



**Summary of Substantive Changes
between the 2015 edition and the 2016 edition of
NSF/ANSI 60, “Drinking Water Treatment Chemicals - Health Effects”**

Presented to IAPMO Standards Review Committee on November 07, 2016

General: The changes to this standard may have an impact on currently listed products. The substantive changes are:

- Added new definitions in sections 2.5 ‘bonded individual’ and 2.9 ‘chain of custody’ to address issues that certifiers have experienced in recent years in implementing the tamper evidence requirements under section 3.9 of NSF/ANSI 60, and to clarify requirements for small packaged products, large reusable containers (totes), tamper-evident seals used in milk run deliveries, and chain of custody.
- Editorially repositioned Notes 1 & 2 to be adjacent to text referring to testing and added section numbers 3.2.3.1 and 3.2.3.2 to respective paragraphs for clarity and citation from another section of the standard.
- Added new section 3.6.1 ‘Hazard assessment procedures for process water’ to help identify steps to be taken to reduce the potential contamination of treatment chemicals during periods of varying water quality changes that can occur seasonally, after heavy rains or droughts, or other events such as chemical spills.
- Decreased the typical use level (TUL) of polyamines from 20 mg/L (ppm) to 10 mg/L (ppm) within Table 4.1 – Coagulation and flocculation products – product identification and evaluation – to better reflect usage in the field.
- Added new like sections 5.3.1 ‘Minimizing risk for pathogen transmittal in chemicals’ and 7.3.4 ‘Minimizing risk for pathogen transmittal in chemicals,’ to address chemicals noted with either a potential to be used post disinfection or used on a water system which doesn’t add disinfectant fall under these sections only.
- Increased the typical use level (TUL) of carbon dioxide from 200 mg/L (ppm) to 600 mg/L (ppm) within Table 5.1 – Chemicals for corrosion and scale control, softening, sequestering, precipitation, and pH adjustment – to better reflect usage of water utilities that may require doses up to 600 ppm for pH control.
- Added a new definition for ‘separation process tracers’ in section 8 for tracers used to verify the integrity of RO membranes and determine whether the membrane has been compromised.
- Added three analytical methods for acrylamide monomer in polyacrylamide under Annex B.



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2 Definitions

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2.5 bonded individual: A bond is a promise that a contractor, or driver, will fulfill his obligations. If a driver is bonded, a third-party company or his trucking company backs his performance and promises he will complete the task as agreed upon. Therefore, a bond provides assurance that the contracted work will be satisfactorily completed. If a loss occurs, however, a separate insurance policy may be required to cover the property, not the bond.

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2.9 chain of custody: A record documenting the existence of positive control and security over an item with counter signatures or other acknowledgements (receiver/deliverer) at each stage of transition of control/security responsibility.

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3 General requirements

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3.2 Formulation submission and review

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3.2.3 If the finished product contains water, the formulation information provided by the manufacturer shall be reviewed to determine if the water source used in the manufacturing of the finished product requires testing.

3.2.3.1 Water sources that require testing include, but are not limited to, the following: non-treated surface water; non-treated ground water; non-treated rain water; and water collected from other non-treated sources. Testing of water sources shall include the following analyses; metals, VOCs, base/neutral/acid scan, radionuclides, herbicides/pesticides, and dioxin/furan scan.

NOTE 1 - Testing related to water sources may be performed on the finished product or on a separate water sample; however, any test conducted on the finished product itself, as part of such product's certification testing battery, may be omitted from testing performed on a separate water sample.

NOTE 2 – Metals analysis shall include antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, mercury, selenium, and thallium.

