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New Legislation and Official Literature Issued During 1978

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Lists are given of some of the documents of 1978 relating to legislation likely to be encountered in Public Analysts' laboratories. Reference is also made to some of the reports issued in 1978.

Every year Public Analysts feel a need to explain why they think it necessary to summarise such of the past years' legislation as may affect them. In 1978, the Department of Health and Social Security in Marsham Street, London issued a document entitled *D of E Central Unit on Environmental Pollution for the Inter-departmental Committee on the Redevelopment of Contaminated Land: Notes on Redevelopment of Gas Works Sites*, and numbered ICRCCL 2/78 and CUEP/198/2, which serves as an excellent illustration for this. The list it contains, of tests to be applied to the soils of old gas works sites before consideration may be given to the redevelopment of the land for housing or other projects, is certainly comprehensive. It appeared to have been assembled as guidance for those concerned with utilising old gas works sites, to follow a report numbered ICRCCL 3/78, called *Redevelopment of Contaminated Land*, which considered every possible hazard and inconvenience which might occur in the reclaiming of old industrial sites. The DHSS document is ambiguous as it uses the term "Total Metals" without defining what is meant by this and thus it often falls to the Analyst to decide what the enquirer really wants, and then to draft his certificate to show not only what his findings were, but how they relate to the document which may state what standards are being set.

The LAA/Bulletins (which serve so well in providing a guide to where the legislators' thoughts lie) have now proceeded to number 11. In number 11 it was announced that issue of the consultative document, on which comment would be invited about the proposed comprehensive review of the Food and Drugs Act, would be postponed until early in 1979. It has been hard not to make premature comment about this review, but all of us who have done so have merely made self-interested pleas for recognition in the new Act. It might have been better to wait and see whether at last some real effort will be made to make the anti-adulteration legislation work. The Food and Drugs Act was the only one of the three important consumer protection Acts of the mid 1800's that was not given its own Inspectorate. In September 1978, the Bank of England stated that there had been a "Consumer Boom". Perhaps it will influence the future of the Food and Drugs Act.

The *MAFF Information Bulletins* of 1978, broadly, were as follows:

Bulletin number	Topics
27	Hopes about methods of analysis, mainly from the Codex Alimentarius Commission.
28	Updated proposals for methods of analysis for concentrated milk products.
29	Some analytical methods relating to coffee products.
30	Analysis of caseinates and a method for copper in milk products.
31	Moisture and solubility of caseinates, NZ method for sediment in milk protein products.
32	Erucic acid, and coffee products.
33	Analytical methods for some sugar products.
34	Synopsis of methods of analysis for foods under discussion by EEC.

The *MAFF/DHSS Joint Announcements* in 1978 drew attention to the following matters.

- No. 47 Antioxidants in Food Regulations 1978.
- No. 64 Proposals to amend the Labelling of Food Regulations. (This was accompanied by a very useful comprehensive document which listed all the present U.K. food standards legislation.)
- No. 156 Labelling of Food (Amendment) Regulations 1978 which provided for changes in the names of certain pilchards.
- No. 162 Issue of a Report by the FSC on the declaration of added water in prepacked foods.
- No. 163 Proposals for regulations for coffee and coffee products.
- No. 173 Proposals for amending the Preservative in Food Regulations 1975 (as amended) to extend the use of benzoic acid in certain fish products and to permit the use of sulphur dioxide for the retention of colour in canned garden peas.
- No. 209 Introduction of the Common Agricultural Policy (Wine) Regulations 1978.
- No. 228 Publication of a FACC Report on a review of solvents in food.
- No. 237 Proposals for the transfer of provisions for the use of mineral hydrocarbons in food to the Miscellaneous Additives in Food Regulations 1974.
- No. 254 Proposals to amend the Colouring Matter in Food Regulations 1973 so that Riboflavin might be added and three colours (Orange G, Chocolate Brown FB and Channel Black) might be removed from the list of permitted food colours.
- No. 263 Publication of an FACC Report on the review of additives and processing aids used in the production of beer.
- No. 290 Appointments to the Food Standards Committee.
- No. 306 Setting up of a review of margarine and other table spreads.
- No. 320 Introduction of the Coffee and Coffee Product Regulations 1978.
- No. 329 Setting up of a review of regulations governing the use of emulsifiers and stabilisers in food.
- No. 408 The publication of a Report by the Food Standards Committee on exemptions from ingredients listing, and generic terms.
- No. 409 The introduction of the Colouring Matter in Food (Amendment)

Regulations 1978 (which make the changes proposed in announcement number 254).

- No. 414 The publication of a FACC Report on the review of nitrates and nitrites in cured meats and cheese (recommending a reduction in the levels permitted for the control of botulism in canned and cured meats).

CHAIS Bulletins

A new kindly venture which was introduced in 1978 for the assistance of Public Analysts in their new fields of endeavour was the *Consumer Hazards Analysis Information Service* (or the CHAIS Bulletins) issued by the Laboratory of the Government Chemist. Five issues were provided, with major topics as follows:

Issue no.	Contents
1	EEC Cosmetics Directive. Restricted substances. Methods provisionally agreed. Methods for nitromethane.
2	Methanol in cosmetic products. Organo-mercurial preservatives.
3	Methods for fluoride in toothpaste.
4	Methods for quinine in shampoos.
5	Cosmetic products—colouring agents.

The *Consumer Safety Bulletins* continued to be issued by the Department of Prices and Consumer Protection, numbers 11 to 19 appearing during 1978.

LACOTS and the EEC

On 1 March, 1978, another advisory body was born, after a two-year gestation, out of the former Local Authority Joint Advisory Committee on Standards. The new body is known as LACOTS, the Local Authority Co-ordinating body on Trading Standards. Its members include representatives of the ACC, AMA and COSLA. Its first Chairman was Mr Norman Best (Chairman of the ACC Consumer Services Committee), and its first task was a weights and measures one associated with the move from the U.K. minimum weight system to the EEC average weight system. The former Joint Secretary of LAJAC wrote to thank the LAJAC technical advisers, and to warn them that they might be called upon to join working parties for the new body. A major part of the new body's function is said to be concerned with EEC Consumer legislation. From the viewpoint of the local authority laboratories themselves, this last mentioned field of endeavour seemed incomprehensible, but the Department of Industry EEC Information Unit in Victoria Street, London, attempted to provide an overall map in a publication called "*EEC. This is your business—A T & I Guide to the European Communities. HOW IT WORKS.*" Another body called the International Union of Local Authorities Council of European Municipalities at 36 Old Queen Street, London, promised to provide a European Information Service, issue no. 1 of which appeared in June, and the third issue dated 8 October found its way to the laboratories because of an assertion that lead from wine bottle neck wrappers might be likely to contaminate wine. Mention was also made in

it of a report about the possible adverse effects of consuming fats. The magazine *Trade and Industry*, on 10 March, tried to provide a checklist of Environmental Directives, and then, on 28 April, provided another list of draft and adopted Directives, only 25 of which dealt with foods. An article by Mr Alan Turner of Cadbury Schweppes Ltd., which appeared in the November issue of the magazine *Environmental Health*, however, stated that, in 1976, the U.K. Food and Drinks Industries Council had identified 106 Directives which concerned food. He mentioned the tide of legislation as running so fast that everyone was being drowned by it, and although MAFF helped to negotiate British Milk Chocolate into the Cocoa and Chocolate Directive, it was only provided that it was called "Household Chocolate" in Germany and "Freak Chocolate" in Belgium. LAJAC produced a draft of a Codex Alimentarius document on Soups and Broths which indicated that, at the time of its distribution, there had been meetings on the topic which had involved representatives from seventeen countries. Then in June, from out of the blue, there appeared Codex Alimentarius Committee deliberations about oils and fats, about "Reduced-fat Margarine", about Low Erucic-acid Rapeseed-oil, about Edible Coconut-oil, Palm oil and Palm Kernel oil, some proposed draft standards for Grapeseed-oil and Babassu-oil, and documentation about Sterols and Fatty acids in edible oils.

