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**Some Applications of Thin Layer Chromatography in the
Public Analyst's Laboratory**

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Some applications of thin layer chromatography are illustrated by reference to the determination of aflatoxin, alkaloids, sugars, tocopherols, *p*-phenylene diamine and pesticides using established methods.

The usefulness of thin layer chromatography (TLC) to the food analyst has already been illustrated in this Journal by accounts of its application to the determination of food additives such as water-soluble colouring matters¹, *p*-hydroxy-benzoic acid and its esters², cyclamates, saccharin, benzoic acid, sorbic acid and antioxidants³. The following are examples of problems encountered in the author's laboratory and which have been solved by the use of TLC. The general apparatus and reagents and thin layer preparations which have been used are those already described in this Journal³.

A. Aflatoxin

Aflatoxin is the generic name given to highly toxic metabolites of *Aspergillus flavus* which can be present on groundnut shells. The metabolites, comprising mainly aflatoxins B1 and G1, can find their way into the kernels which may be used either in animal feeding stuffs or in peanut butter or sold for direct human consumption.

EXTRACTION AND CHROMATOGRAPHY

Use the procedures of Coomes *et al.*⁴

DISCUSSION

The extraction comprises grinding the kernels, defatting the meal with light petroleum, extraction of the aflatoxin by the Soxhlet process, using methanol and further cold separation into chloroform. The chromatogram of the concentrated extract is prepared using 5 per cent. methanol in chloroform as the mobile solvent. The aflatoxins are shown as fluorescent spots when exposed to u.v. light of wave-length 365 m μ . Aflatoxin B1 (usually the major component) shows as a purple-blue fluorescence with R_f value of 0.45-0.50 and

aflatoxin G1 as a green fluorescence with R_f value 0.35–0.40. Limits of detection are given by Coomes⁴ as 0.0004 μg of aflatoxin B1 and 0.0003 μg of aflatoxin G1.

A quicker method of extraction, which dispenses with the methanol extraction step, has been given by Lee⁵ and a comparison of assay procedures has been made by Trager *et al.*⁶

A sample of groundnuts tested by the methods described contained 0.6 p.p.m. of aflatoxin B1 and 0.2 p.p.m. of aflatoxin G1. These amounts were considered by the Tropical Products Institute to be excessive and the consignment was condemned. It is possible that groundnuts containing aflatoxins and condemned for human consumption are used in animal feeding stuffs, and for this reason such samples have been examined periodically, but so far without positive identification. Groundnuts sold for human consumption and for feeding animals at the Bristol Zoo have given no indication of the presence of aflatoxin.

The limits proposed by the Tropical Products Institute⁷ for aflatoxin B1 and G1 are given in the following ranges:

Parts per million

Below 0.05	Low
0.05–0.25	Medium
0.25–1.0	High

B. Alkaloids

Thin layer chromatography of alkaloids can be particularly useful either where a qualitative examination is required or where there are two or more alkaloids present.

REAGENT

Spray Reagent. Mix 3 ml of 10 per cent. aqueous platinum chloride solution with 97 ml of water and add 100 ml of 6 per cent. aqueous potassium iodide solution. Keep the resulting iodoplatinate solution in an amber glass bottle.

EXTRACTION AND CHROMATOGRAPHY

Extract the alkaloids in the conventional manner and use the chromatographic systems of Waldi *et al.*⁸ on layers of Kieselgel G and apply the iodoplatinate solution as the chromogenic agent. This reagent is far more selective than others, commonly used, such as Dragendorff's.

DISCUSSION

Alkaloids are retained on layers of Kieselgel G, more or less strongly according to their basicities and dissociation constants. Consequently, a base

is added to the mobile solvent to decrease this retention and give a wider spread for the various alkaloids which may be encountered.

In order to cover the more common alkaloids of interest in Public Analysts' work, two solvent mixtures have been chosen from the various Waldi systems. These are:—

Solvent A. Chloroform: diethylamine (9:1)

Solvent B. Benzene: ethyl acetate: diethylamine (7:2:1)

The R_f values obtained using these solvents, together with the colours obtained after iodoplatinate spraying, are listed in Table I. Some of these differ from the literature where the colour for morphine is given as deep blue, that of strychnine as yellow and that of physostigmine as pink. Moreover, the R_f values quoted for solvent B for atropine, cocaine and narcotine^s are considerably less than those in Table I. Examination of strychnine, narcotine and quinine under ultra-violet light of wavelength 365 $m\mu$ reveals spots with green, blue-green and blue fluorescence respectively. All alkaloids are dissolved in methanol except quinine which is used as the sulphate and is dissolved in a mixture of 3 parts of ethanol to 7 of chloroform. Ideally, about 50 μg of extract are used for the chromatogram for which a 10-cm development takes 15 minutes.

TABLE I
THIN LAYER CHROMATOGRAPHY OF CERTAIN ALKALOIDS

<i>Alkaloid</i>	R_f value <i>solvent A</i>	R_f value <i>solvent B</i>	<i>Colour of spot</i>
Morphine	0.09	0.06	White
Quinine	0.26	0.21	Yellow
Cinchonine	0.43	0.30	Violet
Atropine	0.45	0.23	Violet
Homatropine	0.45	0.27	Violet
Codeine	0.55	0.29	Violet
Pilocarpine	0.57	0.15	Light brown
Hyoscyne	0.61	0.34	Violet
Brucine	0.64	0.24	Blue-brown
Strychnine	0.68	0.35	Violet
Cocaine	0.86	0.75	Violet
Lobeline	0.87	0.57; 0.64	Red-brown
Narcotine	0.89	0.69	Light yellow
Physostigmine	0.91	0.48	White

A stomach powder which was labelled as containing Pulv. Opii. B.P. 0.6 per cent., was examined for opium alkaloids by chromatographing a concentrated methanolic extract. Morphine was found at a concentration of approximately 0.05 per cent. by comparison with standards. Traces of narcotine and codeine were also found together with a spot which corresponded with thebaine (R_f value; 0.52, using solvent B: colour after spraying with iodoplatinate solution—red/brown). However, no standard was available for comparison. The presence of narcotine was confirmed by its fluorescence in u.v. light.

A quick overall method such as this is extremely useful in confirming the absence of alkaloids. Thus a sample of grain, eaten by a dog that had subsequently been violently ill, was found to be free of alkaloids. (The sample was later found to contain a high concentration of salt).

C. *Sugars*

The clean chromatographic separation of maltose and lactose has always been difficult, and with this problem in mind, the use of TLC for these sugars, together with sucrose, glucose and fructose, has been investigated.

REAGENTS

1. *Spray Reagents* (a) Add 1 ml of saturated aqueous silver nitrate to 20 ml of acetone, with stirring. Add water dropwise until the solid just dissolves. After development and spraying, place the plates in saturated ammonia vapour for 15 minutes, then heat at 90° C for 15 minutes. Spots show as dark areas on a light ground.

(b) Dissolve 5 grammes of phosphomolybdic acid in 95 ml of ethanol. After development and spraying, heat the plates at 90° C for 10 minutes. Spots show blue-green on a yellow ground.

(c) Add an equal volume of a 0.2 per cent. ethanolic solution of 1:3-dihydroxynaphthalene to 20 per cent. aqueous trichloroacetic acid⁹. After development and spraying, heat the plates at 90° C for 15 minutes. Spots show blue-violet on a light ground.

(d) Add 1 ml of conc. H₂SO₄ to 5 ml of anisaldehyde dissolved in 50 ml of glacial acetic acid¹⁰. After development and spraying, heat the plates at 100° C for 10 minutes. Spots show blue-violet on a pink ground.

2. (a) Prepare layers of Kieselgel G in the usual manner using 0.1 N boric acid solution in place of water⁹.

(b) Prepare layers of Kieselguhr G in the usual manner using 0.02 M sodium acetate solution in place of water¹⁰.

