REPORT OF
ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER, 1972

environmental engineering, inc.

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EPA PROJECT REPORT NO. 73-KPM-1-B

ENVIRONMENTAL ENGINEERING, INC.
2324 SOUTHWEST 34TH STREET
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ODOR SURVEY REPORT

WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY

Introduction

On October 3 - 6, 1972, an odor survey was conducted at the Westvaco Kraft Mill, Wickliffe, Kentucky in conjunction with emission measurements made by EPA personnel and equipment (EPA Project Report No. 73-KPM-1-A) to obtain data for the establishment of new source performance standards for kraft pulp mills. These tests took place using the plant non-condensable gas incinerator system as a source of emissions to be tested. This system collects non-condensable gases from various points in the plant process and exposes these to a natural gas flame assuring that all gases reach a temperature of 1000^{o}F. Odor samples were taken both at discharge and inlet conditions of this facility. Simultaneous evaluations of sulfur content were performed by EPA with instrumentation installed in their mobile laboratory. The odor panel was conducted in accordance with a printed procedure provided by EPA and appended hereto as Appendix A.

Summary

This odor survey which lasted for a total of five days is regarded as having been successful. An effort was made to utilize those persons having the most sensitive olfactory system. Prior experience had established that younger persons generally meet this requirement; therefore, this age group was selectively utilized. The panel was organized and surveys conducted at the Paducah-Tilghman High

School in Paducah, Kentucky. Surveys were conducted on the non-condensable gas incineration system, using both incinerated and non-incinerated (inlet) gas samples. As might be expected, the odor characteristics of these two were distinctly different, the former being predominantly sulfur dioxide in character and the latter predominantly malodorous gases including hydrogen sulfide and the mercaptan species. These differences produced no problem with the panel when properly handled.

Procedure

Essentially the procedure followed in the organization and conduction of this odor survey was that appearing as Appendix A of this report. As described above, this procedure was delineated by EPA as a tentative method for the purpose stated. Deviations from this procedure were essentially three in number, the first having to do with the requirement for an odor free room. Due to timing and limited facilities at this distant location, an odor free room was not available. A portable air cleaning system was tried but the tubing used in construction contributed more odor than was removed. This system was abandoned. All tests were conducted in the high school science laboratory which was essentially free of odors and certainly free of all odors associated with pulp and paper operations. The second deviation from the printed procedure dealt with the method of screening candidates for final selection of the odor panel. The method designates vanilla extract and methyl salicyate in solution with benzyl benzolate. For the test in Paducah, substitutions were made for these chemicals. Vanilla extract was used in aqueous solution along with the mouth wash "Listerine" in aqueous solution. The third deviation from the printed

procedure involved the size of hypodermic syringe to be used. The method calls for a 100 ml syringe which was not readily available; therefore, 50 ml syringes were used with no apparent deterioration in the ability of the panel to detect odor.

For this test a panel of ten individuals was selected from an original group of 21 applicants. The selected ten consisted of nine males and one female, all of whom were juniors or seniors in the high school. Figure 1 illustrates the test used to screen this group.

Samples from the Westvaco mill at Wickliffe were collected in syringes and mylar bags thereafter being transported to Paducah, approximately 38 miles distant for purposes of conducting the odor survey.

Results

The results of these odor surveys are presented in Table 1 and Figures 2 through 7 appearing hereafter. As one will note in examining these several figures and associated data of Table 1, there were occasions when the consistency of the panel was poor. One might expect that this would improve with time, as indeed it did.

Throughout the execution of these series of odor surveys, certain observations were made and are summarized as follows:

1. The persons conducting the odor survey program must maintain control of the odor panel group at all times.

2. Dilutions for presentation to the panel should be made in a location not in view of the panel, preferably in an adjacent room.