Attention was also drawn during the year to the following references in the Official Journal of the European Communities.

<i>Date</i>	<i>Page</i>	<i>Subject</i>
14 i 78	L 11/18	Colours in medicinal products.
10 ii 78	L 40/19	Additives in feeding stuffs.
15 ii 78	L 44/15	Directive 78/142/EEC on vinyl chloride monomer in food.
15 ii 78	L 44/17	Limit of 1 mg/kg of vinyl chloride monomer in foodstuffs.
15 ii 78	L 44/18	Antioxidants in foodstuffs (amendment).
15 ii 78	L 44/20	Colouring in foodstuffs (amendment).
15 ii 78	L 44/23	Preservatives in food (amendment).
2 vi 68	L 146/1	Changing the laboratories which may issue wine documents for wines imported from third countries.
17 vi 78	L 160/9	Revising the levy on Isoglucose.
24 vi 78	L 167/36	The method for deciding the minimum bread-making quality of wheat.
22 vii 78	L 197/10	Amending the chocolate directive.
22 vii 78	L 197/22	Amending the emulsifiers, thickeners and gelling agents directive.
29 vii 78	L 206/13	Packaging of pesticides.
29 vii 78	L 206/43	Methods of analysis for feeding stuffs containing Zinc bacitracin, Flavophospholipol, Iron, Copper, Manganese and Zinc.
14 viii 78	L 223/7	Purity criteria for certain food additives.
14 viii 78	L 222/1	Quality of water for fish life.

14	viii 78	L 223/30	Purity criteria for certain antioxidants.
1	ix 78	L 208/2	Proposals for standards for upland surface drinking waters.
28	ix 78	L 272/1	Changing the date of arrangements for the determination of water in frozen chickens.
17	x 78	L 291/15	Amending the chocolate products directive.
20	x 78	L 295/21	Increasing the alcoholic strength of certain wines.
25	xi 78	L 330/30	Amending criteria for certain additives in feeding stuffs (includes a list of preservatives).

In addition the following answer to a question was given:

22	iii 78	L 72/25	Benomyl, used to protect plants and plant products against harmful organisms, but which does not appear in the list of permitted preservatives, may be considered a borderline case and the French Republic's authorisation of its use stands.
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There also appeared to be EEC discussions afoot about proposals for flavourings in foods, date markings, drained weights and the declaration of "EEC Index" numbers for food additives. A letter from MAFF dated 30 January 1978 and bearing ref. FCN 53G, stated that discussion of a Draft Directive on drinking water quality was unlikely to take place at least until mid-year.

Milk

Very little new legislation about milk appeared in 1978.

Under the Drinking Milk Regulations the "Guideline Figure" for the fat content of Standardised Whole Milk which might be imported into the U.K. during the year beginning in May 1978, was 3.78 per cent.

Two amendment orders to the *Brucellosis (England and Wales) Order 1977* extended the eradication areas and made changes in the powers of appointed officials, but these Statutory Instruments (SI 1978 Nos 541 and 689) made no changes to affect Public Analysts.

Two letters of guidance were received from the MAFF at Whitehall Place, SW1, bearing the reference MK 12417. The first mentioned sales of Untreated Milk, stating that the policy which already had resulted in the limitation of sales of untreated milk to that from cows in brucellosis accredited herds would be extended by 1 August 1980. New Milk (Special Designation) Regulations will then be made which will no longer provide for the sale of "Untreated Milk" except where special consents are given. This matter will affect some laboratories. The swings and roundabouts system of costings in some instances depends upon profits from milk testing subsidising losses on food testing, but many inspectors claim that there will be no further need for milk sampling when Untreated Farm Bottled Milk ceases to exist. Already among Inspectors who are so persuaded, there appears to be some misunderstanding about the meaning of milk-as-it-comes-from-the-cow, and those seem to believe that low-fat fore-milk may be accepted as "genuine milk". The Ministry letter MK 12317G re-emphasised the meaning of Section 91 (5) of the Food and Drugs Act 1955, and the last paragraph of the Seventh Schedule, (which refer to "milk of the cows

when properly and fully milked") and pointed out that Regulation 16(f) of the Milk and Dairies (General) Regulations, 1959 makes special provision for the handling of fore-milk—both provisions superseding the ruling of *Grigg v. Smith*, 1917, whose reporting in Bell and O'Keefe had caused the misunderstanding.

A newspaper report in early 1979 suggested that Untreated Milk may be permitted to continue, after all, for another three years.

Food Standards

The new U.K. food legislation of 1978 was the following.

The Antioxidants in Food Regulations 1978, SI 1978 No. 105. The Regulations re-enact the regulations of 1974, with EEC provisions included (i.e. re-defining "Dairy Product", extending the list of "specified foods", permitting Diphenylamine on apples and pears (up to 10 p.p.m.) and allowing baby foods with added Vitamin A to contain BHA or BHT introduced with the Vitamin A preparations).

The Labelling of Food (Amendment) Regulations 1978, SI 1978 No. 646. The Regulations provided for two new designations for pilchards, namely "Pacific Pilchard" and "South Atlantic Pilchard" but names like "Chilian", "Japanese" or "Californian" may still be used for the former, and "South African" may still be used for the latter until 31 March 1980.

The Common Agricultural Policy (Wine) Regulations 1978, SI 1978 No. 861. The Regulations re-enact with amendments the Regulations of 1973 making it much easier to identify the offences which the local authorities (or the Minister, or the Wine Standards Board) are expected to monitor.

The Beer Regulations 1978, SI 1978 No. 893. For the analyst, the parts of these Regulations which are of greatest importance are the instructions about the determination of Original Gravity (which must be carried out at 60°F) and the prohibitions on saccharin or liquorice.

The Beer (Amendment) Regulations 1978, SI 1978 No. 1186. The amendment rewords the prohibitions mentioned above.

The Price Marking (Food) Order 1978, SI 1978 No. 738.

The Food (Prohibition of Repricing) Order 1978, SI 1978 No. 1014. Except in the case of withdrawal of introductory or special offer prices, food items once displayed at a particular price for retail sale may not be repriced to a higher price.

The Coffee and Coffee Products Regulations 1978, SI 1978 No. 1420. The regulations replace, from July 1980, the Regulations of 1967.

The Colouring Matter in Food (Amendment) Regulations 1978, SI 1978 No. 1787. The Regulations remove from the permitted list of food colours the artificial

colours Orange G, Chocolate Brown FB and the Channel Black form of Carbon Black. They add a new colour Riboflavin-5-phosphate (which must meet certain purity criteria).

Further Statutory Instruments to note are the following.

The Price Marking (Cheese) (Amendment) Order 1978, SI 1978 No. 133. The order varies the unit pricing rules so that no ordinary customer will know whether the unit price should be shown or not.

The Bread Prices (No. 2) Order 1976 (Amendment) (No. 6) Order 1978, SI 1978 No. 545. Former references to bread loaf weights of 28 oz., 14 oz., 10 oz. and 8 oz. shall be replaced by the weights 800 g, 400 g, 300 g and 225 g respectively.

Bread Prices (No. 2) Order 1976 (Amendment) (No. 7) Order 1978, SI 1978 No. 1790. Price control.

The Butter Prices (Amendment) Order 1978, SI 1978 No. 835. The order increased the maximum prices of butter.

The Concentrated Butter Prices Order 1978, SI 1978 No. 971. The order regulated the maximum price to be charged for butter for cooking made from butter sold from intervention stocks at reduced prices.

The Provision of Milk and Meals (Amendment) Regulations 1978, SI 1978 No. 959. The Regulations make arrangements for the charges connected with the provision of school milk for pupils up to the age of 12. They are unlikely to affect sampling arrangements which may have been made by local authorities in respect of such milk.

The Authorised Officers (Meat Inspection) Regulations 1978, SI 1978 No. 884. The Regulations revoke the Regulations of 1974 and specify the qualifications to be held by Meat Inspectors.