3. *Mobile solvents* A: Acetone : *n*-butanol : water (5 : 4 : 1)⁹

B: Ethyl acetate : *isopropanol* : water (6 : 2 : 1)¹⁰

EXTRACTION AND CHROMATOGRAPHY

Extract the sugars by established methods and use the chromatographic procedure of Pastuska⁹ for Kieselgel G layers and Stahl and Kaltenbach¹⁰ for Kieselguhr G layers.

DISCUSSION

Because of the marked hydrophilic nature of sugars, Kieselguhr G would appear to be the best medium for their chromatography since adsorption

effects cannot be precluded from chromatography on Kieselgel G. Although the Stahl and Kaltenbach procedure using Kieselguhr layers impregnated with sodium acetate and solvent B gives reasonable separation of mono-, di- and tri-saccharides, the sugars in each group are bunched together. This method was tried in order to use the anisaldehyde reagent (*d*), which fails on layers impregnated with boric acid. This spray reagent gives differently coloured spots for various sugars at the 1 μg level but it has been found that at levels of the order of 5–50 μg the colours of the five sugars examined were the same, *viz.*, blue-violet.

Inactivation of Kieselgel G is achieved by the incorporation of water in mobile solvent A and chromatography on layers impregnated with boric acid then gives good separation of lactose and maltose. A better separation of maltose and sucrose can be achieved on Alusil layers (Kieselgel G and Aluminium oxide G mixed in equal proportion) using a solvent comprising *n*-propanol: water: ethyl acetate: ammonia solution (sp. gr. = 0.880), (24:12:4:1) according to Waldi¹¹. (R_f value for maltose = 0.30 and R_f value for sucrose = 0.39).

The silver nitrate spray reagent (*a*), the phosphomolybdic acid spray reagent (*b*) and the 1:3-dihydroxynaphthalene spray reagent (*c*) were examined in conjunction with the Kieselgel G chromatography and limits of detection are given in Table II together with R_f values. The time required for a run of 10 cm is about 40 minutes. Spots of aqueous solutions of sugars are applied.

This method provides a good basis from which to proceed should analytical problems arise concerning trace amounts of other sugars.

TABLE II
THIN LAYER CHROMATOGRAPHY OF SOME SUGARS

Sugar	R_f value with solvent A on Kieselgel G impregnated with boric acid	Limits of detection (μg) for various spray reagents		
		a	b	c
Fructose	0.19	2	5	10
Lactose	0.34	5	20	40
Glucose	0.47	5	10	10
Maltose	0.53	5	20	40
Sucrose	0.56	5	10	10

Considerable difficulty has arisen in the past in proving the presence of milk in a "milk loaf". Attempts have been made to detect lactose by paper chromatography but problems have arisen from separating the small amount of lactose present in the bread from the greater quantity of maltose which has been co-extracted. The sugars are extracted by established methods and TLC carried out using the silver nitrate spray reagent (if the greatest sensitivity is required) or the 1:3-dihydroxynaphthalene reagent for the greatest specificity. By applying various dilutions of the sample extract and a number of standard dilutions, it is possible to assess the quantity of lactose in a "milk loaf" to within 5 to 10 per cent.

D. *Tocopherols (Vitamin E)*

The application of TLC to vitamins is very widespread. The determination of vitamin E is complicated by the presence of extraneous material which may be readily removed by the following procedures.

EXTRACTION

Saponify vegetable oils in the presence of pyrogallol to prevent oxidation. Extract the unsaponifiable matter with diethyl ether and evaporate carefully to dryness. Dissolve the residue in a small volume of benzene.

CHROMATOGRAPHY

Prepare a chromatogram of the benzene extract on Kieselgel G using benzene as solvent, and spray with 0.2 per cent. aqueous $\alpha\alpha'$ -dipyridyl followed by 0.2 per cent. aqueous solution of ferric chloride hexahydrate¹². For quantitative measurement, remove the silica containing the tocopherols from the chromatographic plate (by using pre-knowledge of the R_f values of standards), carry out the $\alpha\alpha'$ -dipyridyl-ferric chloride reaction, and measure the red colour spectrophotometrically¹³.

DISCUSSION

Three of the more common tocopherols have been studied and the following R_f values were obtained:—

α -tocopherol	0.52
α -tocopheryl acetate	0.63
β -tocopherol	0.40

The development time for a 10-cm run is about 20 minutes.

This system has been successfully applied to wheat germ oil for the quantitative detection of α and β tocopherols. However, γ -tocopherol would not be distinguished from the beta homologue by this procedure.

E. *p-Phenylene diamine*

The necessity for using TLC for the detection of *p*-phenylene diamine has arisen from the possibility of its incorporation in hair dyeing and rinsing preparations. It has been used in the past because, when oxidised to the corresponding quinone, it makes a useful brown hair dye. It has since been discovered to cause dermatitis.

EXTRACTION

Use liquid preparations direct and make ethanolic extracts of solid preparations.

CHROMATOGRAPHY

Prepare a chromatogram on Kieselgel G using a mixture of ethanol:water: ammonia solution (sp. gr. = 0.880), (16:3:1) as the mobile solvent¹⁴.

DISCUSSION

Under these conditions *p*-phenylene diamine has an R_f value of 0.80. When developed, the spot is violet but oxidises to brown on standing. A convenient amount of the substance for this test is 20 to 50 μ g.

Seven hair rinses were examined and *p*-phenylene diamine was proved to be absent. A number of assorted colouring matters were incidentally encountered.

F. Pesticides

The TLC methods used in this laboratory and which have proved satisfactory for organochlorine insecticides¹⁵ and herbicides¹⁶, organophosphorus insecticides¹⁵ and triazine herbicides¹⁷, may be referred to.

In work on pesticides, TLC has proved to be an invaluable tool in the clean-up of samples for gas-liquid chromatography (GLC). In the analysis of the more common organochlorine compounds, extracts in suitable solvents are transferred to layers of Kieselgel G and a chromatogram is prepared using *n*-hexane as mobile solvent (fraction from petroleum; boiling range 67–70° C). *pp'*- and *op'*-DDT and *pp'*-DDE may be removed from the plate and redissolved in *n*-hexane to give clean GLC chromatograms. If γ -BHC is required for GLC examination, it is better to use 10 per cent. acetone in *n*-hexane so that the γ -BHC spot is taken a reasonable distance from the origin and thence away from interfering substances.

This technique also provides strong confirmatory evidence in organochlorine pesticide analysis and this gives the necessary support to any GLC result.

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An Account of Results of a Radio-activity Monitoring Scheme in Monmouthshire

by G. V. JAMES

An account of a scheme for monitoring radio-activity in the County of Monmouthshire to check on pollution of the atmosphere, water, food-stuffs, etc., is given.

The Windscale incident occurred on 11th October, 1957 and the Public Analyst who regularly monitored smoke filters in Leicester obtained the results shown in Table I¹, where the effect of emission of the radio-activity can be seen in a marked degree.

TABLE I
RADIOACTIVE DEPOSITION DUE TO THE WINDSCALE INCIDENT

<i>Date (1957)</i>	<i>Counts per minute</i>
October 10-11	1
11-12	289
12-14	89
14-15	10
15-16	4
16-17	2
17-18	0

The decision to operate a monitoring scheme in Monmouthshire was prompted by the presence of three existing or proposed nuclear generating stations on the Bristol Channel.

The County of Monmouth is approximately rectangular in shape with high ground, rising to 2,600 feet in the north and west, sloping to the east and south. The east is bounded by the River Wye, the south by the northern shores of the Bristol Channel, whilst down the centre runs the River Usk and its tributaries. The prevailing winds are SW but during the spring months may be easterly, whilst the average rainfall is approximately 40 inches per annum on the low ground increasing to 70 to 80 inches per annum on the high ground. This brief description is necessary to appreciate the sampling pattern about to be described.

