DUS 1679

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## Specification for polyvinylchloride (PVC) for its safe use in contact with food stuffs, pharmaceuticals and drinking water



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## Foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Trade, Industry and Cooperatives established under Cap 327, of the Laws of Uganda, as amended. UNBS is mandated to co-ordinate the elaboration of standards and is

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The committee responsible for this document is Technical Committee UNBS/TC 19, Packaging and Packaging products



 

# Specification for Polyvinyl chloride (PVC) for its safe use in contact with food stuffs, pharmaceuticals and drinking water

## 1 Scope

This Draft Uganda Standard specifies the requirements and methods of sampling and test for polyvinyl chloride (PVC) and its copolymers for the manufacture of plastic items used in contact with foodstuffs, pharmaceuticals and drinking water.

## 2 Normative references

The following referenced documents referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

DUS 1667*, Positive list of constituents of polyvinyl chloride (PVC) and its copolymers in contact with food stuffs, pharmaceuticals and drinking water.*

US 1659, *Packaging materials for food contact use – General requirements*

US 1675*, Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs – Method of analysis*

## 3 Terms and definitions

For the purposes of this standard, the following term and definition shall apply.

**3.1**
**Polyvinyl Chloride**
shall mean:

a) the homopolymers of vinyl chloride;

b) copolymers containing at least 50 percent by mass of vinyl chloride with one or several of the following monomers:

1) vinylidene chloride;

2) acrylonitrile;

3) styrene and substituted styrene;

4) butadiene;

5) ethylene;

6) propylene;



 

7) divinyl benzene;

8) vinyl acetate;

9) maleic, fumaric, itaconic, crotonic, acrylic and methacrylic acids ( maximum limit of 8 percent by mass of all these monomers); and

10) esters of maleic, itaconic, crotonic, acrylic and methacrylic acids with saturated monohydric aliphatic alcohols;

c) mixtures of homopolymers of vinyl chloride with one or several of the copolymers indicated in (b); and

d) mixtures of several copolymers indicated in (b).

# 4 Requirements

## 4.1 Material

The material shall comply with the threshold limits of the catalysts, polymerization inhibitors, emulsifying agents, suspension agents, chain-transfer agents, miscellaneous additives, auxiliary items, that is, plasticizers, stabilizers, and other additives, as prescribed in DUS 1667.

As the foils are to be used in contact with food stuffs they shall be produced with rolling oils/ lubricants which do not contain substances which are injurious to health or have any deleterious effect on the flavour, odour or appearance of food stuffs.

## 4.2 Pigments and colorants

In case the coloured material is used for food packaging applications, it shall comply with the list and limits of the pigments and colorants prescribed in US 1659

## 4.3 Monomer content

3.3.1 The vinyl chloride monomer content of PVC suspension resin used for the manufacture shall not exceed 5 ppm, when tested as prescribed in Annex A

3.3.2 The vinyl chloride monomer content (VCM) in the PVC containers/film used for packaging shall not exceed 1 ppm, when tested as prescribed in Annex A.

3.3.3 The residual migration of VCM into foodstuffs being packed shall not exceed 10 ppm.

## 4.4 Overall migration

The material shall also comply with the overall migration limit as detailed below when tested by the method prescribed in US 1675

In the case of liquid foodstuffs or of simulants, the limit shall be 60 mg/l, *Max.* However, the value of the overall migration limit shall be equal to 10 mg/cm$^2$, Max*,* of the surface of the material or article in the following cases:

a) Containers or articles which are similar to containers or which in any case may be filled to a capacity less than 250 ml provided it is possible to calculate the surface of contact with the foodstuff.

b) Sheets, foils and other non-fillable articles for which ratio between the surface area of the material or article and the quantity of foodstuffs in contact may not be calculated.



 

### 4.5 Storage and control

#### 4.5.1 Storage

Plastics materials intended for food contact use shall be stored separately from other materials in closed, properly identified containers.