3.2.3.2 Water sources that do not require testing include the following: treated or non-treated water sources that comply with state or national drinking water standards, deionized water, distilled water, demineralized water, water treated on-site to drinking water quality with the exception of disinfection, drinking water treated with a reverse osmosis system, and ground water treated on-site to drinking water



quality or a higher purity grade ~~and recirculated water or condensate water originating from water sources that do not require testing.~~

~~NOTE 1 – Testing related to water sources may be performed on the finished product or on a separate water sample; however, any test conducted on the finished product itself, as part of such product’s certification testing battery, may be omitted from testing performed on a separate water sample.~~

~~NOTE 2 – Metals analysis shall include antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, mercury, selenium, and thallium.~~

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3.6 Formulation control

The manufacturer shall have practices in place to ensure that the product is manufactured according to the approved formulation, and to ensure that no changes in manufacturing processes, product composition, or raw materials can occur without prior authorization by the certification body. The practices shall ensure that no contamination is introduced by product packaging, transfer and storage equipment, or dilution water. Containers shall either be dedicated to one category of chemical, or written records of cleaning (e.g. wash tickets) must be available for review. Documentation of these practices shall be available for review.

[3.6.1 Hazard assessment procedures for process water](#)

[3.6.1.1 If the finished product contains water supplied by a public water system, the manufacturer shall have procedures in place that identify steps to be taken when utilities issue warnings such as a boil water alert, or do not drink, or do not use order.](#)

[3.6.1.2 If the finished product contains water sourced through other than a public water system, the manufacturer shall have procedures that periodically monitor the water for chemicals of concern. The procedure shall also specify treatment of the source water, or preclude its use, when significant quality changes may introduce unacceptable levels of contaminants to the product.](#)

[NOTE – Significant water quality changes can occur seasonally, after heavy rains or droughts, or other events such as chemical spills. Manufacturers need processes in place that identify steps to be taken when utilities issue warnings such as a boil water alert, or do not drink, or do not use order. Similar hazards can occur with non-utility waters. Steps need to be taken to reduce the potential contamination of treatment chemicals during these periods of varying water quality.](#)

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3.9.2 Security requirements for packaged products

Packaged product shall be stored, shipped, and delivered in tamper-evident packaging as defined in Section 3.9.1. Properly constructed, labeled, and sealed multi-wall containers such as bags and fiber drums constitute two forms of acceptable tamper-evident packaging.



Smaller containers do not require individual tamper evident seals when shipped in a larger container from the manufacturer with acceptable seals or closures on the larger container as noted in the prior paragraph, provided the smaller containers are not intended to be sold individually as certified product (i.e., not labeled for individual sale/use for drinking water applications).

3.9.2.1 Bags and super sacks

Packages for product shipped without reusable openings shall be constructed and properly sealed to make opening or substitution obvious to the purchaser. The packages shall display the company's name, and employ seals that are destroyed upon opening, or that make resealing unlikely (e.g. serialized tags), or other equivalent tamper-evident measures so that once opened, the tamper-evident feature of the seal on the packaging ~~cannot be restored nor readily duplicated~~ is unable to be restored or readily duplicated.

3.9.2.2 Drums and small containers

Drums and small containers used for product shall be constructed and properly sealed to make opening or substitution obvious to the purchaser. Openings in the containers shall be sealed with tamper-evident seals and the packages shall display the company's name. Packages shall employ seals that are destroyed upon opening, or that make resealing unlikely (e.g. ultrasonic seals), or other equivalent tamper-evident measures so that once opened, the tamper-evident feature of the seal ~~cannot~~ is unable to be restored ~~nor~~ readily duplicated

3.9.3 Security requirements for bulk shipments and large reusable containers (totes)

~~Bulk quantities of product~~ Drinking water treatment chemicals shipped in bulk¹² shall be secured during storage and distribution by employing one or more of the following security measures (see 3.9.3.1, 3.9.3.2 and 3.9.3.3). These requirements are applicable to a single load delivered to one or to multiple locations¹³. This requirement applies ~~to all tank truck chemical deliveries~~, and to railcar chemical deliveries that are direct to drinking water utilities or to other end users involved in the addition of the delivered chemical to drinking water and to truck deliveries whether to a single destination or by milk run deliveries.

3.9.3.1 Tamper-evident seals

~~Bulk shipping containers~~ Containers used for bulk shipments shall have tamper protection provided at all openings capable of loading or unloading chemicals. Vents shall have tamper protection provided unless they are protected by construction that makes them incapable of receiving chemicals. Bulk ~~shipping~~ containers may be sealed with a uniquely numbered, non-reusable, tamper-evident seal on each opening in the ~~shipping~~ container. If tamper-evident seals are used, the seals shall remain in place until removed at the point of delivery. Seal numbers shall be recorded and disclosed on shipping documents provided to the purchaser at the time of delivery and kept available for review by the certification body. If tamper-evident seals are used in milk run deliveries, a new seal shall be applied after each partial off-loading and noted in the consignment records after each partial delivery.

