NIOSH/TC/A-001

TEST PROCEDURE FOR GAS DETECTOR TUBE CERTIFICATION

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PREFACE

The authors express their appreciation to Robert Schutz, Michael McCawley, Joan Allender and Lee Hall for their help in preparing the photographs used in this report. Also thanks to Nancy Rice and Nancy Morgan for their efforts in preparing the manuscript. The mention of a manufacturers' product in this report is not to be construed as an endorsement by NIOSH. Equivalent test equipment may be used for this test procedure and should give comparable results.

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LIST OF ILLUSTRATIONS

LIST OF ABBREVIATIONS AND SYMBOLS

MIL-STD – Military Standard

NBS – National Bureau of Standards

NIOSH – National Institute for Occupational Safety and Health

NTS – NIOSH Test Concentration

$\Delta L/M$ – (Maximum Reading - Minimum Reading)/Average Reading

$S/\bar{x}$ – Coefficient of Variation

Max – The Length of the Furthermost extension of an Oblique Stain

Min – The Length of the Least Extended Point of an Oblique Stain

INTRODUCTION

The objective of this paper is to present the operating procedure for the testing of length-of-stain type, gas detector tubes. It is the responsibility of NIOSH to conduct these tests as set forth in 42 CFR 84. The procedure given in this paper is the general procedure applied to all length-of-stain gas detector tubes. Specific methods of gas generation and analysis shall not be presented here. Various methods must be used for each contaminant tested. Gas generation methods, and analytical procedures for several gases have been presented (ref. 1-10), and future papers will cover other gases.

REFERENCE TO TEST REQUIREMENTS

Gas detector tubes are tested according to 42 CFR Part 84. The basis requirements for certification are as follows:

1. The tube must give an accurate reading of the true concentration with a $\pm$ 35% tolerance at 1/2 x NTC, and $\pm$ 25% tolerance at 1, 2, and 5 x NTC.
2. The tube shall either produce a stain greater than 15 mm long at 1 x NTC, or have a coefficient of variation between tube readers of less than 10%.
3. Channeling of airflow through the tube shall be minimized such that $\Delta L/M \leq 0.20$.
4. The packing that interfaces with the indicating reagent at the stained end of the tube shall be at a right angle to the longitudinal axis of the tube. If this interface is oblique, a variation of greater than 2 mm is a defect.

EQUIPMENT LIST

Equipment	Manufacturer	Catalog No.
Optical Micrometer	Bausch & Lomb, Inc.	2542LF
Timer	Fisher Scientific	14-647-20
Gas Chromatograph	Perkin-Elmer	Model 900
Integrator	Autolab	22000-670
Strip Chart Recorder	Perkin-Elmer	056-0009
Syringe	Hamilton	1050
Hygrometer	Arthur H. Thomas	6066H10
Manometer	Datametrics	1174
Sensor	Datametrics	570A-1000T-3A1-H5
Thermometer	Ametek Controls Division	93T2CC1D1
Reader Box	NIOSH	
Clock	Sears Roebuck & Company	7183
Light	General Electric	F15T8.CW
Ruler	NBS	C13.10:376
Camera	Minolta Camera Co.	SR-T-101
Copy Stand	Superior Photo	Stand
Standard Grey	Kodak	Color Test Card
Optical Tube Reader	Food Technology Corporation	1609
Tests for Color Blindness	Graham-Field Surgical Co.	2867-1255

SPECIAL PRECAUTIONS

Gas detector tubes are thin wall glass ampules containing an indicating reagent deposited on inert materials. Often this reagent is corrosive and toxic in itself.

During the conduction of the test, the operator must break off the tips of the ampules (Figure 4). Occasionally, an ampule will shatter, spraying broken glass and reagent. Therefore, face protection (faceshield) and hand safety equipment will be worn during the exposing operation.

CALIBRATION PROCEDURES

No calibration is necessary.

OPERATING PROCEDURE

Packing Interface Testing

After a completed application for testing of gas detector tubes has been received by NIOSH, the detector tubes are checked in, numbered sequentially, and examined for packing interface abnormalities as specified by 42 CFR 84.21(c). A sample of tubes is selected from the lot submitted by the applicant, according to the Sampling Plan, MIL-STD-105D, Level II. The tubes are examined under an optical micrometer, as seen in Figure 1. A count is then made on the number of tubes on which the obliqueness of the packing interface exceeds the 2mm standard. All tubes are then stored according to the manufacturers instructions.