Volumetric sulphur dioxide-smoke instruments were set up at seven different places and filters were submitted for examination in a regular pattern so that each day samples were received from places on high and low ground as well as from the central valley of the Usk.

Normal air filters were replaced with GFL Whatman paper² as this paper absorbs more radio-activity than others.

The most important source of widespread artificial activity is from the "fall-out" following the explosion of nuclear devices. Radio-activity is released

and travels round the world at great heights before falling to earth, or being washed out by rain. The greatest proportion of fission products formed in such an incident emit beta particles which are simple to measure and can give an earlier indication of trends than if a laborious estimation of individual isotopes such as strontium 90 had to be performed.

The samples of filters were despatched so that they reached the laboratory on the day following that on which they were taken off the instrument. This passage of time allowed much naturally occurring radio-activity to decay, hence a high count became indicative of contamination. The contaminated circle on the filter paper was cut out, placed in a platinum planchet and counted. After the time for a pre-set count had been noted, the paper was ashed in a muffle furnace and re-counted. This latter result was usually a shade higher than the first result due to reduced absorption by organic matter and indicated the absence of iodine 131 (which is lost on ashing). Corrections were of course made for background count determined before and after the particular experiment.

TABLE II
COMPARISON OF QUARTERLY MEASUREMENTS OF RADIO-ACTIVE AND SMOKE POLLUTION OF THE ATMOSPHERE

	Station 1		Station 2		Station 3	
	Radio	Smoke	Radio	Smoke	Radio	Smoke
1961 2nd Qtr 2.20 34	1.20	32	1.10	22
3rd " 1.20 —	0.84	30	0.87	15
4th " 1.38 —	0.56	79	1.39	62
1962 1st " 0.83 53	0.83	82	0.80	51
2nd " 0.61 50	0.60	48	0.51	21
3rd " 0.50 51	0.60	38	0.65	19
4th " 1.07 138	1.05	138	1.24	72
1963 1st " 1.20 —	1.17	149	1.21	38
2nd " 0.66 45	0.92	63	1.04	23
3rd " 0.54 47	0.45	42	0.65	22
4th " 0.81 93	0.66	58	0.91	23
1964 1st " 0.37 102	0.62	111	0.62	41
2nd " 0.40 40	0.55	31	0.80	12
3rd " 0.20 35	0.21	25	0.22	12
4th " 0.20 94	0.34	133	0.36	37
1965 1st " 0.34 —	0.37	—	0.37	—
2nd " 0.20 —	0.21	—	0.46	—
3rd " 0.40 —	0.15	—	0.25	—

Radio-activity Total Counts per Minute per 60 cu. ft. of Air.
Smoke Densitometer readings.

Table II shows the results of examination of air filters for three stations over the last five years, averaged to give quarterly figures and compared with smoke records, where available, for the same places. The radio-activity figures are expressed as counts per minute per 60 cubic feet of air, whilst the smoke figures are expressed as micrograms per cubic metre. Results were obtained before March 1961 but the scheme only commenced operating as a whole at that time.

Consideration of this table shows that the general level of radio-activity in the air is falling at the places studied. An increase at the end of 1961 may be due to the explosion of Russian bombs in late October.

During this period daily results of radio-activity were obtained at one station by exposure of Petri dishes coated with Vaseline. After 24 hours, the dishes were collected, replaced with fresh dishes, and the exposed dishes wiped clean with cotton wool and finally rinsed with light petroleum (40°C – 60°C) which was added to the wool in a platinum planchet, evaporated, ashed and counted. Figure 1 shows the results obtained; the peaks on 1st/2nd November correspond with the explosion of a 30 Megaton bomb, whilst the peak on 7th/8th November corresponds with the explosion of the 50 Megaton bomb. During this period the wind was mainly from the W and SW but on 11th/12th November

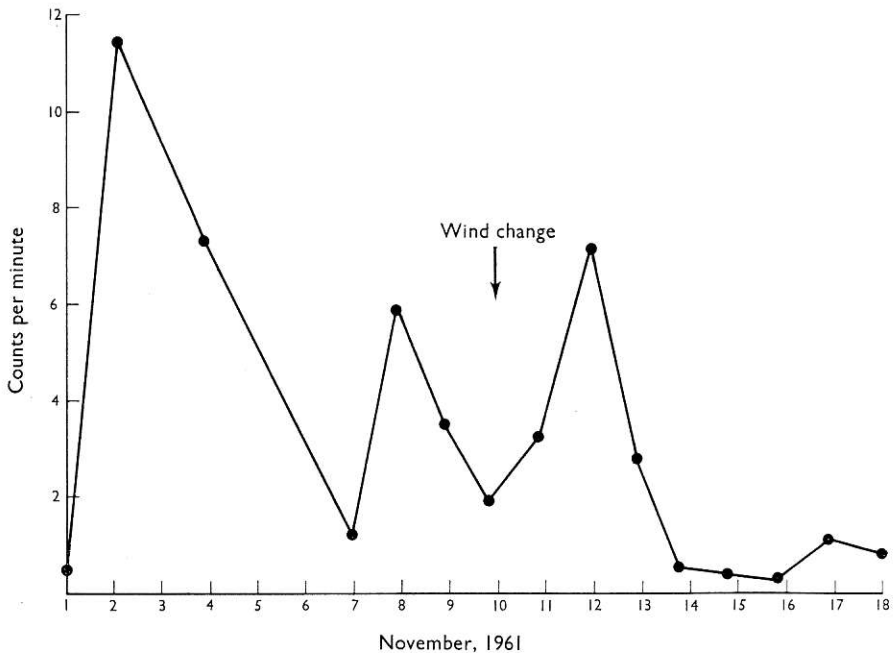


Fig. 1. Deposition of Radioactive Dusts during November, 1961.

it changed to an easterly direction and the radio-active cloud returned and resulted in another peak. Figure II shows the results obtained at the same station using the GFL filters following the explosion of the Chinese bomb on 16th October, 1963 and these results are compared with the smoke results on the same paper. The first peak on 26th October occurred during a spell of easterly winds which changed to W and SW on 12th November when a further peak was obtained.

Concurrently with the monitoring of the air filters, rain water was collected and bulked so that quarterly samples could be examined. Collections were

made in polythene bottles and an aliquot of the water was evaporated to small bulk, gradually transferred to a planchet and evaporated to dryness. Although little potassium 40 is normally present in rain, as many of the sites are affected by industrial pollution it has been customary to correct for this isotope by measuring the potassium with a flame photometer.

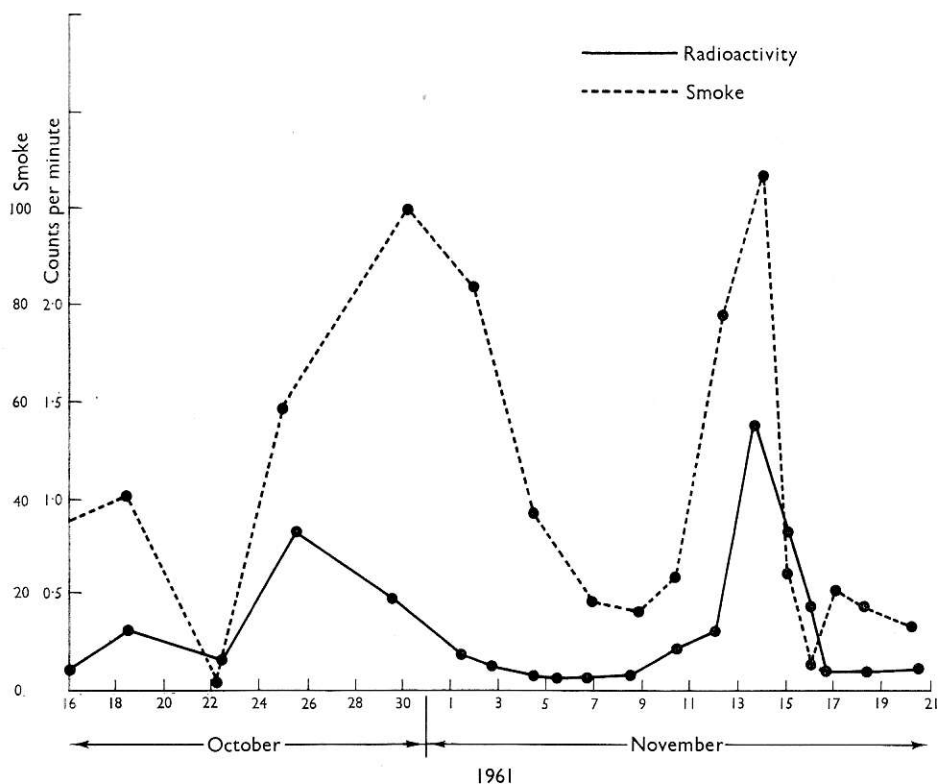


Fig. 2. Deposition of Radioactive Dust, and Smoke measurements: October–November, 1961.

