Volume 14 Part 1 March 1976

JOURNAL

OF THE

ASSOCIATION OF PUBLIC ANALYSTS

The Wine Standards Board

B. S. R. PENNEY

London and South East (Southern Area) Wine Standards Board, The Vintners Company, Upper Thames St, London EC4V 3BS

Summary of a Paper read to a General Meeting of the Association of Public Analysts held at the Royal Institute of Chemistry on 12 June 1975 in which the function of the Wine Standards Board is described.

When the United Kingdom joined the European Economic Community, it was automatically committed to applying existing EEC Regulations and, in particular, putting the EEC Wine Regulations into force from 1 September 1973. Naturally, the Ministry of Agriculture, Fisheries and Food was given the responsibility for implementing the Regulations, and after studying the procedures adopted by other Member States, MAFF held consultations with various trade representatives, trade associations and the Vintners Company.

In order to implement the EEC Wine Regulations, it was necessary to appoint a "competent agency", and the Vintners Company offered not only to set up and operate the field organisation but also to finance it for a limited number of years. The offer was accepted by the Ministry and accordingly the Vintners Company formed the Wine Standards Board, consisting of a controlling Board of part-time Members meeting regularly under the chairmanship of Sir Louis Petch. The Board then appointed eight full-time Inspectors to cover Great Britain and a part-time Inspector to deal with Northern Ireland. The Inspectors are required to visit the premises of all importers, wholesalers and bottlers of wine at least every two years to examine accounts, documents and operations to ensure that the EEC Wine Regulations are being observed. In addition to checking that proper records are being maintained, they may make additional investigations periodically if wine misdescriptions are suspected.

Thus, the Wine Standards Board is a private Company of limited guarantee, appointed as an agency for MAFF on whom final responsibility rests for matters of policy and dialogue with the European Commission in Brussels. It is not surprising that close and frequent contact is maintained between the Ministry and the Board.

There are three separate organisations involved in the application of controls on wine in the United Kingdom, namely Customs and Excise, the Wine Standards Board and Local Authorities. On importation and in bonded warehouses,

control will rest with Customs and Excise who will carry out checks on the composition of wines, documents and records relating to wine under their supervision.

From the time the wine is cleared on payment of duty from the port or inland bonded warehouses until it reaches the retail level of sale, it becomes the responsibility of the Wine Standards Board. At retail level, enforcement of the EEC provisions for wine will rest with the Consumer Protection Officers of the Local Authorities who have the responsibility for enforcement of the Food and Drugs Act and the Trade Descriptions Acts. It is vital that the three organisations maintain contact with one another at all levels in applying the Regulations and that each is supported by expert opinion when needed. For example, the Board may seek the advice of the Legal Department of the Ministry, the Customs and Excise may consult the Government Chemist, and Local Authorities their Public Analysts.

There is a vast number of EEC Regulations on wine, many of which concern matters of vineyard husbandry and vine varieties, but quite a few are of limited interest at the moment in the United Kingdom. However, there are regulations which do cover areas of interest to local authorities, namely composition, description and documentation. The principal ones are:

EEC 816/70. This Regulation covers basic rules on prices, intervention, production processes including coupage and formulations, limits for actual and total alcoholic strength, total and volatile acidity and sulphur dioxide content.

EEC 817/70. This Regulation prescribes the rules concerning quality wines and the restrictions on the use of "quality wine" as a description.

The EEC defines "quality wine" as wine deemed to be quality wine by the producing Member State and produced in specified regions appearing on lists drawn up by those Members States. There is a provision that the name of a specified region may only apply if it is a quality wine entitled to use an expression such as "Appellation Controllée" (AC) or equivalent. As a consequence, such descriptions as "Spanish Sauterne" or "Spanish Chablis" will no longer be permissible and names such as "Beaujolais" or "Entre Deux Mers" can only be used if the wine is entitled to the quality description "Appellation Controllée" which must be shown on the label.

Both EEC 816/70 and 817/70 are being revised and reprinted, and although the former is probably the one of most interest to a Public Analyst, there are a number of other regulations supplementing it in relation to the composition of wine, these being EEC 948/70, EEC 1599/71, EEC 2592/73 and EEC 2805/73.

EEC 1153/75. This basic Regulation covers the documentation of wines and the records to be kept by wine traders. It is a revised version of EEC 1769/72, the original Regulation on the subject. Under this Regulation, all movements of wine within the Community must be covered by officially issued accompanying documents which certify the nature and the description of the wine.

EEC 2247/73. This Regulation is a companion to EEC 816/70 and lists the wines which are entitled to be described as "quality wines".

EEC 2133/74. Originally due to come into force on 1 September 1975 but now deferred by Regulation EEC 1890/75 until 1 September 1976, this Regulation lays down requirements for labelling and descriptions of wine.

The Wine Standards Board is actively concerned in applying all the foregoing regulations, but on a day-to-day basis, it is mostly involved with EEC 1153/75, governing the keeping of records and the use of accompanying documents. These documents can be likened to a "birth certificate" and are of different types according to whether the wine is an EEC "quality wine", EEC "table wine" or a "Third Country Wine". In practice, these documents will accompany all bulk wine until it reaches the bottling stage and, provided that the bottler identifies himself by printing his name and address or code number on the cork or capsule, there is no further need for such documents.

The Board is therefore concerned with the adequacy of records, the absence or misuse of the necessary documents, the description and labelling of wine and the disposal of wine stocks bottled before 1 September 1973. Where English vineyards are involved, the Board's interest commences at the source and it not only controls the bottling and disposal of wine but also the movement of the grapes themselves.

It is apparent then that the primary interest of the Wine Standards Board is at the bottler stage where substitution, dilution or even plain errors may occur, resulting ultimately in the consumer purchasing a wine which he did not intend to buy. The Board's Inspectors do not profess to be technical experts on composition, flavour and "nose", their controls being simply visual and documentary. Indeed, analysis may not be necessary to prove that a fraud has been perpetrated. Simple documentary inspection of records may, for example, show that, although 1000 gallons of Pommard went into an operation, 1500 gallons were bottled and sold. In such a case, the Board, who have legal powers of access to premises and records, but not to undertake prosecutions, would advise the Ministry to institute proceedings.

The problems most likely to involve the Public Analyst are those arising from consumer complaints through the Consumer Protection Officer. The latter, in addition to consulting the Public Analyst, may also refer to the Wine Standards Board Inspector, particularly if the trouble is thought to extend to more than an isolated bottle. Indeed, there is the opportunity now for even wider mutual consultation, particularly where a Public Analyst wishes to give an opinion based on his analysis of wine. In normal circumstances, the local Inspector of the Board will be able to trace the origin of the wine back to the bottler or the importer and may also be able to assist in locating an authentic reference specimen of wine which would allow the analyst to make his pronouncement without hesitation, and it is towards this type of co-operation that the Wine Standards Board looks in the immediate future.

J. Assoc. Publ. Analysts, 1976, **14**, 5–10

The Detection of Polyphosphate Added to Frozen Chicken

R. W. TRUMAN AND G. J. DICKES
Avon County Scientific Services, Canynge Hall, Whiteladies Rd, Bristol BS8 2PR

An appraisal of the protein, meat, phosphorus and sodium contents of chicken breasts usually shows whether the chickens contain added polyphosphate.

It is a common practice to inject chickens with polyphosphate solution prior to freezing them. An aqueous solution of either disodium dihydrogen pyrophosphate or tetrasodium pyrophosphate is normally used as the injection medium. It is claimed that polyphosphate injection reduces fluid loss during the freeze-thaw cycle, assists in the retention of the natural flavour and freshness of the chicken and shortens the cooking time.

The injection, which is carried out in the breast with a pressurised gun, equipped with two or three needles, adds both polyphosphate and water to the carcase. This addition has become the subject of controversy between consumer protection authorities and the frozen chicken trade. For example, is a polyphosphate-treated chicken technically a "chicken" or is it a "processed chicken"? Is the resultant addition of water a contravention of the Food & Drugs Act 1955, Section 2, in that the chicken is no longer of the required quality? Should the presence of polyphosphate be declared in both whole chicken and chicken portions? To cover the addition of polyphosphate, is the declaration "contains emulsifying salts" specific enough and also is it correct supposing that the polyphosphate is not there in this capacity?

