Styrene	7.3×10^{-1}	13
	3.7×10^{1}	13
alpha-Terpineol	3.4×10^{-1}	13
	3.5×10^{-1}	13
Terpinolene	2×10^{-1}	13
1,1,2,2,-Tetrachloroethane	5×10^{-1}	13

Table 5. (Continued) ODOR THRESHOLD CONCENTRATIONS OF CHEMICALS IN WATER

Compound	Threshold (ppm)	Source
iso-Valeric	5.0×10^{0}	12
n-Valeric	1.0×10^{1}	12
n-Undecane	1.0×10^{1}	12
Vanillin	2×10^{-1}	13
	4.0	13
	2.2×10^{0}	12
x-Xylene	2.2	12
	1.8	9
m-Xylene	5.0×10^{-2}	12
	1.0	9
p-Xylene	1.0	12
	5.3×10^{-1}	9

SECTION V

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16. ABSTRACT

This investigation was designed to determine the odor thresholds in water of organic pollutants that have been identified in industrial effluents. Seven to fourteen judges were used to determine the odor threshold values of 13 compounds at room temperature and 60°C. Odor threshold values for the compounds in ppm at room temperature are: acenaphthene, 0.08; 2-ethyl-1-hexanol, 1.28; butanol, 2.77; geosmin, 0.13×10^{-3}; 2-methyl naphthalene, 0.01; 1-methyl naphthalene, 0.02; diacetone alcohol, 44.1; dibenzofuran, 0.12; 2-benzothiazole, 0.08; 2-mercaptobenzothiazole, 1.76; 2-ethyl-4-methyl-1,3-dioxolane, 0.38; caprolactam, 59.7; d-camphor, 1.29. Extreme value calculations were made to predict a concentration below which a certain percentage of the population might still be able to detect the compound(s). The threshold values obtained at 60°C in most cases do not differ or are higher than those determined at room temperature.

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17. KEY WORDS AND DOCUMENT ANALYSIS

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