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New Food and Drugs Legislation and Proposals for Regulations, etc. during 1965

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Only six Regulations which are of interest to Public Analysts were made during the year 1965. During the same period, however, a formidable list of Proposals for Regulations was circulated by the Government to interested parties, for comment. The regulations were:—

The Eggs (Marking and Storage) Regulations, 1965.
The Dried Milk Regulations, 1965.
The Milk (Special Designation) (Amendment) Regulations, 1965.
The Milk (Great Britain) Order, 1965, as amended.
The Therapeutic Substances (Supply of Substances for Analysis) Amendment Regulations, 1965.
The Cheese Regulations, 1965.

New legislation which is suggested included the following:—

Imported Food Regulations.
Regulations on Sausages and other Meat Products.
Revised Proposals for Regulations on Canned Meat.
Revised Proposals for Regulations on Meat Pies.
Revised Regulations on Colouring Matter in Food.
Regulations Amending the Mineral Hydrocarbons in Food Regulations, 1964.
Revised Regulations on Antioxidants in Food.
Replacing certain parts of the Labelling of Food Order, 1953.
Regulations on Butter.
Regulations on Margarine.
Regulations on Salad Cream and Mayonnaise.
Regulations on Coffee, Coffee Mixtures and Coffee Extracts.
Regulations on Ice Cream.

The Eggs (Marking and Storage) Regulations, 1965, replace the Agricultural Produce (Grading and Marking) (Eggs) Regulations, 1936, as amended. The new regulations require preserved eggs, cold stored and chemically stored eggs to be marked in a prescribed manner in letters of not less than one-sixteenth of an inch in height enclosed in a circle of not less than half an inch in diameter. The marking prescribed is: "PRESERVED" for preserved eggs; "SEALED" for eggs coated with mineral oil; "CHILLED" or "COLD STORED" for eggs from cold storage, and "STERILISED" in the case of chemically stored eggs. The previous requirements relating to grade designations and marking have been revoked without replacement.

The Dried Milk Regulations, 1965, came into operation on the 11th March, 1965. They re-enacted with amendments The Public Health (Dried Milk) Regulations, 1923 to 1948 and also revoked the Dried Milk Regulations, 1964, before the latter came into operation. The 1965 Regulations are very similar to the 1964 Regulations and provide for the following six descriptions of dried milk:—dried full cream milk (with not less than 26 per cent. of milk fat); dried three-quarter cream milk (with milk fat less than 26 and more than 17 per cent.); dried half-cream milk (with milk fat not more than 17 and not less than 14 per cent. of milk fat); dried quarter cream milk (with less than 14 and not less than 8 per cent. of milk fat); dried partly skimmed milk (with less than 8 and not less than 1·5 per cent. of milk fat); and dried skimmed milk (or dried low-fat skimmed milk), with less than 1·5 per cent. milk fat. All descriptions of dried milk are required to contain not more than 5·0 per cent. of moisture. Precise labelling requirements for all descriptions of dried milk are included in the Regulations.

The principal difference from the labelling requirements of the 1964 Regulations is that the new regulations make it obligatory for the containers of dried skimmed milk bearing the description "Dried low-fat skimmed milk" to carry a declaration of the maximum milk fat content while containers bearing the alternative description of "Dried Skimmed Milk", are merely permitted to display this declaration.

The Milk (Special Designation) (Amendment) Regulations, 1965, came into operation on the 1st October, 1965, and amend the Milk (Special Designation) Regulations, 1963, by adding the special designation "Ultra Heat Treated" to the existing special designations of "Pasteurised", "Sterilised" and "Untreated". "Ultra Heat Treated" milk is required to be retained at a temperature of not less than 270° F for not less than one second, and then immediately placed into the sterile containers in which it is to be supplied to the consumer. The cap of the container (or, in the absence of a cap, the container itself), is required to be marked either "Ultra Heat Treated Milk" or "U.H.T. Milk". A sample taken at any time before delivery to the consumer is required to satisfy the Colony Count Test prescribed in Part V of the 2nd Schedule to the Regulations. The test ensures that the milk is practically sterile and that no bacterial contamination has occurred after heat treatment. The tests at present used for

sterilised milk and pasteurised milk are unsuitable for U.H.T. milk in that if the last named is heated sufficiently to give a negative turbidity test, the flavour is adversely affected. It has also been found that reactivation of phosphatase may occur within a few days of treatment thereby rendering the Phosphatase Test unsuitable for control purposes.

The Milk (Great Britain) Order, 1965, came into operation on the 1st August, 1965, and it replaces the similar order made in 1962 and its amendments. It is enforced by the Ministry of Agriculture, Fisheries and Food and fixes maximum prices for Channel Islands and South Devon Milk and for milk of seven other special designations and types. It is principally of interest to Public Analysts in that paragraph 2 of the Order contains definitions of Channel Islands milk and South Devon milk; the minimum standard of 4 per cent. of milk fat for these milks is, however, prescribed by the Milk and Dairies (Channel Islands and South Devon Milk) Regulations, 1956.

The Therapeutic Substances (Supply of Substances for Analysis) Amendment Regulations, 1965, further relaxes the control exercised by Section 9 (1) of the Therapeutic Substances Act, 1956 on the sale of supply of Penicillin and certain other therapeutic substances by permitting their supply to persons engaged in testing the substances in the course of manufacture. The original exemptions under the Therapeutic Substances (Supply of Substances for Analysis) Regulations, 1958, only applied to Public Analysts, Agricultural Analysts, Sampling Officers, Inspectors appointed by the Pharmaceutical Society of Great Britain, and certain other persons.

The Cheese Regulations, 1965, were made on the 31st December, 1965, and come into operation on the 1st February, 1967. They are the first statutory regulations made in this country with regard to the composition and labelling of cheese and they follow the Food Standards Committee Report on Hard, Soft and Cream Cheeses published in 1962 and the Proposals and Revised Proposals for Cheese Regulations circulated for comment in 1963. The regulations apply to the following types of cheese: hard cheese, soft cheese (including cream cheese), whey cheese, processed cheese, cheese spread and compound products containing not less than 10 per cent. of cheese. Definitions of all the above types are included in Regulation 2. The regulations do not apply to cheese intended for use for manufacturing purposes. Cheese spread, although it is now the subject of a statutory standard, is still required to bear a list of ingredients. Hard cheese sold under the name of one or other of the 12 varieties specified in Schedule I must contain at least 48 per cent. of milk fat in the dry matter and not more than 39 to 48 per cent. of moisture, depending on the variety. Any other hard cheese must be described as either "full fat hard cheese", "medium fat hard cheese" or "skimmed milk hard cheese" and conform to standards given in Regulation 5 or bear a declaration of the minimum percentage of milk fat content or the minimum percentage of milk fat content in the dry matter and the maximum percentage water content. Descriptions and standards for soft cheese are given in Regulation 6 which also

includes a standard for "Cream Cheese" of not less than 45 per cent. milk fat and for "double cream cheese" of not less than 65 per cent. milk fat. Standards and descriptions of three qualities of whey cheese are given in Regulation 7. Processed cheese is required to conform to similar standards to hard cheese except that processed Cheddar cheese shall contain not less than 48 per cent. milk fat in the dry matter and not more than 43 per cent. water. "Cheese Spread" may also be described as "Cheese Food" and shall contain not less than 20 per cent. of milk fat and not more than 60 per cent. of water. Permitted ingredients other than those included in the definition of "cheese" are given in Regulation 12, which also includes references to sage cheese. The Statutory descriptions required to be marked on labels or tickets apply to cheese in containers and to cheese otherwise than in a container when it is sold by retail. Details with regard to labelling and advertisement are given in Part III and Schedule 2 of the Regulations.