Hamence and Kunwardia[1] have shown that, when considered separately, the moisture, phosphorus and meat contents of whole frozen chickens may not indicate whether such birds have been treated with polyphosphate. They have also shown that, although the paper chromatographic separation of the orthophosphate from the polymerised phosphate by the method of Doro and Remoli[2] might be used to ascertain the form of phosphate in the drip liquor from frozen chicken, the injected polyphosphate undergoes hydrolysis to orthophosphate in the carcase.

An analytical scheme has been devised to discover whether a chicken has been treated with polyphosphate. Several whole frozen chickens and chicken portions known to have been treated, and several known to have been left untreated, have been analysed for those parameters most likely to show treatment differences, viz. drip liquor, moisture after thawing, protein, meat, phosphorus and sodium. When the preliminary results had been considered, it was decided to examine only the breast portion of the chicken, after collecting the drip liquor

from the whole bird, to determine the protein, meat, phosphorus, the ratio of phosphorus to protein, and sodium.

Preparation of Sample

Weigh the frozen chicken after the removal of any giblets. Allow the carcase to drip at normal room temperature for 24 hours and reweigh the chicken. Remove the breast, separating the flesh plus skin from the bone. Mince the flesh plus skin three times, mixing the sample well between each mincing.

If analysis of the flesh on the legs and on the final remnant of chicken is required, separate and prepare in a similar way.

Methods

DRIP LIQUOR

Weigh the drip liquor accumulated after the thawing of the whole frozen bird for 24 hours. Express as a percentage of the total weight of the chicken before thawing commenced.

MOISTURE AFTER THAWING, PROTEIN, FAT, ASH, TOTAL MEAT CONTENT

Analyse according to the standard methods for meat products as given by Pearson[3]. Calculate the percentage total meat from the moisture after thawing, fat, protein and ash contents using the Stubbs and More formulae[4]. Assume that the average nitrogen content per cent. in the fat-free portion is 3·9 for breast, 3·6 for legs, 3·7 for whole chicken and 3·6 for the chicken remnant after removal of breast and legs.

PHOSPHORUS

Determine the phosphorus on the ash by the vanado-molybdate colorimetric method of Hanson[5]. Express as percentage of P_2O_5.

SODIUM

Determine the sodium on the ash using the extract prepared for the determination of the phosphorus. Dilute to obtain a solution containing about 50 p.p.m. of sodium and determine the sodium content in p.p.m. by flame photometry.

Results

An initial investigation was carried out on fresh chicken samples in order to obtain the natural levels of moisture, protein, phosphorus and sodium in different portions of the birds. Chickens were divided into four portions, viz.;

(a) half a chicken;
(b) breast of the other half;
(c) leg of the other half;
(d) the remnant of the other half.

The results of analysis of these portions of six chickens are shown in Table I. The ratio of phosphorus to protein, multiplied by 100 for ease of reading, shows

clearly the difference between polyphosphate-treated and untreated chicken portions.

TABLE I
ANALYSIS OF THE MEAT FROM DIFFERENT PORTIONS OF SIX FRESH CHICKENS (TOTAL WEIGHTS BETWEEN 1600 AND 2000 g)

	Bird no.						Average
	1	2	3	4	5	6	
Moisture, per cent., whole	59·6	61·6	58·4	59·0	56·5	62·8	59·7
Moisture, per cent., breast	66·4	68·6	67·0	67·1	57·8	69·7	66·1
Moisture, per cent., leg	62·0	66·2	59·7	62·2	60·4	64·4	62·5
Moisture, per cent., remnant	53·7	57·7	52·3	50·3	47·5	58·0	53·3
Protein, per cent., whole	17·7	18·2	18·5	18·0	17·4	18·2	18·0
Protein, per cent., breast	21·7	21·9	21·1	20·3	19·7	22·2	21·2
Protein, per cent., leg	17·6	18·6	17·6	17·8	17·2	17·7	17·8
Protein, per cent., remnant	14·6	16·8	15·6	13·9	14·0	16·4	15·2
Total meat, per cent., whole	98	97	102	100	100	97	99
Total meat, per cent., breast	100	98	97	94	102	98	98
Total meat, per cent., leg	97	96	100	98	98	95	97
Total meat, per cent., remnant	96	100	101	97	99	98	99
Phosphorus (as P_2O_5), per cent., whole	0·43	0·38	0·40	0·35	0·43	0·37	0·39
Phosphorus (as P_2O_5), per cent., breast	0·47	0·47	0·47	0·46	0·43	0·48	0·46
Phosphorus (as P_2O_5), per cent., leg	0·41	0·46	0·41	0·37	0·38	0·41	0·41
Phosphorus (as P_2O_5), per cent., remnant	0·29	0·34	0·33	0·29	0·29	0·33	0·31
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, whole	1·06	0·91	0·94	0·85	1·08	0·89	0·96
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, breast	0·95	0·94	0·97	0·99	0·95	0·94	0·96
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, leg	1·02	1·08	1·02	0·91	0·96	1·01	1·00
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, remnant	0·87	0·88	0·92	0·91	0·90	0·88	0·89
Sodium, p.p.m., whole	870	740	730	770	710	750	760
Sodium, p.p.m., breast	690	720	650	690	680	640	680
Sodium, p.p.m., leg	930	830	790	850	850	650	820
Sodium, p.p.m., remnant	740	780	770	720	980	820	800

A similar analysis was carried out on two chickens (birds 7 and 8) which had been put through the full spin-chilling, freezing and polyphosphate injection procedures. As a control, two others (birds 9 and 10) were analysed which had been through the spin-chilling and freezing procedures only. The results are given in Table II, which includes the drip liquor figures.

After these investigations, it was decided to concentrate future analysis on the breast portions of the chicken, since the breast showed greater differences in the chosen parameters. Results for untreated and polyphosphate-treated frozen chicken breast samples cut from whole birds for retail sale are shown in Table III. From the analysis of both untreated and polyphosphate-treated

TABLE II

ANALYSIS OF THE MEAT FROM DIFFERENT PORTIONS OF FROZEN, POLYPHOSPHATE-TREATED AND UNTREATED CHICKENS (TOTAL WEIGHT BETWEEN 1300 AND 1700 g)

	Treated		Untreated	
Bird no.:	7	8	9	10
Drip liquor, per cent.	5·0	5·3	6·2	5·4
Moisture after thawing, per cent., whole	64·6	68·7	63·8	68·2
Moisture after thawing, per cent., breast	71·4	76·3	66·4	72·5
Moisture after thawing, per cent., leg	65·1	69·9	63·7	69·8
Moisture after thawing, per cent., remnant	55·6	63·2	64·3	56·3
Protein, per cent., whole	16·6	16·9	17·1	19·8
Protein, per cent., breast	18·9	17·8	20·1	23·0
Protein, per cent., leg	18·7	18·2	16·6	18·4
Protein, per cent., remnant	13·6	16·2	17·9	15·0
Total meat, per cent., whole	89	86	93	98
Total meat, per cent., breast	86	78	95	99
Total meat, per cent., leg	97	92	93	93
Total meat, per cent., remnant	90	93	97	96
Phosphorus (as P_2O_5), per cent., whole	0·47	0·51	0·42	0·46
Phosphorus (as P_2O_5), per cent., breast	0·67	0·68	0·49	0·55
Phosphorus (as P_2O_5), per cent., leg	0·56	0·42	0·39	0·43
Phosphorus (as P_2O_5), per cent., remnant	0·40	0·42	0·47	0·42
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$; whole	1·24	1·32	1·07	1·01
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$ breast	1·55	1·67	1·06	1·04
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, leg	1·31	1·01	1·03	1·02
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$, remnant	1·28	1·13	1·15	1·22
Sodium, p.p.m., whole	1190	1630	620	620
Sodium, p.p.m., breast	2850	2310	450	630
Sodium, p.p.m., leg	940	970	620	730
Sodium, p.p.m., remnant	1090	1160	610	670

chicken breast samples, ranges of those parameters of diagnostic value have been deduced and are given in Table IV.

