# THE COMPOSITION OF

# CANNED RICE, SAGO AND TAPIOCA PUDDINGS

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Satisfactory analyses of canned rice and similar puddings are obtained by the use of standard methods for estimating their constituents. Calculation of the ingredients used in the preparation of these puddings must, however, be based on acceptable assumptions regarding the average composition of these ingredients. An attempt has been made to show how varying assumptions may influence the calculation of the ingredients present.

Following the publication of the paper on this subject by Dalley and Wood<sup>1</sup> an examination of the results of the analysis of about 150 samples received in this laboratory was undertaken. It is felt that a study of a sufficiently large number of such results including, as they must, both analytical data and the interpretation of such data, would help to clarify the problems involved in these aspects of a Public Analyst's work.

<u>Analytical Methods.</u> The sample is rendered of uniform consistency by passing through a Glen Creston mill and the following determinations are made:- total solids, fat, lactose, sucrose, protein, ash and carbohydrate (by difference.) The fat is determined by the modification of the Werner-Schmidt method appropriate for sugar-containing foods. The protein is calculated as N x 6.38.

While Dalley and Wood assumed that the only adjustment needed in order to obtain the correct figures for milk constituents was the appropriate allowance for protein derived from rice (or corresponding ingredient), a correction has also been made here for the ash due to rice.

<u>Composition of Rice.</u> Published figures 2, 3, 4, give the protein content of rice as 7.0 per cent., 7.6 per cent. and 6.2 per cent. The last of these figures is definitely stated to be N x 5.7 and it is at least implied that the other figures are similarly calculated. This means that the nitrogen contents are respectively 1.23 per cent., 1.33 per cent. and 1.09

per cent. The practice in this laboratory has been to assume that rice contains 7.0 per cent. of protein and since total protein is equal to N x 6.38 in this particular analysis, the assumed nitrogen content is 1.10 per cent. Following the publication of the paper by Dalley and Wood, twelve samples of locally purchased rice were analysed and gave an average nitrogen content of 1.08 per cent. Similarly, both published and locally determined figures indicate that rice contains 0.5 per cent of ash. The figure for carbohydrate by difference has been converted to rice by multiplying by 100/80.

Calculation of Milk Content. Unlike Dalley and Wood it was assumed by the author that, since figures for the lactose, protein, ash and fat of the milk present could be obtained from the results of analysis, the sum of these milk ingredients should be used in calculating the milk content of the sample. Both the Condensed Milk and the Dried Milk Regulations stipulate that milk should contain 12.4 per cent. of total milk solids and the published data of most Public Analysts give figures very close to this as their annual average figure. This figure has, therefore, been used to calculate the amount of full cream milk present.

Calculated Composition of Milk Puddings. Samples which, on the stated basis of calculation, have been found to contain significantly more than 10.0 per cent. of added water have been reported as adulterated and are not included in the following averages. The results obtained on samples received since the end of 1960 until the end of September 1963 are quoted in Table I.

### TABLE I

### AVERAGE PERCENTAGE COMPOSITION OF

#### CANNED RICE PUDDINGS

No of Samples	Lactose	Milk Protein	Milk Ash	Milk S.N.F.	Fat	Total Milk Solids
(	3.55	2.64	0.59	6.78	3.04	9.82
65 (	Milk Con	tent	Rice	Sugar	Add	led Water
(	79.2		8.4	6.6		5.8

These figures give a ratio of lactose:protein:ash (L:P:A:) of 12.55:9.35: 2.10 calculated on a basis of a total of 24 units.

Richmond's ratios, when calculated to the same basis, are (a) 12.82:9.17:2.01<sup>5</sup> and (b) 12.75:9.22:2.03<sup>6</sup>. Richmond also quotes Vieth as suggesting the L:P:A: ratio as 13:9:2, but it has not been possible to find Vieth's publication of this version. Vieth, however, did publish<sup>7</sup> an earlier L:P:A: ratio in 1888 giving figures of 6:5:1. While this suggests that approximations may possibly be slightly misleading, there is no doubt that the generally accepted Vieth Ratio of 13:9:2 has proved of enormous value over the past 70 years. Comparing this method of calculating milk content with alternative methods, the above data vield the results shown in Table II.

### TABLE II

# COMPARISON OF METHODS OF CALCULATING MILK CONTENT OF CANNED MILK PUDDINGS

Basis of	T.M.S.	S.N.F.	Fat	Lactose	Protein	Ash
calculation (per cent.)	12.4	8.8	3.6	$\frac{13}{24}$ of 8.8	$\frac{9}{24}$ of 8.8	$\frac{2}{24}$ of 8.8
Calculated milk conten in sample (per cent.)	t 79.2	77.0	84.4	74.4	80.0	80.8

These findings, in spite of the different allowance for the protein due to rice, confirm those of Dalley and Wood, namely, that the lactose present appears to be low and the fat present to be high. The proportions of solids-not-fat and fat in the 12.4 per cent. of total milk solids calculated from the average figures printed above are 8.56 per cent. and 3.84 per cent. whereas Staffordshire's average annual figures for 1962 were 8.71 per cent.and 3.69 per cent. respectively.

One way of attempting to discover the reason for the apparent abnormalities in these results for the composition of the milk in canned rice puddings is to compare them with the results of analysis obtained by identical analytical methods when applied to canned sago and tapioca puddings where the corrections for protein and ash due to the sago and tapioca present differ greatly from those used in the case of canned rice puddings. It it, perhaps, rash to make any deductions from the results obtained on a batch of only 19 samples but,

nevertheless, these are given as a tentative basis of comparison. The corrections made assume that sago and tapioca contain 86.0 per cent. of carbohydrate, 0.30 per cent. of protein and a negligible amount of ash. On this basis the samples examined gave the following results (Table III):-

### TABLE III

# AVERAGE PERCENTAGE COMPOSITION OF CANNED SAGO AND TAPIOCA PUDDINGS

No. of Samples	Lactose	Milk Protein	Milk Ash	Milk S.N.F.	Fat	Total Milk Solids
(	3.74	2.80	0.64	7.18	2.91	10.09
19 (	Milk Con	tent	Tapioca or Sago	Suga	r	Added Water
{	81.4		6.4	5.7		6.5

