ver. 2014/12/05

Softening point: In conformity with JIS K7234

Total chlorine: In conformity with JIS K7243-3 (ISO 21672-3)

Chlorine ion: In conformity with JIS K7243-1 (ISO 21672-1)

Sodium ion: Measured by means of ion chromatography

Iron: ICP emission spectroscopy

Refractive index: In conformity with ISO 5661

Gardner color scale: In conformity with ISO 4630-1

GPC:

Column (Shodex KF-603, KF-602 × 2, KF-601 × 2)

Consolidated eluent: Tetrahydrofuran

Flow rate: 0.5 mL/min

Column temperature: 40°C

Detection: RI (refractive index detector)

[0055]

Example 1

A one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device was once evacuated in vacuum and substituted with nitrogen (oxygen concentration: 4.9%); and thereafter, 256 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP, ~~purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm~~), 842 parts of epichlorohydrin, and 180 parts of methanol were added while purging with nitrogen (2 L/hr), and a water bath was subjected to temperature rise to 75°C. When the internal temperature exceeded 65°C, 21 parts of flaky sodium hydroxide was added dividedly over 90 minutes, and thereafter, a post-reaction was further performed at 70°C for one hour. After completion of the reaction, water washing was performed, and the excessive solvent such as epichlorohydrin, etc. was distilled off from an oil layer at 140°C under reduced pressure by using a rotary evaporator. To the residue, 600 parts of methyl isobutyl ketone was added for dissolution, and the resulting solution was subjected to temperature rise to 70°C. 26 parts of a 30% by weight sodium hydroxide aqueous solution was added under stirring, and the contents were allowed to react with each other for one hour. Thereafter, water washing was performed until the washings became neutral, and as for the obtained solution, the methyl isobutyl ketone and the like were distilled off at 180°C under

reduced pressure by using a rotary evaporator, thereby obtaining 305 parts of an epoxy resin (EP1). The obtained epoxy resin had an epoxy equivalent of 266 g/eq., a softening point of 89°C, an ICI melt viscosity of 0.42 Pa·s (at 150°C), a total chlorine content of 1,600 ppm, a hydrolyzable chlorine content of 1,540 ppm, a chlorine ion content of 2 ppm, a sodium ion content of 0.5 ppm, and a hue of not more than 0.2 (Gardner in a 40% MEK (methyl ethyl ketone) solution). In addition, the structure of the foregoing formula (1) accounted for 93 area% (GPC).

[0056]

Example 2

A reaction was performed in the same manner as that in Example 1, except that a one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device was once evacuated in vacuum and substituted with nitrogen (oxygen concentration: 5.3%), and thereafter, a phenol compound (DPPI2) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP (purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), to which 500 ppm of phenolphthalein had been added) was used while purging with nitrogen (2 L/hr). The obtained epoxy resin (EP2) had an epoxy equivalent of 266 g/eq., a softening point of 90°C, an ICI melt viscosity of 0.44 Pa·s (at 150°C), a total chlorine content of 2,000 ppm, a hydrolyzable chlorine content of 1,950 ppm, a chlorine ion content of 1 ppm, a sodium ion content of 0.3 ppm, and a hue of not more than 0.2 (Gardner in a 40% MEK solution). In addition, the structure of the foregoing formula (1) accounted for 93 area% (GPC).

[0057]

Example 3

A one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device was substituted with nitrogen (oxygen concentration: 6.5%) at 4 L/h for 30 minutes; and thereafter, 256 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP (purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), 661 parts of epichlorohydrin, and 165 parts of methanol were added while purging with nitrogen (2 L/hr), and a water bath was subjected to temperature rise to 75°C. When the internal temperature exceeded 65°C, 57 parts of flaky sodium hydroxide was added dividedly over 90 minutes, and thereafter, a post-reaction was

further performed at 70°C for one hour. After completion of the reaction, water washing was performed, and the excessive solvent such as epichlorohydrin, etc. was distilled off from an oil layer at 140°C under reduced pressure by using a rotary evaporator. To the residue, 600 parts of methyl isobutyl ketone was added for dissolution, and the resulting solution was subjected to temperature rise to 70°C. 5 parts of a 30% by weight sodium hydroxide aqueous solution was added under stirring, and the contents were allowed to react with each other for one hour. Thereafter, water washing was performed until the washings became neutral, and as for the obtained solution, the methyl isobutyl ketone and the like were distilled off at 180°C under reduced pressure by using a rotary evaporator, thereby obtaining 297 parts of an epoxy resin (EP3). The obtained epoxy resin had an epoxy equivalent of 277 g/eq., a softening point of 96°C, an ICI melt viscosity of 0.62 Pa·s (at 150°C), a total chlorine content of 2,230 ppm, a hydrolyzable chlorine content of 2,100 ppm, a chlorine ion content of 0.5 ppm, a sodium ion content of 0.5 ppm, and a hue of not more than 0.2 (Gardner in a 40% MEK solution). In addition, the structure of the foregoing formula (1) accounted for 82 area% (GPC).

