
International Standard



5498

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Agricultural food products — Determination of crude fibre content — General method

Produits agricoles alimentaires — Détermination de l'indice d'insoluble dit «cellulosique» — Méthode générale

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	France	Netherlands
Austria	Germany, F.R.	Poland
Brazil	Hungary	Portugal
Bulgaria	India	Romania
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

Agricultural food products — Determination of crude fibre content — General method

0 Introduction

There are numerous methods for the determination of the crude fibre content of agricultural food products and, for a given method, numerous variants are used according to the products analysed, or, for the same product, from laboratory to laboratory.

This International Standard therefore fulfils a need for harmonization. It specifies a method of general application based on the Weende method, which is the most commonly used, and basically involves acid treatment followed by alkaline treatment.

Studies carried out have demonstrated the need to specify precisely the conditions for the acid and alkaline treatments and to describe the various procedures used for separating the insoluble matter. These procedures are described in annex B.

1 Scope

This International Standard specifies a conventional method for the determination of the crude fibre content of agricultural food products.

2 Field of application

The method is intended for general application; however, it may be necessary, in certain individual cases, to choose a more appropriate method, particularly in the case of yeasts and products containing less than 1 % of crude fibre for which the method described in ISO 6541 shall be used.

3 Reference

ISO 6541, *Agricultural food products — Determination of crude fibre content — Modified Scharrer method.*

4 Definition

crude fibre content: Conventionally, the whole of the substances which are insoluble and combustible under the operating conditions described in this International Standard.

The crude fibre content is expressed as a percentage by mass, referred either to the product as received or to the dry matter content of the product.

NOTE — In French, the term “indice d’insoluble dit cellulosique” has been adopted for “crude fibre” rather than the alternative “cellulose brute”.

5 Principle

After any necessary grinding and defatting, boiling with sulphuric acid solution of standard concentration, and separation and washing of the insoluble residue.

Boiling this residue with sodium hydroxide solution of standard concentration, then separation, washing, drying and weighing of the insoluble residue, and determination of the loss in mass on incineration.

6 Reagents and materials

All reagents shall be of recognized analytical quality and the water used shall be distilled water or water of at least equivalent purity.

6.1 Sulphuric acid, standard volumetric solution, $c(1/2 \text{ H}_2\text{SO}_4) = 0,255 \pm 0,005 \text{ mol/l}$ (corresponding to 12,5 g of sulphuric acid per litre of solution).

6.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,313 \pm 0,005 \text{ mol/l}$ (corresponding to 12,5 g of sodium hydroxide per litre of solution).

This solution shall be as free as possible from carbonates.

6.3 Acetone, or 95 % (V/V) **ethanol**, or **methanol**, or **propan-2-ol**.

6.4 Extraction solvent :

Technical grade *n*-hexane, or light petroleum (having a boiling range between 40 and 60 °C), or diethyl ether, or another solvent or mixture of solvents more suitable for the extraction of fatty substances from certain products to be analysed.

6.5 Hydrochloric acid, 0,5 mol/l solution (in the case of products rich in carbonates). (See 11.1.)

6.6 Filter aid (in cases where the separation techniques described in clauses B.2 or B.3 of annex B are used).

6.7 Anti-foam agent, if necessary, known to have no effect on the results.

6.8 Anti-bumping agent, if necessary, resistant to attack under the test conditions or known to have no effect on the results.

7 Apparatus

Usual laboratory apparatus, and in particular

7.1 Grinding device, easy to clean, suited to the nature of the product and allowing grinding of the product without causing undue heating or significant change in the moisture content.

7.2 Sieve, of metal wire cloth, aperture size 1 mm, complying with the requirements of ISO 3310/1.

7.3 Drying oven, capable of being controlled at 130 ± 2 °C.

7.4 Wide-mouthed vessel, provided with a condenser, for example a flask having a minimum capacity of 600 ml fitted with a reflux condenser, or a beaker without spout, of capacity 600 ml, covered by a round-bottom flask of 500 ml capacity containing 450 ml of cold water.

7.5 Heating device, for example an electrically heated hot-plate fitted with a magnetic stirrer, capable of maintaining 200 ml of reagents 6.1 and 6.2 at a gentle boil.

