STATUTORY INSTRUMENTS

1954 No. 613

EMERGENCY LAWS

FOOD STANDARDS (MARGARINE)

The Food Standards (Margarine) Order, 1954

Made - - - -	12*th May*, 1954
Laid before Parliament	14*th May*, 1954
Coming into Operation	16*th May*, 1954

The Minister of Food, in pursuance of the powers conferred upon him by Regulation two of the Defence (Sale of Food) Regulations, 1943(**a**), as continued in force by the Emergency Laws (Continuance) Order, 1953(**b**), and of all other powers him enabling in that behalf, hereby orders as follows:—

1. This Order shall come into operation on the 16th day of May, 1954, may be cited as the Food Standards (Margarine) Order, 1954, and shall be construed as one with the Food Standards (General Provisions) Order, 1944(**c**) as amended(**d**).

2. In this Order—

(*a*) any reference to margarine shall be construed as including a reference to any food, whether mixed with butter or not, which resembles butter and is not milk-blended butter.

(*b*) "catering business" includes the business or undertaking of an inn, public-house, hotel, restaurant, café, tea-shop, buffet, coffee-stall or any place of refreshment open to the public, or of a club, boarding-house, apartment-house, refreshment contractor, school feeding-centre, staff dining-room or canteen; and the word "caterer" shall be construed accordingly.

"sale" includes an offer for sale and exposure for sale.

"sale by retail" means a sale to a person otherwise than for the purpose of re-sale and includes a sale of margarine as such by a caterer in the course of his catering business but does not include a sale to a manufacturer for the purposes of his manufacturing business or a sale to a caterer for the purposes of his catering business.

"vitamin D" means the anti-rachitic vitamins.

3. Pursuant to Regulation two of the Defence (Sale of Food) Regulations, 1943, the Minister of Food hereby prescribes that the standard for margarine, as respects vitamin A and vitamin D contained therein, shall be as specified in the First Schedule to this Order.

4. The standard prescribed by this Order shall apply only as respects sales by retail.

(**a**) S.R. & O. 1943/1553, 1945/1454 (1943 II, p. 70; 1945 II, p. 95).
(**b**) S.I. 1953/1768.
(**c**) S.R. & O. 1944/42 (1944 II, p. 505).
(**d**) S.R. & O. 1944/654 (1944 II, p. 508).

5. Proceedings in England and Northern Ireland for an infringement of Article 1 of the Food Standards (General Provisions) Order, 1944 as amended, in respect of margarine may be brought by a Food and Drugs Authority without the consent of the Minister.

G. Lloyd-George,
Minister of Food.

Dated the 12th day of May, 1954.

FIRST SCHEDULE

The standard for margarine shall be as follows:—

Each ounce of margarine shall contain—

(*a*) not less than 760 international units and not more than 940 international units of vitamin A determined in accordance with the method set forth in the Second Schedule to this Order. The vitamin A content shall be calculated as the sum of the vitamin A present as such or as its esters plus 0·8 times the beta-carotene equivalent of any carotenes present; any alpha-carotene being considered as equivalent in potency to half its weight of beta-carotene; and when red palm oil is used as a source of carotenes, the beta-carotene equivalent shall be taken as 53·5 per cent. of the total carotenes;

(*b*) not less than 80 international units and not more than 100 international units of vitamin D.

SECOND SCHEDULE

Method for Determination of Vitamin A Content

Principle

The margarine is saponified, the unsaponifiable matter is extracted with diethyl ether and passed through a process of double column chromatography, which separates carotene from vitamin A. The optical density of the carotene solution is measured over the spectral range 440–450 mμ, and the vitamin A fractions, after being identified by the Carr-Price reaction, are combined in light petroleum. The optical density of the solution is measured at the wavelength of maximum absorption, normally at 324 mμ. All operations should be carried out under subdued lighting conditions.

Apparatus

1. Chromatographic apparatus, (see sketch of assembly and of components). It consists of an upper and lower column. The lower column is fitted with a side arm which can be adjusted so that the eluate from the upper column can either by-pass, or pass through, the lower column. The assembly is fitted with three-way taps to enable air pressure to be applied either to the top column alone or to both columns.

