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# New Food and Drugs Legislation and Proposals for Regulations, etc., during 1967

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Twelve Statutory Instruments which will make an impact on the work of Public Analysts' laboratories were published in 1967. These were:—

The Meat Pie and Sausage Roll Regulations, 1967.

The Canned Meat Product Regulations, 1967.

The Sausage and Other Meat Product Regulations, 1967.

The Artificial Sweeteners in Food Regulations, 1967.

The Solvents in Food Regulations, 1967.

The Food (Control of Irradiation) Regulations, 1967.

The Carcinogenic Substances Regulations, 1967.

The Toys (Safety) Regulations, 1967.

The Labelling of Food Regulations, 1967.

The Coffee and Coffee Product Regulations, 1967.

The Ice-Cream Regulations, 1967.

The Margarine Regulations, 1967.

Two reports were published by the Food Standards Committee of the Ministry of Agriculture, Fisheries and Food, one on compositional standards for Cream, the other on cyclamate sweetening substances.

Proposals for new Regulations, which also appeared last year, included the long-awaited draft dealing with Claims and Misleading Descriptions on Labels. A second document was concerned with Skimmed Milk containing Non-Milk Fat.

The Farm and Garden Chemicals Act became effective in July, 1967, and a white paper dealing with intended legislation on the Safety, Quality and Description of Drugs and Medicines was published in September. Both these subjects are of direct interest in Public Analysts' laboratories.

The first three of the above Statutory Instruments might almost be read together, namely, The Meat Pie and Sausage Roll Regulations, 1967, The Canned Meat Product Regulations, 1967, and The Sausage and Other Meat Product Regulations, 1967.

The Meat Pie and Sausage Roll Regulations were made in May 1967, and will operate from May 1968. They specify requirements for the composition of meat pies, puddings or pasties (including meat pies, etc., containing meat and vegetable, meat and egg, meat and cheese, or meat, egg and cheese) and sausage rolls. They also specify requirements for the labelling and description of these foods, and for their advertisement.

The basic requirement is that meat pies, puddings or pasties shall contain at least 25 per cent. of meat; but if sold uncooked, they are only obliged to contain 21 per cent. meat. In addition, one of those products which seem only to occur when Regulations are made, has loomed up. Under the name of Scottish or Scotch Pie (which, from the definition given, seems as if it may be an open-topped meat pie) must contain at least 20 per cent. of meat if sold cooked, and 17 per cent. of meat if sold uncooked. They are believed to be mutton pies made in a pre-cooked pastry case, with soaked brown bread incorporated into the filling along with the meat. Some of these products have tolerances, which amount to allowing meat contents of only about 85 per cent. of the generally prescribed 25 per cent. meat standard in products of which the weight approaches the maximum of the various weight ranges listed. A further tolerance seems to have been allowed for the specially popular pie size which falls into the weight range 4 ounces to $5\frac{1}{2}$ ounces. In practice, therefore, some $5\frac{1}{2}$ ounce pies having a meat content of only about 73 per cent. of that prescribed will be acceptable. These requirements are slightly complicated by an allowance for fat which might migrate to the pastry. Ordinarily it would appear that pastry recipes require one part of fat to two parts of flour, but in Scotland it must be one part of fat to three parts of flour. Fat in the pastry, therefore, in excess of 50 per cent. of the carbohydrate content, may be counted as meat . . . except with Scottish or Scotch pies, where all fat in excess of 35 per cent. of the carbohydrate may be so calculated. This could be interpreted to allow pies to be sold with very small actual lean meat contents.

Meat and vegetable pies, puddings or pasties are required to contain at least $12\frac{1}{2}$ per cent. of meat, unless sold uncooked, when the requirement is $10\frac{1}{2}$ per cent.

Meat pies which also contain egg or cheese or both, must contain at least $12\frac{1}{2}$ per cent. of meat, and the other above-named protein ingredients must combine with this to make the total contribution from all these sources together up to 25 per cent. For uncooked items, the required amounts are $10\frac{1}{2}$ and 21 per cent. respectively. With sausage rolls, the required meat content is $12\frac{1}{2}$ per cent. (cooked) and $10\frac{1}{2}$ per cent. (uncooked).

Whichever of the above kinds of pie, pudding or pasty is sold, the customer must be made aware of the variety. If the comestible is a cooked, uncomplicated, conforming pie, it should be labelled "Meat Pie" (Pudding or Pasty), and although it takes some time to find (Regulation 11 (1) (*i*)), it seems that it is in order to replace the word "Meat" with words like "Beef" or "Steak" or whatever is appropriate. Only when the 25 per cent. minimum meat

content is not reached is it necessary to look at Regulation 6 (3) to make sure that the words "Uncooked", "Scottish" or "Scotch", or "Sausage Roll" can be used singly or in combination. The notification to the customer may be by labelling the container, displaying a label in close proximity to the pie, or verbally.

There is, at first sight, a maze of labelling requirements for meat pies containing meat and vegetable. In fact, it is all reasonably straightforward, the words "Meat and Vegetable Pie" being reserved for those articles with a meat content between 12½ per cent. and 25 per cent. of meat. This is subject, of course, to the usual codicil concerning "Uncooked" pies and permutations and combinations which allow the word "Meat" to be replaced by a named variety of meat, the word "Vegetable" to be replaced by the name of the vegetable used, "Pasty" to be interchangeable with "Pudding" or "Pie", and "and" to be replaceable by "with". "Forfar Bridie" or "Cornish Pasty" are names reserved for Meat and Vegetable Pies of their peculiar standard only, and may take the place of all or any of the above possible names for Meat and Vegetable Pies.

A pie made from vegetables and less than 12½ per cent. of meat, may be sold, provided that the ingredients in the name are in the reverse order to those in the more "meaty" product—"Vegetable and Meat Pie", or any of the obvious alternatives which preserve this order, being accepted forms. Again, the customer must know what he is getting. One could predict correctly, that if the pie contains no meat, the appropriate name becomes "Vegetable Pie", not forgetting a proviso about "Uncooked" and the possibilities for playing Put-and-Take with vegetable names and "Pie", "Pudding" and "Pasty".

Pies however, may also be made from meat, egg and cheese. The rules for naming them are analogous to those set out above, although they take longer to enumerate. If the meat content is greater than 12½ per cent. and less than 25 per cent., and the total protein-food contribution exceeds 25 per cent., "Meat and Egg Pie", "Meat and Cheese Pie", "Meat Egg and Cheese Pie" or "Meat Cheese and Egg Pie" are the appropriate forms, with the words "Uncooked", "Pudding" or "Pasty", "with" and meat names all being allowed for. Again, provision is made for such pies, having meat contents of less than 12½ per cent. to be sold, provided that they are labelled along the alternative lines of placing the meat part of the name in the subordinate position in the title.

Every description must be conspicuously and legibly printed near the name of the product in light print on a dark ground or in dark print on a light ground.

The entire requirement is summarised in Regulation 11 except that by attempting to take this short cut, the reader will miss the exemptions granted to products which weigh less than 1½ ounces, those products intended for feeding soldiers, or for export, and for any product which contains only fat as its meat ingredient.

The Regulations seem to be very well drawn, in such a complicated situation, but an omission which has worried Public Analysts and Inspectors is the

problem of how a formal sample should be taken. The weight categories make it imperative for the analyst to have whole pies, and the most reasonable suggestion so far made is that a sample for division should consist of nine pies, three of which are taken whole in random fashion for each of the Inspector's three jars. It remains for magistrates to decide whether this procedure is acceptable. If it is not, the Regulations will be unenforceable. The Ministry of Agriculture, Fisheries and Food is aware of the difficulty, and a letter, reference FS 2248B, dated 20th June, 1967, was sent by Mr. J. H. V. Davies of the Food Standards Division, to all Food and Drugs Authorities, somewhat non-committally drawing attention to the need for careful interpretation of analytical findings on pies especially in the case of smaller pies.

S.I. 1967 No. 861 is called The Canned Meat Product Regulations, 1967. These Regulations were made in May 1967, and become operative in May 1969. In them "Can" can mean "Jar". "Canned meat with cereal" will not include faggots, rissoles, croquettes or meat balls. "Cereal" includes potato and soya flour but not pasta, pastry, dumplings, or grain rice. "Container" can mean, as with meat pies, a mere encircling band. The words "Gravy" and "Sauce" need to be considered carefully because the former can include "Gravy sauce". Meat, alas, can mean tripe and a certain amount of skin, rind, etc., and "Lean meat" is meat trimmed free from visible fat. It may therefore be necessary to use some factor such as CALCULATED LEAN $\times$ 1·05 in order to obtain defined lean meat, after which the fat found would have to be reduced by the equivalent plussage. "Meat paste" includes "Potted meat", and "Vegetable" includes mushroom or other edible fungus, tomato and grain rice.

The exemptions also need noting, for they contain such things as canned sausages, canned ham (under certain circumstances), canned whole or part bird carcases, and such gruesome possibilities as canned Haggis, canned black pudding or canned white pudding. There is also a startling exemption for canned sandwiches. The Regulations amend the Labelling of Food Order, so that lists of ingredients shall still be given with canned meat products.