Figure 1

LAYOUT OF SCREENING TEST

A-Vanilla W-Listerine

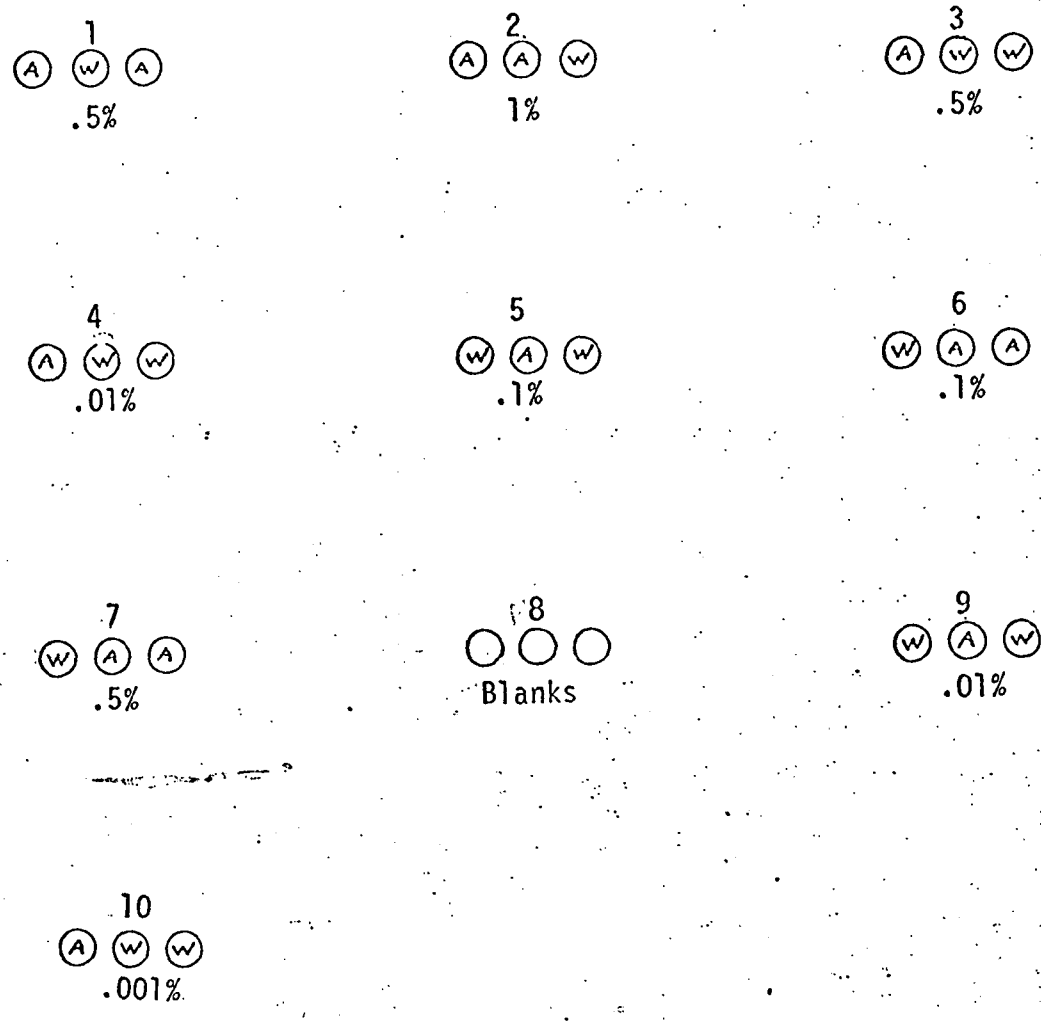

TABLE 1

SUMMARY OF ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER, 1972

Source	Date	Dilution Ratio	Percent Reporting Positive Response
Outlet	10/3/72	0:1	90
		2:1	90
		5:1	100
		5:1	70
		10:1	50
		∞:1	10
		17:1	80
		5:1	40
		17:1	60
		10:1	60
		50:1	20
Outlet	10/4/72	2:1	100
		100:1	80
		500:1	50
		1000:1	30
		100:1	20
		∞:1	60
		100:1	100

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TABLE 1
(continued)

SUMMARY OF ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER, 1972

Source	Date	Dilution Ratio	Percent Reporting Positive Response
		500:1	40
		250:1	40
		1000:1	30
Outlet	10/5/72	500:1	20
		250:1	30
		100:1	80
		66:1	40
		∞:1	30
		50:1	60
Outlet (No Fire)	10/5/72	10,000:1	80
		1000:1	100
		100,000:1	55
		1,000,000:1	20
		100,000:1	10
		100,000:1	60
Outlet	10/6/72	1000:1	80
		2000:1	30

TABLE 1
(continued)

SUMMARY OF ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER, 1972

Source	Date	Dilution Ratio	Percent Reporting Positive Response
		100:1	100
		∞:1	0
		500:1	100
Outlet (No Fire)	10/6/72	10,000:1	20
		1000:1	50
		500:1	90
		100:1	100
		∞:1	10
		100:1	100