It was thought that a preliminary indication of whether a chicken has been polyphosphate-treated would be shown in the differences in the phosphate content of the drip liquors. This was clearly a possibility, since Hamence and Kunwardia[1] showed that there was a distinct difference between the phosphate content of the drip liquors of two treated and two untreated birds. We analysed the drip liquor of eight treated and eight untreated birds, the results being shown in Table V.

Discussion

Table I shows the difference in the analytical figures for whole chicken, breast, leg and the remnant after removal of breast and leg. The breast has

TABLE III
ANALYSIS OF BREAST FROM FROZEN WHOLE CHICKENS

	Polyphosphate-treated								Untreated							
Bird no.:	7	8	11	12	13	14	15	16	9	10	17	18	19	20	21	22
Drip liquor, per cent., (whole bird)	5·0	5·3	5·8	7·5	9·0	12·5	8·7	6·7	6·2	5·4	8·2	5·1	5·5	7·0	2·9	4·4
Moisture after thawing, per cent.	71·4	76·3	74·6	77·6	72·4	78·2	75·1	79·2	66·4	72·5	70·5	–	–	–	–	74·6
Protein, per cent.	18·9	17·8	19·8	18·3	20·0	20·5	18·2	16·2	20·1	23·0	21·8	20·0	20·9	22·5	22·4	23·3
Total meat, per cent.	86	78	85	75	82	84	76	67	95	99	91	97	94	95	100	96
Phosphorus (as P_2O_5), per cent.	0·67	0·68	0·73	0·61	0·46	0·57	0·71	0·88	0·49	0·55	0·53	0·43	0·53	0·53	0·51	0·58
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$	1·55	1·67	1·61	1·46	1·00	1·21	1·70	2·37	1·06	1·04	1·06	0·94	1·11	1·03	0·99	1·09
Sodium, p.p.m.	2850	2310	1470	3150	1230	1500	2500	3500	450	630	600	490	420	380	560	850

the greatest moisture after thawing, protein and phosphorus contents when compared with the other portions.

A study of Table II shows that, as anticipated, the phosphorus and sodium contents of polyphosphate-treated whole chickens are higher than those of untreated ones. The protein and total meat contents of the treated whole birds are lower than those parameters for the untreated chickens. The most marked differences in phosphorus, sodium, protein and total meat contents are in the

TABLE IV
ANALYTICAL RANGES OF CHICKEN BREASTS, POLYPHOSPHATE-TREATED AND UNTREATED

	Treated	Untreated
Protein, per cent.	15·5–22·0	19·5–24·0
Total meat, per cent.	65–90	90–105
Phosphorus (as P_2O_5), per cent.	$\geqslant$ 0·45	0·40–0·60
$\frac{\text{Phosphorus} \times 100}{\text{Protein}}$	$\geqslant$1·00	0·75–1·15
Sodium, p.p.m.	1000–5000	350–1000

breast samples, the breast being the injection site. With a decrease in protein content and an increase in phosphorus content for treated, as compared with untreated, chicken breast, the ratio, phosphorus $\times$ 100/protein, gives a good indication of polyphosphate treatment. The phosphorus $\times$ 100/protein ratios calculated from the results of Hamence and Kunwardia[1] for polyphosphate-treated and untreated whole chickens support our findings.

Table III shows the results of the analyses of the breasts of eight treated and eight untreated birds. It gives an indication of the spread of results likely to be

TABLE V
PHOSPHORUS (AS P_2O_5) IN DRIP LIQUORS OF WHOLE FROZEN CHICKEN

	Polyphosphate-treated							Untreated							
Bird no.:	12	13	14	15	16	23	24	22	25	26	27	28	29	30	31	32
Drip liquor, per cent., of whole chicken	7·5	9·0	12·5	8·7	6·7	8·0	7·6	4·4	4·2	4·2	5·5	5·2	5·7	3·2	6·3	6·8
Phosphorus (as P_2O_5) in drip liquor, per cent.	0·39	0·13	0·23	0·27	0·45	0·13	0·15	0·49	0·09	0·13	0·20	0·17	0·28	0·18	0·32	0·48

obtained in each category. The sodium contents are enhanced if there has been addition of salt to the polyphosphate injection liquor.

Some overlapping of the analytical ranges occurs, e.g., protein and phosphorus contents, but Table IV is a summary of ranges by which it is usually possible to decide if polyphosphate treatment has taken place. However, it is not possible to differentiate when the results of analysis approximate to the following figures.

Total meat per cent.	90
Phosphorus (as P_2O_5) per cent.	0·50
Phosphorus × 100/protein	1·15
Sodium, p.p.m.	1000

These figures are on the border of, or overlap, the ranges given in Table IV.

Hamence and Kunwardia[1] found more phosphate in the drip liquor from treated birds than in that from untreated ones. We found that this difference cannot be guaranteed and Table V indicates the variable results.

Conclusions

By straightforward analytical methods, it is possible to place most frozen whole chickens or chicken breast portions into polyphosphate-treated or untreated categories, the important parameters being total meat, phosphorus and sodium contents and the ratio, phosphorus × 100/protein. It should be noted that for a frozen chicken to be labelled as if it had been treated with polyphosphate is no guarantee that such treatment has taken place.

We thank M. A. Chapman, the Gloucestershire County Trading Standards Officer and B. A. Williams of his Department for stimulating this study; P. Roche and S. Pearce for analytical help; A. J. Harrison, the Avon County Analyst, for permission to publish this paper.

References

1. Hamence, J. H., and Kunwardia, G. H., *J. Assoc. Publ. Analysts*, 1974, **12**, 85.
2. Doro, B., and Remoli, S., *Boll. Lab. Chim. Provinciali*, 1963, **14**, 23.
3. Pearson, D., "The Chemical Analysis of Foods", 6th Edn., Churchill, London, 1970, p. 383.
4. Stubbs, G., and More, A., *Analyst*, 1919, **44**, 125.
5. Hanson, W. C., *J. Sci. Food Agric.*, 1950, **1**, 172.

J. Assoc. Publ. Analysts, 1976, **14**, 11–16

Some Problems in the Disposal of Poisonous Waste

K. T. CHISNALL AND J. MARKLAND

Derbyshire County Council, County Scientific Adviser's Department, Matlock, Derbyshire

Factors of importance in assessing the suitability of waste material for disposal on land are discussed. Chemical composition and quantity of waste, the hydrogeology of the tip site and the possibility of reactions of the waste with incompatible materials already on the site are all important. In order to control disposal of materials a system of records is essential. This paper suggests one simple approach to this problem.

At the present time the disposal of waste is controlled by the Deposit of Poisonous Waste Act, 1972[1] with a reference to its associated Regulations[2]. The Act requires organisations responsible for the removal of waste materials to notify appropriate Local Authorities of the intention to remove waste from premises and also to indicate where the waste will be taken after removal. Such notification must be made at least three days before the time of the proposed tipping. This gives the Local Authorities only a very short time to refuse or accept the proposals or to suggest alternative means of disposal.

For the past few years, this department has been consulted on the disposal of poisonous wastes by the District Councils before Local Government Reorganisation and since then by the County Surveyor of the County Council. The possible hazards are well-known and vary from the tipping of highly inflammable liquids or potentially explosive solids to substances which are themselves poisonous or which by reaction with other substances can be poisonous. In dealing with notifications for the land disposal of these materials we have considered a number of factors of importance in assessing the suitability of a waste for land disposal. These are set out below.

