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DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH

METHODS FOR THE DETECTION OF
TOXIC GASES IN INDUSTRY

LEAFLET No. 11

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DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH

METHODS FOR THE DETECTION OF
TOXIC GASES IN INDUSTRY

LEAFLET No. 11

ANILINE VAPOUR

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LONDON
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1939

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Foreword

Under Regulation 7 of the Chemical Works Regulations, 1922 (made under section 79 of the Factory and Workshop Act, 1901), before any person is allowed to enter, without wearing an approved breathing apparatus and life-belt, any vessel or place which it is thought may contain a dangerous gas or fume, it is necessary that the vessel or place be tested by a responsible person appointed by the occupier, and the appointed person must certify, in writing, that it is free from dangerous gas or fume.

The use of white mice has been suggested for the purpose of this test. Though this method is effective in the cases of carbon monoxide and hydrogen cyanide, experience has shown that it cannot always be relied upon for other important gases and vapours encountered in industry and that there is a need for simple and rapid chemical or other methods for determining low concentrations of dangerous gases, such as may occur in various circumstances in chemical works. The matter was discussed by the Association of British Chemical Manufacturers with the Home Office, and as a result arrangements were eventually made by the Department of Scientific and Industrial Research, at the request of the Home Office and with the financial and technical co-operation of the Association of British Chemical Manufacturers, for a series of tests to be developed by the Chemical Defence Research Department. Although this series of tests has been worked out mainly from the point of view of the chemical industry, the gases and vapours in question are encountered in many other industries to which the tests should also be useful. The tests will also be applicable for making the necessary regular tests on the atmosphere of workrooms, etc.

In most cases, chemical methods involving colour changes have been adopted as best suited for the purpose in view. Each test has been carefully standardized in the laboratory, and tested under practical conditions in actual works. While the information contained in the leaflets describing the tests is addressed primarily to responsible works officials, medical officers, chemists, and other persons in charge of chemical plant, the tests themselves have been made as simple and straightforward as possible in order that they

can be operated, given the necessary materials, by comparatively unskilled personnel. They can be elaborated or modified to a certain extent to suit particular conditions, provided that the fundamental conditions laid down for the tests are not altered in any way. In this connexion it must be remembered, however, that the primary object of the tests is not to obtain an extreme degree of accuracy, but to give a rapid indication of the relative safety of the atmosphere. A result which is close to the danger limit should always be regarded as indicating that conditions are dangerous.

The complete series of tests will cover the following gases and vapours. Leaflets for those marked with an asterisk are not yet published :—

- Aniline (No. 11).
- Arsine (No. 9).
- Benzene (No. 4).
- Carbon bisulphide (No. 6).
- *Carbon monoxide (No. 7).
- Chlorine (No. 10).
- Hydrogen cyanide (No. 2).
- Hydrogen sulphide (No. 1).
- Nitrous fumes (No. 5).
- *Organic halogen compounds.
- Phosgene (No. 8).
- Sulphur dioxide (No. 3).

It must be remembered that each of the above tests will only indicate the presence or absence of the specific gas, and in cases where any other gas is liable to occur, the relevant test for it must be applied before the atmosphere is adjudged as safe to breathe.

Further, none of the tests will indicate the danger which exists in cases where the oxygen content of a closed chamber is dangerously deficient.

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH,
16 Old Queen Street,
Westminster, London, S.W.1.

September 1939.

METHODS FOR THE DETECTION OF TOXIC GASES IN INDUSTRY

ANILINE VAPOUR

Occurrence

Aniline exists in small quantities in coal tar but is produced industrially almost exclusively by the reduction of nitro-benzene. It is used chiefly as a starting point for the preparation of dyestuffs and to a less degree in other chemical industries. Concentrations which may be dangerous may occur in the following :—

- Dyemaking and dye-using works
- Leather-dyeing works
- Leather-polish works
- Pharmaceutical works
- Rubber works
- Textile-dyeing works

This list is intended to be illustrative only, and not exhaustive.

Poisonous Effects

Aniline is a colourless oily liquid boiling at 184° C.