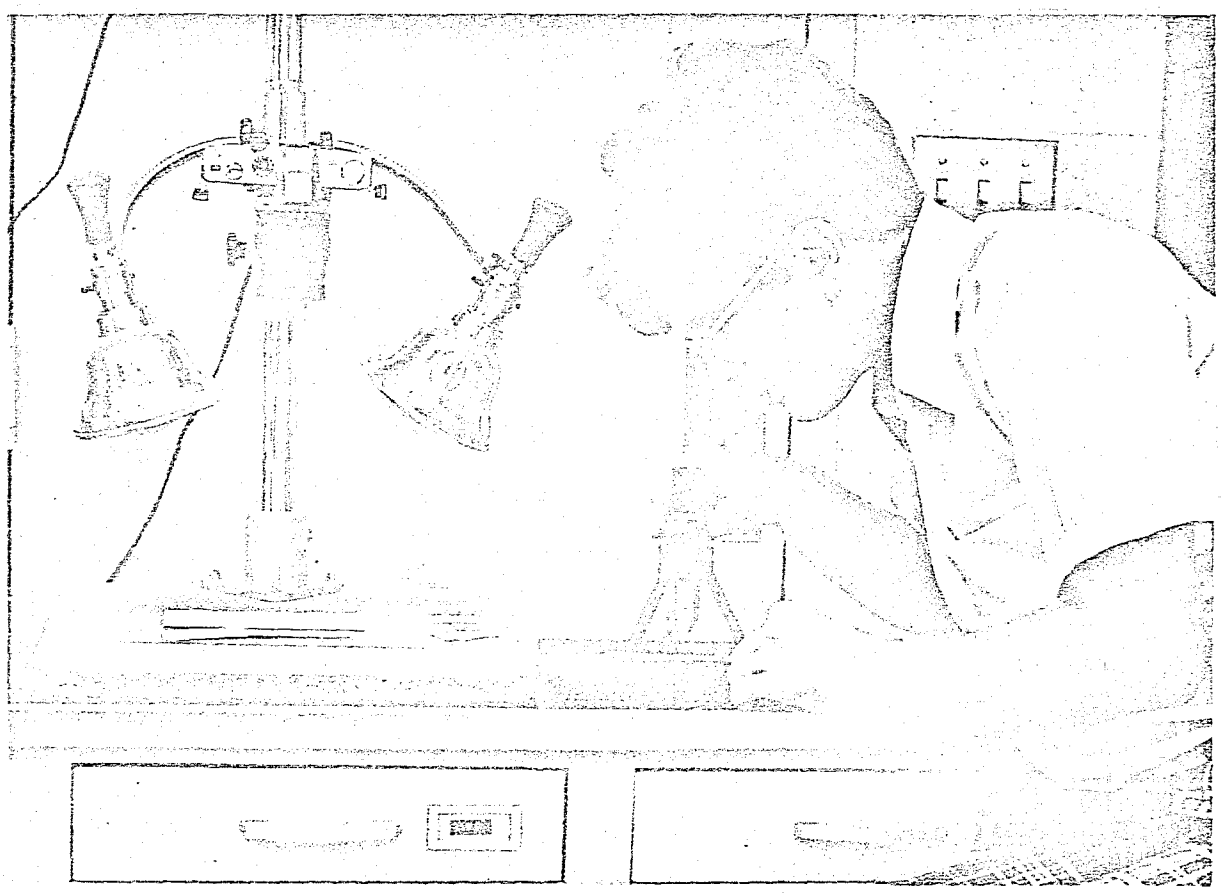


Figure 1 - Packing interface tests. Also pictured is the camera apparatus for maintaining a permanent record.

Accuracy Tests

Using MIL-STD-414, Level II, a sample of tubes is taken from the storage cabinets. The aspirating pumps to be used for the test are given the field tests as specified by the manufacturer. Generally, this consists of a quick test for leaks and a test on flow rate where flow rate is significant.

The gas generation system, such as in Figures 2 and 3, is set up to produce a stable concentration of the contaminant in question. The relative humidity of the test gas atmosphere is adjusted to a value within that stated by the manufacturer, usually 50%. The true relative humidity is determined in one of two ways: (1) for most contaminants, moisture is added to the diluent air stream using a calibrated humidification device built by NBS. (2) Where other methods must be used, e.g. splitting the gas stream and passing one leg through a midget impinger, analysis for water vapor is done on the Gas Chromatograph, utilizing a Hot Wire detector.

A record is kept on the following ambient conditions:

1. Room Temperature
    a. Wet Bulb
    b. Dry Bulb
2. Atmospheric Pressure
3. Test Gas Temperature

The detector tubes are then exposed to the contaminant atmosphere according to the instructions provided by the manufacturer. Figures 2 and 4 show a typical exposure process.