We first have to deal with the waste itself and in considering this it is necessary to know the composition, the quantity and, if possible, the nature and quantities of other wastes previously tipped nearby. Frequently, notifications are presented which give a long list of materials, each of which could fit the popular conception of a hazard. Thus we find heavy metals, cyanide, phenols, and other such well-known toxic materials, each declared as a few parts per million but no other information is given. In deciding on the suitability for tipping, it is essential to know what the waste actually is. Whilst one would not deny the importance of heavy metals and cyanide, yet in parts per million quantities on a solid waste, they may well be less important than the nature of the waste itself. For example, the presence of two parts per million of lead becomes insignificant

in the context of a waste which may well contain 15 per cent. of caustic soda. It is important that the presence of toxic materials should be declared but their presence need not necessarily prevent a waste being tipped if the major constituents are themselves innocuous.

The next consideration is the quantity of waste and this should be tied in with the composition discussed above. For example, when one sees a suggestion that the disposal of 20 grams of sulphanilamide could be dangerous, one wonders in what context such a quantity could possibly be harmful on a tip, unless it is disposed of in its original container rather than distributed. On the other hand, the presence of quite a small concentration of a few parts per million of cyanide or phenol in a liquid waste which is being disposed of in thousands of gallons per week may well be undesirable. Therefore, in considering suitability for tipping, the composition and the quantity together will give an idea of the amount of actual poisonous material being deposited and this is one of the important factors. In saying this, the significance of the total quantity of poison cannot completely override the component factors of high poison concentration or total quantity of material which is to be disposed of.

The physical character of the material, solid, liquid or sludge, needs to be considered in relation to possible movement of the waste within the tip or from the tip.

When considering possible movement, the factor of what other wastes have been deposited in the area becomes important. Records are not always available on what has happened in the past but, nevertheless, this does not excuse anyone from avoiding the responsibility of knowing what is being tipped now and to take steps to ensure that incompatible wastes are not tipped near to each other. For example, the tipping of an acid waste into a waste containing sulphide is a self-evident hazard. The fact that it is probably undesirable to tip acid waste anyway is another matter but accepting that such acids are tipped then it is necessary to make sure they do the least possible harm. In other words, if there is some record of the nature of the materials which have been tipped and of the places on the tip where they have been deposited, it should be possible to avoid dangerous reactions.

The properties and composition of the waste cannot be divorced from the tip itself and here we have to consider both the geology and geography of the tip.

If the problem solely concerns solid wastes then the geological formation near to the tip is only of importance in relation to the solubility of the wastes in rainwater. With liquid wastes, the hazardous materials are already in solution and one would expect quicker percolation under these circumstances. It is obvious that if liquid waste is tipped and it constantly disappears, then either the liquid is evaporating or else there is percolation through the tip. If the waste readily evaporates, it is probably dangerous anyway and if there is percolation, then it is necessary to know where the waste liquid goes to. It seems wiser to err on the side of safety. Thus if seepage is occurring into some stratum beneath, then one presumes that at some stage in the future this stratum might become saturated and one could also presume that the liquid might reappear in some other place. On these grounds, it is better to know where the material goes rather than to say that if it does not come out in any place in a reasonably short time it is safe.

Whilst the geography of the area will be more of a planning than a scientific matter, this must be considered in deciding on the scientific problems. A very faint smell may be acceptable if it is not near any houses. The element of danger which is acceptable will depend on whether or not the public can get access to the tip, either legally or illegally.

Another aspect of the geography of the tip is the access for transport which, whilst this is again a planning matter, should not be neglected as a scientific problem. Transport can present other possible hazards which could be even worse than those of the tip itself.

Some knowledge of degree of supervision provided at the site is also desirable. It is clear, therefore, that close co-operation is needed between a number of officers of different disciplines.

Classification

The need to know what materials have been previously placed on a tip has been stressed earlier. It follows immediately that a system for the recording of deliveries of wastes to tip sites must not only cover the dangers of mixing of incompatible materials but also lead to consistent advice for wastes of similar composition.

In Derbyshire, disposals of toxic materials are made at a number of different sites and operations involve many different waste disposal contractors. The wastes are very variable in character.

Every notification of intention to deposit waste contains the following information:

(a) the tip to which the waste will be sent;

(b) the waste disposal operator making the delivery;

(c) the name of the firm generating waste and from whose premises the waste will be removed;

(d) details of the composition of the waste.

There is clearly scope for a complex card index or even computer scheme to allow for adequate cross reference. There is, however, a need to produce a quick decision on whether a waste may be accepted and for ease and speed we have evolved a fairly simple recording system based upon the card shown in Figure 1.

The card scheme is designed to make full use of the information given in the notification. The cards are classified as shown in Figure 2.

The tip sites give the first classification and this gives the main divisions in the filing system.

Within each tip site section the cards are sub-divided into sections for waste disposal contractors.

Within each section for waste disposal contractors every firm generating waste and using this contractor is allocated a separate card.

These cards are mounted in such a way that they are overlaid and the whole of the right hand column marked "Type" is always visible. This extreme right-hand column of the card is divided into boxes marked 1–30. Each box

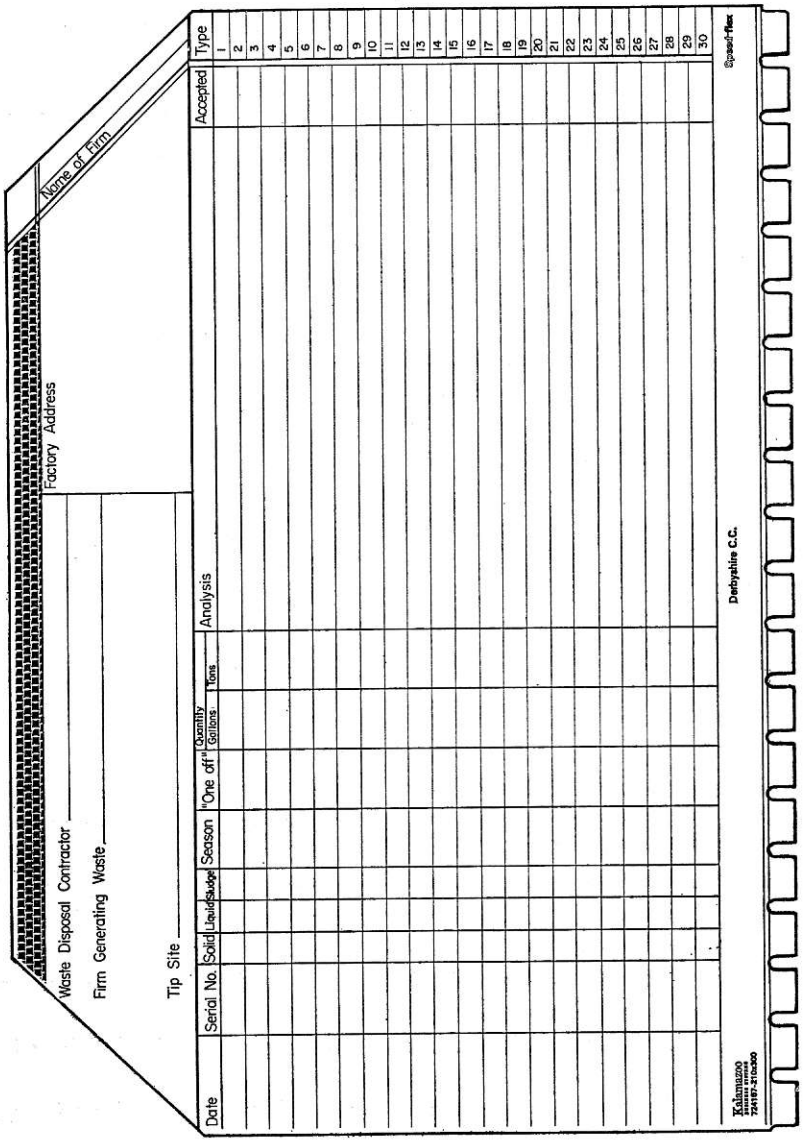

Fig. 1. Record card used for classification.