Provision is made for the possible presence of meat-like extras in meat products. Thus, provided that its presence is stated, egg may be calculated as meat up to one-fifth of the total meat content . . . the calculation should prove interesting. The Regulations specify minimum meat contents, and by Article (5) (2) at least 60 per cent. of such minimum meat content shall be lean meat. Bone in chops, oxtail, etc., can count as meat if its presence is declared, but if the presence of bone is not declared it can count only as part of the total weight of the product, and not as part of the meat content. Sausage skins and similar casings will not count as "meat" although these can add one per cent. to the meat content of an ordinary sausage and canned products containing dehydrated meat shall have the meat contents calculated on the reconstituted material.

The prescribed meat contents of canned meat products are as follows:—

| | Meat Content |
|---|---|
| Canned Meat | 95 per cent. |
| Cured Meat | 90 per cent. |
| Savoury Minced Meat | 85 per cent. |
| Meat with Cereal (or Luncheon Meat) | 80 per cent. |
| Meat with Jelly | 80 per cent. |
| Meat with Gravy | 75 per cent. |
| Meat Loaf | 65 per cent. |
| Meat with Sauce | 60 per cent. |
| Brawn, Collard head, Pressed Meat | 60 per cent. |
| Sliced Meat with Gravy | 60 per cent. |
| Stuffed Meat Loaf | 50 per cent. |
| Meat with Onion (or Mushroom, or Asparagus) and Gravy | 50 per cent. |
| Meat with Stuffing (or Dumpling, or Pasta) and Gravy | 50 per cent. |
| Meat with Onion (or Mushroom, or Asparagus) and Sauce | 40 per cent. |
| Meat with Stuffing (or Dumpling, or Pasta) and Sauce | 40 per cent. |
| Canned Faggots, Meat balls, Rissoles or Croquettes | 35 per cent. |
| Curried Meat | 35 per cent. |
| Curried Meat with Rice | 15 per cent. |

The names "Canned meat with gravy" ("with sauce" or "with cereal") used with the words "Pie filling", will imply a product containing only 35 per cent. of meat. Such products, and canned rissole products, may also opt out of the minimum requirements by mentioning other ingredients before the meat in the product's name, or by omitting the word "meat" altogether. They also evade the need to conform to any minimum meat content if they are sold with at least two kinds of vegetable, or one kind of vegetable with some pasta or dumpling, under the name "Ready Meal".

Regulation 7 will cause some public analytical murmurings. It starts happily enough by saying that when any of the above items are canned as part of another product, or when sausages, pies or sausage rolls are so incorporated, all these things shall, at the time of canning, conform to the Regulations which apply to them. Unfortunately Regulations 7 (3) and (4) permit such items to count as if they were all meat when they provide the meat in a composite product, or when they are canned with another meat product. The simplest example would be with a product like Hamburgers, which are to be considered to be "Meat with Cereal" and therefore to contain 80 per cent. of meat. If sold canned as "Hamburgers in Gravy", the meat content of the can would need only to be that appropriate to 80 per cent. of the 75 per cent. of meat ordinarily required for meat and gravy products, i.e. the can would need to contain only 60 per cent. of meat.

The Regulations specify that unambiguous names be used, but there are many alternative names possible, including those with the insertion of

descriptive terms to describe the meat or the sauce, etc., with various names for the meat or the cereal used, and with other meat names which may replace the "Ham" part of the word "Hamburger".

After seeing what has now been attempted in the previous two orders it seems incredible that the humble English Sausage, banned from sale altogether in the U.S.A. and upon which the British public spends £100 millions each year, should have had no official standards for meat content for about 14 years. In the 1948 Order, renewed in 1952 and revoked in 1953, the pork in a pork sausage was allowed to be diluted with other meat so that it could contain 20 per cent. of meat other than pork. This, almost the only recent example of true adulteration in the sense of diluting an expensive commodity with a cheaper one, has been connived at by the Ministry for so long that even Public Analysts have come to accept the practice. It is said to have been the difficulties associated with the accurate determination of this concessionary 20 per cent. of the permitted adulterant which has held up the introduction of standards for sausages for so long. Now, approximately 20 years later, in a time when meat shortages like those experienced in the 1940's can exist only for very different reasons, the suggested standards for Pork and Beef sausages are almost exactly those of 1948.

The Statutory Instrument, 1967, No. 862, which comes into force in May 1969, deals with more than sausages, however. Its title, "The Sausage and Other Meat Product Regulations, 1967" holds the clue that much of it overlaps with the Canned Meat Products Regulations to regulate similar meat products sold loose instead of in cans.

The standards laid down for sausage products are:—

| | Meat Content |
|---|---|
| Sausage, Sausage Meat, Polony, Hog Pudding | 50 per cent. |
| Pork Sausage or Sausage Meat | 65 per cent. (of which 80 per cent. must be pork). |
| Beef Sausage or Sausage Meat | 50 per cent. (of which 50 per cent. must be beef). |
| Frankfurter, Vienna Sausage or Salami | 75 per cent. |
| Canned Frankfurter, Vienna or Salami Sausage after draining | 70 per cent. |

(*This standard is for less meat than was formerly suggested for Continental and Salami sausages, the* 1952 *Orders having stated* 80 *per cent. of meat for these.*)

| | |
|---|---|
| Liver Sausage, Tongue Sausage | 50 per cent. (of which 30 per cent. must be Liver or Tongue). |

A further requirement concerning the meat content of sausages and sausage meat is that at least 50 per cent. of the specified minimum meat content shall consist of lean. In practice therefore this amounts to a lean meat standard of 25 per cent. in a beef sausage and 32½ per cent. in a pork sausage. A moment's calculation reveals some interesting possibilities. A pork sausage, which is required to contain 65 per cent. meat, may in fact contain only 52 per cent. pork (*i.e.* 80 per cent. of 65 per cent.) so 13 per cent. may be beef or mutton. Since only 32½ per cent. of lean meat need be present, the rest being lard, and assuming the beef or mutton to be lean, this means that only 19½ per cent. of a pork sausage which conforms with these long awaited standards needs to be lean pork! In view of the changing relative prices of the different varieties of meat this may be less serious than it appears to be.

The remaining items mentioned in the Order fall in line with the requirements of The Canned Meat Regulations. Thus egg, when declared, may be counted toward the meat content up to 20 per cent. of the weight of the product. Fat may not be present in excessive amount in other products as well as sausages, and at least 60 per cent. of the minimum meat content in such other products must be lean meat.

*Almost* every kind of product mentioned in previous orders appears to have been considered, although a perusal of the first schedule, paragraph 1, in The Meat Products (No. 2) Order, 1952, reveals that some other esoteric meat products are known to the Ministry, besides those which appear in these three Regulations.

All three of these Regulations have been amended since they first appeared, so that the size of lettering on labels shall comply with the requirements of the new Labelling of Food Regulations.

The Artificial Sweeteners in Food Regulations, 1967, were made in July 1967, and came into operation in two stages. As from August 1967, new sweetening tablets came into being in which both saccharin and cyclamates were allowed to be present. Until December 1967, the newly-accepted cyclohexyl sulphamic acid sweetening agent was not permitted in food, although saccharin remained an acceptable food ingredient by virtue of the Artificial Sweeteners in Food Order, 1953. The old familiar saccharin tablet which contained 11·7 to 14·2 milligrammes of saccharin also remained in being until December when the Food Standards (Saccharin Tablets) Order, 1953 was revoked.

The new Regulations specify standards of purity for saccharin and cyclamic acid and for their sodium and calcium salts, all these being the "Permitted Artificial Sweeteners". They also prescribe the strengths of "Full Strength" and "Half Strength" tablets. If these contain saccharin sweeteners only, the full strength tablets must contain between 11 and 14 milligrammes calculated as the free acid, and half strength tablets must contain between 5½ and 7 milligrammes. If the sweetening tablets contain cyclamate sweeteners only, they

must contain between 183 and 233 milligrammes of cyclamates calculated as cyclamic acid in full strength tablets, and between 92 and 117 milligrammes in half strength tablets.

Where sweetening tablets contain a mixture of saccharin and cyclamate they shall contain not less than 1 milligramme of saccharin and not less than 15 milligrammes of cyclamate. Thus, a perfectly legal mixed tablet could be sold which had only about one-third the sweetening power of a half strength tablet.

Precise labelling is required. The last mentioned mixed tablet must be labelled "Cyclamate and saccharin tablets" or "Saccharin and cyclamate tablets" as appropriate (although it seems unlikely that a tablet containing 16 milligrammes of saccharin and 15 milligrammes of cyclamate would be made), and the other tablets must be named "Saccharin tablets" ("Saccharin Calcium tablets" or "Saccharin Sodium tablets") or "Half strength saccharin tablets" (or "saccharin calcium" (or sodium) as appropriate), or of course, "Cyclamate tablets", "Sodium cyclamate tablets" or "Calcium cyclamate tablets", or the appropriate designation preceeded by the words "Half Strength". Alternative words signifying the same thing as "tablets" are permitted, and mixtures of saccharin and its salts, or cyclamic acid and its salts are permitted.

The Preservatives in Food Regulations, 1962, are amended, as are the Lead in Food Regulations, 1961, to accommodate this new Statutory Instrument.