This gives an L:P:A ratio of 12.50:9.36:2.14, very similar to that obtained on the canned rice puddings. The S.N.F.:Fat ratio is, however, 8.82:3.58. When applied to the figures in Table III, the various methods of calculating Milk Content give the following results:-

#### TABLE IV

Basis of T.M.S. S.N.F. Fat Lactose Protein Ash Calculation

Calculated Milk Content per 81.4 81.6 80.8 78.4 84.8 87.7 cent. in sample

<u>Comment.</u> The outstanding difference between the Rice Puddings and the Sago and Tapioca Puddings is in the ratio S.N.F.:Fat. In the former, as Dalley and Wood pointed out, the fat content was disproportionately high. It was thought that the oil content of rice would have no appreciable effect on the calculation of the milk content, and hence on the added water present in these samples. Allowance for the amount of oil in rice does, in fact, reduce the calculated amount of milk by 0.8 per cent. only, but the effect of this correction on the S.N.F.:Fat ratio is much more striking. Published figures<sup>2</sup>, <sup>3</sup>, <sup>4</sup>, indicate that rice contains about 1.0 per cent. of oil and the twelve samples of rice examined here were found to contain an average of 1.2 per cent. of oil. A further error in the original calculations made and quoted above is due to the fact that the normal figure of 8.80 per cent. for S.N.F. in milk includes 0.15 per cent. of solids in addition to the sum of the figures for lactose, protein and ash. When these two sources of error are corrected, the following figures for the average composition of Canned Milk Puddings are found, (Table V).

### TABLE V

# CORRECTED PERCENTAGE COMPOSITION OF CANNED MILK PUDDINGS

	No. of Samples	Milk S.N.F.	Milk Fat	Milk T.S.
Rice	65	6.90	2.94	9.84
Sago or Tapioca	19	7.30	2.91	10.21
	Milk Content	Rice, Sago or Tapioca	Sugar	Added Water
Rice	79.4	8.5	6.6	5.5
Sago or Tapioca	82.3	6.4	5.7	5.6

The ratio S.N.F.: Fat in 12.4 parts of total milk solids becomes, for rice puddings 8.69:3.70 and for sago and tapioca puddings 8.86:3.54. The comparable ratio for all samples of milk received in Staffordshire in 1962 was 8.71:3.69. This appears to explain the abnormal S.N.F.: Fat ratio obtained before these corrections were made.

The abnormal L:P:A ratio remains to be accounted for. If dried milk powder, or more particularly, skimmed milk powder is used in the manufacture of these products the difficulty of getting the lactose into solution may explain the low figure for this ingredient. This problem has occurred in the preparation of ice-cream from dry ingredients.

Two possible sources of added water have not yet been mentioned. It is understood that sugar is sometimes added in the form of a syrup and this could well account for 3.0 percent.

added water. Another but probably less frequent source results from the practice of heating skimmed milk by steam injection. This can, of course, introduce 10.0 per cent. or more of added water.

This paper was drafted before the publication of Markland's paper<sup>8</sup> where attention is drawn to the two sources of error dealt with above. It is interesting to note that they tend to cancel each other out in the case of rice puddings and that, in the case of sago and tapioca puddings where only one of the corrections applies, the difference in the calculated amount of milk and of added water is just under 1.0 per cent.

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First Submitted, Dec., 1963. Revised, May, 1964.

# THE NITROGEN CORRECTION FOR RUSK FILLER IN SAUSAGES

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The results of analyses of a number of samples of cereal fillers used in sausages indicate that published figures for the nitrogen content of rusk sold for this purpose are excessive.

An investigation of the nitrogen content of rusk fillers, obtained in Staffordshire, showed different figures from those recommended for general use when allowing for this source of nitrogen in estimating the meat content of sausages.

The Report of the Meat Products Sub-Committee of the Society for Analytical Chemistry<sup>1</sup> recommended a figure of 2.3 per cent. of the dry carbohydrate as the correction for nitrogen in rusk filler, while Fraser and Holmes<sup>2</sup> use a factor of 2.5 per cent. for nitrogen associated with the starch which was determined polarimetrically.

As a preliminary to using the method suggested by Fraser and Holmes for determining the starch in sausage, nineteen samples of rusk fillers obtained in Staffordshire in 1963 were analysed in this laboratory. The following determinations were made:- moisture, oil, mineral matter, protein, carbohydrate by difference and starch polarimetri cally. The methods used were those given by Fraser and Holmes<sup>3</sup>. Two methods for estimating moisture and for estimating fat are given in this paper, but only one alternative method was used in each case in this laboratory. It was found that the determination of starch by the A.O.A.C. method<sup>4</sup>, gave results in agreement with those obtained by Fraser and Holmes.

The results of the above analyses, as given in Table I, show that the average nitrogen content of the rusk fillers examined was:-

- (a) 2.02 per cent. of the carbohydrate by difference (Standard Deviation 0.16)
- (b) 2.20 per cent. of the starch, (Standard Deviation 0.17),

and that starch in carbohydrate = 91.8 per cent. (Standard Deviation 0.89.)

It would appear that the figure of 2.3 per cent. for the

nitrogen correction in rusk fillers, as recommended by the Meat Products Sub-Committee, is too high when applied to rusks used in Staffordshire and that a figure of 2.0 per cent. should be used. Similarly the factor of 0.025 recommended by Fraser and Holmes for determining the nitrogen associated with the starch is not applicable and a figure of 0.022 should be used in this area.

Two further differences are apparent, since the percentage of starch in carbohydrate was found to be 91.8 per cent. as compared with the Fraser and Holmes figure of 94.0 per cent. for flour, and the mean starch to protein ratio was found to be 8 to 1 as compared with the Fraser and Holmes figure of 7 to 1.