In spite of its relatively low vapour pressure at low temperatures it may cause poisoning by inhalation of the vapour.

The smell is aromatic and pleasant, and in no way suggests to the uninstructed the dangers resulting from its presence in the atmosphere.

The experience of the Factory Department of the Home Office has shown that most of the cases of acute aniline poisoning arise by absorption through the skin, from splashes either directly on to the skin or indirectly through the clothing. For this reason scrupulous cleanliness of the skin, and the clothing is essential for the prevention of aniline absorption.

The immediate toxic effect of aniline is on the blood, with the formation of methaemoglobin, causing cyanosis (characterised by a blue-grey discoloration chiefly of the lips, the ears, the cheeks, the tip of the nose and the nails). Depending upon the intensity of the intoxication, shortness of breath, rapid feeble pulse, nervous excitement, not unlike drunkenness and, in rare cases, blood in urine (haematuria) may later occur.

In persons exposed continuously to small doses of aniline the cyanosis may be little marked, but anæmia is present with loss of energy, digestive disturbances and headaches.

Convalescence after severe poisoning is slow, due to the subsequent anæmia.

According to Henderson and Haggard ("Noxious Gases," 1927) the effects of exposure to aniline vapour are as follows :—

<i>Concentration of Aniline Vapour in Air</i>		<i>Effects</i>
<i>Parts by volume (approximately)</i>	<i>mg./litre (approximately)</i>	
1 in 140,000— 1 in 40,000	0.03–0.1	Slight symptoms after several hours' exposure.
1 in 10,000— 1 in 6,000	0.4–0.7	Serious disturbance if inhaled for more than one hour.

According to Lehmann (quoted in "Occupation and Health," p. 123, I.L.O., 1930).—

0.3 mg. of aniline per litre (1 in 13,000 approximately) causes no perceptible effects in a 15 minute exposure to man or other animals, but if the exposure is 4 to 6 hours' duration, such an atmosphere is fatal to cats 12 hours later.

It appears undesirable to breathe 1 part in 200,000 (approximately 0.02 mg./litre) for several hours, particularly if the exposure is to be repeated on successive days.

The chemical test described below is capable of detecting concentrations of this order.

Methods of Detection

In order to test for the presence of aniline vapour in the atmosphere, the aniline must first be brought into solution, by drawing a sample of the air under test by means of a hand pump through a small bubbler containing dilute hydrochloric acid. Any aniline vapour present is thereby converted to the hydrochloride, to which any of the ordinary tests for aniline may then be applied.

Normally, however, the concentration of the vapour present in the atmosphere is so small that only very little hydrochloride is obtained without taking an inconveniently large sample of air; several of the ordinary tests are therefore insufficiently sensitive. For example, the best known test for aniline is by adding a few drops of a dilute solution of bleaching powder, which produces a purple coloration, rapidly changing to a dirty red; over the range of concentrations of aniline vapour likely to be encountered in the atmosphere, however, only the slightest trace of colour is obtainable.

A more sensitive test has therefore been developed from the bleaching powder test. If, after the addition of the bleaching powder, the solution is made alkaline with ammonia and a dilute aqueous solution of phenol added, a permanent deep blue colour is produced even with minute quantities of aniline. This test has been adopted as the standard test for aniline vapour in industry. It has been made quantitative by comparing the colours obtained at known concentrations with a series of standard colours prepared from a dye.* In this way a table has been drawn up showing the depth of colour obtained with up to 10 strokes of the standard hand pump over a range of concentrations from 1 part in 5,000 to 1 part in 100,000. The table is included in the detailed instructions for performing the test given below.

Instructions for Carrying out the Test for Aniline Vapour

The test shall be carried out in accordance with the following instructions :—

General

Although the test is simple and accurate, it shall only be carried out by a responsible person appointed by the occupier; this is definitely required by Regulation 7 of the Chemical Works Regulations, 1922, in the case of entry into vessels. All necessary steps shall be taken for the protection of the person carrying out the test.

In determining the concentration of aniline vapour in a workroom samples shall be taken at a point closely adjacent to the workers, as an average sample for the whole workroom may give misleading figures of the concentrations actually inhaled. Regular periodical tests will enable a rising concentration to be detected and ensure that an undesirable level is never attained.