The Solvents in Food Regulations, 1967, were made in October 1967, and will operate from November 1969.

Solvents were defined as any liquid substances not being natural food substances and excluding water, which are capable of the extraction and dissolution of food and are generally used to facilitate the incorporation of ingredients in food.

The Regulations prohibit the sale and advertisement of non-permitted solvents for use in food, and prohibit the sale of foods containing any non-permitted solvents. They also regulate labelling of solvents when sold as such, so that the words "Food Solvent", together with a quantity-graded list of ingredients, shall appear legibly marked in letters at least $\frac{1}{8}$ of an inch high, within a surrounding line. The Regulations do not apply to solvents, or foods containing such solvents, intended for export.

The permitted solvents are ethanol, ethyl acetate, ether, glycerol, monoacetin, diacetin, triacetin, *iso*propanol and propylene glycol, of grades specified in a schedule to the Regulations, which was corrected early in 1968 by The Solvents in Food (Amendment) Regulations, 1967, on account of a printing error in the original formula given for triacetin.

It is interesting to note that newspaper articles appeared after the publication of these Regulations, referring to traces of hydrocarbons which might remain after decaffeination of coffee, and the Journal of the Association of Official Agricultural Chemists, 1967, p. 719, mentions hydrocarbon residues in solvent-

extracted oils. To judge from the Explanatory Note to the Regulations however, their concern may be principally with regulation of solvents which facilitate the incorporation of ingredients into food and therefore remain in it, as was indicated in the 1966 Report. Possibly the biggest surprise is the omission from the list of diethylene glycol mono-ethyl ether, for which a newly issued British Standard (B.S. 4117/1967) recommended a use in foodstuffs.

When certified by a Public Analyst as containing non-permitted solvent, the food becomes "Unfit" for the purposes of Section 9 of the Food and Drugs Act.

The Food (Control of Irradiation) Regulations, 1967, were made in March and operated from June. They took by surprise those who had not kept abreast of World Health Organisation Reports, for generally speaking, electromagnetic radiation has never been considered an important food contaminant, and strawberries, bacon and fish have all been successfully sterilised by subjecting them to gamma radiation of the order of 5 or 6 mega-rads. The Low Temperature Research Laboratories in Cambridge also announced that radiation levels of about one-tenth as much as was required for complete sterilisation would not affect flavour in fish and could be used to prolong cold storage life. Similar doses have been advocated for pest control, and smaller doses still, to inhibit sprouting in stored potatoes.

It appears, however, that certain very important food bacteria are quite difficult to kill by means of radiation and might grow more resistant. Preformed toxins from *Cl. botulinum* would not be destroyed either, so for the time being Statutory Instrument 1967, No. 385 prohibits the use of ionising radiation in the preparation of food intended for human consumption. It also prohibits the sale or importation of irradiated food. Low level radiation devices are, however, still allowed for in the Regulations, so that where these are used for controlling the quantity of food in packages or providing continuous watch on specific gravity, or similar parameters, they may continue in use.

Local Authorities and Port Health Authorities are called upon to enforce these Regulations, and at present, presumably, it will be done mainly by inspection. The development of means of detection of previous radiation by physical and chemical changes in the foods will require investigations into fundamental food science far beyond the financial capacity of local authority testing laboratories.

The Carcinogenic Substances Regulations, 1967, made in June and operating from December 1967, are of interest because they do NOT apply to Public Analysts' Laboratories.

The Regulations refer first to $\beta$-naphthylamine, benzidine, 4-amino-diphenyl and 4-nitro-diphenyl and prohibit the employment of operatives in factories (as defined) where they are made, or produced in other substances in amounts exceeding one per cent. These substances may not be taken into any such

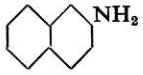
factory nor used in one. At the discretion of the Chief Inspector, however, benzidine hydrochlorides may be manufactured, or substances containing any of the above-named "Prohibited Substances" as impurity, provided that the operation is carried out in a totally enclosed system. The Chief Inspector may also give written permission for such substances to pass through docks, etc., for purposes of export. Manufacture or use could also be authorised by the Chief Inspector for scientific or medical research, investigation, or testing, provided that the director of the laboratory had authorised their use in writing.

The Regulations also control the employment of people in factories engaged in the making of α-naphthylamine, *o*-tolidine, dianisidine, dichlorbenzidine and its salts, auramine and magenta, and control their use. Provision is made for regular, six-monthly medical examinations of people so employed.

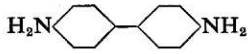
Industrial cancer was first investigated by Sir Percival Pott in 1775 and was probably first recognised as an environmental hazard after the investigation of Cornish copper smelters in 1822. Quite early in the history of the dye industry bladder cancer became associated with working with aniline, and later work showed this to be due to impurities, notably 4-amino-diphenyl. It would seem therefore that these Regulations have not made too early an appearance. The conditions for carcinogenicity in this class of compounds are said to be the presence of two aromatic rings having the position *para* to the amine blocked.

With this in mind, the structures of the controlled materials are worth noting again:—

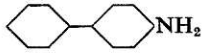
β-Naphthylamine



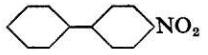

Benzidine



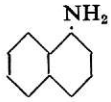

4-Amino-diphenyl



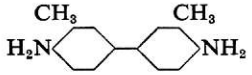

4-Nitro-diphenyl



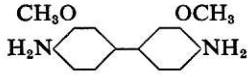

α-Naphthylamine



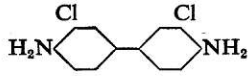

*o*-Tolidene




Dianisidine




Dichlorobenzidine




In common use in most laboratories are α-naphthylamine for Griess Ilosvay's reagent, benzidine for blood tests and for the determination of HCN, *o*-tolidine

for free chlorine, dianisidine for the determination of the antioxidant BHT, and magenta in Schiffs solution. It might therefore be prudent to note that the DPD method is available for chlorine, that Cleve's acid (a sulphonated naphthylamine) may be used for Ilosvay's reagent, luminol, or reduced phenolphthalein with hydrogen peroxide may be used to test for bloodstains, and *p*-toluidine or a slower barbituric acid method may be applied for HCN. Before one grows too neurotic about the possibilities inherent in these Regulations, it is as well to reflect that colour developers for colour-film processing are usually substituted *p*-phenylene diamines which enjoy at least as bad a reputation as the above substances, yet it is only for the amateur photographer that they are made fat insoluble and therefore less liable to be absorbed during handling, by the introduction of hydrophilic groups. This is an area of a Public Analyst's work where it will be necessary to practice good housekeeping. Nevertheless, it might be a happy day when the protective parts of the Factories Acts are made to apply to the small laboratory.

The Toys (Safety) Regulations, 1967, were made in July and became operative on 1st November, 1967. They provided immediately for the prohibition of toys which contained cellulose nitrate (except ping-pong balls) and those on which any paint film contained lead in excess of 1·1 per cent. After 31st October, 1968, the lead allowance will be reduced to not more than 0·5 per cent. of lead in the dry paint film, and at the same time arsenic in excess of 250 parts (as As) per million parts of dry paint film will be prohibited.

Also from 31st October, 1968, the Regulations will prohibit on toys paint which will yield antimony, barium, cadmium or chromium in excess of 250 parts of the element in a million parts of the dry paint film, when the detached paint, reduced in size to pass a B.S. 30 mesh sieve, is shaken for one hour and then allowed to stand for a further hour in contact with 0·25 per cent. hydrochloric acid (approx. 0·066N). A schedule to the Regulations specifies the method of bringing the metals into solution, and seems to imply that the 1963 amendment to the British Standard Specification for sieves affects the sieve specified in the Regulations, whereas, in fact, it does not.

The Regulations are quite specific in establishing that they apply to toys and parts manufactured before the Regulations became operative. This seems rather odd in view of the fact that toy fairs are usually held in January and February, and that buyers would already have completed their contracts before these Regulations were even made. It is perhaps unfortunate, in view of this, that certain hysterical headlines appeared in the pre-Christmas Press about "lethal" toys.

A Home Office Circular issued with the Regulations made the assumption that the tests required would be carried out by Public Analysts, and also drew attention to the parts of the Rag Flock Act which deal with toy fillings. A forthcoming Code of Safety for electrical toys was also forecast.

Four Statutory Instruments were laid before Parliament on 21st December, 1967, and greeted us in 1968 as the last legislation of 1967. The first of these

was The Labelling of Food Regulations, 1967, which comes into operation as far as the newly-permitted cyclamate additives were concerned, on 1st January, 1968, but in all other respects is not operative until January 1971. These Regulations seem rather better than the 1965 "Proposals", although, as with every metamorphosis from Proposals to Regulations, they include their surprises. Some of these are among the definitions. For example, after accepting a mere confining band in several recent Regulations as a "Container", one accepts with reserve that a "crimp case" is not a container. It seems strange that "biscuits" are not "flour confectionery" and are separately defined, and that after so long the question of whether "Rum Butter" is or is not "Sugar Confectionery" remains as it was. The definition of "intoxicating liquor" firmly avoids an old difficulty which sometimes arose with Black Beer, but "Main Ingredient" is surprisingly defined. "Meat", though similarly defined as in the Canned Meat Product Regulations does not specifically mention Tripe, and "Appropriate Designation" appears to put us back with over-specific names like cyamopsis tetragonolobus for half-familiar materials like Guar. One welcomes the clarification of fish names and the settling of the doubts one once entertained about the Syrup in "Canned-fruit-in-syrup". Certain other trivial provisions seem sensible; thus a period of use for existing embossed drink bottles, alphabetical listing of fruit or vegetables present in approximately equal mixtures, and non-declaration of traces of permitted food preservative or antioxidant introduced with ingredients will cause no difficulty; although, reasonably, where permitted additives form part of an ingredient and are present in substantial amount, their presence must be indicated, usually within a surrounding line and in lettering of certain minimum sizes.

