

COEI-1-RESECA Cation exchange resins

1. Objective, origin and scope of application

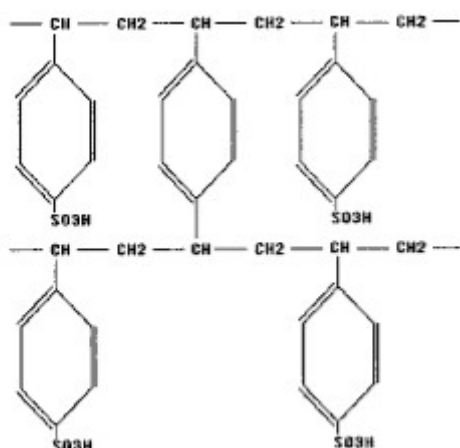
Ion exchange is the reversible exchange of ions between a liquid and a solid, during the course of which the solid does not undergo any substantial changes. When this technique is applied to wine, the solid is an insoluble, permeable synthetic resin capable of exchanging ions with the wine with which it is in contact.

These resins are used in the tartaric stabilization of wine.

2. Composition

Cation exchange resins may be prepared in an appropriate physical form using one or more of the following formulas.

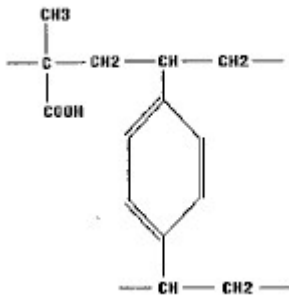
Sulfonated styrene-divinylbenzene copolymer:



Divinylbenzene-methacrylic acid copolymer

INTERNATIONAL OENOLOGICAL CODEX

RESINES ECHANGEUSES DE CATIONS



Resin inertia must be satisfactory.

The substances which can be used in the manufacture of these resins are indicated in Annexes 1 and 2.

The resin should not contain more than 1 mg of extractable organic substance per kg. These organic extracts are obtained with each of the following solvents: a) distilled water, b) alcohol, 15% by volume, c) 3% acetic acid solution (m/m).

The resin must have been washed and conditioned in accordance with the manufacturer's instructions.

Prepare different ion exchange columns for each solvent, using 50 ml of the resin that has previously been weighed.

While maintaining the maximum temperature that may be encountered during use, pass the three solvents used in the analysis (distilled water, 15% hydroalcoholic solution and 3% acetic acid solution (m/m)) through the resins at a flow rate of 350-450 ml per hour.

The first liter of effluent from each solvent should not be considered for analytical purposes; only the following two liters of each solvent should be used to analyze the organic extracts.

Total extract: The two liter sample should be evaporated at 105 °C until a constant weight is obtained.

Ash: This dry residue derived from evaporation of the 2 liters of effluent is then burned in an oven at 850 °C until a constant weight is obtained.

Organic extract: Total extract minus total ash gives the organic extract. If the organic extract is greater than 1 ml/l of solvent used, a "blank" should be made using the solvent and a correction should be made by subtracting the organic extract found in the "blank" from that obtained during the resin test. The solvents used are prepared as follows:

INTERNATIONAL OENOLOGICAL CODEX

RESINES ECHANGEUSES DE CATIONS

Control reagents: Distilled and/or de-ionized water.

Ethyl alcohol at 15% by volume obtained from absolute ethyl alcohol and distilled and/or de-ionized water.

3% acetic acid produced by mixing 3 parts (by mass) of acetic acid with 97 parts (by mass) of distilled and/or de-ionized water.

3. Limits

- The treatment must not alter the nature of the wine.
- The treatment must not reduce the color intensity of the wine.
- The treatment must not decrease the concentration of metallic cations in the wine below 300 mg/l.
- The treatment must not lower the wine's pH below 3.0. The decrease in pH should not exceed 0.3 pH units.
- The resin must not leave substances in the wine or impart to it characteristics (as a result of the resin-based treatment) that do not ordinarily exist in wine.
- The winemaker may use conditioning agents and/or regenerants composed of water and inorganic acids, bases or salts, provided that the conditioned or regenerated resin is washed in water until all conditioning agents and regenerants are removed before adding the wine.

