DRAFT FOR DEVELOPMENT

DD CEN/TS 13130-17:2005

Materials and articles in contact with foodstuffs — Plastics substances subject to limitation —

Part 17: Determination of carbonyl chloride in plastics

ICS 67.250

British Standards

National foreword

This Draft for Development is the official English language version of CEN/TS 13130-17:2005.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature because the method was not evaluated using recognized ring trial procedures. As a consequence there are no reproducibility data for the method. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated 2 years after its publication by the European organization so that a decision can be taken on its status at the end of its 3-year life. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent in writing to the Secretary of BSI Subcommittee CW/47/1, Migration from plastics, at British Standards House, 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled "International Standards Correspondence Index", or by using the "Search" facility of the *BSI Electronic Catalogue* or of British Standards Online.

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Summary of pages

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 17: Determination of carbonyl chloride in plastics

Mat6riaux et objets en contact avec les denizes alimentaires - Substances dans les mati&res plastiques soumises & des limitations - Partie 17: Determination du chlorure de carbonyle dans les mati&res plastiques

Werkstoffe und Gegenstande in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschrankungen unterliegen - Teil 17: Bestimmung von Carbonylchlorid in Kunststoffen

This Technical Specification (CEN/TS) was approved by CEN on 16 December 2004 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Contents

Foreword

This document (CEN/TS 13130-17:2005) has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This part of EN 13130 has been prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, *"Development of Methods of Analysis for Monomers"* and has been prepared by Subcommittee (SC 1) of TC 194 "Utensils in contact with food" as one of a series of test methods for plastics materials and articles in contact with foodstuffs.

This standard is intended to support Directives 2002/72/EC [1], 89/109/EEC [2], 82/711/EEC [3] and its amendments 93/8/EEC [4] and 97/48/EC [5], and 85/572/EEC [6].

At the time of preparation and publication of this part of EN 13130 the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of a test or tests described in this standard.

This part of EN 13130 should be read in conjunction with EN 13130-1.

Further parts of EN 13130, under the general title *Materials and articles in contact with foodstuffs - Plastics substances subject to limitation*, have been prepared, and others are in preparation, concerned with the determination of specific migration from plastics materials into foodstuffs and food simulants and the determination of specific monomers and additives in plastics. The parts of EN 13130 are as follows.

Part 1: *Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants*

Part 2: *Determination of terephthalic acid in food simulants*

Part 3: *Determination of acrylonitrile in food and food simulants*

Part 4: *Determination of 1,3-butadiene in plastics*

Part 5: *Determination ofvinylidene chloride in food simulants*

Part 6: *Determination ofvinylidene chloride in plastics*

Part 7: *Determination of monoethylene glycol and diethylene glycol in food simulants*

Part 8: *Determination of isocyanates in plastics*

Part 9: *Determination of acetic acid, vinyl ester in food simulants*

Part 10: *Determination of acrylamide in food simulants*

Part 11: *Determination of 11-aminoundecanoic acid in food simulants*

Part 12: *Determination of 1,3-benzenedimethanamine in food simulants*

Part 13: *Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants*

Part 14: *Determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline in food simulants*

Part 15: *Determination of 1,3-butadiene in food simulants*

Part 16: *Determination of caprolactam and caprolactam salt in food simulants*

Part 17: *Determination of carbonyl chloride in plastics*

Part 18: *Determination of 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophenone and 4,4'dihydroxybiphenyl in food simulants*

Part 19: *Determination of dimethylaminoethanol in food simulants*

Part 20: *Determination of epichlorohydrin in plastics*

Part 21: *Determination of ethylenediamine and hexamethylenediamine in food simulants*

Part 22: *Determination of ethylene oxide and propylene oxide in plastics*

Part 23: *Determination of formaldehyde and hexamethylenetetramine in food simulants*

Part 24: *Determination of maleic acid and maleic anhydride in food simulants*

Part 25: *Determination of 4-methyl-pentene in food simulants*

Part 26: *Determination of 1-octene and tetrahydrofuran in food simulants*

Part 27: *Determination of 2,4,6-triamino-1,3,5-triazine in food simulants*

Part 28: *Determination of 1,1,1-trimethylolpropane in food simulants*

Parts 1 to 8 are European Standards. Parts 9 to 28 are Technical Specifications.