A real effort has been made to ensure that partial lists of ingredients no longer appear and this even operates to prevent selective declarations of additives which are present. If a label is too small to take all the necessary information, only the name must be given. The old exemption from labelling, given to food prepacked on the premises from which it is sold, now also applies if the food is sold from a van operating from those premises.

Registered Trade Marks will not be allowed to take the place of a name and address (except in a special proviso for soft drinks, which have a five year period of grace).

Mixed food sold loose should be labelled with a name or appropriate designation, and drink, bread, butter, biscuits, cheese, confectionery, flour, fish and ice-cream sold loose do not need a declaration of additives. Food sold from vending machines must either be properly labelled with labels visible from outside the machine or have the appropriate designation of the food marked clearly on the front of the machine.

Intoxicating liquors must be labelled with the name and address of whoever takes responsibility for them, a designation which indicates the geographical origin (subject, however, to there being no misleading geographical implication in this) and in the case of wine-like products there must be a statement of the

fruit basis, or absence of fruit basis of the drink, if this is not grape juice. Brandy, gin, rum, vodka and whisky may now be sold understrength if a statement to that effect is given due prominence on the label. There must also be a statement of alcoholic strength (expressed either as Percentage Proof, or Percentage Alcohol by Volume). Apart from the designation and the bottler's name and address, all the above information must be given in standard forms and sizes within an enclosing line, but for brandy, gin, rum, vodka or whisky which is above the strength required in the Food and Drugs Act, it will be sufficient declaration of strength if the present and familiar statement, like "70° PROOF" appears. The same form is acceptable for other liquors with strengths greater than "40° PROOF".

"Wine" which is wholly or partially derived from saccharine material other than grapes, must be suitably described, in uniform type. Cider and Perry seem to be allowed the adjective "Champagne" if made to sparkle by secondary fermentation, and another victory to the cider industry appears to be an acquiescence in a misuse of the word "vintage" in phrases like "made from vintage apples" (whatever that may mean).

The labelling of processed peas and acetic acid remains very much as in the 1953 Order. In addition, meat treated with proteolytic enzymes must be marked "tenderised" and the word "milk" must refer to whole cow's milk unless otherwise specified.

For the most part all these points are mere dotting of "i"s and crossing of "t"s in the 1953 Order, but one important difference deals with the size of lettering on labels. Both the principal words and those showing ingredients will have to be of certain minimum sizes depending on the size of the container, the ingredients' lettering in general evidently being expected to be half the size of the major lettering. There are also limitations on the proportional differences between the sizes of the smallest and the largest letters. In the principal lettering, in general, small letters must be at least a quarter the height of the tallest letters appearing on the label, and at least half the height of other letters in the designation. Soft drinks in embossed bottles may carry the necessary particulars on the bottle closure in letters at least 1 mm high. Similar specifications are made with regard to the labels for goods sold loose. Although this part of the Regulations seems a little involved, it does clarify the labelling part of the Cheese (Amendment) Regulations which appeared in 1966. As there written, a cheese label like "BRANDEX Processed Cheddar Cheese with Butter" required only that the smallest rectangle capable of enclosing, say, the small "h" should be at least nine-sixteenths of the smallest rectangle capable of enclosing the "X" in "BRANDEX", but provided that it was legible no lower limit was set on small type size, so that micro dot lettering appeared to be in order.

The Coffee Product Regulations will come into operation in 1971 and they gather together standards which formerly appeared in the Liquid Coffee

Essences Order, The Coffee Mixtures Order and The Labelling of Food Order. In addition, standards are made for Dried Extracts of Coffee and of Coffee and Chicory, although these do not have any minimum caffeine requirements. A maximum limit of 100 parts per million for matter not derived from the coffee or coffee and chicory will probably remove from the market certain products which have contained added glucose syrup solids. A maximum limit of 0·1 per cent. of caffeine is imposed for decaffeinated coffee, and a caffeine standard of at least 0·4 per cent. of caffeine will now apply to the new Liquid Extract of Coffee and Fig. Precise designations for the various coffee products are stipulated.

The Ice-Cream Regulations, 1967, will also come into force in 1971, and these restate the existing compositional standards for ice-cream, and they also provide for the proper labelling of the various qualities available.

The Margarine Regulations, coming into operation at the same time, make a slight alteration to the way in which the former prohibition on the presence of more than 10 per cent. of butter in margarine is stated. This always was a fixed maximum, although a 2 per cent. tolerance in effect meant that one could expect from 8 to 10 per cent. butter to be present in a mixed product. The new Regulations require not less than 80 per cent. of fat to be present, of which not more than one-tenth (by weight) may be fat derived from milk. A little arithmetic will reveal that this is a restatement of the former standard. The long established 16 per cent. maximum moisture content and the existing standards for vitamins remain unaltered.

Much of the legislation connected with the sale of Margarine arises from its sales promotion as a substitute for butter. The new regulations place restrictions on the use and size of the words "milk", "cream" and "butter" when used in association with margarine—restrictions which would probably be unnecessary if margarine were sold coloured blue and flavoured with fruit or peppermint—and it is required that in pictorial or audible advertising the word "Margarine" shall be used without its being obscured by brand names, etc.

The former requirement about a minimum size for the word margarine on wrappers has given place to the general requirements for food labelling.

Two reports were published in June 1967. One was a Food Standards Committee Report on Cream and the other was a Food Additives and Contaminants Committee Report, being the Second Report on Cyclamates.

The Report on Cream takes cognisance of the modern methods of making cream, which render it possible at last to apply effective Heat Treatments and effective Regulations to govern these. It is also recognised that there is a potential market for thin cream, for use in coffee, and for a cream which will whip to give an overrun of approximately 100 per cent. The Committee therefore recommends that the following names and minimum fat contents should be introduced.

| | |
|---|---|
| Clotted Cream | 48 per cent. |
| Double Cream | 48 per cent. |
| Whipping or Whipped Cream | 35 per cent. |
| Sterilised Cream | 23 per cent. |
| Cream | 20 per cent. |
| Sterilised Half Cream | 12 per cent. |
| Half Cream | 12 per cent. |

They are also prepared to accept that sugar may be present in cream used in flour confectionery and in whipped cream, up to $1\frac{1}{2}$ lb per gallon, provided that its presence is declared. They would also allow up to 0·3 per cent. by weight of sodium alginate or carboxymethyl cellulose in whipped cream, and they consider that up to 0·2 per cent. by weight of sodium or potassium carbonate, citric acid and orthophosphoric acid and calcium chloride should be permitted in sterilised cream provided that the presence of these materials is also declared.

The Second Report on Cyclamates was published at almost the same time as the Artificial Sweeteners in Food Regulations were issued. Its purpose was to confirm that there was no risk to health in allowing the use of cyclamates in food. The Committee recommended, however, that a break-down product, cyclohexylamine, should be further examined from a toxicological point of view.

The first feeling that one had, when the "Proposals for Regulations to deal with Claims and Misleading Descriptions on Labels and Advertisements of Food" were published in November, was that the considered recommendations of an expert committee were being set aside. The second feeling was that it might be better now to set aside the Proposals. The Report, issued in 1966, was the second half of a two part report on food labelling of which the first part was the Report on labelling itself, which came out in 1964. The 1966 Report on claims, etc., was a booklet running to about 20,000 words, in comparison with which the Proposals occupy a mere 4,500 or so words.

The Report expressed the view that regulations were needed to clarify doubts about what constituted misleading claims and to ensure that the consumer was given adequate information in respect of claims affecting health. Under the former heading there were eleven pages making recommendations about:—

1. Pictorial devices
2. Designatory phrases used in food names
3. Adjectives
   (i) "natural"
   (ii) "pure"
   (iii) "home made"
   (iv) "made of" and "made from"
   (v) words of foreign origin, etc.
   (vi) "butter"
   (vii) "cream" and "creamed"
   (viii) "digestive"

4. Dried foods
5. Cake and Sauce Mix Powders
6. Meat products with names like "steak", "cutlet", "chop" and "fillet", and those with derivatives of such names such as "steakette"
7. "Burgers" and "Fritters"
8. Vinegar
9. Shandy
10. Mustard and Cress.