The background count was determined before and after the sample was counted, and the average background count obtained. This was deducted from the count due to the water solids, and the result corrected in turn for potassium 40. The method was similar to that of Clark³. Table III shows the results of analyses of rain water taken in the same period as that for Table II and from the same stations.

As it is necessary to calibrate the instrument, strontium 90 was used and the results are expressed as pico curies of strontium 90 per litre. It must be stressed that this is merely a useful method of expression and does not signify that the whole of the beta-activity is in fact due to strontium 90.

TABLE III
RADIOACTIVE CONTAMINATION OF RAINWATER

(Total β -Activity from all Sources)

		Station 1	Station 2	Station 3
1962	1st Qtr.	170.0	202.7	157.1
	2nd "	133.3	133.9	140.9
	3rd "	115.6	91.4	104.9
	4th "	88.2	114.0	127.2
1963	1st "	173.5	145.2	344.0
	2nd "	20.0	23.1	20.3
	3rd "	18.6	—	16.7
	4th "	23.9	40.8	44.5
1964	1st "	41.30	66.85	72.50
	2nd "	11.60	33.10	48.00
	3rd "	36.70	13.11	26.48
	4th "	8.17	13.76	16.34
1965	1st "	—	—	—
	2nd "	21.03	21.24	20.68
	3rd "	7.08	6.96	8.85

The results confirm the observation that the general level of radio activity is tending to fall at the places studied. The higher results noted during the severe winter of 1962/63 are thought to be due to the very heavy snowfalls which probably carried down more radio activity.

Drinking water and sewage were also collected over quarterly periods and examined by the same method used for rain water. As drinking water is supplied by various Water Authorities only two series of results are quoted since the areas of these authorities are most nearly congruent with the boundaries of the stations where rain water is collected and air is sampled. Unlike results quoted in the Metropolitan Water Board reports⁴ it is not possible to give individual sources for comparison with the air and rain as the supplies are bulked after being derived from various sources, some sources being upland surface, some springs and some deep sources. The sewage sample is collected before treatment at the works and the area of the sewage board quoted includes Station 2 and 3 but not Station 1 and covers a large industrial area, which may in part, account for the radio-activity of the sewage being greater than that of the drinking water. Also it must be remembered that in the human diet only a small part of the intake of radio-active substances is due to water but the excreta contain residues from milk, cereals and other sources.

The results in Table IV are for the total beta-activity expressed as picocuries Sr 90 per litre and again it must be emphasized that the true Sr 90 is only a fraction of this. It is better to watch the total activity of the supply, and sample regularly in order to achieve this and to make a specific examination if the level rises, than to make a sporadic examination for specific isotopes which may be very time-consuming and possibly of less value.

The above results are in the main similar to the results obtained by the Metropolitan Water Board⁴.

The results show that radio-activity in air, rain and drinking water is declining in quantity, that radio-activity is deposited to a lesser extent in dry weather, and to some extent runs parallel with the smoke present in the atmosphere.

Apart from the work of the Agricultural Research Council at Wantage, which is done on a national basis, similar work has been carried out by Hawthorne *et al.*⁵ in Scotland. It is intended to continue the present series of reports with a description of further work on foodstuffs.

TABLE IV
RADIO CONTAMINATION OF WATER AND SEWAGE

				(Total β -activity from all Sources)		
				Drinking Water		Sewage
				Water Board	Water Board	
				No. 1	No. 2	
1961	4th Qtr.	—	10.15	20.41
1962	1st	"	..	—	5.01	12.27
	2nd	"	..	5.54	6.08	10.81
	3rd	"	..	10.75	4.70	12.18
	4th	"	..	12.88	5.18	12.90
1963	1st	"	..	11.02	5.55	8.75
	2nd	"	..	9.94	8.27	9.77
	3rd	"	..	6.04	4.93	12.66
	4th	"	..	11.9	5.02	18.05
1964	1st	"	..	20.3	2.00	—
	2nd	"	..	2.47	2.00	8.16
	3rd	"	..	2.07	1.22	8.80
	4th	"	..	4.52	5.20	20.22
1965	1st	"	..	1.33	4.63	10.79
	2nd	"	..	3.05	3.35	8.60
	3rd	"	..	6.18	—	6.56

The question of what standards to apply to these results is a very vexed one as numerous recommendations have been made for various purposes, but the following are suggested for drinking water (rain water and sewage are liable to variable sources of contamination beyond control):—

20 picocuries per litre for strontium 90 and 1000 picocuries per litre for "other beta-emitters".

The International Committee for Radiological Protection recommended^{8,9} that for air, the following standards were acceptable maxima:

Any mixture of beta and gamma radiation

except strontium 90 10^{-9} microcuries/ml.

Strontium 90 plus yttrium 90 2×10^{-10} microcuries/ml.

An average person breathes 20,000 litres of air per day, and this is equivalent to approximately 1 picocurie per litre or 395 counts per minute per 60 cu. ft. based on the particular instrument used, which has been standardised against Sr 90.

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Analytical Note

A Note on the Determination of Methyl Salicylate by Gas-Liquid Chromatography

by S. COLLINGS and R. SINAR

Stevens and Warren¹ described a method based on Gas-Liquid Chromatography for the determination of methyl salicylate in pharmaceutical preparations. Because of reported difficulties with existing methods, the technique was examined with a view to its adoption in an official standard book of reference*.

Initially the conditions imposed by the authors were rigidly adhered to, except that a flame ionisation detector was used instead of a katharometer detector. In view of the increased sensitivity of this detector less inert material was injected on to the column.

The accuracy claimed by the authors could not be reproduced, the difficulty of measuring accurately the base of the peak being undoubtedly the cause of error. The retention times of methyl salicylate and diphenyl are insufficiently far apart to allow peak height ratios to be used, the diphenyl peak being too broad-based under the conditions used. Although methods of increasing the accuracy of measurement of peak base are available, it was considered better to search for an alternative internal standard and to simplify the calculations by basing them solely on peak heights.

A suitable standard was found in benzyl alcohol. This had a retention time relative to methyl salicylate of 1.55, the two peaks just separating under the conditions used. The conditions were chosen to give narrow peaks completely separated from the solvent peak. The same column was used throughout the determinations. It shows no sign of becoming fouled by the small amounts of inert material injected on the flash heaters. The results obtained were extremely promising, variation from the mean of injection of standard solutions never exceeding 2 per cent. even when the peaks differed in height by a factor of 5. It is suggested that each batch of benzyl alcohol used should be standardised against methyl salicylate in view of the possibility of traces of impurities. The batch used showed a very small subsidiary peak with a retention time much lower than methyl salicylate. It did not interfere with the analysis.

Apparatus and Running Conditions

A Gas Chromatography Ltd. model S.6 gas chromatograph incorporating a flame ionisation detector was used. Copper columns, measuring 2 ft. ×

* Work carried out on behalf of the Revision Committee of the British Pharmaceutical Codex.