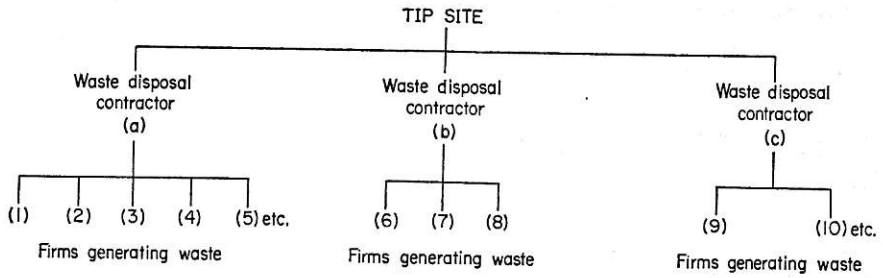

Fig. 2. Classification for one tip site. Note that each section has unlimited expansion.

represents one chemical or physical attribute. For each waste, marks are made in the appropriate boxes. So far only 21 of the boxes are used, the remainder being held in reserve. Table I shows the classification scheme adopted. For example,

TABLE I
CLASSIFICATIONS OF MAJOR CHEMICAL AND PHYSICAL PROPERTIES

Card box no.	Attribute
1.	Water soluble
2.	Organic
3.	Inorganic
4.	Acid
5.	Alkaline
6.	Inflammable
7.	Combustible
8.	Putrescible
9.	Oxidiser
10.	Organic halogen
11.	Organic nitrogen
12.	Lead, cadmium and mercury
13.	Zinc, copper, chromium and nickel
14.	Arsenic, antimony, selenium and tellurium
15.	Cyanide
16.	Asbestos
17.	Sulphide
18.	Mineral oils
19.	Detergent
20.	Plastics, rubber
21.	Phenols

The spare boxes on the card are left for future amendments.

a waste declared to contain inorganic acids would be marked in boxes 3 and 4. An asbestos waste would be marked in box 16. The card in Figure 1 shows these classifications.

If the same waste has been dealt with at some other time, there will be a card in the system and rapid scan of the visible right hand corners of the cards will find the record of previous notifications. It is easy to withdraw the card and so refer to previous action on the waste.

If there is no card present then the waste is new. It is necessary to decide whether the waste is acceptable on the basis of chemical composition and the

quantity of waste declared. Reference is then made to all the right-hand column classifications from all contractors using the tip. It is thus possible to check on possible incompatibilities and on the action taken on similar wastes.

We are grateful to Miss C. Lowton for help with organising the card filing system.

References

1. "Deposit of Poisonous Waste Act." H.M.S.O., London, 1972.
2. "Deposit of Poisonous Waste Act (Notification of Removal or Deposit) Regulations". S.I. 1972 No. 1017. H.M.S.O., London, 1972.

J. Assoc. Publ. Analysts, 1976, **14**, 17–22

Estimation of the Original Gravity of Beer

J. R. HUDSON
The Brewing Research Foundation, Nutfield, Redhill, Surrey, RH1 4HY

(This article was published in the Journal of the Institute of Brewing, 1975, **81**, 318–321 and is reproduced here with the permission of that Journal.)

A method is described for the estimation of the original gravity of beer, which is accepted as a reference method by the Laboratory of the Government Chemist.

The Original Gravity of a beer is the strength of the wort from which that beer was made, as defined by a distillation procedure[1]. As Original Gravity is used officially to monitor beers and, for example, to check on the reclamation of duty for exported beers, the method of estimation is of obvious importance in brewing laboratories. It is therefore surprising that there has been only one published description[2] of the apparatus and technique employed. Even in the extensive account of the work[3] which provided the basis of the tables which still remain in official use, it is stated that, "The samples removed were shaken to dispel the carbonic acid gas, and measured quantities . . . distilled in the usual way". Not surprisingly, somewhat different apparatus and techniques have come to be employed in different laboratories and there has been no method of checking the reliability of values between laboratories. Furthermore, the lack of a recognised method could lead to difficulties in obtaining agreement between countries on the methods to be employed to monitor international trade.

The Committee sought co-operation from the Laboratory of the Government Chemist in providing a reference method. The method described in the Appendix was derived from that in current use in the Government Laboratory and, having been tested in two collaborative trials in which that Laboratory participated, will be officially recognised as a reference method[4]. The trials were carried out according to an approved design[5] and Table I gives the statistical summary of information extracted from the results. Although from other collaborative experience[6], the results of the first trial appeared to be as good as could be expected for this type of analysis, it was decided to proceed with a second trial using slight modifications in technique. As seen from Table I these did not bring any improvement in reliability and indeed for Beer Types B and C, results from one laboratory were eliminated and the statistics recalculated for 9 laboratories rather than the 10 which participated. It is seen that the within-labora-

TABLE I
PRECISION AND VARIATION IN ESTIMATION OF ORIGINAL GRAVITY

Beer type	Trial no.	No. of collaborators	Grand average $\bar{X}$	Within lab. error S_r	Between lab. error S_b	Coefficients of variation: Precision $100CV_r$	Coefficients of variation: Between labs. $100CV_b$	Precision within lab. $\pm 2S_r$	Diff. between single results from any two labs. $\pm 2\sqrt{S_r^2 + S_b^2}$
A	1	11	33·28	0·151	0·279	0·454	0·838	0·30	0·63
	2	10	33·71	0·240	0·308	0·712	0·914	0·48	0·78
B	1	11	39·44	0·214	0·230	0·543	0·583	0·43	0·63
	2	10	39·76	0·194	0·500	0·488	1·257	0·39	1·07
		9	39·91	0·151	0·198	0·378	0·496	0·30	0·50
C	1	11	46·77	0·148	0·391	0·316	0·836	0·30	0·84
	2	10	47·22	0·151	0·307	0·318	0·650	0·30	0·68
		9	47·31	0·160	0·124	0·337	0·263	0·32	0·40
D	1	11	79·51	0·319	0·663	0·401	0·834	0·64	1·47
	2	10	80·51	0·201	0·333	0·250	0·414	0·40	0·78

$100CV_r = 100S_r/\bar{X}$; $100CV_b = 100S_b/\bar{X}$; S_r and S_b are standard deviations (see Reference 5)
Results expressed as 1000 (S.G.—1000).

TABLE II
COMPARISON OF ERRORS

	Beer	Within lab. error precision $\pm 2S_r$: Trial 1	Within lab. error precision $\pm 2S_r$: Trial 2	Overall error $\pm 2\sqrt{S_r^2 + S_b^2}$: Trial 1	Overall error $\pm 2\sqrt{S_r^2 + S_b^2}$: Trial 2
O.G.	A	0·30	0·48	0·63	0·78
	B	0·43	0·39 (0·30)	0·63	1·07 (0·50)
	C	0·30	0·30 (0·32)	0·84	0·64 (0·40)
	D	0·64	0·40	1·47	0·78
S.I.	A	0·045	0·126	0·109	0·202
	B	0·092 (0·081)	0·085 (0·071)	0·117	0·200 (0·128)
	C	0·074	0·063 (0·065)	0·167	0·189 (0·086)
	D	0·095 (0·074)	0·071	0·295 (0·123)	0·239
R.G.	A	0·119	0·149 (0·158)	0·147	0·564 (0·266)
	B	0·078 (0·083)	0·114	0·172 (0·089)	0·314
	C	0·091	0·127 (0·129)	0·170	1·345 (0·199)
	D	0·116	0·128	0·180	0·252
P.G.	A	0·094	0·174 (0·184)	0·142	0·840 (0·303)
	B	0·083	0·081	0·269	0·372
	C	0·107	0·151 (0·160)	0·191	1·695 (0·207)
	D	0·145 (0·131)	0·102	0·542 (0·138)	0·335

Figures in brackets ( ) are "corrected" estimates.

tory error [$\pm 2S_r$] is rather less than 1 per cent. and the between-laboratory error [$\pm 2\sqrt{S^2_r + S_b^2}$] is below 2 per cent.

Examination of the detailed measurements showed that the determination of the Spirit Indication is performed best and the determination of Present Gravity is performed least well. This is seen in Table II where the figures in brackets are estimates corrected from statistical considerations. As well as showing that performance of the method was a little less good in the second trial than in the first, Table II reveals that the errors increase somewhat as the Original Gravity increases.