Turning now to Proposals for Regulations circulated for comment during the year, the first related to Imported Food Regulations. These were intended to replace the Public Health (Imported Food) Regulations, 1937 to 1948, the Food and Drugs (Whalemeat) Regulations, 1949, and the Food and Drugs (Whalemeat) (Amendment) Regulations, 1950. The proposals would prohibit the importation of food intended for, but unfit for human consumption and would require "Official Certificates" from the export country in relation to certain carcases and parts of carcases and certain meat products on importation into this country. It would be possible for food to be held at a port for a stated period, not exceeding 6 days, to enable examination by any special procedure to be completed. An authorised officer of a port health authority may procure a sample under Section 91 of the Food and Drugs Act for analysis by a Public Analyst and Section 92 of the Act would apply as if the sample had been procured within the area of the Food and Drugs Authority which appointed the Public Analyst to whom it was submitted.

The circulation of Proposals for Regulations with regard to Sausages and other Meat Products came as a complete surprise, as previously the Government had refused to fix standards because of the technical difficulty of determining the proportions of the various types of meat in a sausage containing mixed meats. The Proposals have surmounted this difficulty by requiring sausages which bear the name of a single variety of meat to contain meat consisting entirely of that variety. Sausages will in general be required to contain 50 per cent. of meat, but certain "sausages", including pork, liver, luncheon, polony, black puddings and canned frankfurters, must contain not less than 65 per cent. of meat. Frankfurters (not canned) and similar sausages must contain not less than 80 per cent of meat. There is a difficulty in respect of black puddings in that the definition of meat in the proposals does not include blood; black puddings are made with blood and fat and not flesh yet

the proposals require them to have a meat content of 65 per cent. Standards are also proposed for the meat content of meat with jelly (80 per cent.); meat with cereal (80 per cent.); meat loaf (65 per cent.); meat loaf with stuffing (50 per cent.), and for any other meat product, 35 per cent. These products would thus be brought into line with similar canned products covered by the proposals described in the next paragraph.

The Revised Proposals for Regulations on Canned Meat were circulated for comment in May 1965 and referred to 13 categories of canned meat products as compared with only five or six categories in the original proposals made in March, 1964. In addition to those mentioned above the following categories would be included: corned meat (to consist wholly of corned meat); canned meat (95 per cent. meat); chopped or cured meat (90 per cent.), and meat with gravy (70 per cent.). Meat with gravy is an important category and in the original proposals, a minimum of 80 per cent. of meat was suggested. The Food Standards Committee report in 1962 suggested 75 per cent. and this last figure was upheld by an Appeal in the case of *A. J. Mills and Co. Ltd.* v. *Williams* in 1964, in respect of a sample of canned steak in gravy. It is, therefore, unexpected to find a standard of only 70 per cent. of meat in the Revised Proposals.

The Revised Proposals for Regulations on Meat Pies were also circulated for comment in May 1965, and followed the original proposals of March 1964. The standard proposed for the meat content of meat pies is 25 per cent., for uncooked meat pies and Scottish pies, 20 per cent., and for Meat and Vegetable pies, $12\frac{1}{2}$ per cent. The proposals also state that the standard shall be deemed to be complied with if a pie of not more than 3 ounces contains not less than $\frac{5}{8}$ths of an ounce of meat, a pie of not more than 4 ounces contains not less than $\frac{7}{8}$ths of an ounce of meat and a pie of not more than $5\frac{1}{2}$ ounces contains not less than 1 ounce of meat. This last *proviso* corresponds to only 18·2 per cent. of meat, which seems unnecessarily lenient in a pie weighing as much as $5\frac{1}{2}$ ounces. Provision would be made for fat in the pastry in excess of a ratio of fat to carbohydrate of 60 : 100 to be included as part of the meat content and egg up to a maximum of 10 per cent. by weight of the meat content would also be regarded as meat. No special provisions are made in the Revised Proposals with regard to sampling, although the original proposals suggested that a formal sample should consist of at least three pies and the Food Standards Committee Report on Meat Pies in 1963 stated that some special provision with regard to sampling would have to be laid down. Special provisions are necessary for unwrapped pies sold singly, because it is not possible to divide a pie into three exactly equal and representative portions. In regard to prepacked pies, section 97 of the Food and Drugs Act could be applied and each part of a formal sample could consist of one or more whole, wrapped pies. It does not seem feasible, however, to make regulations which could only be successfully enforced in the case of prepacked pies.

The Proposals for Revised Regulations on Colouring Matter in Food

follow, in general, the recommendations of the Food Standards Committee report published in 1964. Six colours permitted by the 1957 Regulations would be deleted and one colour (Black 7984) would be added. The number of permitted coal-tar colours under the Proposals would, therefore, be 25 as against 30 under the 1957 Regulations. The six coal-tar colours to be withdrawn are:—Ponceau 3R, Ponceau SX, Naphthol Yellow S, Blue VRS, Yellow RFS and Yellow RY. There would, thereby, be no permitted "blue".

The Mineral Hydrocarbons in Food Regulations 1964 were to come fully into operation on the 15th February, 1965. The need for proposals to amend these very recent regulations is due principally to the fact that the Government had received representations with regard to the difficulty of making a microcrystalline wax which would comply with the specification given in Part 3 of the 1st Schedule to the Regulations. Furthermore, it was said that the limit test for polycyclic hydrocarbons given in Part II of the Schedule was possibly dangerous to carry out and was not sufficiently strict to prevent the use of impure wax. The proposals, therefore, replace the words "Paraffin or Microcrystalline Wax" in the regulations by the words "Solid Mineral Hydrocarbon". The specifications of paraffin and Microcrystalline Wax would be replaced by completely new specifications for solid mineral hydrocarbons (mineral waxes) and the whole of Part II of the 1st Schedule would be replaced by a new and more stringent test for the presence of polycyclic hydrocarbons in solid mineral hydrocarbons. This last test has been given in very great detail, including detailed instructions for the preparation of the reagents used, as it was felt that any departure from the prescribed conditions could lead to erroneous results. One kilogramme of the solid mineral hydrocarbon is required for the test as against 100 grammes in the 1964 Regulations. A larger representative sample has, therefore, to be taken and this will only be possible on manufacturing premises where it is being used. It would also not be possible to carry out the test on solid mineral hydrocarbons after their addition to chewing compounds.

The Proposals for Revised Regulations on Antioxidants in Food were circulated for comment in September, 1965. They follow, in general, the recommendations of the Food Standards Committee Report, 1963, and of the supplementary report published early in 1965. The proposals would prohibit the sale of foods, intended for infants, which contained any antioxidant. Butylated hydroxytoluene (B.H.T.) would only be permitted in specified foods at half the present amounts pending a further report from the Pharmacology Sub-Committee of the Food Additives and Contaminants Committee with regard to the possible toxicity of this compound. Partial glycerol esters would be permitted to contain antioxidants and the presence of up to 3 parts per million of ethoxyquin would be permitted on apples and pears. There is also a tentative inclusion of diphenylamine as an antioxidant for use on apples and pears but this would depend on the advice of the Food Additives and Contaminants Committee who are at present considering the matter. Prohibition

of the use of both diphenylamine and of butylated hydroxytoluene was advised by the Food Standards Committee in their 1963 Report. It is therefore surprising that the former should have been tentatively included and the latter included at half the present permitted amounts before the safety of their use could be definitely established.