The Materials and Articles in Contact with Food Regulations 1978, SI 1978 No. 1927. Public Analysts are specified as the analysts to carry out tests on materials expected to come into contact with food and which may use the restricted description "for food use". The method of testing for transfer of components to food is not shown in the Regulations.

Other Food Standards

Imported Milk. Circular FSH 9/78 (in the form of an MAFF letter ref. MK 13719A) reminded District Authorities that any UHT prepacked imported milk needed to be re-processed and packed at registered premises in this country as a precaution against the possible importation of Foot and Mouth Disease, and that the importers' Continental packs containing a litre contravene Schedule 4 Part V 3 (a) of the Weights and Measures Act.

Pasta. A Food Manufacturers Federation Code of Practice for dry pasta made in the U.K. seemed to be being circulated in 1978. It called for a maximum moisture content of $12\frac{1}{2}$ per cent., ash between 0.6 per cent. and 0.85 per cent., protein at least $11\frac{1}{2}$ per cent. ($N \times 5.7$) and beta-carotene colour at least $2\frac{1}{2}$ p.p.m. None of the imported pasta products appear to comply with these criteria.

Metallic Mercury in Oranges. A DHSS letter ref. CMO(78)2 asked that the discovery of metallic mercury in oranges be notified to Alexander Fleming House but that the metal did not constitute a significant hazard to health.

Water Content of Deep-frozen Turkeys. The Commission of the European Communities *Information on Agriculture* series No. 42 is mainly a statement of experimental work on the title subject, but it does state that the physiological water of turkey carcasses is related to the protein by the equation:

$$\text{Water} = 3.21 \text{ protein} + 89.8.$$

Water Content of Poultry Meat. Two circulars about poultry meat were issued by the MAFF. Circular FSH 1/78, issued by the branch at Surbiton, was about interim arrangements for health marking of carcasses sent to other premises for cutting, and would scarcely affect Public Analysts except that it lists EEC Directives concerned with poultry meat.

Circular FSH 3/78 bears an earlier date than FSH 1/78 but it was issued by MAFF at Whitehall Place. It explains that Food and Drugs Authorities are the designated authorities for ensuring that regular checks on water uptake of poultry carcasses during washing and chilling are carried out at slaughterhouses, and suggests that when "in-plant" tests indicate that there has been less than 5.5 per cent. water uptake, the ANNEX III test will be satisfied. It also contained a reminder that at least one test in each four-hour working-period is required.

Council Regulation (EEC) No. 2238/78 of 26 September 1978 (O.J., 28 September 1978, p. L 221(1)) postponed implementation of the control measures which involve ANNEX III testing.

Proposed Standard for Canned Mackerel. This was a proposed draft standard only, which had reached Step 5 in the procedures of the Codex Alimentarius Commission, and bore the number ALINORM 78/18A. It consisted of product quality detail for manufacturers. It included a "Defects Score" which seemed not to consider parasites.

Ministry Guidance Letters

The MAFF is kind enough to send guidance letters with most of its circulations of proposals, and with some of the new regulations. In addition it will sometimes provide informal opinions about matters which are found not to be straightforward but it is always careful to include the caveat that only a Court could give an authoritative interpretation of legislation. The letters are, nevertheless, extremely valuable as indications of what was probably intended by the various regulations.

MAFF letter ref. FS 6336B. Labelling of Sausages. It was not intended that the 1976 Amendment Regulations should permit excess preservative in rusk to carry over significant quantities into the sausages and yet remain undeclared, simply because it was an ingredient of the rusk. Additionally a sausage does not opt out of the minimum meat standards simply by substituting for the usual adjective "Beef" or "Pork" a word like "Farmhouse", and making up the protein content with milk powder.

MAFF letter on Benomyl Residues on Citrus Fruit. The letter drew attention to "Chemicals Used in Agriculture and Food Storage" leaflet "RECS/1172", issued in April 1976, which states that Benomyl is not a hazardous chemical and is recommended as a post-harvest dip for fruit and potatoes. The CODEX committee had put forward a guideline recommendation of about 10 p.p.m. for residues on citrus fruit.

MAFF letter ref. ADF99. Polyethylene Glycol Tablet Lubricant in Campden Tablets. The letter stated that polyethylene glycol is a non-permitted stabiliser, and the regulations prohibit chemicals not on the permitted list but which have capabilities of emulsifying and stabilising, regardless of the function actually performed.

MAFF letter ref. FS 4878A. Slimcea Lightmilk is not a concentrated milk but a mixture of milk and skimmed milk. It should not be labelled "enriched" and it should bear a list of ingredients.

MAFF letter ref. FS 6336. The designation "Whole Orange Drink". The letter recalled that the FSC's 1959 report objected to the term "drinks made from whole oranges" and in the review FSC/REP/65 (published in 1976) recorded that the term "Orange Drink" had come to replace it. While appreciating the ambiguity in the designation "Whole Orange Drink" it seemed that if the term "Orange Drink" were to be considered acceptable for diluted and sweetened orange juice, the name "Whole Orange Drink" might have to be accepted for a similar drink, whose orange component consisted of comminuted whole oranges.

MAFF letter from Local Advisory Service. This is mentioned because it raised the interesting question of whether a poultry processing plant might use octadecylamine to protect its steam-raising equipment. The local Veterinary Officer thought the local authority would object. When this was looked into, however, it was found that Paragraph 24 of the *Food Additives and Contaminants Committee Review of the Emulsifiers and Stabilisers in Food Regulations* (H.M.S.O., 1970) had already taken a rather indulgent attitude toward the small amounts which would be carried over into food by steam.

Fertilisers and Feeding Stuff

Because farming is one of Britain's largest industries, one might expect it to be able to look after its own raw materials by the usual industrial process of buying

against Specifications and Warranty, but probably because farming affects everyone, the control of fertilisers and animal feeding stuffs is a Consumer activity. For many years, control was effected by means of the Fertilisers and Feeding Stuffs Act 1926, but since the U.K. became associated with the EEC, the legislation itself has been stimulated. Difficulties which have been mentioned in the past arise not only from incorporation of the earlier legislation into parts of the newer Agriculture Act 1970, and that part of the Medicines Act 1968 which deals with medicated animal feeds, but also because the moment of transfer coincided almost exactly with Britain's acceding to the EEC, and with the EEC having, at that stage, two Directives almost at completion. The Council Directive 76/116/EEC then had to be enforced in Britain by January 1978 and the Directive 77/535/EEC (which appeared in the Official Journal of the European Communities for 22 August 1977) had to be implemented in Britain by December 1978. Methods of analysis were in the latter Directive and most of them were included in the following.

Agriculture. The Fertilisers (Sampling and Analysis) Regulations, 1978, SI 1978 No. 1108. Guide to the Requirements of the Fertilisers Regulations 1977. This very necessary MAFF Guide was issued in November but distributed after Christmas. It draws attention to such matters as the overall variation which applies in the case of compound fertilisers, and which may narrow the limits of variation for individual constituents. Examples of how the limits should be applied are given.

MAFF letter ref. DW 334 (17 August 1978). This letter announced the publication of the Fertilisers (Sampling and Analysis) Regulations 1978 and explained that the sampling procedures for liquid fertilisers, and methods of analysis for fertilisers which are not sold as "EEC Fertilisers", would continue to be those in The Fertilisers and Feeding Stuffs Regulations 1973.

MAFF letter ref. DW 334 (18 September 1978). This letter suggested that in order to enable certificates of analysis to occupy only one side of a sheet of paper the pre-printed forms need not include all the analytical headings shown in the form of certificate detailed in Schedule 3 (p. 98) of the Fertilisers (Sampling and Analysis) Regulations 1978.

MAFF letters ref. DW 302 (December 1977 and 1978). These were summaries of local authority sampling for 1976 and 1977 respectively (certain corrections followed later in 1978).

