

ANALYTICAL AND TECHNICAL CONTRIBUTIONS

LEAD IN FOOD DERIVED FROM
TINNED STEEL FRYING PANS

by G.S. Meadows
(City Laboratory, Salford, Lancs.)

It was thought that food cooked in a tinned steel frying pan might have been responsible for symptoms resembling those of lead poisoning in a person who had eaten several meals prepared in the pan. The pan was therefore submitted to the laboratory for examination, and, in view of the surprising results obtained, several more tinned steel frying pans of different brands were also purchased and examined.

The proportion of lead in the tinning on each of the pans was found by carefully scraping the underside of the base and determining the lead in the scrapings by the usual sulphate precipitation method. In addition, the 'complaint' pan and another having a similar proportion of lead in the tinning, were examined more fully by cooking test meals in them and determining the lead in the meals, and also by finding the coating weight on the upper surface of the base. The test meals consisted of bacon, egg, and tomato, and were cooked over a gas flame; the conditions were kept as uniform as possible throughout the cookings, and the flame was a size that would probably be normally used in the home. During cooking it was noticed that there was a tendency for the tinning to become molten and, particularly in the case of the complaint pan, for the molten tinning to run and form thicker patches on the base of the pan.

Results

Table 1 gives the results of the various determinations and also any claim on the labels as regards the quality of the tinning. It can be seen that in only two cases was there a negligible amount of lead, a definite claim being made about the quality of the tinning in each case, whereas the other four all contained a high proportion of lead.

TABLE 1
Examination of Tinned Steel Frying Pans

<u>Pan</u>	<u>Labelled</u>	<u>Per cent Lead in Tinning</u>	<u>Weight of Tinning</u>
A	"Tinned Steel"	54.9	109.5
B	"Tinned Ware"	51.3	72.4
C	"Tinned Steel"	52.0	
D	"Pure Tinned Steel"	Less than 0.5	
E	-	51.7	
F	"Pure Tinned, Guaranteed free from Lead"	Less than 0.5	

The last column shows in ounces per basis box the thickness of the coating on the upper surface of the base.

TABLE 2
Lead Content of Test Meals

<u>Pan</u>	<u>Lead in Test Meals, Parts per Million</u>	<u>Average</u>
A	50, 80, 35.	55
B	7.3, 11.	9.2

Each result represents a separate test meal.

The amount of lead in all the five test meals (Table 2) was very much in excess of the general limit of two parts per million for the lead in food, and although the proportion of lead in the tinning was approximately the same for both pans, the meals cooked in pan A showed a much higher lead content than those cooked in pan B. Microscopical examination showed some of the lead in the meals to be in the form of particles of very finely divided metal, and this, together with the fact that much more lead was taken up from the pan with the thicker lining, suggests that some of the lead is acquired mechanically rather than chemically due to the food being in contact with the semi-molten lining.

A meal weighing 150 grammes cooked in pan A would, on the average result, give a total intake of approximately 8 milligrammes of lead. It is interesting to note that this is some four times the figure of 1 to 2 milligrammes which is generally accepted as the maximum amount that can be ingested daily without toxic effects.

Conclusion

The Food Standards Committee of the Ministry of Food recommended in their Report on Lead in 1954 that "the sale of domestic cooking equipment lined with lead - containing tin or pottery glaze should be controlled".

These findings indicate that it is still not uncommon for frying pans to be lined with lead - containing tin and that there appears to be no control over their sale. Although much of the lead taken up by meals cooked in these frying pans is probably not in a soluble form, there nevertheless exists a possible lead poisoning hazard through using them, a hazard which is increased if the weight of tinning is unduly heavy.

RUM CONTENT OF RUM BUTTER

by J. G. Sherratt and R. Sinar

(Public Analyst's Laboratory, Warrington, Lancs.)

Rum butter is a confection made traditionally by mixing together sugar, butter, rum and, sometimes, spices. It is popular in the north country, and particularly in Cumberland and the Lake District. Latterly its popularity appears to have extended geographically, and a compound described as "Genuine Cumberland Rum Butter" is made by at least one firm in London, and sold at chain stores throughout the country.

No legal standard is prescribed for the amount of rum that should be present in rum butter. Recipes in cookery books for preparing home-made rum butter usually recommend the addition of a quantity of rum that would give a concentration of proof spirit in the finished article substantially higher than 2.0 per cent. If a similar formula were used in making commercial rum butter, the sale of the confection would, in theory at least, be restricted to licensed premises. Rum butter is, in fact, sold regularly from unlicensed premises, and a problem that confronts Food and Drugs Authorities is to decide the minimum quantity of rum that may reasonably be expected by a purchaser. Two per cent of proof spirit is equivalent to 3 per cent of rum (35° u.p.). Clearly, it would not be reasonable to demand as a minimum standard under the Food and Drugs Act a concentration of rum that would also be a maximum limit under Customs and Excise Regulations, because such a standard would leave no latitude for manufacturing variation or loss. On the other hand, tasting tests suggest that the presence of less than 1 per cent of rum can barely be recognised by an ordinary palate, particularly if the butter component has the strong flavour characteristic of farm butter from which the confection was traditionally prepared.

Table No. 1 gives the concentration of rum, of 35° u.p., found in 34 samples of rum butter analysed in this laboratory under the Food and Drugs Act since 1958. The average rum content of all the samples was 2.5 per cent. Categories (a), (f) and (g) deviate so far from the average that they may reasonably be excluded as non-typical. The average of the remaining categories is then 2.9 per cent of rum.

TABLE 1

	(a)	(b)	(c)	(d)	(e)	(f)	(g)
Rum Content	below 1.0%	1.0% to 1.9%	2.0% to 2.9%	3.0% to 3.9%	4.0% to 4.9%	5.0% to 5.9%	6.0% to 6.9%
No. of Samples	5	7	7	4	9	1	1

Total number of samples = 34

Mainly on the basis of these data, it was suggested in a recent prosecution for the sale of rum butter alleged to be deficient in rum that a reasonable standard for a commercial article at the time of sale would be 2 per cent of rum.

Rum butter is perishable and does not maintain its characteristic flavour during long storage. Table 2 shows the loss of proof spirit in two samples kept in the laboratory and analysed at intervals over a period of approximately ten weeks. Sample (a) was kept in a plastic jar with a tight lid; sample (b) was contained in a screw-capped glass jar with a large air space above the rum butter. The lids of the containers were removed before each test, and the samples stirred. Hence, the atmosphere above the rum butter was changed each time the containers were opened for testing. Under these conditions the sample in the plastic container lost 30 per cent of its proof spirit in 10 weeks; the sample in the glass jar lost 77 per cent.