The Proposals for Regulations on Labelling of Food were circulated for comment in September, 1965, and they are intended to replace those parts of the Labelling of Food Order, 1953 which affect specific aspects of labelling. The parts of the 1953 Order which are concerned with claims are to be the subject of another report by the Food Standards Committee after which further proposals may be considered. The proposals on labelling follow in general the recommendations of the Food Standards Committee Report on labelling published in 1964. The list of exemptions in the Labelling of Food Order, 1953, as amended, has been curtailed; it would no longer be possible to use a registered trade mark instead of the name and address of the packer; voluntary lists of ingredients on exempted foods would, in future, be required to be complete lists conforming fully with the regulations; certain prepacked foods which are otherwise exempt (and many foods sold non-prepacked), must be labelled appropriately if they contain preservative, antioxidant, natural colour, artificial colour or artificial sweetener; vending machines would be required to be clearly labelled with the appropriate designations of the foods they contain; "tenderised" meat would be required to be labelled as such, and fish, meat and edible offal, certain fruits and potatoes sold by retail would be required to be marked with the variety or type and, where appropriate, the joint (of meat). In certain respects, however, the proposals do not follow the recommendations of the Food Standards Committee Report. For example, the report recommended that the size of the print of wording on labels should be directly related to the size of the surface area of the container but the Third Schedule to the Proposals relates the size of all print on a label to that of the word with the largest print irrespective of the size of the container. The report also recommended that declarations of alcoholic strength in terms of "Proof Spirit" should be discontinued and replaced by a declaration in terms of "percentage alcohol content by volume". The proposals have, however, retained Proof Spirit but would allow an alternative declaration in terms of per cent. alcohol by volume for certain liquors; this could lead to confusion, in that numerical statement of the same strength of alcohol by the two systems is quite different. Finally, the definition of "pre-packed" in the proposals does not include "made up in advance in a wholly transparent wrapper". This would appear to exempt all foods packed in this way from the general requirements relating to prepacked foods and is contrary to the view of the Food Standards Committee expressed in para. 171 of their Report.

The Proposals for Regulations on Butter are intended to replace the Food Standards (Butter and Margarine) Regulations, 1955, in so far as the latter concern butter. The proposals would require butter to have a fat content of

not less than 80 per cent. by weight, a milk solids-not-fat content of less than 2 per cent. by weight and a moisture content of not more than 16 per cent. by weight. It would also be made an offence to sell or advertise unsalted butter unless it is clearly labelled as such.

The Proposals for Regulations on Margarine would require margarine to contain not less than 80 per cent. by weight of fat (of which not more than 1/10th should be fat derived from milk) and not more than 16 per cent. water. Margarine sold by retail would also be required to contain, per ounce, not less than 760 i.u. and not more than 940 i.u. of Vitamin A present as such or as its esters and not less than 80 i.u. and not more than 100 i.u. of Vitamin D. The limits given for Vitamin A are identical with those contained in the Food Standards (Margarine) Order, 1954, which permitted a proportion of the carotenes present to be included as Vitamin A. The proposals do not refer to carotenes and do not include a statutory method for determination of Vitamin A. Manufacturers may have to increase the actual Vitamin A content and disregard any carotenes present. The labelling of margarine will be controlled both by the Proposals on Labelling of Food and the Proposals on Margarine. The latter require that whenever any brand name is used on a label the word "Margarine" must be in letters not less than 9/16ths of the area of the letters of any other word on the label, but in an advertisement the word "margarine" must be in type substantially as prominent as the brand name. The Proposals for Margarine also require that where the words "butter", "cream", or "milk" appear on labels they must not be in letters larger than those used for the word "margarine". Claims with regard to the presence of cream or milk will only be permitted if they conform to the precise wording stated in the proposals and claims that margarine contains butter would have to include a statement of the minimum percentage of butter present.

The Proposals for Regulations on Salad Cream and Mayonnaise include the same standards for edible vegetable oil and egg yolk solids content as the Food Standards (Salad Cream and Mayonnaise) Order, 1945. In addition, however, the proposals include a definition of "Salad Cream" but they do not specify the statutory disclaimer which has, under the present order, to be printed on the label when a product is not claimed to be a salad cream and does not comply with the statutory standard.

The Proposals for Regulations for Coffee, Coffee Mixtures and Coffee Extracts lay down tentative standards for ten types of coffee extracts and mixtures, and include, with additions, those at present in the Food Standards (Liquid Coffee Essences) Order, 1945 and in the Food Standards (Coffee Mixtures) Order, 1952. In addition to the standards at present in force the proposals would require (1) dried extract of coffee to comprise at least 95 per cent. of soluble solids of coffee, with no matter not derived from coffee; (2) dried extract of coffee and chicory to have a content of caffeine derived from coffee of not less than 1 per cent.; (3) dried extract of coffee and fig to have a content of caffeine derived from coffee of not less than 2·7 per cent.; (4) liquid extract of

coffee and fig to contain at least 0·4 per cent. *w/v* of caffeine derived from coffee; (5) decaffeinated coffee not to contain more than 0·1 per cent. of caffeine and (6) any decaffeinated coffee extract, essence, or mixture to have a caffeine content of not more than 1 per cent. by weight of the coffee solids.

The Proposed Regulations on Ice-Cream were circulated for comment in November, 1965. They reproduced, unchanged, the standards for Ice-Cream, Dairy Ice-Cream, Milk Ice and "Parev" (Kosher) Ice, contained in the Food Standards (Ice Cream) Regulations, 1959 but amended the labelling requirements for ice-cream contained in the Labelling of Food (Amendment) Regulations, 1959. A definition of ice-cream is included in the proposals specifically excluding water ices and iced lollies. The present requirement that prepacked ice-cream containing non-milk fat must be labelled either "contains non-milk fat" or, where appropriate, "contains vegetable fat" would be extended to apply to all ice-cream whether pre-packed or not. The Second Schedule to the Proposals stipulated the requirements which would apply to labels and tickets used with ice-cream.

In addition to the above proposals for regulations, two reports by the Food Standards Committee were published during 1965, one on Flavouring Agents and the other on Fish and Meat Pastes. There are in this country no statutory regulations with regard to the use of flavouring agents in food. In their report on flavouring agents the Food Standards Committee have considered a very large number of both natural and synthetic flavourings submitted by the food industry and have also considered the control on these substances exercised in the U.S.A., Canada and Germany. In view of the very large number of flavouring agents and the absence of adequate toxicological information, the Committee recommends that, as a first stage, there should be published a list of flavourings suspected to be harmful but that eventually regulations should be based on a list of permitted flavouring agents. The list of flavouring agents recommended to be prohibited is as follows:—

Coumarin	Dihydrosafrole	Dulcamara	Birch Tar Oil
Tonka bean	*iso*Safrole	Pennyroyal Oil	Cade Oil
Safrole	Agaric Acid	Oil of Tansy	Volatile Bitter Almond Oil
Sassafras Oil	Nitrobenzene	Rue Oil	Male Fern

The Report on Fish and Meat Pastes follows, in general, the recommendations that were made in regard to the composition and labelling of these products in the Food Standards Committee Report on Food Labelling published in 1964. It is recommended that the meat content of meat paste should be increased to the figure for fish paste, *viz.*, 70 per cent. Potted Meat or Fish should contain not less than 95 per cent. of the named meat or fish, and no added binder (except dressed crab which may contain added cereal). Products described as being "with butter" would have to contain not less than 6 per cent. butter fat, and limits are recommended for the fat contents of various types of meat and fish products which may be included as meat. Another recommendation is that the word "spread" should be confined to products containing at

least 70 per cent. of the named variety of meat or fish and the word "paste" used where there is at least 70 per cent. of meat or fish but less than that amount of the named meat or fish. Furthermore, a paste could be described simply as "meat" or "fish" paste without naming the variety. The arbitrary distinction suggested between "spread" and "paste" would appear little, if any, less confusing to the general public than that at present in use in the voluntary Codes of Practice where "X paste (or spread)" refers to products containing only the named meat or fish while "fish (or meat) paste, X" refers to products containing meat or fish other than that named "X". Finally, it is recommended that the full labelling requirements, including a declaration of ingredients, should apply to hermetically-sealed products, but that open-pack products should only be required to comply with the general recommendations for non-pre-packed foods in the proposals on labelling, i.e., they should be labelled with their usual name only, without an accompanying list of ingredients.

