Notes

IDENTIFICATION OF DEMOLITION DUST

SHORTLY before the war we were asked to examine several new costumes which, it was alleged, had been spoiled by the dust produced in the demolition of an adjoining building penetrating into the room in which the costumes were hung. Samples of the demolition dust were submitted for comparison.

To extract the dust from the garments, a piece of finely woven white silk was stretched across the penultimate joint of the tube of an ordinary vacuum cleaner and over it was fitted the nozzle joint. By passing the nozzle over the surface of the different dresses a few mg of dust were extracted from each and could afterwards be brushed from the silk diaphragm without detaching any significant amount of silk fibres.

Microscopical examination of the demolition dust showed that it contained numerous light particles which could be separated by flotation on water. These were identified as wood by their cellular structure and analogous particles were separated from the dust extracted from the dresses. It was concluded that other yellow particles floating on the water consisted of paint, and similar particles were present in the dust from the dresses (see Figs. 1, 2 and 3). The wood was separated from the paint by treatment with

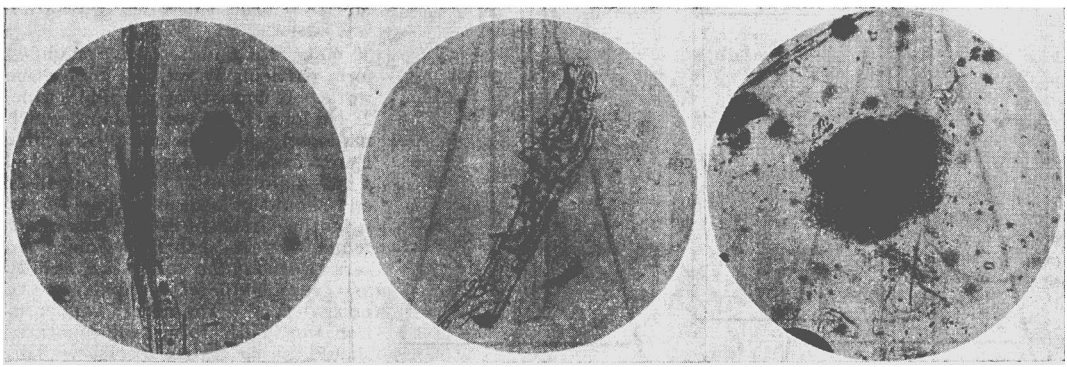

Fig. 1
Wood and paint in demolition dust
× 125

Fig. 2
Wood in dust from dress
× 125

Fig. 3
Paint and wood in dust from dress
× 125

50% alcohol, in which the paint sank. The heavy sediment which settled in the treatment with water had sp.gr. 2·28. It contained calcium sulphate and an aluminium salt and was probably Keene's cement (sp.gr. *ca* 2·3).The following table shows the comparative results obtained in typical tests:

	Demolition dust	Violet dress	Black coat
Micro-crystalline sediment from water:	$CaSO_4$.Al salt	$CaSO_4$.Al salt	$CaSO_4$.Al salt
Paint:	Lead, iron, trace of zinc	Lead, iron, no zinc found	Lead, iron, trace of zinc

Samples of ordinary street dust did not contain wood or paint and consisted mainly of siliceous material and organic débris.

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REFLUX APPARATUS FOR AUTOMATIC DISPERSION OF FROTH IN THE DETERMINATION OF FIBRE

THE estimation of fibre in cocoa and feeding stuffs by successive heatings under reflux with acid and alkali is invariably accompanied by some frothing and necessitates constant attendance. Frothing can be checked by applying a damp cloth to, or by blowing on the walls of the flask. The use of anti-froth agents does not comply with the official method of the Fertilisers and Feeding Stuffs Regulations (S.R. & O., 1932, No. 658).

Even with moderate frothing, particles of material tend to adhere to the walls of the flask, and are very difficult to swill down by rotation. Such particles as remain attached are removed from the hydrolysing action of the reagent, and hence are more or less incompletely digested, depending upon the stage of the process at which they are deposited.