TABLE 2

<u>Date of Test</u>	<u>Sample (a).</u>	<u>Sample (b)</u>
	<u>- Proof Spirit, % -</u>	
16. 11. 62	1.02	1.22
4. 12. 62	0.96	0.85
10. 1. 63	0.77	0.44
29. 1. 63	0.71	0.28

During the hearing before magistrates of the case referred to above, the defendants agreed that the proof spirit content of rum butter could diminish on storage. An expert witness called on their behalf stated that the apparent loss of alcohol was due primarily to fat-splitting of the butter and subsequent esterification by combination of acid and alcohol, although no conclusive

analytical evidence in favour of the theory was produced. This witness regarded esterification of the alcohol as "maturing", and the defendants claimed that, as the manufacturer had mixed rather more than 2 per cent of rum with butter and sugar in making the rum butter, they as the vendors had not committed any offence in selling the "matured" article, which, at the time of purchase, contained proof spirit equivalent to only 0.5 per cent of rum. Ultimately the magistrates accepted the prosecution's contention that, even if the theory of esterification was valid, which was not admitted, the article at the time of sale did not contain 2 per cent of rum; moreover, esterification of the alcohol, if it had occurred, must have altered the characteristic flavour. The defendants were fined £10. and costs.

The interesting theory that ethyl alcohol was not lost during storage, but had "matured", appeared to warrant further investigation. The main channels by which alcohol can be lost on storage are:-

- (a) Evaporation.
- (b) Oxidation to aldehyde.
- (c) Oxidation to acetic acid.
- (d) Combination with organic acids to form ethyl esters.

Other theoretical possibilities of change appear improbable under the ordinary conditions of room-temperature storage.

In order to investigate these possibilities, sample (b) in Table 2 was selected for further examination. Approximately 36 g were steam distilled in a closed system to avoid loss of alcohol. The distillate was made up to 100 ml, and determinations of alcohol (by the Kozelka and Hine method), acidity (titration with standard alkali), aldehyde (with Schiff's reagent), and ester-alcohol (after hydrolysis with caustic potash), were made. The results were as follows:-

Alcohol present 16th November, 1962,	
as proof spirit	1.22% w/w.
<u>4th February, 1963</u>	
Alcohol, as proof spirit	0.14 per cent.
Alcohol, as proof spirit + ester alcohol	
after hydrolysis with caustic potash.	0.13 per cent.
Alcohol due to ester	negligible
Acidity, as acetic acid	0.01 per cent.
Aldehyde	none detected, i.e.
	less than 0.02%.

The above figures lend no support at all to the theory that the apparent loss of alcohol during storage was due to chemical

change. The inference may be drawn that, unless rum butter is contained in an hermetically sealed jar, with little air space, very substantial loss of alcohol may occur during a period of a few weeks. Manufacturers might safeguard themselves from the consequences of deterioration by a cautionary label.

THE IDENTIFICATION OF OIL IN BREAD

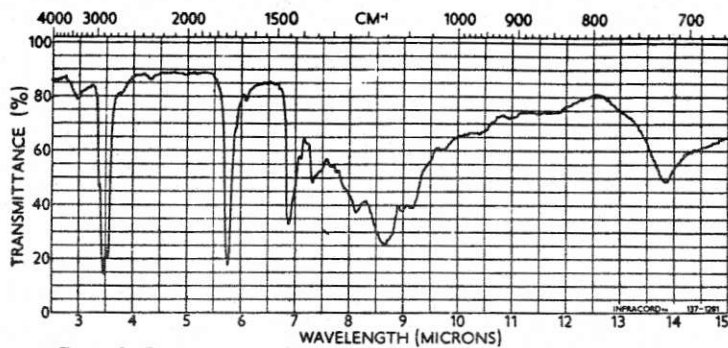
by L. E. Coles
(City Laboratory, Cardiff, Glam.)

A sample of bread, complained of by a private purchaser, contained a small area of dark green discoloration. Preliminary extraction with petroleum ether (40° - 60° C.) and saponification of the residue yielded 11 mg of unsaponifiable matter. Spot-tests showed the presence of traces of copper, iron and zinc on the bread itself.

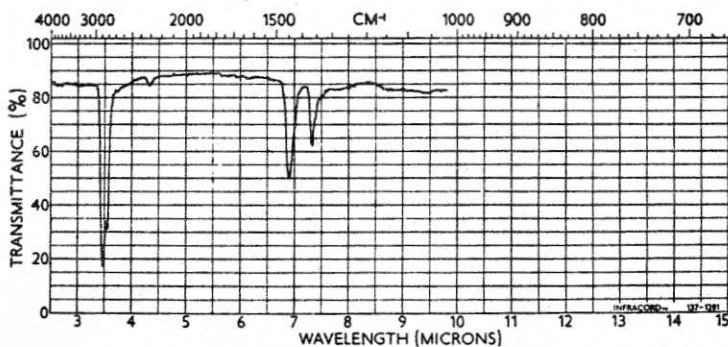
Communication with the bakery concerned revealed that they understood that all the lubricating materials used were edible, and supplied a sample of each one for examination. They were labelled as follows:-

- A. Ground-Nut Oil
(See Graph I). This is, of course, edible and saponifiable, and used for greasing the trays before baking.
- B. Edible White Oil
(See Graph II). This is unsaponifiable, resembling liquid paraffin, and would be more properly described as Refined Mineral Oil. It is used for certain moving parts of the machinery in the bakery.
- C. Alvania Grease
(See Graph III). This contained a small amount of lime soap, had a high viscosity and was largely unsaponifiable. This is also used for certain moving parts of the machinery in the bakery.

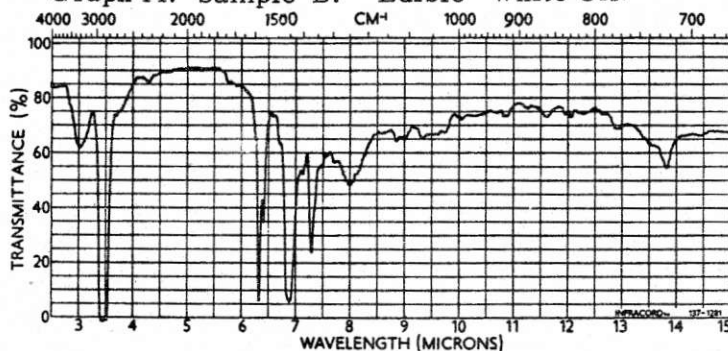
It was evident from observation of the unsaponifiable matter that it was the material described as "Edible White Oil", but the use of infra-red spectroscopy proved beyond doubt that this was the case. Graph IV is the I.R. spectrum of a few milligrams of the unsaponifiable matter extracted from the bread and is identical with Graph II, the I.R. spectrum of the so called "edible white oil".