# TABLE I

## COMPOSITION OF RUSK FILLER

Carbo- hydrate by difference	Starch %	Nitrogen % (±0.005)	Nitrogen calculated as % of carbo - hydrate	Nitrogen calculated as % of starch	Starch % in carbo- hydrate
85.59	77.50	1.596	1.86	2.06	90.5
84.06	77.50	1.680	2.00	2.17	92.2
81.70	75.20	1.540	1.89	2.05	92.0
84.31	77.50	1.736	2.06	2.24	91.9
84.29	77.50	1.792	2.13	2.31	91.9
83.79	77.50	1.722	2.06	2.22	92.5
84.33	77.50	1.736	2.06	2.24	91.9
83.47	76.20	1.680	2.01	2.21	91.3
85.61	77.20	1.554	1.81	2.01	90.2
81.95	75.00	1.708	2.08	2.28	91.5
85.69	78.00	1.708	1.99	2.19	91.0
81.50	75.00	1.740	2.13	2.32	92.0
81.99	74.70	1.680	2.05	2.25	91.1
85.22	78.00	1.778	2.09	2.28	91.5
84.60	77.50	1.750	2.07	2.26	91.6
85.10	79.00	1.520	1.79	1.92	92.8
79.95	74.00	1.944	2.43	2.63	92.6
86.33	81.30		1.70	1.81	94.2
85.21	78.50	1.773	2.08	2.26	92.1

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62

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# THE ANALYSIS OF CANNED RICE PUDDINGS

### by F.B. Pim

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Interpretation of the results obtained in the analysis of canned rice pudding is discussed.

Analyses of samples of rice used for pudding manufacture have been carried out to determine a mean composition for the rice itself as this is required to establish the appropriate factors to be used in subsequent calculations.

Estimations of the milk content of the pudding by calculation from either the milk protein or the lactose are both possible and no systematic difference in the results so obtained occurs if the figure for lactose hydrate is used in the Vieth ratio.

It has been shown that there is a small error in the figure derived from lactose because of the formation of some lactose-protein complex during processing. This is less significant than the effect of seasonal variations in the composition of the bulk milk used in manufacture. For this reason it is preferable to report the mean of the figures from protein and lactose as the "milk content".

Two papers 1, 2 have recently been published on the subject of the analysis of canned rice pudding, and on the interpretation of the results in order to calculate the milk content of the pudding and establish the absence of added water.

It is intended to put forward evidence that the methods of calculation and conclusions in these papers are not necessarily valid for all rice puddings.

# Analytical Methods.

The method for estimation of sucrose and lactose is as follows: Weigh 13  $\pm$  0.5 g of the thoroughly macerated sample into a 70 ml centrifuge tube with a lip. Add 30 ml industrial spirit to precipitate the starch. Add approx. 0.5 g light CaCO<sub>3</sub> and then 70 per cent. alcohol (w/v) gradually, while stirring, to within  $\frac{3}{4}$  inch of the top of the tube. Warm to 50°C and centrifuge at 2000 r. p. m. Pour off the supernatant liquor into a 250 ml beaker, add 0.2 - 0.5 g light CaCO3 and evaporate off the alcohol on a boiling water bath. Meanwhile re-extract the residue in the tube twice with 70 per cent. alcohol warming to 50°C and centrifuging as before, and add the washings to the beaker. Make small additions of water 3 or 4 times to wash down the sides of the beaker and to prevent evaporation to dryness while completely removing the Transfer the solution remaining in the beaker to a alcohol. 250 ml graduated flask with the minimum amount of water, and cool. Add saturated normal lead acetate and alumina cream in equal amounts (about 1.5 ml. of each), make up to the mark at 20°C and filter. Add approximately 1 g potassium oxalate to the filtrate to precipitate excess lead (and calcium), stand for one hour and filter. (With this serum, the use of Calgon in the titration is not necessary.) Using the Lane and Eynon method estimate reducing sugars on this serum before and after inversion, and calculate the sucrose and lactose using the appropriate tables.

Other methods of analysis used follow closely those of Dalley and Wood  $l_{\,\cdot}$ 

# The Composition of Rice.

As Dalley and Wood state, any calculations must take account of the composition of the rice. Recent analyses (Table I) of a number of samples of rice as used in the manufacture of puddings suggest that these author's assumptions of 1.0 per cent. nitrogen and 80 per cent. starch are questionable. The samples of rice analysed were commercial samples but probably all of Italian origin.

# TABLE 1

# ANALYSES OF RICE SAMPLES

Sample No.	Moisture %	Nitrogen %	Protein % (N x 6.25)	Fat %	Ash %	% "Starch by difference"*
1	15.1	1.067	6.65	0.89	0.38	77.0
2	13.8	1.075	6.7	0.86	0.38	78.3
3	15.2	1.075	6.7	0.98	0.35	76.8
4	15.3	1.060	6.6	0.90	0.36	76.9
5	15.1	1.121	7.0	0.89	0.37	76.6
6	14.8	1.100	6.9	0.90	0.33	77.1
7	13.5	1.118	7.0	0.92	0.38	78.2
8	15.45	1.060	6.6	0.89	0.38	76.7
		* inclue	des fibre			

The sum of the average figures for moisture (5 hours at  $100^{\circ}C$  under vacuum) protein, fat and ash is 22.8 per cent.; therefore a figure of 80 per cent. for "starch by difference" is too high and 77-78 per cent. would be more appropriate. A figure of 1.1 per cent. for rice nitrogen is nearer the average than 1.0 per cent.

Markland<sup>2</sup> discusses the respective merits of the factors 6.25 and 5.70 for the conversion of rice nitrogen to rice protein. Authorities may be quoted for both these figures, while a third factor of 5.95 for rice is quoted by Orr and Watt<sup>3</sup>, who list actually determined factors for different grains, legumes, nuts, etc. Kent-Jones and Amos<sup>4</sup> use 5.70 for wheat and flour and 6.25 for other cereals; McCance and Widdowson<sup>5</sup> use 6.25 in the 1940 edition and 5.70 in the 1960 edition for all cereals, but state no reason for the change. In Table I, N x 6.25 was used, but had N x 5.95 or N x 5.70 been used instead, the figure for "starch by difference" would only have been increased by 0.3 per cent or 0.6 per cent. respectively.

## Calculation of the Milk Content.

Three methods of calculation of milk content using the fat, the protein and the lactose respectively are described by Dalley and Wood. Their calculation from the fat, assuming as it does the fat content of the original milk to be 3.6 per cent. seems to be of questionable validity. The fat content of milk is very variable and much quite genuine whole (i.e. full cream) milk contains less than 3.6 per cent. fat. Likewise the ratio of fat to solids-not-fat in bulk milk is by no means constant, and the figures quoted by Dalley and Wood in discussing their samples C and D are not unusual. Although 8.5 per cent. is the legal presumptive minimum S.N.F., much genuine milk fails to reach this standard at certain times of the year.