Annex 1: List of substances used in the manufacture of adsorbant ion-exchange resins used to condition foodstuffs.

| List 1 Substances assessed by an international organization | | | |
|---|--------|------------|--------------|
| Name | PM/REF | Case | Restrictions |
| Monomers and other Starting substances | | | |
| n-butyl acrylate | 10780 | 00141-32-2 | - |
| Ethyl acrylate | 11470 | 00096-33-3 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|-----------------------------------|-------|------------|-------------------|
| Acrylonitrile | 12100 | 00107-13-1 | SML = ND |
| | | | (DL = 0.02 mg/kg) |
| | | | SML = 15 mg/kg |
| Formaldehyde | 17260 | 00050-00-0 | |
| Methyl methacrylate | 21130 | 00080-62-6 | - |
| Methanol | 21550 | 00067-56-1 | - |
| Styrene | 24610 | 00100-42-5 | - |
| Chemical Modifiers | | | |
| Carbonic acid, salts | 42500 | - | - |
| Hydrochloric acid | 72640 | 07664-38-2 | - |
| Silicic acid, salts | 85980 | - | - |
| Sulfuric acid | 91920 | 07664-93-9 | - |
| Acetic anhydride | 10150 | 00108-24-7 | - |
| tert-butyl-4-hydroxyanisole (BHA) | 40720 | 25013-16-5 | SML=30 mg/kg |
| Diethylene triamine | 15790 | 00111-40-0 | SML= 5 mg/kg |
| Dimethylamine | 49225 | 00124-40-3 | SML=0.06 mg/kg |
| 2-(dimethylamino)ethanol | 49235 | 00108-01-0 | SML=18 mg/kg |
| Formaldehyde | 54880 | 00050-00-0 | SML=15 mg/kg |
| Hexamethylenediamine | 18460 | 00124-09-4 | SML=2.4 mg/kg |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|---|-------|------------|------------------|
| Potassium hydroxide | 81600 | 01310-58-3 | - |
| Sodium hydroxide | 86720 | 01310-73-2 | - |
| Sodium nitrite | 86920 | 07632-00-0 | SML=0.6 mg/kg |
| Ethylene oxide | 17020 | 00075-21-8 | MQ=1 mg/kg in FP |
| 2-propanol | 81882 | 00067-63-0 | - |
| Polymerization Additives | | | |
| Akylsulfonic acids (C ₈ -C ₂₂) | 34230 | - | SML=6 mg/kg |
| Linear, primary alkylsulfuric | 34281 | - | - |
| acids (C₈-C₂₂) having an even number of carbon atoms | | | |
| Formic acid | 55040 | 00064-18-6 | - |
| Carboxymethylcellulose | 42640 | 09000-11-7 | - |
| Stannic chloride(IV) | 93420 | 07646-78-8 | - |
| Methylene chloride | 66620 | 00075-09-2 | SML=0.05 mg/kg |
| 1,4-dihydroxybenzene | 48620 | 00123-31-9 | SML=0.6 mg/kg |
| Gelatin | 55440 | 09000-70-8 | - |
| Ammonium hydroxide | 35600 | 01336-21-6 | - |
| Magnesium hydroxide | 64640 | 01309-42-8 | - |
| Hydroxyethylcellulose | 60560 | 09004-62-0 | - |
| Hydroxethylmethylcellulose | 60880 | 09032-42-4 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|------------------------------|-------|------------|---------------|
| Methanol | 65960 | 00067-56-1 | - |
| Methylcarboxymethylcellulose | 66200 | 37206-01-2 | - |
| Methyl isobutyl ketone | 66725 | 00108-10-1 | SML=5 mg/kg |
| Toluene | 93540 | 00108-88-3 | SML=1.2 mg/kg |

Annex 2: Substances that may be used provisionally to manufacture ion-exchange resins.