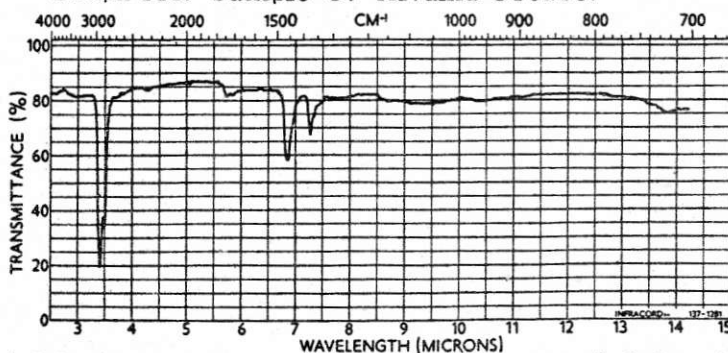
Graph I. Sample A. Ground-Nut Oil.



Graph II. Sample B. "Edible" White Oil.



Graph III. Sample C. Alvania Grease.



Graph I V. Unsaponifiable matter from bread. (cf. Graph II)

THE DETERMINATION OF
THE ALMOND CONTENT OF MARZIPAN:
COMPOSITION OF ALMONDS.

by A. Houlbrooke,
(County Laboratory, Stafford)

The results of the analysis of 219 samples of Ground Almonds examined under the Food and Drugs Act in the Staffordshire County Chemical Laboratory since mid-1950 and reported as genuine confirm the figures for oil content published by Monk (J.A.P.A., 1963, 1, 20). Estimations of protein were not carried out but the ash content was determined on 190 of these samples and this determination provides an equally satisfactory basis for estimating the almond content of Marzipan.

The results obtained are as follows:-

	<u>per cent</u>	
	<u>Oil</u>	<u>Ash</u>
Highest	65.6	3.50
Lowest	52.7	2.40
Average	58.29	2.92

The results also confirm the comparatively small degree of variability found by Monk. 196 of the 219 samples, or 89.5%, gave oil contents within the range of 55.5% to 60.4% while 170 of the 190 samples, or 89.5%, gave ash contents within the range of 2.65% to 3.24%.

FIRST REPORT OF THE A.P.A. PESTICIDE PANEL
(Circulated to A.P.A. members, May 1963).

This Panel was formed to investigate methods of examination which could be applied to food stuffs to detect, and if necessary determine, pesticide residues.

The members of the Panel are as follows:-

T. E. Rymer (Convener), J. B. Aldred, A. C. Bushnell, L. E. Coles, R. A. Dalley, H. Dediccoat, C. N. Grange, P. S. Hall, A. Houlbrooke, W. E. Jones, D. D. Moir, J. D. Peden, J. G. Sherratt, R. Sinar, E. P. Underwood, R. W. Watridge.

In view of the current interest being shown by the Press, in Parliament and by the public at large, it is thought that many Public Analysts will feel that it is their duty to be in a position to examine food stuffs without delay. It is thought, therefore, that they would like to hear the results of the preliminary

investigations carried out by this Panel, although these are by no means complete, so that they may be in a position to carry out tests for themselves during the coming spring and summer. There follows, therefore, a report of the first series of collaborative tests carried out by the Panel.

It was decided in the first instance to carry out collaborative tests using the biological sorting tests described by Hall¹. Some time had necessarily to lapse before some members were in a position to carry out this work since it was necessary for them to establish a colony of the test insect. Members were requested in the first place to determine L T 50 values for Dieldrin for residues of 1.0 and 0.1 μg contained in 5 mg of paraffin wax, and also to determine the L D 50 for 24 hours exposure, using in each case the procedure mentioned above. Secondly, they were requested to carry out a recovery experiment using potatoes to which had been added 0.1 p.p.m. of dieldrin. (The U.S. Food and Drug Administration establishes a tolerance of 0.1 p.p.m. of Dieldrin for potatoes).

Breeding Drosophila

After some teething troubles most members were able to establish a satisfactory colony although it was found that many casualties occurred during the cold weather due to inadequate heating arrangements.

Media

About half the members who reported found the Rothamsted medium to be satisfactory, although one or two found that it could be improved by the use of slightly less water than the formula recommends.

Alternative formulae which have the merit of simplicity were also suggested:

1. Mashed banana with or without the addition of a little maize meal to make it less wet.
2. A mixture of "Farex" and treacle.

In both cases the media are preserved by the addition of 0.1 per cent of methyl parahydroxy benzoate.

Determination of L T 50 and L D 50 Values

Results of the collaborative tests are shown in the accompanying table. These show a very wide inter-lab. variation. An experiment carried out by one of us indicates that the temperature at which the test is carried out may be quite critical, and

it is probable that part of the variation is due to this cause, since some members have not yet achieved adequate temperature control of the test room. It cannot in any case be expected that different breeds of the test organism produced quite independently should show precisely the same response. There would appear to be no significant difference between the vestigial and the winged variety of drosophila from the point of view of susceptibility. It is considered that the wide inter-lab. variation is not too important but it indicates quite clearly that control tests must be carried out with each experiment.

Recovery of Dieldrin added to Potatoes Extraction with dichloro methane

A number of members reported that intractable emulsions were formed. This is unfortunate, but it is usually possible with patience to achieve a satisfactory separation. Alternative extraction procedures have been suggested (e.g. acetone); dichloro-methane, however, was used in this instance because it forms the first step in the Laws & Webley² procedure which is thought to be generally applicable to both organo-chlorine and organo-phosphorus pesticides.

Recovery

The results obtained for the dieldrin extracted from potatoes are shown in the attached table. Once again a wide variation between laboratories is shown and in most cases a very poor recovery. It is worth noting, however, that three laboratories show substantially 100 per cent recovery when working with the smallest aliquot, i.e. equivalent to 0.1 µg of Dieldrin, and that the recovery experiment contained a significantly lower weight of residue than the experiment using paraffin wax. The recovery falls off very significantly with increasing aliquot, i.e. with increasing weight of residue in the test jar. It is shown that very little advantage in sensitivity is obtained by increasing the aliquot.