¹²The term "bulk" is used for shipments being transported in a container having a volume of more than 1,000 L and applies to containers holding solid, liquid and gaseous products. Such containers can be multi-modal containers, tank trucks or tank cars appropriate to the physical characteristics of the product being transported.

¹³Multiple destination shipments are referred to as "milk run deliveries".



3.9.3.2 Chain of custody

An auditable continuous chain of custody protocol may be used to ~~document~~ record secure distribution of product. Maintaining a continuous chain of custody requires that the product is under the continuous control of bonded and designated ~~responsible~~ individuals, that direct access to the product is restricted to those individuals, and that the shipping container is sealed or ~~locked~~ or secured at all times during ~~shipping~~ transport from the place of shipment to the place of delivery. If chain of custody is used, a ~~copy of a~~ completed, ~~signed~~ chain of custody ~~form~~ record showing continuous and secure custody between the certification holder ~~and~~ to the purchaser shall be provided by the transporter to the certification holder and to the purchaser at the time of delivery. The completed chain of custody record returned to the certification holder shall be ~~and~~ kept available for review by the certification body.

NOTE - For the custody procedure during transport by road of certain drinking water treatment chemicals, there may be a requirement for two persons to be assigned to the distribution activity, with the vehicle being under the direct supervision of at least one person at all times.

Where a paper-based chain of custody procedure is used for milk run deliveries, the documentation shall have sufficient copies that a copy of the documentation shall be signed and provided to each consignee noting the quantity delivered at that destination, and the balance remaining in the shipment. A copy of the complete series of deliveries shall be provided by the transporter to the certification holder.

Where an electronically-based chain of custody procedure is used for milk run deliveries, the record of the custody and deliveries shall be provided by the transporter to the certification holder.

NOTE - It is normal transport procedure for the transporter to retain duplicate records of all cargo acceptances and deliveries, including chain of custody documents or records. These may be accessed if necessary to verify chain of custody.

3.9.3.3 Alternative method

An alternative method or methods¹⁴ agreed upon by the certification holder and the purchaser may be used for bulk shipments if the alternative method provides protection against tampering that is equivalent to this Standard. If alternative methods are used, the agreement with the purchaser and description of the alternative methods shall be in written form and kept available for review by the certification body.

3.9.4 Tamper-evident integrity

The tamper-evident features employed on all final product packaging, seals, and ~~bulk shipping containers~~ containers used for bulk shipments shall be designed to remain intact when handled in a reasonable manner during manufacture, storage, shipment and delivery to the purchaser.

¹⁴Alternative methods may include secured electronic tracking and communication methods.

4 Coagulation and flocculation chemicals



The following table is a generic listing of the types of products covered in this section. This table is not intended to be a complete list of all products used for coagulation and flocculation applications. Inclusion of a product does not indicate either a use endorsement of the product or an automatic acceptance under the provisions of this Standard. Annex D, Table D1, includes a cross-reference index of the various chemicals (and the more common synonyms) contained in this table.

Table 4.1 – Coagulation and flocculation products – product identification and evaluation

Chemical type (Description)	Synonyms	Formula (CAS number)	Approximate molecular weight	Preparation method	Typical use level (mg/L) ¹	Minimum Test Batteries of Chemistry-specific analyses ²
poly (epichlorohydrin/dimethylamine) (polyamines) (polyelectrolytes)	EPI/DMA, polyamine	(25988-97-0) or (42751-79-1)	30 thousand - 3 million	—	2 10.0 ¹⁰	epichlorohydrin, 1,3-Dichloro-2-propanol, 1,2-dichloro-3-propanol, glycidol, dimethylamine, ethylenediamine (if used as a branching agent)
<p>¹ The typical use level is an application level which has been used historically in water treatment. The typical use level is not the maximum use level for the product unless specifically stated.</p>						

² Analysis for all chemistry-specific analytes in these minimum test batteries shall be performed each time the product is evaluated. Analysis shall also include formulation-dependent analytes as identified during formulation review. Testing for specific repackages, blends, or dilutions of previously certified products may be waived.