Calculation from protein is liable to errors from the allowance which has to be made for the rice protein. These errors arise not only from deviations in rice composition discussed above, but also from the fact that the figure for starch (and hence the total rice) in the pudding has to be obtained by difference and must therefore be affected by the experimental errors in all the other analytical determinations.

The most reliable method is calculation of the milk content from the lactose, since this is solely derived from the milk and (subject to qualifications referred to below) should give a direct estimate of milk S.N.F. Vieth<sup>6</sup> in propounding the ratio of 13:9:2 for lactose:protein:ash in milk did not define the state of the lactose, but it can be assumed from an earlier publication<sup>7</sup> that he intended the figure for anhydrous lactose to be used.

Experience in this Laboratory is that the mean analytical figures for genuine bulk milks come much closer to this ratio if the figure for lactose hydrate is used. Markland<sup>2</sup> refers to the possibility of "a little seasonal variation in the composition of milk". Monthly variations in the ratio of lactose to protein in milk from different herds have been described by Overman<sup>8</sup> and variations according to the stage of lactation by Vanschoubroek<sup>9</sup>, and by Leydolph and Ulrich<sup>10</sup>. Our experience with bulk milk as used for manufacture in this country is that the seasonal variation is very marked (ranging from about 1.3 to 1.6 for lactose hydrate divided by protein). Since the analyst is unlikely to know the time of year when the pudding was manufactured, he cannot allow for this variation and has to rely on a mean figure. We found that the overall average for analyses covering a period of six years was very close to 1.45, i.e. 13/9, and the average for S.N.F./ Lactose hydrate was  $\frac{24}{13}$ . Therefore we use this ratio when calculating the milk S.N.F. content of rice puddings, and with the laboratory-prepared puddings this does give results which agree well with the known composition.

Attention should also be drawn to another factor which can affect the calculation of milk content from lactose.

When milk is heated, a lactose-protein compound may be formed; it has been shown by several workers<sup>11</sup>, 12, 13 that this reaction is irreversible, and that the bound lactose is not recovered by dialysis or when a serum is prepared by protein precipitation. In a laboratory experiment "puddings" were made from mixtures of milk and pre-boiled rice and lactose was estimated before and after sterilising. The results showed that such a reaction does occur, a lower figure for lactose being found in the sterilised pudding. The error in the calculated milk content consequent on this was about 1 - 2 per cent.

### RESULTS AND DISCUSSION.

The combined effect of the above considerations may be illustrated by the analyses and calculations shown in Table II. (facing)

Samples A, B and C were puddings prepared in the laboratory. The ingredients were weighed into the cans, the milk and rice (sample 1, Table I) being of known composition. In B and C, 3 per cent. and 5 per cent. respectively of water was included to see if this could be detected in the analysis. Samples D and E were of factory manufacture made at different times of the year.

For purposes of calculation, a figure for S.N.F. of 8.5 per cent., the legal presumptive minimum, has been assumed for milk. If the actual S.N.F. of the milk is higher than this then the calculated milk content of the pudding will be higher than the true value. In this case the method of calculation gives the benefit of the doubt to the manufacturer. On the other hand when the S.N.F. is below 8.5 per cent., and also in puddings containing added cream where the S.N.F. of the enriched milk is lowered, a low value for milk content would be obtained.

In calculating the starch in the pudding by subtracting the sum of the other constituents from the total solids, it has been assumed that the lactose in the dry residue is in the anhydrous form, in agreement with Dalley and Wood<sup>1</sup>. This point is under investigation, but results to date support this view rather than that of Halliday et al. <sup>14</sup> who suggested that the lactose in a total solids estimation for milk is in the hydrated state.

Comparison of the composition derived for samples A, B and C (Table II) with their known composition is given in Table III.

### TABLE II

ANALYSIS OF CANS OF RICE PUDDING

Sample	A.	В.	С.	D.	E. ø
Analyses:	%	%	%	~/0	70
Total solids	23.2	22.3	22.2	22.9	21.9
Fat	3.37	2.60	2.66	3.08	2.98
Nitrogen	0.516	0.535	0.517	0.539	0.496
Protein $(N \times 6.38)$	3.29	3.41	3.30	3.44	3.17
Sucrose	5.57	5.50	5.45	5.42	5.30
Lactose (anhydrous)	3.81	3.66	3.63	3.73	3.8
Ash	0.63	0.63	0.62	0.69	0.72
Starch (by difference	6.53	6.52	6.50	6.52	5.9
Calculations.					
Lactose (as hydrate)	4.01	3.86	3.82	3.93	4.0
Milk=Lactose hydrate <sup>*</sup> x 21.72'	87.1	83.9	83.0	85.3	87.1
Rice=Starch $\times \frac{100}{78}$	8.35 <sup>7</sup>	8.35	8.33	8.35	7.55
RiceN=Ricex0.011	0.092	0.092	0.092	0.092	0.083
Total N	0.516	0.535	0.517	0.539	0.496
Milk N	0.424	0.443	0.425	0.447	0.413
Milk=MilkN $\ge 200\phi$	84.8	88.6	85.0	89.4	82.6
Mean Milk Content	86.0~	86.3	84.0	87.3	84.8
$Milk+Rice+Sugar^{\theta}$	99.9	100.2	97.8	101.1	97.7
Fat content of original milk = Fat x 100 milk content	3.91	3.02	3.18	3.53	3.52
Ratio Lactose hydra Milk Protein	te∕ 1.48√	1.37	1.41	1.38	1.52
$\frac{24}{13} \times \frac{100}{8.5} = 21.72^{4}$	, ,	¢6.38 ×	$\frac{24}{9}$ x	$\frac{100}{8.5} = 20$	0.1

θ The mean of the two figures for milk, calculated from lactose and protein, has been used as, in view of the variations in composition already discussed, this is probably nearer the correct figure.