The results of one member (B) must be mentioned since they appear to indicate that the potato extract shows a synergistic action. No other member observed this. The member concerned records that an extract of the potatoes in question without any added Dieldrin showed no toxic effects.

A.P.A. PESTICIDES PANEL

Summary of Results of
Collaborative Experiment using *Drosophila*

	Analyst						
	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>	<u>G</u>
Type of Fly*		V		V	W	W	V
Test Jar (size in oz.)					4	4	4
A. Test Jar containing 5 mg of paraffin wax.							
L T 50, 1 µg Dieldrin (hr.)	5	16	16	4½	6-8 ^a	3½ ^b 16 ^c	7½
L T 50, 0.1 µg Dieldrin (hr.)	31	96	64 72+ 72+	23	40-55 ^a	24 ^b 62 ^c	29
24 hr. exposure, L D 50, µg Dieldrin	0.1	0.6	0.5	0.1	0.3 ^a	0.1	0.13
B. Tests using extracts of potatoes containing 0.1 p.p.m. Dieldrin, 50 g to 100 ml of extract.							
2 ml extract, 0.1 µg. Wt. residue in jar, mg.		0.23 0.4		0.2	0.6	0.3 0.3	} un- read- able
L T 50, hr.		16 6¾		24	48	25 62	
10 ml extract, 0.5 µg. Wt. residue in jar, mg.		1.15 2.0		2.9	3.0	1.7 1.7	} 10% to 20% reco- very
L T 50, hr.		8¼ 4¾		7.5	12	27 52	
20 ml extract, 1 µg. Wt. residue in jar, mg.		2.3 4.0		5.9	6	3.4	} 5% to 13% reco- very
L T 50, hr.		7½ 3¼		6	9	58	

* W = Winged, V = Vestigial.

(a) Used beeswax, not paraffin wax.

(b) Test thermostatically controlled at 22° C.

(c) Test in laboratory, average temp. about 15° C.

Recommendations

As a result of work so far carried out the Panel make the following recommendations:-

Breeding

It is desirable that the temperature of the breeding room should be thermostatically controlled. One of the cheap domestic thermostats now available is quite satisfactory for this purpose. A temperature range of 18-25°C is probably satisfactory. The choice of media is largely a matter of personal choice and convenience. It is important to ensure that all ingredients are satisfactorily free from pesticide residues since the presence of even low pesticide contents may breed a strain of insects which have acquired tolerance.

Testing

It is not intended that this test should be any other than a sorting test and no attempt is made to produce any quantitative estimate of the amount of pesticide present, other than to say that the amount present, if any, does not reach a dangerous level. Nevertheless, it is necessary to carry out controls with known amounts of pesticide. It is clear from the experiments so far carried out that the response of the test organism to a given amount of pesticide varies not only with the weight of residue in which the pesticide is embedded, but with the nature of the residue.

The originally proposed use of paraffin wax for control experiments is therefore no longer tenable, at least at very low levels of pesticide residue.

It is desirable, therefore, to carry out control tests using an extract of the type of crop under examination but which is known not to have been treated with the pesticide.

Our results indicate also that little advantage is obtained in using an aliquot providing more than 2-3 mg of residue, and it is suggested that as a maximum no more than 5 mg of residue should be present in the test jar.

Our experiments indicate also that the temperature at which the test is carried out may be critical, and it is recommended, where it may not be possible to control the temperature of the breeding room within narrow limits, that the test should be carried out in an incubator controlled at a temperature between 20-25°C.

References

1. J.A.P.A., 1963, 1, 5.
2. Laws, E.Q. & Webley, D.J. Analyst 1961 86 249

THE ANALYSIS OF
CANNED RICE (AND SAGO) PUDDINGS

by R.A. Dalley and Eric C. Wood.
(City Laboratory, Leeds; and
Public Analyst's Laboratory, Norwich.)

During 1961/2, we were engaged in collaborative analyses of several samples of canned rice pudding of different brands, in connection with a sample that contained 18 per cent of added water (see Monthly Report, March 1962, 1, 24). This led us to investigate in some detail the analytical methods available and the assumptions made when calculating the composition of the article in terms of ingredients from the analysis. Some of our conclusions may be of interest.

Analytical Methods

The samples were emptied out of the cans and thoroughly mixed by using a high-speed macerator. Water and protein were determined by standard methods, using a factor of 6.38 to convert N to protein; usually 80 - 85 per cent of the total N is derived from milk, so that this is nearer the truth than 6.25. Fat was determined by a modified Werner-Schmidt method as for ice-cream, weighing out 4 - 5 g., adding 5 ml. conc. HCl + 10 ml. water, mixing, and heating on a water-bath at 50° - 55° C for 30 minutes. Ash is determined by the standard method in doubtful cases but for routine purposes may be assumed to be 0.6 per cent., which we have found to be always within 0.1 per cent of the correct figure. Lactose and sucrose were determined by R.A.D. with Fehling's solution, using the Lane and Eynon methylene blue titration method, before and after inversion; although the presence of calcium is said to interfere with this method, he found experimentally that the addition of Calgon as recommended by Gaskin¹ to complex the calcium made no appreciable difference to the titrations. E.C.W. determined the total reducing sugars after inversion with Fehling's solution and the lactose separately by the chloramine-T method. With either method, the accuracy of the calculation will be affected if any reducing sugar other than lactose is present, e.g., if any of the sucrose has become inverted. Our results, however, do not suggest that any significant degree of inversion takes place; if it did, the apparent lactose figures would be too high, whereas usually they appear to be too low (see below). Starch was estimated by difference.

The Composition of Polished Rice

In order to calculate from the analysis the composition of the pudding in terms of ingredients, the nitrogen content and starch content of the rice used must be assumed. Published data from various sources^{2, 3, 4}, give the protein content of polished rice as from 6.2% to 8.1%, i.e., a nitrogen content of 0.99% to 1.30%. Four samples of rice as actually used for making canned rice puddings were obtained from a manufacturer of this product and analysed by E.C.W.; the countries of origin and nitrogen contents were (Italy) 1.04%; (Argentine) 1.02%; (United States) 0.99%; (Australia) 0.86%. For the purpose of calculation we now assume the nitrogen content of rice to be 1.00%, which has the advantage of simplifying the arithmetic. If the assumed nitrogen content is increased to 1.1%, the calculated milk content of a sample of canned rice pudding is decreased by a maximum of 2%, so that our assumption of 1% is almost always in the manufacturers' favour.