In the proposals we do find some attempt to limit the more flamboyant use of pictures, and it is specified that the word "flavour" should follow designations which seem to name a contributory food which has not in fact been used in the food, *e.g.* Raspberry flavour Jelly. Of the Report's eight offending adjectives only two are dealt with, these being "Butter" and "Home made". The somewhat unsatisfactory code of practice which required a minimum of 4 per cent. of butter fat irrespective of the presence of other fat to be present in sugar confectionery or chocolate confectionery which bears the adjective "Butter", has been re-stated in the Proposals without any mention of the use of the word in flour products. The proposals also require dried foods to be named "dried", "desiccated" or "dehydrated", and make provision for the buyer to be notified when dry cake mixes, etc., are incomplete. The specific names of different grades of vinegar are sorted out, and "Shandy" must contain at least $1\frac{1}{2}$ per cent. of proof spirit. Almost everything else in the Report has been abandoned. Some very muddled thinking has appeared concerning liqueur chocolates. If the Proposals stand, an infinitesimal amount of spiritous liquor will qualify those chocolates for the name.

On the Health side, the Energy recommendations made in the Report have been so watered down in the Proposals that it has been remarked that provided the calorie content is declared, even so humble an energy provider as a saccharin tablet will qualify as a source of energy. A claim for high protein content can be made when 13 per cent. is present (instead of the recommended 20 per cent.), if the calorie contribution of a food comes from protein. Although claims relating to obesity and slimming are dealt with along the lines recommended in the Report, in simplifying the provisions of the Report an odd feature has been introduced into the Proposals. Almost anything may be sold as a slimmer's food, provided the calorie content is stated, advice to consult a doctor when slimming is given, a list of ingredients is given, and the only actual claim made is that to eat the product will relieve any feeling of hunger felt by a patient on slimming diet. Such material is indeed already on the market. It has a normal calorie content for the kind of article in question.

The proposals make vitamin claims more confusing than they are at present, by allowing a twofold way of declaring them instead of the current single way, and we shall have to wait to see whether the old Code of Practice will still be

observed, in which no claim about vitamin content appeared unless 1/6th of the normal daily requirement could reasonably be contributed by the food.

Claims that foods had tonic properties were frowned on by the Report, but in the Proposals the use of the word "Tonic" is merely regulated and not prohibited. The Report also recommended that no special benefits should be promised to heart sufferers who consumed only certain dietary fats. This has been omitted from the Proposals.

In some respects one can appreciate that to adopt all the recommendations of the Report would have been tantamount to making life very colourless. "Hope" is an important ingredient in much that we consume, and the Ministry has evidently conceded that a man is not necessarily misled when he encounters it. One can also well understand that a recommendation to change the name "Mustard and Cress" to "Rape and Cress" should have gone into limbo. If the recommendations of the Food Standards Committee were as impractical as these Proposals suggest, however, it might have been better if an earlier recommendation had been resuscitated, and that each case be dealt with on its own merits.

Proposals for the amendment of the Skimmed Milk with Non-milk Fat Regulations, 1960, set out to extend to three manufacturers (Trufood, B.D.H. and John Wyeth) an exemption from the requirement to label certain of their products "Unfit for Babies" or "Not to be used for Babies" which was extended to certain other manufacturers in 1966. Specification for the said products would be added to Part II of the Second Schedule to the regulations.

In January 1967, a Press Notice was issued jointly by the Department of Education and Science, the Ministry of Agriculture, Fisheries and Food, the Ministry of Health, and the Home Office about Toxic Chemicals in Agriculture and Food Storage, and about Aldrin and Dieldrin residues in Food.

Two reports were being published: one, by the Advisory Committee on Pesticides and Other Toxic Chemicals, reviewed safety arrangements and recommended that a compulsory licensing scheme for pesticide products should replace the existing Safety Precautions Schemes. It further recommended that it should be made an offence to misuse certain pesticide and veterinary products in specified ways. It was felt that more data should be collected before residue limits for foodstuffs are established in Great Britain.

The second report, prepared by the Food Additives and Contaminants Committee, considered the specific question of aldrin and dieldrin residues in food. They recommended that immediate steps should be taken to obtain information about amounts of these chemicals already present in foods, and that Statutory limits should be laid down for their residues in foods sold in the United Kingdom. It was proposed that these should be: mutton (1 p.p.m.), general foods (0·1 p.p.m.), Baby food (including dried milk) (0·02 p.p.m.) and liquid milk (0·003 p.p.m.).

An official publication which contained much of potential interest for the Public Analyst service was the white paper, "Forthcoming Legislation on the Safety, Quality and Description of Drugs and Medicines", which was presented to Parliament in September 1967. Proposals for legislation were outlined which followed those of France, Germany, Holland, Canada and other Commonwealth countries, and was broadly comparable with what is thought likely to be adopted by the European Economic Community. The legislation will cover drugs for human and animal use.

The general framework for control will be provided by a licensing system for drugs, licences being needed for marketing, importing, manufacturing or wholesaling. Products for clinical trials etc. will need a certificate to ensure that toxicity testing has taken place before the beginning of the trials. Particulars to be supplied to the licensing authority are likely to be similar to those now requested by the Dunlop Committee on Safety of Drugs, and by the Advisory Committee on Pesticides and Toxic Chemicals; and an appeals system will be devised for applicants and licence holders who are dissatisfied with decisions made by the authority. The licensing authority will also be given power by Statutory Instruments to control toiletries, cosmetics, disinfectants etc.

A medicines commission will be set up to advise the Ministers of Health and Agriculture. It will also recommend the pattern of expert committees, consider representations from applicants for licences and from licence holders, and will arrange for the preparation of the British Pharmacopoeia. Paragraph 77 somewhat enigmatically refers to the provision of compensation for loss or diminution of emoluments to local government officers, and to employees of the General Medical Council engaged on the British Pharmacopoeia.

Labels will be required to give specified information depending on the product and circumstances, and labels bearing false or misleading information will be prohibited. Provision will be made for the licensing authority to stipulate types of containers to be used, or the materials or colours used on them. Ministers will be able to specify drugs for which advertising will not be permitted and they could also regulate content, format, and manner of presentation of advertisements. Brand names will be allowed due prominence but direct mailing to doctors will be restricted.

*The Farm and Garden Chemicals Act*, 1967.

The above Act, which was effective as from 14th July, 1967, is a permissive Act allowing the Minister of Agriculture Food and Fisheries, jointly with the Secretary of State, to make Regulations from October 1968 regarding the labelling and marking with a suitable colour code indicating the toxic hazard, of any pesticide or animal repellents, weed killers, growth regulators, defoliants, desiccants or thinning agents.

The usual defences regarding due diligence, or default of other persons are

provided. The prosecutor (it is not stated who will administer the Regulations) must give the defendant a sample in time for him to have it analysed.

A document purporting to be a certificate issued by an analyst possessing the requisite qualification for appointment as a Public Analyst under section 89 of the Food and Drugs Act, 1955, shall be admissible as evidence of matters stated therein, provided that a copy has been given to the defendant together with a suitably-sized sample of the product, and due notice is given.

The court may, on the request of either party, send a portion of the sample to the Government Chemist.

Although a review of this kind might not ordinarily include comment on rules of evidence, it might prove helpful to mention the Criminal Justices Act, 1967, Part I, Paragraph 9, and Statutory Instrument, 1967, No. 1661, L. 13 (Magistrates Courts Rules) (Paragraph 7). These two documents materially alter procedure at Court, and in future it may become a very rare event for an Analyst actually to appear in person to give evidence. There is just one question which seems at present to be unanswered and this concerns the production of the sample. It seems unlikely that perishable or inconvenient samples should be expected to become part of legal files, but perhaps one may have to think in terms of photographing many "Complaint" samples in order to be able to present visual evidence along with the written evidence.

Finally, a draft Statutory Instrument was later followed by S.I., 1967, No. 1901 (The Merchandise Marks (Imported Goods) No. 7 Order, 1934, Amendment Order, 1967), which allowed marking of certain imported edible offals to be in letters of reduced size, as from March 1968.

# The Determination of Dimexan in Crops

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Residues of Dimexan on vegetables and other crops can be determined by treating the sample with ethanolic sodium hydrosulphide, then boiling with dilute sulphuric acid and absorbing the $CS_2$ evolved in a modified Viles' reagent. The colour developed is measured spectro-photometrically. With onions, high results were obtained in recovery tests unless the onions were fresh and examined at once.

The substance known as Dimexan in Gt. Britain (Dimexano in Europe) is bis-(methyl-xanthic) disulphide, $CH_3O–\overset{\overset{\|}{S}}{C}–S–S–\overset{\overset{\|}{S}}{C}–OCH_3$. It is used as a haulm-killer and defoliant.

At ordinary summer temperatures in Britain it is a fairly volatile oil. The crops referred to in this report were sprayed with the proprietary preparation "Tri-P.E.", consisting of an emulsifiable concentrate containing about 40 per cent. of dimexan, and made by Fabriek van Chemische Producten, Vondelingenplaat N.V., of Rotterdam.

The method employed for the determination of Dimexan is an adaptation of a method, first published by Clark[1] and improved by Cullen[2], for the determination of residues of dithiocarbamates. These substances yield carbon disulphide ($CS_2$), on treatment with acids, whereas Dimexan requires preliminary treatment with ethanolic sodium hydrosulphide. The reactions are as follows—

$$CH_3O{\cdot}CS{\cdot}S_2{\cdot}CS{\cdot}OCH_3 + 2NaSH \rightarrow 2CH_3O{\cdot}CS{\cdot}SNa + H_2S + S$$
$$CH_3O{\cdot}CS{\cdot}SNa + H^+ \rightarrow CH_3OH + Na^+ + CS_2$$

A stream of nitrogen is passed through the apparatus and carries with it the evolved gases, first through a solution of zinc acetate to remove the $H_2S$ and then into a modification of Viles' reagent[3]—cupric acetate and diethanolamine in ethanol—which reacts with the $CS_2$ to form yellow chelates[2]. The optical density of the solution is measured spectro-photometrically. This method was first devised in the Vondelingenplaat Laboratories, but various improvements were made in the Norwich laboratory and all the analyses referred to in this paper were made in Norwich.