Two Codes of Practice were issued by the Local Authorities' Joint Advisory Committee on Food Standards in October, 1965. The first of these, Code of Practice No. 4—Canned Fruit and Vegetables, describes standards of fill, composition and sizes for canned fruit and vegetables and represents the recognised practice of the British Canning Industry. The other, Code of Practice No. 5—Canned Beans in Tomato Sauce, has been agreed with the Food Manufacturers' Federation and includes limits for total solids, tomato solids and sugar in canned products described either as "Beans in Tomato Sauce" or "Baked Beans in Tomato Sauce".

Finally, it should be mentioned that during the year 1965 the Food Standards Committee was asked by the Government to review the Food Standards (Preserves) Order, 1953, as amended. In addition to a general review of the order, the Committee has been asked to give special consideration to a number of points including:—the use of lactic acid in jams; a possible lowering of the soluble solids content of melon jams; an increase in the fruit content of raspberry jam; that only lemon and orange curds be required to contain oil of lemon or oil of orange; and whether there would be any advantage in having two standards for jams, i.e. a normal standard and one for extra quality jams.

The Determination of Cyclamate in Citrus Fruit Squashes

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A volumetric method for determining cyclamate in citrus fruit squashes is described. Sulphate is released from cyclamate by the action of nitrous acid and this is precipitated with barium. The excess barium is titrated with E.D.T.A. using a fluorescent indicator. A blank determination eliminates possible errors due to calcium, sulphate, or sulphur dioxide present in the squash.

The use of cyclohexanesulphamic acid and its sodium and calcium salts (hereafter referred to as cyclamate) for the sweetening of soft drinks is permitted by the Soft Drinks Regulations 1964[1].

At present the only officially recognised method for the determination of cyclamate is the A.O.A.C. method[2] which is due to the work of Wilson[3,4,5]. This method involves the gravimetric determination of sulphate produced by the action of nitrous acid on cyclamate and is not very suitable for determining cyclamate in citrus fruit squashes because of the difficulty of obtaining clear solutions.

Rees[6] has recently published a gas chromatographic method based on the analysis of the organic products of the nitrous acid reaction.

The proposed method is a volumetric determination of the generated sulphate using E.D.T.A. The determination of sulphate by precipitating with standard barium chloride solution and titrating the excess barium with standard E.D.T.A. was first proposed by Munger[7] and has been improved by Sijderius[8] and Bond[9]. All these methods use eriochrome black T as indicator and for most of the work a method combining those of Sijderius and Bond was used. This gave excellent results on pure cyclamate solutions, but when applied to squashes, the end-point tended to be masked by the artificial colour in the squashes. In some strongly coloured squashes the end-point could not be observed at all. This has been overcome by the use of fluorescent indicator, calcein blue, discovered by Wilkins[10]; rhodamine B is used to mask the residual fluorescence.

The presence of sulphur dioxide can cause high results because it is oxidised to sulphate by nitrous acid. Theoretically, this could be allowed for by determining the sulphur dioxide content (1 p.p.m. of $SO_2 \equiv 3{\cdot}14$ p.p.m. of cyclamate) but in practice this has not been found to give accurate results. It appears that some of the sulphur dioxide complexes are not oxidised in acid solution. Accurate results have been obtained by oxidising the sulphur dioxide in the blank determination with a dilute solution of hydrogen peroxide, the excess being removed when the solution is boiled. The blank determination is necessary to eliminate possible errors which would be caused by the presence of sulphate or calcium in the squashes.

Method

Apparatus

u.v. Lamp; principal wavelength 350 mμ*.

Reagents

1. *Barium chloride solution:* 0·02 M.
2. *E.D.T.A. solution:* 0·010 M
3. *Dilute hydrochloric acid:* 2 N HCl.
4. *Hydrogen peroxide solution:* 2-volume O_2.
5. *Sodium nitrite solution:* 10 per cent.
6. *Potassium hydroxide solution:* 8 M soln. of KOH.
7. *Calcein blue solution:* 0·05 per cent. (filtered if necessary) available from Hopkin & Williams Ltd.
8. *Rhodamine B solution:* 0·01 per cent.

Procedure

To two 250-ml beakers, add 10·0 ml of sample (or a volume containing 2–35 mg of cyclamate), 1·0 ml of 2 N hydrochloric acid and 10·0 ml of 0·02 M barium chloride solution. To one beaker add 1·0 ml of 2-volume hydrogen peroxide solution, place the beaker on a hotplate, cover with a clock glass and bring to the boil with occasional swirling. Boil the solution for one minute, remove it from the hotplate, wash down the clock glass and the sides of the beaker with water and cool the solution. When cool, add distilled water to increase the volume of solution to about 150 ml, add 4 ml of 8 M potassium hydroxide solution and place on the inverted u.v. lamp (which should be shielded and be positioned slightly above eye level) and stir the solution by an electric stirrer. Add rhodamine B solution until there is a slightly red fluorescence and then add three to five drops of calcein blue solution. Titrate the solution with 0·010 M E.D.T.A. solution until the blue fluorescence is quenched. The titration should be carried out under subdued lighting conditions; darkness is not required.

To the other beaker add 1·0 ml of 10 per cent. sodium nitrite solution (instead of the 2-volume hydrogen peroxide solution), place on a hotplate and continue as for the first beaker.

Calculation

$$\text{Cyclamate content} = \frac{\text{titre difference} \times 100 \times \text{F}}{\text{volume of sample taken}} \text{ per cent.}$$

* Most commercially-available lamps sold for chromatographic purposes have a principal wavelength at about 250 mμ and are not suitable. A suitable lamp is made by Camag and marketed by Camlab (Glass) Ltd., Cambridge. The Osram "black glass" mercury lamp is also suitable.

where

F = 0·001782 g for cyclamate anion
= 0·002012 g for sodium cyclamate
= 0·002163 g for calcium cyclamate dihydrate

Results

The results obtained with pure cyclamate solutions are given in Table I and with samples of orange and lemon squashes in Table II. The squashes contained various amounts of sugar and saccharin and no interference was observed.

TABLE I

RECOVERY OF "CYCLAMATE" BY E.D.T.A. TITRATION

Solutions employed	Concentration of "Cyclamate" Added *per cent.*	Found *per cent.*
Sodium Cyclamate	1·00	1·03 1·03 1·03
Sodium Cyclamate	0·50	0·505 0·503 0·503
Sodium Cyclamate	0·10	0·0967 0·0997 0·0997
Sodium Cyclamate (containing 250 p.p.m. SO_2)	0·25	0·250 0·250 0·250
Calcium Cyclamate Dihydrate ..	0·50	0·500 0·504 0·502

TABLE II

DETERMINATION OF "CYCLAMATE" IN CITRUS SQUASHES

Sample	Approx. "Cyclamate" Concentration	"Cyclamate" determined Found *per cent.*	Average *per cent.*
Orange A	0·35 per cent. Sodium Cyclamate	0·379 0·367 0·367 0·368	0·370
Orange B	0·35 per cent. Sodium Cyclamate	0·361 0·362 0·364	0·362
Lemon A	0·25 per cent. Sodium Cyclamate	0·228 0·230 0·232	0·230
Lemon B	0·25 per cent. Sodium Cyclamate	0·252 0·252 0·250	0·251

The precision of the method was estimated by calculating a mean for each squash and determining the standard deviation of all results from their respective mean values. The standard deviation after applying Bessel's correction was 0·0033 per cent. Thus for a probability of 95 per cent. the precision is better than ±0·01 per cent for the range covered.