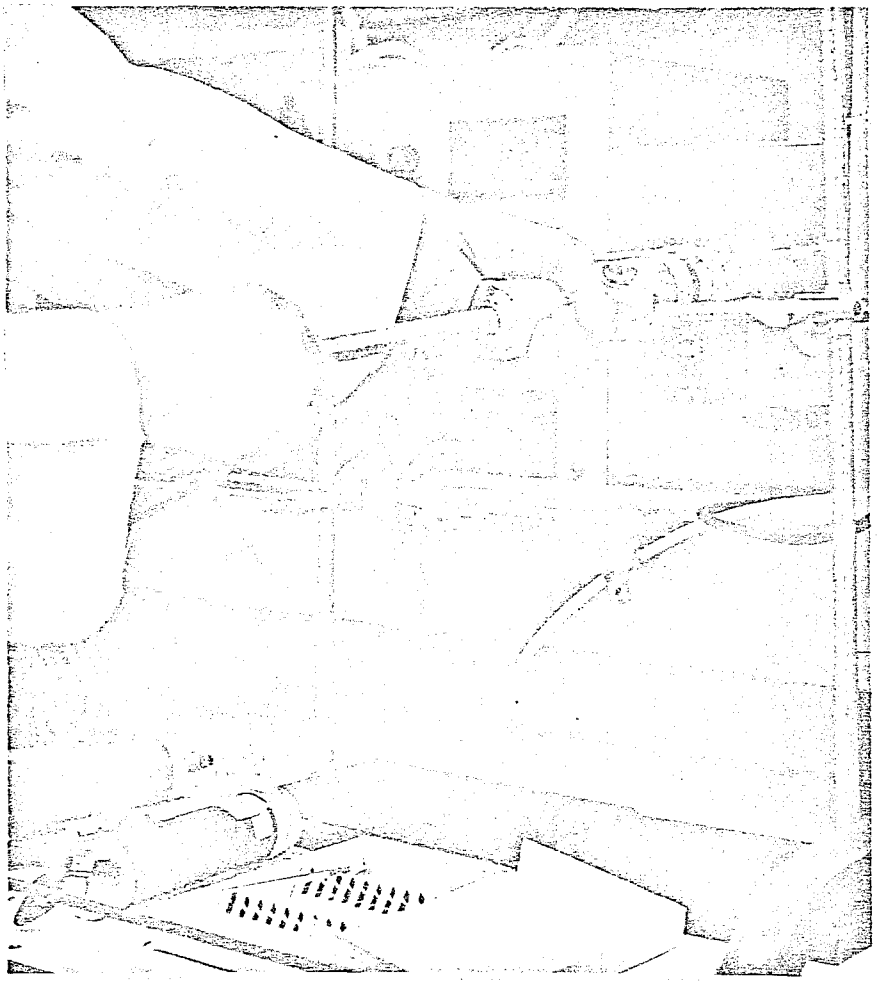


Figure 2 - Exposing detector tubes, gas sampling.

