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UNITED STATES ARMY
CHEMICAL CORPS BIOLOGICAL LABORATORIES
Fort Detrick, Maryland

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Colorimetric Method for Determining Ammonia in the Air with Phenol and Sodium Hypochlorite.

by M.I. Poletayev and N.A. Andreyeva

Translated from *Gigiena i Sanitariya*, 24: 73-74, 1959, by
SFC Eldon E. Ewing, Technical Library, Technical Information Division.

The detection of ammonia in the air of industrial and housing installations is chiefly made by the colorimetric method with Nessler's reagent. By its sensitivity this method completely fulfills the requirements that are necessary for sanitary control of the air medium. Therefore it has found wide use in the conduct of sanitary - chemical investigations. However, the chemical reaction on which the given method is based is nonspecific. Thus, with a content of aldehydes (formaldehyde, acrolein) and hydrogen sulfide in the air this method cannot be used.

In the conduct of sanitary-chemical investigations one must often contend with conditions where aldehydes and hydrogen sulfide are contained in the air simultaneously with ammonia. In view of this we attempted to develop a specific method that would enable us to detect slight amounts of ammonia with a simultaneous content of aldehydes and hydrogen sulfide in the air. It is commonly known that a blue stain is produced from the action of phenol and sodium hypochlorite on ammonium salts. The intensity of the staining depends on the concentration of ammonium salts in the solution.

By using different variants in the preparation of the sodium hypochlorite and also various absorbents for absorbing ammonia from the air, we were able, by using the given test, to detect 0.0003 milligram of ammonia in a five-milliliter sample, which is fully adequate for a sanitary evaluation of the air in industrial and housing installations.

Method of detection. The air samples for analysis are taken in two series connected absorbents of the Petri type, filled with a 0.002 n. solution of sulfuric acid with 10 milliliters in each absorbent. The rate of air flow was 60 liters/hour. The sample is transferred from the absorbents to colorimetric test tubes: 5 ml from the first absorbent into the first test tube and 5 ml from the second absorbent into the second test tube. Then 0.05 - 0.1 - 0.2 - 0.3 - 0.4 - 0.5 - 0.6 - 0.7 - 0.8 - 0.9 and 1.0 milliliter of a standard solution of ammonium sulfate, containing 0.01 milligram of ammonia per liter, is pipetted into a series of colorimetric test tubes, beginning with the second. A standard scale of solution from 0.0002 to 0.01 milligram of ammonia is received. The first test tube remains as a control: all reagents are inserted into it except the standard solution of ammonium sulfate.

The volume of liquid in all of the test tubes of the scale and in the samples equals 5 ml with a 0.02 n. solution of sulfuric acid. Then to each of the test tubes of the scale and to the samples add 1 ml of a 4-% solution of phenol (aqueous) and 0.5 ml of sodium hypochlorite, which is prepared by the following method. First, the active chlorine content in the calcium hypochlorite is determined. It must be in the range of 30 - 35-%. For this, dissolve 3.5 grams of the calcium hypochlorite in a measured liter flask, take 50 ml of the solution in a conical flask with a ground stopper, add 10 - 15 ml of a 10-% solution of potassium iodide and 10 ml of 30-% acetic acid, and titrate the iodine that has been precipitated with a 0.1 n. solution of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) to a weak yellow color and then slowly add several drops of starch drop by drop, until decolorization.

One milliliter of 0.1 n. solution of sodium thiosulfate corresponds to 0.003456 gram of chlorine. After the active chlorine content has been established, take (1) 100 grams of calcium hypochlorite, stir carefully for 15 minutes with 170 ml of water and add to the mixture, while constantly stirring, a solution of 70 grams of Na_2CO_3 in 170 ml of water. The mass will at first thicken and then liquify. The fluid is drawn off the sediment through a linen filter or through a porous glass filter. Before use, the resultant reagent is diluted with non-ammoniacal distilled water, 1:1. Within 10 minutes after the addition of the sodium hypochlorite to the scale and to the samples the intensity of the blue color in the samples are compared with the standard scale. The standard scale is stable for 24 hours. The sodium hypochlorite prepared by the indicated method is good for a period of 15 days.

The sensitivity of the method is 0.0005 milligram in a colorimetric volume. (2) Aldehydes (formaldehyde, acrolein) and hydrogen sulfide in concentrations up to 1 milligram in a sample do not interfere with the determination of the ammonia. The method is non-specific in the presence of amines (methyl amine, dimethylamine, etc.). For a comparative evaluation of the results that are received by the proposed method we made parallel analyses of water solutions containing ammonia by the two methods.

The results of the analyses are presented in the table.

The parallel analyses for ammonia that are presented in the table show a satisfactory convergence of results.

The difference between the individual determinations do not exceed 0.001-0.003 milligram in the majority of cases. The analyses that have been conducted by the proposed method for the detection of ammonia in the air under industrial conditions have given positive results.

Footnotes: (1) Yu. V. Koryakin and I.I. Angelov: Pure Chemical Reagents. M., Goskhimizdat, 1955, p 421.

(2) The sensitivity of the method requires checking and should be more precise (Editors).

Literature

Zhitkova, A.S.: Express methods for detecting harmful gases and fumes in the air of industrial installations, M. - L., 1946.

You, D.G.: Photometric chemical analysis (colorimetry and nephelometry). M., 1935, V. 1.

Kogan, I.B.: in the book: Methods of investigating labor hygiene in industry. M., 1953, 145 - 238.

Ammonia concentrations received in parallel analyses of water solutions containing Nessler's reagent or phenol and sodium hypochlorite.

Amount of ammonia detected with the Nessler reagent (in mg)	Amount of ammonia detected with the phenol and sodium hypochlorite (in mg)	The difference in the readings (in mg)
0.241	0.238	0.003
0.143	0.15	0.004
0.12	0.118	0.002
0.113	0.112	0.001
0.042	0.045	0.003
0.022	0.023	0.001
0.018	0.023	0.005
0.016	0.012	0.004
0.009	0.007	0.002
0.006	0.008	0.002