Vinyl chloride

II

(Preparatory Acts)

COMMISSION

Proposal for a Council Directive on the approximation of the laws of the Member States relating to materials and articles containing vinyl chloride monomer and intended to come into contact with foodstuffs

(Submitted by the Commission to the Council on 21 December 1976)

THE COUNCIL TO THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Directive 76/893/EEC of 23 November 1976 on the approximation of the laws of the Member States relating to materials and articles intended to come into contact with foodstuffs [1], and in particular Article 3 thereof,

Having regard to the proposal from the Commission,

Having regard to the opinion of the European Parliament,

Having regard to the opinion of the Economic and Social Committee,

Whereas Article 2 of Directive 76/893/EEC provides that materials and articles must not transfer to foodstuffs any constituents in quantities liable to endanger public health;

Whereas Article 3 of the same Directive provides that the Council, under the procedure provided for in Article 100 of the Treaty, shall adopt by a Directive special provisions applicable to certain groups of materials and articles ('specific Directives'); whereas these provisions may include specific limits on the migration of certain constituents into or onto foodstuffs as well as other rules to ensure compliance with Article 2 of the said Directive;

Whereas the administration of large doses of vinyl chloride monomer to experimental animals has been shown to produce harmful effects; whereas such effects could also occur in man;

Whereas the Scientific Committee for Foodstuffs has given the opinion that 'the levels of vinyl chloride monomer in polyvinyl chloride and related polymers should be reduced as far as possible', and at the same time recommended that 'no trace of vinyl chloride should be detectable in food or potable water by a method which can be generally applied to the majority of foodstuffs by most laboratories';

Whereas further research is at present in progress on vinyl chloride monomer, but as a precaution the absorption of vinyl chloride monomer should be restricted until these results are known;

Whereas the adaptation of the Directive to technical progress is an implementing measure; whereas, in order to simplify and accelerate the procedure, this should be the responsibility of the Commission;

Whereas, in all cases in which the Council confers on the Commission authority to implement rules relating

[1] OJ No L 340, 9. 12. 1976, p. 19.

to materials and articles intended to come into contact with foodstuffs, a procedure should be laid down establishing close cooperation between Member States and the Commission within the Standing Committee on Foodstuffs set up under the Council Decision of 13 November 1969,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Pursuant to Article 3 of Directive 76/893/CEE of 23 November 1976, this Directive concerns materials and articles prepared with vinyl chloride polymers or copolymers which in their finished state are intended to come into contact with foodstuffs, or which are in contact with foodstuffs and are intended for that purpose. They are hereinafter called 'materials and articles'.

Article 2

Materials and articles shall not contain vinyl chloride monomer in quantities exceeding the limits laid down in Annex I.

Foodstuffs having come into contact with materials and articles shall not contain vinyl chloride monomer in quantities exceeding the limit laid down in Annex I,

Article 3

Compliance with the limits laid down in Annex I shall be checked by the method described in Annex II.

Article 4

Amendments to adapt the Annexes to this Directive in accordance with developments in scientific and technical knowledge shall be adopted in accordance with the procedure laid down in Article 10 of Directive 76/893/EEC of 23 November 1976.

Article 5

This Directive shall not affect national provisions relating to other possible standards provided for in Article 3 of Directive 76/893/EEC of 23 November 1976.

Article 6

1. To comply with the provisions of this Directive, the Member States shall, where necessary, amend their respective laws by 1 January 1978 and inform the Commission forthwith.

2. The laws as thus amended shall be applied with effect from 1 July 1978 both to materials and articles, and to foodstuffs with which such materials and articles have come into contact.

Article 7

This Directive is addressed to the Member States.

ANNEX I

LIMITS OF VINYL CHLORIDE MONOMER IN MATERIALS, ARTICLES AND FOODSTUFFS

Materials and articles:

1 mg/kg in the final product. However, in the case of materials and articles prepared with vinyl chloride copolymers and not intended for use in packaging or containing liquids for human consumption, the limit shall be 5 mg/kg.

Foodstuffs:

0·050 mg/kg.

ANNEX II

DETERMINATION OF VINYL CHLORIDE (VC) MONOMER IN MATERIALS, ARTICLES AND FOODSTUFFS

1. PRINCIPLE

The presence of VC in foodstuffs, materials or articles is determined by means of gas-liquid chromatography using the 'head space' method after solution or suspension of the sample in dimethylacetamide (DMA).

2. REAGENTS

2.1. VC, of purity greater than 99·5 %.

2.2. N-N-DMA, not containing any volatile impurity, with the same chromatography retention time as VC.

2.3. Any appropriate internal standard solution, such as diethyl ether in DMA.

3. APPARATUS

NB:

An instrument or piece of apparatus is mentioned only if it is special or made to particular specifications; usual laboratory apparatus is assumed to be available.

