ethyl acrylate content limits prescribed in paragraph (b) of this section, and the extractability limits prescribed in paragraph (c)(2) of this section, when tested by the methods prescribed for polyethylene in § 177.1520.

(1) *Specifications*—(i) *Infrared identification*. Ethylene-ethyl acrylate copolymers can be identified by their characteristic infrared spectra.

(ii) *Quantitative determination of ethyl acrylate content*. The ethyl acrylate can be determined by the infrared spectra. Prepare a scan from 10.5 microns to 12.5 microns. Obtain a baseline absorbance at 11.6 microns and divide by the plaque thickness to obtain absorbance per mil. From a previously prepared calibration curve, obtain the amount of ethyl acrylate present.

(iii) *Specific gravity*. Ethylene-ethyl acrylate copolymers have a specific gravity of not less than 0.920 nor more than 0.935, as determined by ASTM method D1505–68 (Reapproved 1979), ''Standard Test Method for Density of Plastics by the Density-Gradient Technique,'' which is incorporated by reference. Copies may be obtained from the American Society for Testing Materials, 1916 Race St., Philadelphia, PA 19103, or may be examined at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC 20408.

(2) *Limitations*. Ethylene-ethyl acrylate copolymers or the blend may be used in contact with food except as a component of articles used for packaging or holding food during cooking provided they meet the following extractability limits:

(i) Maximum soluble fraction of 11.3 percent in xylene after refluxing and subsequent cooling to 25 °C.

(ii) Maximum extractable fraction of 5.5 percent when extracted with *n*-hexane at 50 °C.

(d) The provisions of paragraphs (b) and (c)(2) of this section are not applicable to ethylene-ethyl acrylate copolymers used in the formulation of adhesives complying with § 175.105 of this chapter.

[42 FR 14572, Mar. 15, 1977, as amended at 49 FR 10108, Mar. 19, 1984]

§ 177.1330 Ionomeric resins.

Ionomeric resins manufactured from either ethylene-methacrylic acid copolymers (and/or their ammonium, calcium, magnesium, potassium, sodium, and/or zinc partial salts), ethylene-methacrylic acid-vinyl acetate copolymers (and/or their ammonium, calcium, magnesium, potassium, sodium, and/or zinc partial salts,), or methacrylic acid polymers with ethylene and isobutyl acrylate (and/or their potassium, sodium and/or zinc partial salts) may be safely used as articles or components of articles intended for use in contact with food, in accordance with the following prescribed conditions:

(a) For the purpose of this section, the ethylene-methacrylic acid copolymers consist of basic copolymers produced by the copolymerization of ethylene and methacrylic acid such that the copolymers contain no more than 20 weight percent of polymer units derived from methacrylic acid, and the ethylene-methacrylic acid-vinyl acetate copolymers consist of basic copolymers produced by the copolymerization of ethylene, methacrylic acid, and vinyl acetate such that the copolymers contain no more than 15 weight percent of polymer units derived from methacrylic acid.

(b) For the purpose of this section, the methacrylic acid copolymers with ethylene and isobutyl acrylate consist of basic copolymers produced by the copolymerization of methacrylic acid, ethylene, and isobutyl acrylate such that the copolymers contain no less than 70 weight percent of polymer units derived from ethylene, no more than 15 weight percent of polymer units derived from methacrylic acid, and no more than 20 weight percent of polymer units derived from isobutyl acrylate. From 20 percent to 70 percent of the carboxylic acid groups may optionally be neutralized to form sodium or zinc salts.

(c) The finished food-contact article described in paragraph (a) of this section, when extracted with the solvent or solvents characterizing the type of food and under the conditions of time and temperature characterizing the conditions of its intended use as determined from tables 1 and 2 of § 176.170(c)

of this chapter, yields net acidified chloroform-soluble extractives in each extracting solvent not to exceed 0.5 milligram per square inch of food-contact surface when tested by the methods described in paragraph (e)(1) of this section, and if the finished food-contact article is itself the subject of a regulation in parts 174, 175, 176, 177, 178 and § 179.45 of this chapter, it shall also comply with any specifications and limitations prescribed for it by that regulation.

