

THE DETERMINATION OF TRACE AMOUNTS OF NITROFURAZONE AND FURAZOLIDONE IN MILK

by R.J.M. Ratcliffe

(Smith Kline and French Laboratories Ltd,
Welwyn Garden City, Hertfordshire)

A method for the determination of trace amounts of nitrofurazone and furazolidone in milk is described, based on the formation and colorimetric determination of 5-nitro-2-furfurylidene phenylhydrazone. A preliminary separation of the furazolidone by extraction with benzene enables an individual assay of the mixed nitrofurans at a level of 1 μg per ml to be carried out.

The incidence of mastitis in cows leading to an inflammation of all or part of the udder is of sufficient importance to justify treatment by therapeutic means.

Both nitrofurazone [5-nitro-2-furfurylidene semicarbazone]^{1, 2, 3} and furazolidone [3-(5-nitro-furfurylidene-amino) 2-oxazolidone]⁴ have been shown to be effective against many of the organisms associated with mastitis. Field trials have demonstrated that treatment is more effective when the drugs are used in combination.

Residual nitrofurans in milk in excess of 5 μg per ml may interfere with the normal development of the organisms necessary for the production of cheese and butter flavours. Therefore there is a need for a method for the determination of low concentrations of these compounds in milk drawn from treated cows.

Buzard Vrablic and Paul⁵ have described a method of general application to the assay of 5-nitro-2-furfurylidene compounds based on the hydrolysis of the compound in an aqueous acid solution of phenylhydrazine with simultaneous formation of 5-nitro-2-furfurylidene phenylhydrazone which was subsequently extracted into toluene and the absorbance measured at 4350 \AA .

In order to use this method for the individual assay of mixed nitrofurans a preliminary separation of the components was necessary, since all 5-nitro-2-furfurylidene compounds gave the same phenylhydrazone.

It was found that furazolidone at low concentrations could be extracted from aqueous solutions into benzene or toluene

whereas the partition coefficient of nitrofurazone is such that it remains predominantly in the aqueous phase. When a 25 ml sample was extracted with 4 portions of 25 ml benzene, 94% of the furazolidone and 8% of the nitrofurazone were removed without the formation of intractable emulsions (Table 1).

In current practice nitrofurazone and furazolidone are used in equal proportions for the treatment of mastitis, and since investigation has shown that they disappear from the milk in the udder at approximately equal rates, the incomplete separation is largely compensated for in practice.

Experimental

Reagents

Benzene. Analar grade.

Toluene. Analar grade.

Dilute hydrochloric acid (approximately 5.5 M). Hydrochloric acid (Analar grade) diluted 1 + 1 with distilled water.

Phenylhydrazine solution. Dissolve 1.5 g Analar grade phenylhydrazine hydrochloride in distilled water and dilute to 100 ml. Prepare fresh daily.

Standard Nitrofurazone solution. Dissolve exactly 40 mg nitrofurazone in 25 ml N,N-dimethylformamide and dilute to 100 ml with distilled water. Dilute 10 ml of this solution to 100 ml, with milk from untreated cows preferably from the same herd. This solution contains 40 µg per ml.

Standard Furazolidone solution. Dissolve exactly 40 mg furazolidone in 30 ml N,N-dimethylformamide and dilute to 100 ml with distilled water. Dilute 10 ml of this solution to 100 ml with milk from untreated cows, preferably from the same herd. This solution contains 40 µg per ml.

Preparation of calibration curves

Prepare five stoppered 50 ml tubes as follows.

- (1) 3 ml normal nitrofurazone-free milk.
- (2) 0.5 ml standard nitrofurazone solution and 2.5 ml normal milk.
- (3) 1.0 ml standard nitrofurazone solution and 2.0 ml normal milk.

- (4) 2.0 ml standard nitrofurazone solution and 1.0 ml normal milk.
- (5) 3.0 ml standard nitrofurazone solution.

To each tube add 1 ml dilute hydrochloric acid and 1 ml phenylhydrazine solution, mix and place in a well stirred water bath at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 30 minutes. Cool rapidly to 20°C and to each tube add 10 ml toluene. Shake vigorously 40 to 50 times. Decant the emulsion into a centrifuge tube, and spin at 4000 r.p.m. for 3 minutes. Break the curdy top layer of toluene by gently stirring with a thin glass rod, and decant the toluene into a 1 cm absorptiometer cell.

Measure the absorbance of each toluene solution at 4350 \AA using a similar cell containing toluene as a blank. Subtract the absorbance of tube (1) from the standards (tubes 2, 3, 4 and 5) and plot a calibration curve relating absorbance to μg nitrofurazone per ml of milk.

Repeat the process using the standard furazolidone solution.

Determination of Nitrofurazone in milk

Pipette 25 ml of the milk sample into a 125 ml separating funnel and extract four times with 25 ml of benzene. Pipette 3 ml of the extracted milk into a stoppered 50 ml tube and proceed as described for the preparation of the calibration curves from "To each tube add 1 ml dilute hydrochloric acid." Since a "blank" milk truly representative of the sample is not usually available, subtract 0.035 from the absorbance found in the test, and from the nitrofurazone calibration curve read off the nitrofurazone content of the milk in μg per ml.

Determination of Furazolidone in milk

Pipette 3 ml of the original milk sample into a stoppered 50 ml tube and continue as described for the preparation of the calibration curves from "To each tube add 1 ml dilute hydrochloric acid." Subtract the total absorbance found in the nitrofurazone assay from the absorbance measured at 4350 \AA , and convert the residual absorbance to μg furazolidone per ml of milk from the furazolidone calibration curve.

Discussion

In the assay method described the furazolidone content of the milk is obtained by subtracting from the absorbance due to the total nitrofurans present, the absorbance due to an essentially furazolidone-free preparation. The absorbance obtained

in the nitrofurazone assay, however, includes the small absorbance found when nitrofurazone-free specimens of milk are carried through the assay procedure. It has been found that the milk from any single cow is likely to yield blank absorbancies which vary from day to day and even between different quarters of the udder (Table 2). The blank is small, usually 0.010 to 0.045 absorbance, although Cox and Heotis⁶ quote a wider range of values. The average of 60 measurements on milk from several cows over a period of time was 0.035. This value has been used as an empirical correction to be subtracted from the absorbance found in the nitrofurazone assay, in which it represents the equivalent of approximately 1 μ g nitrofurazone per ml.

The hydrolysis of the 5-nitro-2-furfurylidene compounds and their conversion to 5-nitro-2-furfurylidene phenylhydrazones is temperature and time dependent, the rate of conversion varying from compound to compound. The conditions chosen for the assay are a compromise suited to the two nitrofurans under consideration, and it is important to adhere strictly to these conditions.

Calibration curves run over a period of several months gave a range of absorbancies shown in Table 3. The calibration curves for both nitrofurazone and furazolidone obey the Beer-Lambert law over the range 0 - 40 μ g per ml.

Results

Recovery experiments were carried out within 3 hours of adding known equal quantities of nitrofurazone and furazolidone to milk from untreated normal cows. Results of these experiments are shown in Table 4. From consideration of the data in Table 3 and the fact that 8 per cent nitrofurazone and 94 per cent furazolidone are extracted by the benzene, it can be shown that 97 per cent of the nitrofurazone and 103 per cent of the furazolidone would be recovered when the two compounds are present in a 1:1 ratio.