[0058]

Example 4

A one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device was once evacuated in vacuum and substituted with nitrogen (oxygen concentration: 5.2%); and thereafter, 256 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP, purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), 661 parts of epichlorohydrin, and 200 parts of dimethyl sulfoxide were added while purging with nitrogen (2 L/hr), and a water bath was subjected to temperature rise to 45°C. When the internal temperature exceeded 40°C, 57 parts of flaky sodium hydroxide was added dividedly over 90 minutes, and thereafter, a post-reaction was further performed at 70°C for one hour. After completion of the reaction, water washing was performed, and the excessive solvent such as epichlorohydrin, etc. was distilled off from an oil layer at 140°C under reduced pressure by using a rotary evaporator. To the residue, 600 parts of methyl isobutyl ketone was added for dissolution, and the resulting solution was subjected to temperature rise to 70°C. 16 parts of a 30% by weight sodium hydroxide aqueous solution was added under stirring,

and the contents were allowed to react with each other for one hour. Thereafter, water washing was performed until the washings became neutral, and as for the obtained solution, the methyl isobutyl ketone and the like were distilled off at 180°C under reduced pressure by using a rotary evaporator, thereby obtaining 300 parts of an epoxy resin (EP4). The obtained epoxy resin had an epoxy equivalent of 270 g/eq., a softening point of 92°C, an ICI melt viscosity of 0.48 Pa·s (at 150°C), a total chlorine content of 1,050 ppm, a hydrolyzable chlorine content of 960 ppm, a chlorine ion content of 0.3 ppm, a sodium ion content of 0.1 ppm, and a hue of 0.6 (Gardner in a 40% MEK solution). In addition, the structure of the foregoing formula (1) accounted for 90 area% (GPC).

[0059]

Example 5

A reaction was performed in the same manner as that in Example 1, except that a flask equipped with a stirrer, a reflux condenser, and a stirring device was once evacuated in vacuum and substituted with nitrogen, and thereafter, a phenol compound (DPPI3) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP (purity: 99% or more, residual phenolphthalein: 210 ppm, iron: <5 ppm), to which 1% of phenolphthalein had been added) was used while purging with nitrogen (2 L/hr). The obtained epoxy resin (EP5) had an epoxy equivalent of 262 g/eq., a softening point of 89°C, an ICI melt viscosity of 0.42 Pa·s (at 150°C), a total chlorine content of 3,200 ppm, a hydrolyzable chlorine content of 2,700 ppm, a chlorine ion content of 5 ppm, a sodium ion content of 0.5 ppm, and a hue of 3 (Gardner in a 40% MEK solution). In addition, the structure of the foregoing formula (1) accounted for 90 area% (GPC).

[0060]

Comparative Example 1

In a flask equipped with a stirrer, a reflux condenser, and a stirring device, 256 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC PPPBP (purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), 842 parts of epichlorohydrin, and 3 parts of benzyltrimethylammonium chloride were added, and a water bath was subjected to temperature rise to 70°C. 100 parts of a 49% sodium hydroxide aqueous solution was added dropwise thereto over 90 minutes, and thereafter, a post-reaction

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(amended page)

[0070]

Example 13

In a one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device, 295 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC BPPBP, purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), 694 parts of epichlorohydrin, and 173 parts of dimethyl sulfoxide were added, and a water bath was subjected to temperature rise to 50°C. When the internal temperature exceeded 45°C, 66 parts of flaky sodium hydroxide was added dividedly over 90 minutes, and thereafter, a post-reaction was further performed at 45°C for 2 hours and subsequently at 70°C for one hour. After completion of the reaction, water washing was performed, and the excessive solvent such as epichlorohydrin, etc. was distilled off from an oil layer at 140°C under reduced pressure by using a rotary evaporator. To the residue, 760 parts of methyl isobutyl ketone was added for dissolution, sodium chloride and the like formed by water washing were removed, and thereafter, an organic layer was subjected to temperature rise to 70°C. 30 parts of a 30% by weight sodium hydroxide aqueous solution was added under stirring, and the contents were allowed to react with each other for one hour. Thereafter, water washing was performed until the washings became neutral, and as for the obtained solution, the methyl isobutyl ketone and the like were distilled off at 180°C under reduced pressure by using a rotary evaporator, thereby obtaining 355 parts of an epoxy resin (EP10). The obtained epoxy resin had an epoxy equivalent of 268 g/eq., a softening point of 99°C, an ICI melt viscosity of 1.03 Pa·s (at 150°C), a total chlorine content of 305 ppm, a hydrolyzable chlorine content of 270 ppm, a chlorine ion content of 0.1 ppm, a sodium ion content of 0.1 ppm, and a hue of 0.6 (Gardner in a 40% THF solution). In addition, the structure of the foregoing formula (1) accounted for 85.2 area% (GPC).

[0071]

Example 14

In a one-liter four-necked flask equipped with a stirrer, a reflux condenser, and a stirring device, 295 parts of a phenol compound (DPPI1) (a compound represented by the foregoing formula (P), wherein all the substituent Rs are a hydrogen atom, SABIC BPPBP, purity: 99% or more, residual phenolphthalein: 200 ppm, iron: <5 ppm), 971 parts of epichlorohydrin, and 165 parts of dimethyl sulfoxide were added, and a water