# TABLE III

# COMPARISON OF CALCULATED COMPOSITIONS OF CANNED RICE PUDDINGS

Sample.	Α.		В	•	с.	
	Calcu- lated %	Known %	Calcu- lated %	Known %	Calcu- lated %	Known %
Milk Content - calcd.from Lactose	87.1	_	83.9	_	83.0	-
- calcd.from Protein	84.8	-	88.6	-	85.0	-
Mean	86.0	86.0	86.3	83.2	84.0	81.2
Sugar	5.57	5.6	5.50	5.6	5.45	5.6
Rice	8.35	8.2	8.35	8.2	8.33	8.2
Water(by difference.)	Nil	Nil	Nil	3	2.2	5
Fat content of original milk	3.91	3.88	3.02	3.02	3.18	3.14
S.N.F. of original milk		8.48	_	8.78		8.57

It will be seen that in sample A, figures show quite good agreement, but that with B and C (made using milks of higher S.N.F.) the calculated milk contents are about 3 per cent. high so that the 3 per cent. of added water in B and part of the 5 per cent. in C have not been detected. This gives some indication of the limitations of the method for detecting added water.

The figures given by Dalley and Wood<sup>1</sup> in Table II of their paper have been recalculated using  $\frac{24}{13}$  x hydrated lactose for M.S.N.F. and assuming 78 per cent. starch and 1.1 per cent. nitrogen in rice; the results are shown in Table IV. Dalley and Wood's figures are included in brackets.

### TABLE IV

RECALCULATION OF DALLEY AND WOOD'S ANALYSES

	Can A.	Can B.	Can C.	Can D.
Hydrated lactose	3.93	4.17	3.51	3.70
Milk (Hydrated lactose x 21.72)	85.5 (81.0)	90.6 (86.0)	76.4 (72.3)	80.5 (76.2)
Milk nitrogen	0.413	0.431	0.396	0.429
$Milk(MilkN \times 200)$	82.6 (84.8)	86.2 (88.4)	79.2 (82.0)	85.8 (88.2)
Milk+Rice+Sugar	98.9 (100)	101.3 (101.3)	92.9 (97.1)	97.1 (102.2)
Hydrated lactose/ Milk Protein	1.49	1.51	1.38	1.35

On this basis there is no systematic difference between the milk contents derived from the lactose and from the protein. The differences are now random and numerically smaller than those of Dalley and Wood<sup>1</sup>, and are attributable, probably, to variation in the milk used from the assumed mean value for Vieth's ratio.

The use of abnormal milk powder as a possible cause of anomalous results has been suggested both by Dalley and Wood<sup>1</sup> and Markland<sup>2</sup>, but this seems very unlikely. Manufacturers would be in danger of infringing the Labelling of Food Order if milk powder were included in rice puddings without suitable declaration on the label.

It is concluded therefore that since the composition of the ingredients, notably milk and rice, is variable and mean values have to be assumed for purposes of calculation, it is not possible to assess, from analyses of the type discussed, small amounts of added water present in canned rice puddings. Quantities such as 7.1 per cent. in Dalley and Wood's Can C, however, as recalculated, would seem to be significant.

# SUMMARY AND CONCLUSIONS.

1.

Analyses of samples of rice actually used for canned rice puddings have given average figures for starch, protein and moisture of 77.2 per cent, 6.75 per cent. and 14.8 per cent. respectively. These figures are significantly different from those used by Dalley and  $W \operatorname{ood}^1$ .

No systematic difference is found between milk contents calculated from the lactose hydrate and from the protein figures, but considerable random differences are found arising from variations in the lactose/protein ratio of the bulk milk used in manufacture of different batches.

3. The best figure for milk content is probably obtained by taking the mean of figures calculated from lactose and protein.

4. In calculating the milk content a figure for S.N.F. must be assumed. For obvious reasons this should be the legal presumptive minimum 8.5 per cent. The magnitude and sign of any error in this calculation will therefore depend upon the amount by which the actual S.N.F. of the milk differs from this figure.

5. Detection of added water would thus not be certain from these analyses unless the amount is at least, say, 3 or 4 per cent.

The author wishes to acknowledge the helpful co-operation of Miss M.A. House in this work.

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70

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### SOME PUBLIC ANALYSTS OF THE PAST

### by C.H. Manley

(3, Great Brockeridge, Westbury-on-Trym, Bristol)

Dr. D.T. Lewis, the Government Chemist, in May 1962 delivered at the A.P.A's Annual General Meeting in Oxford a lecture entitled "Public Analysts - Ancient and Modern". Whilst, however, he dealt in some detail with the work undertaken by public analysts since 1860, he actually mentioned but a few public analysts, and indeed, not having been one himself, he could hardly have been expected to speak with first hand knowledge of more than a few. In fact, the only ones mentioned were Wigner, Dyer, Augustus Voelcker, amongst the earlier ones and Hamence and Garratt amongst the later.

Prior to my attending Dr. Lewis's lecture it had been suggested that as one who was now one of the older members of the profession my own recollections of former public analysts might be well worth recording.

As the son of the head of one of the Oxford College laboratories I had, whilst still an undergraduate, made the acquaintance of W.W. Fisher, M.A., F.I.C., a former Fellow of Corpus Christi, who for many years had held the official position of Aldrichian Demonstrator in the University Department of Chemistry. As such, he was not only a Senior Lecturer, but also, under Professor William Odling, in control of the Inorganic Chemistry Laboratories, in one of which he had his own quarters where he practised as Public Analyst for the Counties of Berks, Bucks, and Oxon and as Water Examiner for the State of Jersey. During my school days,I remember my father, J.J. Manley, carrying out analyses for a local dairyman, whose milk had been certified by Fisher to contain added water. Soon after graduating,I had the good fortune to join Fisher in his food and drug work and whilst with him passed my Branch E examination. Fisher, at the time of my entering his laboratory, was 69 and had been President of the Society of Public Analysts in 1899-1900, following Bernard Dyer and preceding J.A. Voelcker in this office. I liked to think of him as resembling the former Swedish Chemist Berzelius. However that might be, I learnt much from him about the composition of foods and drugs and waters, on the geological aspect of which he was an acknowledged expert.