The starch content of polished rice is given by the same authorities as from 79.4% to 79.8%. For the purpose of calculation we have assumed it to be 80%, which again simplifies the arithmetic. If a figure of 79% had been used the calculated rice content of a sample of pudding would have been increased by only 0.13%. We learned from discussions with another P.A. that he had taken the starch content of rice to be 86.8%, on the alleged authority of the well-known publication "The Composition of Foods"³, in which the carbohydrate content of rice is given as 86.8%. All the figures for carbohydrate in this book, however, are expressed as monosaccharides, and to convert them to starch they must be multiplied by 162/180, i.e., by 0.900; this gives a starch content for this particular sample of rice of 78.1%. Our assumed figure of 80% may be a little too high but if so, this leads to a slight under-estimate of added water, thus again giving the manufacturer the benefit of the doubt.

Calculation of Milk Content

Estimates of the milk content can be obtained in three different ways.

- (a) The lactose content is multiplied by $24/13 = 1.846$ (Vieth's ratio of s.n.f. to lactose) to obtain an estimate of the s.n.f.
- (b) The milk nitrogen is multiplied by 17 (i.e., $6.38 \times 24/9$) to obtain an estimate of the s.n.f. In either case, a further assumption must be made regarding the s.n.f. content of the original milk; two such assumptions are shown in the calculations in Table I, both using the protein content and not the lactose content for reasons discussed below.

- (c) The fat content of the pudding is divided by the assumed fat content of the original milk and multiplied by 100.

The method actually used by R.A.D. before this collaborative experiment began is best illustrated by means of the analysis of a sample given in Table I.

Table I

Analysis by R.A.D. of a typical canned rice pudding

Water		76.70	per cent
Fat		3.06	" "
Ash		0.64	" "
Protein (N x 6.38)		3.23	" "
Lactose (anhydrous)		3.52	" "
Sucrose		5.85	" "
Starch (by difference)		7.00	" "
		<hr/>	
		100.00	" "
		<hr/> <hr/>	
	^{10%} 100		
Rice (Starch x 1.25)	=	8.75	" "
Rice N (Rice x 0.010)	=	0.088	" "
Total N	=	0.507	" "
Milk N	=	0.419	" " <i>by difference</i>
Milk S.N.F. (Lactose x 24/13)	=	6.50	" "
Milk S.N.F. (Milk N x 17)	=	7.12	" "
Milk (7.12 x 100/8.8)	=	80.9	" "
Milk (7.12 x 100/8.5)	=	83.8	" "
Milk (Fat x 100/3.6)	=	85.0	" "

	<u>Found</u>	<u>Reported as:</u>
Milk	85.0 per cent	85.0 per cent
Rice	8.8 " "	9.0 " "
Sugar	5.9 " "	6.0 " "
Added Water	0.3 " "	Nil
	<hr/>	<hr/>
	100.0 " "	100.0 " "
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When the milk content calculated by method (b) and assuming 8.8% s.n.f. in the milk leaves room for added water, as in the example, it is re-calculated assuming 8.5% s.n.f. and also by method (c) assuming 3.6% fat in the milk.

The manufacturer is given the benefit of the more favourable of the two figures. An even higher estimate of the milk content could of course be obtained by assuming the fat content of the milk to have been lower than 3.6%; but R.A.D. considered this a reasonable figure to adopt for the following reasons.

First, if the fat content of the original milk was lower than 3.6%, the milk was below average quality and should not have been described - as it invariably is - as 'full-cream milk' on the label. Secondly, some manufacturers start with full-cream dried milk, which if correctly reconstituted will then contain 3.6% of fat.

The other of us (E.C.W.) had always calculated the milk content from the milk N x 200, which is equivalent to assuming that the s.n.f. of the original milk was 8.5% (by a happy arithmetical chance, $6.38 \times 24/9 \times 100/8.5 = 200.1$), because the determination of N is at least as precise as that of fat, and the probable variability of the milk s.n.f. (say 8.5 to 9.0, or 3% either side of the mean) is less than that of the fat (3.0 to 3.6 or more, i.e., at least 10% either side of the mean). However, if one calculates the estimated milk content from the nitrogen and then uses this to estimate the fat content of the original milk, one sometimes obtains rather odd-looking figures.

Table II
Analysis of four cans of rice pudding

Analyst	<u>Can A</u>	<u>Can B</u>	<u>Can C</u>	<u>Can D</u>
	<u>Per Cent</u>			
	<u>E.C.W.</u>	<u>E.C.W.</u>	<u>E.C.W.</u>	<u>R.A.D.</u>
Water	76.06	77.62	76.53	77.00
Fat	2.78	3.22	3.18	3.31
Ash	0.62	0.60	0.65	0.68
Protein (N x 6.38)	3.24	3.35	3.29	3.42
Sucrose	6.90	4.60	4.50	4.50
Lactose (anhydrous)	3.73	3.96	3.33	3.51
Starch (by difference)	6.67	6.65	8.52	7.58
	100.00	100.00	100.00	100.00
Rice (Starch x 1.25)	8.3	8.3	10.6	9.5
Total N	0.507	0.525	0.516	0.536
Rice N (Rice x 0.01)	0.083	0.083	0.106	0.095
Milk N	0.424	0.442	0.410	0.441
Milk (N x 200)	84.8	88.4	82.0	88.2
Milk (Lactose x 21.72)	81.0	86.0	72.3	76.2
Milk + Rice + Sugar	100.0	101.3	97.1	102.2
Added Water (minimum)	nil	nil	2.9	nil
Fat in orig. milk	3.28	3.64	3.88	3.75

Table II shows the analysis of four separate cans of rice pudding, all claimed to be made from 'full-cream milk'; cans A, B and C are of three different brands analysed by E.C.W. and

= Fat content of pudding x 100
milk (N x 200)

the fourth, can D, is the same brand as C but was analysed by R.A.D. and did not have the same code number. (The estimate of milk content from the lactose, though shown in Table II, was not used for a reason discussed below).