List 2

| Substances not fully evaluated by an international organization | | | |
|---|--------|------------|--------------|
| Name | PM/Ref | Case | Restrictions |
| Monomers and other starting substances | | | |
| Ethylene glycol dimethacrylate | 20440 | 00097-90-5 | - |
| Divinylbenzene | 16690 | 01321-74-0 | - |
| Diallyl ether of 1,1,1-trimethylolpropane | 25645 | 00682-09-7 | - |
| 2,3-epoxypropyl methacrylate | 20590 | 00106-91-2 | - |
| 2-methyl-1,3-butadiene | 21640 | 00078-79-5 | - |
| 1,7-octadiene | 22585 | 03710-30-3 | - |
| 1,1,1-trimethylolpropane trimethacrylate | 25840 | 03290-92-4 | - |
| Chemical Modifiers | | | |
| N,N-dimethyl-1,3-diaminopropane | 49380 | 00109-55-7 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|---|--------|------------|--------------|
| Triethylamine | 95270 | 00121-44-8 | - |
| Triethylene tetramine | 25520 | 00112-24-3 | - |
| Polymerization Additives | | | |
| Polyvinyl alcohols | 81280 | 09002-89-5 | - |
| 4-tert-butylcatechol | 40640 | 00098-29-3 | - |
| Diisobutyl ketone | 49050 | 00108-83-8 | - |
| Sodium hypochlorite | 62110 | 07681-52-9 | - |
| Isobutanol | 62270 | 00078-83-1 | - |
| 4-methoxyphenol | 66030 | 00150-76-5 | - |
| Methylene bis(sodium naphtha- lenesulfonate) | 66600 | 26545-58-4 | - |
| 2-methyl-2-pentanol | 66860 | 00108-11-2 | - |
| Dibenzoylperoxide | 46440 | 00094-36-0 | |
| Partially hydrolyzed vinyl polyacetate | 81260 | - | - |
| Substances not evaluated by an international organization | | | |
| Name | PM/Ref | Case | Restrictions |
| Monomers and other starting substances | | | |
| Dimethoxymethane | - | 00109-87-5 | - |
| Diethylene glycol divinyl ether | - | 00764-99-8 | |
| Ethyl vinyl benzene | - | 28106-30-1 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|---|--------|------------|---------------|
| 1,2,4-trivinyl cyclohexane | - | 02855-27-8 | - |
| Chemical Modifiers | | | |
| Chlorosulfonic acid | - | 07790-94-5 | - |
| Monochloroacetic acid | - | 00079-11-8 | - |
| Phosphoric acid | - | 13598-36-2 | - |
| Bromine | - | 07726-95-6 | - |
| 2-chloroethanol | - | 00107-07-3 | - |
| Methyl chloride | - | 00074-87-3 | - |
| 1,2-dichloroethane | - | 00107-07-3 | - |
| 1,2-dichloropropane | - | 00078-87-5 | - |
| 3-(dimethylamino)propane | - | 03179-63-3 | - |
| Name | PM/Ref | Case | Prescriptions |
| Monomers and other starting substances | | | |
| Methylic chloromethyl ether | - | 00107-30-2 | - |
| Nitrobenzene | - | 00098-95-3 | - |
| Potassium nitrite | - | 07758-09-0 | - |
| Phthalimide | - | 0085-41-6 | - |
| Sulfur trioxide | - | 07446-11-9 | - |
| Trimethylamine | - | 00075-50-3 | - |
| Polymerization additives | | | |
| Lignosulfonic acid | 63940 | 08062-15-5 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|--|-------|------------|---|
| Peracetic acid | - | 00079-21-0 | - |
| Polyacrylic acid | 76460 | 09003-01-4 | - |
| Poly(styrenesulfonic) acid | - | 09080-79-9 | - |
| Acrylamide/acrylic acid copolymer | - | 09003-06-9 | - |
| Ethoxylated, propoxylated tert-alkylamines (C ₁₂ -C ₁₄) | - | 68603-58-7 | - |
| Maleic anhydride-styrene copolymer, ammonium salt | - | 26022-09-3 | - |
| Attapulgate | - | 12174-11-7 | - |
| Azobisisobutyronitrile | - | 00078-67-1 | - |
| 1,1-bis(tert-butylperoxy)-3,3,5-trimethylcyclohexane | - | 06731-36-8 | - |
| n-Dodecyl mercaptan | - | 25103-58-6 | - |
| Poly(ethylene/propylene)glycol monobutyl ester | - | 09038-95-3 | - |
| Polyethylene glycol octylphenyl ether | 78560 | 09002-93-1 | - |
| Poly(ethylene-propylene/glycol ether with 1,1,1-trimethylolpropane | - | 52624-57-4 | - |
| tert-hexadecyl mercaptan | - | 25360-09-2 | - |
| Cumyl hydroperoxide | - | 00080-15-9 | - |
| Isododecane | 62405 | 31807-55-3 | - |
| Isooctane | - | 26635-64-3 | - |

INTERNATIONAL OENOLOGICAL CODEX
RESINES ECHANGEUSES DE CATIONS

| | | | |
|--|-------|------------|---|
| Mono- and dialkyl (C ₁₀ -C ₁₈) Sulfonamides | - | - | - |
| Silver nitrate | - | 07761-88-8 | - |
| n-Octane | - | 00111-65-9 | - |
| tert-Butyl peracetate | - | 00107-71-1 | - |
| tert-Butyl perbenzoate | - | 00614-45-9 | - |
| bis(4-tert-butylcyclohexyloxy) percarbonate tert | - | 15520-11-3 | - |
| Butyl per(2-ethyl-hexanoate) | - | 03006-82-6 | - |
| tert-Butyl peroctanoate | - | 13467-82-8 | - |
| Dilauroyl peroxide | - | 00105-74-8 | - |
| Poly(diallyldimethylammonium chloride) | - | 26062-79-3 | - |
| Polyvinylpyrrolidone | 81500 | 09003-39-8 | |