7.6 Incineration dish, of capacity 25 to 50 ml, resistant to attack under the test conditions, or a **filter crucible**, suitable for separation and incineration of the residue.

7.7 Muffle furnace, provided with air circulation and temperature control, suitable for carrying out incineration at 550 ± 25 °C.

7.8 Desiccator, containing an efficient desiccant.

7.9 Separating device.

Various types of separating devices are described in annex B.

7.10 Analytical balance.

8 Sampling

Refer to the International Standard appropriate to the product concerned.

9 Procedure

9.1 Preparation of test sample

9.1.1 Preliminary drying

In the case of products having moisture contents too high for them to be mixed or ground as received, carry out a preliminary drying of the product at an appropriate temperature. In this case, weigh the product before the preliminary drying and again just before preparation of the test sample (9.1.2 or 9.1.3).

9.1.2 Products not requiring grinding

9.1.2.1 Products that pass through the sieve (7.2) without leaving a residue do not need to be ground before the determination.

Mix well before taking the test portion.

9.1.2.2 If the results are to be expressed relative to the dry matter content, determine beforehand the dry matter content of the test sample (9.1.2.1) by an appropriate method.

9.1.3 Products requiring grinding

Products that do not pass through the test sieve (7.2) without leaving a residue shall be ground.

9.1.3.1 If the results are to be expressed relative to the product as received, determine beforehand the dry matter content of the sample by an appropriate method.

9.1.3.2 Grind the laboratory sample in the grinding device (7.1) so that the product passes through the sieve (7.2) without leaving a residue.

9.1.3.3 Determine the dry matter content of the test sample (9.1.3.2) by an appropriate method.

9.2 Test portion

NOTE — If it is necessary to remove fatty matter (see 9.3.1) and the technique described in A.1.3 of annex A is to be used, the extraction shall be carried out before the test portion is taken.

Weigh, to the nearest 1 mg, about 3 g of the test sample (except in special cases) which has been prepared as described in 9.1, and which is presumed to contain more than 1 % of crude fibre.

9.3 Determination

9.3.1 Extraction of fatty substances (see annex A)

If the fatty matter content is less than 1 %, extraction of fatty matter is unnecessary.

Extraction is not absolutely essential, but is, nevertheless, recommended if the fatty matter content is between 1 and 10 %.

If the fatty matter content is more than 10 %, prior extraction is essential.

For products containing fatty substances which cannot be removed directly, the extraction shall be carried out after the acid treatment (see A.1.5 of annex A).

9.3.2 Acid treatment

9.3.2.1 Transfer the test portion, which may have had the fat and oils (see 9.3.1) and carbonates (see 11.1) removed, into the vessel (7.4). Add, if appropriate, the prescribed quantity of filter aid (6.6) (in the case of the variants described in clauses B.2 and B.3 of annex B) and, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).

Measure 200 ml of the sulphuric acid solution (6.1) at room temperature, bring it to a temperature of 95 to 100 °C (see 11.2) and add it to the contents of the vessel (7.4).

Fit the condenser. Bring rapidly to the boil (in about 2 min) using the heating device (7.5) and continue boiling gently for 30 ± 1 min. Swirl the vessel from time to time so that any particles adhering to the interior wall are returned to the solution.

9.3.2.2 After the specified boiling period, add about 50 ml of cold water and separate rapidly the insoluble residue using the separating device (7.9) chosen. Wash the vessel (7.4) with 50 ml portions of hot water (temperature 95 to 100 °C) and pour the washings over the insoluble residue remaining in the separating device.

Repeat the washing of the insoluble residue until the filtrate is substantially neutral to litmus paper. The separation and washing of the insoluble residue shall be completed in less than 30 min.

9.3.3 Alkaline treatment

9.3.3.1 Return the washed insoluble residue to the vessel (7.4) and add, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).

Measure 200 ml of the sodium hydroxide solution (6.2) at room temperature, bring it to a temperature of 95 to 100 °C (see 11.2), and add it to the contents of the vessel (7.4) in small portions.