The opportunity was taken to test the "check procedure":

Residue Gravity (R.G.) — Present Gravity (P.G.) = Spirit Indication (S.I.)

The cumulative values given in Table III show that the differences between the

TABLE III
COMPARISON OF OBSERVED AND CHECK SPIRIT INDICATIONS

Beer type	Calculated S.I. R.G. — P.G. = S.I.	Actual S.I.	Difference
A	10·83 — 5·86 = 4·97	5·15	−0·18
B	12·95 — 7·06 = 5·89	6·05	−0·16
C	18·91 — 12·81 = 6·10	6·33	−0·23
D	31·49 — 21·00 = 10·49	10·65	−0·16

actual and predicted Spirit Indications are very consistent and justify the use of the check. If the difference between these values is greater than 0·3 then the estimation must be repeated. Another test as to whether the distillation technique is satisfactory is that there should be no significant change in alcohol content if the distillate is re-distilled five times. For example, it has been stated[7] that a 10 per cent. alcohol solution should have a strength of at least 9·9 per cent. after five distillations. The results in Table IV revealed that 9 of the 11 laboratories

TABLE IV
EFFECT OF REPEATED DISTILLATION ON SPIRIT INDICATION (BEER D)

Collaborator	Spirit Indication: 1st distillation	Spirit Indication: 5th distillation	Difference
1	10·67	10·29	0·38
2	10·80	10·28	0·52
3	10·91	10·97	−0·06
4	10·83	10·71	0·12
6	10·53	10·48	0·05
7	10·65	10·55	0·10
8	10·68	10·70	−0·02
9	10·90	10·92	−0·02
10	10·80	10·78	0·02
11	10·68	10·65	0·03

had achieved a satisfactory standard in this part of the estimation but that improvement was needed in the other two.

The Committee is grateful to Dr D. B. Lisle of the Laboratory of the Government Chemist for his help and suggestions and to Mr D. G. W. Brown of Allied Breweries (Production) Ltd., Burton-on-Trent, who provided the statistical analysis of results.

References

1. "Beer Regulations, 1952," S.I. 1952, No. 2232. H.M.S.O., London, 1952.
2. "Brewing Scientific Reviews," A. Clark Doull (Edit.), Brewers Guild Publications Ltd., London, 1949, 111.
3. Thorpe, E., and Brown, H. T., *J. Inst. Brewing*, 1914, **20**, 569.
4. Laboratory of the Government Chemist (private communication), July 1975.
5. Howard, G. A., *J. Inst. Brewing*, 1973, **79**, 315.
6. 4th Report, Research Committee on the Analysis of Potable Spirits, *J. Assoc. Publ. Analysts*, 1974, **12**, 45.
7. "Report of the Government Chemist, 1973". H.M.S.O., London, p. 116.

Appendix: Reference Method For Estimation of Original Gravity

APPARATUS

Volumetric flask, 100 ml., class A.
Medium fast qualitative filter paper, 185 mm diameter.
Filter funnel.
Clock glass.
Water bath with temperature controlled at 60°F.
Flat bottom distillation flask, 500 ml.
Thorpe (Inland Revenue) condenser, BS1848.
Still head, an upwards inclining (15°) tube fitted with silicone rubber bungs.
Asbestos baffle, 150 × 150 × 10 mm with a hole 65 mm in diameter.
Asbestos shield, 300 × 300 × 5 mm.
Density bottle, 50 ml, BS733 or equivalent.
Thermometer, range 57–63 °F with a scale length not less than 5 mm per °F.

METHOD

Distillate Gravity

Adjust the temperature of the beer to 60°F and filter into a 100 ml volumetric flask. Cover the filter funnel with a clock glass to prevent loss of alcohol. Adjust the volume to 100 ml at 60°F. Transfer the contents to the distillation flask rinsing with three portions (5 ml) of distilled water. Assemble the apparatus as shown in Figure 1 using the original 100 ml volumetric flask as the receiver. Distil over approximately 85 ml taking care not to char the residue in the distillation flask. Using a few millilitres of distilled water, rinse any liquid from the inside of the condenser into the receiver. Make up to volume at 60°F and determine the specific gravity at 60°F using a density bottle.

Residue Gravity

Cool the residue in the distillation flask and transfer the contents to the same 100 ml volumetric flask previously used. Adjust the volume to 100 ml at 60°F and determine the specific gravity at 60°F.

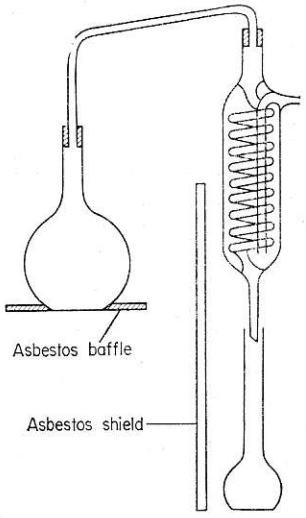

Fig. 1. Distillation apparatus.

CALCULATION

Calculate the Spirit Indication from:
Spirit Indication = 1000 (1·00000—Specific Gravity of distillate). Obtain the corresponding degrees of gravity lost from the statutory gravity lost table[1,2] for 60°F/60°F.

Calculate the Residue Gravity from:
Residue Gravity = 1000 (Specific Gravity of residue—1·00000).

To obtain the Original Gravity add to the Residue Gravity of the beer the degrees of gravity lost corresponding to the Spirit Indication.

The precision of the method is about ± 1 per cent. of the original gravity value.

CHECK

Determine the specific gravity of the filtered beer at 60°F and convert the figure obtained into "degrees of gravity".

The equation:

Spirit Indication = Residue Gravity—Present Gravity

may be used to check the experimental determination of the Spirit Indication. The value obtained by subtracting the Present Gravity from the Residue Gravity is usually about 0·16 less than the value obtained by subtracting the distillate gravity from 1000. If the difference between these values is greater than 0·3 then the determination must be repeated.

NOTES

1. The 750 ml Thorpe distillation flask is not readily available. It has been found that the use of a 500 ml flat bottom flask does not affect the precision of the determination.

2. An asbestos baffle is used to prevent charring of the residue above the liquid level in the distillation flask. A wire gauze does not give adequate protection.
3. An asbestos shield is placed between the receiving flask and the burner, to prevent loss of alcohol by evaporation.
4. The volume of rinse water must be limited to 3 × 5 ml making a total volume of 115 ml in the flask. Because it is necessary to distil 70 per cent. of the total volume of liquid to ensure complete recovery of alcohol, the volume collected should be about 85 ml.
5. As a check on the distillation, it should be possible to take the distillate and redistil it five times without significantly altering the alcohol content.
6. For large numbers of samples the re-use of the 100 ml volumetric flask for making up the residue may not be practical. For routine operation class A flasks or matched (numbered) pairs of flasks can be used, but the original volumetric flask must be used for the distillate, since this is the more critical determination.

J. Assoc. Publ. Analysts, 1976, **14**, 23–25

The Determination of Nitrate and Nitrite in Food

R. FAWCETT, D. A. TAME AND T. E. JOHNSON
Kent County Council Laboratory, County Hall, Maidstone, Kent

A method is presented for the determination of nitrate and nitrite which overcomes the difficulty in obtaining a clear solution from some foods. Results of the analysis of various foods, using the method, are given in the Appendix.

Until recently nitrates and nitrites were determined in the Kent County Council Laboratory by the method of Follett and Radcliff[1] with the modification that Cleve's acid was substituted for alpha-naphthol, as in the method of Bunton, Crosby and Patterson[2]. Difficulties were experienced in obtaining a clear solution at the stage where sulphanilic acid and Cleve's acid were added. Since the intensity of the resulting colour was to be measured on a spectrophotometer it was essential that a clear solution should be produced.

To overcome the difficulty, arsanilic acid and naphthyl ethylene diamine were used for colour development and the colour was extracted with *n*-butanol.