NOTE: In testing the finished food-contact article, use a separate test sample for each required extracting solvent.

(d) The finished food-contact article described in paragraph (b) of this section, when extracted according to the methods listed in paragraph (e)(2) of this section and referenced in this paragraph (d), using the solvent or solvents characterizing the type of food as determined from table I of paragraph (f) of this section, shall yield net acidified chloroform-soluble extractives as follows:

(1) *For fatty food use.* (i) For films of 2 mil (0.002 inches) thickness or less, extractives shall not exceed 0.70 milligram/square inch[1] (0.109 milligram/square centimeter) of food-contact surface (*n*-heptane extractions) when extracted by the abbreviated method cited in paragraph (e)(2)(i) of this section.

(ii) For films of greater than 2 mils (0.002 inch) thickness, extractives shall not exceed 0.40 milligram/square inch[1] (0.062 milligram/square centimeter) of food-contact surface (*n*-heptane extractions) when extracted by the abbreviated method cited in paragraph (e)(2)(i) of this section, or

(iii) Alternatively, for films of greater than 2 mils thickness, extractives shall not exceed 0.70 milligram/square inch[1] (0.109 milligram/square centimeter) of food-contact surface (*n*-heptane extractions) when extracted by the equilibrium method cited in paragraph (e)(2)(ii) of this section.

(2) *For aqueous foods.* (i) The net acidified chloroform-soluble extractives shall not exceed 0.02 milligram/square inch[2](0.003 milligram/square centimeter) of food-contact surface (water, acetic acid, or ethanol/water extractions) when extracted by the abbreviated method cited in paragraph (e)(2)(i) of this section.

(ii) Alternatively, the net acidified chloroform-soluble extractives shall not exceed 0.05 milligram/square inch[3] (0.078 mg/square centimeter) of food-contact surface (water, acetic acid, or ethanol/water extractions) when extracted by the equilibrium method cited in paragraph (e)(2)(ii) of this section. If when exposed to *n*-heptane, a particular film splits along die lines, thus permitting exposure of both sides of the film to the extracting solvent, the results for that film sample are invalid and the test must be repeated for that sample until no splitting by the solvent occurs. If the finished food-contact article is itself the subject of a regulation in parts 174, 175, 176, 177, 178 and § 179.45 of this chapter, it shall also comply with any specifications and limitations prescribed for it by that regulation.

NOTE: In testing the finished food-contact article, use a separate test sample for each required extracting solvent.

(e) *Analytical methods*—(1) *Selection of extractability conditions for ionomeric resins.* First ascertain the type of food (table 1 of § 176.170(c) of this chapter) that is being packed or used in contact with the finished food-contact article described in paragraph (a) of this section, and also ascertain the normal conditions of thermal treatment used in packaging or contacting the type of food involved. Using table 2 of § 176.170 (c) of this chapter, select the food-simulating solvent or solvents and the time-temperature test conditions that correspond to the intended use of the finished food-contact article. Having selected the appropriate food-simulating solvent or solvents and time-temperature exaggeration over normal use, follow the applicable extraction procedure.

(2) *Selection of extractability conditions for ionomeric resins.* Using table I of

[1] Average of four separate values, no single value of which differs from the average of those values by more then ±10 percent.

[2] Average of four separate values, no single value of which differs from the average of those values by more than ±50 percent.

[3] See footnote 2 to paragraph (d)(2)(i) of this section.

paragraph (f) of this section ascertain the type of food that is being packed or used in contact with the finished food-contact article described in paragraph (b) of this section, and also ascertain the food-simulating solvent or solvents that correspond to the intended use of the finished food-contact article.