If the ratio of the nitrofurans is changed, then using the data mentioned in the previous paragraph, expected recoveries can be calculated. Table 5 shows a comparison of these, and the actual recoveries.

It has been found that samples of milk from normal untreated cows to which nitrofurans at low concentration have been added show no significant decrease in the level of nitrofurans present over a period of 24 hours when stored at room temperature (Table 6).

Field Trials

Three series of trials were carried out using the proprietary mastitis preparation "Nefuran" containing 150 mg nitrofurazone and 150 mg furazolidone in a mineral oil base, per 3 g tube.

In the first series, three quarters of each of four cows were treated with one tube of Nefuran per quarter after morning and evening milking on three consecutive days. Samples from all quarters of each cow were examined for the presence of nitrofurans after the morning milking. The maximum nitrofurans content detected during the trial in three of the cows was 7 μg per ml of furazolidone and 8 μg per ml of nitrofurazone. However, the fourth cow showed a maximum content of 34 μg per ml furazolidone and 4.7 μg per ml of nitrofurazone. No nitrofurans could be detected in any milk sample 36 hours after the final injection.

Four cows in full lactation were used in the second trial, two quarters of each cow being treated with one tube of Nefuran after morning and evening milking on three consecutive days. Samples of the bulked milk from the treated and control quarters were examined for nitrofurans content at both milkings. The maximum content found was less than 1 μg per ml of either nitrofurans, and 12 hours after the final injection no nitrofurans could be detected.

In the final trial, four cows in full lactation were used, between one to four quarters of each cow being treated with one tube of Nefuran after evening milking on two consecutive days. Samples from all quarters of each cow were examined for nitrofurans content after both milkings.

The maximum amount of nitrofurans detected during the trial was 6 μg per ml of furazolidone and 2 μg per ml of nitrofurazone. No nitrofurans could be detected in any cow 24 hours after the final injection.

Smith and Scott⁷ in an investigation of the clinical and bacteriological aspects of these and other trials concluded that the nitrofurans intramammary preparation "Nefuran" was effective in 75 per cent of the cases of bovine mastitis treated.

The author expresses his thanks to those colleagues who provided valuable assistance in the investigation and to the Directors of Smith Kline and French Laboratories Limited for permission to publish the results.

The separation of Nitrofurazone and Furazolidone in aqueous solution by four extractions with an equal volume of benzene

µg per ml before extraction		µg per ml after extraction	
Nitrofurazone	Furazolidone	Nitrofurazone	Furazolidone
11	12	10	1
22	25	21	1
34	37	31	2

Table 2

Absorbancies measured in Nitrofurazone assay when applied to milk from a normal untreated cow

Day	1		2		3		4		5	
	a.m.	p.m.	a.m.	p.m.	a.m.	p.m.	a.m.	p.m.	a.m.	p.m.
Right-fore quarter	0.037	0.022	0.027	0.028	0.026	0.020	0.029	0.033	0.027	0.016
Left-hind quarter	0.038	0.029	-	0.032	0.026	0.035	0.030	0.027	0.022	0.013
σ limits	0.027 ± 0.007									

Table 3

Reproducibility of the calibration curves of Nitrofurazone and Furazolidone

Concentration µg per ml	Nitrofurazone			Concentration µg per ml	Furazolidone		
	Absorbance in 1 cm cell at 4350 Å				Absorbance in 1 cm cell at 4350 Å		
	Max.	Min.	Average		Max.	Min.	Average
6.6	0.185	0.169	0.179	6.6	0.162	0.138	0.149
13.3	0.352	0.266	0.322	13.3	0.328	0.262	0.291
26.7	0.686	0.636	0.667	26.7	0.619	0.550	0.589
40	1.021	0.996	1.011	40	0.972	0.861	0.909

Table 4

Recovery of Nitrofurazone and Furazolidone added to normal milk

µg per ml added	Nitrofurazone				µg per ml added	Furazolidone			
	µg per ml found					µg per ml found			
	A	B	C	D		A	B	C	D
5	4.5	5.0	4.3	4.0	5	5.5	5.2	5.3	5.7
10	8.3	10.1	9.0	8.0	10	11.0	10.6	10.7	10.3
15	14.7	14.3	13.0	13.0	15	15.1	14.0	16.6	17.0
20	20.6	18.9	20.5	17.7	20	20.7	21.7	20.0	24.1

Table 5

The effect of the ratio of Nitrofurazone to Furazolidone content on their recovery when added to normal milk

µg Nitrofurazone added per ml	µg Furazolidone added per ml	Ratio	Per cent Nitrofurazone recovery		Per cent Furazolidone recovery	
			Theory	Found	Theory	Found
			23.6	12.0	2:1	95
23.4	4.7	5:1	93	95	140	142
3.9	19.4	1:5	115	128	96	96
11.3	22.4	1:2	102	102	99	117

Table 6

The effect of raw milk on the concentrations of Nitrofurazone and Furazolidone in 24 hours at room temperature

µg per ml added	Nitrofurazone			µg per ml added	Furazolidone		
	µg per ml found				µg per ml found		
	initial	4½ hr.	24 hr.		initial	4½ hr.	24 hr.
1.1	1.0	0.7	0.7	1.3	1.4	1.0	1.3
2.5	2.0	1.8	1.7	3.3	4.0	3.7	3.2

References

1. Jackson R.A. Vet. Med. 1959, 56, 379 - 381.
2. Mires M.H. and Chadwick R.H. Vet. News. 1947, 10, 3 - 5.
3. Mires M.H.J. Am. Vet. M. Ass. 1950, 117, 49 - 51.
4. Smith J.E. 1958. Private Communication.
5. Buzard J.A., Vrablic D.M. and Paul M.F. Antibiotics and Chemotherapy. 1956, 6, 702.
6. Cox P.L. and Heotis J.P.J. Agric. Food Chem. 1962, 10, (5) 402 - 403.
7. Smith I.M. and Scott W.N. To be published, 1963.

THE DETERMINATION OF FAT AND CARBOHYDRATE IN THE PASTRY OF MEAT PIES

by H. Dediccoat

(Public Analyst's Department, Town Hall, Burnley)

A method is described for the direct determination of the fat and carbohydrate content of pastry as a step towards the estimation of the meat content of meat pies.

The Food Standards Committee's Report on Meat Pies (H.M.S.O., 1963) recommends on page 8, para. 26 B "In analysing the pastry of a meat pie the excess fat above the ratio of fat to carbohydrate of 60:100 should be credited to the meat." This is to allow for any meat fat absorbed by the pastry.

The usual method of obtaining the carbohydrate content is by first determining the water, fat, protein, mineral matter, etc. and then the carbohydrate by difference from 100 per cent. The following method has been used in this laboratory and has been found to have advantages (See discussion of results).

Method

Sample preparation

The pastry, which has been separated from the meat pie and weighed, is thoroughly mixed by either passing through a mincer or by pestle and mortar depending on the consistency of the pastry.

Reagents

Dilute Hydrochloric Acid solution, approximately N
(Concentrated Hydrochloric Acid diluted ten times).