In determining the concentration of aniline vapour in the atmosphere inside a tank prior to the entry of a workman, it may be insufficient to do only one test. When there is any chance of the concentration inside the tank rising, sampling shall be carried out at intervals throughout the period of work.

Apparatus

The atmosphere being tested shall be sampled by means of a hand exhausting pump with a barrel of approximately 1.25 in. bore and a capacity of 126 ml. The pump shall be fitted with a nozzle opening for attaching rubber tubing; it may also be provided, if desired, with a device for counting the number of strokes made.

The sample shall be 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)

* See footnote on page 8.

reaching nearly to the bottom of the test tube, and terminating in a fine jet.* It is convenient previously to calibrate the test tubes to avoid measuring out the reagent each time a test is made.

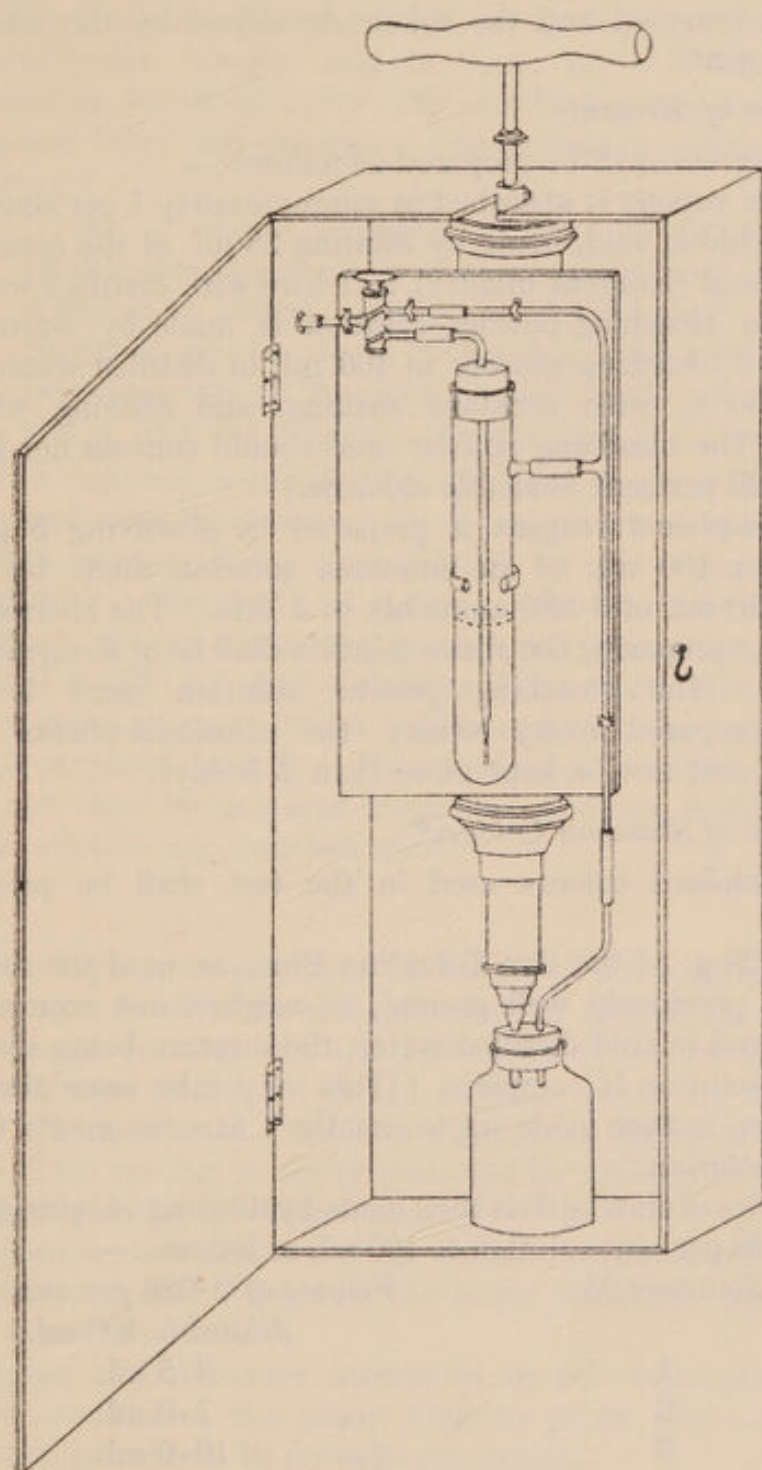
The bubbler shall be mounted on the barrel of the pump in such a way that the reagent is easily seen, and the tube readily removable for comparison with the standard and replacement by a fresh tube. In order to prevent the reagent being accidentally drawn into the pump by too rapid pumping, a trap shall be inserted between the pump and the bubbler.