Fit the condenser. Bring rapidly to the boil (in about 2 min) using the heating device (7.5) and continue to boil gently for 30 ± 1 min.

9.3.3.2 After the specified boiling period, add about 50 ml of cold water and rapidly separate the insoluble residue using the separating device (7.9) chosen. Wash the residue with 25 ml of the sulphuric acid solution (6.1) measured at room temperature and then raised to a temperature of 95 to 100 °C (see 11.2). Wash with water as described in 9.3.2.2. Dry the residue with one of the reagents (6.3); wash with solvent (6.4) to remove unsaponifiable fatty matter.

According to the filtration technique chosen (see annex B), collect all the residue in the incineration dish (7.6) or in the filter crucible (7.6).

9.3.4 Drying

Dry the incineration dish or the filter crucible with its contents in the oven (7.3) (see the note) at 130 ± 2 °C.

Allow to cool to room temperature in the desiccator (7.8) and quickly weigh to the nearest 0,5 mg.

Repeat these operations until the difference between two successive weighings, following drying in the oven and cooling in the desiccator, does not exceed 1 mg.

NOTE — The length of the periods in the oven depends on the methods of separation used. A total drying period of 2 h is usually sufficient.

9.3.5 Incineration

After drying, incinerate the dry residue in the muffle furnace (7.7) at 550 ± 25 °C to constant mass. Allow to cool to room temperature in the desiccator (7.8) and weigh again to the nearest 0,5 mg.

9.3.6 Number of determinations

Carry out at least two determinations on the same test sample.

9.4 Blank test

If asbestos is used as a filter aid (see clause B.2 of annex B), carry out a blank test under the same conditions as described in 9.3.

10 Expression of results

10.1 Method of calculation and formulae

10.1.1 Crude fibre content relative to product as received

The crude fibre content, expressed as a percentage by mass relative to the product as received, is given by the formula

a) *for products not requiring grinding*¹⁾

$$[m_1 - (m_2 + m_3)] \times \frac{100}{m_0}$$

b) *for products requiring grinding*¹⁾

$$[m_1 - (m_2 + m_3)] \times \frac{100}{m_0} \times \frac{100}{M'_S} \times \frac{M_S}{100}$$

where

m_0 is the mass, in grams, of the test portion (9.2);

1) If there was no blank test, delete m_3 from the formula.

m_1 is the total mass, in grams, of the dry residue and its support after drying (9.3.4);

m_2 is the total mass, in grams, of the dry residue and its support after incineration (9.3.5);

m_3 is the difference in mass, in grams, observed during the incineration process in the blank test (9.4), taking into account the quantity of filter aid used;

M'_S is the dry matter content, expressed as a percentage by mass, of the product as received, determined as indicated in 9.1.3.1;

M_S is the dry matter content, expressed as a percentage by mass, of the test sample, determined as indicated in 9.1.2.2 or 9.1.3.3.

10.1.2 Crude fibre content relative to the dry matter content of the product

The crude fibre content, expressed as a percentage by mass relative to the dry matter content of the product, is given by the formula¹⁾

$$[m_1 - (m_2 + m_3)] \times \frac{100}{m_0} \times \frac{100}{M'_S}$$

where m_0 , m_1 , m_2 , m_3 and M'_S have the same meanings as in 10.1.1.

10.1.3 Case of preliminary drying

If a preliminary drying was carried out (see 9.1.1), the crude fibre content, expressed as a percentage by mass relative to the product as received, is obtained by multiplying the result calculated according to 10.1.1 by the ratio

$$\frac{m_5}{m_4}$$

where

m_4 is the mass, in grams, of the initial moist sample before preliminary drying;

m_5 is the mass, in grams, of the same sample after the preliminary drying.

10.1.4 Result

Take as the result the arithmetic mean of the two determinations (9.3.6), provided that the requirements for repeatability (see 10.2) are satisfied.

10.2 Repeatability

The difference between the results of the two determinations (9.3.6), carried out simultaneously or in rapid succession by the same analyst, shall not exceed :

0,4 (absolute value) for a crude fibre content less than 10 % (m/m);

4 % (relative value) for a crude fibre content greater than 10 % (m/m).