0.25 inch O.D. were packed with 10 per cent. Carbowax 20 M on 60/80 mesh Celite. The carrier gas was nitrogen, flowing at 50 cu. cm. per min. Temperatures used were: oven, 110° C isothermal, injection port, 250° C. The chart speed was 12 inches per hour.

Calibration Curve

The calibration plot of methyl salicylate/benzyl alcohol peak height against the ratio (weight methyl salicylate/weight benzyl alcohol) gave a straight line passing through the origin and corresponding to the expression $y = mx$, where $m = 0.9786$.

Standard Benzyl Alcohol Solution

Accurately weigh benzyl alcohol to give approximately a 4 per cent. w/v solution in petroleum spirit (boiling point 80/100° C).

Method

Into a 20-ml, calibrated flask, accurately weigh an amount of ointment or other preparation containing approximately 200 mg of methyl salicylate. Measure 5 ml of standard benzyl alcohol soln. together with 10 ml of petroleum spirit (boiling point 80/100° C) into the flask and shake until the ointment has completely dissolved or has been uniformly dispersed. Dilute the solution to 20 ml with petroleum spirit. Inject approximately 2 microlitres of the dispersion on to the column. Calculate the peak height ratio of methyl salicylate to benzyl alcohol and from the calibration curve determine the weight of methyl salicylate and hence its concentration in the original sample.

Results

The method has been applied to the three official preparations listed below with the following results.

	<i>Methyl salicylate found</i>	
	<i>Proposed Method</i> <i>per cent. w/w</i>	<i>B.P.C. Method</i> <i>per cent. w/w</i>
Ointment of methyl salicylate	49.6	49.4
Ointment of iodine and methyl salicylate ..	6.0	5.3
Liniment of methyl salicylate	{ 24.7	24.0
	{ 25.2	25.5

Reference

1. Stevens, S. G. E., and Warren, B., *J. Pharm. Pharmacol.*, 1964, **16**, 32T-34T.

R_f Values of Ponceau 6R

by D. PEARSON

(National College of Food Technology (University of Reading), Weybridge, Surrey)

Spalding¹ has pointed out that the sample used² (and which is referred to in the "Solmedia" set of non-permitted colours as "Ponceau 6R") is incorrectly labelled. The R_f values of what appears to be an authentic sample have now been determined using the method of ascending paper chromatography² and the 12 solvents previously described³:

R_f VALUES OF PONCEAU 6R USING SPECIFIED SOLVENTS

Solvent No.	R _f value	Solvent No.	R _f value
1	0.93	7	0.00
2	0.53	8	0.07
3	0.12	9	0.10
4	0.01	10	0.01
5	0.03	11	0.70
6	0.03	12	0.27

The sample of Ponceau 6R used was kindly supplied by Mr. E. I. Williams, Senior Analyst, Central Laboratory, Spillers Ltd., Cambridge. The manufacturer's container was labelled as "Farben fabriken, Bayer A.G. Leverkusen: 131386/10; Ponceau 6R bes. rein 70; 453708; 500gr."

References

1. Spalding, R. C., *J. Ass. Publ. Analysts*, 1964, 2 (4), 111.
2. Pearson, D., and Chaudhri, A. B., *ibid.*, 1964, 2 (2), 22.
3. Pearson, D., and Walker, R., *ibid.*, 1965, 3 (2), 45.

Legal Notes

MANUFACTURE AND SALE OF SHANDY

On the 1st February, 1966, an Appeal was heard in the High Court (*Hall v. Hyder*) which may at first appear to have little bearing on the work of the Public Analyst but which may nevertheless be worth recording for its implications.

A publican had been charged in the Magistrates Court with selling an intoxicating liquor, namely shandy, to a person under the age of 18. The Defence contended that since the liquid sold was a mixture of beer and lemonade, there was no proof that its alcoholic strength exceeded 2° proof, and that therefore the Prosecution could not prove that what had been sold to the young person was intoxicating liquor. The Magistrates dismissed the case on this ground and the Prosecution appealed against this decision.

The Lord Chief Justice said in his judgment that when a publican sells shandy made up to order for the purchaser, he is selling "beer, and quite separately, lemonade" and that he then mixes these two as the customer's agent. He said that this must be so because according to Section 163, Sub-Section 1, of the Customs and Excise Act, 1952, he is not allowed to sell beer diluted with any other substance. He then quoted the Sub-Section as follows. "If any dealer in or retailer of beer dilutes any beer or adds anything to beer other than finings for the purpose of clarification he shall be liable to a penalty of £50." He cited a case taken in 1887 (obviously under an earlier Act with the same provision) in which it was held on Appeal that a publican may legally sell what is known as "half and half", which involves mixing strong beer with a weaker beer, providing that this is done "in the customer's presence and as his agent". In other words, the publican is selling the two halves separately, and then as agent for the purchaser pours one into the other.

It was now clear, said the Lord Chief Justice, that what the publican in the case had sold to the young person was beer, which was certainly intoxicating liquor, and the fact that it had afterwards been mixed with lemonade was irrelevant. The prosecution must succeed and the case was sent back to the Magistrates with instructions to convict.

The question was raised by a Public Analyst whether this judgment did not mean that it was illegal for anyone to buy beer and lemonade or ginger beer, mix the beer with the soft drink, can or bottle the mixture, and offer the product for sale. The person or firm carrying out these operations must surely be dealing in beer and it could not be suggested that they were acting as an agent for the purchaser when at the mixing stage they would have no idea who the purchaser was going to be. However, the point was referred to the Solicitor of the Local Authority who gave it as his opinion that this was not so. A "dealer" in beer is defined under the Customs and Excise Act as a person who holds an Excise Licence under Section 146, and this is necessary for a person

dealing in beer by wholesale. A wholesaler is a person who buys an article in bulk and sells it again, usually in smaller quantities than those in which he buys it, but without doing anything to it except possibly changing the container or the labelling. The canner or bottler of shandy is not a wholesaler but a manufacturer, buying beer in bulk and then submitting it to a process (mixture with a soft drink) whereby its nature is changed. He is therefore not a wholesaler but a manufacturer, and is not committing an offence under Section 163 of the Act.

E. C. WOOD.

HIGH PROTEIN FOODS

Allegedly misleading claims were made in an advertisement for Gerber Strained Beef and Egg Noodles with Vegetables which appeared in a women's magazine. The entry ran:—

“Feed him” (a five month old baby) “a jar of Beef and Egg Noodles for dinner. It's high in protein—just like egg”.

Analysis showed that the baby food contained 2.84 per cent. protein, whereas egg contained 12.9 per cent. protein on the edible portion.

It was considered that the correct means of comparison was one of percentages; a number of mothers, after having read the advertisement, also thought that a spoonful of the baby food would contain as much protein as a spoonful of egg. However, even if one had compared the total weight of protein in a full jar of the food it was still only about one half of that in a standard egg. The Prosecution also quoted the protein contents of a number of other foods including baked potatoes, which were certainly not classed as foods high in protein and which contained some 3 per cent. of protein and so were about the same as the baby food.

The Medical Officer of Health for Salford gave his opinion that it was usual to feed babies with whole egg, yolk and white, starting with a “taste” at 8 or 10 weeks and gradually increasing the amount if this proved satisfactory. Indeed the advertisement itself suggested the feeding of egg to baby, that is the yolk and the white, not just the yolk.

The Defence was based on the following points:—

(a) Many leading authorities on baby feeding held the view that babies under 6 months should not be given white of egg.

(b) The calculations on which the allegations were based were meaningless; assessment by percentages or by weight or against different sizes of egg produced different results and one method of comparison showed that the food was superior to an egg yolk in the amount of protein present.

