

UNCLASSIFIED

Copy 15 of 17A

~~RESTRICTED~~

KIA-387

Classification changed to Unclassified by
authority of Paul B. David
by Paul B. David 8-24-70
Revised by C.W. Huntington 8/27/79

Contract Number AT-33-1-GEN-53

MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

APR 16 1955

OSTI

L. M. Haring
Laboratory Director

TESTS FOR HYDROGEN CYANIDE AND HYDROGEN SULFIDE

(Information Report)

This document is
PUBLICLY RELEASABLE

James H. Hara (OSTI)
Authorizing Official

Date: 6-30-09

Date: August 24, 1949

Prepared by: E. F. Joy

UNCLASSIFIED

UNCLASSIFIED

MLM-387

DISTRIBUTION

1. - M. M. Haring
2. - J. J. Burbage
3. - R. A. Staniforth
4. - J. L. Svirbely
5. - Area Manager
6. - Area Manager
7. - Area Manager
8. - D. L. Scott
9. - J. R. Wiesler
10. - C. H. Pittenger
11. - R. K. Harris
12. - C. M. Alspaugh
13. - E. F. Joy
14. - Central Files
15. - Central Files
16. - Central Files
17. - Central Files

Issued: NOV 16 1949

UNCLASSIFIED

INTRODUCTION

A potential source of dangerous concentrations of hydrogen cyanide exists in the plating room of the Machine Shop where open plating baths containing cyanide salts are maintained and where solid cyanide salts are stored. Also the use of hydrogen sulfide in certain steps of the waste disposal process has lead to noticeable and sometimes objectionable concentrations of this gas in the air of the "TD" Building. In view of the toxic properties of these two gases, it was desirable to set up suitable tests to determine the actual concentrations present in the air of the respective working areas.

SUMMARY AND CONCLUSIONS

Standard tests for hydrogen cyanide and hydrogen sulfide described in "Manual of Industrial Health Hazards" by J. B. Ficklen were adapted for use with the wall-type air samplers (Mine Safety Appliances Company) now in use at Mound Laboratory, Miamisburg, Ohio. Suitable test papers were prepared and tested in the laboratory and in the areas in question. The concentration of hydrogen cyanide found in the plating room was very low and should cause no concern until there is a change in operating conditions in this area. Hydrogen sulfide was found in objectionable concentrations during one test in the "TD" Building. However, when the ventilating fans were operating effectively and connecting lines were tight, there was no detectable concentration of hydrogen sulfide in the area. The procedures used and the results of preliminary tests follow.

DETAILED REPORTTest for Hydrogen Cyanide

This method depends upon the blue coloration produced on a moist paper impregnated with a mixture of copper and benzidine acetates when exposed to air containing hydrogen cyanide.

Reagents - Copper Benzidine Acetate Paper - Solution I, dissolve 3.0 grams of copper acetate, $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot \text{H}_2\text{O}$, in 100 cc. of water. Solution II, heat 2.3 grams of benzidine acetate to 80°C . in 100 cc. of water for 10 to 15 minutes with constant shaking. Cool and filter. Solution is stable for two days. To prepare paper, mix 25 cc. of the benzidine solution with 2 cc. of the copper acetate solution. Dip two-inch wide strips of extra thick white filter paper (Whatman No. 3) into this solution immediately. After one minute allow to drain, dry in warm atmosphere free from fumes until just moist. Cut out 4.25 cm. circles of the paper to fit the air sampler and use immediately. Place piece of paper in holder and sample the air at the rate of 10 cc. per sec. for a period of time in seconds, as given in Table I, such as will give a suitable stain for comparison. Compare the test paper with the color chart on page 106, in the book by J. B. Ficklen.

Notes

1. A rate of 10 cc. per sec. is obtained with the wall-type sampler by setting the rotor at the lowest calibration mark. This rate was checked by using a calibrated rotameter in the air line just beyond the sampler.
2. A portable air sampler can be used as a source of vacuum if an installed vacuum line is not at hand.

UNCLASSIFIED

Table ISAMPLING TIME WITH WALL-TYPE SAMPLER CORRESPONDING
TO SAMPLING WITH HAND PUMP FOR HYDROGEN CYANIDE

<u>Sampling Time Seconds*</u>	<u>No. of Strokes**</u>	<u>Volume of Air cc.</u>
25	2	250
38	3	375
63	5	625
100	8	1000
125	10	1250

* At sampling rate of 10 cc./sec., wall-type sampler.