³ If nitrogen-containing initiators are used in these chemical types, evaluation shall include analysis for the initiator and any initiator by-products.

⁴ The typical use level for this product is based on an acrylamide polymer application of 1 mg/L and an acrylamide monomer level of 0.05% in the polymer, or equivalent (40 CFR 141.111) for a carryover of not more than 0.5 ppb of acrylamide monomer into the finished water.

⁵ Metals = antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, mercury, selenium, thallium

⁶ A GC/MS analysis shall also be performed on this chemical type when recycled materials are used in the manufacturing process.

⁷ The first value is the typical use level as indicated by the chemical formula. The second value is the typical use level as aluminum oxide for the aluminum salts (aluminum chloride, aluminum sulfate, polyaluminum chloride, and sodium aluminate).

⁸ The first value is the typical use level as indicated by the chemical formula. The second value is the typical use level as Fe for the iron salts (ferric chloride, ferric sulfate, ferrous chloride, and ferrous sulfate).

⁹ The typical use level for this product is based on a polyDADMAC polymer application of 25 mg/L and a carryover of not more than 50 ppb of DADMAC into the finished water.



¹⁰ The typical use level for this product is based on a EPI/DMA polymer application of ~~2~~1 0 mg/L and a epichlorohydrin monomer level of 0.01% in the polymer, or equivalent (40 CFR 141.111) for a carryover of not more than ~~2~~1 ppb of epichlorohydrin monomer into the finished water.

¹¹ The typical use level of this product is expressed as mg/L of active polymer in the product as sold.

¹² Sodium silicate may be used in conjunction with an acid-forming substance to produce activated silica. The net concentrations of sodium silicate and acid-forming substance are not to exceed the maximum use levels for these chemicals individually.

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5 Chemicals for corrosion and scale control, softening, precipitation, sequestering, and pH adjustment

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5.3 General requirements

General information and evaluation requirements for the products covered in this section are summarized in Table 5.1.

5.3.1 Minimizing risk for pathogen transmittal in chemicals

To minimize the risk for pathogen transmittal in chemicals evaluated under this section, those that contain water in the finished product shall only be produced using waters meeting the criteria of 3.2.3.2.

NOTE: The chemicals in section 5 may be added to drinking water post disinfection or in drinking water systems not adding disinfectant to the treated water. As such, this section is intended to minimize the potential for pathogen introduction from treatment chemicals where other measures are not in place to prevent it.

The following water treatment chemicals are exempted from this restriction.

- Those with a pH less than or equal to 2 or greater than or equal to a pH of 11, or
- Those where the product literature limits the use of the treatment chemical to applications where the water is disinfected post addition of the chemical.

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Table 5.1 – Chemicals for corrosion and scale control, softening, sequestering, precipitation, and pH adjustment – product identification and evaluation

Chemical type (primary use)	Synonyms	Formula (CAS number)	Molecular weight (g)	Preparation method	Typical use level ¹ (mg/L)	Minimum Test Batteries of Chemistry-specific analyses ²
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carbon dioxide (pH adjustment)	—	CO2 (124-38-9)	44	method E, Annex B, section B.3.6	200 600	VOCs
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7 Miscellaneous treatment applications

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7.3 General requirements

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[7.3.4 Minimizing risk for pathogen transmittal in chemicals](#)

[To minimize the risk for pathogen transmittal in chemicals evaluated under this section, those that contain water in the finished product shall only be produced using waters meeting the criteria of 3.2.3.2.](#)

[NOTE: The chemicals in section 7 may be added to drinking water post disinfection or in drinking water systems not adding disinfectant to the treated water. As such, this section is intended to minimize the potential for pathogen introduction from treatment chemicals where other measures are not in place to prevent it.](#)

[The following water treatment chemicals are exempted from this restriction.](#)