(This occurred when one compared the weight of protein in a full jar of the food with the weight of protein in one egg yolk: but when using this method of comparison the Defence conveniently omitted to state that the jar of food considered was seven times heavier than an egg yolk.)

(c) It was unfair to take a phrase out of context and put a false interpretation on it as had been done in this case. To read a phrase like "It's high in protein—just like egg" as meaning it was exactly equivalent to egg would make nonsense of present-day advertising. To emphasise this point the Defence produced advertisements for Mother's Pride Bread, which was "Just like mother used to bake" and one for toilet tissue "Soft and strong—just like Andrex".

The Stipendiary Magistrate said that although there were differences of opinion about the feeding of egg to babies, what had to be decided was whether or not the advertisement was misleading to the ordinary person. He found that this baby food was, in fact, high in protein. Therefore, there was no case to answer and it would be dismissed. Although the case was dismissed the Magistrate, nevertheless, suggested that the advertisement ought to be changed.

G. S. MEADOWS.

The Changing Nature of Chocolate

Scarcely any branch of the Confectionery Trade has been the subject of more controversial assessment of its products in recent years than that concerned with chocolate and chocolate coverings.

Judging by the reports of the proceedings of the International Standards Organisation, British Manufacturers appear to be alone in their views on the composition of confections which, although resembling the public conception of chocolate in appearance, are of such variable composition that it is hard to believe that they can virtually all be described by one name.

Proposals by the Food Standards Committee of the Ministry of Agriculture, Fisheries and Food, for Regulations governing the Labelling of Food, seek to define "chocolate" and "chocolate confectionery". These definitions do not dispel the confusion.

At Redditch Magistrates' Court on 8th September, 1965, a case was brought in which the Public Analyst, Mr. W. E. Jones, M.Sc., F.R.I.C., gave it as his opinion that Chocolate (as distinct from Milk Chocolate) should contain no fat other than cocoa butter. The case for the prosecution relied upon the established and traditional composition of chocolate. Nevertheless, the statements by the defence that modern trends were towards making other additions or substitutions evidently found some acceptance and the case was dismissed.

At this hearing the defence were supported by Dr. H. G. Harvey, M.Sc., Ph.D., F.R.I.C., a Specialist Consultant in Foods, by whose permission we reproduce the substance of the arguments in favour of the "New Chocolate", and contained in a memorandum published internally for members of the Association of Public Analysts.

MEMORANDUM ON THE MEANING OF THE WORD "CHOCOLATE"

(with particular reference to the character of the Added Fat)

(1) *The essential character of products* designated by the term "chocolate", whether used as an adjective or a noun, is a characteristic flavour and colour. In addition, in most but not all products to which the designation "chocolate" is applied, a characteristic physical quality of typical eating texture is also implied for the product to be recognised as "chocolate" *e.g.*, chocolate bars, chocolate Easter eggs, chocolate covered products all imply a typical eating texture but products such as chocolate cake mixture, chocolate sponge mixture, etc., do not generally require this characteristic eating *texture* but instead require just the characteristic flavour and colour.

(2) *The characteristic flavour and colour* are obtained by a suitable mixture of ground roasted cocoa beans or cocoa powder with sugar usually with suitable particle size reduction of the mixture.

(3) *The characteristic textural eating quality* in those "chocolate" products which require it (in order to be generally recognised by the designation "chocolate") is conferred essentially by the presence of added fat other than that which is present in a mixture of either ground roasted cocoa beans with sugar or of cocoa powder with sugar

proportions which give the characteristic flavour and colour. Other but minor ingredients may be required for various purposes.

The historical evolution of the chocolate industry naturally resulted, in the earliest stages of development of chocolate, in the use of cocoa butter as the main or almost sole type of added fat. This occurred for two reasons:—

(a) The physical properties of cocoa butter arising from its particular glyceride composition were found to be particularly suited, with the processing means then available, to provide acceptable physical characteristics and eating qualities to the limited range of products such as moulded chocolate bars, etc., then marketed.

(b) The consumption of chocolate was then sufficiently small for the economic problems associated necessarily with the processing of additional cocoa beans (to provide the "added fat") and leading to a by product (cocoa powder)—to be of no great significance—especially as the cocoa powder could then be utilised as a widely acceptable basis for cocoa drinks.

Subsequent developments involved—

(i) The need for modification of the compositional and physical characteristics of "chocolate" to suit its particular and developing range of applications.

(ii) The greatly increased consumption of chocolate.

(iii) The declining popularity of cocoa-type drinks.

(iv) The increased understanding of the physical properties of fats, their modification by processes such as fractionation, hydrogenation and inter-esterification and the realisation that, by use of different methods of tempering (other than those conventionally used when cocoa butter was the main added fat), normal "snap", gloss and textural eating properties could be obtained by using added fats other than cocoa butter.

(v) The development of blended fats which closely match the compositional and physical characteristics of cocoa butter with such an exactness that differentiation from cocoa butter by normal analytical techniques is impossible. One such fat indeed shows smaller differences—even when examined by elaborate techniques—from cocoa butter than occur between different cocoa butters.

(vi) The occasional incidence of cocoa disease or crop failure which raises the price of cocoa butter very high relative to other fats and generally because cocoa butter is dearer than other fats, have for long resulted in the use of fats other than cocoa butter in many chocolate applications.

The significance of the foregoing developments—(i) to (vi) requires, possibly, further explanation as follows:—

(i) Added cocoa butter as the only type of added fat was found not to result in the best product for all purposes. Thus, covering chocolate for many flour confectionery products requires a more flexible or plastic character than results from the use of cocoa butter alone. The flexible character of a sponge sandwich requires a chocolate coating which does not contract too much on setting (quite the opposite of what is required in moulded chocolate bars). Indeed, it is maintained by some technologists that even when the added fat is entirely a more plastic one than cocoa butter, nevertheless the cocoa butter present from the use of ground roasted cocoa beans is sufficient to prevent attainment of the best physical properties and that a more satisfactory product is obtained by use of low-fat content cocoa powder together with sugar and a fat of required physical characteristics (not cocoa butter).

Again, cocoa-butter type chocolate, when used in association with other fatty products, such as nuts, is particularly prone to the defect of fat-bloom and it has long been claimed that partial replacement of cocoa butter by other types of added fat can greatly reduce the risk of such spoilage.

A number of other such instances could be cited—and there is, of course, no logical or scientific or nutritional reason for believing that cocoa butter is the ideal added fat for all forms and applications of chocolate. It is a matter of practical fact that it is not.

(ii), (iii) and (vi) all result in a position whereby, if cocoa butter should be the only added fat permissible in chocolate, serious economic disadvantages would arise, involving unnecessarily high imports of cocoa beans into the country, increase in chocolate prices and occasionally, at least, shortages of chocolate.

(iv) and (v) are developments which cannot be denied and which have, to the credit of the industry, enabled chocolate of better quality to be made for many applications and of equal quality to those based on cocoa butter for other forms of chocolate, with economic advantages to suit all ranges of incomes.

It must indeed be recognised that the flavour, aroma and colour of chocolate products are quite independent of the character of the added fat. It has been argued that cocoa butter contains some chocolate aroma not possessed by other fats. It only requires a moment's reflection to realise that the amount of chocolate flavour (if it be admitted) contributed from cocoa butter cannot be more than would be contributed by a microscopic increase in the proportion of fat-free cocoa solids.

This argument has been tacitly admitted in those instances where compositional standards or codes of practice, in Britain, have been adopted at different times. Thus, in respect of chocolate couverture (1946), chocolate swiss rolls and in relation to flour confectionery the meaning of "chocolate" has always referred to a minimum content of dry fat-free cocoa solids without limitation as to the type of added fat.

There are no scientific, technological or nutritional or other quality grounds which necessitate that the added fat (where it is called for) should be cocoa butter. The initial use of cocoa butter was solely related to the limited economic, technical and range of requirements applicable at that time. To insist on the exclusion of other types of fat in relation to present circumstances is as logical as insisting that the original "man of war" of the British Navy should not have progressed from sail to steam to nuclear propulsion.