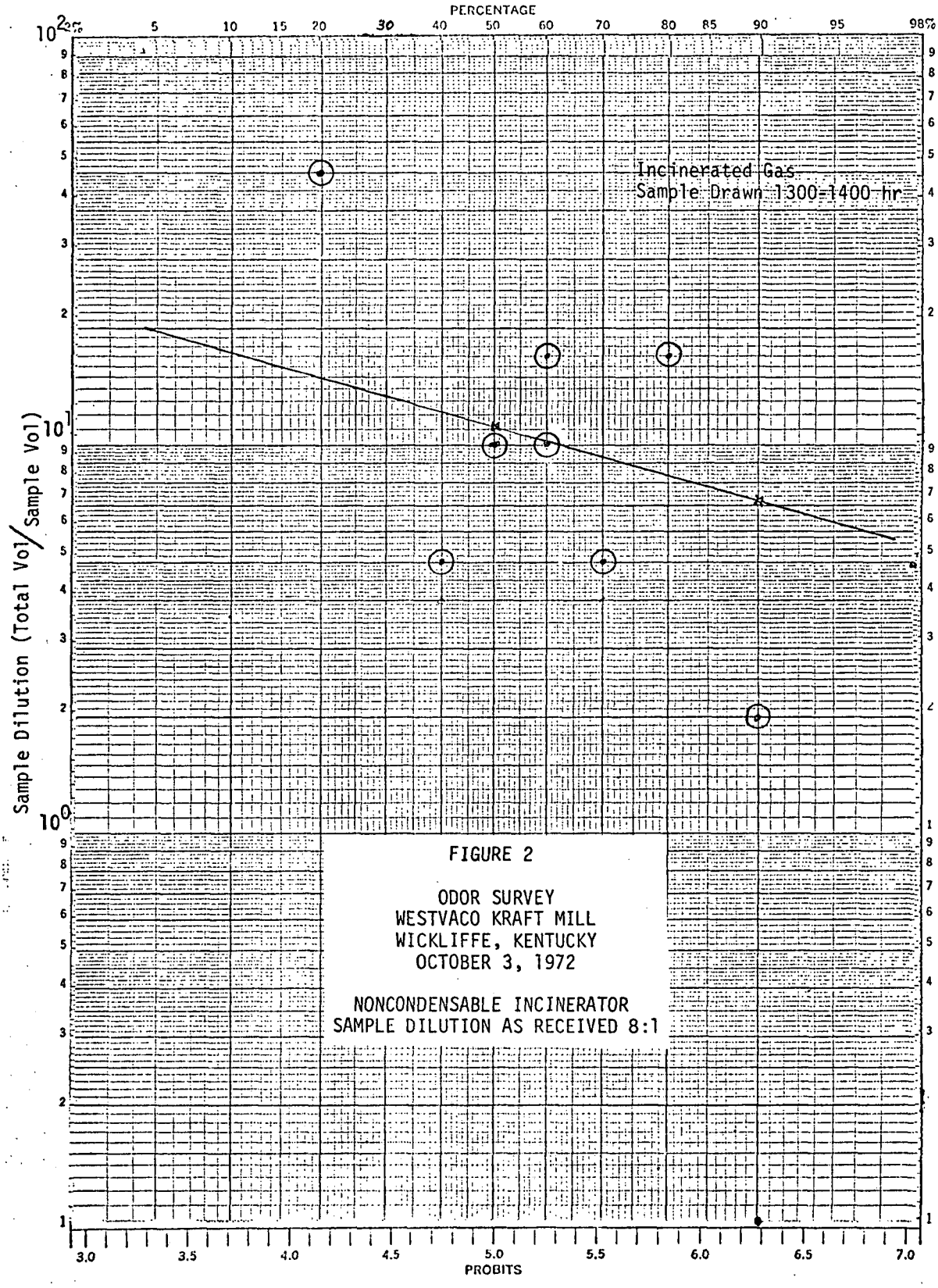
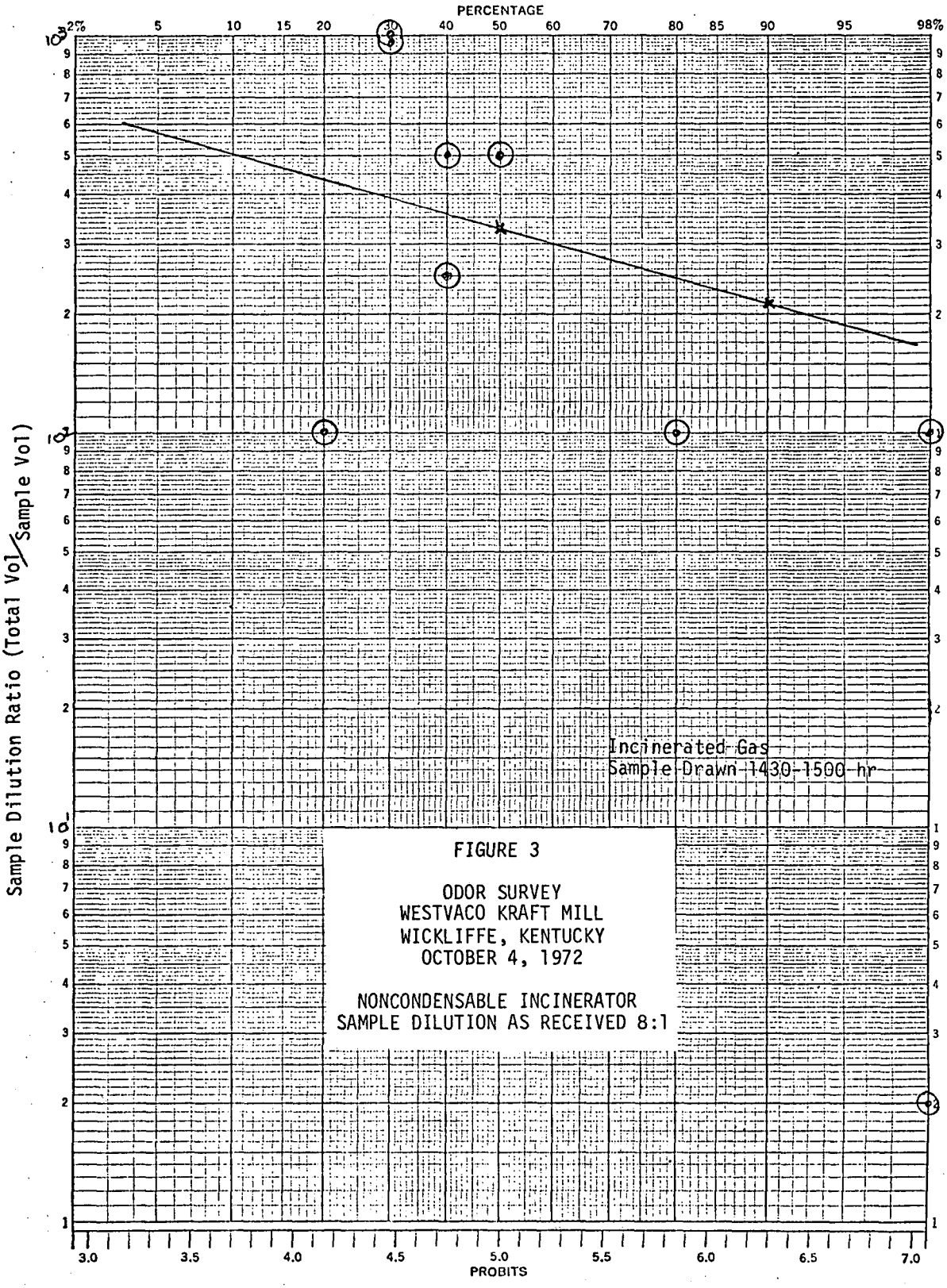


FIGURE 2

ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER 3, 1972

NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 8:1


PERCENTAGE
2%
5
10
15
20
30
40
50
60
70
80
85
90
95
98%
Sample Dilution Ratio (Total Vol/Sample Vol)
Incinerated Gas
Sample Drawn 1430-1500 hr
FIGURE 3
ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER 4, 1972
NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 8:1
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
PROBITS
Percentage of Panel with Odor Detection