11 Notes on procedure

11.1 If the sample has a carbonate (CO_3^{2-}) content greater than 1 %, add to the test portion 100 ml of the hydrochloric acid solution (6.5) at room temperature. After 5 min, filter and wash the sample with water at room temperature. Proceed as described in 9.3.2.

Without this pre-treatment, 1 % of carbonate would reduce the concentration of the sulphuric acid.

11.2 Precautions are necessary to avoid any change in the concentration of reagents 6.1 and 6.2 whilst heating them to a temperature between 95 and 100 °C, for example by using a flask fitted with a reflux condenser.

12 Test report

The test report shall show the method used and the result obtained (in particular whether it is expressed relative to the product as received or to the dry matter content). It shall also mention the separating device used and all operating conditions not specified in this International Standard or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all the details required for the complete identification of the sample.

1) If there was no blank test, delete m_3 from the formula.

Annex A

Methods of extraction of fatty substances

A.0 Introduction

If it is necessary to extract the fatty substances (see 9.3.1), use as the test portion the residue of a direct determination of the fatty matter content of the product or extract the fatty substances from the test portion using a solvent (6.4) using one of the techniques described below.

In all procedures, the extraction of the fatty substances shall be carried out in the absence of any material capable of introducing cellulosic fibres (paper, absorbent cotton) or of causing loss of fine particles when decanting.

A.1 Methods of extraction

A.1.1 Carry out one or more washings of the test portion directly in the vessel used for treatment, followed by removal of the solvent by decantation without loss of the product. After washing, dry the receiver containing the defatted material in order to remove the rest of the solvent.

A.1.2 Carry out a continuous extraction for 1 or 2 h, transferring the test portion to a suitable apparatus, for example a continuous extraction apparatus.

A.1.3 Carry out an extraction on a known amount of the test sample substantially larger than the test portion needed. After extraction, dry the residue and remove an aliquot portion corresponding to the mass of the test portion for the determination of the crude fibre content. When expressing the result, take into account the percentage of the fatty substances extracted and of the moisture removed.

A.1.4 In the case of the variant described in B.2.2 of annex B, proceed as follows.

Carry out an extraction by placing the test portion on the filter plate of the separating device (7.9) previously filled with 2 g of asbestos (6.6) weighed to the nearest 1 mg. Cover three times with 50 ml of solvent (6.4) and apply suction each time with care.

A.1.5 Products containing fatty substances which are not directly extractable can be treated by the above procedures only after acid hydrolysis.

After having eliminated the sulphuric acid by washing (see 9.3.2.2), dry the residue three times, using a total volume of 100 ml of one of the reagents (6.3), then extract the fatty substances three times with 50 ml of the solvent (6.4).

In this case, the method of separation described in clause B.1 of annex B cannot be used.

Annex B

Various devices for the separation of crude fibre and supplementary information on the procedures for their use

B.0 Introduction

When using the general method described in this International Standard, the separating device (7.9) and the corresponding procedure differ according to the separation technique used.

These techniques, given as examples, include

- separation by centrifuging;
- separation by filtration with or without filter aid (asbestos, sea sand, filter cloth, filter paper).

When using one of these methods, follow the text of the general method but include the following modifications.

B.1 Separation by centrifuging

Clause 6

Add :

“**6.9 Bromothymol blue**, 0,2 g/l indicator solution.”

Clause 7

Replace the text of 7.6 by :

“**Filter crucible**, of material resistant to attack under the test conditions.”

Replace the text of 7.9 by :

“**Centrifuge**.”

Clause 9

Replace the text of 9.3.2.2 by :

“After the specified boiling period, separate the condenser from the vessel and transfer the contents of the vessel to the tube(s) of the centrifuge (7.9) while retaining as large a part as possible of the insoluble residue in the vessel.

Centrifuge the tube(s) immediately until the liquid is completely clear. Remove the greater part of the supernatant liquid by decantation, and neutralize the remainder with the sodium hydroxide solution (6.2), using 5 drops of the bromothymol blue solution (6.9) as indicator, until the colour changes from yellow to green.