Pumice Powder.

Sodium Hydroxide aqueous solution, 10 per cent.

Phosphotungstic Acid aqueous solution, 10 per cent.

Bromo-phenol blue indicator solution.

Fehling's No. 1 and No. 2 solutions.

Methylene Blue aqueous solution, 0.5 per cent.

Light Petroleum, boiling point 40° - 60° C.

Procedure

Weigh 5 (\pm 0.01)g of prepared sample into a 250 ml wide-mouthed flat-bottomed flask, preferably fitted with a standard socket (B 34). Add from a cylinder 50 ml dilute hydrochloric acid and about 0.05 g pumice powder. Attach an air or water condenser and gently simmer under reflux for 2 hours. (In this laboratory the heating under reflux is carried out on the multi-unit solvent extraction apparatus). Cool the flask and contents to below 25° C, add sufficient bromo-phenol blue indicator to give a marked yellow colour and neutralise by the addition of 10% caustic soda solution (about 20 ml of caustic soda solution will be required). Add 10 ml of phosphotungstic acid solution, shake thoroughly and filter through a wet 12.5 cm No. 4 Whatman paper into a 500 ml graduated flask. Wash the residue on the paper at least four times with at least 100 ml of hot water, catching the filtrate in the same 500 ml flask.

Fat Content

Allow the filter funnel and its contents to drain, insert it into the neck of the 250 ml flask and transfer to the drying oven at 100-110° C. Dry for 2 - 3 hours. Remove the dry paper and contents from the funnel, carefully fold, (taking all precautions to prevent loss of fat) and place in an extraction thimble or wrap in a 15 cm filter paper. Transfer the enclosed paper to an extraction tube (Bolton or Soxhlet) attached to a tared flask. Wash the filter funnel and 250 ml flask thoroughly with light petroleum and transfer the washings to the extraction apparatus. Extract for at least one hour, evaporate off the solvent and weigh the residue, dried at 100° C.

Percentage fat in pastry = Residue x 20.

Carbohydrate Content

Make up the filtrate to 500 ml with water, mix and titrate into accurately measured 10 ml volumes of mixed Fehling's solution, using the Lane and Eynon technique and methylene blue indicator.

$$\begin{aligned} & \text{Percentage of carbohydrate present in the pastry} \\ & = \frac{500}{5} \times 0.045 \times \frac{100}{\text{Titration}} = \frac{450}{\text{Titration}} \end{aligned}$$

(10 ml mixed Fehling's solution = 0.050 g glucose = 0.045 g starch).

Calculation of the meat fat associated with the pastry

$$\begin{aligned} & \text{Total weight of carbohydrate in the pastry} \\ & \quad \text{Total weight of pastry} \times \text{percentage carbohydrate} \\ & = \frac{\text{in the pastry}}{100} \end{aligned}$$

$$\begin{aligned} & \text{Total weight of fat in the pastry} \\ & = \frac{\text{Total weight of pastry} \times \text{percentage fat in the pastry}}{100} \end{aligned}$$

"Meat fat" in the pastry

$$= \text{Total weight of fat in pastry} - 0.6 \times \text{total weight of carbohydrate in the pastry.}$$

This weight of meat fat is added to the total weight of meat obtained from an analysis of the filling of the pie and the total obtained is calculated as a percentage of the original pie.

Discussion of the results

The carbohydrate content determined by this method assumes that all the carbohydrate present is in a form which will convert to glucose by acid hydrolysis, i.e., that it is substantially starch. This is no less accurate than determining the carbohydrate by difference, as the determination by difference depends on the accuracy of 4 other determinations of which the moisture determination is probably the one with least accuracy and also on the factor used for converting nitrogen to protein (5.7 or 6.25). Results on about one dozen samples examined in this laboratory show that the carbohydrate content is 1 - 2 per cent less by this method than by difference. This is to the advantage of the manufacturer in that it will show a slightly higher meat content.

The acid hydrolysis releases the fat from the cereal of the pastry and ensures complete extraction, and as it is necessary to determine the fat no matter which method is used, one can

by this method obtain both fat and carbohydrate on the one sample weight.

The removal of the fat by filtration and drying avoids the troublesome emulsions usually associated with the Werner-Schmidt method and the extraction of the dry residue with light petroleum gives a colourless fat residue free from non-fatty extractives.

Application of the method to starchy filler in meat products

The method has been used over the past twelve years for the determination of starchy filler in all kinds of meat and fish products, usually as a check on the carbohydrate content obtained by difference in doubtful samples. The fat obtained by this method on meat products containing cereal filler has usually been 1 to 2 per cent higher than that obtained by direct extraction and the carbohydrate content usually ninety per cent of that obtained by difference.

If this method is used for sausage meat and other meat products containing about ten per cent starch the amount of sample should be increased to 10 g and the filtrate made up to 200 ml instead of 500 ml in order to give a satisfactory titration with Fehling's solution.

THE PAPER-CHROMATOGRAPHIC IDENTIFICATION OF FOOD DYES

by J.H. Shelton and J.M.T. Gill

(Public Analysts' Laboratory, 7 Tudor St., London, E.C.4.)

The method developed by Y. Yanuka et al.² has been extended for the identification of those food colours permitted in the United Kingdom and not included in the original paper.

Yanuka's method², using a single solvent, has been found of considerable value in our laboratory for the separation and identification of mixtures of colours frequently found in food-stuffs. The identification can frequently be made in one operation on one sheet of paper overnight, thus eliminating the necessity of a preliminary separation of the colours present.

Dyes Tested

Ponceau MX, Red 10 B, Erythrosine, Red 2 G,
Ponceau 3 R, Fast Red E, Orange G, Orange RN,

Yellow 2G, Yellow RFS, Yellow RY, Indigo Carmine, Violet BNP, Brown FK, Black PN, Chocolate Brown FB and Chocolate Brown HT.

Procedure

The colours are first deposited on and removed from wool in the manner described in the publication of the Association of Public Analysts¹ and subsequently spotted on to the large sheet of chromatographic paper in the following manner :-

Prepare sheets of Whatman No. 1 filter paper measuring 46 cm x 28 cm by cutting slots parallel with the larger side so as to divide the paper into 10 strips, each strip beginning $4\frac{1}{2}$ cm from the bottom and ending 10 cm from the top of the paper. Ignoring the outer two strips apply the aqueous solution of the dyestuff to the centre of each of the eight strips at a point $6\frac{1}{2}$ cm from the bottom edge of the paper, so that the diameter of the spot is not more than 1 cm. Successive amounts may be dried with warm air between application. To each of the dye spots add, in order, working from left to right, 10 μ l of one of the solutions listed below :-

- (1) N potassium hydrogen carbonate
- (2) 0.1 N potassium hydrogen carbonate
- (3) 0.01 N potassium hydrogen carbonate
- (4) buffer solution, pH 7
- (5) 0.01 N perchloric acid
- (6) 0.1 N perchloric acid
- (7) N perchloric acid
- (8) N sulphuric acid.

Along a line $4\frac{1}{2}$ cm above the bottom edge of the paper, apply in the same order at the centre of each strip similar amounts of the eight solutions above. Allow all the spots to dry and develop the chromatogram for 20 hours with the solvent system n-butanol, ethanol and water (1 : 1 : 1) by the ascending technique.