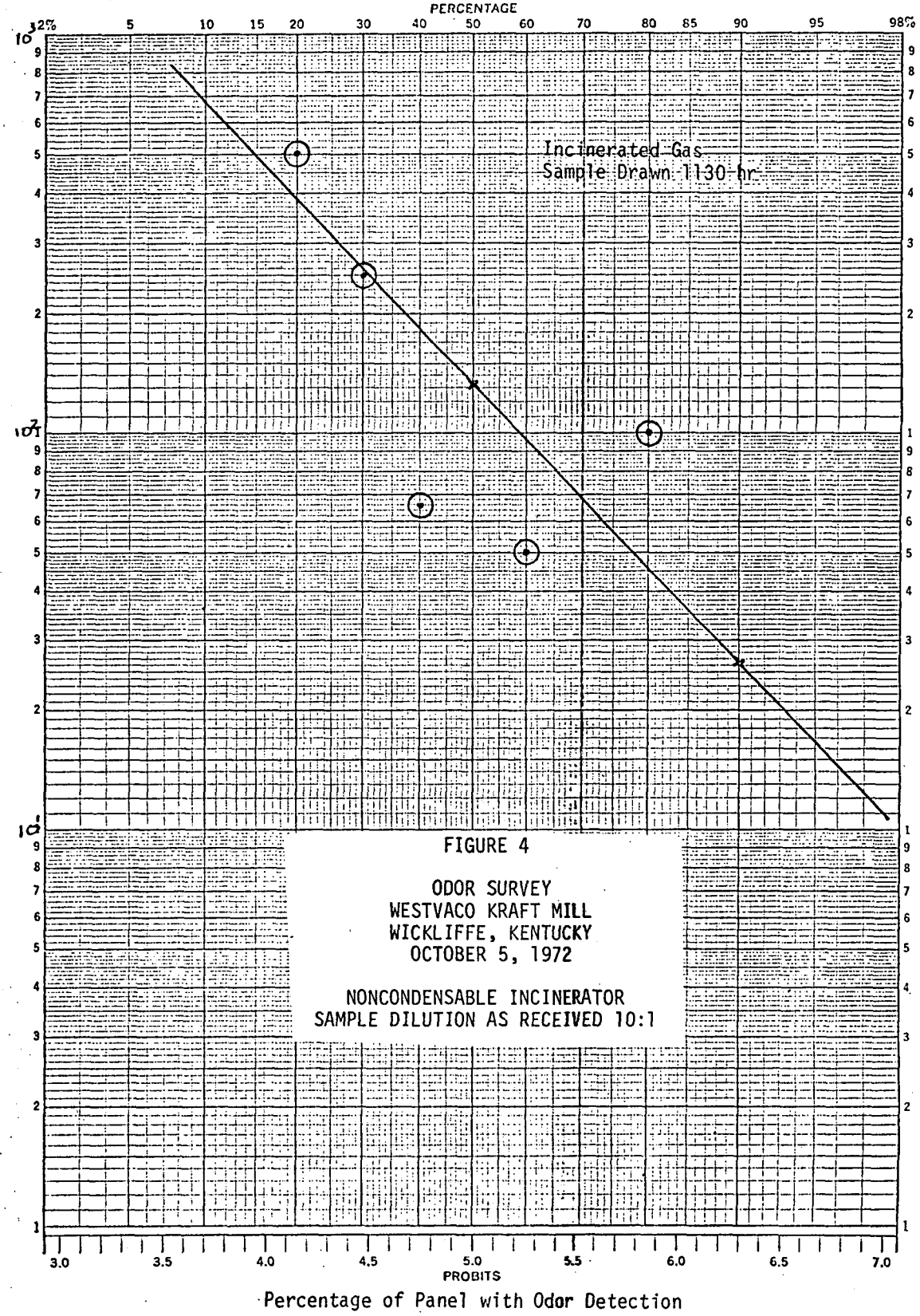


FIGURE 4

ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER 5, 1972

NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 10:1

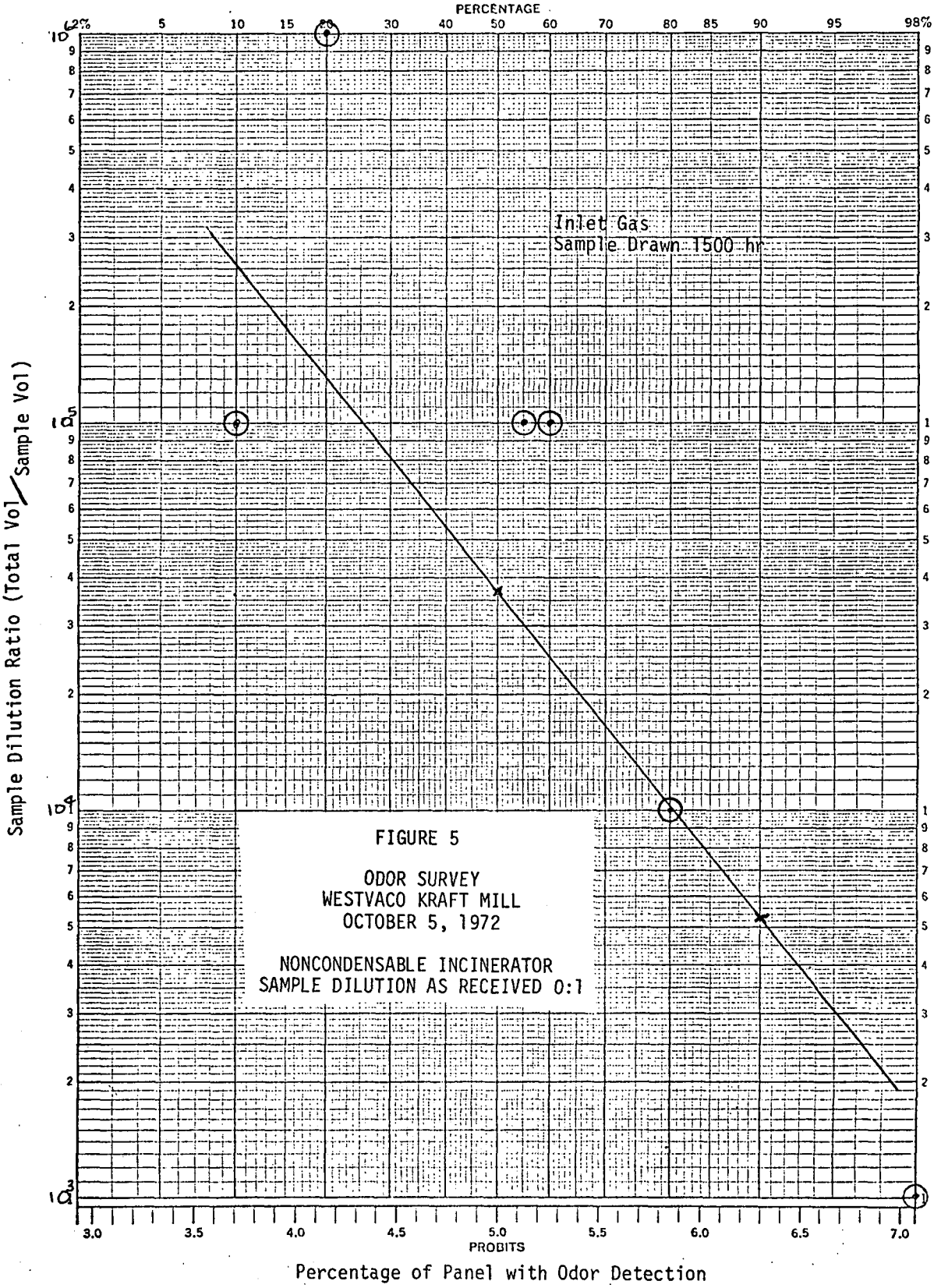
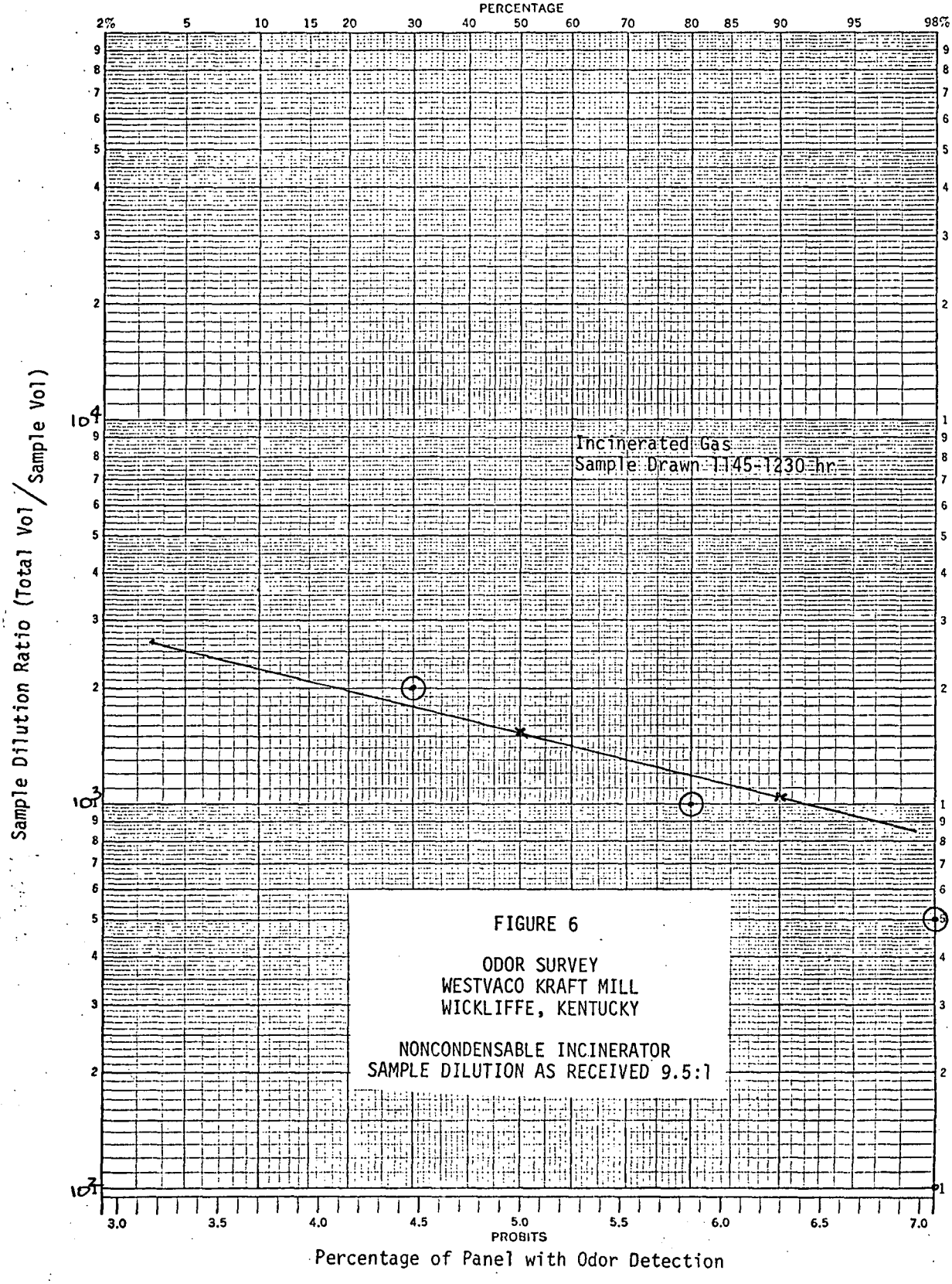