The first two samples give results that are consistent with probability, except that milk containing at least 3.64 per cent fat does not usually contain only 8.5 per cent s.n.f. as is assumed in the calculations. But the calculations on can C imply that the sample contained either 82 per cent of milk with 8.5 per cent s.n.f. and 3.88 per cent fat, with 2.9 per cent added water, or (if there were in fact no added water present, which was strenuously maintained by the reputable manufacturers of this article) 85 per cent of milk with 8.2 per cent s.n.f. and 3.74 per cent of fat. This is not a likely composition. Since R.A.D.'s sample (can D) adds up to 102.2 per cent without any added water, the implication is that his sample actually contained 86 per cent of milk of composition 8.72 per cent s.n.f. and 3.85 per cent fat. It is possible that some manufacturers may add a little extra fat, e.g., margarine, as the housewife sometimes does; but we have no evidence of this and it is certainly not declared in the list of ingredients on any label we have seen.

The Lactose Content of the Puddings

We have already said that an estimate of milk content based on the lactose content is not used, and the figures in Table II show the reason. In our experience, based on more than 80 samples, this estimate is almost always lower than that obtained from the nitrogen content; sometimes (cans A and B) the difference is not more than 2 - 3 per cent, but sometimes (cans C and D) the "lactose estimate" is 10 per cent less than the "nitrogen estimate".

This discrepancy is not likely to be due to experimental error, because both R.A.D.'s and E.C.W.'s methods for determining lactose are well-established and known to give accurate results in normal circumstances. If any sucrose had become inverted, or if any starch were hydrolysed to reducing substances, during the canning process, the apparent lactose content would be too high, not too low.

The phenomenon would be largely explained if some manufacturers use reconstituted New Zealand dried milk, or a mixture of this with home-produced milk. Halliday *et al.*⁵ have shown that for this milk the Vieth ratio, protein:lactose:ash is not 9:13:2 but (weighted average of 22 samples) 10.4:11.55:2.05. If these figures are applied to our analyses both the 'nitrogen estimate' and the 'lactose estimate' of milk content are affected.

The milk N must be multiplied not by 200 but by $6.38 \times 24/10.4 \times 100/8.5 = 173$. Similarly, the lactose must be multiplied not by 21.72 but by $24/11.55 \times 100/8.5 = 24.44$.

Sago Puddings

Sago contains more starch and less nitrogen than rice and for this reason the calculation is modified as follows:-

$$\text{Sago} = \text{Starch (by difference)} \times 100/85.$$

$$\text{Sago N} = \text{Sago} \times 0.0004$$

After subtracting the sago N from the total N, the rest of the calculation is the same as for rice pudding. For tapioca, the factor is 0.007.

Conclusions

1. The milk content of canned rice pudding is best calculated from the 'milk nitrogen'; the rice used may reasonably be assumed to have contained 80 per cent starch and 1.0 per cent nitrogen.
2. The fat content of the original milk as estimated from the analysis sometimes seems to be higher than is likely to occur in practice, having regard to the assumption in the calculations that the milk contained only 8.5 per cent s.n.f.
3. The lactose content of the rice pudding is almost always too low, and sometimes much too low, to be consistent with the rest of the analysis, if it is assumed that the milk was of normal composition. This might possibly be due in some samples to the use of New Zealand milk in high proportion. We would welcome the experience of other Public Analysts and the comments of manufacturers, both on this point and on the rather high fat content already mentioned.
4. The majority of the canned rice puddings we have examined do not contain any added water at all, and even when present there is rarely more than two or three per cent. It is clear that it is commercially quite practicable to make canned rice pudding without any added water, and the cookery-book recipes we have seen do not provide for any. We therefore regard any significant quantity of added water in a canned rice pudding as an adulterant.

References

1. Gaskin J.G.N. Analyst, 1935, 60, 318.
2. Kent-Jones D.W. and Amos A.J. "Modern Cereal Chemistry", 1957, pp. 125 and 127.

3. McCance R.A. and Widdowson E.M. "The Composition of Foods", 1960, pp. 26 and 27.
4. Bacharach A.L. and Rendle T. (Edited by). "The Nation's Food", 1946, p.200.
5. Halliday J.H., Burden E.H.W.J., and Lamont J.J. Analyst, 1960, 85, 839.

Editor's Note: We have been asked by several members of the Association to reprint in the Journal certain contributions made to our confidential Monthly Bulletin in the years before we had any non-confidential publication, and which therefore if not reprinted cannot be made generally available. Three such articles follow, the first being by the late D.G. Allen, formerly Public Analyst for Northampton C.B., and elsewhere, who died in January 1961.

THE DECOMPOSITION OF SAUSAGES ON KEEPING:
THE EFFECT ON APPARENT MEAT CONTENT.

by D.G. Allen.

(Reprinted from the A.P.A. Bulletin, 1958).

It is generally known that the apparent meat content of sausages, as determined by the routine Stubbs and More method, will usually increase as samples are kept and decompose. This is due to the decomposition of the starch in the cereal filler and the consequent concentration of the other constituents and the inclusion of some of the nitrogenous material from the filler with the meat nitrogen.

Many public analysts will have had the experience of being confronted during a prosecution for meat deficiency with an analysis carried out on behalf of the defendant some weeks or even months after the sample was taken. This may show a meat content substantially higher than that reported by the public analyst when the sample was fresh.

As I had not seen any figures published for the apparent meat contents of sausages after decomposition, the following information may be of interest.

Four samples of sausage were analysed within a day or two of being received. They were then kept in glass jars in the ordinary domestic-type refrigerator for 8-9 weeks and then re-examined. The four samples contained 200-300 parts per million SO₂ at the time of sampling. The samples were not all from the

same source and the four samples were not examined concurrently.

<u>Sample</u>	<u>First Analysis</u>			<u>Second Analysis</u>			
	<u>Moist.</u> <u>%</u>	<u>Carbo.</u> <u>%</u>	<u>Total</u> <u>Meat</u> <u>%</u>	<u>Moist.</u> <u>%</u>	<u>Carbo.</u> <u>%</u>	<u>Total</u> <u>Meat</u> <u>%</u>	<u>Apparent</u> <u>Increase</u> <u>%</u>
1. Pork	52.7	13.5	53	58.0	5.2	63	10
2. Pork	47.1	14.8	58	51.3	6.4	70	12
3. Pork	52.7	13.5	64	59.3	2.4	74	10
4. Beef	40.0	12.5	79	45.5	5.7	88	9

The carbohydrate figures were obtained by difference.

It is not suggested that all sausages would decompose in this way, but the figures do show the possible magnitude of the apparent increase, sufficient to make an unsatisfactory sample appear satisfactory. The "no change" statement is particularly important in such cases and defence analysts need close cross-examination.