WARNING All chemicals are hazardous to health to a greater or lesser extent. It is beyond the scope of this Technical Specification to give instructions for the safe handling of all chemicals, that meet, in full, the legal obligations in all countries in which this Technical Specification may be followed. Therefore, specific warnings are not given and users of this Technical Specification should ensure that they meet all the necessary safety requirements in their own country.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

Carbonyl chloride or phosgene, $CCl_{2}0$, PM/Ref. No 14380, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual carbonyl chloride can remain in the polymer and may migrate into foodstuffs coming into contact with the plastics material or article.

The method has been pre-validated by a collaborative trial with two laboratories.

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of carbonyl chloride monomer in polymers. The method is applicable to polycarbonate as well as to other polymers and copolymers where these are soluble in methylene chloride. The level of carbonyl chloride monomer determined is expressed as milligrams of carbonyl chloride per kilogram of polymer. The method is appropriate for the quantitative determination of carbonyl chloride at a minimum level of 0,3 mg per kilogram of polymer.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 13130-1:2004, *Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 1: Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants.*

3 Principle

The level of carbonyl chloride in the polymer is determined by dissolution of the polymer and concurrent derivatization with 2-aminophenol. The resulting 2-benzoxazolinone is analyzed by high performance liquid chromatography (HPLC) with ultra violet (UV) detection. Quantification is achieved by standard addition of carbonyl chloride to the test polymer. Confirmation of the identity of carbonyl chloride is carried out by diode array detection.

4 Reagents

NOTE 1 Carbonyl chloride reacts rapidly with moisture. Suitable precautions should be taken to protect carbonyl chloride solutions from moisture. The carbonyl chloride derivative is not sensitive to moisture.

NOTE 2 All reagents should be of recognized analytical quality unless otherwise stated.

4.1 Analytes

4.1.1 Carbonyl chloride, CCl_20, molecular weight, 98,9, commercially available as a 20 % (w/w) solution in toluene, density at 20 °C 0,935 kg per litre, corresponding to a concentration of 1,93 mole per litre or 191 g per litre.

Store the commercial carbonyl chloride solution in a closed container for up to 6 months at + 1 °C in the dark.

4.1.2 2-benzoxazolinone, $C_7H_5N0_{2l}$ molecular weight: 135,1, purity 98 %.

4.2 Chemicals

4.2.1 2-aminophenol (2-hydroxyanilin), C_6H_7NO, molecular weight: 109,1, purity 98 %.

4.2.2 Dichloromethane (DCM), dried over a bed of molecular sieve (5A), H_20 < 0,005 %, purity > 99,5 %.

4.2.3 **Toluene, dried over a bed of molecular sieve (5A),** H_20 < 0,005 %, purity > 99,5 %.

NOTE The water content of the toluene should be as low as possible. Toluene dried over a molecular sieve can be obtained from chemical suppliers. Alternatively, toluene can be dried before use, in which case the molecular sieve should be activated for maximum water absorption.

4.2.4 Methanol

4.2.5 Acetone

4.2.6 Acetonitrile, purity > 99,5 %, HPLC grade

4.2.7 Distilled water, HPLC grade

4.2.8 Sodium dihydrogen orthophosphate, $NaH_2P0_4 . 2H_20$

4.2.9 Glacial acetic acid

4.2.10 Hydrochloric acid, 1 N

4.2.11 Sodium sulfate, anhydrous

4.2.12 Mobile phase for high performance liquid chromatography, prepared as follows.

Dissolve 7,5 g of sodium dihydrogen orthophosphate (4.2.8) in 700 ml of water (4.2.7), add 250 ml acetonitrile (4.2.6) using a measuring cylinder and adjust to pH (3,6 ± 0,1) with glacial acetic acid (4.2.9).