FIGURE 5

ODOR SURVEY
WESTVACO KRAFT MILL
OCTOBER 5, 1972

NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 0:1


PERCENTAGE
2%
5
10
15
20
30
40
50
60
70
80
85
90
95
98%
Sample Dilution Ratio (Total Vol / Sample Vol)
Incinerated Gas
Sample Drawn 1145-1230 hr
FIGURE 6
ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 9.5:1
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
PROBITS
Percentage of Panel with Odor Detection

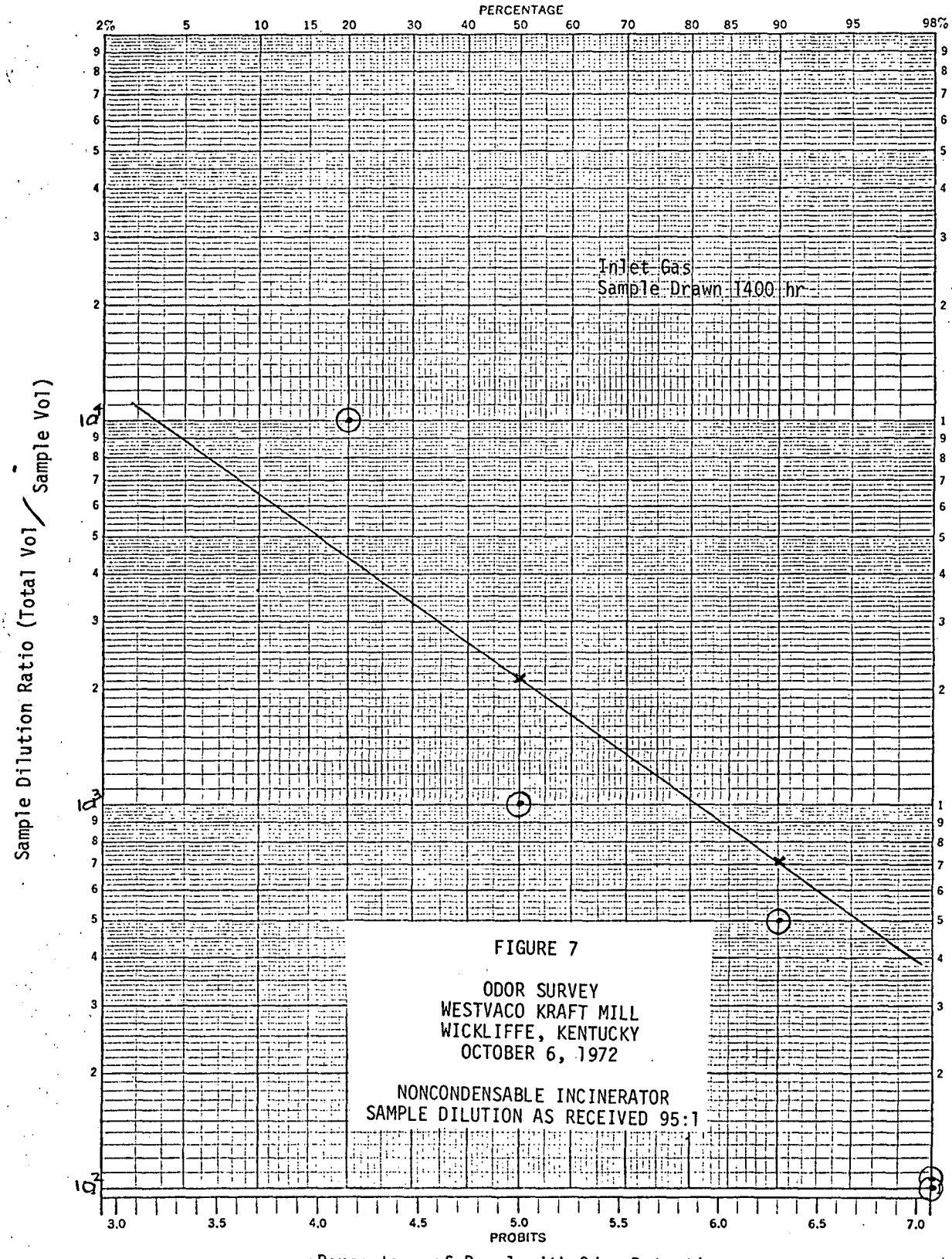


FIGURE 7

ODOR SURVEY
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER 6, 1972

NONCONDENSABLE INCINERATOR
SAMPLE DILUTION AS RECEIVED 95:1

3. Each individual member should keep his detection or non-detection confidential until the entire panel has recorded their respective findings.

4. Where the prevalent odor is not vividly distinct, the panel should be allowed a reasonably strong "refresher" sample between each evaluation sample.

5. It is preferable to introduce samples to the panel generally from a weak to stronger odor progression.

6. The entire series should be conducted as rapidly as permissable but not at a frequency in excess of one sample per 10-15 minutes. It was found that after a continuous evaluation period of approximately two hours, the panel members became somewhat fatigued and psychologically exhausted and required a short recess; preferably leaving the odor evaluation room and returning approximately one half hour later. This procedure seemed to improve consistency among the panel members.

7. A psychological problem exists in that panel members seem to have an innate feeling that it is a virtue to detect an odor. The repeated positive responses to pure air demonstrates this problem. Through repeated lecturing, however, this tendency was reasonably overcome toward the end of the test program.

The original data sheets from which the data of Figures 2 through 7 were derived are presented in Appendix B.

Table 2 has been prepared through which total odor emission rates may be estimated, upon the addition of volume flow data available to EPA.

TABLE 2

ODOR EMISSIONS
WESTVACO KRAFT MILL
WICKLIFFE, KENTUCKY
OCTOBER, 1972

Source	Date	C	VA	E
Outlet	10/3/72	90	----	----
Outlet	10/4/72	2640	----	----
Outlet	10/5/72	1320	2610	3.45×10^6
Outlet (No Fire)	10/5/72	37,000	3000	1.11×10^8
Outlet	10/6/72	14,490	2223	3.22×10^7
Outlet (No Fire)	10/6/72	203,300	2608	5.30×10^8

C = Odor concentration in odor units per cubic foot (derived at 50 percentile detection point)

VA = Volume flow rate in stack, standard conditions, SCFM (70°F and 29.92 in. Hg)

E = Odor emission rate, in odor units per minute, E = CVA

APPENDIX A

DILUTION METHOD OF ODOR MEASUREMENT

METHOD 11 - DILUTION METHOD OF ODOR MEASUREMENT

1. Principle and Applicability

1.1 Principle. A sample of gas is extracted from the emission source to be measured, and is diluted with odor-free air until a dilution is achieved in which the odor can barely be perceived. The ratio of the total volume of the diluted sample to the volume of original sample taken for dilution, is a measure of the odor concentration of the original sample. The technique is not intended to identify individual odor-causing materials or their concentrations, and does not take into account the character of an odor.

1.2 Applicability. This method is applicable for the determination of odorous emissions from stationary sources only when specified by test procedures for determining compliance with the New Source Performance Standards.

2. Range and Sensitivity

2.1 Range. The lower limit of this method is that concentration of odor which can just barely be perceived (odor threshold) at a 1:9 dilution ratio. The upper limit is that concentration which produces the odor threshold at a dilution ratio of 1:9999.

2.2 Sensitivity. The sensitivity depends upon the human olfactory sense, and is subject to variations of this sense, from person to person or from hour to hour in the same person.

3. Interferences

3.1 Extraneous odors interfere in the test and all foreign odors must therefore be eliminated from the test environment. Hands and clothing of the observers, and any necessary equipment, must be clean and odor-free.

3.2 Colds and other physical conditions affecting the sense of smell will interfere with the observers' perception of odors. Use of tobacco, gum, or even eating, can affect the sense of smell, and should not be indulged in for at least 30 minutes prior to the evaluation of odors. To avoid fatigue of the olfactory sense, the observer should carry out the odor test for no longer than fifteen minutes at a time, with a fifteen minute rest period between tests.

3.3 Some aromatic compounds desensitize the olfactory response and will cause erratic results. The only solution to this problem is longer rest periods between tests.

4. Precision and Accuracy

4.1 The precision and accuracy of this method depends on the number, physical condition, experience and skill of the observers. Consistent and reproducible results have been found to be obtained with a panel consisting of at least eight qualified observers. Any single observer should be able to attain results that are reproducible within ± 50%, on any given day.