THE DETERMINATION OF GLYCERIN IN MIXTURES

by A.L. Williams
(City Laboratory, Portsmouth).

(Reprinted from the A.P.A. Bulletin, 1955)

Some Public Analysts may not have come across the following method of determining glycerin in such preparations as Compound Glycerin of Thymol. It is given in Thorpe's Dictionary, 4th edition, Vol. 6, p. 57, where it is stated to be taken from Oil and Soap, 1941, 18, 14. Ordinary sugars, oxalic acid, hydroxy-acids such as citric and tartaric acids, do not interfere (here lies much of the value of the method); ethylene and propylene glycols, mannitol and sorbitol, interfere somewhat, showing from 2% to 5% of the amount present as apparent glycerol. Polyglyceryl ethers, and alkylolamines, interfere.

Weigh not more than 10 ml, containing not more than 0.8 g of glycerin, into a 100 ml volumetric flask, and dilute to 10 ml with water. In a second flask take 10 ml of water for a 'blank'. To each add 10 ml of 30% w/v. NaOH solution, followed by 60 ml of 95% alcohol, and mix well. Add from a burette an alcoholic 10% solution of cupric chloride dihydrate until a permanent undissolved precipitate of cupric hydroxide remains after shaking; then add 0.5 ml more and adjust to 100 ml at 20°C (maintain at

this temp. throughout) with 95% alcohol. Centrifuge at 1300 r.p.m. for 10 minutes and decant a 50 ml aliquot into a conical flask of about 300 ml capacity. Add 100 ml water and make just acid with glacial acetic acid. Then add 2 ml more, cool in ice, add 10 g of potass. iodide and titrate with 0.1 N thiosulphate, using starch indicator and adding 2 g of ammonium thiocyanate just before the end-point. If the weight of sample taken is S, and the titrations in terms of exactly 0.1 N for sample and blank are T and B respectively, the percentage of glycerol in the sample is $18.41 (T - B)/S$.

THE DETERMINATION OF GLYCERIN IN PRESENCE OF SUGARS

by W. B. Chapman.
(City Laboratory, Portsmouth)

(Reprinted from the A.P.A. Bulletin, 1956.)

The determination of glycerin using cupric chloride, to which A. L. Williams has drawn attention, (see above) was recommended only for use with Compound Glycerin of Thymol. Whilst sucrose does not interfere with this method, the presence of appreciable amounts of reducing sugars prevents the direct application of the method, due to their reducing action on the copper-glycerol complex. For this reason attempts to use the method for the determination of glycerol in cough mixtures etc. have failed.

Separation of glycerin from reducing sugars can be effected by the chromatographic method of Sporek and Williams (Analyst, 1954, p. 63). This process gives a glycerin solution sufficiently low in reducing sugars to enable the cupric chloride method to be applied.

Recoveries of added glycerin in the presence of dextrose, of honey and of several types of cough mixtures have ranged from 100 to 108% for amounts of added glycerin from 0.1 to 0.5 gram. The cough mixtures tested have contained the following substances:- Honey, Syrup of Blackcurrant, Tincture of Ipecacuanha, Acet. Scillae, Citric Acid, Chloroform, Camphor, Liquid Glucose, Lemon Juice, Quinine Hydrochloride, Oil of Cinnamon, Colour and Syrup basis.

Method

Weigh an amount of sample (up to 5 g.), containing up to 0.5 g of glycerin, into a 150 ml beaker and carry out the chromatographic separation as given in Analyst, 1954, 79, 65, obtaining

250 ml of acetone solution containing the glycerin. Transfer to a 600 ml beaker and evaporate on the water bath until all the acetone has been volatilised and the volume is reduced to 2 or 3 ml. (Loss of glycerin may occur if heating is continued beyond this point).

Transfer solution to a 10 ml cylinder, record volume to 0.1 ml and pour into a clean dry 100 ml volumetric flask. Rinse out the 600 ml beaker with 2-3 ml H₂O, transfer to cylinder, record volume and again pour into flask. Repeat until a total volume of 12.5 ml is obtained in the flask. (Usually 2 or 3 washings are possible and all the glycerin should thus be transferred to the volumetric flask). Add 7.5 ml of 40% w/v NaOH and proceed as in the normal cupric chloride method (see A. L. Williams, above).

Note:- It has been found that it is not essential to maintain only 0.5 ml excess of cupric chloride reagent. A standard addition of 10 ml of this solution to each flask is much simpler, and will be sufficient for up to about 0.5 g of glycerin. This technique, although producing much larger amounts of copper hydroxide, does not affect the final result.

CHANGES IN PUBLIC APPOINTMENTS

BAKER G.H.	Add - D.P.A., Stockport C.B.
CHAPMAN W.B.	Formerly D.P.A., Portsmouth - now appointed as P.A. for Bethnal Green and Poplar Met. B.'s. Address - County Hall, Westminster Bridge, London, S.E. 1.
BARKER J.H.	Now D.P.A. for the above Met. B.'s. Address as above.
GREENBURGH S.	Add - P.A., Soke of Peterborough C.
WOODHEAD J.E.	Retired on the 31st March last and has resigned all his public appointments.

LEGAL NOTES

"Non-Alcoholic Lager" - Successful prosecution.

At Wellingborough Magistrates' Court on the 2nd April, 1963, Carlsberg Distributors Ltd. were convicted on two charges under Sect. 6 of the Food and Drugs Act. They had pleaded not guilty to the following charges :-

1. Giving with an article sold as 'Carlsberg Consort' a label which was calculated to mislead as to the quality of the said food, and -
2. Being a party to the publication of an advertisement which falsely described 'Carlsberg Consort' as non-alcoholic.

Evidence was given that the article was contained in bottles similar in size, shape and colour to the same firm's ordinary lager, and were labelled in a similar manner; the label bore only the words 'Carlsberg Consort, Product of the Carlsberg Breweries Copenhagen'. Mr. R.E. Smith, prosecuting for the Northamptonshire County Council said that the label of 'Carlsberg Consort' was likely to mislead an ordinary person into thinking that this was a lager beer of the quality and type normally produced by the company.