** Using hand pump of 125 cc. capacity per stroke as given by
J. B. Ficklen, see reference on page 3.

The method was tested in the laboratory by drawing a known amount of hydrogen cyanide corresponding to 20 parts per million in a liter of air through a test paper and comparing the stain produced with that given in the chart for this concentration. The match was good. The small amount of hydrogen cyanide required was obtained by transferring a 25 microliter portion of a sodium cyanide solution containing 0.182 grams of NaCN in 100 cc. of water to a flask and adding a drop of sulfuric acid. All of the hydrogen cyanide released (0.025 mg.) was drawn through the test paper by aspirating air through the flask.

Tests for Hydrogen Cyanide in the Plating Room of the Machine Shop

Tests were made on the air in the plating room on August 19, 1949 at a time while plating was not being carried on and while the ventilating fans were not operating. Qualitative tests were made in the air immediately over the surface of each of the baths containing cyanide. Hydrogen cyanide was present only over the silver plating bath which is known to contain the highest concentration of dissolved cyanide. Quantitative tests were therefore carried out by sampling the air approximately 1-1/2 feet above the rim of this bath and at a level somewhat below the normal breathing zone of a person working at the bath. Duplicate tests gave a concentration of less than five parts per million. This value is at the lower limit of sensitivity of the test and is well below the accepted limit of 20 parts per million for exposure during the whole work period. Tests for hydrogen cyanide in the storage cabinet for solid cyanide salts were completely negative.

Under the present conditions the level of hydrogen cyanide in the plating room is very low even without the operation of ventilating fans and should cause no concern until there is a change in operating conditions.

Test for Hydrogen Sulfide

This method depends on the blackening of lead acetate paper.

Reagents - Dissolve ten grams of lead acetate in 100 cc. of water and add 5 cc. of glacial acetic acid.

Test Papers - Immerse 2 by 4-inch strips of Whatman No. 1 filter paper in the solution for one minute. Allow to drain, suspend vertically and allow to dry in an atmosphere free from hydrogen sulfide at room temperature. When dry, cut 4.25 cm. circles from the center of the paper.

~~CONFIDENTIAL~~

UNCLASSIFIED

MLL-387

Test papers may be kept for ten days in an air tight container in which there is a drying agent.

Collection of Sample - Mount a test paper in the wall-type air sampler and draw air through at a rate of 10 cc. per sec. for a period of time in seconds as given in Table II such as to give a suitable stain to the paper. Compare the stain formed within ten minutes with those on the standard chart opposite page 113, in the book by J. B. Picklen.

Table II

SAMPLING TIME FOR WALL-TYPE SAMPLER, HYDROGEN SULFIDE

<u>Sampling Time Seconds</u>	<u>No. of Strokes</u>	<u>Volume of Air cc.</u>
13	1	125
25	2	250
38	3	375
63	5	625

Tests for Hydrogen Sulfide in the "TD" Building - Tests were made in the "TD" Building on August 19, 1949 and August 23, 1949 during operations involving the use of hydrogen sulfide when the concentration was expected to be the highest. These operations consist of bubbling hydrogen sulfide through a Pfaudler tank during a half-hour period and the subsequent filtration of the solution.

UNCLASSIFIED

~~RESTRICTED~~ UNCLASSIFIED

MLM-387

<u>Location</u>	<u>Concentration of Hydrogen Sulfide Parts per Million</u>	<u>Remarks</u>
Neutralization Tank	< 5	Test paper used dry.
Neutralization Tank	< 7	Test paper used moist.
At Holding Tank, Final Filter Room	20	Remainder of tests made with dry paper.
Between Final Filter Tanks	33	
Beside Vent Fan, Final Filter Room	50	

The tests on August 23, 1949 were made while bubbling hydrogen sulfide through the Pfaudler at the usual pressure of 5 p.s.i.

<u>Location</u>	<u>Concentration of Hydrogen Sulfide Parts per Million</u>	<u>Remarks</u>
At H ₂ S Cylinder next to Pfaudler	None Detectable	No odor, no change in color.
At Top of Pfaudler, Cover off	None Detectable	No odor.
Final Filter Room	< 7	Slight odor, trace of stain on paper.

The tests on August 23, 1949 indicate that treatment of the solution in the Pfaudler with hydrogen sulfide can be carried out without the escape of appreciable amounts of the gas into the room. The ventilating system was adequate to remove excess hydrogen sulfide from the Pfaudler at such a rate that none was detectable by smell or test paper in the area around or over the Pfaudler. Effort should be directed toward maintaining the conditions which prevailed during this test.

~~RESTRICTED~~

UNCLASSIFIED

 **UNCLASSIFIED**

11.-387

Under other conditions when the ventilating fans are not operating as effectively or when there are leaks in the connecting lines, the concentration of hydrogen sulfide can reach higher levels. The tests on August 19, 1949 show levels as high as 50 p.p.m.

 **UNCLASSIFIED**