- [Those with a pH less than or equal to 2 or greater than or equal to a pH of 11, or](#)
- [Those where the product literature limits the use of the treatment chemical to applications where the water is disinfected post addition of the chemical.](#)

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8 Miscellaneous water supply products

8.1 Coverage

This section covers products used in a variety of drinking water supply applications. These products are not routinely used to produce a treatment effect in the water they may contact. The products can be fed continuously, applied intermittently, or flushed from the water supply system prior to its return to use. These products include, but are not limited to, antifoamers, separation process scale inhibitors and cleaners, [separation process tracers](#), water well drilling aids, water well rehabilitation aids, well pump lubricating oils, backfill materials for cathodic protection or electrical installations, and distribution system cleaning aids.

8.2 Definitions

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8.2.7 separation process tracers: *Chemical products used in reverse osmosis and nano-filtration systems to verify the integrity of the seals, membranes, etc. These products are dosed into the feed water and their absence verified via an analytical method in the permeate water to show that the membrane and seals are intact.*

8.2.78 well drilling aids: Products used in drilling and development of wells used as drinking water sources.

8.2.89 well rehabilitation aids: Products used in the rehabilitation and the cleaning of wells used as drinking water sources.

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Annex B
(normative)
Sampling, preparation, and analysis of samples

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B.4 Analysis methods

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B.4.3 Organics

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B.4.3.2 Acrylamide monomer in polyacrylamide

Acrylamide monomer shall be determined using one of the following methods. ~~the method described in "Determination of acrylamide monomer in polyacrylamide and in environmental samples by high performance liquid chromatography," Analytical Chemistry 50: 1959 (1978).~~ Alternate methods shall be allowed to be used but shall be validated.

B.4.3.2.1 Method A

Sample analysis shall be by High Performance Liquid Chromatography (HPLC) with ultraviolet (UV) detection.

B.4.3.2.1.1 Apparatus

The following apparatus shall be used in this analysis:



- Vacuum apparatus or Sonicator to degas mobile phase.
- HPLC pump.
- HPLC-UV spectrophotometric detector.
- YMC ODS-AL column, 4.6x150mm, (AL12S05-1546WT); Guard Housing (XPEF43WTI); and YMC ODS-AL S-5 Guard Column (AL12S05 G 304WTA).
- Bio-Rad HPLC Fast Acid Analysis Column Cat. No. 125-0100 and Micro-Guard Refill Cartridges Cat. No. 125-0129.
- Autosampler 100µl capabilities.
- Analytical Data Acquisition System.
- Millipore 0.1 VV µm filter disc and 0.22 µm GS filter paper.
- Volumetric pipettes.
- Analytical balance accurate to 0.0001 gm.
- Multi-plate stirrer and 1 inch stirring bars.
- Vacuum filtration flasks
- 100ml volumetric flasks
- 400ml beakers
- Vacuum manifold for 0.1 µm Millex-VC filters
- For Latex: Cage stirrer, Jiffy Mixer, Model LM and Cone Driven Stirring Motor

B.4.3.2.1.2 Reagents

The following reagents shall be used in this analysis:

- Concentrated sulfuric acid (H₂SO₄) reagent grade
- Acrylamide of 99+%
- Milli-Q water

B.4.3.2.1.3 Procedure

B.4.3.2.1.3.1 Preparation of mobile phase

The mobile phase shall be prepared in the following manner.

- Add 1.0 ml of concentrated sulfuric acid to a 2L volumetric flask, QS with DI water and mix well. This yields a solution of sulfuric acid at approximately 0.01 M
- Filter through 0.22 µm GS Millipore filter paper.
- Vacuum or ultrasonicate to degas.

B.4.3.2.1.3.2 Sample Preparation

a) Dry Polymer Preparation:

- 1) Weigh 199.5 +/-0.1 gm DI water into a 400 ml tall form beaker. Record the weight as Wwt.