Conclusions

It is considered that this method is suitable for the routine determination of cyclamate in citrus fruit squashes and that with slight modification it should be suitable for the determination of cyclamate in food if the legal use of cyclamate is extended.

Acknowledgements

I should like to thank Mr. D. F. J. Bird for suggesting the use of calcein blue indicator and Dr. E. C. Wood for his interest and advice. This work was mainly carried out at the Norwich City College under the supervision of Mr. G. Matthews, as part of a course in advanced analytical chemistry while the author was on day-release from J. & J. Colman Ltd., Research Department.

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The Determination of the Composition or Purity of Animal Feeding Stuffs by Microscopy

by J. HUBERT HAMENCE

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The use of lobelia seeds for evaluation of areas of characteristic fibres in the estimation of the constituents of animal feeding stuffs, is described.

The fibre constituents of most animal feeding stuffs have specific microscopical characteristics which are invaluable in their detection, and also in assessing their purity.

In this laboratory the fibre portion is prepared by boiling the finely ground cake or meal (ground to pass the 1-mm sieve) successively with acid and alkali following the procedure laid down in the Fertilisers and Feeding Stuffs Regulations. The fibrous tissue remaining after the alkali treatment is washed with water and then with 1 per cent. hydrochloric acid.

Portions of the prepared fibre are then placed between two microscope slides and examined under a 1-inch objective.

Examination of several (say twenty), fields, will show if a feeding stuff, for example a linseed cake, is pure linseed or whether it contains an admixture of other seeds or vegetable matter, chaff, etc. If foreign fibres are found to be present, indicating admixture, the question arises how the extent of the admixture can be determined by microscopical examination.

Where the impurity has the same amount and type of fibre as the main constituent, such as rape seed in a linseed cake, the proportion of the two constituents can be determined by estimating the area of the different fibres in about fifty fields. The area of the different fibres is assessed by means of a graticule divided into squares, and the ratio of the areas of the characteristic fibres is directly proportional to the amounts of the two constituents.

This technique has been used for many years to determine the proportion of admixture of rape in linseed cakes.

The efficacy of the method depends entirely on the fact that the fibres of linseed and rapeseed are similar in character and amount. When the constituents of the mixture contain different types of fibre, a simple counting of the areas of the different fibres is no longer capable of giving an assessment of the constituents present. Thus, it would be of little use trying to determine the proportion of linseed and rice bran meal in a mixture by determining the ratio of the areas of the different fibres. Clearly, a new technique is required to deal with such cases. For a solution to the problem, it is necessary to devise a method whereby the area of fibre obtained from a given weight of one feeding stuff can be compared with the area given by a similar weight of another.

Counting the whole area from a given weight is a tedious and difficult operation and therefore a simpler technique is required.

A similar problem arose some years ago in connection with the determination of the composition of a starch mixture by microscopy[1], and it was solved by the use of lycopodium powder as a "counting aid". In the method used a standard amount of lycopodium powder was added to the farinaceous mixture and by counting a number of fields, the ratio of the various starches to lycopodium spores could be obtained. If this ratio was determined for the individual starches then it was possible to determine the composition of a mixture.

Considerable difficulty was experienced in finding a standard material which could be used to replace lycopodium in the starch counting. An easily obtainable, uniform, organic material about the size of the average piece of vegetable fibre was required for the purpose. Many different organic materials such as finely ground straw, and sawdust from different woods carefully graded for particle size, were tried but without success.

Finally, small seeds were examined and found to be more suitable for the purpose, and lobelia seeds were selected as being the best.

Method

The "Lobelia Count" of a pure feeding stuff is determined in the following manner:—

Prepare the feeding stuff as described in the Fertilisers and Feeding Stuffs Regulations by grinding until it passes a 1-mm sieve. Weigh 0·9 gramme of the prepared feeding stuff, add 0·1 gramme of lobelia seed and prepare the fibre portion by boiling for 15 minutes successively with 1·25 per cent. sulphuric acid and 1·25 per cent. sodium hydroxide. Wash the fibre with water and finally acidify with 0·1 per cent. hydrochloric acid. Wash again with water. No fibre must be lost during the process. Nylon cloth is used for filtration.

Place the prepared fibre on a white tile and quarter down until a suitable portion, enough to cover a microscope slide, is obtained. Place on a microscope slide and mix with sufficient mucilage of tragacanth (1 per cent.), so that the mixture covers the slide. Spread over the slide, cover with another slide and press down.

Examine under a microscope, using a one-inch objective, and count 20 fields, selected at regular intervals, counting the number of lobelia seeds and the area in squares occupied by the "characteristic fibre" of the feeding stuff under examination in each field examined. Quarter down the fibre portion once more, prepare a second slide and make a second count of 20 fields.

If the crude fibre of the feed is also known the following constants can be calculated:—

1. The Lobelia Count, i.e. the number of squares of the characteristic fibre of a feeding stuff per 1 lobelia seed.

2. The Lobelia Count to fibre ratio, i.e. $\frac{\text{The Lobelia Count}}{\text{The Percentage Fibre}}$.

In simple mixtures, the Lobelia Count enables the percentage of the constituents to be calculated by comparison with standards obtained with pure feeding stuffs. The second constant, (the lobelia count to fibre ratio), enables the proportions of fibre in a mixture of feeding stuffs due to the different constituents to be calculated, and is of considerable value in calculating the composition of complicated mixtures.

An example will make the method of calculation of these two constants clear.

A cotton seed cake containing 9·9 per cent. of fibre determined by the official method gave the following results using 0·9 g cotton seed cake + 0·1 g lobelia. The number of seeds of lobelia counted was 103, the number of squares of cotton fibre was 937. Thus, the Lobelia Count = 9·1 and the Lobelia Count to fibre ratio = 0·9.

The characteristic fibre of a particular feeding stuff must depend upon the preference of the individual analyst and no useful purpose would be served in this paper in giving details of the fibres chosen in our laboratory.

The following results, however, will serve as a guide to the type of figures obtained.

TABLE I

ANALYSIS OF FEEDING STUFFS: LOBELIA COUNT AND FIBRE RATIO

Feeding Stuff	Lobelia Count	Fibre *per cent.*	Lobelia Count to fibre ratio
Linseed Cake	13·2	8·0	1·65
Cotton Seed Cake	9·1	9·0	0·92
Sesame Cake	21·8	23·0	0·95
Poppy Seed	25·2	8·0	3·15
Ground Nut	6·1	15·3	0·40
Rice Bran	5·0	9·9	0·51
Soya Meal	7·6	4·0	1·90
Barley Meal	3·2	3·5	0·92
Rape Meal	16·0	9·7	1·65
Dried Grass	28·7	—	—
Thistle Seeds	21·5	47·9	0·45
Coffee Husk	17·0	27·2	0·62

It will be seen from the foregoing table that there is a wide variation between the different feeding stuffs. The lobelia count shows the bigger variation, which is only to be expected due in part to the wide variation in fibre contents of different feeding stuffs.

The lobelia count to fibre ratio shows a smaller variation, but in reality this is the more important factor since it is a measure of the difference between the fibre contents of different feeding stuffs. The relative thick fibre of ground nut has a constant 0·40 whereas the thin fibre of poppy seed has a constant of 3·15.