MAFF letter ref. DW 356 (12 December 78). The letter drew attention to the forms in which nitrogen content should be declared in addition to *total N*, and explained that the declarations required for two of the soluble forms of phosphorus salts, namely " P_2O_5 soluble in neutral ammonium citrate and in water" and " P_2O_5 soluble in water" are mutually exclusive and should therefore be treated as separate entities each to be reported in its own right.

The Agriculture Act 1967 (Amendment) Regulations 1978, SI 1978 No. 244. This Statutory Instrument is mentioned only to indicate that it will in future be difficult to know which Agriculture Act legislation affects Public Analysts. In Stationery Office lists, items marked "Agriculture Act" may in future refer, as this Instrument does, to matters such as afforestation areas. SI 1978 No. 244 set out to metricate acreages, "four hectares" replacing former references to "ten acres".

Directory of Garden Chemicals (British Agrochemicals Association). The third edition of this booklet is a useful quick guide to uses, trade names and suppliers of certain chemicals.

Medicines

From 1 February 1978, "Drugs" were removed from the Food and Drugs Act 1955 in all places except the title. The Medicines Act 1968 provides for a comprehensive system of controls over medicinal products by a licensing system operated by the Health and Agricultural departments of central government, and enforced mainly by the Secretary of State for Social Services, or, when animal diseases are concerned, by the Minister of Agriculture, Fisheries and Food. Except for "counter prescribing", certain products sold from the manufacturing pharmacy without advertisement, and herbal practitioners' remedies prescribed for specific patients, licensing covers all manufacture and importation of medicines. The product licences usually make provisions about specifications (such as those of the B.P.), and about advertising, labelling, etc. The need, therefore, for checks at retail level has been considered to have been much reduced.

Some local authority involvement is anticipated by the Act, since in Section 128 (4) there is provision for the Minister to pay the local authorities such sums as may reasonably be incurred in the enforcement of certain provisions of the Act. So far, the only provisions (other than those imposed under Section 108, relating to medicated animal feeding stuffs as detailed in Section 62(1) (b) and Section 90 of the Act) to have been entrusted to the local authorities relate to Sections 53, 54 and 66 of the Act, which are mainly to do with the supervision of premises. In this connection however, it has been pointed out that no-one outside a pharmacy is allowed to sell loose medicine, so a local authority does have power to go into a shop where loose medicine might be on sale and take action. They would have similar powers over wrong labels on General Sale List Medicines. The only regulations so far to have been made under Section 66 concern segregation of veterinary drugs not on the General Sale List.

The *British Food Journal* for March/April 1978, p. 41, drew attention to the role under the Act of the Public Analyst in cases of "complaints" about medicines.

It has already been pointed out that local authorities were given an enforcement role in the case of the prohibition order, made under Section 62(1), to the Indian medicine, Bal Chivan Chamcho, and they may have a role in the control of herbal remedies mentioned in Section 12 of the Act. It is also suggested that the local authorities may have a role in checking false labels or misleading

labels as indicated by Section 85(5), in cases where drugs may be sold in premises other than registered pharmacies. They have also performed a role under Section 87 when local authorities checked retail outlets for aspirin and paracetamol preparations which were not in accordance with the requirements of the *Medicines (Child Safety) Regulations*. It is also believed that the local authorities could be given a function to fulfil under Section 95(1) of the Act, in connection with the advertisement of non-medicinal materials or of remedies for diseases which used to be listed in the Pharmacy and Medicines Act 1941 and now are listed in the *Medicines (Labelling and Advertising to the Public) Regulations 1978*.

Such views were broadly indicated in a press release ref. PCY 222.37 issued in July by the Association of County Councils, possibly following representations made in May by Hampshire County Council. Under the Food and Drugs Act 1955 most criticism of drugs arose through deterioration due to excessively prolonged storage, but this aspect appears to have been disregarded in the new legislation.

In the present circumstances therefore, it may not be appropriate to attempt to list Medicines legislation, because the impression grows that the DHSS is easing the local authority function out of the Medicines Act enforcement arrangements, and the paragraph headed "Sampling" on page 90 of the *Annual Report for 1977 of the Medicines Commission* (H.M.S.O., ISBN 010 257778 1, price £2) makes this almost a certainty. In case the ground needs to be made up, however, such guidance as may be found in the *Pharmaceutical Journal* 1978 for 21 January, p. 45, 28 January, p. 70, 4 February, p. 98, 25 February, p. 174 and 5 August, p. 110 may be worth noting.

The Statutory Instruments connected with Medicines to reach the Analysts' laboratories in 1978 were as follows.

The Medicines (General Sale List) Order 1977, SI 1977 No. 2129.

The Medicines (Fluted Bottles) Regulations 1978, SI 1978 No. 40.

The Medicines (Labelling and Advertising to the Public) Regulations 1978, SI 1978 No. 41.

The Medicines (Prescription Only) Amendment Order 1978, SI 1978 No. 187.

The Medicines (Labelling) (Special Transitional) Regulations 1978, SI 1978 No. 190.

The Medicines (Prescription Only) (Amendment (No. 2) Order 1978, SI 1978 No. 987.

The Medicines (Pharmacy and General Sale—Exemption) Amendment Order 1978, SI 1978 No. 988.

The Medicines (Sale or Supply) (Miscellaneous Provisions) Amendment Regulations 1978, SI 1978 No. 989.

The Medicines (Labelling) Amendment Regulations 1977, SI 1978 No. 996.

The Medicines (Exemptions from Restrictions on the Retail Sale or Supply of Veterinary Drugs (Amendment) Order 1978, SI 1978 No. 1001.

BRITISH PHARMACOPOEIA 1973 ADDENDUM 1978

This third Addendum, effective from 1 December 1978, amends the B.P. 1973 and the B.P. Addenda for 1975 and 1977. It was published on the recommendation of the Medicines Committee in accordance with Section 99(b) of the Medicines Act.

The 44 new monographs include the following:

Alcoholism treatment:	Disulfiram.
Anabolic steroid:	Oxymetholone.
Analgesics:	Alclofenac and Naproxen.
Antibiotic:	Rifampicin.
Antifungal:	Miconazole nitrate.
Diuretic:	Polythiazide.
Gonadotrophin:	Menotrophin.
Neuromuscular blocking agent:	Pancuronium bromide.
Vasodilator:	Methyl nicotinate.

The two monographs on *Charcoal* and *Piperazine Hydrate* are transferred to the Addendum from the B.P.C., to give effect to the European Pharmacopoeia requirements. Twenty-nine monographs of the B.P. 1973, and the 1975 and 1977 Addenda have been withdrawn in favour of the European Monographs. The requirements of the former general monograph on *Tablets* have been replaced by those of the European Pharmacopoeia and include a change in the method of performing the disintegration test.

New assay methods, and tests for related impurities, are given for the antibiotics *Ampicillin* and *Carbenicillin*, while G.L.C. becomes the official method for the assay of *Atropine* and hyoscine preparations. A new T.L.C. method is included for the detection of impurities in *Saccharin* and *Sodium Saccharin* (the impurity level having been reduced from 0.1 per cent. to 100 parts per million).

A new monograph for *NYSTATIN (Dermatological)* was issued, to have effect from May 1978.

Two series of amendments to the B.P.C. and the British National Formulary 1976-1978 were published in the *Pharmaceutical Journal* of 18 February and 30 September 1978. They concerned the B.P.C. preparations (i) Prepared Storax, (ii) Paraffin Gauze Dressing, (iii) Hydromellose Eye Drops, (iv) Calcium with Vitamin D Tablets, (v) Vitamins A & D Capsules, (vi) Vitamin capsules, and (vii) the B.P. preparation, Neomycin Tablets.

Guide to the Misuse of Drugs Act 1971 and to Certain Regulations made under the Act. This useful guide was published by the DHSS at Elephant and Castle, London, SE1 6TE in 1977, and became generally available early in 1978.

WHO Technical Report Series 615. Includes a model list of effective drugs such as might benefit developing countries which need to limit their purchases of drugs to those which are most cost-effective.