### *Apparatus*

This is shown in Figure 1. A litre flask with three necks has a condenser fitted vertically to the middle neck, the two side necks being used for the

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admission of nitrogen and for the addition of liquids *via* a dropping funnel fitted with a stop-cock. From the top of the condenser, a tube leads the issuing gases through two gas-washing tubes (which absorb the $H_2S$ present) and then through a sintered glass gas distributor (porosity 2) in the final absorption tube which contains the modified Viles' reagent. Each absorption tube to be used in this position is calibrated by measuring into it 5·0 ml of water with an accurate pipette and marking the level reached by the water. It is important that the internal diameter of this tube should be only 2–3 mm more than that of the gas distribution tube (the authors' pattern has a maximum external diameter of 13 mm), so that the latter is immersed in as great a depth as possible when 5 ml of the modified Viles' reagent is introduced. To use a larger volume of reagent would result in a loss of sensitivity, and for the same reason the reagent is not diluted before determining the optical density, as is done in Cullen's method[2]. The volume of 5 ml (less drainings) is ample for use in a 10-mm spectrophotometer cell. All joints are standard glass cones and sockets or spherical joints, introduced to obtain some degree of flexibility.

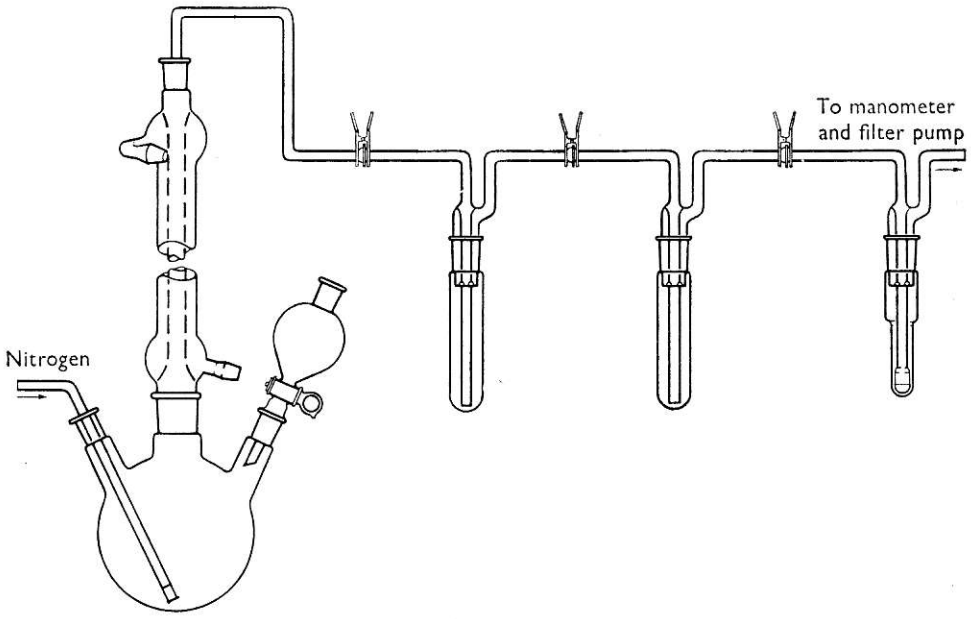


Fig. 1

### *Reagents*

1. *Industrial Methylated Spirit:* I.M.S., 74 degrees o.p.
2. *Sodium Hydrosulphide Solution:* 5 per cent. in I.M.S. Dissolve 6·6 g of sodium hydrosulphide, $NaHS.H_2O$, (Fluka A.G. Buchs, Switzerland) in I.M.S., filter and make up to 100 ml. (Alternatively, dissolve 22 g of sodium sulphide, $Na_2S.9H_2O$, in a minimum amount of water in a 100-ml

volumetric flask. Add I.M.S. to a total volume of about 80 ml and cool nearly to 0° C in ice. Add, gradually and with constant swirling, 17·8 ml of cooled, 5 N hydrochloric acid solution. Allow the mixture to come to room temperature, make up to 100 ml with I.M.S. and mix well. A precipitate may form; always use the clear solution and discard it after one week.)

3. *Zinc Acetate Solution:* 10 per cent. w/v.
4. *Sulphuric Acid:* 5 N $H_2SO_4$ soln.
5. *Viles' Reagent (Modified):* Dissolve 10 mg of cupric acetate monohydrate and 25 g of diethanolamine in I.M.S. and dilute with I.M.S. to 250 ml. 5 ml of this reagent are suitable for the absorption of at least 200 $\mu$g of $CS_2$, which is equivalent to about 280 $\mu$g of Dimexan, or 2·8 p.p.m. on a 100-g sample.

***Method***

Introduce into the three-necked flask a weighed sample (usually 100 g) of the plant material to be examined. Add 50 ml of I.M.S. and 2 ml of sodium hydrosulphide solution; swirl for one minute, connect the flask to the condenser and allow to stand for 15 minutes. Into each $H_2S$ trap introduce 12 ml of zinc acetate solution, and pipette 5·0 ml of Viles' reagent into the final absorption tube. With the apparatus completely assembled, turn on the nitrogen and apply slight suction from the vacuum pump, adjusting both so that with a reasonable flow of nitrogen as shown by the bubbles in the traps, a slight negative pressure is shown on a manometer, to reduce risk of loss of gas (and therefore of $CS_2$) at the spherical joints. Add, from the dropping funnel, 50 ml of water followed by 50 ml of 5 N sulphuric acid, and after nitrogen has been passing for three to four minutes, heat the contents of the flask to boiling point and boil for 30 minutes. The flow of nitrogen must be adjusted so that frothing in the first $H_2S$ trap is controlled; the rate of bubbling may have to be restricted to 2 to 3 bubbles per second until boiling begins but can be increased towards the end of the boiling period, when the frothing has died down, in order to ensure that all $CS_2$ has been carried through to the final absorption tube. The contents of the second $H_2S$ trap should remain clear throughout the determination, indicating that all $H_2S$ has been absorbed in the first trap.

At the end of the 30-minute boiling period, disconnect the final absorption tube and rinse the gas distributor into it with the few drops of I.M.S. necessary to restore the volume of the contents to the 5-ml mark. Mix, and allow to stand for 15 minutes. (Disconnect the $H_2S$ traps and open the top of the dropping funnel before turning off the flame and the nitrogen.) Determine the optical density of the contents of the absorption tube in a 1-cm cell at 435 m$\mu$, with Viles' reagent (as modified) in the reference cell.

NOTES

1. Dimexan residues will be present on, rather than in, the crop, and as with the dithiocarbamates, there is danger of decomposition if the crop is cut or mashed and the dimexan comes into contact with water, enzymes, etc. before the I.M.S. and the sodium hydrosulphide are added. For this reason the crop is handled as little as possible; peas are shelled and weighed out whole, beetroot is cut up into cubes small enough to enter the neck of the reaction flask but no smaller, and so on.
2. Zinc (or cadmium) acetate solution is better than lead acetate solution in the $H_2S$ traps because the tubes are easier to clean afterwards, the precipitated sulphide dissolving readily in hydrochloric acid.
3. The volume of sodium hydrosulphide solution used (2 ml) is theoretically in large excess of the amount required to react with any amount of Dimexan likely to be met with; 1 ml would probably be sufficient. If more than 2 ml are used, zinc sulphide may appear in the second $H_2S$ trap, destroying the analyst's conviction that no $H_2S$ is reaching the Viles' reagent.

### *Calibration Graph*

A calibration graph is constructed for each crop to be examined, by adding to 100-g portions of the crop (free of Dimexan) known amounts of standard Dimexan solution from zero upwards. It was found convenient to use 0, 25, 50, 75 and 100 $\mu$g amounts of Dimexan solution, corresponding to 0, 0·25, 0·5, 0·75 and 1·0 p.p.m. respectively. Under the conditions described, using a Unicam SP 500 spectrophotometer, the optical density corresponding to 100 $\mu$g of Dimexan was 0·66. The "blank" using 100 g of untreated material, was satisfactorily low; for example, with peas the optical density was 0·02 to 0·04. It was found that the relation between weight of Dimexan and optical density was linear up to 100 $\mu$g, the highest amount which was to be determined.

### *Abnormal Results*

Analyses of shelled peas and of red beet at intervals of one to seven days after spraying were made, using as control samples, peas and beet from parts of the same field that had been left unsprayed. The results were in accordance with expectation and exhibited no unusual features; determinations on triplicate samples from different parts of the sprayed area on any one day were in satisfactory agreement with each other and the "blanks" on the control samples were low (optical density 0·02 to 0·04). When known amounts of Dimexan were added to portions of sprayed peas, the Dimexan content of which had already been determined, the recovery of the added Dimexan was 95 per cent. or better.

