4210 Determination of vinyl chloride monomer and vinylidene chloride monomer in plastics

This method applies to determine residual vinyl chloride monomer in polyvinyl chloride products and residual vinylidene chloride monomer in polyvinylidene chloride products for pharmaceutical packaging.

This method based on gas-liquid equilibrium, the specimen is dissolved in a suitable solvent in a sealed container. At a certain temperature, vinyl chloride monomer and vinylidene chloride monomer diffuse into space, and after reaching equilibrium, take a quantitative headspace gas and inject it into the gas chromatograph, characterize it by retention time and quantify it by peak area.

Carry out the method for gas chromatography <0512>.

Chromatographic conditions and system suitability tests The stationary phase is a capillary column of polyethylene glycol or (6%) cyanopropylphenyl-(94%) dimethylsiloxane or a capillary column of similar polarity; procedure of heating, the initial temperature of 40□ hold for 12 min, increase to 200□ at 60□/min, maintain for 5 min, and then decrease the temperature to 40□ at 60□/min, maintain for 2 min; the temperature of the gasification chamber is 190□, and the flame ionization detector temperature is 210°C; the flow rate of the carrier gas is 0.8ml/min (recommended, which can be adjusted according to the selected chromatographic column).

In the chromatogram of the reference solution, the resolution of the peaks of each component should meet the requirements.

Reference solution Take five 20ml headspace vials, add 3ml of N,N-dimethylacetamide (DMAC) to each of them beforehand, accurately measure an appropriate amount of vinyl chloride monomer and vinylidene chloride monomer reference solution, quickly inject into the headspace vials, press the cap to seal, and mix well with shaking to form a series of mixed reference solutions containing 0.5μg, 1.0μg, 2.0μg, 3.0μg, 4.0μg of vinyl chloride monomer and 1.5μg, 2.0μg, 3.0μg, 4.0μg, 5.0μg of vinylidene chloride monomer.

Test sample Cut the sample into small particles of about 0.3cm×0.3cm, weigh accurately 1.0g, put it into a 20ml headspace vial, accurately add 3ml of DMAC, quickly press the cap to seal, and shake to dissolve or expand fully, then get the test solution. Parallel preparation of 2 copies.

Determination Equilibrate the headspace vials containing the reference solution and test sample solution, in a headspace oven at 70°C±1°C for 30 minutes. Take a quantitative amount of headspace gas and inject into the gas chromatograph. Record the chromatogram.

Make a linear regression between the content of vinyl chloride monomer and vinylidene chloride monomer reference solution and the corresponding peak area respectively, obtain the linear regression equation (the correlation coefficient should

not be less than 0.99), and calculate the content of vinyl chloride monomer and vinylidene chloride monomer in the test sample (μg/g).

Note： 1. If only determine the content of vinyl chloride monomer or vinylidene chloride monomer, the preparation of the reference solution can be selected from a reference product, there is no need to prepare a mixed reference solution.

2. The preparation of the reference solution should be operated in a fume hood.

3. Because of the low boiling point of the reference product, it is very easy to volatilize and lead to changes in concentration, it should be sampled quickly and at low temperatures (e.g., in an ice bath).

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