MEDICINES—RADIOACTIVE SUBSTANCES

The Medicines (Radioactive Substances) Order 1978, SI 1978 No. 1004. (Laid before Parliament in draft in April and made in July.) Brought within the control of the Medicines Act certain pieces of sealed-source radioactive apparatus intended for carrying out medical tests upon the human body.

The Medicines (Committee on Radiation from Radioactive Medicinal Products) Order 1978, SI 1978 No. 1005. Set up an advisory committee on medicinal radiation.

The Medicines (Administration of Radioactive Substances) Regulations 1978, SI 1978 No. 1006. Implement provisions in Council Directive 76/579/Euratom for a system of prior authorisation for use of ionising radiation in medicine.

Consultative Document: Health and Safety Commission "Ionising Radiations". Includes a summary of present legislation, maximum levels of surface contamination, etc.

Poisons

The Poisons Act 1972. This Act was brought into effect on 1 February 1978 by *The Medicines Act 1968 (Commencement No. 7) Order 1977* and deals with the control of non-medicinal poisons. In general terms, the Act replaces the control of those poisons which under the *Pharmacy and Poisons Act 1933* might only be sold by an *Authorised Seller of Poisons* or a *Listed Seller of Part II Poisons*. Enforcement remains with the local authorities.

The Poisons Rules 1978, SI 1978 No. 1. Apply only to listed poisons.

The Poisons (Amendment) Rules 1978, SI 1978 No. 672. This amendment was made in consequence of the *Packaging and Labelling of Dangerous Substances Regulations 1978* (SI 1978 No. 209) which implemented an EEC Directive on the classification, packaging and labelling of dangerous substances. (One effect is to relieve suppliers of liquid poisons, packed in quantities of not more than 1.14 litres, of the obligation to use fluted or ribbed bottles.)

The Poisons List Order 1978, SI 1978 No. 2. The new list contains only substances which have a non-medicinal use, so the Rules and the List relate only to the Poisons Act.

This legislation affects local authority laboratories most, in all probability, with questions involving the sale of disinfectants, so that attention should be paid to Schedule 3 which exempts preparations which do not contain phenol itself and contain less than 2½ per cent. of other phenols. British Standard 5917: 1976 "Specifications for Aromatic Disinfectant Fluids" will also be of interest, since this retains the old pine oil disinfectants.

Diseases of Animals

The Diseases of Animals (Approved Disinfectants) Order 1978, SI 1978 No. 32.

The Diseases of Animals (Approved Disinfectants) (Amendment) Order 1978, SI 1978 No. 934.

The Diseases of Animals (Fees for the Testing of Disinfectants) Order 1978, SI 1978 No. 708.

Weights and Measures

Weights and Measures legislation affects the laboratories only indirectly and in a manner similar to the legislation in such documents as The Bread Prices Order (i.e. to indicate what the original weight of certain commodities should have been). This is sometimes important when a sample has suffered deterioration, such as, for example, evaporation of natural moisture. The following list will almost certainly be incomplete:

The Weights and Measures Act 1963 (Potatoes) Order 1978, SI 1978 No. 741.

The Weights and Measures Act 1963 (Various Goods) (Termination of Imperial Quantities) Order 1978, SI 1978 No. 1080.

The Weights and Measures Act 1963 (Coffee Extracts and Chicory Extracts) Order 1978, SI 1978 No. 1081.

The Trade Marks (Amendment) Rules 1978, SI 1978 No. 1120.

The Trade Descriptions (Indication of Origin) (Exemptions No. 11) Directions 1978, SI 1978 No. 1153.

Consumer Protection

The Consumer Safety Act 1978.

Consumer Protection. The Babies Dummies (Safety) Regulations 1978, SI 1978 No. 836. Refers to BS 5239: 1975.

The Nightwear Safety Order 1978, SI 1978 No. 1728.

The Tobacco Products (Higher Tar Cigarettes) Regulations 1978, SI 1978 No. 1156. Higher duty is charged on cigarettes with a tar yield exceeding 20 mg per cigarette. This extraordinary piece of legislation may indicate a benign government or an avaricious one. Smokers seem to smoke for the nicotine stimulation, so when they change to low-tar cigarettes they smoke more of them. Since 1974, the low-tar cigarettes have already increased their share of the market from 5 to 12 per cent., during which time the number of smokers has fallen but the remaining smokers have increased the number of cigarettes smoked per person. While lung cancer deaths (associated with tar) have declined in men

under 60 years of age in rough proportion to the decline in men who smoke, the incidence of death from coronary heart disease (associated with the carbon monoxide, and therefore with the number of cigarettes smoked) has increased. It is rather strange that none of these things was discovered before Rhodesian tobacco became scarce, and Virginia tobacco cost valuable dollars. In Japan, where exchange rates do not hamper tobacco studies, twice as many cigarettes are smoked per person and the lung cancer death rate is only a fifth of that in the West—which is not to say that tobacco is without hazard.

✓ *Consumer Protection. The Cosmetic Product Regulations 1978, SI 1978 No. 1354.*

D of E Waste Management Paper No. 19. Wastes from the Manufacture of Pharmaceuticals Toiletries and Cosmetics (including a code of practice).

Consumer Law Statutes (ISBN 0 906533 00 7, Monitor Press).

The Environment: Water

D of E/National Water Council—Standing Committee of Analysts First Report on Methods for Quality Control of the Water Cycle (ISBN 0 904561 26 7). Published in 1977 and available in 1978, this is standard Technical Committee Report No. 7, and it reports on surveys of water analysis methods.

D of E Circular 20/78 and River Pollution Survey Report updated to 1975, D of E Circular 72/78. The D of E/National Water Council Sub-committee on the Treatment of Water of Swimming Pools has recommended that the I.C.I. cessation of supply of chlorine gas in cylinders to swimming pools, which is intended to operate from the end of 1980, should be postponed until the end of 1984.

The Water Authorities (Control of Discharges) Order 1978, SI 1978 No. 1210. Concerns procedures for granting and varying "Consents to discharge" effluents to streams and controlled waters.

Miscellaneous Environmental Matters

Harwell Environmental and Medical Sciences Division Report AERE—R 8869 "A Survey of Atmospheric Trace Elements in the U.K." Results for 1976.

D of E Eighteenth Progress Report of the Standing Technical Committee on Synthetic Detergents.

The Detergents (Composition) Regulations 1978, SI 1978 No. 564. When tested by the OECD Method, the surfactants in synthetic detergents must be at least 80 per cent. biodegradable.

Control of Pollution Act 1974 (Commencement No. 13) Order 1978, SI 1978 No. 954 (corrected).

Health and Safety Commission Draft Regulations, etc. "Control of Lead at Work".

Health and Safety Executive Guidance Note EH 15/77, Threshold Limit Values for 1977.

D of E Central Unit on Environmental Pollution Report of Joint Working Party: Pollution Paper No. 14. "Lead Pollution in Birmingham". The earlier pollution papers that most laboratories would benefit from holding are numbers 1, 2, 3, 4, 11 and 12.

D of E Waste Management Paper No. 17. Wastes from Tanning, Leather Dressing and Fellmongering (including a Code of Practice).

Proposals for New Legislation

- May 1978 Proposals for Revised Lead in Food Regulations.
 May 1978 Proposals for Regulations on Coffee and Coffee Products (seven days later a revised and corrected Schedule to the proposals was issued)—(later implemented).
 July 1978 Proposals to amend further the Miscellaneous Additives in Food Regulations 1974 to incorporate Mineral Hydrocarbons in Food legislation.
 July 1978 Proposals to amend the Colouring Matter in Food Regulations —(later implemented).
 December 1978 Proposals to amend and consolidate the Preservatives in Food Regulations 1975.

FSC Review of Food Labelling Part 2 *Exemptions from Ingredient Listing and Generic Terms* (FSC/REP/698) (dated 1977 but available in January 1978).

Reports, Etc.