2. Graduated tubes.—1 ml. (see sketch).

3. Spectrophotometer.—For measuring ultra-violet absorption of vitamin A solution.

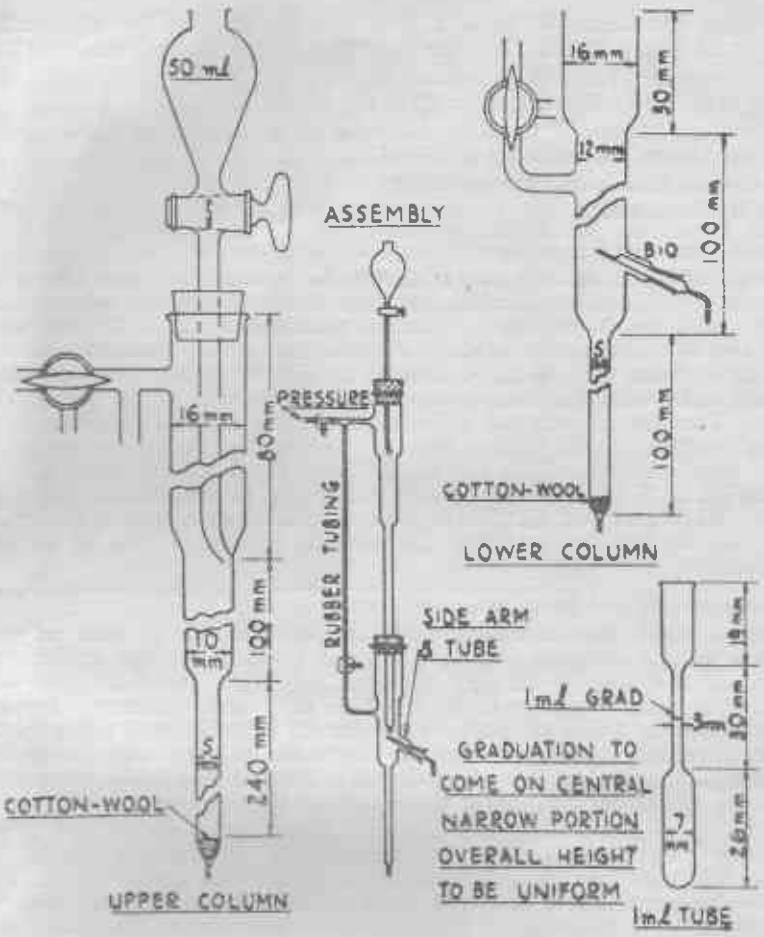
4. Quartz cuvettes.—Two; each of 1 cm.

5. Saponification flasks.—300 ml. resistance glass.

6. Separators.—500 ml. capacity.

7. Graduated flasks ("A" grade).—10 ml., 50 ml.

8. Pipette ("A" grade).—1 ml., the tip drawn out to a capillary to fit graduated tubes.



CHROMATOGRAPHIC APPARATUS FOR VITAMIN A ASSAY

SKETCH OF ASSEMBLY AND OF COMPONENTS

Reagents

All reagents must be of the quality required for quantitative chemical analysis.

1. Alumina (Type 1).—Prepare from alumina trihydrate.

Activate the fraction passing through a 150 mesh British Standard sieve by heating at 800°C. for 7 hours. After cooling, add distilled water, 2 g. per 98 g. activated alumina, and mix well. Keep in glass stoppered bottle.

2. Alumina (Type 2).—Place 10 g. of activated alumina (Type 1) in a 50 ml. round bottom flask, add 10 ml. of sodium hydroxide solution, and mix to a thin paste. Attach the flask to a vacuum pump and evacuate for 30 minutes at room temperature at 20 mm. pressure or lower. While still under this reduced pressure, place the flask in an oil bath at 135°C. for 30 minutes. Cool under reduced pressure and shake out the alumina-sodium hydroxide into a small mortar, ignoring any which adheres to flask. Add 1·2 ml. water slowly while stirring gently with a pestle. Immediately transfer the smooth powder to a small well stoppered bottle.

3. Antimony trichloride.—A saturated solution in chloroform (Carr-Price reagent).

4. Diethyl ether.—Freshly distilled over sodium hydroxide pellets.

5. Ethyl alcohol.—Absolute

6. Light petroleum (40 to 60°C.).—To obtain an optically pure fraction it may be necessary to distil and use only that portion boiling below 45°C.

7. Potassium hydroxide solution.—Dissolve 60 g. of potassium hydroxide pellets in distilled water and make up to 100 ml.

8. Sodium hydroxide solution.—Dissolve 10 g. of sodium hydroxide pellets in distilled water and make up to 100 ml.

9. Carbon dioxide or hydrogen supply.

10. Hydroquinone.

Saponification and Extraction of Unsaponifiable Matter

Weigh 10·0 g. margarine into a 300 ml. flat bottom flask, add 20 mg. hydroquinone, 40 ml. absolute alcohol, 10 ml. potassium hydroxide solution and boil under reflux for 20 minutes. Transfer quantitatively to a 500 ml. separator using two 40 ml. quantities of distilled water and extract once with 100 ml. and then three times with 50 ml. quantities of diethyl ether. Combine the ether extracts and wash with four successive portions, each of 50 ml. of distilled water. Transfer the ethereal solution in suitable aliquots quantitatively to a 300 ml. wide-mouth flat-bottom flask. All subsequent operations are conducted in an inert atmosphere e.g. carbon dioxide. Distil off the ether, add to the residue about 5 ml. absolute alcohol and reheat to remove the last traces of water. Make sure that no trace of alcohol remains, as otherwise the chromatogram will not develop properly. Dissolve the residue in 5 to 10 ml. of light petroleum.