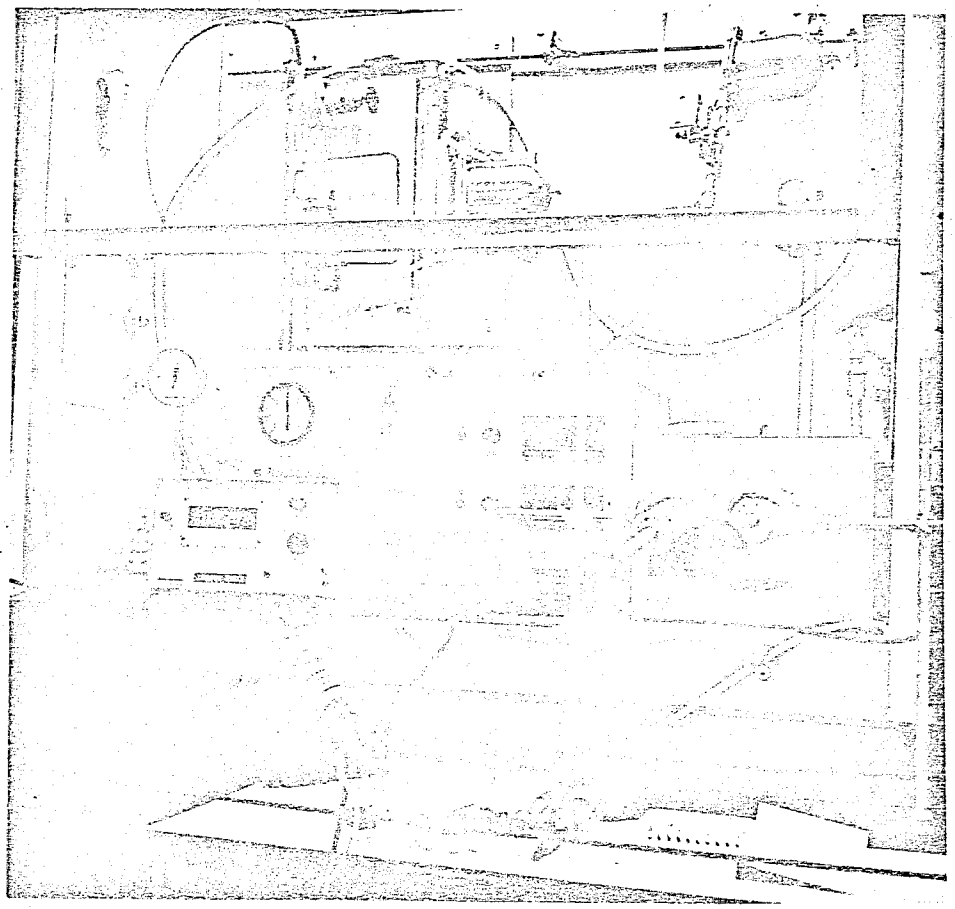


Figure 3 - A gas generation system and test condition monitors.

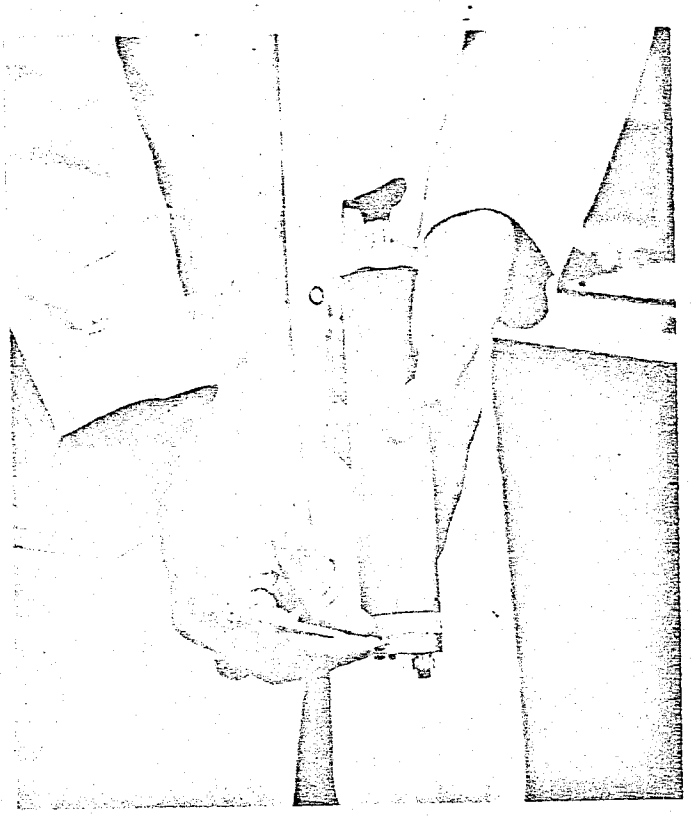


Figure 4 - Breaking the tips of the tube during exposing.

Evaluation of the length-of-stain is made by a panel of three independent tube readers. Each tube reader was checked for their color vision with standard test charts. Only individuals with normal color vision are selected as tube readers. A tube reader has no knowledge of the true test concentration and has no knowledge of the readings of the other two readers. Each reader is provided with a copy of the manufacturers instruction sheet and a general set of instructions, (Appendix A) to guide in evaluation of the channeling effect and stain length.

After the tube has been exposed, it is immediately handed to the first of the three tube readers. The stain length and channeling effect measurements are evaluated in millimeters, and are interpreted for concentration readings using the scale provided by the manufacturer, as shown in Figure 5. The tube is then passed to the next reader for evaluation. After each reader has evaluated each tube, a few representative exposed tubes are selected and are photographed, Figure 1, for a permanent record of the stain characteristics. To determine the effects of time on the tube stain, an optical tube reader, Figure 6, is utilized to evaluate the endpoint of the stain. This device produces a step shaped curve. In most cases, the point of steepest slope of the output curve represents the length-of-stain which would be evaluated by an average human tube reader. A typical optical tube reader output is given in Figure 7. Successive curves are generated for the same tube over the 15 minute period following exposure. In this way, the rate of stain degradation can be determined. If this test determines the three readers do not have enough time to evaluate the tube, the test procedure is modified to allow each reader to evaluate one third of the tubes.