From Fisher also, I learnt of other public analysts, whose names were entirely new to one whose knowledge of chemists had been limited almost entirely to those associated with the academic side of the science. There were, for instance, Harvey of Canterbury, Liverseege of Birmingham, Richardson of the West Riding and Ellis Richards of London. In the spring of 1914, during Fisher's absence on holiday in Somerset, Chaston Chapman and his wife called at the Laboratory, which, following the appointment of W.H. Perkin junior as Odling's successor, had been moved to a room on the ground floor of the University Museum facing Parks Road. Chapman was Public Analyst for St. Albans, but his main source of revenue was his consulting practice in Duke Street, Aldgate, one of his specialities being brewing chemistry.

Originally known as A.C. Chapman (A for Alfred) he in due course signed himself "A. Chaston Chapman", which added impressiveness to a practice of increasing importance. Hitherto, the only Chapman familiar to me had been D.L. Chapman, Fellow and Tutor of Jesus College, Oxford, well-known for his work on the actinic combination of gases and whose lectures on Inorganic Chemistry I had attended after H.B. Baker had resigned the Lee's Readership to become a professor at the Imperial College, South Kensington. I saw in Chaston Chapman a resemblance to the former great French Chemist, Dumas. His manner and general appearance were striking and his "Quite, quite" as I showed him items of interest in the large hall of the Museum itself seemed to be one of his characteristic expressions. When in London about a year later, I had the pleasure of returning the call and I met him again and for the last time soon after the First World War at the Manchester Literary and Philosophical Society in John Dalton Street during his presidency of the Institute of

Chemistry. It transpired that he was a native of Pool-in-Wharfedale, and following his untimely death at the age of 63 in 1932, I was present at the opening of a library to his memory in the University Chemistry Department at Leeds, for which city I had in 1928 become the first full-time public analyst. (Chaston Chapman it will be recalled was the first public analyst to receive the F.R.S.) The principal speaker at the library opening was Dr. Bernard Dyer, who had been my Food and Drugs examiner and a friend of Fisher. Fisher had died in 1920, when his practice closed down and his appointments went to others outside Oxford.

Dyer had extensive laboratories in Great Tower Street, London, his original partner in the practice being James Nimmo, who was away when I called on Dyer in 1915. Many years later (in 1933) I met Dyer again when first serving on the Council of the Society of Public Analysts and sat near him at its 60th Anniversary Dinner held in the Empire Room of the Trocadero Restaurant. Sir Bernard Spilsbury was one of the guests of honour. After Dyer's health had been proposed by Arnaud, the retiring president, and the hope expressed that Dyer would be present also at the 70th Anniversary Dinner, Dver responded, saying that he had every intention of being so present, and indeed he would have been but for the Second World War, during the later years of which he had been prevailed upon to move to Burford in Oxfordshire. Meantime the practice had been converted into a limited liability company, with George Taylor and Hubert Hamence as Dyer's partners. We continued to correspond until shortly before his death in 1948, and I was one of those present at the 1st Bernard Dyer Memorial Lecture delivered by Sir John Russell in the Royal Society's rooms in March 1950.

As with other chemists, my food and drug career was interrupted by war work, but in due course I was appointed an Additional Public Analyst for the City of Manchester to Harri Heap, M.Sc., F.I.C. Heap had held the Senior appointment since 1920 and was experiencing the effect of overstrain through having no deputy to share the burden of increasing work. Heap was 38 when I joined him at the pleasantly situated laboratories in York Place off the Oxford Road and adjoining the Royal Infirmary. On the other side of the corridor which divided the building were the quarters of the Professor of Bacteriology, who with Heap lectured to D.P.H. Part II Students, Heap thus holding a University appointment as well as his City one. Heap proved a most congenial chief, who was, moreover an excellent raconteur

even to the extent of being at times (though possibly unconciously) somewhat of a romancer. Once when asked the reason for the unusual spelling of his Christian name he said that he understood that it meant "Land of the Indian Sun!" My first job (in Heap's absence at Whitsuntide) was the assay of some morphine hydrochloride tablets which a nurse addict, by means of forged prescriptions, had been obtaining in a considerable quantity and this involved my giving evidence before the magistrates within a fortnight of my assuming office. Heap returned from holiday to be confronted with a fatal case of belladonna poisoning, two boys having eaten Deadly Nightshade berries in Heaton Park on the north side of the city. One was saved by his mother administering a dose of castor oil and the other succumbed. We gave joint evidence in a safe-breaking charge (ammonia alum being found in the safe packing and the trouser turn-ups of the two accused). Also we appeared in a case in which a spiteful Greek had attempted to cause grevious bodily harm to a rival by introducing mercuric chloride into a well known brand of cocoa put through his intended victim's letter box. On one occasion Heap found himself opposed by T.R. Hodgson, M.A. (Cantab.), F.I.C., Public Analyst for Blackpool, who had a private practice in Manchester and had published work in "The Analyst" but was never a member of the S.P.A. When several years later I read a paper at the Royal Sanitary Institute Congress in Blackpool, Hodgson proposed the vote of thanks.

The formation of the North of England Section of the S.P.A. in 1925 brought me into initial contact with W.H. Roberts (Liverpool), John Evans (Sheffield) and H.T. Lea (Halifax). I already knew S.E. Melling (Cheshire) and G.D. Elsdon (Salford). Roberts came over one day to give Heap supporting evidence in a Gruyere cheese case, Heap having reported adversely on the cheese for its.being a partly skimmed one. Roberts, grey-haired, gave his evidence with eyes half-closed and with fingers playing on the edge of the witness box. The case was lost as the stipendary magistrate accepted the defending solicitor's submission that as the food inspector had specifically pointed at the cheese and said "Iwant that ", he had been given what he asked for. John Evans, successor to A.H. Allen, became in turn chairman of the North of England Section and President of the S.P.A. He was round faced, bespectacled and jovial, and smoked numberless cigarettes. As a non-smoker myself I felt that the latter must have contributed materially to the heart trouble to which he eventually succumbed. In due course from Sheffield University he

received the degree of Hon. M.A. in recognition of his services to analytical chemistry, which included one of the earliest methods for the determination of alcohol in urine and the microscopical recognition of leaf adulterants of mint.