Recently a series of fibre determinations was carried out on a cattle food which, when digested under reflux, frothed excessively in every test, and this could be suppressed only by frequent removal of the Rose burner. The resulting fibre figures were not concordant, ranging in 4 tests from 10·31 to 10·64; mean 10·47%. Under such conditions it is essential to suppress frothing.

We have found that froth can be dispersed automatically by maintaining the cooling of the condensate after it has passed from the condenser to the mixture under reflux. This is effected by allowing the condensate to flow down a secondary water-cooled condenser supported inside the reflux flask, thus effecting a cooling at the surface of the liquid.

The anti-froth device (Fig. 1) can be used in the form of an adaptor by permanently connecting the secondary condenser in series with the reflux condenser. When required for fibre determinations the delivery end of the latter condenser is merely inserted into the socket at B, and lowered until contact is made with the feed-bridge C. The dimensions assigned to the secondary condenser are suitable for a litre Pyrex flask. Although the secondary condenser can be considerably elaborated to give a more efficient cooling of the condensate, it is shown here in a simple form.

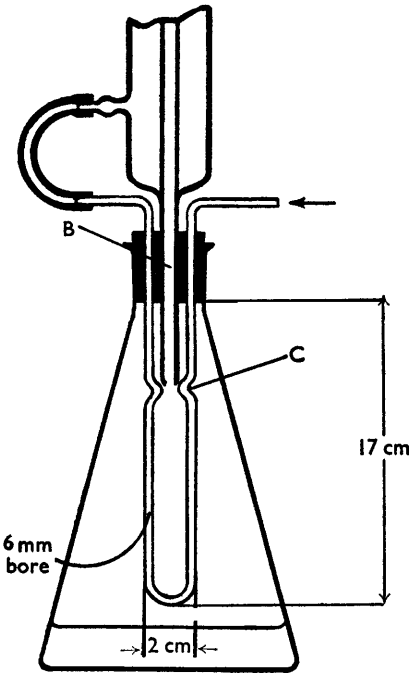

Fig. 1

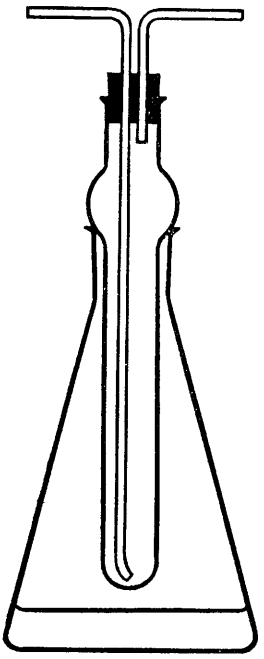

Fig. 2

A parallel set of fibre determinations on the same sample was made with the aid of this device, and the results were much more consistent than before, ranging in 4 tests from 10·24 to 10·28; mean 10·27%. The mean figure obtained was a little lower than in the previous test, suggesting more complete digestion. Moreover, the subsequent filtration was easier.

Fig. 2 is an alternative modified form particularly suitable for routine work. It consists of a boiling tube, 24 cm long × 2·5 cm in diam., which fits loosely into the neck of a litre Pyrex flask, and is supported by the 4-cm diam. bulb. The condenser end should be approximately 1·5 cm from the surface of the mixture under reflux. Units can be ganged in series without the congestion usually associated with reflux condensers of normal type. The rate of condensation for both forms of apparatus should be *ca.* 1 drop per sec. This corresponds to gentle boiling. In most instances frothing is completely eliminated.

It is important when transferring the material to the reflux flask to reserve an adequate portion of the reagent for rinsing down adhering particles from the walls of the flask. Since particles can be deposited by external agitation as well as by frothing, the normal procedure of rotating the flask is not only unnecessary but undesirable.

We intend to apply a similar technique to other problems involving frothing.

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THE DETECTION OF INDOLE BY MEANS OF XANTHYDROL

During a study of the effect of interfering substances on the pptn. of urea with xanthydrol it was observed that indole forms a violet pigment with xanthydrol in acid solns. As this colour reaction was not given by any other familiar biological compound examined, it should be of value in the detection and estimation of indole.