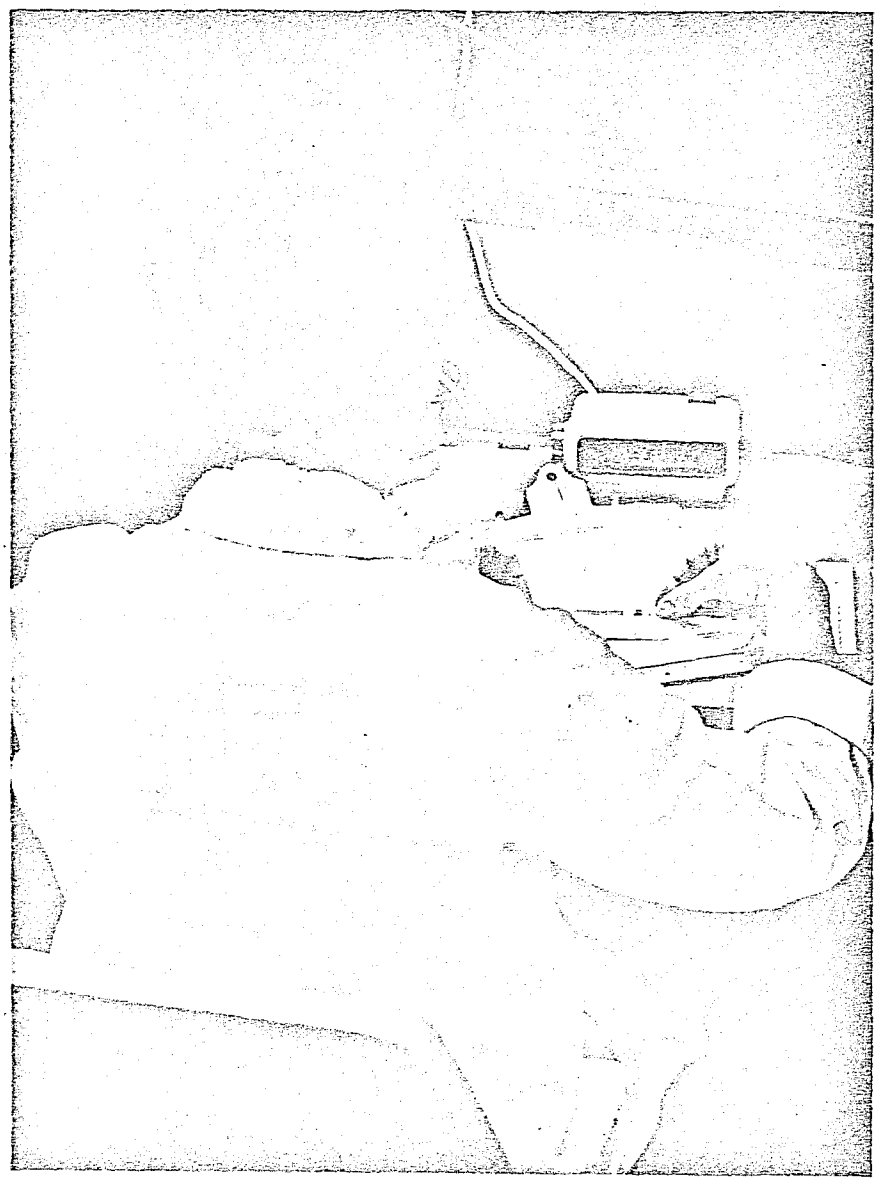


Figure 5 - Tube reading.