(i) *Abbreviated test.* For intended use involving food contact at or below 120 °F (49 °C), the appropriate food-simulating solvent is to contact the food-contact film for the time and temperatures as follows:

Solvent	Time	Temperature
n-Heptane	[1] 2	120 °F (49 °C).
Water, 3% acetic acid, or 8%/50% ethanol.	[1] 48	120 °F (49 °C).

[1] Hours

(ii) *Equilibrium test.* For intended use involving food contact at or below 120 °F (49 °C), the appropriate food-simulating solvent is to contact the food-contact film at a temperature of 120 °F until equilibrium is demonstrated.

Solvent	Minimum extraction times (hours)
n-Heptane	8, 10, 12
Water, 3% acetic acid, or 8%/50% ethanol	72, 96, 120

The results from a series of extraction times demonstrate equilibrium when the net chloroform-soluble extractives are unchanging within experimental error appropriate to the method as described in paragraphs (d) (1)(i) and (2)(i) of this section. Should equilibrium not be demonstrated over the above time series, extraction times must be extended until three successive unchanging values for extractives are obtained. In the case where intended uses involve temporary food contact above 120 °F, the food-simulating solvent is to be contacted with the food-contact article under conditions of time and temperature that duplicate the actual conditions in the intended use. Subsequently the extraction is to be continued for the time period and under the conditions specified in the above table.

(3) *Reagents*—(i) *Water.* All water used in extraction procedures should be freshly demineralized (deionized) distilled water.

(ii) *n-Heptane.* Reagent grade, freshly redistilled before use, using only material boiling at 208 °F (97.8 °C).

(iii) *Alcohol.* 8 or 50 percent (by volume), prepared from undenatured 95 percent ethyl alcohol diluted with demineralized (deionized), distilled water.

(iv) *Chloroform.* Reagent grade, freshly redistilled before use, or a grade having an established, consistently low blank.

(v) *Acetic acid.* 3 percent (by weight), prepared from glacial acetic acid diluted with demineralized (deionized), distilled water.

(4) *Selection of test method.* The finished food-contact articles shall be tested either by the extraction cell described in the *Journal of the Association of Official Agricultural Chemists,* Vol. 47, No. 1, p. 177–179 (February 1964), also described in ASTM method F34–76 (Reapproved 1980), ''Standard Test Method for Liquid Extraction of Flexible Barrier Materials,'' which are incorporated by reference, or by adapting the in-container methods described in § 175.300(e) of this chapter. Copies of the material incorporated by reference are available from the Center for Food Safety and Applied Nutrition (HFS–200), Food and Drug Administration, 5100 Paint Branch Pkwy., College Park, MD 20740, and the American Society for Testing Materials, 1916 Race St., Philadelphia, PA 19103, respectively, or may be examined at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC 20408.

(5) *Selection of samples.* Quadruplicate samples should be tested, using for each replicate sample the number of finished articles with a food-contact surface nearest to 100 square inches.

(6) *Determination of amount of extractives*—(i) *Total residues.* At the end of the exposure period, remove the test container or test cell from the oven, if any, and combine the solvent for each replicate in a clean Pyrex (or equivalent) flask or beaker, being sure to rinse the test container or cell with a small quantity of clean solvent. Evaporate the food-simulating solvents to about 100 milliliters in the flask, and transfer to a clean, tared evaporating dish (platinum or Pyrex), washing the flask three times with small portions

of solvent used in the extraction procedure, and evaporate to a few milliliters on a nonsparking, low-temperature hotplate. The last few milliliters should be evaporated in an oven maintained at a temperature of 221 °F (105 °C). Cool the evaporating dish in a desiccator for 30 minutes and weigh the residues to the nearest 0.1 milligram, *e*. Calculate the extractives in milligrams per square inch of the container or material surface.

(*a*) *Water, 3 percent acetic acid, and 8 percent and 50 percent alcohol.* Milligrams extractives per square inch=*e/s*.