#### 4.5.2 Good Manufacturing Practices (GMP)

GMP shall be maintained at all times and personnel shall be trained in GMP.

## 5 Packing and marking

**5.1** The material shall be packed as agreed between the purchaser and the manufacturer/supplier, in a manner so as to ensure that the items do not become contaminated during storage.

**5.2** Each package shall be clearly marked with the name and type of the material, month and year of manufacture of the material, name of the manufacturer and manufacturer's trade mark, if any.

## 6 Sampling

The method of preparing representative test samples of the material and the criteria for conformity shall be as prescribed in Annex B.



 

# Annex A
(normative)

# Determination of vinyl chloride monomer in basic materials and articles

## A.1 General

In this method the vinyl chloride monomer level in basic materials or articles is determined by means of gas chromatography using the 'headspace' method after dissolution or suspension of the sample in N, N-dimethylacetamide

## A.2 Reagents

**A.1.1** Vinyl chloride (VC) of purity greater than 99.5 %

**A.1.2** N, N-dimethylacetamide (DMA), free from any impurity with the same retention time as VC or as the internal standard (A I.3) under the conditions of the test.

**A.1.3** Diethyl ether or cis-z-butene, in DMA (A I.2) as the internal standard solution. These internal standards shall not contain any impurity with the same retention time as VC, under the conditions of the test.

## A.3 Apparatus

### A.3.1 Gas-chromatograph

Fitted with automatic head space sampler or with facilities for manual sample injection.

### A.3.2 Flame ionization detector or other detectors mentioned in A 6.

### A.3.3 Gas-chromatographic Column

The column shall permit the separation of peaks of air, or VC and of the internal standard, if used. Furthermore, the combined *A 2.2* and *A 2.3* system shall allow the signal obtained with a solution containing 0.02 mg VC/litre DMA or 0.02 mg VC/kg IMA to be equal to at least five times the background noise.

### A.3.4 Sample vials or flasks fitted with silicon or butyl rubber septa

When using manual sampling techniques the taking of a sample from the headspace with a syringe may cause a partial vacuum to form inside the vial or flask. Hence, for manual techniques where the vials are not pressurized before the sample is taken, the use of large vials is recommended

A.3.5 Micro-syringes

A.3.6 Gas-tight syringes for manual headspace sampling

A.3.7 Analytical Balance; accurate to 0.1 mg.

## A.4 Procedure

**CAUTION** VC is a hazardous substance and a gas at ambient temperature, therefore, the preparation of solutions should be carried out in a well ventilated fume cupboard. Take all the necessary precautions to ensure that no VC or DMA is lost.

NOTE 1 When employing manual sampling techniques an internal standard (A.1.3) should be used.

NOTE 2 When using an internal standard, the same solution shall be utilised throughout the procedure.

### A.4.1 Preparation of Concentrated Standard VC Solution at Approximately 2000 mg/kg

Accurately weigh to the nearest 0.1 mg a suitable glass vessel and place in it a 50 ml quantity of DMA. Reweigh. Add to the DMA 0.1 g of VC in liquid or gas form, injecting it slowly on to the DMA. The VC may also be added by bubbling it into the DMA, provided that a device is used which shall prevent loss of DMA. Re-weigh to the nearest 0·1 mg. Wait for two hours to allow equilibrium to be attained. Keep the standard solution in refrigerator**.**

### A.4.2 Preparation of Dilute Standard VC Solution

Take a weighed amount of concentrated standard solution of VC (A 3.1) and dilute, to a known volume of a known weight, with DMA or with internal standard solution (1.3). The concentration of the resultant dilute standard solution is expressed as mg/l or mg/kg respectively.

### A.4.3 Preparation of Calibration Curve

NOTE The curve shall comprise at least 7 pairs of points.