NOTE 1 Approximately 5 ml should be sufficient.

Make up to 1 l with water (4.2.7).

NOTE 2 Degassing the mobile phase may be necessary with some HPLC equipment.

4.3 Solutions

4.3.1 Standard solution of carbonyl chloride in toluene, 95,5 mg/l

Pipette into a 20 ml vial (5.3) 20,0 ± 0,1ml of toluene (4.2.3) and close with the septum and cap. Using a 10 pi syringe (5.5), add 10 pi of the 20 % carbonyl chloride solution (4.1.1) by injection through the septum and dispensing after the needle has immersed into the toluene. Remove the syringe and mix.

NOTE 1 The exact concentration of this standard solution is 95,5 mg/l.

Repeat the procedure to provide a second standard solution.

NOTE 2 This standard solution can be stored at + 4 °C for up to 4 weeks in the dark.

4.3.2 Derivatization reagent solution, 500 pg/ml

Accurately weigh 25 mg ± 0,2 mg of 2-aminophenol (4.2.1) into a 50 ml volumetric flask and add approximately 45 ml dichloromethane (4.2.2). Close and dissolve the derivatization reagent using an ultrasonic vibration bath at room temperature. Finally make up to the mark with dichloromethane (4.2.2), mix and close.

Prepare fresh derivatization reagent solution on the day when required.

4.3.3 Preparation of 2-benzoxazolinone standards in acetonitrile at defined concentrations between 0,4 pg per millilitre and 7 pg per millilitre

NOTE 1 Preparation and use of benzoxazolinone standards is optional, but are advised when the method is used for the first time and when problems are experienced in the HPLC determination or the derivatization procedure.

NOTE 2 The standards of the carbonyl chloride derivative are particularly useful to establish the analytical system and to check linearity of detector response as well as for the recovery check (7.4.3).

NOTE 3 Benzoxazolinone concentrations can be converted into carbonyl chloride equivalent concentrations by multiplying by 0,732.

Weigh 22 mg + 2 mg to an accuracy of 0,1 mg into a 10 ml volumetric flask and make up to the mark with acetonitrile (4.2.6). Close and mix to obtain a standard stock solution of approx 2,2 mg per millilitre.

Pipette into each of five 10 ml vials 10,0 ml acetonitrile (4.2.6). Add by micro-syringe 2 pi, 6 pi, 10 pi, 20 pi and 30 pi of the 2,2 mg per millilitre benzoxazolinone stock solution, close and mix. The resulting standard solutions contain approximately 0,44 pg, 1.32 pg, 2,20 pg, 4,40 pg and 6,60 pg benzoxazolinone per millilitre solution corresponding to 0,32 pg/ml, 0,97 pg/ml, 1,61 pg/ml, 3,22 pg/ml and 4.83 pg carbonyl chloride equivalents per millilitre of solution.

Calculate the exact concentrations of carbonyl chloride equivalents in milligrams per litre of solution.

NOTE 4 These standard solutions can be stored for maximum 8 weeks at temperatures up to + 10 °C in the dark.

5 Apparatus

NOTE An instrument or item of apparatus is listed only where it is special, or made to a particular specification, usual laboratory glassware and equipment being assumed to be available.

5.1 High performance liquid chromatograph with a 10 pi injection loop, and a variable wavelength UV detector, set to 270 nm, connected to a strip chart recorder or integrator.

5.2 HPLC column, capable of fully resolving benzoxazolinone from 2-aminophenol and polymer constituents such that the peak of benzoxazolinone does not overlap by more than 1 % peak area with the peak of 2-aminophenol and with interferences arising from the polymer and from injection media.