3.1. Gas chromatograph fitted with an automatic head-space sampler and flame ionization detectors. Alternatively, a gas chromatograph fitted for manual sample injection and equipped with a flame ionization detector may be used. It has also been found that it is satisfactory to use a specific detector in the gas chromatograph instead of a flame ionization detector, e.g. a micro-electrolytic conductivity detector of the kind described in the Journal of Chromatographic Science, volume 12 (March 1974), page 152.

3.2. A potentiometer.

3.3. Sample phials or flasks fitted with a silicone or butyl rubber septum.

When using the manual technique, the taking of a sample in the head space by means of a syringe may cause the formation of a partial vacuum inside the phial or flask. Hence, for manual techniques where the phials are not pressurized before the sample is taken, the use of large phials is recommended.

3.4. Micro-syringes.

3.5. Gas-tight syringes for manual head-space sampling.

4. PROCEDURE

4.1. Preparation of standard VC solution

NB:

VC must be handled in a ventilated fume cupboard.

Weigh a suitable glass vessel accurately and place in it a quantity (e.g. 50 ml) of DMA. Re-weigh. Add to the DMA a quantity of VC (e.g. 0·1 mg) in liquid or gas form, in the latter case by bubbling in or injecting. After again re-weighing, calculate from the difference the quantity of VC in solution and hence the concentration of the solution obtained (standard solution). This standard solution will keep for a long time if stored in a refrigerator.

Prepare a dilute VC solution by taking an aliquot of standard solution and adding it to a predetermined quantity of DMA contained in an already tared glass phial, at the same time reducing the volume of air (head space) as much as possible. Re-weigh and calculate the VC concentration by difference.

4.2. Preparation of sample

NB:

— Take all the necessary precautions to ensure that no VC is lost through volatilization.

— Take samples of foodstuffs, materials or articles in such a way that they are representative of the product under investigation. For this purpose, in the case of non-liquid foodstuffs, homogenize the samples under investigation accurately before taking an aliquot.

4.2.1. *Foodstuff sample*

Weigh accurately into each of a set of three phials a suitable quantity of foodstuff under investigation. If considered necessary, add an appropriate quantity of DMA (this sometimes promotes the attainment of a state of equilibrium) and/or of the internal standard. Seal immediately and homogenize.

4.2.2. *Calibration curve for the determination of VC in foodstuffs*

Weigh into each of a set of not less than five phials a proportionate quantity of the same foodstuff which has never been in contact with materials or articles containing VC. If some DMA and/or some of the internal standard has been added to the foodstuff sample under 4.2.1, add proportionate quantities to each reference sample.

Finally, add diluted standard VC solution in quantities such as give VC concentrations equal to 0·000, 0·050, 0·075, 0·100, 0·150 mg/kg, etc. Seal immediately and homogenize.

4.2.3. *Sample of material and article*

Weigh accurately into each of a set of three phials about 200 mg of the material or article, sampled from the product under investigation and reduced to small size. Insert a magnetic stirrer and about 2 ml of DMA containing, if considered necessary, an appropriate quantity of internal standard solution. Seal the phial, suspend it in a waterbath held at 60 to 70 °C and dissolve the sample by stirring the solution vigorously.

4.2.4. *Calibration curve for the determination of VC in materials or articles*

Place about 290 mg of a material or article of the same type as described in 4.2.3., not containing any detectable amount of VC, in each of a set of not less than five phials. Insert a magnetic stirrer and add about 2 ml of DMA containing a suitable quantity of internal standard, if this has been used under 4.2.3.

Dissolve the material or article by stirring. Add diluted standard VC solution in quantities such as give a range of concentrations comprising the concentration of the VC in the sample under investigation. Seal immediately and homogenize.

4.3. Gas-chromatography determination

4.3.1. *Bring the sample to a state of equilibrium*

Allow the samples contained in the sealed phials a sufficient time to attain a state of equilibrium before beginning the head-space sampling operation. A period of two hours at 50° C is usually sufficient for this purpose.

4.3.2. *Determination*

Choose a gas-chromatography column and operating conditions such that the product under investigation does not interfere with the VC or, if applicable, with

the internal standard. Periodically flush the column so as to eliminate DMA peaks from the chromatogram. Measure the area (or height) of the peaks representing the VC or the internal standard, if used, and deduce the VC concentration of the sample of foodstuff, material or article under investigation from the constructed calibration curves.

4.4. Interference peaks on the chromatograms

Certain foodstuffs give rise to interference peaks. In that event, confirm that VC is actually present in the foodstuff sample, either by using a micro-electrolytic conductivity detector as mentioned in 3.1 or by means of mass spectrometry. In this event, the fact that molecular ions with parent masses (m/e) of 62 and 64 are present in the ratio of 3 : 1 is regarded as confirming that VC is present. If such interference is confirmed, use a different column.