- 2) Clamp beaker under the mixer with the impeller centered about 1 cm above the bottom of the beaker.
- 3) Set mixer speed to 800 +/-20 rpm.
- 4) Place 0.5 gm (to the nearest 0.1mg) of dry polymer into the beaker. Record the weight as DPwt.
- 5) Mix at 800 rpm for 30 minutes.

a.1) Chromatography Sample preparation for Dry Polymer:

- 1) Weigh 1.0 gm (to the nearest 0.1mg) of the solution prepared in section B.4.3.2.4.2.a into a glass jar. Record the weight as DPS.
- 2) Add 10 ml of mobile phase weighed to the nearest (0.1mg) into the same jar. Record the total weight as DPT.
- 3) Add a stir bar and stir for 30 minutes at a medium speed.
- 4) After 30 minutes, filter through a 0.1µm Millex-VC using a vacuum manifold.
- 5) The sample is now ready for injection.

b) Chromatography Sample Preparation for Latex Polymer:

- a) Weigh 0.1 gm (to the nearest 0.1mg) of latex polymer into a 100ml volumetric flask. Record the weight as LPwt.
- b) QS the flask with mobile phase.
- c) Add a stir bar and stir for 30 minutes at a medium speed.
- d) After 30 minutes, filter through a 0.1µm Millex-VV filter unit.
- e) The sample is now ready for injection.

B.4.3.2.1.3.3 Calibration Standards

Five calibration standards shall be prepared at concentrations of 0.005, 0.05, 0.1, 1 and 5 ppm using the dilution sequence noted below:

Prepare a 1000 ppm stock solution then prepare standards as listed in Table I noted below. Dilute all standards with mobile phase. Pipet volume in column A into volumetric listed in column B to yield standard concentration listed in column C. Adjust accordingly for the actual concentration of the stock solution.

Table I

	<u>A</u>	<u>B</u>	<u>C</u>
<u>Working</u>	<u>Standard Used</u>	<u>Volumetri</u>	<u>Standard</u>
<u>Standard</u>	<u>Size</u>	<u>C</u>	<u>Concentration</u>
	<u>(ml)</u>		
<u>A</u>	<u>0.5 ml of 1000 ppm stock</u>	<u>100</u>	<u>5 ppm</u>
<u>B</u>	<u>0.1 ml of 1000 ppm stock</u>	<u>100</u>	<u>1 ppm</u>
<u>C</u>	<u>2.0 ml of 5 ppm solution</u>	<u>100</u>	<u>0.1 ppm</u>



<u>D</u>	<u>1.0 ml of 5 ppm solution</u>	<u>100</u>	<u>0.05 ppm</u>
<u>E</u>	<u>0.1 ml of 5 ppm solution</u>	<u>100</u>	<u>0.005 ppm</u>

B.4.3.2.1.3.4 Instrumentation Conditions

The sample solution of the polymer sample shall be analyzed using the following conditions.

a) Columns:

1) YMC ODS-AL S-5 Guard Column

2) YMC ODS-AL C18, 4.6x150mm column

3) Bio-Rad HPLC fast acid analysis column and guard cartridge

b) The columns are connected in series, C18 guard column, C18 column, Bio-Rad guard column, Bio-Rad Fast Acid column

c) Mobile Phase: 0.01 M H₂SO₄

d) Flow Rate: 0.6 ml/min

e) Detection: 210 nm UV 0.002 a.u.f.s. (adjustable)

f) Injection volume: 100 ul

g) Temperature: Ambient

B.4.3.2.1.4 Calculations

A linear regression of the five calibration standards shall be used to calculate the concentration of each analyte in the sample preparation (ppm). The following equations shall be used to calculate the concentration of the analyte in the respective polymer sample.

a) Dry Polymer Calculation:

$$\frac{\text{Curve concentration (ppm)} \times [(Wwt + DPwt)/DPwt] \times DPT/DPS}{= \text{PPM of acrylamide in polymer product}}$$

b) Latex Polymer Calculation:

$$\text{Curve concentration (ppm)} \times 100/LPwt = \text{PPM of acrylamide in polymer product}$$

B.4.3.2.2 Method B²⁴

Sample analysis shall be by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection.