This shows clearly why a straight calculation of areas of fibre is of little value in composition analysis unless the fibres are similar.

The following comments and examples may be helpful to show how the foregoing technique is applied in practice.

Where a single impurity in a cake is the subject of investigation it is only necessary to make a lobelia count of the impurity, thus avoiding the additional time which in the past has been spent in also counting the main constituents of the cake. For instance, a linseed cake which was contaminated with thistle seed was found to give a lobelia count for the thistle seed of 5·5, which was equivalent to approximately 25 per cent. of thistle seed.

Similarly a sample of sunflower seed which was contaminated with rape gave a lobelia count for the rape of 1·9, which was equivalent to approximately 12 per cent of rape seed. In neither instance was the major constituent of the cake counted.

In the majority of instances apart from a microscopical lobelia count we find it advisable to make a partial chemical analysis, at least for protein and fibre. The composition of the sample under examination may then be calculated from the average figures for the constituents found to be present. If the calculated composition differs from that found by any significant amount then clearly an explanation for the difference must be sought.

An interesting example of this is the case of a ground nut meal which was found to be contaminated with cotton seed meal. Although the protein was found to agree substantially with the theoretical figure, nevertheless the fibre calculated from the lobelia count—fibre ratios, was found to be some 5 per cent. short. During the lobelia count it had been noted that the sample included a large proportion of cotton lint, and a further determination of the lint constituent showed that 4 per cent. was actually present, which explained the missing percentage of fibre.

An example of a complicated mixture consisted of a cotton seed which was heavily contaminated with coffee husk and ground nut.

From the lobelia count we calculated that the sample consisted of:—

Cotton Seed	..	..	52 parts
Coffee Husk	..	..	28 parts
Ground Nut	..	..	12 parts

The lobelia count therefore accounted for 92 per cent. of the sample.

The lobelia count—fibre ratio indicated the presence of 14·6 per cent. of fibre, representing only 90 per cent. of the total fibre, which was actually 16·2 per cent. Further examination of the sample showed no other constituents to be present, and the apparent deficiency in the fibre content was clearly due to experimental error inherent in the method. To calculate the composition of the feeding stuff therefore the percentages found by the lobelia count were all multiplied by a factor of 1·09.

One of the major difficulties which arises in applying this technique to the examination of compound feeding stuffs is that some cakes, such as groundnut and cotton seed, may be used in both the decorticated and undecorticated form, and in most instances we would have no knowledge of the fibre content of the original cake. In such instances quite clearly the chemical analysis of the cake must be taken into consideration in calculating composition.

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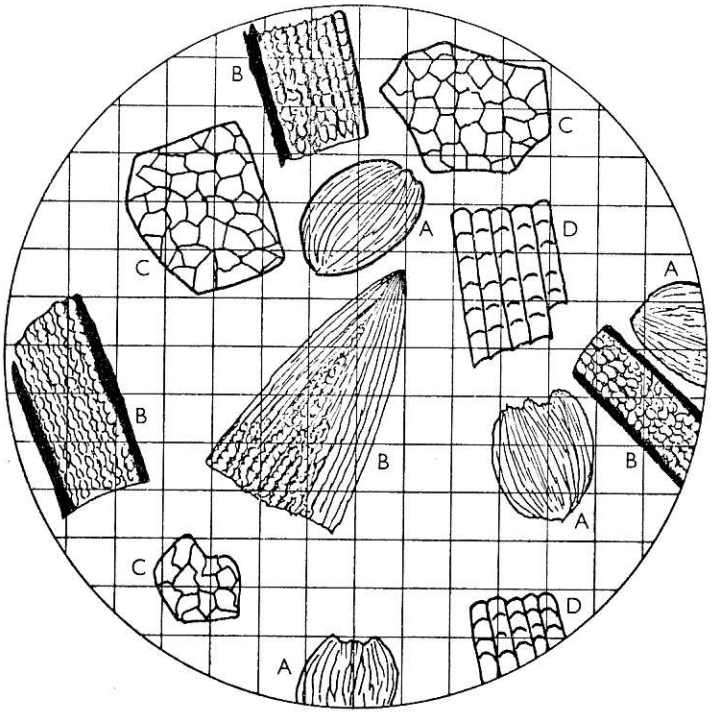


THE MICROSCOPIC STRUCTURE OF FEEDING STUFF FIBRES

The diagram shows a typical field obtained during a lobelia count of a sample of linseed cake heavily contaminated with Rice bran and brassica (rape).

A. Lobelia seeds.
B. Linseed
C. Brassica (rape)
D. Rice bran.

The squares on the graticule in the eye piece which are used for counting the "squares of fibre" are seen in the background.

Photography and the Public Analyst

by MISS A. COOK AND A. J. HARRISON
(*Public Analyst's Dept., Clive Road, Portsmouth*)

A description is given of the Edixa Reflex camera and accessories, and ways in which they can be applied to the work of the Public Analyst. Details of the adaptation to photomicrography, document copying, etc., are supplemented by a summary of the lighting, development and printing techniques involved.

Two years ago it was decided that it would be an advantage to have a camera in this laboratory capable of a wide range of uses and not limited to photomicrography only. A thorough examination of the available types of camera was made with a view to obtaining an instrument with maximum flexibility. The type most suited to this requirement is that known as the "Single Lens Reflex", with a "seen through the lens" viewing system. Of the models available we purchased an Edixamat Reflex Model D camera with various accessories.