Appropriate operating conditions shall be established for the specific equipment used for the determination.

NOTE The following column has been found to be suitable:

250 mm x 4,6 mm I.D. octadecylsilane, 10 % carbon loading with Hypersil 5 pm connected to a pre-column of the dimensions 20 mm x 4,6 mm, packed with the same stationary phase.

The following operating conditions were found to be suitable for this column:

Injector:	10 pi total loop fill
Mobile phase:	25/75 acetonitrile/water, buffered to pH=3,6 (4.2.12)
Flow rate :	1 ml per minute
Temperature:	ambient
Detector (UV)	270 nm

With other reverse phase columns, adjustments can be made to the acetonitrile composition of the mobile phase to give acceptable retention times and resolution.

5.3 Glass vials, 10 ml, 20 ml and 50 ml capacity with polytetrafluoroethylne-faced butyl rubber septa and crimp-closure.

The vials shall be dried by baking at 105 °C and subsequent storage in a desiccator until required for use.

5.4 HPLC sample vials

5.5 Microsyringes, 10 pi, 50 |j|, 100 pi and 250 pi.

5.6 Orbital shaking device

6 Samples

NOTE 1 The following precautions are advisable:

a) to avoid cross contamination, carry out preparation of the polymer samples in an area remote to that used for handling carbonyl chloride and benzoxazolinone;

b) to avoid loss of carbonyl chloride through hydrolysis, carry out preparation of the polymer and calibration samples in an area of low relative humidity and away from sources of moisture;

c) ensure that all glassware and syringes are dry before use.

NOTE 2 Samples can be kept refrigerated at + 4 °C in closed containers with the exclusion of light for up to four weeks.

6.1 Preparation of test samples

6.1.1 Derivatization reaction of aminophenol with carbonyl chloride

Ensure that the given derivatization reaction time is not exceeded, (see also NOTE in 6.1.2).

Weigh into a 50 ml glass vial 1,00 g ± 0,01 g of the polymer, add 10,0 ml dichloromethane (4.2.2) and 2,5 ml of the derivatization reagent solution (4.3.2) and close immediately. Shake the sealed vial on an orbital shaker until the polymer has been dissolved (this takes approximately 10 min to 15 min), and then continue shaking for further 30 min.

6.1.2 Protonation and extraction of excessive aminophenol

NOTE The derivatization agent aminophenol is capable of reacting not only with carbonyl chloride, but also, at a much slower rate, with oligomers or the polymer. Therefore, it is necessary to remove excessive aminophenol.

Open the vial and add, by pipette, 5 ml acetone (4.2.5) and 5 ml 1N HCl (4.2.10) and close the vial again. Shake vigorously for one minute, let the phases separate, open the vial and withdraw by pipette, as quantitatively as possible, the upper HCl layer, approximately 3 ml to 4 ml, which contains the excess aminophenol.

6.1.3 Precipitation of dissolved polymer and further work-up procedure

Open the vial again and add slowly 10 ml methanol (4.2.4) under gentle manual shaking, close the vial again and shake for 15 min on an orbital shaker to precipitate the dissolved polymer. Open the vial and filter the mixture quantitatively into a 100 ml flask with the aid of approximately 5 ml methanol as a washing solvent. Concentrate the solution obtained by gentle warming in a water bath at 40 °C, to a volume of approximately 5 ml by evaporation with a rotavapor, firstly removing dichloromethane at a pressure of approximately 850 mbar and secondly removing methanol at approximately 330 mbar.

Avoid splashing in the rotavapor and reduce slowly the pressure to remove methanol. Addition of some anti bumping granules may facilitate evaporation of the solutions.