Method—Mix 0·5–1 ml of the test soln. with 2–5 ml of glacial acetic acid. Add a small quantity (5 mg) of xanthydrol and boil. In presence of indole in concns. as low as 1 : 50,000 in the final mixture a bright violet colour develops in *ca.* 1 min. and deepens as the mixture is shaken with air. The reaction also proceeds, but much more slowly, at room temp. The lower limit of the test is *ca.* 1 : 10^6, and hence it approximates in delicacy to the indole-aromatic aldehyde reactions studied by Deniges.[1] The pigment is stable in acid solns. and survives at least 6 months' exposure to light and air, in absence of direct sunlight. As the unchanged xanthydrol is pptd. by addition of water, it is necessary to use acetic acid or alcohol as diluents for colorimetric work.

Selectivity—The test appears to require the presence of an unsubstituted H in the β-, or 3-, position in the indole nucleus. The reaction is not given by scatole, β-indole propionic acid, tryptophan or indoxyl, or by any of the common amino acids, proteins, purines, amides, amines, ureides, guanidines or water-sol. vitamins. The test is also negative with normal urine, blood plasma, milk, saliva, gastric juice, bile, pancreatic juice, and cerebro-spinal fluid, although it will reveal traces of indole that have been added to these liquids. The presence of urea in urine does not seriously interfere with the test, and the characteristic colour can be seen even before the ppt. of dixanthyl urea has separated out. Among non-biological compounds, free pyrrole gives a similar reaction, while barbituric acid, but not the substituted barbiturates, yields a deep purple colour (Kidd[2]).

Reagents—The xanthydrol may be applied as the solid or as a 5–10% soln. in aldehyde-free methyl or ethyl alcohol. Such solns. are active for at least a year, although xanthydrol is unstable, and even in

solid form slowly reverts to a mixture of xanthene and xanthone (Kny-Jones and Ward)[3], neither of which gives a colour with indole. Acids stronger than acetic, if used as condensing agents, have the disadvantage that they form a yellow oxonium salt with the xanthydrol. Even the soln. in acetic acid acquires a faint yellow on boiling, but this disappears when the soln. cools. With conc. hydrochloric acid colour formation occurs rapidly in the cold and, if the mixture be boiled, the test loses some of its selectivity, and both free tryptophan and tryptophan-containing proteins eventually react, presumably owing to indole formation by decomposition. Acetic acid contaminated with glyoxylic acid will give a pink colour when boiled with indole; this effect, however, is suppressed in presence of excess of xanthydrol.

Mechanism—The properties of the pigment and the circumstances of its preparation suggest that it is a compound of the indolidene-methane type investigated by Burr and Gortner,[4] formed by oxidation of the 3-xanthyl-indole precursor (Illari)[5]. Fosse[6] has obtained a colourless, crystalline dixanthyl-indole, but does not refer to the formation of the violet pigment, although he observes that the dixanthyl derivative decomposes at 205°–214° C., "pour donner un liquide rouge fonce." Fosse does not appear to have published the exact details for the preparation of dixanthyl-indole. The compound is readily obtained when a conc. soln. of indole in glacial acetic acid is boiled with excess of xanthydrol. Under these conditions the acid is buffered to such an extent that the violet pigment does not form, which may be the reason why Fosse has not referred to it. The pigment can be isolated as an acetate (m.p. 170–172° C.) from the interaction between equimolecular proportions of indole (117 mg) and xanthydrol (198 mg) in 90% acetic acid (100 ml). The mixture is gently boiled for 10 min., left overnight, and treated with 30–40 ml of aldehyde-free ether, which ppts. any dixanthyl-indole that may have formed. Excess of ether bleaches the pigment, and the pigment-base is pptd. After filtration from any xanthyl-indole present, the mixture is concentrated *in vacuo* until the pigment separates as a crust on the side of the container. It is extracted with 50–70% acetic acid, and recrystallised.

SUMMARY—Dil. solns. of indole yield a stable purple pigment on being boiled with xanthydrol and excess of acetic acid. Among biological compounds, the reaction appears to be selective for indole.

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