

## Department of Scientific and Industrial Research

### METHODS FOR THE DETECTION OF TOXIC GASES IN INDUSTRY

#### V. NITROUS FUMES\*

THE situations in which nitrous fumes may be encountered in dangerous concentrations include ammonium nitrate works, celluloid works, dyestuffs works, explosives works, nitric acid works, nitrocellulose paint, lacquer and leather cloth works, photographic film works and sulphuric acid works (chamber process). They are also encountered in electro-plating, engraving, metal cleaning and photo-gravure processes, and are formed during oxyacetylene welding, particularly when an oxyacetylene flame plays on cold steel in a confined space. They have caused fatalities during the heat treatment of metals in molten nitrates.

**POISONOUS EFFECTS.**—There may be, and generally are, no immediate effects, and therefore it is impossible to foretell the serious consequences that may result from the inhalation of these fumes. A workman, unaware that he has inhaled

\* Leaflet No. 5. H.M. Stationery Office, York House, Kingsway, London, W.C.2. March 14th, 1939. Price 3d. net.

the fumes, continues at work, often remaining well until after he has returned home. Some hours later he becomes restless with a dry cough and shortness of breath. These symptoms increase, accompanied by a frothy sputum, tinged with blood. If appropriate treatment is not applied, death follows from oedema (water-logging) of the lungs. The following table, based on various authorities (*e.g.* Henderson and Haggard, *Noxious Gases*, 1927; International Labour Office, etc.), shows the results following the inhalation of nitrous fumes in various concentrations:

Parts by vol. (approx.)	Mg. per litre (approx.)	Effects
1 in 100,000	0.03	Maximum concentration allowable for an exposure of several hours' duration.
1 in 10,000	0.3	Only sufficient to produce mild irritation of the throat and chest, but dangerous to inhale for only a few minutes.

At concentrations higher than 1 in 10,000 the fumes are frequently fatal if breathed for more than a few minutes. Hence any atmosphere in which nitrous fumes are noticeable by odour, irritating effects or colour should be regarded as dangerous.

**METHODS OF DETECTION.**—Starch and potassium iodide papers are fairly sensitive to low concentrations of nitrous fumes, but owing to their lack of specificity and to the difficulty of obtaining complete absorption of the gas by the paper, their use was not considered as a standard test.

Two tests, which depend on the diazo reaction, are specific for nitrous fumes and are very sensitive; they are: (i) the Griess-Ilosvay reaction; (ii) the Bismarck Brown test. In the former the gas is bubbled through a solution of  $\alpha$ -naphthylamine and sulphanilic acid in acetic acid, a rose-pink colour being produced; in the latter test the gas is bubbled through a solution of *m*-phenylenediamine hydrochloride in acetic acid, and an orange colour, due to the formation of Bismarck Brown, is produced. As the Griess-Ilosvay test is somewhat more sensitive than the Bismarck Brown test, it was adopted as the standard test. It is sufficiently sensitive to be capable of detecting nitrous fumes at a concentration of 1 part in 100,000. The test is made by drawing the atmosphere under examination through a tube containing the reagent by means of a hand-pump until a rose-pink colour of standard depth is reached. From the number of strokes of the pump required to produce the standard colour, the concentration of nitrous fumes present can be obtained by reference to a table given in the Leaflet.

**INSTRUCTIONS FOR CARRYING OUT THE TEST.**—Under Regulation 7 of the Chemical Works Regulations, 1922, the test shall be carried out only by a responsible person appointed by the occupier, and all necessary steps shall be taken for the protection of that person. When testing the concentration of nitrous fumes inside a tank prior to the entry of a workman, one test may be insufficient; if there is a chance of the concentration inside the tank rising, the atmosphere must be sampled at intervals throughout the period of work.