Allow the final chromatogram to dry and then view by ultra-violet light together with spots of known dyestuffs on similar paper. The curve so obtained, together with the appearance of the spots, is usually sufficient evidence by which to identify the colour present (See Figure 1).

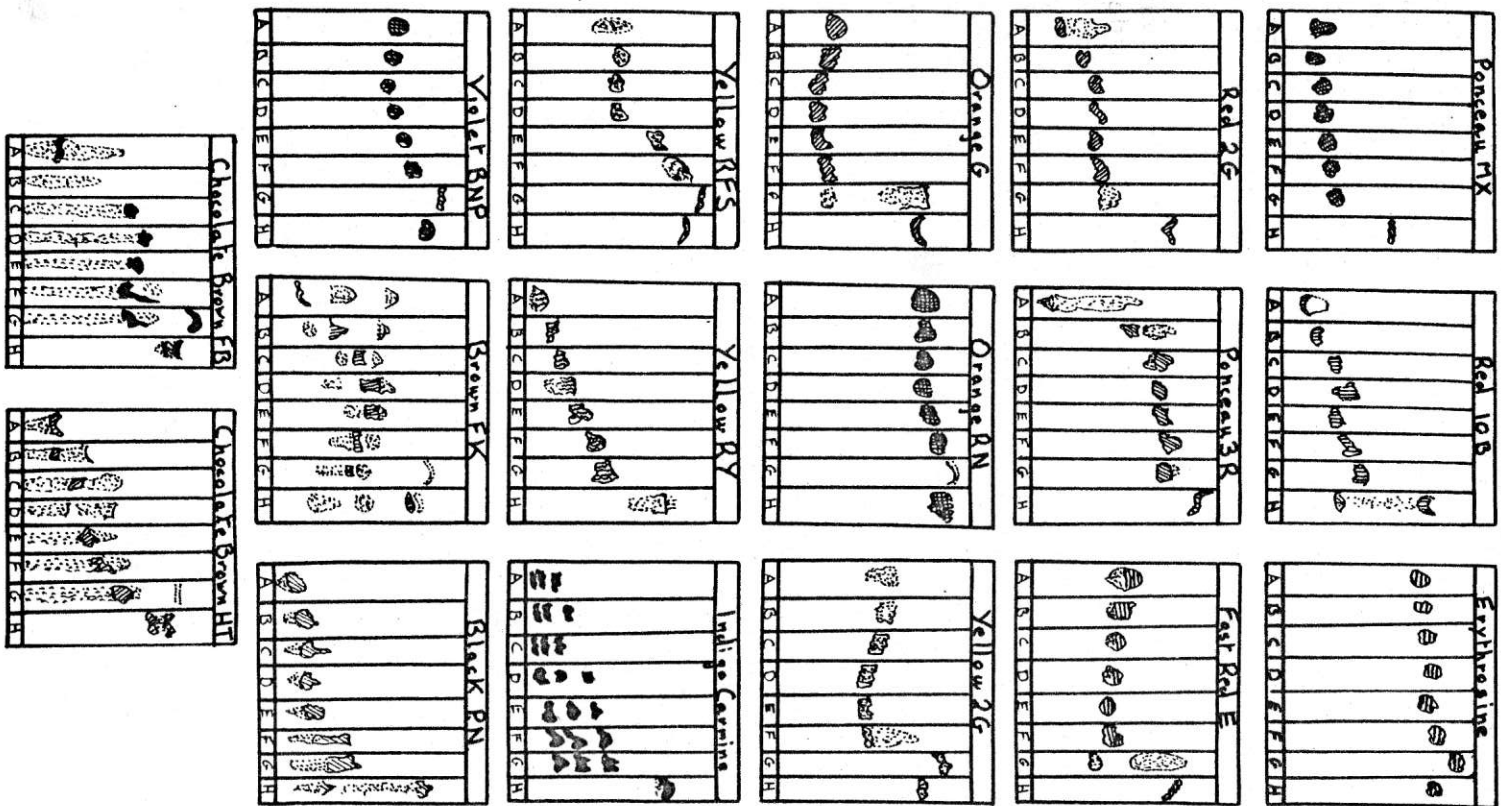


Fig. 1. The Chromatographic Pattern of Various Food Colours

Discussion of results

It will be noted that with Indigo Carmine, subsidiary spots are produced additional to the characteristic spot of the main colour. This phenomenon occurred with two commercial samples of the dyestuff, and it must be concluded that it is due to traces of isomeric dyes present in the original.

The colours Chocolate Brown FB, HY and brown FK (being mixtures of dyestuffs) exhibit more than one spot.

Thanks are due to Mr. Y. Yanuka for permission to publish this extension of his original work.

References

1. "Separation and Identification of Food Colours Permitted by the Colouring Matters in Food Regulations 1957," Association of Public Analysts, London, 1960.
2. Y. Yanuka et al., Analyst, 1962, 87, 791.

THE LEAD CONTENT OF GAME

by Eric C. Wood and E. P. Underwood
(Public Analyst and Deputy Public Analyst, Norwich)

Carcases of game killed by gunshot have been found to have a relatively high content of lead, even after removing visible lead shot and portions of shot. An analytical technique involving the maceration of rather large samples of flesh is described which much reduces the sampling error when contamination in the form of fine discrete particles is present.

We recently examined a sample of pheasant pâté and found it to contain 4 p.p.m. of lead. Following correspondence with the makers we were asked by them to investigate the lead content of whole pheasants that had been killed by gunshot. Two carcasses were submitted, one raw and the other cooked; the latter had already been searched for shot (the normal practice of the firm before making the pâté) and some had been removed. We were asked to examine separately (a) the flesh surrounding any shot or its track in either carcass; (b) flesh not visibly near any wound. A careful dissection was therefore carried out;

one whole shot was found in the cooked carcass and nine in the other. The four portions of flesh were each weighed, minced and thoroughly mixed. 10 g sub-samples were taken for analysis (except that the portion weighing only 3 g was taken in its entirety) and the results were as follows.

	<u>Flesh near shot</u>		<u>Rest of Flesh</u>		<u>Total Flesh</u>
	<u>Weight</u>	<u>Lead, p.p.m.</u>	<u>Weight</u>	<u>Lead, p.p.m.</u>	<u>Lead, p.p.m.</u>
Raw	15 g	21.7	464 g	5.0	5.5
Cooked	3 g	99	444 g	22.5	23.0

These results suggest that (a) lead shot entering the body contaminate the flesh surrounding their track with relatively large amounts of lead; (b) during the cooking process the lead migrates at least in part and contaminates the flesh in general. It was also realised, however, that if the contamination was in the form of minute particles of metallic lead, the sampling error involved in taking 10 g or less for analysis must be very large (1 particle weighing 0.1 mg would contribute 10 p.p.m.).