Rinse the vessel with hot water (temperature between 95 and 100 °C) and neutralize the washings as described above.

Transfer the washings to the centrifuge tube(s) and, if necessary, bring to pH 7, checking with indicator paper.

Centrifuge again until the liquid is completely clear.”

Replace the text of 9.3.3.1 by :

“Add, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8) to the vessel (7.4). Remove the major part of the supernatant liquid from the centrifuge tube(s) and transfer the residue quantitatively to the vessel, extracting it from the centrifuge tube(s) with 200 ml of the sodium hydroxide solution (6.2) measured at room temperature and raised to a temperature between 95 and 100 °C (see 11.2) and added in successive portions.

Fit the condenser to the vessel. Bring rapidly to the boil (in about 2 min) using the heating device (7.5) and maintain gently boiling for 30 ± 1 min.”

Replace the text of 9.3.3.2 by :

“After the specified boiling period, separate the condenser from the vessel and transfer the contents to the tube(s) of the centrifuge (7.9) taking care to transfer as much of the insoluble residue as possible.

Centrifuge the tube(s) immediately until the liquid is completely clear. Remove the greater part of the supernatant liquid by decantation and neutralize the remainder with the sulphuric acid solution (6.1) using 5 drops of the bromothymol blue solution (6.9) as indicator, until the colour changes from yellow to green. If necessary, bring to pH 7, checking with indicator paper.

Rinse the vessel with hot water (temperature between 95 and 100 °C) and neutralize the washings as described above.

Quantitatively transfer the residue in the vessel and in the tube(s) to the filter crucible (7.6), previously mounted on a suction flask, using a wash bottle with hot water.

Wash the residue with boiling water and then dry using one of the reagents (6.3).”

B.2 Separation by filtration through asbestos

WARNING — Asbestos should be handled with care as it can be injurious to health.

B.2.1 First variant

Clause 6

Replace the text of 6.6 by :

“**Asbestos**, suitable for use in a Gooch crucible, prepared as follows.

Add dilute hydrochloric acid solution [1 volume of hydrochloric acid ($\rho_{20} = 1,19 \text{ g/ml}$) + 3 volumes of water] to the asbestos and boil for about 45 min.

Filter through a Buchner funnel and wash with water until free from the hydrochloric acid.

Then wash with acetone, dry and incinerate in the muffle furnace (7.7) at $550 \pm 25 \text{ }^\circ\text{C}$ for 16 h.

Allow to cool. Place the asbestos in a suitable quantity of the sulphuric acid solution (6.1) and boil for 30 min.

Decant the asbestos on to a Buchner funnel, wash abundantly with water and dry.

Treat the asbestos by boiling it for 30 min in the sodium hydroxide solution (6.2).

Filter again, wash once with the sulphuric acid solution (6.1), then rinse with water until the filtrate is neutral.

Dry, wash with one of the reagents (6.3), dry in the oven (7.3) at $130 \pm 2 \text{ }^\circ\text{C}$ and incinerate in the muffle furnace (7.7) at $550 \pm 25 \text{ }^\circ\text{C}$ to constant mass (usually at least 2 h).”

Clause 7

Replace the text of 7.9 by :

“**Separating device**, for example a Buchner funnel provided with a stainless steel screen.”

Clause 9

Replace the second sentence of 9.3.2.1 by :

“Add, as the filtration aid, about 1 g of asbestos (6.6) weighed to the nearest 1 mg.

Replace the last paragraph of 9.3.3.2 by :

“Collect all the residue in the incineration dish (7.6).”

B.2.2 Second variant

Clause 6

Replace the text of 6.6 by the corresponding text in B.2.1

Clause 7

Replace the text of 7.9 by :

“**Separating device**, comprising :

7.9.1 Filter flask, of capacity about 2 litres.

7.9.2 Glass funnel, of diameter 120 mm, provided with a rubber stopper to fit the flask (7.9.1) and a filter plate consisting of a porcelain disc of diameter 40 mm and 4 mm thick, having 16 perforations of 4 mm diameter, each of conical shape corresponding to the angle of the funnel; the disc shall be covered by a wire gauze of about 1 mm aperture size constructed from a material resistant to the test conditions.”