The Memorandum does not, however, express the united views of the Public Analysts and cogent arguments have been made in reply to it, both by W. E. Jones and by Mr. D. D. Moir, M.Sc., F.R.I.C. (Public Analyst for Surrey). An extract from a letter to the Editor from the former Analyst runs as follows:—

"In terms of economics, I must agree with many of the opinions expressed in Dr. Harvey's memorandum. But it must be emphasised that most of the points are just as valid for the development of margarine as a substitute for butter. This was also an economic necessity, but it has always been an offence to sell margarine as butter, and rightly so.

"In my opinion, I think that on behalf of the public we should object to changes in the composition of a familiar, traditional commodity such as chocolate. The use of other types of fat in chocolate can be justified only if their presence is suitably declared on the label.

"It may be of interest to know that one British Importer lists the Lausanne definitions of plain and milk chocolate as the U.K. requirements for these products.

"In conclusion, I would draw attention to the fact that the sample under discussion contained virtually no cocoa butter, this ingredient being replaced by a vegetable fat resembling hydrogenated palm kernel oil. The problem is, surely, that if we allow one substitute how can we prevent all the others?"

The communication from D. D. Moir comprises a very full statement of the evidence for the 'prosecution'.

CHOCOLATE, CHOCOLATE COUVERTURE AND CHOCOLATE COATINGS

"I have read the report of the case at Redditch Magistrates' Court in the October issue and Dr. Harvey's general apologia in the December issue* with considerable trepidation. The former decision is contrary to that given in other Magistrates' Courts and it is important that there is not a repetition in other places. Dr. Harvey's appearance on behalf of the defence is one which is to be deplored and his special pleading in relation to chocolate as such is surely against the interests of the chocolate and confectionery trade.

"I have no doubt that the technological reasons which Dr. Harvey adduces do create the necessity for some slight relaxation in the definition of chocolate as being the product of the cocoa nib with sugar and with or without added cocoa butter. This has been accepted for many years in the case of lecithin and, although we may not have known it or directly approved of it, the addition of a small quantity of fats other than cocoa butter has been the practice of some manufacturers for special conditions and purposes for as long or longer. But Dr. Harvey is not content with this. He would have the composition of chocolate completely altered by the unlimited substitution of foreign fats: and that not even restricted to coberine or similar products which are very good imitations of cocoa and possibly not distinguishable analytically. Complete substitution by hydrogenated palm kernel oil gives a product which is readily distinguishable by taste alone and is undoubtedly inferior. That it can be produced and sold for nearly half the price is immaterial. If the product is called chocolate people have a right to get chocolate and another name must be invented for the substitute article.

"There is no doubt that in the case of straight chocolate and couverture the technological troubles can be overcome by the inclusion of no more than about 10 per cent. of other fats in the total fat content of the chocolate and, since the latter is usually between 30 and 35 per cent., the need for substitute fats is only about 3 or 4 per cent. of the whole chocolate. An outside allowance of 5 per cent. is ample and this is the figure which, until recently, was allowed by Swiss law. I am in favour of making a relaxation to this extent for straight chocolate and couverture.

"The case of milk chocolate is a little different since for some purposes, such as prevention of 'bloom', butter fat itself will 'do the trick'. However, there is still scope needed for a softer or harder consistency according to external conditions and it may well be that a similar relaxation is necessary as for plain chocolate. I am informed on good authority, that the addition or substitution of other fats is self-limiting and that proportions larger than 10 per cent. of the total fat would usually result in an unsatisfactory product unless the *whole* of the cocoa butter were replaced. The only permissible ingredient other than those allowed for plain chocolate should be whole milk solids. This is the practice for all reputable makers of milk chocolate sold in this country and the proportion of milk solids used is far greater, often about twice as much, as any minimum standard which has been suggested. Where I fear there is liable to be a dispute is that if a minimum standard for whole milk solids were laid down, would it then be regarded as permissible to add skimmed milk powder over and above the minimum? I repeat that this is not British Practice except in the case of some milk chocolate couverture used on chocolate biscuits, and my view is that the moment the whole milk solids are diluted with skimmed milk the chocolate ceases to be milk chocolate. My reasons for this are as follows:—

"(1) The suggested standards for milk chocolate are set so low in respect of whole milk solids (or milk fat and skimmed milk solids equivalent to whole milk solids) that a milk chocolate conforming to the minimum would not normally be sold in this country.

"(2) The usual procedure as indicated above is to produce a milk chocolate containing up to twice this minimum amount of whole milk solids and the public have come to

* Internal "Bulletin of the Association of Public Analysts."

accept this as normal. Since the proportions of other major ingredients, sugar and cocoa solids including cocoa butter, are largely controlled by the demand for a sweet chocolate the milkiness of milk chocolate is judged by the colour. This may be rough and ready, but a very light chocolate is frequently very milky.

"(3) The position being what it is in Great Britain, I consider it to be deception and to the prejudice of the purchaser if the skimmed milk is used to produce a light coloured 'milk' chocolate, and any legal standard which attempts to justify this practice is to be deplored.

"I know that contrary views are held by some people and that articles such as malted milk have been sold in which the whole milk solids, although conforming to a reasonable minimum standard, have been supplemented by skimmed milk, but I do urge Public Analysts to think very carefully about this issue before they accept the principle. It would lead to a lowering of present standards.

"I now turn to the question of couverture, and any standards for the type of article used for enrobing chocolate confectionery would and should follow those for chocolate. The permission to use additional fats up to 5 per cent. of the product would overcome any technological difficulties. Larger proportions of fats other than cocoa butter would do more harm than good and the only alternative would be to allow complete substitution, which is unthinkable. The definition of chocolate given in the Proposals for the New Labelling of Food Regulations is a reasonable one and if this is accepted the position outlined above would automatically follow.

"Biscuits coated with chocolate or milk chocolate should present no problems and the same standards as for couverture are applicable. Incidentally, the minimum proportion of cocoa butter would probably be higher for couverture than for block chocolate.

"Dr. Harvey extends his remarks to chocolate used in flour confectionery and the soft 'chocolate' coatings used to cover sandwiches, buns, eclairs, fancy cakes, etc., do perhaps need further consideration. It has been said that although chocolate used as a noun should rightly conform to certain standards in which the fat content is restricted mainly to cocoa butter, it is unnecessary and illogical to require the same when 'chocolate' is used as an adjective. I cannot see the force of this argument. The important point is to try to fathom what the public expects, and if standards are made for chocolate used in blocks and as couverture for chocolate confectionery and biscuits, I cannot see that a purchaser would not expect the same basic composition if the same description, used adjectivally, were made for an article which includes a distinct coating of a substance which is similar in appearance to chocolate. A justifiable argument is the one implied in Section 47 of the Food and Drugs Act for 'cream' cakes. Although a substitute for cream had been used for many years, the Act managed to re-enforce the common sense view that cream means genuine cream if it has that appearance. In judging chocolate coatings I would have thought that Public Analysts ought to take the same view.

"The Trade contends that the technological difficulties are too great and that it is impossible to produce satisfactory coatings or genuine coatings of the right degree of consistency, which would stand up to long storage without cracking or developing 'bloom', or other defects. If this be true the products should not be referred to as 'Chocolate X' and another name should be sought. But the fact is that some of these commodities are produced and advertised as 'covered with real chocolate' and if one manufacturer can do it, why not others. I believe the truth to be that it is much easier and cheaper to use the substitute article and that no real effort has been made by the Bakery Trade to find a way out of the difficulties. I am open to be convinced, but if the same concession were made to allow certain small proportions of other fats as for chocolate and chocolate couverture and the Bakery Trade were to work on a slightly modified chocolate, if they have not already done so, I believe the basic conception of chocolate as being mainly a product of the cocoa nib could be maintained. Public Analysts should be very cautious before they yield to the idea of complete fat substitution. I believe

that the pressure for change is derived mainly from the shortage of cocoa butter and the consequent high price and that it is not unduly difficult to produce a high quality article if price is not regarded as the first consideration.