Accordingly, another raw carcass was examined by a different technique. The whole of the breast flesh, which had visibly been penetrated by many shot, was carefully removed by hand, and all shot found were picked out, every care being taken to ensure that none remained in the flesh. Flesh from the legs and other parts that had not apparently been penetrated was also removed separately; this was searched for shot also, but none was found. 100 g of each portion of flesh was then pulped with water in a high-speed macerator and the resulting slurry was allowed to stand for a short time before being poured off into a 2-litre flask, leaving behind about 100 ml. More water was added with stirring and the process of standing and decanting off was repeated, leaving behind the last 50 ml or so. This was repeated several times until the flask was nearly full and the residue consisted of water containing some visible small black particles but little flesh. The whole of this residue was examined for lead; the contents of the flask were made up to the mark and well shaken, an aliquot being then examined for lead.

	<u>100 g breast flesh</u> <u>(penetrated by shot)</u>	<u>100 g other flesh</u> <u>(not apparently penetrated)</u>
Slurry	2.2 p.p.m.	3.0 p.p.m.
Residue	0.91 mg = 9.1 p.p.m.	0.01 mg = 0.1 p.p.m.
	<u>11.3 p.p.m.</u>	<u>3.1 p.p.m.</u>

It was obvious, from the appearance of the slurry from the first portion of flesh, that fine particles of lead were present, although it is almost certain that no whole shot, or even large pieces of shot, had escaped detection when the dissection was done. It is interesting to note that the slurry from the breast flesh contained a relatively small amount of lead; clearly not more than traces of soluble lead compounds could have been formed by contact between the lead shot and the flesh, the lead contamination being almost entirely in the form of metallic lead. The fact that the apparently unpenetrated flesh contained slightly more lead than the breast flesh is somewhat anomalous, but was checked by duplicate analyses. It may be due to sampling error, or possibly the flesh concerned had been penetrated by shot without our realising it.

Some time later we examined a canned whole roast pheasant in jelly, from another manufacturer. The pheasant carcass was separated as completely as possible from the jelly and the flesh was then removed by hand from the bones and other inedible parts. The separated flesh, which weighed 814 grammes, was very carefully picked over and five whole lead shot were found and removed.

150 grammes of the flesh was then reduced to a fine pulp in a high-speed macerator with the addition of water and examined by the process described above. The lead content of the jelly was also determined. The results were as follows.

Slurry	4.3 p.p.m.
Residue	8.8 mg = 58.7 p.p.m.
Total in flesh	<u>63.0 p.p.m.</u>
Jelly	1.5 p.p.m.

There seems to be little doubt that much of the lead in the flesh was in the form of very fine particles of metallic lead.

Discussion

The results recorded above are few, and moreover, we have no 'controls', i.e., game that had not been shot. Too much importance must not be attached to them, and we would welcome further investigation of the matter by others. From the health aspect, there is little risk of lead poisoning from the consumption of game, even if our results are confirmed. The ordinary man or woman does not eat game very often, and one might expect that since the lead present is almost entirely in

the metallic form, most of it would either be detected in the mouth and rejected or if consumed would be excreted unchanged. But the fine particles found present in our investigation would be swallowed and might be soluble in the gastric juices.

There is, however, the legal aspect to be considered. Neither the 'Report on Lead' of the Food Standards Committee of the Ministry (H.M.S.O., 1954) nor the Lead in Food Regulations, 1961, contains any reference to game or other animals killed by gunshot, and the lead limit for otherwise unspecified foods of 2 p.p.m. must therefore apply to game and game products sold by retail. In other words, a game dealer who sells pheasants or grouse by retail is committing an offence if they contain even less lead than was present in the carcasses examined in this investigation. There is also the further problem of manufactured products such as game pâté; jugged hare and other dishes sold by caterers; and the like.

Finally, the question of sampling error on foods such as these requires emphasis. With any food that may contain fine particles of metallic lead, no sample of the order of 10 g can be expected to give accurate or reproducible results. The procedure described above, of pulping 100 g (more would be even better) and analysing separately the slurry and any deposit present, should be reasonably satisfactory, and is certainly more practical than the obvious alternative of submitting the whole of a sample of the same size to either wet combustion or dry ashing.

THE ENFORCEMENT OF THE LAW RELATING TO FOODS AND DRUGS IN THE UNITED STATES

by F. Bullock
(Formerly Public Analyst, Leicester.)

The work of the American Food and Drug Administration is well known to British Public Analysts. In recent years we have had first hand information of its activities on several occasions when the Deputy Commissioner, John L. Harvey, and Dr. Arnold J. Lehman, Director of the Pharmacology Division, have attended conferences in this Country, and Charles A. Adams paid visits to the U.S.A. and recounted his experiences to us on his return. Harold Monk also crossed the 'herring pond' a few years ago and, having met some of the F.D.A. personnel in their own

laboratories, brought back his personal views of some of their ideas and methods.

In February this year the A.P.A. "Monthly Report" contained a review by Chas. Adams of the Annual Report of the F.D.A. for 1961, and this furnished us with a useful reminder of the scope and volume of the work carried out by this 'unique organization.' The Report for 1962 is now available and is a most interesting document. Enforcement appropriation for the year 1963 is 28 million dollars, an increase of 5 million dollars over 1962, providing for an increase of staff of 542. Written in the same vivid style as the previous number, it takes the story a stage further in 52 pages of largely factual matter, much of it bordering on the sensational and as enjoyable and easy to read as much modern crime fiction. It would be difficult to review in a conventional way and perhaps selected extracts will serve the better purpose of whetting the appetite of at least some readers to obtain the Report for himself and read it in toto.

Without beating about the bush, the Report begins boldly with the statement ". . . consumer protection was dynamically on the move in 1962" and it proceeds to give a listing (sic) of some of the year's accomplishments :-

Congress on Medical Quackery held
 First seizures of unlabelled hazardous household substances
 Campaign on Packaging Violations intensified
 Drug Counterfeiting Ring ended
 Bootleggers of Dangerous Drugs jailed
 Incubator Reject Egg Handlers sentenced
 Physicians' Samples abuses curbed
 Overhaul of Special Dietary Regulations proposed
 Fake Medical Devices seized
 etc. etc.

Specific cases illustrating and justifying the above claims form a considerable part of the subsequent Report. 'Consumer Protection' in general and the debunking of 'Nutritional Quackery' in particular are principal and recurring themes. Three cases out of many are given here as being of particular interest to P.As:

- (1) Nature Food Centres, a corporation selling "natural and organic" foods, and two of its officers were convicted for shipments of food products represented for the treatment of many serious diseases. One of the defendants during public lectures in Philadelphia and Chicago, in an effort

to promote sales of the products, represented them to be effective for the treatment of conditions ranging from heart diseases and hepatitis to mental sluggishness, impotency and colds. Fines of \$5,000 for each individual and \$10,000 for the firm represented the highest total fines assessed during the year.

- (2) Royal jelly, a substance secreted by bees to feed the queen bee, has been promoted as a "miracle" ingredient to increase sexual vitality, extend the life span, normalize the growth of underdeveloped children, cope with the ailments of old age, improve memory, stimulate the appetite, etc. A vigorously contested trial of the seizure of a shipment of royal jelly, Jenasol RJ Formula, in 1962 gave F.D.A. its first court victory to establish such claims as unfounded. Following the decision in favour of the Government, the court granted its request for an injunction to prevent further misbranding of royal jelly by the firm. Numerous other actions against royal jelly products have been won by default.
- (3) In another case involving the product of bees, the Government seized $1\frac{1}{2}$ tons of honey misbranded with misleading claims for the treatment of waning virility, arthritis, weak heart, and as a cure for premature death. Lelord Kordel, a "health food" lecturer, is president of the distributing firm. The honey did not differ from ordinary honey.