A convenient arrangement† of the apparatus is shown in the diagram on page 5. The bubbler is fitted into a spring clip attached to a thin board fixed to the barrel of the pump; it can thus be readily withdrawn when required. The face of the board should be painted white or covered with a sheet of thin white porcelain or similar material. The trap employed consists of a small wide-necked bottle, carrying a rubber bung through one hole in which passes the nozzle of the pump. Through a second hole passes a glass tube (approximately 0.25 in. bore), terminating immediately below the bung, and connected by rubber tubing to one arm of a T-piece fastened to the board on the pump. The remaining arms of the T-piece are similarly connected to the side-arm of the bubbler and to one branch of a three-way tap, which is also fastened to the board. The remaining branches of the tap are connected as shown to the delivery tube of the bubbler and to the sampling tube. By suitable manipulation of the tap the bubbler can thus be bypassed, and air pumped directly from the sampling tube. This arrangement is convenient when tests are being carried out at a distance and it is necessary first to clear the pure air from the sampling tube. (See page 9.)

The whole apparatus may be mounted in a wooden case by means of two spring clips into which the pump fits, the top face of the case being slotted so that the handle of the pump remains outside the case. The pump can thus be operated without removing the apparatus from the case; if necessary, however, the whole apparatus is readily

* A convenient method of obtaining a jet of the correct size is to draw out the end of the delivery tube in a flame, and then to insert a wire of approximately the required diameter (18-25 S.W.G. is suitable) until it is stopped. The jet is then cut at this point.

† This arrangement is intended merely to suggest a convenient way of assembling simple apparatus which would be likely to be available in any laboratory. Modifications in non-essentials may, of course, be made to suit individual circumstances or preference, e.g. by altering the lay-out, using all-glass bubblers or using permanent connexions. Some users may also prefer to purchase complete sets of apparatus offered by laboratory outfitters, which may differ in lay-out from the arrangement suggested. Such alternative arrangements are quite permissible so long as they preserve the essential features of the apparatus described above, and may present practical advantages in manipulation.



withdrawn in one piece. The sampling tube passes into the case through a small hole in the side near the three-way tap.

It will be noted that no provision is made for mounting the standard colour alongside the bubbler because the bubbler has first to be removed and the colour developed by the addition of various reagents.

Preparation of Reagents

The reagents shall be prepared as follows :—

- (i) The vapour is absorbed in approximately 1 per cent hydrochloric acid, made by diluting 25 ml. of the concentrated acid (Reagent quality) to 1 litre with distilled water.
- (ii) The bleaching powder solution is made by warming 5 g. of bleaching powder in 100 ml. of distilled water to 50°–60° C. with constant shaking, and filtering while hot. (The bleaching powder used should contain not less than 25 per cent available chlorine.)
- (iii) The phenol reagent is prepared by dissolving 5 g. phenol in 100 ml. of an ammonia solution made by diluting 50 ml. of 0.880 ammonia to 1 litre. The chemicals used in preparing the above solution shall be of Reagent quality.

The bleaching powder solution must be freshly prepared every week; the ammonia-phenol solution must not be kept more than 2–3 days.