"Incidentally, I do not think that many of these articles are now described as 'chocolate X . . .' and if a purchaser were to ask for them under this description it would not be a difficult matter for bakery sales staff to be trained to explain that the coating is only 'chocolate flavoured'. A suitable disclaimer could be printed for pre-packed articles. A provision of this kind would very quickly provide the incentive to produce the real article because of its sales value.

"To summarise then, I would be in favour of a relaxation in the definition of chocolate and chocolate couverture to permit the addition of up to 5 per cent. of fats other than cocoa butter and milk fat, and that chocolate coatings for flour confectionery be allowed the same additions or, possibly, a slightly increased allowance if it could be shown to be an advantage. Flour confectionery products carrying coatings other than those conforming to the standards should be described as 'chocolate flavoured' in the absence of any other suitable name.

"I utterly reject the argument adduced that because we accepted the word chocolate for chocolate swiss rolls and sponge goods we must logically do so for coated goods. That which has the appearance of chocolate should consist of chocolate. A sponge flavoured with cocoa does not come under this category."

In conclusion, Dr. Harvey replied to the above contentions and here the matter rests until a further test-case is brought or until the ideas of the negotiators on the composition of "chocolate" are clarified.

"I think your members will agree that, in a seriously competitive world, British industry stands or falls by its ability continually to modify and improve the quality and economic worth of its products, not the least of which are manufactured foods. It is therefore surprising to read that your members fight vigorously to prevent changes which are contrary to tradition and accepted practice. Such changes, which you suggest should be opposed, most people consider necessary for our economic survival, as your members must know.

"It is also common knowledge that today in this country almost everyone has ample money for the purchase of food products and with the keen competition prevailing there is virtually no scope at all for the successful marketing of poor quality foods and certainly not for poor quality chocolate.

"In common with virtually all food products the quality of chocolate has consistently improved and such improvement will no doubt continue with increasing competition and availability of money to purchase this and other food products.

"It is quite untrue to say, with reference to chocolate (or to almost any other food)—that 'all the time' it is being degraded.

"My article, I submit, gave factual reporting in respect of chocolate and it is certainly true that, in those chocolate products where *textural* eating qualities is important (*i.e.*, chocolate bars, etc.), the *characteristic* quality of the chocolate does not (with modern advances in fat technology) require that the added fat present be solely cocoa butter. If this be granted then why should anyone try to put a brake on the application of scientific and technical advances? You do not change the name of the 'motor car' when it runs on diesel fuel instead of petrol or if you give it twin carburettors.

"The function of the Public Analyst is surely never intended to prevent the constant improvement of quality from application of technical progress: it surely is the proper function of the Public Analyst to confine himself to ensuring that nutritional quality and intrinsic eating characteristics are maintained or improved.

"To attempt to go further is surely only to stultify progress, prevent quality advances, and impede the economic improvement on which every one of us depends."

Liquid Glucose in Soft Drinks

The Soft Drinks Regulations, 1964, included in the section devoted to the interpretation of terms, a definition of the word "sugar" as used in the Regulations. It was held to mean, for this purpose, "any soluble carbohydrate sweetening matter". To a pure chemist this would present little difficulty of understanding, but the position is complicated by the views of the manufacturers of soft drinks who wish to include that controversial substance "Liquid Glucose", otherwise known as "hydrolised starch", etc., as a part of or as the whole of the sweetening agent in their products.

The problem results from the fact that "Liquid Glucose" contains not only sweetening compounds but other solids from the degradation of starch as well as a substantial amount of water. The question is, whether the whole "Liquid Glucose" or only the soluble solids (or the sweet fraction) should be considered as "soluble carbohydrate sweetening matter" for the purposes of the Soft Drinks Regulations.

With a view to clarifying the position, the Association of Public Analysts approached the Ministry of Agriculture, Fisheries and Food for a further definition of their intention in this specific case. The Ministry justifiably refuses to interpret its own regulations in this way. Administering Authorities or the Courts have this responsibility. Nevertheless, some satisfaction was gained from a reply in April 1965, which is reproduced here by kind permission of the Ministry.

"It will be recognised that only a Court can give an authoritative ruling as to the interpretation to be placed on a legal definition, but subject to this proviso our comments on the views expressed in your letter are as follows:—

"The definition of "sugar" in the Food Standards (Soft Drinks) Order 1953 reads:—
"Sugar" means the product usually known as sugar in commercial usage, consisting principally of sucrose.

"The above Regulations which replaced this Order were framed to cover all types of soft drinks including glucose beverages, low calorie drinks and drinks for diabetics. This necessitated a widening of the definition in the 1953 Order and "soluble carbohydrate sweetening matter" was considered appropriate for this purpose.

"In applying this definition to "glucose syrup" which is now the official designation for the carbohydrate sweetener previously known in the trade as "liquid glucose" it is clear that the component water not being sweet nor of carbohydrate nature cannot be reckoned as contributing to a soluble carbohydrate sweetening matter. In the same way any insoluble matter although this may be carbohydrate in nature may not be reckonable. Glucose syrup is used as an entity and is sweet although it may well be that certain portions of the carbohydrates present in this syrup contribute little to the sweetness. It is therefore our opinion that the total soluble solid carbohydrates present in the glucose syrups constitute "sugar" for the purpose of the Regulations.

"It is noted that you state in your letter "that it is only the sugar fraction of the liquid glucose solids which counts in the compositional standards". It is doubtful if this is correct for the following reasons. In the first place, since glucose syrup is an entity,

its constituents cannot easily be separated into sweet and non-sweet carbohydrates. Secondly even if it were accepted that the sweetness of a starch conversion syrup was essentially due to contributions of dextrose and maltose, variations in the relative proportions of these sugars will cause large variations in the actual sweetness since dextrose is much sweeter than maltose. Lastly there is the problem of finding a satisfactory analytical criterion for "sweetness". The commonly used dextrose equivalent is obviously of little value since it can give only a rough approximation for acid inverted syrups and is of even less value if the inversion has been carried out enzymatically. In consequence individual determinations of dextrose and maltose would have to be used. For all these reasons we regard it as logical and proper to regard "sugar" as including the total content of soluble carbohydrate solids.

"The definition will of course be re-examined in the light of experience when the current regulations are reviewed".

Book Review

BELL'S "Sale of Food and Drugs". Service Volume—Issue No. 11. Butterworth & Co. Ltd., London, 1966. Price 45s. 0d. (plus 2s. 0d. postage).

As 1965 saw the introduction of so much new legislation and proposals for new measures of control, it is pleasing to see another issue to the Service Volume of Bell's Sale of Food and Drugs. Much of the new matter, filling 200 pages, is included in this eleventh re-appraisal of the previous literature and brings the references up to date to the 1st December, 1965. Its prompt appearance is once more due to the industry of Mr. O'Keefe. Amendments and additions are made in all sections, and the value of "*Bell*" is universally acknowledged by all concerned with the "Sale of Foods and Drugs". It is unnecessary to mention any particular changes except, perhaps, to refer to an unreported amendment to a Code of Practice relating to Meat Food Substances. The Code was agreed between the Food Manufacturers Federation Inc. and the Association of Public Analysts in 1958. The amendment was recorded in the Journal of the latter Association in September 1965.

The activities of the Local Authorities Joint Advisory Committee are manifest by the appearance of two Codes of Practice which this body had been instrumental in introducing. Three previous Codes, however, have still escaped mention. Nevertheless, this Service Issue, like the Main Volume and Service Volume, is an indispensable source of information in the field.

J. H. E. MARSHALL.

Notice to Subscribers

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