One interesting section covering six pages refers to a message from the President of U.S.A. to a Congress on Consumer Protection in which he enumerated the following for 'Basic Consumer Rights' :

- (1) The Right to safety
- (2) The Right to be informed
- (3) The Right to choose
- (4) The Right to be heard

Under (1) the Report claims that "the safety clearance requirements of the Federal Food, Drug and Cosmetic Act" operated so as to ensure the ' . . . narrow escape by American families from the tragedy of grotesque deformity in babies caused in European Countries by a sleeping pill containing the drug Thalidomide.'

It adds that 2,200 chemicals are used in food and food packaging and have posed many research problems.

Under (2) the right to be informed is secured by ". . . affirmatively requiring that information needed by consumers for intelligent purchase and use be prominently and truthfully stated in product labelling. . . Enforcement reached an all-time high in 1962. There is no field in which misinformation is more vicious or more dangerous than that of medical quackery. Quackery is big business. . . Consumers spend needlessly more than a billion dollars a year on falsely represented foods, drugs and cosmetics." "Dietary products, vitamins and mineral and so-called health foods represent a major area of exploitation of the consumer through misinformation."

The right to choose is valueless without knowing what to choose and this is impossible in the confusion caused by exaggerated claims made for competing products. The Report does not say how the F.D.A. helps the consumer to know which product is exaggerating least, but it does say that "in 1962 85 seizures were made of products so labelled that important consumer information was hard to find, hard to read, or partly or entirely absent."

Implementing the right to be heard, numerous consumer conferences were held in 1962 for forum discussions directly with consumers. We can well imagine that the various organizations of American women made the most of these opportunities to voice their opinions. How unanimous or effective may have been the resulting recommendations is a matter for conjecture!

Under the heading "Radioactivity in Foods" we are told that the installation of radiological equipment was stepped up in 10 districts following resumption of nuclear testing by the Soviets in September 1961 and the number of samples of food examined for Strontium 90 was tripled (sic).

A considerable section is devoted to "Chemicals in Food" although the F.D.A. finds, as we do in this Country, that most cases of food poisoning are traced to bacteriological contamination. The following cases are typical:-

A consignment of biscuits was seized containing 6,700 p.p.r mineral oil. The biscuits were being brushed with a mixture of mineral and soybean oil to prevent them sticking. Later 140 gallons of extra heavy mineral oil labelled for both food and drug use (excellent non-fattening oil in salads!) were seized under the Food Additives Amendment.

The use of stilboestrol pellets for artificial caponization of chickens had created a cancer hazard from eating liver, skin and kidneys of treated birds. Firms who refused to discontinue the use of this process were penalised and 25,000 pounds of

stilboestrol treated poultry and 3,000 live chickens were destroyed in New York during the year.

A meat seasoning containing niacin and polyvinyl pyrrolidine (a stabiliser) were seized. The amount of niacin was far greater than would be used as a food supplement; severe flushing of the face is said to result when too much is consumed.

Lead contamination of puffed cereals resulted from continuing use of worn lead gaskets in the "popping guns."

Though devoid of wisecracks, the Report as might be expected has an unmistakable American flavour, and was clearly written with gusto by an enthusiast. The occurrence of long words is typically American, but there is no trace of affectation in their use. One feels that they are in some sort a measure of the conviction and keenness of the author. If in such an expression as "dynamically on the move" the present writer detects a faint suspicion of pleonastic tautology (see dictionary if necessary!) it is probable that he is not sufficiently familiar with the American idiom.

In a nation of 150 million people, where according to the Report "An estimated 500 million dollars is wasted by the public each year on unneeded vitamins and faddist food products foisted upon them by false representations", one wonders if even an organisation like the F.D.A. is not fighting a losing battle against the appearance of new rackets while suppressing existing ones as long as the policy of 'impulse buying and consumption' is fostered by commercial interests. The Commissioner himself has no delusions, stating in his final section ". . . But the coverage is still inadequate to appraise and act upon all of the problems existing in food, drug, device and cosmetic areas."

"The consumer is receiving increased recognition and will receive added protection in the year to come through new tools . . . more staff, better facilities and new laws. . ."

I commend all P.As to consider the worthwhileness of maintaining and cherishing such contacts as are already established between the F.D.A. and the A.P.A. Working in quite different environments our respective objects - protection of the consumer - are identical and our respective successes up-to-date probably of the same order of magnitude. Mutual enlightenment must inevitably result from continued contact, and as a concrete suggestion I recommend that the 1962 Report be perused by P.As generally, all of whom according to their line of specialisation will find one or more of the sections useful and thought provoking and having a direct bearing upon their own work.

REPORT ON FOODS EXAMINED BY PUBLIC ANALYSTS
FOR PESTICIDE RESIDUES DURING 1962

by J.H. Hamence
(Bernard Dyer and Partners,
Peek House, Eastcheap, London, E.C.3.)

About the middle of 1962 a questionnaire was sent to all public analysts requesting information regarding samples examined during 1961 for the presence of pesticide residues. The response to this questionnaire was very encouraging and the following is a summary of the results obtained.

During 1961, 1,145 samples of foods were examined by public analysts for the presence of pesticide residues. The samples included both home-grown and imported foods and were made up as follows:-

<u>Type of Food</u>	<u>No. of Samples</u>
Fruit	504
Tomatoes	101
Green Vegetables	128
Potatoes	38
Other Vegetables (Carrots, etc.)	151
Miscellaneous (Flour, Tea, etc.)	<u>223</u>
	<u>1145</u>

The nature of the examinations made and the results obtained are shown in the following summarised results:-

Arsenic, Lead and Mercury

<u>Type of Food</u>	<u>No. of Samples</u>		
	<u>Arsenic</u>	<u>Lead</u>	<u>Mercury</u>
Fruit	230	236	26
Tomatoes	24	22	48
Potatoes	35	8	-
Other Vegetables	45	25	-
Miscellaneous	138	181	5

No samples were found to contain mercury in excess of 0.1 part per million.

Only two samples contained arsenic and/or lead in excess of statutory limits, these were :-

- (1) Apples - 3.4 parts per million lead.
- (2) Apples - 3.3 parts per million lead and
1.6 parts per million arsenic.

A number of samples were also examined for copper but no excessive amounts were found.

Organic Insecticides

Where organic insecticides are concerned no attempt was made to examine each sample for every likely insecticide by individual tests for different compounds. The time involved for such a procedure clearly rendered this impossible.

The usual procedure therefore adopted by public analysts, in the results shown below, was to employ a preliminary biological sorting test using *Drosophila melanogaster* and in some cases *Daphnia*.

The conditions of the tests were designed to show if the residues present were significant, i.e., in excess of the tolerances allowed in the U.S.A. In many cases lesser amounts of residual pesticides would have been detected. If a positive response was obtained in the biological sorting test, a further detailed examination was made to determine the group to which the residual insecticide belonged and finally its true nature and the amount present.