*Preparation of Standard Colours**

The standard colours used in the test shall be prepared as follows :—

0.28 g. of the dye Toluidine Blue, as used for microscope stain, previously well ground, is weighed out accurately and dissolved in cold distilled water, the mixture being shaken well until solution is complete. (This may take some time.) The solution is then made up to exactly 1 litre, to give a 0.028 per cent solution.

The series of standards is then made by diluting varying quantities of the 0.028 per cent solution to 100 ml. as follows :—

<i>Standard No.</i>		<i>Volume of 0.028 per cent solution diluted to 100 ml.</i>	
1	3.5 ml.
2	7.0 ml.
3	10.0 ml.
4	15.0 ml.
5	20.0 ml.

The standards should be well corked or sealed and kept in the dark when not in use. They should be freshly prepared every 4 weeks.

* See footnote on page 8.

It is possible that slight variations may occur in the intensity of the colour given by different batches of the Toluidine Blue stain. A new supply of the latter should therefore be tested before use as follows :—

The dyestuff is finely ground and thoroughly mixed, and a representative sample used to make up a 0.028 per cent solution as described above; the standard colour No. 3 is then prepared from this solution. The following solution is then made up :

30 g. copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) of Reagent quality are dissolved in distilled water and the volume made up to 100 ml. To this is added 1.2 ml. of a freshly prepared solution made by dissolving 0.3 g. of potassium permanganate of Reagent quality in 100 ml. of distilled water, and the solutions are thoroughly mixed.

The coloured solution thus obtained is then immediately compared with the standard No. 3 prepared from the dyestuffs, the two solutions being observed side by side in small tubes of equal bore. The depth of colour in the two tubes should be identical; if any difference is observed, the strength of the original 0.028 per cent Toluidine Blue stock solution is adjusted until an exact match is obtained with the new standard No. 3 prepared from it. The standard colours for use in the test can then be prepared from this modified 0.028 per cent stock solution as described above.

Method

Before carrying out any test, the valves of the hand pump shall be tested (with the bubbler disconnected) by closing the inlet with the finger, and drawing out the piston slowly to its full extent. On releasing the piston, it should rapidly return to its original position. The washer on the pump piston may shrink in the course of time. (This trouble is easily remedied by soaking the piston in warm liquid paraffin for a few hours.)

To prepare for a test, 10 ml. of the hydrochloric acid are measured into the side-arm test tube, and the latter connected up with the pump as described above.

To obtain a preliminary indication of the atmosphere to be tested, five strokes of the pump shall be made very slowly and steadily (approximately 10 seconds per stroke).

Five strokes is convenient for a preliminary test. The complete test takes about half an hour, but the bulk of the colour develops in the first 10 minutes. Concentrations up to 1 part in 15,000 can thus be roughly estimated in $\frac{1}{4}$ -hour by using 5 strokes. Further,

more accurate tests can then be made, if desired, using the most suitable number of strokes, according to the concentration indicated in the preliminary test, and allowing the full time for the development of the colour.

When the desired number of strokes has been made, the bubbler is detached from the pump and the contents transferred to an ordinary test tube. Two drops of the bleaching-powder solution are added, the mixture allowed to stand for 5 minutes, and then brought to the boil. 5 ml. of the phenol reagent are run in, and the mixture allowed to stand for 15 minutes, during which a blue colour develops if aniline is present. The liquid finally is transferred to a small Nessler tube and the depth of colour compared with that of the standard colours contained in similar tubes.* The concentration, corresponding to the depth of colour obtained and the number of strokes used, is then read off from the following table :—

<i>No. of Strokes</i>	1	3	5	10
<i>Concentration Parts by Volume</i>	<i>Standard No.</i>	<i>Standard No.</i>	<i>Standard No.</i>	<i>Standard No.</i>
1 in 5,000	3-4	—	—	—
1 in 10,000	2	4	—	—
1 in 15,000	—	3-4	5	—
1 in 20,000	1	3	4	—
1 in 30,000	—	2	3	5
1 in 50,000	—	—	2	3-4
1 in 100,000	—	—	1	2
1 in 200,000	—	—	—	1

When possible, the preliminary test should be completed before the atmosphere is entered by workmen. In cases where the test is being made in a room already occupied, if a colour deeper than Standard No. 5 develops within a few minutes after adding the reagents, immediate steps should be taken to evacuate the room, as a concentration harmful for even short exposures may be present.