**A.4.3.1** The repeatability of the responses shall be lower than 0.02 mg VC/l or kg of DMA. The curve shall be calculated from these points by the least squares technique, that is, the regression line shall be calculated using the following equation:

$$y = a_1 x + a_0$$

where,

$$a_1 = \frac{n\sum xy - (\sum x) \times (\sum y)}{n\sum x^2 - (\sum x)^2}$$

$$a_0 = \frac{(\sum y)(\sum x^2) - (\sum x)(\sum xy)}{n\sum x^2 - (\sum x)^2}$$

where

$y$ the height or area of peaks in any single determination,



 

*x* the corresponding concentration on the regression line, and

*n* number of determinations carried out *(n.*≥ 14).

**A.4.3.2** The curve shall be linear, that is, the standard deviation, (s) of the differences between the measured responses ($y_1$) and the corresponding value of the responses calculated from the regression line ($z_1$) divided by the mean value ( $\bar{y}$ ) of all the measured responses shall not exceed 0.07.

This shall be calculated from:

$$\frac{s}{\bar{y}} = \leq 0.07$$

$$s = \sqrt{\frac{\Sigma\left(y_i - \bar{y}\right)^2}{n-1}}$$

where

$y_i$ each individual measured response,

$Z_i$ the corresponding value of the response ($y_i$) on the calculated regression line, and

*n* ≥14.

**A.4.3.3** Prepare two series of at least 7 vials (A.3.4). Add to each vial volumes of dilute standard VC solution (A.4.2) and DMA (A.2.3) or internal standard solution in DMA (A.2.3) such that the final VC concentration of the duplicate solutions shall be approximately equal to 0, 0.050, 0.075, 0.100, 0.125, 0.150, 0.200 mg/l or mg/kg of DMA and that all the vials contain the same quantity of DMA that is to be used under A.4.4. Seal the vials and proceed as described under A.4.5. Construct a graph in which the ordinate values show the areas (or heights) of the VC peaks of the duplicate solutions or the ratio of these areas, (or heights) to those of the relevant internal standard peaks and the abscissa values show the VC concentrations of the duplicate solutions.

**A.4.4** Repeat the procedure described under A.4.1 and A.4.2 to obtain a second diluted standard solution with a concentration equal to 0.1 mg VC/l or 0.1 mg/kg of DMA or internal standard solution. The average of two gas-chromatographic determinations of this solution shall not differ by more than 5 % from the corresponding point of the calibration curve. If the difference is greater than 5 %, reject all the solutions obtained in A.4.1, A.4.2, A.4.3 and A.4.4 and repeat the procedure from the beginning.

**A.4.5** Prepare two vials (A.3.4). Weigh into each vial not less than 200 mg, to the nearest 0.1 mg, of the sample obtained from a single material or article under investigation which has been reduced to small pieces. Try to ensure that an equal quantity is weighed into each vial. Close the vial immediately. Add to each vial for each gram of the sample 10 ml or 10 g of DMA (A.2.3) or 10 ml or 10 g of internal standard solution (A.2.3). Seal the vials and proceed as described under A 5.

## A.5 Gas chromatographic determinations

**A.5.1** Agitate the vials avoiding contact between the contained liquid and the septum ( A.3.4 ) to obtain a solution or suspension of the samples of material or article ( A.4.5) as homogeneous as possible

**A.5.2** Put all the sealed vials (A.4.3, A.4.4 and A.4.5) in a water bath for 2 hours at 60 $^0$C ±1 $^0$ C to allow equilibrium to be attained. Agitate again if necessary



 

A.5.3 Take a sample from the headspace in the vial. When utilising manual sampling techniques care shall be exercised in obtaining a reproducible sample (see A.3.4), in particular the syringe shall be pre - warmed to the temperature of the sample. Measure the area (or height) of the peaks relating to the VC and to the internal standard, if used.

A.5.4 Remove from the column (A.3.3) excess DMA using an appropriate method as soon as peaks of DMA appear on the chromatogram.