5. Apparatus

5.1 Sampling

5.1.1 Sampling Syringe - Two 100-ml Luer-type hypodermic syringes.

5.1.2 Syringe Caps - One Luer syringe cap for each syring.

5.2 Analysis

5.2.1 Odor-Free Room - Maintained at comfortable temperature and humidity conditions.

5.2.2 Dilution Syringe - 100-ml Luer-type hypodermic syringes identical to sampling syringes.

5.2.3 Transfer Syringe - Two or more 2-ml Luer-type hypodermic syringes and one 100-ml Luer-type hypodermic syringe.

5.2.4 Transfer Needle - A fitting for connecting the transfer syringe with the sampling and dilution syringes, made from two standard 25 gage hypodermic needles, 1 1/2 in. long (available from Becton-Dickinson and Company, Rutherford, New Jersey). The mating head of one needle is cut off at a point where its inside bore is equal to the outside diameter of the needle shaft. The mating head is slid over the other needle, with the mating opening toward the tip of the needle, and silver soldered in place.

5.2.5 Syringe Caps - One Luer syringe cap for each syringe.

6. Reagents

6.1 Odor-free air.

6.2 Vanillan - 1.0 percent solution in benzyl benzoate.

6.3 Methyl Salicylate - 1.0 percent solution in benzyl benzoate.

7. Procedure

All syringes and transfer needles used in this procedure must be thoroughly washed with an unperfumed detergent, rinsed thoroughly in odor-free tap water, and allowed to dry in the test room atmosphere for at least 15 minutes.

7.1 Sampling. Fill two sampling syringes with the gas whose odor concentration is to be measured, by pushing the plunger all the way in, inserting the tip into the gas stream to be sampled, and pulling the plunger out to the 100-ml mark. Place caps over the tips of the syringes, and transfer to the odor evaluation room. Perform associated measurements such as velocity determination at the time of sampling according to EPA recommended procedures.

7.2 Analysis.

7.2.1 Sample Dilution: Dilution samples are prepared by an assistant and presented blindly to the observer in random order to prevent possible bias due to the anticipation of the observer. In preparing the dilution, place the transfer needle on the transfer syringe, remove the cap from the sampling syringe, and with the transfer syringe empty, insert the transfer needle tip into the sample syringe, and tightly connect the two syringes. Withdraw the desired volume, V_s, of sample into the transfer syringe. Withdraw the needle from the sampling syringe and recap the sampling syringe. Insert the transfer needle tip into the dilution syringe, which has been partially filled with odor-free air. Inject the sample volume, V_s, into the dilution syringe, withdraw the transfer needle, and fill the dilution syringe to the 100-ml mark with odor-free air. Cap the dilution syringe and let it stand for at least 15 sec. to allow mixing by diffusion. The diluted sample is then ready for odor evaluation.

When it is necessary to dilute volumes of 2 ml or less, use the 2-ml transfer syringe. When diluting volumes of less than 0.2 ml, make an intermediate dilution of 1:9 with odor-free air and inject a portion of this intermediate dilution into the dilution syringe. Use the 100-ml transfer syringe for diluting volumes greater than 2 ml.

7.2.2 Odor Evaluation: Prepare samples having dilution ratios of 1:9, 1:99, 1:999, and 1:9999 in random order as described in section 7.2.1. Uncap the dilution syringe and (1) insert the tip of the syringe into one nostril; or (2) hold the tip of the syringe near the nose. Each panel

member should chose the method of smelling the sample which yields the most accurate and reproducible results for him. Suspend breathing for a few seconds, and during this period, expel the 100-ml diluted sample into the nostril or near the nose at a uniform rate over about 2-3 sec. Record whether odor is perceived by the observer, and note the greatest dilution at which odor is perceived.

Based on the above results, prepare a series of dilutions in the range between the greatest dilution in which odor was perceived and the next greatest dilution. The order of dilutions should be random, and at least one out of every four consecutive dilutions should be a "scramble" dilution in no way related to the fundamental trend. The "scramble" odor may range from no odor to considerably above the threshold concentration, and is used to assure that the observer cannot anticipate what the next concentration will be. In this way, he is forced to concentrate only on what he perceives on any given sample. Proceed with dilutions until the difference between the greatest dilution at which odor is consistently perceived and the next greatest dilution measured is less than 50 percent of the greatest dilution at which it is consistently perceived.

8. Calibration

The odor panel shall consist of at least eight persons. Since not all persons are capable of carrying out the test, a group of at least twice the number of observers required shall be screened to select the most sensitive individuals for observers. The screening test consists of a "triangle" test in which two identical samples and one odd sample are presented to the prospective observers in increasingly dilute concentrations. Each person is scored on his ability to distinguish the odd odorant from the two identical ones as dilution increases.

To conduct the screening test, prepare two identical solutions of 1.0 percent vanillan in benzyl benzoate, and a solution of 1.0 percent methyl salicylate in benzyl benzoate. Present these to the group in increasingly dilute concentrations until 50 percent are unable to distinguish the odd odorant from the two identical ones. The remaining members of the group, with better olfactory perception shall be selected as observers.

9. Calculations

9.1 Odor Concentration. Calculate the odor concentration in odor units per cubic foot.

$$C = \frac{100}{V_s}$$

where:
C = Odor concentration in odor units per cubic foot, which is equal to the number of cubic feet that one cubic foot of sample will occupy when diluted to the odor threshold. One odor unit is defined as one cubic foot of air at the odor threshold.
V_s = Milliliters of original sample present in the most dilute sample in which odor is perceptible.
100 = Milliliters of diluted sample.

9.2 Odor Emission Rate. Calculate the odor emission rate in odor units per minute.

$$E = CVA$$

where:
E = Odor emission rate, in odor units per minute
C = Odor concentration, in odor units per cubic foot, as calculated in Section 9.1.
V = Velocity of stack or vent discharge in feet per minute
A = Cross-sectional area of stack or vent, in square feet.