Remove precipitated solids from the flask wall by ultrasonic vibration for a few minutes. Filter the solution, to remove the precipitated polymer, into a 25 ml pointed flask using approximately 5 ml methanol as a washing solvent. Concentrate the filtered solution carefully to approximately 1 ml to 2 ml by evaporation with a rotavapor under the conditions mentioned above to obtain an aqueous residue. Extract this residue twice each time with 4 ml dichloromethane and dry the combined dichloromethane extracts over anhydrous sodium sulfate (4.2.11). Filter the dichloromethane solutions from the sodium sulfate using a few millilitres of dichloromethane to wash the sodium sulfate and filter. Evaporate the dichloromethane solution completely to dryness at room temperature using a nitrogen stream. If necessary, i.e. if an aqueous residue remains, repeat the dichloromethane extraction step. Re-dissolve the remaining residue in 1 ml acetonitrile (4.2.6), if necessary, with the aid of a short ultrasonic vibration, and transfer the solution into a sample vial for HPLC injection (5.4).

If further precipitation of dissolved polymer occurs in the final acetonitrile solution, let the precipitate settle and take an aliquot of the solution for HPLC injection.

6.2 Preparation of blank samples

6.2.1 Reagent blank sample

Prepare a sample as in 6.1, but omit the addition of the polymer sample.

6.2.2 Polymer blank sample

Prepare a sample as in 6.1, but omit the addition of the derivatization reagent solution (4.3.2).

6.3 Preparation of calibration samples (standard addition)

Repeat the procedure described in 6.1 four times, but add also 5 pi, 10 pi, 20 pi and 40 pi of the carbonyl chloride standard solution (4.3.1) to the polymer sample immediately after the addition of the derivatization reagent solution (4.3.2).

NOTE The calibration samples obtained in this way are equivalent to standard addition concentrations of 0,48 mg/kg, 0,96 mg/kg, 1,92 mg/kg and 3,84 mg carbonyl chloride per kilogram polymer.

Repeat the procedure using the second standard solution (4.3.1).

7 Procedure

7.1 HPLC analysis

Each sample shall be determined at least in duplicate.

Examine the baseline stability and response linearity of the detector when starting GC headspace measurements.

Maintain the same headspace GC operating conditions throughout the measurement of all samples and calibration solutions.

NOTE 1 Optimization of the analytical system can be achieved using the carbonyl chloride derivative standards prepared under 4.3.3.

NOTE 2 Under the conditions given in 5.2, 2-aminophenol and the carbonyl chloride derivative were found to have retention times of 4,0 and 8,0 min, respectively.

7.2 Sample treatment

Inject the samples prepared as described in 6.1 to 6.3, under the conditions given in 5.2, applying duplicate injections. Identify the benzoxazolinone peak on a retention time basis and measure the peak heights or peak areas (PA).

NOTE For the purpose of identification, recovery and detection limit, analysis of the solutions prepared in 4.3.3 may be useful.

7.3 Calibration

From measurements of test samples (6.1) and calibration samples (6.3), establish a standard addition curve. This calibration curve shall be obtained by plotting peak height or area of the carbonyl chloride derivative peak against the spiked concentration values of carbonyl chloride in milligrams of the polymer in kilograms.

The calibration curves shall be rectilinear and the correlation coefficient shall be 0,996 or better.

The two sets of calibrant solutions made from independently prepared stock solutions shall be cross-checked by generating two calibration curves which on the basis of peak area measurement shall agree to ± 5 % of one another.

7.4 Evaluation of data

NOTE The following calculations assume that for all measurements exactly the same mass of polymer sample, 1,0 g, and the same volume of acetonitrile, 1,0 ml, used to re-dissolve the final residue for HPLC-analysis, has been applied.