Mr. E. Voelcker, Public Analyst for Northamptonshire, said that in his opinion the product was an alcoholic beverage but with a non-exciseable alcoholic content of 1.8% proof spirit. He added "Had I not known this was less than 2% proof spirit I would have said it was lager beer". The Chief Inspector of Weights and Measures, Mr. F.J. Evans, produced an advertisement from the Daily Telegraph containing the words "Call for a Carlsberg - the right brew for every lager drinker" to show that the word "Carlsberg" would normally be used to mean "Lager".

Mr. F.G. Milton, managing director of Carlsberg Breweries Ltd., said that in his view there was no doubt that 'Consort' was a lager. The company in 1960 wanted to introduce a low-gravity drink into this country; it took twelve months to find a suitable name. Another firm was producing canned fruit under the name of Consort, but they agreed to withdraw their objections to Carlsberg Breweries using the name provided the word Carlsberg was inserted before the word Consort. He added that the product was intended for unlicensed premises such as cafes and youth clubs, and the word Consort was chosen so that it could be used all over the world. A chemist from the Danish company said in evidence that apart from the last fermentation, Consort was made in exactly the same way as lager; the alcoholic content of Danish lagers varied from the 15% of a de-luxe brand to under 2% in their product Let Pilsener.

Mr. John May, counsel for the defence, asked for a dismissal of the first summons on the ground that in every respect except alcohol content, Consort was a lager beer. The prosecution

claimed that the label was misleading because it signified alcohol content; but, he submitted, a member of the public did not just see the label and say "Carlsberg - that is a 5% alcoholic content beer".

As regards the second summons, Mr. Evans said that he had drawn the attention of the defendants to two advertisements in the press, both of which described Consort as non-alcoholic. Mr. Milton, on oath, said that in the beverage trade, an alcoholic drink was understood to be one that contained more than 2% proof spirit. He thought the advertisement was correct in the trade if not in law. The Editor of "The International Beverage News" said that his considerable experience of the beverage trade led him to believe that "non-alcoholic" was the phrase used in the trade to describe drinks with either no alcohol or less than 2% proof spirit.

The Bench found the case proved. Carlsberg Distributors Ltd. were fined £25 in respect of the first charge and were ordered to pay £27. 18s. 6d. costs in respect of the second. It is understood that the firm gave notice of appeal but afterwards withdrew it.

(We are indebted to Mr. F. J. Evans for the above information).

LOCAL AUTHORITIES' JOINT ADVISORY COMMITTEE ON FOOD STANDARDS

CODE OF PRACTICE NO. 1 - USE OF THE WORD "CHOCOLATE" IN FLOUR CONFECTIONERY

1. We are directed by the Local Authorities' Joint Advisory Committee on Food Standards to state that a Code of Practice has been agreed between the Committee and representatives of the Bakery Industry on the use of the word "chocolate" or a synonym in the description of bakery products. The Code has been approved by the constituent bodies.

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

Except as otherwise provided herein, where the word "chocolate" or an abbreviation or a synonym thereof is used in the description of such a product, it shall contain not less than 3 (three) per cent of dry non-fat cocoa solids in the moist crumb.

Provided that the requirement herein as to cocoa content shall not apply:-

normally composed of public analysts with particular experience of the commodities under discussion, together with such representatives of the local authority Associations as may be considered desirable.

The codes of practice, when endorsed by the Joint Advisory Committee, are submitted to the constituent bodies, any of which has the right to reject such recommendations.

Constituent Bodies -

Association of Municipal Corporations
 County Councils Association
 Urban District Councils Association
 Association of County Councils in Scotland
 Convention of Royal Burghs (Scotland)
 Scottish Counties of Cities Association
 Association of Public Analysts.

The accompanying Code of Practice was negotiated with the following organisations representing the Bakery Industry, namely:-
 The Federation of Wholesale and Multiple Bakers, The National Association of Master Bakers, Bakery Allied Traders' Association, Co-operative Bakery Trade Association, The British Baking Industries Research Alliance and the Cake and Biscuit Alliance, Ltd.

ITEMS OF INTEREST

Mustard and Cress, substitution of rape for mustard in.

A number of Public Analysts have recently had submitted to them by their Sampling Officers samples of mustard and cress sold growing in square blocks of soil surrounded by a brightly coloured wrapper drawing attention to the novel pack. In consequence, this popular sandwich filling has been receiving special attention, and it has been realised that the so-called 'mustard' is in fact largely or wholly rape. Some Public Analysts have regarded this as a 'passing off' offence; but E. C. Wood was told that as long ago as 1935, an Order was made under the Agriculture Grading and Marking Regulations in which rape was regarded as equivalent to mustard. He wrote to the Ministry of Agriculture, Fisheries and Food asking if this was so.

The Ministry sent in reply copies of (a) the Order in question (S.R. & O., 1935, No. 107) on the first page of which is a

footnote "For the purposes of this specification the term 'mustard' includes rape", (b) a Ministry of Agriculture and Fisheries "Pre-Leaflet No.34" entitled "National Recommended Grades for Home-Grown Mustard and Cress", dated 1950, which again refers to rape as being equivalent to mustard; (c) the Ministry's Bulletin No.143, issued in 1955, entitled "Salad and Other Food Crops in Glasshouses", in which it is stated "the crop marketed as mustard is almost invariably rape, the true white mustard being rarely grown now for salads. Preference is given to rape in large establishments because its seed leaves, which are the parts used for salad purposes, are of a more intense green colour, and when packed in punnets it does not deteriorate in warm weather so quickly as mustard. . . Some growers claim that the addition of 15 to 20 per cent of true white mustard seed to the rape sowings during December to February improves the flavour of the seed". This last sentence is interesting; after growing both rape and mustard side by side in his laboratory, Wood found that members of his staff agreed unanimously after tasting tests that the mustard was definitely more pungent than rape of the same age, which by comparison was tasteless.

This might suggest that rape sold as mustard is 'not of the quality demanded' in spite of the assumption of equivalence in the publications quoted; but as the Ministry say in their letter enclosing these documents, ". . . This product has been sold in this manner for at least 30 years. . . This is an instance where a product has attracted to itself a wrong name, but has for so long been accepted, that it might now be difficult to do anything about it."

The Ministry Bulletin No. 143 quoted above, also states that the mixed crop is sold "on the basis of one punnet of cress to six punnets of mustard." The growing mixed article referred to at the beginning of this note often contains much less cress than the 14% or so implied by the quoted sentence; the proportion is variable but in some samples is as low as 5%. This although the wrapper describes the article as Mustard and CRESS, the last word being so much bolder than the first that it can be easily read at such a distance that the first word is only a blur.