- ✓ FACC Report on the Review of Solvents in Food (FAC/REP/25).
 FACC Report on the Review of Additives and Processing Aids, used in the production of beer (FAC/REP/26).
 ✓ Food Standards Committee Report on Water in Food (FSC/REP/70).
 FACC Report on the Review of Nitrites and Nitrates in Cured Meats and Cheese. Recommends maxima for sodium nitrate and nitrite (both expressed as sodium nitrite). The first number following shows as mg/kg the recommended limit for the two together, and the second number represents the maximum nitrite component of the first. Thus:
- | | |
|---------------------------|---------------|
| Uncooked ham and bacon | 500/200 mg/kg |
| Sterile cured meat packs | 150/50 mg/kg |
| Salami and cured sausages | 400/50 mg/kg |
| Other cured meat packs | 250/150 mg/kg |
| Continental cheeses | 50/5 mg/kg |

Evaluation of Certain Food Additives. Joint FAO/WHO Technical Report Series No. 617.

Food Control Laboratories (ICP/FSP 003). WHO Regional Office for Europe Report on a Conference.

Nitrates, Nitrites and N-nitroso Compounds. WHO Environmental Health Criteria 5 (ISBN 92-4-154065-6).

MAFF Food Surveillance Paper No. 1. First Report of the Steering Group on Food Surveillance. The Surveillance of Food Contamination in the United Kingdom. It is interesting to read alongside this report, a paper in *New Scientist* for 1 December 1977 by Dr Robin Wilson of Brunel University, and called, "Zinc: A Radical Approach to Disease".

MAFF Food Surveillance Paper No. 2. Second Report of the Steering Group on Food Surveillance. "Survey of Vinyl Chloride Content of Polyvinyl Chloride for Food Contact, and of Foods".

D of E Circular 50/78. Report of the Advisory Committee on Aggregates. Building Research Station Information Sheet IS/78 August 78. "Carbonation of Concrete Made with Dense Natural Aggregates".

Other Publications

Council of Europe 1977. Advice and Recommendations to be used by Authorities Concerned with the Registration of Pesticides. Pesticides (4th ed.).

A.D.A.S.: A Guide to Veterinary Pesticides (ISBN 011-241102-9).

Report of the FAO Global Survey of Pesticide Susceptibility of Stored Grain Products (ISBN 9205-1000-22-0). FAO Plant Production and Protection Series 5.

Second Report of the WHO Expert Committee on Vector Biology. "Chemistry and Specifications of Pesticides". WHO Technical Report Series 620.

British Association for the Advancement of Science Reports. "Salmonella, the Food Poisoner 1975-1977".

DHSS Local Authority Circular LAC(78)25. Annual Return of Food Poisoning: SBL 640.

Reprint from Chemical Society Reviews, Vol. 7, No. 2, 1978. "Chemistry and Flavour".

Industrial Training. The Industrial Training (Transfer of the Activities of Establishments) Order 1978, SI 1978 No. 448.

This final SI is of interest to Public Analysts as a reminder that there are no training arrangements for Public Analysts whatever.

A Field Test Method for the Estimation of Gamma-Hexachlorocyclohexane in Sheep Dips

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A colorimetric method for the estimation of gamma-hexachlorocyclohexane (γ -HCH) in sheep dips based on that of Yuen is described. γ -HCH is extracted from the dip into hexane and hydrolysed using alcoholic sodium hydroxide. Thiocyanate ions, displaced from mercuric thiocyanate by the chloride ions produced during hydrolysis, are complexed with ferric ions to give an intense red colour. The effects of the conditions for extraction, hydrolysis and colour reaction are discussed, and important modifications necessary for the application of the method under field conditions are described.

The dipping of sheep in solutions of the insecticide γ -hexachlorocyclohexane (γ -HCH) has been used in the eradication and control of sheep scab for three decades. Recommendations for correct procedure have been made by the Ministry of Agriculture, Fisheries and Food¹, one of which is that for effective dipping the concentration of γ -HCH in the bath should not fall below 50 mg litre⁻¹. Local Authorities are responsible for the enforcement of the Sheep Scab Order (1977) and supervision of the dipping operation includes sampling the bath to ensure that the concentration of γ -HCH does not drop below the recommended minimum figure. During the time between taking samples and obtaining analytical results from the laboratory the sheep have usually been put out to graze so that, if analysis shows that they have been ineffectively dipped, it becomes a tiresome and costly operation to repeat the procedure. The field test described here is designed to allow on-the-spot estimation of the γ -HCH content of a dip solution and hence to ensure that efficient dipping is being accomplished.

The method chosen as a basis for development was that described by Yuen² for the colorimetric analysis of Timber Dips. It depends on the hydrolysis of γ -HCH extracted from the dip to form chloride ions which displace an equivalent number of thiocyanate ions from mercuric thiocyanate. These ions are then reacted with ferric ions to form a complex which has an intense red colour. Several important modifications to the procedure and formulation of reagents were found to be necessary to allow rapid reliable application of the method to sheep dips in the field.

Experimental

APPARATUS

U.V. Visible Spectrophotometer. A Perkin-Elmer Model 124 spectrophotometer was used for the laboratory investigation of the method.

Gas Chromatograph. Pye Model GCV.

REAGENTS

1. *Ethanol* (analytical reagent grade).
2. *Ammonium nitrate solution, 60 per cent. w/v.*
3. *Hexane* (fraction from petroleum 67 to 70°C).
4. *Sodium hydroxide solution, 5 g litre⁻¹ (alcoholic).* Dissolve sodium hydroxide (12.5 g, analytical reagent grade) in water (100 ml). Dilute 10 ml to 250 ml with ethanol (analytical reagent grade). The reagent will remain stable for 2 to 3 weeks.
5. *Ammonium ferric sulphate solution, 1 per cent. w/v.* Dissolve ammonium ferric sulphate (1.0 g, analytical reagent grade) in a little warm water. Add concentrated nitric acid (15 ml), cool and dilute to 100 ml with water.
6. *Mercuric thiocyanate solution, 0.1 per cent. w/v (alcoholic).* Wash mercuric thiocyanate (10 g) with distilled water (100 ml) by stirring a slurry of the reagent. Filter and repeat the operation. Wash the filter-cake with two portions (50 ml each) of ethanol and dry under vacuum at room temperature. Dissolve 0.10 g of this reagent in 100 ml ethanol and store in a dark amber bottle. This reagent is stable for about 3 to 4 weeks.
7. *Cobalt (III) nitrate solution.* Dissolve cobalt (III) nitrate (8.06 g) in water and dilute to 100 ml.
8. *Potassium dichromate solution.* Dissolve potassium chromate (0.44 g) in water, add molar sulphuric acid (10.0 ml) and dilute to 100 ml.
9. *Colorimetric standard solution.* Mix the cobalt (III) nitrate solution (10.0 ml) with the potassium dichromate solution (2.0 ml). The resultant solution has the same optical spectrum as the iron-thiocyanate complex formed during colorimetric estimation of γ -HCH. It can be diluted with water to provide a range of standards to be used for the estimation of γ -HCH.

Method

Measure 30 ml well-mixed sheep dip solution into a 100 ml stoppered measuring cylinder. Make up to 100 ml with ammonium nitrate solution and add hexane (10 ml). Shake the tube for one minute then allow the layers to separate, tapping the base of the cylinder if necessary.

Transfer 1.0 ml from the hexane layer to a test tube (150 mm \times 16 mm i.d.). Add alcoholic sodium hydroxide (10.0 ml), mix and allow to stand for 10 minutes at not less than 20°C. Add ammonium ferric sulphate solution (2.0 ml) and mercuric thiocyanate solution (2.0 ml) and mix. Compare the red colour produced with the colorimetric standard solutions to obtain the γ -HCH content of the dip.

Discussion of the MethodEXTRACTION OF γ -HCH

Used sheep dip consists of a complex emulsion of γ -HCH, organo-phosphorus insecticide, phenols (in the form of tar acids), emulsifiers, grease from fleeces, faecal matter, urine and general dirt. In order efficiently to extract the γ -HCH into hexane it is necessary to break this emulsion. Some dip concentrates contain soap as emulsifier, whilst others are formulated using detergents. The

former type are destabilised mainly by the acidic nature of the ammonium nitrate solution whilst its high ionic strength overcomes the effect of the detergent in the second type and "salts out" the γ -HCH.