## A.6 Calculation of the test results

**A.6.1** Find by interpolation on the curve, the unknown concentration of each of the two solutions of the sample taking account of the internal standard solution if used. Calculate the amount of VC in each of the two samples of material or article under investigation by applying the following formula

$$X = \frac{C \times V}{M} \times 1000$$

where

X concentration of VC in the sample of the material or article expressed in mg/kg,

C concentration of VC in the vial containing the sample of material or article *(see* A.4.5) expressed in mg/l or mg/kg,

V volume or mass of DMA in the vial containing the sample of material or article *(see* A.4.5) expressed in Litres or kg, and

M amount of the sample of the material or article expressed in grams.

**A.6.2** The concentration of VC in the material and article under investigation expressed in mg/kg shall be the average of the two concentrations of VC (mg/kg) determined under A.6.1 provided that the repeatability criterion under A.8 is satisfied.

## A.7 Confirmation of the VC level

**A.7.1** In cases where the content of VC in materials and articles as calculated under A.6.1 exceeds the maximum permissible amount the results obtained by the analysis of each of two samples (A.5.1 and A.6.1 ) shall be confirmed in one of three ways:

a) by using at least one other column (A.3.3 ) having a stationary phase with a different polarity. This procedure should continue until a chromatogram is obtained with no evidence of overlap of the VC and/or internal standard peaks with constituents of the sample of the material or article; or

b) by using other detectors, namely, the micro-electrolytic conductivity detector; or

c) by using mass-spectrometry. In this case, if molecular ions with parent masses *(m/e)* of 62 and 64 are found in the ratio of 3:1, it may be regarded with high probability as confirming the presence of VC. In case of doubt the total mass spectrum shall be checked.

## A.8 Repeatability

The difference between the results of two determinations (A.6.1) carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.2 mg VC/kg of material or article.



 

# Annex B
(Normative)

# Sampling of PVC and its copolymers

## B.1 General

**B.1.1** In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

**B.1.2** Samples shall not be taken in an exposed place.

**B.1.3** The sampling instrument, wherever applicable, shall be made of stainless steel or any other suitable material on which the material shall have no action. The instrument shall be clean and dry.

**B.1.4** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B.1.5** The samples shall be placed in a suitable, clean, dry, air-tight metal or glass containers on which the material has no action. The sample containers shall be of such a size that they are almost completely filled by the sample.

**B.1.6** Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, etc.

**B.1.7** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

## B.2 Scale of sampling

**B.2.1 Lot** - In a single consignment all the packages of the same class, same type, same form and belonging to the same batch of manufacture shall be grouped together to constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture or different forms, the packages belonging to the same batch of manufacture and same form shall be grouped together and each such group shall constitute a lot.

The packages may consist of containers of PVC and its copolymers, rolls, films or vials.

**B.2.2** For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested from each lot separately. The number of packages to be sampled shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

These packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in ISO 24153 may be followed.

**Table B1 — Scale of sampling**

| Number of packages in the lot | Sample size |
|---|---|
| Up to 15 | 2 |
| 16 to 50 | 3 |
| 51 to 100 | 4 |
| 101 to 300 | 5 |
| 301 to 500 | 6 |
| 501 to 1000 | 8 |
| 1001 and above | 10 |

NOTE When the number of packages in the lot is less than three, all the packages shall be sampled.

## B.3 Preparation of test samples

**B.3.1** From each of the packages of material selected, small portions of material shall be drawn with the help of a suitable sampling instrument. The total quantity of material collected from each package shall be sufficient to test all the requirements given in 3 of the specification.

B.3.2 In the case of packages consisting of containers, vials, rolls or films, the number of items to be selected from a package for testing each of the requirements given in3 of the specification, shall be one.

## B.4 Number of tests

Tests for determining all the requirements given in clause 4 of the specification shall be carried out on the individual test samples.

## B.5 Criteria for conformity

The lot shall be declared as conforming to the requirements of this specification if all the test results on individual samples meet the relevant specification requirements.



 

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