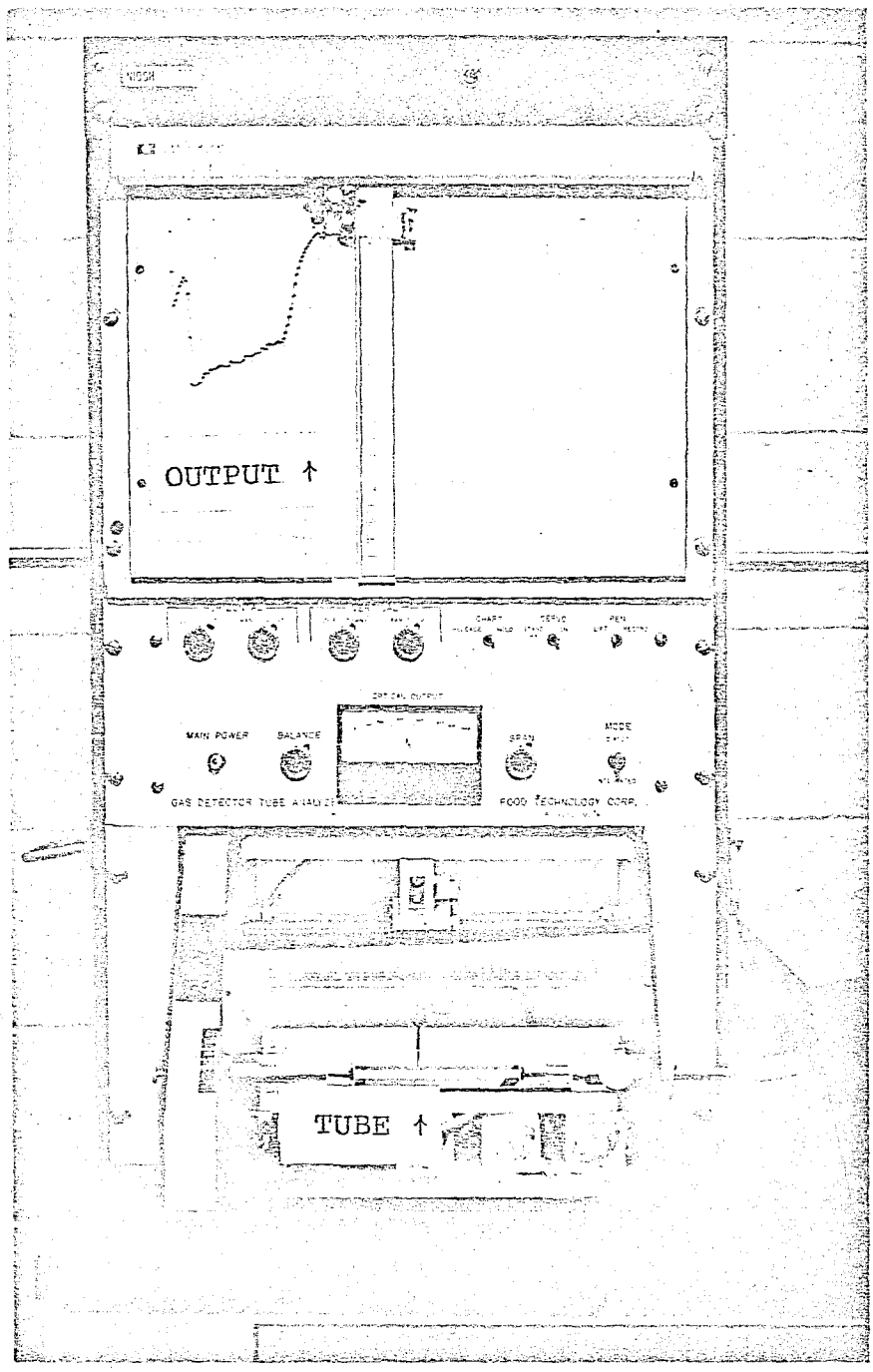


Figure 6 - Optical tube reader.