Hexane was found to be a better solvent for extraction than the toluene suggested by Yuen.² It allows complete extraction within one minute's shaking, better separation on standing and it is compatible with the colour forming reagents. Toluene was found to extract a much greater proportion of tarry matter from the dip than hexane, forming dark brown solutions which interfered with the colorimetric determination.

HYDROLYSIS OF γ -HCH

Full details of the mechanism of hydrolysis of γ -HCH are described elsewhere.³ It was found in our study that the rate of hydrolysis of γ -HCH is extremely temperature dependent. Aliquots (1 ml) of a solution of γ -HCH in hexane (370 mg litre⁻¹) were hydrolysed at temperatures of 20°C, 26°C, 41°C and 61°C for various times. At the end of the hydrolysis period the reaction was rapidly quenched by the addition of the ammonium ferric sulphate solution and then cooled before addition of the mercuric thiocyanate solution. The absorbances of the resultant solutions are shown as a function of time and temperature in Figure 1, which also includes results for a solution prepared by extraction of γ -HCH from a used sheep dip.

Examination of Figure 1 shows that, to reduce the hydrolysis time in the field, where temperatures can be close to freezing during the autumn dipping period, a suitable means of maintaining the temperature close to 20°C is required.

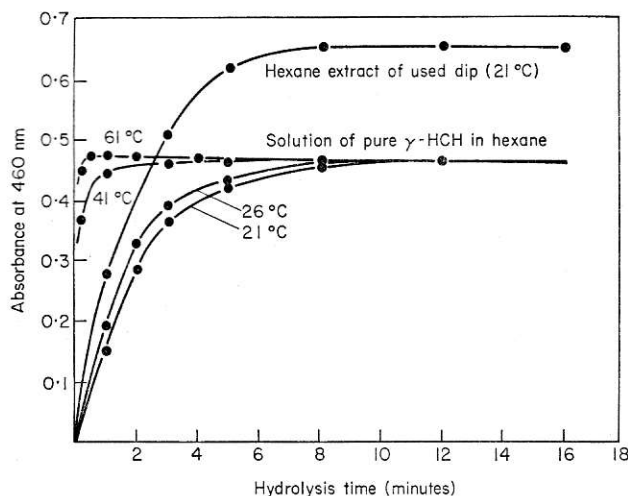


Fig. 1. Absorbance as a function of hydrolysis time and temperature for the colorimetric determination of solutions of γ -HCH in hexane.

FORMATION OF THE FERRIC THIOCYANATE COMPLEX

The solvent in which the ferric thiocyanate complex is prepared consists of a mixture of alcohol, hexane and water. To prevent separation of these phases and precipitation of sodium nitrate formed by neutralisation of sodium hydrox-

ide with nitric acid, the formulation of all the reagents must be strictly controlled.

Mercuric thiocyanate made up in absolute ethanol was found to be considerably more stable and gave lower blanks than a similar alcoholic solution containing 5 per cent. water, which was recommended by Yuen.²

COLORIMETRIC MEASUREMENT

For the purposes of laboratory investigation of the method absorbances were measured in a 10 mm cell at 460 nm. For the field-test trials nine stable colour standards were made to cover a range of absorbances equivalent to 30–150 mg litre⁻¹ γ -HCH in the dip. 15 ml of each standard solution were sealed into a glass test tube of a type identical to those used for analysis of the samples. Visual comparison of these with the colours produced from unknown samples was then used to estimate their γ -HCH content.

Results

CALIBRATION PROCEDURE

1 ml aliquots of solutions of γ -HCH in hexane (100 to 500 mg litre⁻¹) were hydrolysed and colorimetrically estimated according to the field-test method. The results of replicate determinations are shown in Table I.

TABLE I
REPLICATE COLORIMETRIC DETERMINATIONS OF γ -HCH IN HEXANE SOLUTIONS OF KNOWN CONCENTRATIONS

Concentration of γ -HCH, mg litre ⁻¹	Absorbance (corrected for blank)				
	1	2	3	4	Mean
100	0.125	0.120	0.120	0.125	0.123
200	0.260	0.250	0.255	0.260	0.256
300	0.385	0.370	0.380	0.380	0.379
400	0.515	0.510	0.520	0.510	0.514
500	0.640	0.635	0.640	0.635	0.638

The absorbance at 460 nm, obtained using 1 ml of hexane in place of the γ -HCH solution, (blank absorbance) varied between 0.035 and 0.050. Table I shows that the concentration of γ -HCH is linearly related to the absorbance over the range studied and that the precision obtained is excellent. The mean values given in the table were used to draw up a calibration graph and to determine the absorbances required of the colorimetric standard solutions for estimations carried out by visual comparison.

COMPARISON OF THE METHOD WITH GAS LIQUID CHROMATOGRAPHIC METHODS

Two aliquots of diluted samples of commercially available sheep dip concentrates were analysed: one by the field-test method and the other by gas liquid chromatography (G.L.C.). Two methods were used for extraction of the γ -HCH for G.L.C. determination. These were as follows.

G.L.C. 1. Shake 1.0 ml of dip with hexane (about 30 ml) in a 50 ml volumetric flask for one minute. Dilute to the mark with hexane, mix and accurately dilute a suitable volume to a concentration of approximately 0.1 mg litre⁻¹.

G.L.C. 2. Take a suitable volume of the hexane layer, obtained by extraction of γ -HCH by the same method as that used for colorimetric estimation, and accurately dilute to a concentration of approximately 0.1 mg litre⁻¹. This method gives a direct comparison between G.L.C. and colorimetric determinations on the same extract.

Inject 5 μ l of the diluted hexane extract on to a gas chromatographic column operated under the following conditions:

Column: 1 m \times 4 mm i.d. packed with Apiezon "L" (3 per cent.) on

—Chromosorb "W" (60–80 mesh).

—Carrier gas: Nitrogen. Flow-rate: 90 ml min⁻¹.

Injector temperature: 210°C.

Column temperature: 190°C.

Detector: Nickel 63 electron capture detector operated at 230°C.

Response to γ -HCH (retention time about 160 seconds) was found to be linear between 0.05 and 0.15 mg litre⁻¹.

The results of analysis by the three methods are presented for solutions prepared by dilution of two commercial dip concentrates in Table II.

TABLE II
COMPARISON OF γ -HCH CONTENTS OBTAINED BY THE FIELD TEST METHOD WITH THOSE OBTAINED BY G.L.C. FOR DIPS PREPARED IN THE LABORATORY FROM COMMERCIAL CONCENTRATES

Dip concentrate formulation	Nominal concentration of γ -HCH in dip, mg litre ⁻¹	γ -HCH concentration, mg litre ⁻¹		
		Field test method	Method G.L.C. 1	Method G.L.C. 2
1. 2.0 per cent. γ -HCH	150	143	158	175
2.5 per cent. Dichlofenthion	125	115	123	141
15 per cent. Phenols	100	96	102	115
	75	66	74	77
2. 3.2 per cent. γ -HCH	120	130	127	115
10 per cent. Chlorfenvinphos	90	100	85	81
28 per cent. Phenols	60	70	57	60
	30	42	32	28

The figures for the first dip formulation show good agreement between the nominal concentrations, and the results obtained by the field-test procedure and by method G.L.C. 1. Method G.L.C. 2, however, appears to give somewhat higher values. Dichlofenthion was found to have the same retention time as γ -HCH when using the apiezon column, but the use of OV17 silicone oil as stationary phase is reported to effectively separate these two compounds.⁴ The G.L.C. determinations of the γ -HCH contents of the dip solutions described in Table II were, therefore, repeated using an OV 17 column under the following conditions:

Column: 1.5 m \times 4 mm i.d. packed with OV 17 (1.5 per cent.) on

—Chromosorb "W" (80–100 mesh).