Analyses of onions, however, gave unusual results. Whereas red beet, two days after spraying, had been found to contain less than 0·5 p.p.m. of Dimexan,

onions two days after spraying were found to contain 1·3 to 1·8 p.p.m. The "blanks" on control samples were low (optical density 0·03 to 0·05) but when recovery tests were made in duplicate two days later, adding 50 $\mu$g of Dimexan to control samples, recoveries of 77 and 75 $\mu$g were unexpectedly obtained.

This clearly required to be looked into further; the control onions had been chopped up 2 days before the recovery tests were made and might have decomposed with formation either of $CS_2$ or of some compound that behaved similarly towards Viles' reagent. Accordingly, some unsprayed onions that had been lying in the field for 7 days were obtained and chopped up; blanks and recovery tests were made immediately. The blank was a little higher than before (optical density 0·065) and the apparent recovery was 114 per cent.

Three pounds of onions were then purchased from a local shop. They were "topped and tailed", then chopped up into cubes as before. Three Kilner jars were filled and kept under various conditions (see below); "blank" and recovery tests were then made. The results are shown in the following table:

TABLE I

APPARENT RECOVERY OF DIMEXAN FROM ONION SAMPLES

| | Sample | | Sample plus 50 $\mu$g of Dimexan | | |
|---|---|---|---|---|---|
| | Date of Testing | Optical Density | Date of Testing | Optical Density | Recovery *per cent.* |
| Examined on day of preparation .. .. .. | 14 Sept. | 0·027 | 14 Sept. | 0·370 | 101 |
| Jar kept in deep-freeze for 4–5 days .. .. .. | 18 Sept. | 0·059 | 19 Sept. | 0·415 | 114 |
| Jar kept at room temperature for 4–5 days .. | 18 Sept. | 0·029 | 19 Sept. | 0·432 | 119 |
| Mean control optical density | | 0·038 | | | |

Although the variability in the "blanks" as shown by the figures in Table I is rather large and prevents any firm conclusion from being drawn, the matter seems to warrant further investigation. If onions are being examined for Dimexan, both the sprayed and the unsprayed samples should be examined on the day on which they are taken.

**References**

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# A Survey of Fruit and Vegetables for Dithiocarbamate Residues

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Results of a small survey of dithiocarbamate fungicide residues in fruits and vegetables, sampled in the Autumn of 1967, are given, together with details of a TLC method used to distinguish four common dithiocarbamates.

The dithiocarbamate group of fungicides is being used extensively in this country. They are recommended for use in the control of certain fungal diseases on fruits and vegetables[1] as listed in Table I.

TABLE I
AGRICULTURAL APPLICATIONS OF DITHIOCARBAMATE FUNGICIDES

| Fruit or Vegetable | Fungal Disease | Dithiocarbamate used (M – Maneb; T – Thiram; Z – Zineb) |
|---|---|---|
| Apple | Scab, Rust | T, Z |
| Blackcurrant | Leaf spot, Rust | T, Z |
| Celery | Leaf spot | Z |
| Grape | Downy mildew | Z |
| Hop | Downy mildew | Z |
| Lettuce | Botrytis | T |
| Mushroom | Dactylium, Mycogone | Z |
| Onion | Botrytis, Downy mildew | T, Z |
| Pear | Scab | T, Z |
| Potato | Blight | M, Z |
| Raspberry | Cane spot | T |
| Spinach | Downy mildew | Z |
| Strawberry | Botrytis | T |
| Tomato | Blight | M, Z |
| | Didymella stem rot | M |
| | Leaf mould | M, Z |
| Wheat | Rust | Z |

At present, there is no legislation in this country laying down maximum limits for fungicide residues on crops. Maximum limits for residues of certain dithiocarbamates have been fixed in Canada and the United States. These are of the order of up to 20 p.p.m. Several crops are controlled in this way, including celery, green beans, brocolli, rhubarb, lettuce and apricot. The Netherlands limits are 7 p.p.m. for zineb and ziram, 3 p.p.m. for thiram and 10 p.p.m. for maneb.

During the Autumn of 1967, a selection of fruits and vegetables were analysed for dithiocarbamate residues, *viz.* apple, celery, lettuce, mushroom, pear and tomato, to discover primarily if any residues were present, and secondly to determine the approximate concentration ranges of these residues.

* Present Address: Imperial Tobacco Company, Ltd., Bristol.

### *Apparatus*

1. Distillation apparatus as described by Cullen[2].
2. TLC apparatus (Shandon).
3. Spectrophotometer (Unicam SP 600).

### *Reagents*

1. *Industrial Methylated Spirit:* I.M.S., 74° o.p.
2. *Sulphuric Acid Solution:* 10 N $H_2SO_4$ soln.
3. *Chloroform:* Of AR quality.
4. *Pyridine:* Of AR quality.
5. *Iodine:* Of AR quality.
6. *Sodium Azide:* A 3 per cent. solution in water[3].
7. *Zinc Acetate Dihydrate:* A 1·5 per cent. solution of analytical grade reagent in water.
8. *Cupric Acetate Reagent:* To 4 mg of cupric acetate monohydrate, (AR), add 25 g of diethanolamine (laboratory grade) and dilute the mixture to 250 ml with I.M.S. Prepare freshly every day.
9. *Standard Solutions of Dithiocarbamate Fungicides:* 0·01 M solutions of individual fungicides in pyridines.

### *Method*

QUALITATIVE

Chop the whole of the sample submitted, and weigh 50 g of material (in the case of apples and pears, take a representative 20-g sample of peel only). Place the sample in a separating funnel, add 25 ml of pyridine (10 ml for peel) and shake the mixture for one minute. Shake a further three times at twenty-minute intervals and filter the pyridine extract through a No. 1 Whatman filter paper. Transfer separate amounts of 0·5 and 2·5 microlitres of the filtrate to a layer of Kieselgel G, 250 $\mu$ thick. Also apply 1 microlitre of each of the thiocarbamate standard solutions as separate spots on the same plate.

Develop the chromatogram with chloroform for a distance of 10 cm; dry the plate and spray the layer with sodium azide reagent. Place the plate in an atmosphere of iodine until the grey-brown spots develop. This is a simplified version of the method of McKinley and Magarvey[3].

Table II lists the $R_f$ values of four dithiocarbamates.

TABLE II

THIN LAYER CHROMATOGRAPHY OF CERTAIN DITHIOCARBAMATE FUNGICIDES

| Dithiocarbamate | Common Name | $R_f$ Value |
|---|---|---|
| Zinc ethylene-bis-dithiocarbamate .. .. | Zineb | 0·30 |
| Tetramethyl thiuram disulphide .. .. | Thiram | 0·50 |
| Zinc dimethyl-dithiocarbamate .. .. | Ziram | 0·70 |
| Manganese ethylene-bis-dithiocarbamate .. | Maneb | 0·80 |

QUANTITATIVE

Use the method of Cullen[2], which is an adaptation of the original method of Clark[4].

This method depends on the hydrolysis of the dithiocarbamate with sulphuric acid to yield carbon disulphide which is passed through zinc acetate solution to remove any hydrogen sulphide present and thence into the cupric acetate reagent (10 ml) to form the yellow cupric N:N-bis(2-hydroxyethyl) dithiocarbamate.

It is essential to include a second trap containing 10 ml of cupric acetate reagent, because some samples may yield excessive carbon disulphide and the first trap may become saturated. If this should occur, a smaller sample must be taken. Measure the extinction of the solution in the first trap within 10 minutes of disconnecting it, using a 1-cm cell and a wavelength of 435 m$\mu$.

Prepare standard curves for zineb, thiram, maneb and ziram using the concentration ranges suggested by Cullen[2], equivalent to a concentration of up to 200 $\mu$g of carbon disulphide.

In practice, it is quicker to carry out the quantitative determination first and refer only the positive results to TLC for identification.

### *Results and Discussion*

A total of 155 samples has been analysed and the results are given in Table III. Eighty-seven samples contained zineb and one sample contained thiram. Zineb appears to be the most common dithiocarbamate in use, although thiram is used on strawberry, blackcurrant, raspberry and onion, which have yet to be examined.

TABLE III

THE OCCURRENCE OF DITHIOCARBAMATE FUNGICIDES ON FRUIT AND VEGETABLES

| Fruit or Vegetable | Number examined | Number of samples containing zineb | | |
|---|---|---|---|---|
| | | Range 0–5 p.p.m. | Range 5–10 p.p.m. | Range >10 p.p.m. |
| Apple .. .. | 24 | 2 | 1 | 0 |
| Celery .. .. | 7 | 0 | 0 | 0 |
| Lettuce .. .. | 18 | 0 | 1* | 0 |
| Mushroom .. .. | 56 | 24 | 6 | 10 |
| Pear .. .. | 26 | 8 | 8 | 4 |
| Tomato .. .. | 24 | 22 | 1 | 1 |

* Contained thiram only.