Henry Lea was the Section's first Honorary Secretary and once unsuccessfully opposed me in a watered milk case in which I had applied the Hortvet test while he had relied on the ash figure. He died unexpectedly at the age of 49 in 1939 whilst under an anaesthetic for a throat operation. Two other analysts and myself attended the funeral on a high spot outside the town. Lea's practice was in due course acquired by the present holder, Raymond Mallinder, later to become West Riding County Analyst.

With my move to Leeds in 1928 I was invited to join the Yorkshire Analysts' Association, to which belonged not only men like John Evans, J.A. Foster (East Riding), L.G. Paul (Huddersfield), F.W. Richardson (Bradford and West Riding), Arthur Scholes (Middlesborough) and A.R. Tankard (Kingstonupon-Hull), but also men from across the county's borders, J.T. Dunn (Newcastle), C.J.H. Stock (County Durham) and G.D. Elsdon (now Lancashire County). The Association met quarterly over lunch in the old Queen's Hotel, Leeds, adjoining the City Station and was convened by W.D. Mackey, whose name is associated with the well known oil test. He, however, held no public appointment and practised in Victoria Chambers, South Parade. At the meetings problems of mutual interest were discussed and much helpful information exchanged. On occasion argument became somewhat heated, especially when Foster and fees were mentioned, and suggestions of undercutting made. It did not take much to ruffle Captain Foster (as he liked to be called) and at one meeting it was jocularly stated that in Hull anything crushed except Captain Foster!

Following the death in 1927 of B.A. Burrell, owner of the practice of T. Fairley and Partner in Park Square, Richardson had carried on the Leeds work pending my assumption of the role of the City's first full time public analyst. He was 69 when I made his acquaintance and about the time I joined Fisher had devised a neat method for the determination of boric acid in cream, involving the use of copper sulphate and phenolphthalein. The stories of his <u>causes celèbres</u> caused me considerable amusement. One of these concerned a wine claimed to contain egg, which Richardson had found absent, the wine being condemned accordingly and the manufacturers summoned. Whereupon the defence brought three

silk-hatted gentlemen, namely the learned Sir Archibald Bodkin, K.C., Colonel Cassels, Editor of the British Food Journal and the great metropolitan analyst, Otto Hehner, all the way from London to Bradford to crush a local analyst! Otto Hehner in evidence was said to have stated that he had witnessed the manufacture of the wine and had actually seen the egg introduced, but whether or not this was prior to or subsequent to the introduction of the legal preceedings I cannot say. Much to Richardson's distress (and possible disgust) the Bench gave the defendant company the benefit of the doubt and dismissed the case. A second story related to a case of watered milk, about the composition of which there was no dispute. The offending vendor was, however, represented by the well-known Leeds solicitor, Mr. Arthur Willey, renowned for his fighting qualities. Accordingly, as he could not challenge Richardson on either analysis or his conclusions, he suddenly rounded on him with the question "I suppose, Mr. Richardson, that you will receive your fee for this?" to which in a flash he received the reply "And I am perfectly certain, Mr. Willey, that you will already have received yours! " for Richardson said he was well aware that Arthur Willey refused to take a case without at least a 'fiver' having first been deposited! Yet a third cause celèbre concerned a Bradford doctor, one of whose patients had exhibited symptoms of chemical poisoning, suspicions falling upon a tin of coffee, on tasting a little of which on the tip of a spatula Richardson stated he had detected strychnine. "Doctor", he said, "Your patient has been murdered! " Unexpectedly, the outcome was not the Criminal Assize Court, for the doctor, despite receiving the confirmation of his suspicions, was loath to proceed with the matter and there, apparently, it disappointedly ended.

In June 1931, Richardson moved to Bournemouth with the intention of retiring, but with the onset of the financial crisis the following September he decided to retain his interest in the Bradford practice, in which he was partnered by his nephew Frederick Jaffe. Whenever I visited my parents at Queen's Park I invariably called upon Richardson, who, incidentally, had a laboratory attached to his house bordering some pinewoods. After arranging to meet him in the town one morning for coffee, a downpour of rain looked likely to wreck our plans. Accordingly, I telephoned Richardson's house as I was anxious that at his advanced age my would-be host should take no risks. To my surprise I learnt that he was about to set off despite the rain "For", said he "we are not yet soluble in water!" Of the other members of the Y.A.A., Tankard for many years did splendid work as City Analyst and Bacteriologist

for Kingston-upon-Hull before enjoying honourable retirement and living to see his former deputy and successor, Douglas Bagnall himself retire in 1962. As regards looks he almost reminded me of Molotov. Like others of us he was much to the fore in advocating extended standards for foods in the period between the two world wars and that at a time when the Ministry of Health seemed almost indifferent to the question. He has had the satisfaction of seeing most of his hopes more than realised.

Elsdon may be said to have 'made' the Salford appointment not only by his extensive work on the determination of butter in margarines containing coconut and palm kernel oils in addition to a basic fat like lard, but also by the publicity which he persuaded the local press to give to his general work in which he sought to obtain proper standards for commodities like lemon cheese and jam, especially when the former was sold as 'home made'.

His move to Lancashire County in 1926 took him to spacious and well equipped laboratories at Liverpool which the members of the North of England Section officially visited and so saw the work which he and his deputy, J.R. Stubbs, were carrying out on the refractive index of the serum of milk. One worker in the industry had erroneously claimed that determination of this property made it possible to distinguish between a watered milk and one that was naturally deficient in non-fatty solids. Elsdon also exhibited his polarimeter illuminated by a mercury vapour lamp and equipped with a protective eyepiece. For myself, I preferred to play for safety and use a sodium lamp for the Zeiss-Winckel model which I had installed.

Having completed his refractive index work, Elsdon turned his attention to the freezing point method being used by Hortvet in the U.S.A. which was a simplification of that described by Monier-Williams some years previously. As a result, the majority of public analysts in England and Wales were using the apparatus to good effect in the early 1930's and in due course Elsdon and Stubbs were able to summarise the results of the determination of the freezing points of 1000 milks. Surprisingly, in 1937 Elsdon resigned his appointment to become Chief Inspector to the Lancashire Rivers Board, and eight years afterwards at a North of England Section committee meeting on January 27th, 1945, came the news of his sudden death earlier that month at the comparatively early age of 56.