The following samples were examined:-

Fruit	224
Tomatoes	79
Green Vegetables	128
Potatoes	11
Other Vegetables	108
Miscellaneous	29

Only in the case of six samples were positive responses in the biological test obtained. The results were as follows:-

Apples	1.4 parts per million D.D.T.
Apples	4 parts per million D.D.T.
Lettuce	1.3 parts per million organic phosphorus.
Lettuce	1.7 parts per million organic phosphorus.
Strawberries	Derris ?
Strawberries	Derris ?

Both organo-chlorine and phosphorus compounds were found to be absent from the two samples of strawberries and

from further tests and enquiries which were subsequently made it was concluded that the positive biological response was due to the use of derris.

The nature of the organo-phosphorus compounds in the two samples of lettuce was not identified owing to the smallness of the sample. These were imported lettuces and further samples from the same source were satisfactorily free from residues.

Organic Fungicides

Two hundred and twenty-five samples of fruit and four miscellaneous foods were examined for the presence of specific organic fungicides and negative results were obtained in all cases.

The thanks of the Council of the Association are due to all public analysts who replied to the questionnaire and enabled the foregoing summary of results to be prepared.

While the bulk of the work carried out on organic insecticide residues during 1961 was mainly confined to biological sorting tests, it is hoped that in the future it will be possible for public analysts to make more quantitative determinations of pesticide residues, particularly organo-chlorine compounds present in foodstuffs, even though the foodstuff in question passes the biological sorting test.

LOCAL AUTHORITIES' JOINT ADVISORY COMMITTEE ON FOOD STANDARDS

CODE OF PRACTICE NO. 2 - LABELLING OF BRANDY

The Code of Practice has been negotiated between the Local Authorities' Joint Advisory Committee and the Wine and Spirit Association of Great Britain (Incorporated) and the Brandy Shippers Association.

The Labelling of Food Order S.I. 1953 No. 536, and subsequent amendments lays down that every alcoholic liquor shall include on the label of the bottle in which it is prepacked its appropriate designation. Appropriate designation is defined in the Order as "a name or description . . . which shall indicate to a prospective purchaser the true nature of the product to which it is applied." It is, moreover, an offence under the Food and Drugs Act, 1955, to label or advertise a product in terms which falsely describe it, or which are calculated to mislead as to its nature, substance or quality.

The Local Authorities' Joint Advisory Committee on Food Standards have discussed with The Wine and Spirit Association of Great Britain (Incorporated), and the Brandy Shippers Association the application of this legislation to the description of brandy.

While only a court can decide with finality what is the law, the following agreed code of practice, which has the approval of the Joint Advisory Committee has been prepared for the guidance of the trader. It is felt that observance of this code will help traders to avoid committing offences against the appropriate legislation, and will be in the general interest of fair trading practice - a protection both to the legitimate trader and the consumer.

However, the Council of Europe are at present considering definitions of spirits including brandies and an international definition may ultimately be issued. It is considered unlikely that there will be any material difference in substance from descriptions now following.

I. Descriptions to be used

(1) The only spirit which is entitled to be imported, manufactured or sold in Great Britain under the unqualified description "brandy" is the distillate of the fermented juice of fresh grapes without the admixture of any other spirits.

(2) Cognac Brandies or Armagnac Brandies or Cognac or Armagnac are brandies as defined above, produced in the Cognac and Armagnac regions, respectively. Both these areas and the methods of production are prescribed by the laws of the French Republic. These names should not be used in the description of any brandies produced elsewhere, or by any method other than that prescribed by French law.

(3) Fruit Brandy. - Products derived exclusively by the distillation of the fermented juice of one fruit, and containing no brandy, as defined at (1), shall be described as "... Brandy" and this should be followed by the words "a spirit distilled from..." The blank space would in each case be filled by the name of the fruit used - e.g., "Plum Brandy, a spirit distilled from fermented plums."

(4) No exception will be taken to the established names for liqueurs - e.g., Cherry Brandy and Apricot Brandy - provided that they do contain substantial proportions of genuine brandy as defined at (1). It is agreed that this would be met if not less than 20 per cent of the spirit

content of the product is genuine brandy. In the opinion of the Ministry and the trade associations concerned any lesser proportion of genuine brandy would constitute a false trade description under the "Merchandise Marks Acts, 1887 to 1903."

(5) The description "brandy" or "fruit brandy" should only be applied to potable spirits derived from the distillation of the fermented juice of fresh grapes or from distillation of the selected juice of other fruits. Imported products distilled from other materials and made to simulate brandy, and imported compounded spirits made to simulate brandy should be labelled and sold as "imitation brandy."

(6) Mixtures of genuine brandy, as defined at (1), and imitation brandy should be labelled and sold as at (5), above, e.g., "imitation brandy." The labels may bear a description stating that the contents of the bottle are a blend of wine brandy and imitation brandy, e.g., "a blend of Israel Brandy and highly rectified spirit" (provided, of course, that the brandy is the greater of the two ingredients, when both are calculated at the same alcoholic strength).

(7) Potable spirit distilled from the skin and pulp of grapes, after the withdrawal of the juice or wine therefrom, should be described as "Marc Brandy."

NOTES FOR THE GUIDANCE OF THE TRADE

II. The Importer

(1) The importer, for his own protection, should demand from his overseas supplier a certificate of origin and certificate of age covering all shipments of brandies, fruit brandies, marc brandies, or imitation brandies. These certificates should be issued or verified by the official authority in the country of origin and conform to the requirements of paras. 2.260-2.263 of H.M. Customs and Excise Warehousing Manual.

(2) The trader or dealer is entitled to require his importer to produce for his inspection the certificate under which brandy sold to him has been shipped.

The regulations of the French Government are as follows:-

The authorities have for many years insisted on certain certificates or acquits in respect of all exports

of brandies and alcohols. They are as follows :-

- (1) Acquit Jaune d'Or (Yellow Permit). - Under a Decree dated 25th August, 1937, only spirits distilled from wines produced in the delimited areas of Cognac or Armagnac are entitled to the controlled appellation of Origin "Cognac" or "Armagnac," and can benefit by the use of this permit for shipment overseas or for interior circulation.
- (2) Acquit Blanc (White Permit). - This permit is reserved for spirits distilled exclusively from wine, cider or fruits. This permit indicates and guarantees the nature of the substance from which the spirit has been obtained and the customs also guarantee that the spirit was distilled under their supervision.
- (3) Acquit Rose (Pink Permit). - This permit gives no indication or guarantee as to the source or nature of the substance from which the spirit has been distilled.

As will be seen from the above, the Acquit Jaune d'Or and Acquit Blanc (Eau de Vie de Vin) are the only guarantee of pure brandy.

III. Vintage or Age Statement

A new practice under this heading was announced in 1962 on which guidance was sought concerning the labelling of brandy with a vintage or age statement. The ruling by the Cognac Authority, namely : The Bureau National Interprofessional du Cognac, is as follows :-

- (1) The machinery for granting certificate of age for brandy does not extend beyond the maximum of five years; but in the case of the United Kingdom it is only three years. Any brandy declared to be older than this can carry no recognised proof beyond the good faith of the shipper. There is nothing new in this as it has been the case for many years past, but is perhaps not generally known to the trade in this country.
- (2) What might be considered extravagant declarations of ages beyond the scope of this certificate or unreliable claims to a "vintage year" come under the responsibility of the "Service de Repression des Fraudes" who have recently reaffirmed that they actively dispute any such claims that cannot be backed by irrefutable proof.