10. Bibliography

10.1 ASTM Book of Standards, Part 23, pp. 301-304, 1971.

APPENDIX B

ORIGINAL DATA SHEETS

Bag Sample

DATE 10-3-72

OBSERVERS	Straight	1/2	1/5	1/5	1/10	0
Brockman	+	+	+	−	−	−
B. Scott	+	+	+	−	−	−
Cave	+	+	+	+	+	−
Fowler	+	+	+	+	+	⊕ DOUBTFUL
Street	+	+	+	+	+	−
Johnson	+	+	+	+	−	−
Bourne	+	+	+	+	−	−
Morreau	+	+	+	+	+	−
Smith	+	+	+	+	+	−
Davis	−	−	+	−	−	−
	9/10	9/10	10/10	7/10	5/10	1/10
					✓	

Bag contents out of EPA dilution system.
Bag as filled was ~ 8 times diluted
Incinerator operating
Sample drawn 1 - 2 PM

DATE 10-3-72

OBSERVERS	1/16.67 3/50	1/5 10/50	1/16.67 3/50	1/10 5/50	1/50	
BROCKMAN	+	−	−	−	−	
B. SCOTT	+	(−) DOUBTFUL	+	−	−	
CAVE	+	+	+	(+) DOUBTFUL	−	
FOWLER	+	−	+	(−) DOUBTFUL	−	
STREET	+	+	(+) DOUBTFUL	+	+	
JOHNSON	−	+	−	+	−	
BOURNE	+	−	+	(+) DOUBTFUL	−	
MORREAU	+	−	−	(−) DOUBTFUL	−	
SMITH	+	+	+	+	−	
DAVIS	−	−	−	+	+	
	8/10	4/10	6/10	6/10	2/10	

✓

DATE 10-4-72

OBSERVERS	1/2	1/100	1/500	1/1000	1/100	
BROCKMAN	+	+	+	+	−	
SCOTT	+	+	−	−	−	
CAVE	+	+	−	−	−	
FOWLER	+	+	+	⊕ DOUBTFUL	−	
STREET	+	−	−	−	−	
JOHNSON	+	+	+	−	−	
BOURNE	+	−	−	−	−	
MORREAU	+	+	+	−	+	
SMITH	+	+	−	⊕ DOUBTFUL	−	
DAVIS	+	+	+	+	+	
	10/10	8/10	5/10	3/10	2/10	

Bag contents had dilution of ~ 8 from stack
Taken from EPA dilution System
Sample drawn 2:30 - 3:00 PM

2nd Set.

30 MIN. BREAK – CLEAN SYRINGES

DATE 10-4-72

OBSERVERS	CLEAN AIR	20 cc SAMPLE GAS ✓ 1/100	20 cc SAMPLE GAS ✓ 1/500	20 cc sample gas's ✓ 1/250	20 cc sample gas ✓ 1/1000	
Brockman	+	+	(+)	–	(+)	
Scott	–	(+)	–	–	–	
Cave	+	+	+	+	+	
Fowler	+	+	–	–	+	
Street	+	+	–	+	–	
Johnson	+	+	–	+	–	
Bourne	–	+	–	–	–	
Morveau	–	+	–	–	–	
Smith	–	+	+	+	–	
Davis	+	+	+	–	–	
	6/10	10/10	4/10	4/10	3/10	

7/100 - 10/10

BAG SAMPLE

DATE 10-5-72

OBSERVERS	20cc BAG SAMPLE 1/500	20cc BAG SAMPLE 1/250	20cc BAG SAMPLE 1/100	NO REFRESHER 1.5/100	20cc BAG SAMPLE PLAIN AIR	20cc BAG SAMPLE 1/50
BROCKMAN	−	+	+	+	−	+
SCOTT	+	−	−	−	−	−
CAVE	−	−	+	+	−	+
FOWLER	−	−	+	+	+	+
STREET	−	−	+	−	−	+
JOHNSON	+	+	+	+	−	+
BOURNE	−	−	−	−	−	−
MORREAU	−	−	+	−	−	+
SMITH	−	+ DOUBTFUL	⊕	−	+	−
DAVIS	−	⊕	+	−	⊕	−
	2/10	3/10	8/10	4/10	3/10	6/10

This bag sample from EPA dilution system with dilution ratio ~10.

Incinerator operating

Odor apparently not

Sample drawn ~ 11:30 AM

SOURCE: EVAP. - DIGESTOR - GUMS

SYRINGE SAMPLE

DATE 10-5-72

OBSERVERS	1/10,000 1/100,000	1/1000 1/10,000	1/100,000 1/1,000,000	1/1,000,000 1/10,000,000	1/100,000 1/1,000,000	KNOWN CONC. 1/100,000 1/1,000,000
BROCKMAN	−	+	+	−	−	−
SCOTT	+	+	−	−	−	−
CAVE	+	+	+	−	−	+
FOWLER	+	+	LOST SAMPLE	−	−	+
STREET	+	+	−	+	−	+
JOHNSON	−	+	−	−	−	−
BOURNE	⊕	+	+	−	+	+
MORREAU	⊕	+	+	−	−	−
SMITH	+	+	+	⊕	−	+
DAVIS	⊕	+	−	−	−	⊕
	8/10	10/10	5/9	2/10	1/10	6/10

This was straight stack gas, no dilution

Incinerator turned off.

Odor strong in H_2S & mercaptan type odor

Sample drawn ~ 3:00 PM

INCIN. OPERATING

BAG SAMPLE

DATE 10/6/72

OBSERVERS	Refrigerator 2000 cc SAMPLE ∧ 1/1,000	2000 SAMPLE ∧ 1/2000	20 cc SAMPLE ∧ 1/100	2000 SAMPLE AIR	20 cc SAMPLE ∧ 1/500	
BROCKMAN	+	−	+	−	+	
SCOTT	−	−	⊕	−	⊕	
CAVE	+	−	+	−	+	
FOWLER	+	−	+	−	+	
STREET	+	+	+	−	+	
JOHNSON	+	−	+	−	+	
BOURNE	+	+	+	−	+	
MORRELL	−	−	+	−	+	
SMITH	+	⊕	+	−	+	
DAVIS	⊕	−	+	−	+	
	8/10	3/10	10/10	0/10	10/10	

Sample drawn 1145 - 1230 hrs
Incinerator Operating
Dilution 9.5:1 before bag.

Direct Sample

DATE 10-6-72

OBSERVERS	1/10,000	1/1000	1/500	1/100	0	1/100
BROCKMAN	−	−	+	+	−	+ ↓ oil drip
SCOTT	−	−	+	+	−	+
CAVE	−	−	+	+	−	+
FOWLER	−	−	−	+	−	+
STREET	+	+	+	+	+	+
JOHNSON	−	+	+	+	−	+
BOURNE	−	−	+	+	−	+
MORREAU	−	⊕	+	+	−	+
SMITH	−	+	+	+	−	+
DAVIS	+	⊕	+	+	−	⊕
	2/10	5/10	9/10	10/10	1/10	10/10

Sample drawn 2PM
Diluted 95:1 in EPA system before I got it