I never visited Melling's laboratories in Higher Broughton, Manchester, but I met Melling himself many times both before and after leaving Manchester for Leeds. Sam Melling, or S. Ernest Melling, as he signed himself, was tall with dark wavy hair, glasses and a good bearing. I have no doubt that he was also an able expert witness. At social functions he was at times persuaded to recite, his favourite piece being "How Kissing Cup won the race". He readily responded to appreciation, as for instance, to my congratulatory telegram on his installation as President of the S.P.A. in 1943, a lengthy letter resulting. As a past chairman of the Manchester Section of the Institute of Chemistry and of the North of England Section of the S.P.A., he had administratively served the profession of chemistry well. His sudden death in his study in 1954 came as sad news to his many friends in the analytical profession. Like Heap, but in a totally different way, he was a 'character'. His colleague, T.W. Lovett, took over much of his work and in due course himself served his two years as chairman of the North of England Section before dying well before the allotted span.

This account would be incomplete without reference to Dr. J.A. Voelcker and Dr. J.T. Dunn. From the former and Mrs. Voelcker I twice enjoyed hospitality at their South Kensington home when attending Annual General Meetings and Dinners, it being the custom in those pre-second world war days for the London members to entertain those coming up from the provinces. On the first occasion Eric Voelcker, J.A's partner (son of E.W., and now successor in the practice) was present at the weekend gathering which included a visit to the family laboratories at Stuart House in Tudor Street. Unlike H.E. Cox's laboratories in Billiter Square these were spared destruction by Nazi bombers. The old man had a thermometer hanging outside his dining room window and before breakfast each morning as he entered the room would call out "Temperature!" He was an excellent correspondent and we exchanged many a letter on a variety of analytical matters. He died in 1937 in his 84th year. Dr. J.T. Dunn, who was in turn President of the S.C.I. and the S.P.A. (holding the latter office in 1930-31), was an active member of the North of England Section, of which he served his time as Chairman. In my first year at Leeds he came down from Newcastle to support me in my presentation of the paper I had been asked to read on the recently introduced Preservatives Regulations 1925-27 before the joint Local Sections of the Institute of Chemistry and the Society of Chemical Industry. Boric acid had been prohibited as an added preservative and in the course of his investigations Dunn discovered it occurring naturally in minute

amounts in oranges. At meetings he was usually seen with a cigar. He was tall and wore a moustache when I first made his acquaintance but latterly was clean shaven. He was interested in atmospheric pollution especially in the metallic contents of dust and in the effects on herbage of metallic compounds emitted by factory chimneys in its vicinity. His partner, Bloxham, whom I never met, but with whom I once corresponded, had the appropriate initials of H.C.L. One of Dunn's outstanding experiences was in 1928 and concerned his investigation of the cause of the sudden illness of 50 to 60 of the staff of a Newcastle general store after drinking lemonade prepared for them overnight in enamelled buckets in a spell of hot weather. The sickness and abdominal pain proved to be due to antimony present in the glaze and dissolved by the tartaric acid present in the lemonade powder, the equivalent amount of tartar emetic in a half-pint tumbler being over 1.5 grains, compared with the B.P. emetic dose of 0.5 to 1 grains.

If I remember rightly, a similar case occurred at Hastings not long afterwards. Dunn died in his 81st year early in 1939, being therefore spared the anxieties of the second world war.

On the occasion of my second visit to the Voelckers, C.J.H. Stock was my fellow guest. He was Public Analyst not only for County Durham, but also for the remaining northern counties, and was the son of the late W.F.K. Stock, the previous owner of the practice, amongst whose useful contributions to food analysis was an attempt to make the Belfield test for the detection of beef fat in lard quantitative.

I never met the elder Stock but was associated with Cyril Stock in the application of the Hortvet test in watered milk cases and in connection with egg and milk substitute powder prosecutions his evidence was particularly helpful. Ill health eventually enforced his retirement from the practice in Darlington. Of the northern counties appointments, that of County Durham is now a full-time one, the first holder being Joseph Markland. Cyril Stock was tall, handsome, debonair, and withal impressive, being almost all that one would expect an expert analytical witness to be. Once, when Stock gave me supporting evidence in a hotly contended watered milk case, the defending solicitor, who acted for the regional branch of the National Farmers Union, failing to tie up Stock in his initial cross examination, proceeded to put a further question before the answer to the preceding one had been completed; whereupon Stock retaliated with: - "As I was saying when the defending solicitor so unnecessarily interrupted me. .... ".

Cyril Stock had three sons, but unfortunately for him none of them felt the urge to join him in the profession.

Others are better qualified than myself to write in detail about men like Hawkins, Hinks and Tickle.

I served on the Council of the S.P.A. in 1934 with Ernest Mostyn Hawkins and later came to know his son, Ernest Stephen, who, after being on the Liverpool City Analyst's staff, went out to Irak, returning to join his father in the consulting practice at Canterbury. Both are now dead.

Edward Hinks, a past president and former Surrey County Analyst, visited my laboratories in 1929 before returning to London after attending one of the first annual general meetings of the Chemical Society to be held outside London. Subsequently I met him several times at S.P.A. gatherings in the Chemical Society's headquarters in Piccadilly, where there was the impressiveness of the lecture room, with the oil painting of Thomas Graham in the background and the photographs of all the past presidents hung round three of the walls.

On Thomas Tickle, the Devon County Analyst, I paid a courtesy visit when on holiday in the vicinity in the 1950's. My former deputy, R.W. Sutton, the present Derby City and County Analyst, was his chief assistant before joining me in Leeds in 1928. Tickle (like Hawkins senior) was bearded, and sailed a yacht on the Exe. He was nicknamed "The Admiral" and was remarkably active even in his latter years.

Those of us who have had acquaintance with these giants of the past now find ourselves the senior members of the profession and therefore expected to carry on its worthy traditions, thereby giving the lead to the younger members. Concerning one thing we can take heart. During our long apprenticeship we have obtained that experience which in the end counts for so much when allied to academic qualifications and initial practical training.

> Printed and published for the Association of Public Analsts by Technical Administration Ltd., 74 South Street, Reading, Berks.