(3) We are advised by the Bureau National that the existing system of identifying the actual age of stocks makes the provision of such proof very difficult, and it is for the shipper concerned to substantiate beyond dispute any claim as to age that he may make.

To summarise the position, there is no French law forbidding the export of brandy bearing a statement of age over five years, or a vintage date. Where such age is printed on the labels of brandy exported in bottle the authorities in Cognac will themselves have controlled the authenticity of the statement; where brandy is shipped in cask for bottling in this country, however, the importer should for his own protection require that the statement of age which he may wish to make on his own label should be substantiated by a similar declaration on the supplier's invoice.

A copy of this code of practice has been circulated to the appropriate department of the governments of wine-producing countries and the secretariat of the Council of Europe.

Additional copies of this Code may be obtained at a charge of 2s. 6d. per copy from the Joint Secretaries to the Local Authorities' Joint Advisory Committee on Food Standards, Victoria Station House (2nd Floor), Victoria Street, London, S.W.1

1st November, 1963.

CODE OF PRACTICE NO. 3 - CRAB MEAT CONTENT IN NORWEGIAN CANNED CRAB PRODUCTS

1. We are directed by the Local Authorities' Joint Advisory Committee on Food Standards to state that an agreement has been reached between the Association of Public Analysts and the Research Laboratory of the Norwegian Canning Industry on the method of determination of the amount of crab meat in Norwegian canned crab products. This Code of Practice embodies the terms of the agreement negotiated by the Association of Public Analysts on behalf of the Local Authorities' Joint Advisory Committee on Food Standards and has been approved by the constituent bodies.

2. The terms of the Code of Practice are as follows :-

(a) To estimate the meat content of canned crab products it is necessary to determine the protein content of the sample and then convert this to raw crab meat

content by using a protein factor based on the assumed protein content of genuine crab meat.

(b) In calculating the crab meat content of canned Norwegian crab meat products 15 per cent of protein shall be regarded as equivalent to 100 per cent crab meat. The figure of 15 per cent includes fat content of any crab meat so that the fat content of the crab product is not added to the meat content as calculated from the protein.

(c) Provided all the nitrogen in the canned crab product is derived from crab meat the amount of crab meat in the product is to be calculated in accordance with the formula

$$\frac{N \times 6.25}{15.0} \times 100$$

where N is the nitrogen content (expressed in a percentage) determined by the normal Kjeldahl process and the figure 15.0 is the protein referred to above.

(d) Any analyses and calculations are based on the whole of the edible part of the crab.

(e) If the product contains a large amount of cereal filler a correction is to be made to allow for the nitrogen content of the cereal filler. This is calculated by doubling the carbohydrate content and taking 1 per cent of the product as nitrogen.

3. Additional copies of this Code may be obtained at a charge of 1s. per copy from the Joint Secretaries to the Local Authorities' Joint Advisory Committee on Food Standards, Victoria Station House (Second Floor), Victoria Street, London, S. W. 1.

2nd December, 1963.

(For the constitution and functions of the Local Authorities' Joint Advisory Committee on Food Standards, see J.A.P.A., 1963 (2nd Quarter), 1, 50.)

BOOK REVIEWS

DETERMINATION OF TRACE ELEMENTS, WITH SPECIAL REFERENCE TO FERTILISERS AND FEEDING STUFFS. Prepared by the Trace Elements in Fertilisers and Feeding Stuffs Sub-Committee of the Analytical Methods Committee of

the Society for Analytical Chemistry. Published by W. Heffer & Sons Ltd. Pp. vii + 39. 1963. 21/- (special price of 12/6d. to members of the S.A.C.)

The sub-committee appointed in November 1958 included practising analysts well known in the field of trace analysis techniques. The methods described are acknowledged to be complementary to those given in the book "Recommended Methods for the Analysis of Trade Effluents" published in 1958.

Whilst the importance of sampling is emphasised it is considered that the existing regulations (S.I. No. 1165. 1960) should be adequate for obtaining a representative sample. It is possible to determine on the same solution obtained by the general preparation of the sample nine of the fifteen elements considered. These elements include boron, calcium, chloride, chromium, cobalt, copper, fluorine, iodine, iron, magnesium, manganese, molybdenum, nickel, selenium and zinc. A general oxine extraction method followed by paper-chromatographic separation has also been described as a useful preliminary test when more than one constituent is to be determined in the same sample.

In common with previous books of this nature the format is convenient and the working details are precise. The methods given have all been thoroughly investigated and are the result of many collaborative tests of potentially useful methods. For each estimation the methods described are as simple as possible, that is, involving in the main chemical procedures. Indeed, most elements may be estimated by a colorimetric procedure and therefore absorption spectrophotometry is the only physical method which is absolutely necessary. In addition, flame photometry (and its limitations) is mentioned for calcium, a fluorimetric method is described for selenium and a spectrochemical method is given for magnesium. Careful consideration must have been given to the general applicability of the methods since each one may be carried out with the minimum of specialised equipment and no mention is made of the polarograph or atomic absorption apparatus. In the laboratory of the public analyst and official agricultural analyst, who may also be consultants, this book should be welcomed as a useful collection of methods already well tried and recommended. The non-routine laboratory will find that these methods can also be applied to the analysis of many other substances and the useful notes and references at the end of each procedure are very valuable as any practising analyst will appreciate. The sub-committee are to be congratulated for producing such a valuable book of methods and at such a small cost.

L.E. Coles.

BELL'S SALE OF FOOD AND DRUGS. Service Volume Issue No. 7. Butterworth and Co. Ltd., London. 1963. Pp. 284. 37s. 6d. (+ 1s. 4d. postage). Main work combined with Service Volume including present Issue, £6. 18s. (+ 4s. postage).

Once again the indefatigable Mr. O'Keefe has produced a "Service Volume" issue to bring his famous 13th edition of 'Bell', in conjunction with earlier Service Volume issues, up-to-date to the 1st August 1963. It speaks well not only for the author but also for the publishers that the new issue is available less than three months later.

The only Statutory Instrument of importance to Public Analysts included in issue No. 7 is the Soft Drinks Regulations, 1963, and Section 4 of the volume (Ministry Circulars, etc.) contains nothing of moment. But Section 1, the 'Noter-up' to the main volume, contains much new matter, including reports of Appeal cases that have been heard since the last issue, and a very full summary, including long verbatim quotations, of the Report on Meat Pies of the Food Standards Committee. It is a pity, incidentally, that the Index refers only to Sections 2, 3 and 4 of the Service Volume and does not include the 'Noter-up', so that (for example) the many references to meat in the index do not help one to find the Food Standards Committee's Report on Canned Meat Products. Mr. O'Keefe would place all his readers under an even greater debt of gratitude if he could provide a complete index with the next issue; but even as it stands, issue No. 7 is as useful and as indispensable to the Public Analyst (and indeed to every firm of food manufacturers) as its predecessors.

Eric C. Wood.