7.4.1 HPLC interferences

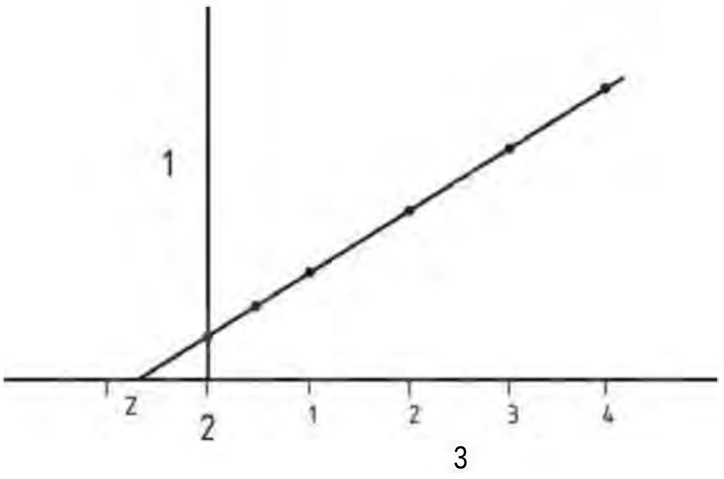
If the polymer blank sample (6.2.2) shows a peak interfering with the benzoxazolinone peak, the peak area of the interference shall be measured and used for correction of the standard addition curve values, i.e. for subtraction from the test sample and the calibration sample values.

NOTE So far no interferences have been observed when following this method.

7.4.2 Determination of carbonyl chloride in the test samples

7.4.2.1 Graphical determination

Read the carbonyl chloride concentration of the test sample (6.1) from the calibration graph (7.3) by back extrapolation to the x-axis where the magnitude of the intercept Z is equal to the carbonyl chloride concentration (see below).



Key

1 UV absorbance

2 Sample

3 mg carbonyl chloride added per kg of polymer

Figure 1 — Typical calibration graph

7.4.2.2 Calculation from the regression parameters

Calculate the regression line of the calibration graph including the sample value from the formula:

y —(axx)+b

where

y	is the peak height or area of the carbonyl chloride derivative;
a	is the slope of the regression line in kilograms per milligram;
x	is the concentration of carbonyl chloride in the polymer in milligrams per kilogram;
b	is the intercept of the regression line.

The residual carbonyl chloride concentration, C_{c}cp»^{is} obtained from the regression parameters a and b according to the following formula:

$$C_{^CCP} = -\frac{b}{a}$$

where

CQCP is the concentration of carbonyl chloride in the polymer in milligrams per kilogram.

Both procedures yield directly the carbonyl chloride concentration in the test material of milligrams of carbonyl chloride per kilogram of polymer.

The method applying calculation from the regression parameters is the preferred method.

An optional recovery and detection limit check may be carried out as follows:

From measurements of the benzoxazolinone standards (4.3.3), establish a standard addition curve. This calibration curve shall be obtained by plotting peak height or area of the benzoxazolinone peak against the concentration values given in carbonyl chloride equivalents in milligrams per kilogram of the polymer.

Calculate the regression line of the calibration graph from the following formula:

y = {a xx) +*b*

where

y	is the peak height or area of the benzoxazolinone;
a'	is the slope of the regression line in kilograms per milligram;
x	is the concentration of carbonyl chloride equivalents in the polymer in milligrams per kilogram;
b'	is the intercept of the regression line.

The percentage recovery is then given by the following formula:

%re co*very* = -^-X 100
a

where

a is the slope obtained in 7.4.2;

a' is the slope.

NOTE The recovery should be higher than 70 %. If not, consider whether or not the values obtained are valid. It is advisable to start the determination again from the beginning.

The requirement for the detection limit can be considered as being fulfilled if the standard with the lowest concentration, approximately 0,32 pg carbonyl chloride equivalents per millilitre of solution, provides a satisfactory peak with higher signal to noise ratio than 10:1.

8 Precision

8.1 Validation

This method was pre-validated by a collaborative trial with two laboratories. In each laboratory a within-laboratory precision experiment was carried out by spiking a polycarbonate sample at the restriction criterion.

8.2 Repeatability

Evaluation of the within-laboratory precision experiment results, in accordance with ISO 5725, at a concentration of 1,0 mg/kg, yielded the following performance characteristics at the 95 % probability level (lab 1/lab 2):

Repeatability r = 0,23/0,32 mg carbonyl chloride per kilogram of polymer.