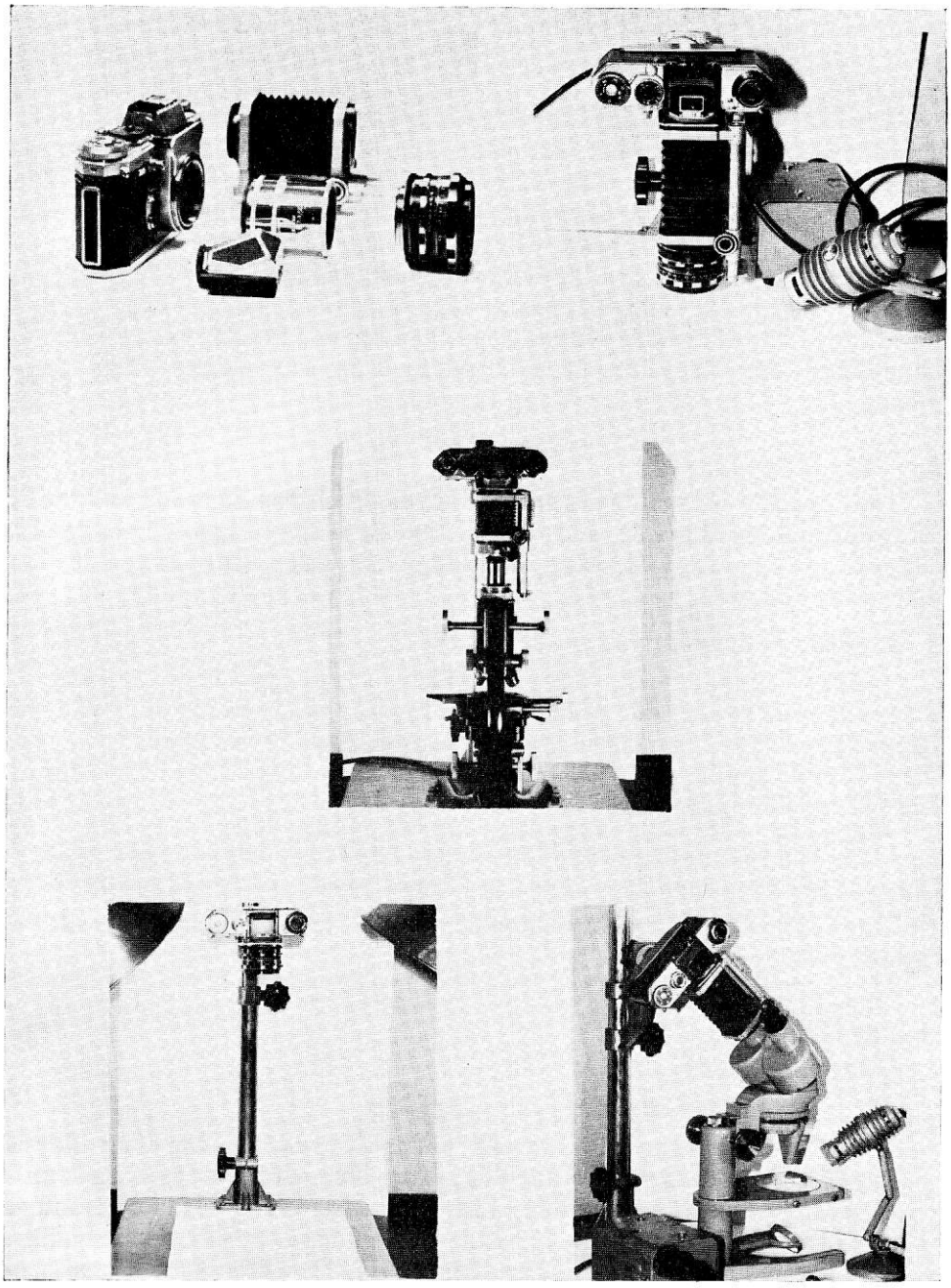
The camera is a 35-mm model. The viewing screen is fitted with a waist-level viewfinder and a plano-convex lens ground on the under-side, incorporating a 4× magnifier for more accurate viewing. The camera lens serves both for taking the picture and also for focusing, the proposed picture being seen the right way up but laterally reversed. This is achieved by a mirror system built into the camera. The waist-level viewfinder may be replaced by a penta-prism for eye level viewing in which case the screen image is seen the right way up and not laterally reversed. The camera has a focal plane shutter which enables the lens or accessories to be changed whilst the camera is loaded with film. The shutter incorporates a delayed action release system with adjustable delay from 2 sec. to 9 sec. and a total range of automatic shutter settings from 9 sec. to 1/1000th sec. It is fully synchronised for bulb or electronic flash.

The lens is a "Xenar", f2·8, of focal length 5 cm, with a fully automatic, spring loaded, iris diaphragm. This is set to the aperture required beforehand, when the iris will remain fully open for reflex viewing, but stops down automatically to the pre-selected opening an instant before the shutter opens. The iris then returns to the full opening immediately after use. The lens is capable of focusing from infinity to 1·25 feet.

Accessories

1. *Extension Bellows* (*see Fig.* 1)

For greater adaptability and a wide range of close up work this attachment is essential. It screws in between the camera body and the lens. The focusing



Top left: Fig. 1. *Top right:* Fig. 2. *Centre:* Fig. 3.
Bottom left: Fig. 4. *Bottom right:* Fig. 5.

range with bellows is continuous and very extensive; with a lens of focal length 5 cm, the scale of reproduction is from 0·5 × image size to 2·3 × image size.

2. *Extension Rings* (*see Fig.* 1)

A set of 4 extension rings of varying length act similarly to the bellows attachment but are somewhat more limited in use. Subject area, when using these rings in various combinations, ranges from 24 in × 36 in to $\frac{7}{8}$ in × $1\frac{3}{8}$ in which gives a scale of reproduction of 0·04 × to 1·02 × on the film in relation to the natural size of the subject.

3. *Copying Unit* (*see Fig.* 4)

This consists of a stand with an adapter clamp which allows the camera to be attached and rotated to any angle required for copying and photomicrography.

4. *Light Meter*

The Japanese Sekonic light meter is used, which has high and low scales ideal for photomicrography and similar conditions involving low light values. The meter is operated by a cadmium sulphide cell which works in conjunction with a small battery.

5. *Lighting Equipment* (*see Figs.* 2, 3, 4, *and* 5)

Various lighting sources are used which include photofloods of selected powers housed in aluminium holders (see Fig. 4); an electronic flashlight, the Bactil light, is fitted permanently in the Watson High Power Binocular microscope with a variable power control and small microscope lights with focusing lens and power control unit.

6. Other equipment includes an Edixa automatic focusing enlarger with f3·5 lens, developing tanks and the usual basic photographic equipment.

The total cost of the above equipment for professional purposes is in the region of £130.

Applications

1. *Photomicrography*

(*a*) *High Power Microscopy* (*see Fig.* 3)

Photomicrographs are easily obtained using the camera with the bellows attachment and no lens. The camera is used with the waist-level viewfinder. The ground glass viewing screen is a distinct advantage when focusing the image, particularly when used with the 4 × magnifier. The camera is set up as in the diagram (Fig. 3), with the bellows attachment fitted over the monocular head of the microscope which contains the 10 × compensating eyepiece. It is loaded with Ilford Pan F film which

is a fine grain film ideal for this kind of work. When the image is focused, the Bactil light fitted to the microscope is set at 6 volts and the photograph taken using the cable release together with the "delayed action release" to avoid camera shake. A normal exposure time for photographs of this nature is 1/10th to 1/5th second.

(*b*) *Low Power Microscopy* (*see Fig.* 5)

In a similar manner, for photographs of slightly larger objects, the camera is used in conjunction with the low power Watson Stereoscopic microscope. The camera, in this instance, is fitted over the focusing eyepiece of the microscope with general illumination from two No. 1 photofloods and highlighting the object with a small microscope light with focusing lens (see Fig. 5). Exposure is carried out as for high power work, suitable exposures ranging from one tenth to a half second, depending upon the nature of the subject.

Determination of Exposure Times for Photomicrography

In our experience it is an advantage to carry out a series of test exposures under fixed lighting conditions, using a suitably mounted specimen for the subject. The negatives are used as reference standards for future work carried out under similar conditions.

Estimation of Final Magnification

It is often desirable to know the final magnification achieved on the print of the photomicrograph. This is determined by the use of a graticule on the microscope stage. The camera, with bellows attachment and no lens, is positioned at different heights above the eyepiece of the microscope and the image focused on the "seen through the lens" viewing screen. With a knowledge of the height of the camera above the microscope and a simple measurement of the distance between the graticule lines seen in the screen, a graph of the magnification can be obtained. One must also bear in mind that this should be multiplied by the magnification produced when enlarging the print to give the final magnification.

2. *Document Copying* (*see Fig.* 4)

Here again the advantages of the ground glass viewing screen with magnifier are apparent, giving a sharp image of the type. For all copying work, Ilford Fine Grain Safety Positive film is used which has a fine grain and gives faithful reproduction of coloured print, an advantage over some commercial copying machines which have varying record response with blues and reds.

The camera is attached to the adapter clamp and focused on to the document to be copied. Two photofloods (No. 2), are placed approximately 1 foot from the base of the stand (see Fig. 4). The light meter is held 10 cm from the

document in order to obtain a suitable exposure time, taking care that the meter aperture is parallel with the document to be copied so that an accurate reading of the illumination of the latter is obtained. Again the exposure is made using the cable release and "delayed action" release to avoid camera shake. Some difficulty due to reflections may be experienced if the document is highly glossy, but this can be avoided by careful positioning of the lights.