(*b*) *Heptane.* Milligrams extractives per square inch=*(e)/(s)(F)*

where:

e=Milligrams extractives per sample tested.

s=Surface area tested, in square inches.

F=Five, the ratio of the amount of extractives removed by heptane under exaggerated time-temperature test conditions compared to the amount extracted by a fat or oil under exaggerated conditions of thermal sterilization and use.

e′=Acidified chloroform-soluble extractives residue. *e′* is substituted for *e* in the above equations when necessary (See paragraph (e)(6)(ii) of this section for method to obtain *e′*).

If when calculated by the equations in paragraphs (e)(6)(i) (a) and (b) of this section, the extractives in milligrams per square inch exceed the limitations prescribed in paragraphs (c) or (d) of this section, proceed to paragraph (e)(6)(ii) of this section (method for determining the amount of acidified chloroform-soluble extractives residue).

(ii) *Acidified chloroform-soluble extractives residue.* Add 3 milliliters of 37 percent ACS reagent grade hydrochloric acid and 3 milliliters of distilled water to the evaporating dish containing the dried and weighed residue, *e*, obtained in paragraph (e)(6)(i) of this section. Mix well so every portion of the residue is wetted with the hydrochloric acid solution. Then add 50 milliliters of chloroform. Warm carefully, and filter through Whatman No. 41 filter paper (or equivalent) in a Pyrex (or equivalent) funnel, collecting the filtrate in a clean separatory funnel. Shake for 1 minute, then draw off the chloroform layer into a clean tared evaporating dish (platinum or Pyrex). Repeat the chloroform extraction, washing the dish, the filter paper, and the separatory funnel with this second portion of chloroform. Add this filtrate to the original filtrate and evaporate the total down to a few milliliters on a low-temperature hotplate. The last few milliliters should be evaporated in an oven maintained at 221 °F. Cool the evaporating dish in a desiccator for 30 minutes and weigh to the nearest 0.1 milligram to get the acidified chloroform-soluble extractives residue, *e′*. This *e′* is substituted for *e* in the equations in paragraphs (e)(6)(i) (a) and (b) of this section.

(f) The types of food and appropriate solvents are as follows:

TABLE 1

Types of food	Appropriate solvent
1. Nonacid (pH above 5.0), aqueous products; may contain salt or sugar or both, and including oil-in-water emulsions of low- or high-fat content.	Water, *n*-heptane.
2. Acidic (pH 5.0 or below), aqueous products; may contain salt or sugar or both, and including oil-in-water emulsions of low- or high-fat content.	*n*-heptane, water, 3% acetic acid.
3. Aqueous, acid or nonacid products containing free oil or fat; may contain salt, and including water-in-oil emulsions of low- or high-fat content.	Water, *n*-heptane, 3% acetic acid.
4. Dairy products and modifications:	
Water, *n*-heptane.	
i. Water-in-oil emulsions, high or low fat.	
ii. Oil-in-water emulsions, high or low fat.	
5. Low moisture fats and oils	*n*-heptane.
6. Beverages:	
i. Containing up to 8% alcohol	8% ethanol/water.
ii. Nonalcoholic	3% acetic acid.
iii. Containing more than 8% alcohol.	50% ethanol/water.
7. Bakery products	Water, *n*-heptane.
8. Dry solids (without free fat or oil).	No extraction test required.
9. Dry solids (with free fat or oil) ..	*n*-heptane.

(g) The provisions of paragraphs (c) and (d) of this section are not applicable to the ionomeric resins that are used in food-packaging adhesives complying with § 175.105 of this chapter.

[45 FR 22916, Apr. 4, 1980, as amended at 49 FR 10108, Mar. 19, 1984; 49 FR 37747, Sept. 26, 1984; 53 FR 44009, Nov. 1, 1988; 54 FR 24898, June 12, 1989]