B.4.3.2.2.1 Apparatus

The following Apparatus shall be used in this analysis:

- HPLC equipped with UV detector
- Column: BioRad Fast Acid 100 x 7.8mm (Catalog # 125-0100) or equivalent
- Analytical balance, 0.1mg accuracy



- Syringes
- Analytical Shaker
- Centrifuge
- Volumetric flasks
- 0.45um syringe filters

²⁴ Based on method in Analytical Chemistry 50: 1959 (1978). "Determination of acrylamide monomer in polyacrylamide and in environmental samples by high performance liquid chromatography."

B.4.3.2.2 Reagents

The following reagents shall be used in this analysis:

- HPLC grade Acetonitrile
- HPLC grade Methanol
- HPLC Deionized water
- 93-98% Trace Metal grade Sulfuric Acid

B.4.3.2.2.3 Procedure:

B.4.3.2.2.3.1 Preparation of mobile phase:

5mM Sulfuric Acid: Add 280ul Sulfuric acid into 1L Deionized water

B.4.3.2.2.3.2 Preparation of standard:

Acrylamide >99% Sigma (Catalog #A8887) or equivalent

Stock Standard: Accurately weigh 10mg Acrylamide into 10mL Methanol

B.4.3.2.2.3.3 Calibration standards:

Five calibration standards shall be prepared at concentrations of 10, 50, 100, 500 and 1000ug/L by serial dilution of the Acrylamide stock standard using Deionized water.

B.4.3.2.2.3.4 Extraction solvent:

70% Acetonitrile/30% Deionized water:

- Into a 500mL Volumetric flask add 350mL Acetonitrile and 150mL of Deionized water and mix.
- If the sample does not disperse in the Acetonitrile/Water solution an extraction solvent with higher aqueous content can be used (e.g. 50% methanol / 50% Acetonitrile).

B.4.3.2.2.3.5 Analysis solution:

- Mix/shake polymer sample to insure homogeneity
- Add 1g of sample into 10mL of Extraction Solvent (70% Acetonitrile/30% DI water) to a 40mL vial



- Cap vial and place on the shaker for at least 15 min.
- Centrifuge at 2000 rpm for 5 minutes.
- Filter supernatant through 0.45 μ m filter and collect into an 8ml vial
- Dilute supernatant 1:100 into Deionized water

B.4.3.2.2.4 Instrument conditions:

The analysis solution containing the polymer sample shall be analyzed under the following conditions:

- Column temperature: ambient
- Injection volume 100 μ l
- Detector: Scan 200 - 360 nm @ 2nm intervals Quantitate @ 205nm

B.4.3.2.2.5 Calculations:

A linear regression of the five calibration standards shall be used to calculate the concentration of Acrylamide in the sample extract (in μ g/L). The following equation shall be used to calculate the concentration of the analyte in the polymer sample. (Calculations assume a 100-fold dilution prior to analysis. In the event a greater dilution factor is required that value should be used in the equations):

$$\frac{\text{Curve Concentration (ug/L)} \times \text{dilution (100)} \times 1\text{L} \times \text{Extraction Volume (10mL)}}{\text{Sample Mass (g)}} = X \text{ ug/g} = \text{mg/kg}$$

B.4.3.2.3 Method C

Sample analysis shall be determined by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection.

B.4.3.2.3.1 Apparatus and equipment

- HPLC equipped with UV detector and acquisition system
- Column: Atlantis dC18 150 x 4.6 mm – 3 μ m (Ref Waters # 186001342)
- Volumetric cylinders,
- Beakers,
- Glass bottles,
- Pipets,
- 30 ml vials with cap,
- Mechanical stirrer,
- Analytical balance (0.001 g),
- Calibrated pH-meter,
- Volumetric flasks,
- 0.45 μ m syringe filters (compatible with solvent).



B.4.3.2.3.2 Reagents

The following reagents shall be used in this analysis:

- HPLC grade Methanol,
- HPLC grade Ethanol,
- Isopropanol,
- Acetone,
- HPLC Deionized water,
- Potassium phosphate monobasic, KH₂PO₄, 99%+,
- Acrylamide of 99+%,
- Phosphoric acid.