3. *Record of Foreign Materials in Food*

Every analyst has the problem of foreign materials in food and the added problem of how to preserve records of their appearance at the time of receipt. The Edixa plus either the bellows attachment or extension rings, depending on the size of the object, is ideal for recording these details. A series of photographs can be taken, e.g. the object in the article as received, and eventual breakdown photographs increasing in magnification to illustrate the true nature of the object. Pan F film is used, lighting systems varying according to the object. For large objects two No. 1 photofloods are adequate for overall illumination and, using the light meter, a suitable shutter speed and lens f-number can be selected. It is advisable to work with a small iris opening in order to obtain the maximum depth of field.

For very small objects the small microscope lights can be incorporated to eliminate shadows when using photofloods for general illumination.

4. *Miscellaneous Uses of the Equipment*

(*a*) The preparation of a reference collection of photomicrographs of common animal hairs.

(*b*) Photographs of foreign materials in foodstuffs which have been submitted as a sample or complaint to Local Authorities and which might subsequently be the subject of a prosecution.

(*c*) The camera has been used successfully for recording on film, information only available from reference libraries, etc. Of course prints can easily be obtained if required, but film is much easier to store.

(*d*) Permanent records of paper chromatograms can be made using this equipment and it is hoped that this facility will prove invaluable for records of thin layer chromatograms.

(*e*) Photographs of laboratory equipment set up for special, non-routine determinations have proved to be well worth while when it is desired to reconstruct the apparatus.

(*f*) Site photographs taken when investigating allegations of excessive atmospheric pollution were used to supplement the report.

(*g*) Photographs taken in this Department have been used to supplement lectures given to local organisations, and it is intended in the future to produce a series of colour slides for this purpose.

Films and Printing Papers

Experience has shown that the purchase of 35-mm film in bulk is more economical than the purchase of 20- or 36-exposure refills. Variable lengths of film can be used with minimum wastage provided that the film is loaded into the cassette and camera in complete darkness. A suitable notched bench in the darkroom facilitates accurate measurement of cut lengths for loading into cassettes. At each loading, $1\frac{1}{2}$ frames are wasted as "lead in" for cassette and camera.

For photo-copying, Ilford Copying film is used and may also be purchased in bulk lengths (i.e. approximately 100 feet for both Pan F and Copying film). This film has the additional advantage that it may be cut and loaded in a darkroom illuminated with an Orange 902 safelight.

This laboratory has standardised on the Kodak range of printing papers which have been found to be generally applicable to the techniques previously described. The white smooth glossy paper gives good quality prints and faithful reproduction of detail.

Hard, normal and soft grades of this bromide paper are used, depending upon the intensity of the negative. Generally speaking, a negative of average contrast gives a good print on the "normal" grade of paper, showing a full range of tone. A white fine lustre surface is sometimes considered more suitable for site photographs, apparatus, etc.

As with film, printing paper may be purchased more economically in bulk. Large sheets (8 in $\times$ 10 in) are purchased which may be cut into a suitable size with little or no wastage.

Film Development and Fixing

It is more convenient to prepare "home-made" stock solutions of Developer and Fixer as required.

1. *Pan F Films*

These are developed for $6\frac{1}{2}$ minutes at 20° C using ID.11, fine-grain developer, used at full strength and prepared as follows:—

Metol	4 g
Sodium sulphite (anhydrous)	200 g
Hydroquinone	10 g
Sodium borate	4 g

Dissolve and make up to 2,000 ml with distilled water, adding each reagent in the order given above. Filter into a dark brown bottle for storage.

The exposed film is loaded into the developing tank in the dark room and processed according to the instructions given by the makers of the tank.

There are two Fixers which may be used, either IF.2 Acid Fixer or IF.9 Acid Fixer and Hardener, as follows:—

IF.2 Fixer

This fixer arrests development immediately and is used at full strength.

Sodium thiosulphate (anhydrous)	512 g
Sodium metabisulphite	42·5 g

Dissolve and make up to 2,000 ml with distilled water.

IF.9 Fixer and Hardener
(used at full strength)

Sodium thiosulphate (anhydrous)	384 g
Sodium metabisulphite	42·5 g
Chrome alum	25 g

Dissolve the thiosulphate and metabisulphite in water (Solution A); dissolve the chrome alum in approximately 200 ml of water. Add this solution to soln. (A) and make up to 2,000 ml with distilled water.

After fixing for 10 minutes, rinse the film once with distilled water and then wash for ½ hour in running tap water. A final rinse with distilled water containing 1 drop of "Teepol" will "wet" the film and allow even drying. The film should be hung in a warm, dust-free atmosphere until dry. In this laboratory, processed film is cut into lengths of up to 6 frames, which are filed in transparent cases attached to information cards. On these cards full details of the subject and conditions of exposure are recorded.

2. *Copying Film*

This is developed in a similar manner to Pan F film but using ID.2 Developer, prepared by diluting 1 part of stock solution with 2 parts of distilled water *immediately before use*. The film is developed for 4–6 minutes depending on the contrast required (average time 4 minutes), at 20° C. The stock solution has the following formula:—

Metol	2 g
Sodium sulphite (anhydrous)	75 g
Hydroquinone	8 g
Sodium carbonate (anhydrous)	37·5 g
Potassium bromide	2 g

Dissolve and make up to 1,000 ml with distilled water, and filter into a brown storage bottle. Ensure that the reagents are dissolved in the order given above.

Either IF.2 or IF.9 Fixers may be used for fixing the negative. The film is then washed and dried as for Pan F films above.

Printing

The Automatic Focusing Edixamat enlarger is convenient and efficient particularly from the time-saving point of view and it has the added advantage *that the number of the negative being printed is always visible.*

Having placed the negative in the enlarger, and selected the area to be printed and the size of the print required, a series of exposures in geometric progression can be made. A simple way of doing this is to give the whole of a

Printing

The Automatic Focusing Edixamat enlarger is convenient and efficient particularly from the time-saving point of view and it has the added advantage that the number of the negative being printed is always visible.

Having placed the negative in the enlarger, and selected the area to be printed and the size of the print required, a series of exposures in geometric progression can be made. A simple way of doing this is to give the whole of a test strip of paper a 5 seconds exposure, and then to cover up successive strips with a card whilst giving further exposures of 5, 10, and 20 seconds. This results in 4 bands having exposures of 5, 5 + 5 + 10, 5 + 5 + 10 + 20 seconds respectively. The test strip is then developed and fixed to give graded exposures from which a suitable exposure time for the print may be deduced.

The developer used for printing is ID.22 which must be prepared fresh immediately before use as follows:—

Sodium sulphite (anhydrous)	12·5 g
Amidol	3·0 g
Potassium bromide	0·8 g
Make up to 500 ml with distilled water and filter.	

At 20° C the developer will produce a good quality print after $1\frac{1}{2}$ minutes total development time, provided the exposure has been correctly assessed.

After some experience, the test strip procedure will rarely be required as suitable exposure time can be estimated visually with a knowledge of the quality of the negative, etc. The average time for fixing prints is 10 minutes and we use the following IF.4 fixer which arrests development immediately.

Sodium thiosulphate (anhydrous)	256 g
Sodium metabisulphite	42·5 g
Dissolve and make up to 2,000 ml with distilled water.	

Prints, after fixing for 10 minutes, should be washed for $\frac{1}{2}$ hour in running water before drying.

Note: If a halfpenny or penny is suitably placed on the paper before exposing, a blank white spot will be produced on the print in which details of the sample, date, etc., can be recorded.

ERRATA

3rd Quarter, 1965 issue, p. 77,

Line 14*: For* "35 ml of chloroform" *read* "35 ml of glacial acetic acid".

4th Quarter, 1965 issue, p. 135,

Line 12: *For* "Lawson" *read* "Lowson".
Line 18: For "Burnell" *read* "Burrell".