Chromatography and Measurement of Optical Density

Place a small wad of cotton wool in the lower tip of each of the two columns of the chromatographic apparatus. Fill the upper column to the middle of the tube of 10 mm. diameter with light petroleum and pour in sufficient alumina (Type 1) to fill the tube of 5 mm. diameter. Prepare the lower column in a similar manner with alumina (Type 2), half filling the 5 mm. diameter section. Close the tip of the lower column with rubber tubing plugged with a short piece of glass rod. Connect the lower column to the upper column so that the outflow passes through the side arm.

Apply air pressure to the top column. When the excess of light petroleum has been forced through the column, release the pressure and transfer quantitatively the light petroleum solution of unsaponifiable matter to the column. Develop under pressure, first with 5 ml. of light petroleum, and then successively with 5 ml. quantities of light petroleum and diethyl ether mixtures containing respectively 4, 8, 12, 16, 20, 24 and 36 per cent. of diethyl ether. During these operations the level of liquid should not fall below the upper surface of the alumina.

Carotene

Carotene passes quickly down the column and is eluted before the 16 per cent. diethyl ether solvent mixture is introduced, the eluate being by-passed through the side tube into a receiver. Reduce the volume of the carotene solution, if necessary, by evaporation in an inert atmosphere, transfer to a 50 ml. graduated flask and make up to volume with light petroleum.

Measure the optical density of the solution in a 1 cm. quartz cuvette, against a light petroleum blank over the spectral range 440–450 mμ. at 2 mμ. intervals. From the optical density at λ max.* calculate the E (1 per cent., 1 cm.) value and multiply this by 358 to obtain the equivalent β-carotene potency in International Units per g. This value multiplied again by 0·8 will give the equivalent vitamin A potency of the carotenoids in International Units per g.

Vitamin A

Immediately prior to adding the 16 per cent. diethyl ether developing solvent, remove the plug from the tip of the lower column and the tube from the side arm of the lower column and plug the side arm orifice. Adjust the flow into

* λ max. means the wavelength of light at which the optical density reaches its maximum value.

and out of the lower column by means of the three-way cock and collect the eluate in calibrated 1 ml. tubes. Mix the solution in each tube by blowing through the liquid a few air bubbles with the drawn out pipette. Withdraw, from each tube, approximately 0·3 ml. of liquid and test for vitamin A by adding about five drops of antimony trichloride solution. The development of a blue colour indicates the presence of the vitamin. From each tube in which a positive test is obtained, take exactly 0·5 ml. of solution, combine together in a 10 ml. graduated flask and adjust to volume with light petroleum. Measure the optical density of the solution in a 1 cm. quartz cuvette, against a light petroleum blank, over the spectral region 290 to 340 mμ. at the following wavelengths 290, 307, 309, 311, 322, 324, 326, 328, 332, 334, 336 and 340 mμ. Transfer the test solution to the blank cuvette, and the blank solution to the test cuvette, re-measure the optical density at λ max. and from the mean of the two measurements calculate the value of E (1 per cent., 1 cm.) at λ max. Multiply this value by 1,830 to obtain the vitamin A potency in International Units per g. The total vitamin A potency is equal to the sum of the potency of the vitamin A and the equivalent vitamin A potency of the carotenoids.

The validity of the test is assessed from the optical density measurements at (λ max. minus 15 mμ.) and (λ max. plus 10 mμ.). The ratio of the optical density at each of these wavelengths to the optical density at λ max. (normally 324 mμ.) should fall within the range of 0·86 ± 0·02. If this is not so the whole determination must be repeated.

EXPLANATORY NOTE

(This note is not part of the Order, but is intended to indicate its general purport.)

This Order, which should be read with the Food Standards (General Provisions) Order, 1944, as amended, prescribes a standard for margarine as respects vitamin A and vitamin D to be contained therein.

The standard applies on sale by retail (as defined) only and applies alike to imported and home-produced margarine.

(33202) (V. 21) K6 6/54 D.L.