Carrier gas: Nitrogen. Flow-rate: 90 ml min⁻¹.

Injector temperature: 300°C.

Column temperature: 200°C.

Detector: Nickel 63 electron capture detector operated at 220°C.

The results are shown in Table III.

TABLE III
COMPARISON OF TWO G.L.C. METHODS USING AN OV 17 COLUMN FOR
DETERMINATION OF γ -HCH IN DIPS PREPARED IN THE LABORATORY FROM
COMMERCIAL CONCENTRATES

Dip concentrate formulation	Nominal concentration of γ -HCH in dip, $mg\ litre^{-1}$	γ -HCH concentration, $mg\ litre^{-1}$	
		Method G.L.C. 1	Method G.L.C. 2
1. 2.0 per cent. γ -HCH	150	160	165
2.5 per cent. Dichlofenthion	125	124	120
15 per cent. Phenols	100	100	96
	75	73	71
2. 3.2 per cent. γ -HCH	120	127	120
10 per cent. Chlorfenvinphos	90	84	85
28 per cent. Phenols	60	57	54
	30	32	30

It can be seen that the discrepancies between methods G.L.C. 1 and G.L.C. 2 for the solutions containing dichlofenthion have been eliminated. OV 17 was, therefore, subsequently adopted as the column of choice for evaluation of the field-test method. Comparison of the results for the second dip shows a small but significant positive difference between the field-test values and those obtained by both G.L.C. methods. It was thought that this might be due to the presence in the dip of the chlorinated organophosphorus insecticide, chlorfenvinphos (2-chloro-1-(2,4 dichlorophenyl) vinyl diethyl phosphate), which could produce a red colour by the field test procedure. This was shown to be the case by reaction of a hexane solution of Shell Supona (chlorfenvinphos, 10 per cent. e-isomer 90 per cent. z-isomer) which resulted in the formation of a red colour with absorption maximum at 460 nm.

IDENTIFICATION AND ELIMINATION OF INTERFERENCES FROM ORGANOPHOSPHORUS INSECTICIDES

Almost all sheep dip concentrates contain an organophosphorus insecticide in addition to the γ -HCH present. Accordingly, commercial samples of a range of the insecticides commonly used⁵ were obtained and tested for possible interferences in the determination of γ -HCH by the field test method.

A pure solution in hexane of each of the organophosphorus compounds was prepared to simulate an extract obtained by the normal field test method. 1 ml of solution was then subjected to the colorimetric determination procedure. Chlorfenvinphos, bromophos, chlorpyriphos and carbophenothion were all found to interfere to some extent with the field-test by production of a red colour in the same way as γ -HCH.

It was found that these interferences could be considerably reduced without significant loss of γ -HCH by vigorous shaking of the hexane solution (5 ml) with 0.5 g Fuller's Earth (BDH, adsorption grade) for 30 seconds, allowing the solid to settle and withdrawing 1 ml of the supernatant liquid for analysis. Recoveries of γ -HCH from solutions in hexane subjected to similar treatment varied between 95 and 102 per cent. Introduction of this further operation into the field-test procedure was thought, however, to be undesirable since the errors likely to be involved in the method carried out without the use of Fuller's Earth

were thought to be acceptable for a field test, an assumption which was further validated by results obtained on used sheep dips (Table V).

APPLICATION OF THE METHOD TO USED SHEEP DIPS

The field test method described above was used to monitor two sheep dipping operations carried out in Somerset in December 1977. Samples taken from the dip bath were divided into two portions, one for estimation of γ -HCH on the spot and the other for subsequent analysis in the laboratory by the method G.L.C. 1. The first trial was carried out at temperature close to freezing-point, and since at this time the effect of temperature on hydrolysis was not known, results obtained by the field test method were very low. The second trial was carried out at comparatively higher temperature (approximately 10–12°C) using a dip concentrate containing γ -HCH and dichlofenthion. The results are shown in Table IV.

TABLE IV
DETERMINATION OF γ -HCH BY THE FIELD TEST AND BY G.L.C. IN SAMPLES
TAKEN FROM A DIP BATH DURING DIPPING

Number of sheep passed through the bath	γ -HCH concentration, <i>mg litre</i> ⁻¹	
	Field test method	Method G.L.C. 1
0	130	152
25	70	85
50	60	71
75	45	60
100	40	38
120 (Bath replenished)	60	63
200	35	40
224 (Bath replenished)	60	65
300	40	46

There is good correlation between field-test and G.L.C. 1. The slightly low results obtained by the field-test procedure are a reflection of the temperature at which the tests were carried out. Further comparisons of field-test and G.L.C. methods have been made in the laboratory at 20°C, using dips of unknown origins, some of which were supplied by the Ministry of Agriculture, Fisheries and Food. The results of these comparisons are shown in Table V. The level of contamination is assessed on the basis of transition of the colour of the dip from cream in the clean state to dark khaki green in the heavily fouled state.

TABLE V
DETERMINATION OF γ -HCH BY THE FIELD TEST AND BY G.L.C. IN SHEEP DIP
SAMPLES OF UNKNOWN ORIGIN

Sample	γ -HCH concentration, <i>mg litre</i> ⁻¹	
	Field test method	Method G.L.C. 1
Dip concentrate 1 diluted 1 to 100	203	196
Dip concentrate 2 diluted 1 to 100	198	204
Dip concentrate 3 diluted 1 to 200	150	158
Used dip (clean)	170	174
Used dip (contaminated)	75	66
Used dip (contaminated)	80	84
Used dip (very heavily contaminated)	20	1
Used dip (contaminated)	49	52

Tables IV and V show that there is generally good agreement between the results obtained by the field-test method and those obtained by G.L.C. for a wide range of samples. In only two cases has it been impossible to get a clean separation between hexane and aqueous layers during extraction and these were for dips which had been used by more than 1,500 sheep.

Conclusion

The concentration of γ -HCH in sheep dips can be adequately determined in the field by the colorimetric method described above. Interferences from organophosphorus insecticides present in dips are, in most cases, low enough to be neglected but can be reduced if required by adsorption of these compounds on to Fuller's Earth.

The author is grateful to his colleagues in the Somerset Scientific Adviser's Department for the help received in carrying out this work, and to the Somerset County Council for permission to publish. Thanks are also due to Messrs Robert Young and Co. Ltd, and Shell Chemicals U.K. Ltd (Agricultural Division) for the provision of commercial samples of organophosphorus insecticides.

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Food Microscopy

(An Annotated Bibliography)

PART IIG. MAJOR INGREDIENTS: WATER

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The microscopy of water includes such topics as the examination of natural and municipal water supplies, water conditioning and treatment problems, water in food products, ice crystals, boiler deposits and scale, and the effects of various waters on dental enamel.

Many textbooks on the microscopy of drinking water have been published over the years and helpful papers can be located through *Water Pollution Abstracts*. The microscopical examination of water supplies may involve the identification of particulate matter (inorganic and organic) or of animal and vegetable life, including bacteria. The optical microscope, in conjunction with membrane filters, has been much used in these studies, and increasing use of the electron microscope is apparent, particularly for bacteriological examination. Both instruments are widely used in the control and development of water treatment for re-cycling. Immobilised or bound water in foods has also been investigated by electron microscopy.

The nucleation and growth of ice crystals in water, aqueous solutions and biological systems, including food, have been extensively studied and the development of snow and frost crystals is also of interest in connection with frozen products and cold storage. The growth of scale in boilers and pipes is sometimes a problem in the food industry and many different inorganic salts of Ca, Mg, Pb, Si and Sr have been identified in these deposits by microscopy and x-ray diffraction. Extravagant claims have been made for magnetic water treatment in the prevention of boiler scale, but these have not been substantiated.

Several electron microscopical studies of human dental enamel have been reported, and the effect on enamel of sodium fluoride in water supplies have been investigated.

The following references provide an introduction to these various aspects of water microscopy.

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