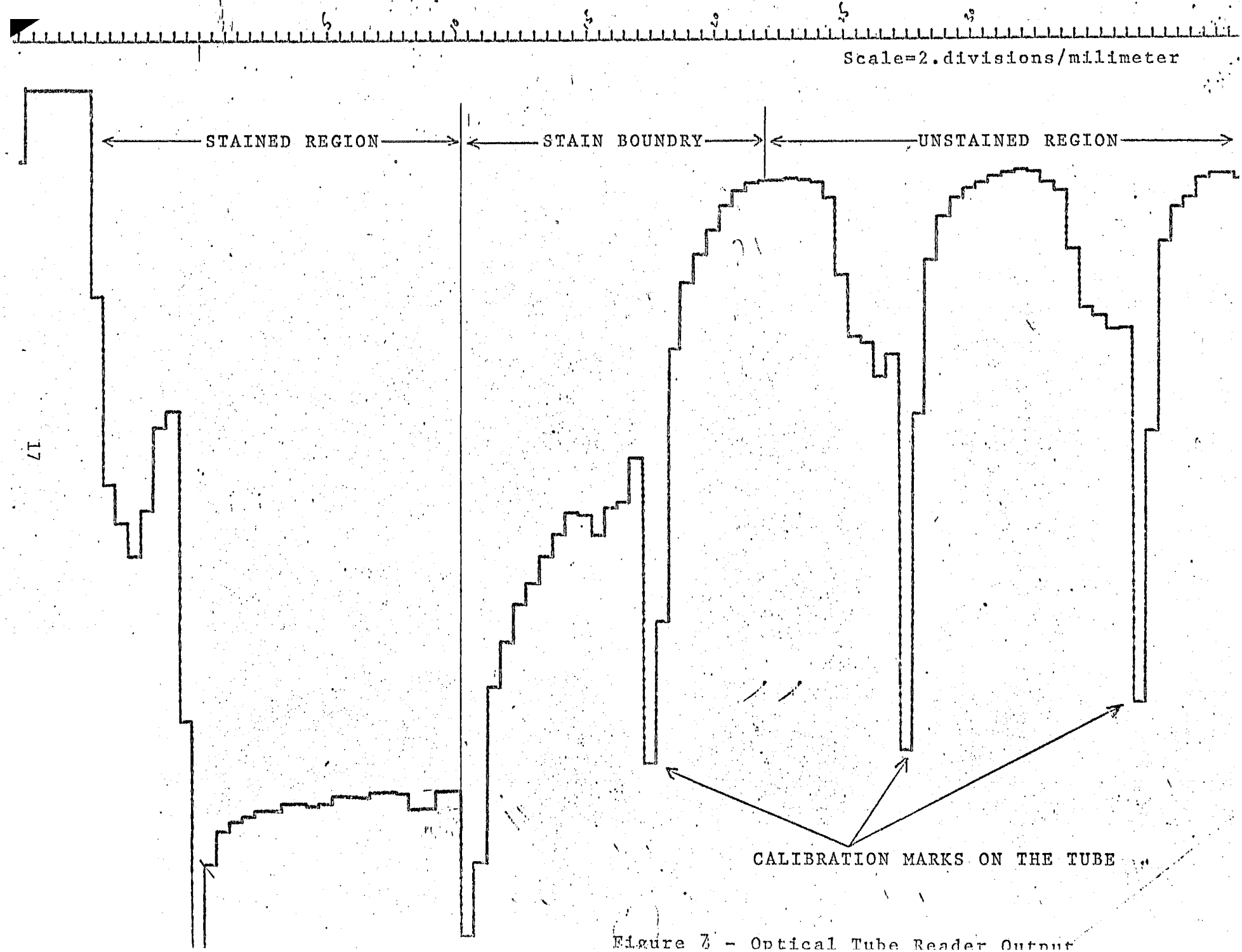


Figure 7 - Optical Tube Reader Output

ANALYSIS OF DATA

The following calculations are made. All calculations are made on individual tube readings which have been corrected per the manufacturer's instructions.

A. Tube accuracy - 42 CFR 84.20(e).

The calculation procedure used is MIL-STD-414 (11 June 1957) double specification, variability unknown. An example (B-3) of this appears on page 43. The variables are specified as:

Sample Size .....Level II
Sample Selection .....Random
Acceptable Quality Level (normal inspections).....6.50
Specification Limits .....Table value times generated concentration

CONCENTRATION	UPPER	LOWER
1/2 x NTC	1.35	.65
1 x NTC	1.25	.75
2 x NTC	1.25	.75
5 x NTC	1.25	.75

Values used - The average of three readers' average reading.

B. Tube channeling - 42 CFR 84.21(b).

The calculation procedure used is MIL-STD-414 (11 June 1957) single specification, variability unknown. An example (B-1) of this calculation appears on page 37. The variables are specified as:

Sample Size .....Level IV
Sample Selection .....Equal number selected randomly from each concentration
Acceptable Quality Level (normal inspections) 2.50
Upper Specification 0.2

Values used - The average of three readers' $\Delta L/Ms$ where

$\Delta L/M$ = (Maximum reading - Minimum reading)/average reading

C. Tube reader deviation - 42 CFR 84.21(a).

If the scale stain length at the NIOSH test concentration (NTC) is less than 15mm, this test is applied. The procedure used is MIL-STD-414 (11 June 1957) single specification, variability unknown. An example (B-1) of it appears on page 37. The variables specified are:

Sample Size .....Level II
Sample Selection .....Those run at 1 x NTC
Acceptable Quality Level (normal inspection 2.50
Upper Specification 0.1

Values Used - S/X where:

S - standard deviation of three or more readers' average readings.

X - the average of the readers' average readings.

D. Packing interface - 42 CFR 84.21(c).

The calculation procedure used is the single sampling plan for MIL-STD-105D (29 April 1963). Reduced inspection is used if the required number of lots (all models) submitted by a manufacturer is satisfactory. The variables specified are:

Sample Size .....Level II
Sample Selection .....Random
Values Used.....count of those greater than 2mm

MAINTENANCE INSTRUCTIONS

Performance of a field test for leaks and flow should be carried out as per manufacturers instructions. Service to the aspirating pumps is performed as needed according to the method specified by the manufacturer.