During the development of this method it was noticed that Whatman No. 4 15 cm filter papers contained 100–150 μg of nitrate, as sodium nitrate, which on a 5 g sample of food was equivalent to 20–30 p.p.m.

The modified method finally adopted was based on that of Elliott and Porter[3]. The details are as follows.

Reagents

1. *Alumina cream.* Neutralise a saturated solution of potassium alum (A.R.) to pH 7 with ammonia (A.R.).
2. *Naphthyl ethylene diamine dihydrochloride.* Prepare a 0·1 % w/v solution in water.
3. *Arsanilic acid monohydrate.* Prepare a 0·1 % w/v solution in 5 N hydrochloric acid.
4. *Buffer pH 9·6.* Adjust a 0·7 M ammonium chloride solution in water to pH 9·6 by the addition of 0·880 ammonia.
5. *Spongy cadmium.* Prepare by placing zinc rods in 20 % aqueous cadmium sulphate (A.R.) and leave for 3–4 hours. Separate the precipitated cadmium, wash twice with de-ionised water and then macerate with de-ionised water for 2–3 minutes. Activate by shaking with 2 N hydrochloric

acid and then wash five times with de-ionised water. Prepare the cadmium fresh for each batch of determinations and store under de-ionised water.

6. *Standard sodium nitrite solution.* A solution containing 10 mg per litre.
7. *n-Butanol.*

Procedure

Weigh 5 g of the macerated sample into a 150 ml beaker, add 50 ml of de-ionised water, and heat with stirring to 80°C. After 10 minutes at 80°C add 20 ml of alumina cream, mix well and transfer to a 100 ml calibrated flask. Cool, make up to the mark with de-ionised water and filter through a 15 cm No. 4 filter paper which has been previously washed with 100 ml of hot water. Reject the first 10 ml of filtrate.

NITRITE

Pipette 10 ml of the filtrate into a 50 ml graduated flask, add 2 ml of the arsanilic acid reagent and mix. After 5 minutes add 2 ml of naphthyl ethylene diamine reagent, mix and stand 10 minutes. If clear make up to 50 ml with de-ionised water and read the absorption in a 1 cm cell at 538 mμ. If the solution is cloudy transfer to a 100 ml separator, saturate with salt and extract the colour with 20 ml, 15 ml and 5 ml of *n*-butanol. Run the butanol extracts through a cotton wool plug into a dry 50 ml calibrated flask and make up to the mark with *n*-butanol. Read the optical density in a 1 cm cell at 545 mμ.

NITRATE

Pipette 10 ml of filtrate into a 60 ml stoppered bottle, add 5 ml of buffer solution and 1 g of the wet cadmium. Stopper the bottle and shake for 5 minutes. Filter through a washed 7 cm No. 4 filter paper into a 50 ml calibrated flask rinsing the cadmium and filter with 5 ml of de-ionised water. Proceed as under the determination of nitrite from "add 2 ml of the arsanilic acid . . . ".

From the figure obtained deduct that obtained in the determination of nitrite.

PREPARATION OF STANDARD GRAPHS

Prepare graphs with a freshly-made standard solution of sodium nitrite, using aliquots containing 10 μg, 20 μg, 30 μg, 40 μg, 50 μg, 60 μg, 70 μg and 80 μg of sodium nitrite, following the procedure detailed under "NITRITE" from "add 2 ml of the arsanilic acid reagent. . .". It is necessary to prepare separate graphs using water and *n*-butanol.

It was found that consistent readings were obtained for the standard nitrite solutions and, therefore, reliance could be placed on a standard graph, thus doing away with the necessity of preparing standards every time the method was used. Similarly, each batch of reagents gave a reproducible blank.

References

1. Follett, M. J., and Radcliff, P. W., *J. Sci. Food Agric.*, 1973, **14**, 138.
2. Bunton, N. G., Crosby, N. T., and Patterson, S. J., *Analyst*, 1969, **94**, 585.
3. Elliott, R. J., and Porter, A. G., *Analyst* 1971, **96**, 522.

Appendix

RESULTS OF ANALYSIS FOR NITRATE AND NITRITE OF VARIOUS SAMPLES OF FOOD

Baby Foods

46 samples were examined, all of which were devoid of nitrite. 22 samples contained nitrate as follows:

	Sodium nitrate, p.p.m.
Cheese Savoury (strained)	12
Cheese and Tomato savoury (dry)	80
Cauliflower Cheese (dry)	120
Vegetable and Lamb (strained)	8
Bacon and Vegetable (strained)	50
Ham and Vegetable	25
Roast Turkey and Vegetable with Bacon	15
Country Lamb and Carrot Purée	50
Lamb Casserole with Vegetable	30
Orange Cereal Breakfast	10
Apple and Bilberry Dessert	20
Chicken and Ham Dinner	15
Tomato and Beef Supper	5
Cheese, Ham and Egg	5
Vegetable and Cereal and Bacon	20
Banana Dessert	5
Chicken Casserole with Vegetables	25
Braised Lamb and Liver Dinner	10
Chicken and Ham Dinner	10
Vegetable and Rice Baby Soup (dry) (to be diluted 15 times for a 6 month-old child)	330
Bone and Vegetable Instant Baby Food (to be diluted 4 times)	275
Vegetables and Chicken Casserole (dry) (to be diluted 4 times)	340

Vegetables

12 samples, all devoid of nitrite:

	Sodium nitrate, p.p.m.
Frozen Chopped Spinach	960
Frozen Peas	15
Frozen Peas	Not detected
Canned Green Beans	130
Canned Green Beans	110
Canned Green Beans	120
Canned Carrots	130
Canned Carrots	90
Canned Peas and Carrots	50
Canned mixed Vegetables (Carrots, Peas, Potatoes and Swedes)	86
Canned mixed Vegetables (Carrots, Peas, Potatoes and Swedes)	80
Canned Broad Beans	Not detected

Meats

	Sodium nitrite, p.p.m.	Sodium nitrate, p.p.m.
Sausages		
Pork (16 samples)	Not detected	0–25
Average	—	5
Beef (3 samples)	Not detected	0–25
Saveloys (1 sample)	75	50
Salami (1 sample)	5	35
Tyrolerwurst (1 sample)	10	25
Luncheon (1 sample)	50	50
Liver (1 sample)	15	75
Meat pastes		
10 samples	Not detected	10–40
Average	—	22
Corned beef		
23 samples	0–5	0–30
Average	—	11
Pre-packed bacon		
5 samples	5–50	5–250
Average	21	100
Canned meats (21 samples)		
Ham cured shoulder	50	215
Ham (Holland)	5	300
Ham (Czech.)	220	Not detected
Remaining samples	0–80	0–130
Average	13	37

J. Assoc. Publ. Analysts, 1976, **14**, 27–28

Review

CHEMICALS IN FOOD AND ENVIRONMENT. SCIENTIFIC EDITOR, M. WEBB. *British Medical Bulletin*, **31** (3), September 1975. Pp. 181–268 + x. London: Medical Department, The British Council. Price £3.

Fifteen papers, together with an introduction by Professor Neuberger, provide the reader with brief reviews and assessments, by acknowledged experts, of the current concern with the composition of food and the state of the environment.

The authors write for medically qualified readers and some chemically qualified readers may find the articles inadequate in depth. However, as a broad picture of naturally toxic as well as contaminated foods and environments, this number of the *British Medical Bulletin* should be on the desks of Public Analysts if not on their book shelves. Many candidates for the Mastership in Chemical Analysis would wish to read this and other *Bulletins*.

Glancing through the index of titles of parts of volumes still in print one finds Causation of Cancer (Vol. 14, No. 2), Antibiotics in Medicine (Vol. 16, No. 1), Electron Microscopy (Vol. 18, No. 3), Mechanisms of Carcinogenesis: Chemical, Physical and Viral (Vol. 20, No. 2), The Separation of Biological Material (Vol. 22, No. 2), Mechanisms of Toxicity (Vol. 25, No. 3), and Drugs: Development and Use (Vol. 26, No. 3), all of which titles suggest the presence of material of some interest to a Public Analyst and, if of the standard of this number, well worth reading.