8.3 Detection limit

The within-laboratory detection limit (WDL), based on the calibration curve method according to DIN 32645, was found to be equivalent to 0,3 pg carbonyl chloride/g of polymer. Thus the method is capable of quantitative detection at a minimum level of 0,3 mg carbonyl chloride per kilogram polymer.

NOTE Detector and pump design, injection loop size, etc., can influence the detection limit values significantly.

9 Confirmation

9.1 Requirement for confirmation

If the residual concentration of carbonyl chloride in the polymer, as determined in accordance with 6.4.2, exceeds the restriction, e.g. a compositional limit of 1,0 mg/kg of polymer, the determination shall be confirmed by the method described in 9.2.

The confirmation is qualitative in the sense that it should demonstrate the correct identity of the measured analyte and the absence of interferences. For the purposes of quantification the result as calculated in accordance with 7.4 shall be taken as the true value.

9.2 Confirmation by diode array detection

By use of diode array detection, record the spectral profiles of the samples, blanks and calibration samples over the range of 200 nm to 320 nm at the front, apex and tail of the peak identified as the carbonyl chloride derivative. The 2-benzoxazolinone can be identified as having an absorbance peak maximum at 272 nm and a minimum at 245 nm with an absorbance ratio of 40 at 260 nm, 100 at 272 nm, and 70 at 280 nm.

NOTE If the peak is pure, the overlaid spectral profiles of the front, apex and tail of the peak should be identical. Therefore, if the three profiles are normalized, they should superimpose on top of each other.

10 Test report

The test report shall include at least the following, where applicable:

a) identification of the sample;

b) name of the laboratory;

c) name of responsible analyst;

d) date of report;

e) date of analysis;

f) analyte;

g) a reference to this method;

h) sample details, such as:

  1) type of material or article;

  2) origin and denotation of the sample;

  3) date and method of obtaining the laboratory sample;

  4) storage conditions;

i) results expressed in milligrams of carbonyl chloride per kilogram of polymer and reported as the average value from two or more determinations satisfying the repeatability criterion of 8.2;

j) details of confirmation procedure, if any;

k) reasons for modifications introduced into the test method, if any.

Bibliography

[1] Commission of the European Communities, Commission Directive of 23 February 1990 relating to plastics materials and articles intended to come into contact with foodstuffs (90/128/EEC), Official Journal of the European Communities, 13 December 1990, no. L349, p26. Corrigendum of the previous publication, Official Journal of the European Communities, 21 March 1990, no. 75. p19.

[2] Commission of the European Communities, Council Directive of 21 December 1988 on the approximation of the laws of the Member States relating to materials and articles intended to come into contact with foodstuff (89/109/EEC), Official Journal of the European Communities, 11 February 1989, no. L 40, p 38.

[3] Commission of the European Communities, Council Directive of 18 October 1982 laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (82/711/EEC), Official Journal of the European Communities, 23 October 1982, no. L 297, p 26.

[4] Commission of the European Communities, Commission Directive of 15 March 1993 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (93/8/EEC), Official Journal of the European Communities, 14 April 1993, no. L 90, p 22.

[5] Commission of the European Communities, Commission Directive of 97/48/EC of 29 July 1997 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs, Official Journal of the European Communities, 12 August 1997, no. L 222, p 10.

[6] Commission of the European Communities, Council Directive of 19 December 1985 laying down the list of simulants to be used for testing migration of constituents of plastics materials and articles intended to come into contact with foodstuffs (85/572/EEC), Official Journal of the European Communities, 31 December 1985, no. L372, p14.

[7] ISO 5725, *Accuracy (trueness and precision) of measurement methods and result.*

[8] DIN 32645, *Chemical analysis; decision limit; detection limit and determination limit; estimation in case of repeatability; terms, methods, evaluation.*

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