Whilst the incidence of dithiocarbamate in apple, celery and lettuce is small, most samples of mushroom, pear and tomato had retained a residue. Apple and pear samples yielded comparatively small amounts of zineb in contrast to mushroom and tomato, where results were as high as 16 and 11 p.p.m. respectively. Twelve samples of mushroom and one sample of tomato exceeded 7 p.p.m. of zineb. The lettuce sample containing 10 p.p.m. of thiram

would be condemned as excessively contaminated under some foreign regulations.

The mushroom samples were English and experiments have shown that most zineb is present in the cap peel[5]. The tomatoes were Spanish or Canary Island in origin and most of the pears were either English or Italian.

Neither maneb nor ziram residues have yet been found.

The Author thanks Robinson Bros. Ltd. and Mi-dox Ltd. for supplying the dithiocarbamate compounds, Miss K. Evason for assistance and Mr. E. G. Whittle, the Scientific Adviser, for permission to publish this paper.

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# A Note on the Detection of Pentachlorophenol in Water

*by* B. J. SANDERS and A. R. PHILLIPS

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A recent examination was made of water from a gold-fish pool in which most of the fish had died suddenly. The pool had been loosely covered with wooden boards for two days for frost protection. During this time a heavy fall of snow occurred and it was found, on lifting the boards, that most of the fish, some of which were 6–8 inches in length, were dead or dying.

The timber had been previously treated with a colourless proprietary timber preservative and weathered for about a week before use. Leaching from the timber was suspected but no copper, organo-chlorine pesticides, or phenols (using the diazotised sulphanilic acid method) could be found in the water.

The timber itself was then examined. A petroleum ether extract gave a positive reaction with the copper wire test, indicating the presence of organo-chlorine compounds, and a strong positive colour reaction for pentachlorophenol was obtained with copper sulphate and pyridine reagent. The latter test, carried out on an extract from 500 ml of water, gave a faint indication of pentachlorophenol.

It was thought that gas chromatography with electron capture detection would provide a sensitive method of confirmation but injections of solutions of pentachlorophenol in petroleum ether gave no peaks on a QF column normally used for pesticide residue analysis. Successful results, however, were obtained on a column including Carbowax/terephthalic acid, using the conditions given below. The determinations were made with Aerograph 600D G.L.C. apparatus.

GAS CHROMATOGRAPHY OF PENTACHLOROPHENOL

1. *Column:* 5 ft × $\frac{1}{8}$ in. O.D., glass, containing 5 per cent. of Carbowax 15,000, and 0·5 per cent. of terephthalic acid on acid-washed Supasorb (BDH) (100–120 mesh). The column must be well purged before use.
2. *Temperature:* 185° C.
3. *Detector:* Electron capture (Tritium).
4. *Carrier Gas:* Nitrogen flowing at 40 ml per minute.
5. *Volume injected:* 5 $\mu$l.
6. *Standard Solution of Pentachlorophenol:* 0·001 g of pentachlorophenol per litre of petroleum ether (boiling range: 40° C to 60° C).

A good response at 20× attenuation was obtained with solutions of 1 part of pentachlorophenol per million of petroleum ether. The chromatogram indicated that two components were present, with a minor peak at 6 min.

and a major peak at 19 min. (Retention times relative to aldrin were 1·3 min. and 4·2 min. respectively.) On a peak area basis, and assuming similar detector response, the ratio of the two components was approximately 86 : 14. Commercial pentachlorophenol is known to be a mixture of pentachlorophenol and tetrachlorophenol in the approximate ratio of 80 : 20. It is therefore probable that the major peak was pentachlorophenol itself. Full-scale deflection corresponded to a current of $6 \times 10^{-11}$ amps.

ESTIMATION OF PENTACHLOROPHENOL

The water sample (100 ml) was acidified and shaken with 10 ml of petroleum ether (boiling range 40° C to 60° C) and 5 $\mu$l of the extract injected. Two peaks corresponding to retention times identical with those for commercial pentachlorophenol were obtained. The ratio of the two components was similar in both sample and standard. Quantitative analysis was made by comparison of peak height of the major component, using several successive injections of sample and standard. The precaution was taken of working within the linear range of the detector and adjusting the concentrations of standard solutions to be fairly close to that of the sample. The results indicated a concentration of 0·2 p.p.m. of pentachlorophenol in the water. (At least 95 per cent. of the total amount present was obtained by a single extraction of the water with 10 ml of petroleum ether.)

TOXICITY OF PENTACHLOROPHENOL

Information on the toxicity of pentachlorophenol to goldfish is sparse, but from data kindly supplied by the Water Pollution Research Laboratory, on American fishes, concentrations exceeding between 0·05 and 0·1 p.p.m. could be toxic. All species of fish studied were killed at a concentration of 0·5 p.p.m. and the more sensitive were affected at about one tenth of this concentration.

On this basis, the level of pentachlorophenol found in the water of the gold-fish pool was within the toxic range. The toxicity would have been accentuated by the fairly low dissolved oxygen content of the sample (40 per cent. saturation at a temperature of 6° C).

# Book Reviews

**BELL'S "Sale of Food and Drugs".** Service Volume—Issue No. 13. Butterworths & Co. Ltd., London, 1967. Price £2 15s. 0d. (plus 2s. 0d. postage).

So many words of praise and admiration have been written about the magnificent work of the compiler and publishers of the service volume of Bell's "Sale of Food and Drugs" that it becomes very difficult to review this 13th Edition without repetition. This new issue completes the service volume up to 1st September, 1967. It is refreshing in this day and age of apathy, working to rule and devaluation, to find a service which is not only first class but really up-to-date and delivered with the minimum of delay.

With this 13th issue, the additions and revisions have necessitated the Noter-up section being bound separately owing to lack of space in the binder. Cross-referencing between the Noter-up and other parts of the service volume remains unchanged. The 294 pages of this section include many new entries and revisions since the 12th issue, including summaries of legal proceedings, reports of the Food Standards Committee of the Ministry, etc.

Many new additional Statutory Instruments are published in the 100 pages of Section 3; these include the Colouring Matter in Food Regulations, Cheese (Amendment) Regulations, the long-awaited Meat Pie and Sausage Rolls, Canned Meat Product, Sausages and Other Meat Product Regulations, together with the Artificial Sweeteners in Food Regulations and the Food (Control of Irradiation) Regulations.

As in previous issues, the Supplementary Tables and Index are renewed. In view of the rapidly increasing size of this publication one wonders if, in future issues, the revised Index could be extensively expanded to give more comprehensive information. It is realised that the revision of such an Index may necessitate some delay before publication but it would, surely, enhance this invaluable reference book.

Judging by the well-worn appearance of the reviewer's copy of "*Bell*", and the fact that the Noter-up section has now outgrown the service binder, can we hope to see, in the near future, a completely new edition gracing our library shelves?

(Miss) A. Cook.

SUPPLEMENT TO OFFICIAL, STANDARDISED AND RECOMMENDED METHODS OF ANALYSIS. Pp. xiv + 424. S. C. Jolly (Editor). London: Society for Analytical Chemistry, 1967. Price £7 7s. 0d. (post free).

This book has such an authoritative standing that the reviewer feels rather like a curate asked to review the New Testament for a church magazine; a certain deference must therefore be pardoned.

The word "Supplement" strictly applies only to the first 73 pages. The remainder and bulk of the book consisting of a bibliography is complete in itself and every section, except that on soils, has been revised and brought up to date, with references to work published up to 1966. In addition, there is a new section on coffee, which did not appear at all in the 1963 edition. All these sectional bibliographies have been revised by acknowledged experts in their various fields and provide a valuable and complete work of reference.

Part one, consisting of detailed accounts of standard methods, recommended by A.M.C. sub-committees, contains two amendments to methods previously published in the 1963 volume. One of these concerns the wet oxidation of organic matter, method IVa, and the other is the revised process for the determination of small amounts of mercury published in the *Analyst* in 1965.

There is one addition to the section on pesticide residues (determination of demeton methyl) and two to that on crude drugs (chemical assay of pyrethrum, and the assays of

anthraquinone glycosides and rhein glycosides in senna). There is also a new section on the semi-micro determination of chlorine in organic combination.

The bulk of the seventy-three pages of part one is devoted to a new section on "Additives in Animal Feeding Stuffs" and this comprises twenty separate methods for the determination of antibiotics, hormones, prophylactics and vitamins.

This book, like its predecessor in 1963 is a mine of information for the analyst, and is almost as necessary in the laboratory today as an ultra-violet spectrophotometer or gas liquid chromatograph.

S. J. BUSH.

JOURNAL OF RADIO-ANALYTICAL CHEMISTRY. Amsterdam: Elsevier Publishing Co. Ltd., Published quarterly. Price £8 5s. 0d. per annum.

The reviewer of a new journal must first consider whether or not there is a real need for the publication. There is no doubt that radioanalytical chemistry as a discrete subject has so far been neglected by the publishing world, and in view of the ever-increasing and certainly permanent importance of radioisotope techniques in analysis the decision to produce a "Journal of Radio-analytical Chemistry" is well justified.

The first issue, and the list of other papers received, show a commendably practical bias, which one may hope the editors will maintain; this is an approach which will be welcomed by all who are directly responsible for analysis involving radionuclides, to whom the more esoteric flights of theory are of little practical concern. Those laboratories which are equipped for radiochemical analysis will find this new journal of great interest and of practical value.

T. M. COTTON.