B.4.3.2.3.3 Procedure:

B.4.3.2.3.3.1 Preparation of mobile phase:

* 20 mM/L KH₂PO₄, pH = 3.8 solution:

Weigh 2.72 g of potassium phosphate monobasic and complete to 1L with deionized water in a volumetric flask.

After dissolution, adjust the pH to 3.8 with phosphoric acid.

* Mobile phase:

Take 850 mL of the previous solution and add 150 mL of methanol

Filter through 0.20 µm acetate cellulose filter paper.

B.4.3.2.3.3.2 Preparation of standard:

Prepare a 10,000 ppm stock solution: Accurately weigh 1.00 g of acrylamide and dissolve it with deionized water in a 100 mL volumetric flask.

B.4.3.2.3.3.3 Calibration standards:

Five calibration standards shall be prepared at concentrations of 10, 20, 40, 50 and 100 ppm by dilution of the acrylamide stock solution using deionized water.

For example: pipet 1 mL of stock solution and complete with deionized water to 100 mL in a volumetric flask to obtain a standard at 100 ppm.

B.4.3.2.3.3.4 Extraction solvent:

Solution A: 540 mL of isopropanol + 450 mL of deionized water + 10 mL ethanol.

Solution B: 740 mL of isopropanol + 250 mL of deionized water + 10 mL ethanol.



Solution C: 990 mL of isopropanol + 10 mL ethanol.

Solution D: 900 mL of acetone + 100 mL of deionized water.

Solution E: 800 mL of isopropanol + 200 mL of deionized water.

B.4.3.2.3.3.5 Sample preparation:

The extraction is made in function of the physical form of the polymer. All supernatants are filtrated through a 0.45 μ m filter before injection.

a- Dry polymer

- Anionic:
 - Weigh 1.99-2.01 g of polymer
 - Add 10 mL of solution A – shake for 40 min
 - Add 10 mL of solution B – shake for 40 min
- Low cationic:
 - Weigh 1.99-2.01 g of polymer
 - Add 10 mL of solution A – shake for 40 min
 - Add 10 mL of solution B – shake for 40 min
- Mid to high cationic:
 - Weigh 0.99-1.01 g of polymer
 - Add 10 mL of solution A – shake for 40 min
 - Add 10 mL of solution C – shake for 40 min
- For all products:
 - Weigh 1.99-2.01 g of polymer
 - Add 20 mL of solution D – shake for 4 hours.

b- Emulsion

- Anionic:
 - Place 50 mL of ethanol in 100 mL beaker and place under stirring.
 - Add 5.00 of polymer (measured by double weighing).
 - Let stir for 30 min.
- Low to middle cationic:
 - Place 50 mL of ethanol in 100 mL beaker and place under stirring.
 - Add 5.00 of polymer (measured by double weighing).
 - Let stir for 30 min.
- High cationic:
 - Place 50 mL of isopropanol in 100 mL beaker and place under stirring.
 - Add 5.00 of polymer (measured by double weighing).
 - Let stir for 30 min.
- For all products:
 - Weigh 1.99-2.01 g of polymer
 - Add 20 mL of solution D – shake for 4 hours.



c- Liquid

- Weigh 1.99-2.01 g of polymer

- Add 20 mL of solution E – shake for 2 hours.

NOTE - if the obtained value after injection is higher than the standard, the sample shall be diluted and new injection made.

B.4.3.2.3.4 Instrument conditions:

The analysis solution containing the polymer sample shall be analyzed under the following conditions:

- Mobile phase: 85% 20 mM/L KH₂PO₄ pH = 3.8 / 15% Methanol (v/v)
- Flow Rate: 1.0 ml/min
- Detection: 205 nm UV
- Injection volume: 10 µl
- Temperature: Ambient

B.4.3.2.3.5 Calculations:

A linear regression through zero of the five calibration standards shall be used to calculate the concentration in the sample preparation (ppm):

$$\text{Area} = B \times \text{Conc (ppm)}$$

The following equations shall be used to calculate the concentration in the respective polymer sample:

$$\text{Acrylamide (ppm)} = \text{Conc (ppm)} \times \frac{\text{Extraction volume (ml)}}{\text{Sample mass (g)}}$$

In the event a greater dilution factor is required that value should be used in the last equation.