* There are alternative methods of comparing the colours obtained, e.g. by means of a comparator using coloured glasses as standard, a photo-electric colorimeter, or an optical density meter. Provided that the standards employed have been properly calibrated and are used according to standard instructions, such methods can be quite satisfactory and may have advantages of permanence and accuracy of standards, and of simplicity of technique. They are likely to be particularly applicable in works where a large number of tests is carried out.

When a colour less than Standard No. 1 is obtained with 10 strokes of the pump, indicating a concentration of less than 1 part in 200,000, the number of strokes made may, if desired, be increased until this standard is reached, and the actual concentration present then calculated by proportion from the figures given for a smaller number of strokes.

Care should be taken that the tube and delivery tube are thoroughly cleaned before each test. The delivery tube should on no account touch the side of the test tube, but should be approximately central.

Where a test is required on air from a space which is not readily accessible, or where there is a possibility of a highly toxic concentration of aniline vapour being present, the atmosphere shall be sampled from a distance according to the following procedure :—

The apparatus shall be connected to the space to be tested by means of glass tubing (approximately 0·25 in. bore) as far as possible, with rubber connexions where necessary, all the tubing to be thoroughly dry. If a by-pass is incorporated in the apparatus as suggested on page 4, the three-way tap is turned so as to establish direct connexion between the pump and sampling tube, and the atmosphere then pumped through, approximately one stroke being made per 12 feet of extension tube. (If no by-pass is provided, the bubbler must be removed for this operation, and the sampling tube attached directly to the pump or trap.) When the sampling tube has in this way been filled with the atmosphere under test, the bubbler is restored to the circuit, and a test carried out in the usual way.

First Aid

In the case of an accident whereby liquid aniline is splashed on the skin or on the clothing, sufficiently to wet the skin, the clothing should be removed at once, and the skin very thoroughly washed. Splashed clothing should be most carefully washed before being worn again. If the patient is cyanosed (blue) oxygen should be administered before the doctor arrives. Cases showing any cyanosis must receive medical treatment without delay. Full details of the treatment of cases of gassing are given in Factory Form No. 395 (H.M. Stationery Office, price 1*d.* net).

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When a column less than standard No. 1 is obtained with 10
 percent of the sample, indicating a concentration of less than 1
 part in 100,000, the amount of sample may be doubled, or
 increased until the standard is reached, and the amount of
 excitation present then calculated by proportion from the
 amount given for a smaller number of atoms.

It may often be found that the tube and electrode tubes are
 thoroughly cleaned before each test. The glass tube should be
 immersed in the acid in the test tube, and should be kept
 in the acid for some time.

When a test is repeated in a test tube which is not readily
 accessible, it is better to use a test tube of a slightly larger size,
 and to replace the electrode in the test tube, and to replace the
 electrode in the test tube.

The electrode should be kept in the test tube for 10 to 15
 minutes, and the tube should be kept in the acid for 10 to 15
 minutes, with the tube immersed in the acid. All the
 tubes to be thoroughly dry. If a test is repeated in the
 test tube as suggested on page 1, the electrode should be turned
 so as to give the electrode a new position between the tube and
 the electrode, and the electrode should be turned through
 approximately one-half turn, and the tube should be turned
 tube. If no test is needed, the electrode should be removed
 for the operation, and the electrode tube should be turned
 the group or tube. When the electrode tube has in this way
 been turned with the electrode tube, the electrode
 removed to the right, and a test should be made in the next tube.

Final Aid

In the case of an electrode which is found to be defective
 the test tube should be turned, and the electrode should be
 turned so as to give the electrode a new position between the tube and
 the electrode, and the electrode should be turned through
 approximately one-half turn, and the tube should be turned
 tube. If no test is needed, the electrode should be removed
 for the operation, and the electrode tube should be turned
 the group or tube. When the electrode tube has in this way
 been turned with the electrode tube, the electrode
 removed to the right, and a test should be made in the next tube.

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