**Apparatus.**—The hand-pump used for sampling the atmosphere has a barrel of approximately 1.25 in. bore and a capacity of 126 ml. It is fitted with a nozzle opening for attaching rubber tubing, and, if desired, may be provided with a device for counting the number of strokes made. A convenient type of pump is described (*cf.* Leaflets Nos. 1 and 2, *ANALYST*, 1937, **62**, 607; 1938, **63**, 658).

The sample is drawn through the reagent contained in a bubbler consisting of a side-arm test-tube of approximately 0.75 in. internal diameter, and about 6 in. in length, fitted with a rubber bung through which passes a delivery tube (approximately 0.125 in. bore) reaching nearly to the bottom of the test-tube and terminating in a fine jet. It is convenient to calibrate the test-tubes to avoid measuring out the reagent each time a test is made. The bubbler is mounted on the barrel of the pump in such a way that the reagent is easily seen, and the tube

readily removable, for comparison with the standard and replacement by a fresh tube.

A convenient arrangement of the apparatus is described and illustrated in the Leaflet.

*Preparation of Reagent.*—(a) 0.5 g. of sulphanilic acid is dissolved, with slight warming, in 150 ml. of a solution of 70 ml. of glacial acetic acid in 500 ml. of water. (b) 0.1 g. of  $\alpha$ -naphthylamine is boiled for a few minutes, in 20 ml. of water, and the mixture is poured into 150 ml. of a solution of 70 ml. of glacial acetic acid in 500 ml. of water. (c) *Immediately before the test is made* 5 ml. of each of the solutions (a) and (b) are mixed in the bubbler.

The solution should remain colourless after mixing; if any pink colour is developed the solution must be discarded and a fresh portion of reagent measured out.

*Preparation of Standard Colour.*—(a) 0.1 g. of dimethylamino-azo-benzene (of reagent quality) is dissolved in acetone, and the solution is made up to 100 ml. with acetone (of reagent quality) in a graduated flask—Solution A. (b) Five ml. of Solution A are made up to 250 ml. with acetone—Solution B. This solution must be kept in a stoppered bottle in the dark. (c) When required, the standard colour is prepared as follows:—One ml. of Solution B is pipetted into a tube of diameter equal to that of that of the side-arm test-tubes used for the test, and made up to 10 ml. with dilute (approximately 7 per cent.) hydrochloric acid. The standard, when not in use, must be well corked or sealed and kept in the dark.

*Method.*—The strokes with the hand-pump are made very slowly (approximately 10 seconds per stroke), and the pumping is continued until the depth of colour developed is a little less than that of the standard colour (the number of strokes made being counted). The bubbler is then removed, and *after 2 minutes* (to allow of full development of the colour) it is compared with the standard tube, the two tubes being observed side by side by transversely transmitted daylight. If the colour given in the test is less than that of the standard, the bubbler is replaced and further strokes of the pump are made until exact equivalence is obtained after a standing period of 2 minutes.

The following table shows the number of strokes necessary for various ranges of concentration in order to obtain the standard depth of colour:

Concentration	Number of strokes
Above 1 in 8,000	Less than 1
1 in 8,000 to 1 in 13,000	1 to 2
1 in 13,000 to 1 in 20,000	2
1 in 20,000 to 1 in 27,000	3
1 in 27,000 to 1 in 35,000	4
1 in 35,000 to 1 in 45,000	5
1 in 45,000 to 1 in 55,000	6
1 in 55,000 to 1 in 65,000	7
1 in 65,000 to 1 in 75,000	8
1 in 75,000 to 1 in 85,000	9
1 in 85,000 to 1 in 100,000	10

Under certain conditions a cloudiness may be produced in the test solution, but this does not interfere with the comparison with the standard colour.

Directions are given for a procedure for sampling the air from a space that is not readily accessible or where there is a possibility of highly toxic concentrations of nitrous fumes being present, *e.g.* in acid tanks.

**FIRST AID.**—Full details of the treatment of cases of gassing are given in Factory Form No. 395 (H.M. Stationery Office, price 1d. net). As there is invariably a delay of some hours before serious symptoms may develop, strict confinement to bed for not less than two days is advisable.