Clause 9

Complete the text of 9.3.1 as follows :

“If the test portion has to be defatted, use the method described in A.1.4 of annex A.”

Replace the first paragraph of 9.3.2.1 by :

“a) If the test portion has been defatted

Transfer the defatted test portion and the asbestos introduced (see 9.3.1) to the vessel (7.4) and add, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).

b) If the test portion has not been defatted

Transfer the test portion to the vessel (7.4), introduce 2 g of asbestos weighed to the nearest 1 mg, and add, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).”

Add the following paragraph at the end of 9.3.2.1 :

“During this time, place the filter plate in the funnel (7.9.2) of the filter flask (7.9.1). Disperse 2 g of the asbestos (6.6), weighed to the nearest 1 mg, in about 100 ml of water and pour on to the filter plate in the funnel. Re-filter to trap any asbestos that may have passed through the filter.”

Add the following paragraph at the end of 9.3.3.1 :

“During this time, prepare a new filter with 2 g of asbestos (6.6), weighed to the nearest 1 mg, proceeding as described for filtration after acid treatment.”

Replace the last paragraph of 9.3.3.2 by :

“Collect all the residue in the incineration dish (7.6).”

Replace the text of 9.4 by :

“Carry out a blank test with 6 g (3 times 2 g) of asbestos (6.6). The loss in mass during the incineration of the 6 g of asbestos shall not exceed 10 mg.”

B.3 Separation by filtration through sea sand

Replace the text of 6.6 by :

“**Sea sand**, prepared as follows.

Sieve the sand successively through sieves of wire gauze of aperture sizes 160 µm and 125 µm, complying with the requirements of ISO 3310/1.

Boil the sand fraction retained by the 125 µm aperture size sieve for 30 min in 4 mol/l hydrochloric acid solution. Rinse the sand with water until the rinsing water is free from chloride, as indicated by the absence of a reaction with silver nitrate solution, and incinerate in the muffle furnace (7.7) at 550 ± 25 °C to constant mass (usually at least 6 h are required).”

Clause 7

Replace the text of 7.6 by :

“**Filter crucible**, for example a glass filter crucible with a sintered glass disc, or a silica filter crucible with a sintered silica filter disc, of 40 mm diameter and with a pore size index in the range 16 to 40 µm (grade P 40).

The crucible is used as the separating device (7.9) when mounted on a filter flask.”

Clause 9

Add the following at the beginning of 9.3.2.1 :

“Spread on the filter plate of the crucible about 8 to 10 g of the sea sand (6.6) weighed to the nearest 1 mg.

Replace the text of 9.3.5 by :

“Place the crucible (7.9) and its contents into the cold furnace (7.7) and raise the temperature to 550 ± 25 °C. Incinerate the dry residue for 1 h at this temperature. Then allow to cool in the furnace, with the door open, to about 200 °C.

Place the crucible on a refractory plate, then cool in the desiccator (7.8) for 1 h and weigh to the nearest 0,5 mg.”

B.4 Separation by filtration through filter cloth

Clause 7

Add to the text of 7.9 :

“**7.9.1 Fine filter cloth**, (for example 18 fibres of 0,42 mm thickness per centimetre) in a conical funnel.

and, if necessary,

7.9.2 Gooch crucible, provided with a layer of fine, but compact, asbestos.”

Clause 9

Add in 9.3.2.2 :

“Use the fine filter cloth (7.9.1) as the separating device.”

Add in 9.3.3.2 :

“Use, as the separating device, the fine filter cloth (7.9.1) or the Gooch crucible (7.9.2) and asbestos.”

Add to the end of 9.3.3.2 :

“If a fine filter cloth is used, separate the residue from the filter (7.9.1), and transfer the whole to the incineration dish (7.6).”

B.5 Separation by filtration through filter paper

Clause 7

Replace the text of 7.9 by :

“**Separating device**, comprising :

7.9.1 Filter flask, of capacity about 1 litre.

7.9.2 Buchner funnel, of diameter about 100 mm, provided with ashless filter paper, that can be used with acid solutions and of such quality that the paper fibres are resistant during filtration and washing.”