APPENDIX A

GENERAL SET OF INSTRUCTIONS PROVIDED TO EACH TUBE READER

TUBE READERS GENERAL INSTRUCTIONS

WE ARE CERTIFYING A MANUFACTURER TO PRODUCE A NIOSH CERTIFIED PRODUCT. THE MANUFACTURER PAYS NIOSH $350 TO DO THE TESTING. THE PRODUCT IS THEN USED BY OSHA, MESA, AND INDUSTRY TO DETERMINE SAFE WORKING ATMOSPHERES. THE FOLLOWING IS A GENERAL GUIDE.

1. READ THE INSTRUCTIONS CAREFULLY AND THOROUGHLY. ASK QUESTIONS ABOUT ANYTHING OF WHICH YOU ARE NOT SURE.

2. DO NOT OBSERVE THE OTHER READER'S RESULTS. INDEPENDENT READINGS ARE NECESSARY.

3. READ THE TUBE AS ACCURATELY AS POSSIBLE. INTERPOLATE BETWEEN SCALE MARKS IF PRACTICAL. (CHECK TO SEE IF SCALE IS LINEAR WHEN INTERPOLATING). WRITE DOWN ALL READINGS.

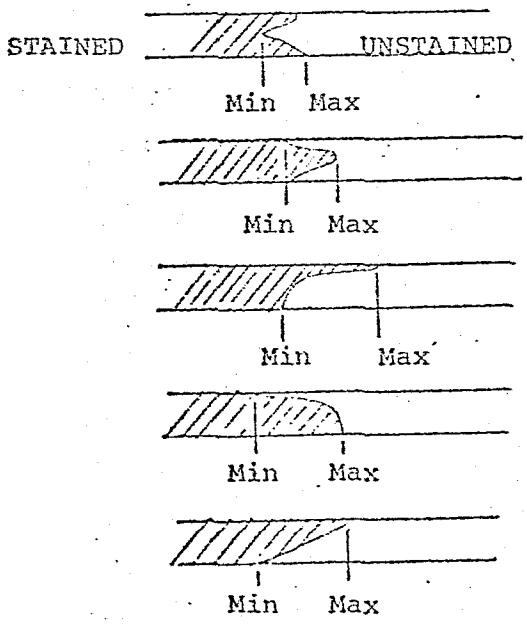
4. TRY DIFFERENT LIGHTS (FLUORESCENT, INCANDESCENT, OR NATURAL), DIFFERENT BACKGROUND COLORS, DIFFERENT DISTANCES FROM EYES, AND ROTATION OF THE TUBE TO GET THE PLAINEST END POINT.

5. RECORD ONE AVERAGE READING FOR THE ACCURACY TEST.

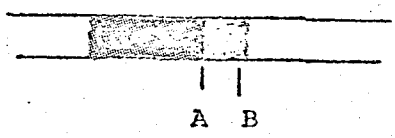
6. RECORD THE MAX AND MIN READINGS FOR THE CHANNELING TEST (SEE BELOW). NOTHING SAYS THAT THE THREE READINGS YOU OBTAIN MUST BE DIFFERENT OR THE SAME - IT'S THE WAY YOU INTERPRET IT.

INTERPRETING DETECTOR TUBE "MAX" AND "MIN" READINGS

MAX AND MIN - DIFFERENT



MAX AND MIN - SAME



MAX & MIN SHOULD BE THE SAME- EITHER A, B, OR SOME VALUE IN BETWEEN. THE VALUE CHOSEN WOULD BE DETERMINED BY HOW THE INSTRUCTIONS SAY TO INTERPRET "END OF STAIN"

7. NOTE ANY DIFFICULTIES ENCOUNTERED OR GENERAL COMMENTS. EXAMPLES
   - END POINT VERY DIFFICULT TO DETERMINE
   - END POINT MUCH EASIER TO READ WITH ORANGE BACKGROUND
   - VERY DIFFICULT TO READ UNDER FLUORESCENT LIGHTS

APPENDIX B

REFERENCES

REFERENCES

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2. E. E. Hughes, W. D. Dorko, J. K. Taylor, System for Preparation of Known Concentrations of Methane in Air, NBS Report NBSIR 73-255, October 1973.

3. P. A. Pella, E. E. Hughes, J. K. Taylor, A Gas Dilution System for Hydrogen Fluoride-Air Mixtures, NBS Report NBSIR 73-261, October 1973.

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9. MIL-STD-105D "Sampling Procedures and Tables for Inspection by Attributes" Government Printing Office 1971 0-414-996.

10. MIL-STD-414 (11 June 1957) "Sampling Procedures and Tables for Inspection by Variables for Percent Defective" Government Printing Office 1971-433-696/9052.