In the *Bulletin* under review, Sir Edward Pochin considers the delicate balance between acceptable and unacceptable risk and records details of fatal accident rates associated with occupation together with the risk of disability incurred, and also non-occupational risks.

Dr Goulding writes of "Chemical Hazards in the Home" including gaseous or vapour poisons used as refrigerants, propellants, cleaning fluids and fuels as well as caustic and corrosive cleaning materials, toxic garden chemicals and toxic plants, residual medicaments and "borrowed" chemicals. Despite a sixfold increase between 1957 and 1971 in hospital admissions caused by poisoning, he concludes that toxicologically the danger in the home has probably received disproportionate emotively-presented attention and publicity.

Dr J. M. Barnes writes on "Assessment of Hazards from Low Doses of Toxic Substances". In his conclusions he draws attention to the difficulty of determining the amount of scientific effort to be invested in discovering the effects of compounds which may induce changes about which there may be doubt that they represent a material disadvantage to exposed individuals.

Dr H. Egan and Mr A. W. Hubbard describe the "Analytical Surveys of Food" undertaken for statutory and other bodies both on specified foods and on total diet studies. They discuss the strategy of food contamination surveys and

present details of contaminants including organo-chlorine pesticides. Work carried out in assessing the proportions of non-essential and essential heavy metals also is described together with investigations of mycotoxins, nitrosamines and polynuclear hydrocarbons.

Dr R. F. Crampton and Mrs Frances Charlesworth write of the "Occurrence of Natural Toxins in Food" including natural carcinogens and oxalates.

Dr A. G. Lloyd and Mr J. J. P. Drake discuss the "Problems Posed by Essential Food Preservatives" including permitted and non-permitted preservatives.

Professor Spicer briefly introduces the subject of the "Toxicological Assessment of New Foods".

Mr P. K. C. Austwick provides a substantial paper on "Mycotoxins", which deals with their natures, production in the environment, classification and effects as well as detection and control.

Professor Higgins surveys the "Importance of Epidemiological Studies Relating to Hazards of Food and Environment" and concludes that better information on exposure to potentially hazardous chemicals in the environment is needed.

Professor Barbara Clayton reviews "Lead: the Relationship of Environment and Experimental Work" and relates the intake of lead and the effect on enzymes. The relationship between concentration of lead and mental development is considered, as also are some effects of lead on the foetus. After mentioning the incidence of lead in soil, water and air, Professor Clayton concludes that there is much awareness in the United Kingdom of the dangers of contamination of the environment with lead but that biochemical assays alone cannot show whether or not mild continuous exposure results in important clinical change.

Dr Magos writes about "Mercury and Mercurials", describing concentration processes in ecosystems, and the forms of combination in which mercury has biological significance. He describes tolerable and toxic daily intake.

Dr Webb reviews "Cadmium, its Sources and Incidence in Living Organisms and Man". He describes the sources of ingestion by man, the uptake and excretion, the interaction with essential metallic ions and the attendant hazards. Perhaps it is disappointing that there is not any mention by Dr Webb, when describing the concentration of cadmium found in the brown meat of the edible crab, of the paper in the *Journal of the Association of Public Analysts*, December 1971.

Dr Martin writes about "Water Supplies of the Future and the Recyling of Drinking Water" and considers the attendant risks and disadvantages of such ventures.

Finally Professor Lawther reviews "Carbon Monoxide", its sources, distribution and fate, the absorption and excretion by man, and the effects in general on the central nervous system and on the cardiovascular system.

The papers are provided with extensive reference lists, but mainly of journals which may not readily be available to Public Analysts. It is gratifying to note that Dr Egan and Mr Hubbard mention this Association and refer to the Pesticides Residues Surveys. But it does not do any credit to this Association that in the other papers about topics of real interest to Public Analysts, there is not any reference to their work or publications.

E. BRAXTON REYNOLDS

J. Assoc. Publ. Analysts, 1976, **14**, 29–30

Letter to the Editor

Catalyst in the Kjeldahl Determination of Nitrogen

Sir,

Reading the paper by Stirrup and Hartley on the use of titanium dioxide and copper sulphate as a catalytic mixture in the determination of nitrogen in feeding stuffs[1] has led me to realise that analysts, who for unofficial purposes still use a copper sulphate catalyst and 2 hours digestion after clearing and are not members of the A.P.A., may like to know of my work published in 1966 in the Bulletin of that Association for the private information of members and their staffs[2]. This work, actually carried out in Leeds in 1956, was undertaken after the discarding, by the Fertilisers and Feeding Stuffs Regulations, 1955, of the alternative copper sulphate (previously permitted by the 1932 Regulations). It was an extension of the work of Alcock on flour, soya flour, milk powder and desiccated yeast, copper sulphate being the only catalyst used, as he had found some evidence of nitrogen loss in very protracted (e.g. 12-hour) digestions with mercury[3]. The products studied by myself consisted of both feeding stuffs and fertilisers, my concern being to compare the results obtainable from both copper and mercury under the most favourable conditions. Having previously found with wholemeal flour that 3 hours' digestion after clearing gave no higher result than 2 hours, I examined several samples using both 1 hour's and 2 hours' digestion with both copper and mercury catalysts. Wood[4] examined my data and pointed out that, with animal feeding stuffs and 1 hour digestion period, copper gave a significantly lower result than mercury, the average difference being 0·1 per cent. N (= 0·63 per cent. of protein). For this reason Table I gives only the results obtained with 2 hours' digestion after clearing. Samples of about 1·5 g were weighed and treated with 25 ml of conc. sulphuric acid, 10 g of potassium sulphate and either 0·5 g of copper sulphate pentahydrate or 0·4 g of yellow mercuric oxide. All-glass apparatus was used throughout and methyl red indicator. Tryptophane and histidine hydrochloride were also examined for recovery from ring-nitrogen compounds.

It will be seen that with these two latter substances results less than theoretical were obtained with both catalysts and that mercury gave a better recovery than copper. The proportion of ring-nitrogen amino-acids in the protein molecule is usually small and the effect of using a copper catalyst in the analysis of ordinary feeding stuffs and fertilisers is thus likely also to be small. This is borne out by the results on the 11 other samples, which taken as a whole show no significant advantage in the use of mercury.

If a copper catalyst could be regarded as of equal value to its mercury counterpart, then it would have the advantage over its rival of cheapness, non-volatility and relative non-toxicity.

TABLE I

COMPARISON OF THE NITROGEN CONTENT OF SOME FERTILISERS AND FEEDING STUFFS, USING COPPER AND MERCURY CATALYSTS

	Nitrogen, per cent.	
	Catalyst	
	Cu	Hg
1. Wholemeal flour	2·13	2·11
2. Cooked flaked maize	1·31	1·29
3. Sow & weaner meal	1·87	1·86
4. Intensive layer pellets	2·88	2·92
5. White fish meal	10·45	10·32
6. Flower fertiliser	3·81	3·75
7. Bone meal	5·49	5·56
8. Fish & bone meal	6·25	6·29
9. Meat & bone meal	6·30	6·40
10. Hoof & horn meal	13·83	13·84
11. Dried blood	14·44	14·40
Acetanilide (control)*	10·41 (theoretical	10·51 10·36)
Tryptophane	12·88 (theoretical	13·30 13·70)
Histidine hydrochloride	17·95 (theoretical	18·30 18·42)

* 1 hour's digestion only after clearing.

References

1. Stirrup, J. E., and Hartley, A. W., *J. Assoc. Publ. Analysts*, 1975, **13**, 72.
2. Manley, C. H., *Assoc. Publ. Analysts Monthly Bulletin*, 1966, **13**, 64 (restricted circulation).
3. Alcock, R. S., *Analyst*, 1946, **71**, 233.
4. Wood, E. C., *Assoc. Publ. Analysts Monthly Bulletin*, 1966, **13**, 114 (restricted circulation).

C. H. MANLEY
Bristol