

Technical Information MERACRYL MAamide

Determination of Methacrylic acid in Methacryl amide by HPLC

Principle

Determination of methacrylic acid in Methacryl amide by means of HPLC.

Terms

HPLC: high-performance liquid chromatography
RP: reverse-phase chromatography

Eluent composition

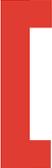
Mix 960 ml demineralised water thoroughly with 40 ml acetonitrile and adjust the mixture to a pH of 2.5 with o-phosphoric acid. Then degas in an ultrasonic bath for 5 minutes.

Sample preparation

Weigh approx. 500 mg of sample accurate to 1 mg into a 50ml volumetric flask, dissolve in approx. 45 ml of demineralised water, heat to 20 °C and fill to the mark. Mix well and inject. Enter the sample weight in "g" und "Calc. method", "sample quantity".

Test conditions chromatography

Apparatus:	HPLC chromatography system by Merck/Hitachi
HPLC column:	LiChrospher 100 RP-8 (5 µm) 125-4 mm 4-4 cartridge precolumn
Eluant:	water/acetonitrile 96/4, pH 2.5
Flow rate:	1.5 ml/min
Pressure:	95 bar
Detector:	210 nm
Sample quantity:	20 µl solution



Technical Information

MERACRYL MAamide

Calibration

The quantity of the calibration standard depends on the actual methacrylic acid content of the Methacryl amide and must be prepared by intermediate dilution.

1. Preparation of the methacrylic acid stock solution: Weigh approx. 100 mg accurate to 0.1 mg into a 50 ml volumetric flask, dissolve in approx. 40 ml demineralised water, fill to the mark at 20 °C and mix well.
2. Dilution: weight 5ml from the stock solution into a 50ml flask, dissolve in demineralised water, fill to the mark at 20°C and mix well.

Methacrylic acid calibration standard: Transfer 1ml of the dilution to another 50ml volumetric flask and fill to the mark with water (20 °C).

The standard is best before one week in a brown volumetric flask.

Integration

Enter the concentration of the calibration standard in "mg" under "Table, Parameter Conc." Start a calibration run to calibrate the integrator with the data of the calibrating solution and to calculate the factor for determining the sample concentration. The integrator will then automatically perform the quantitative evaluation.

Technical Information

MERACRYL MAamide

General remarks

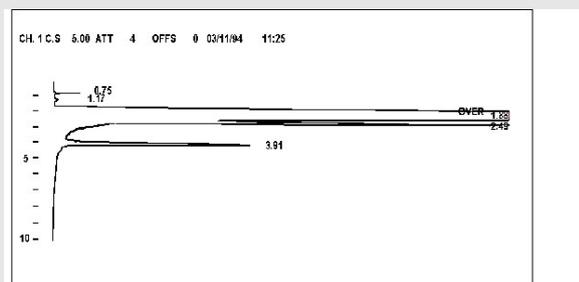
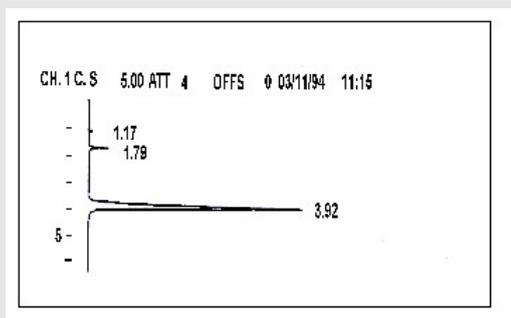
Principle of the method:

Separation of the sample occurs by RP chromatography. Using silylated silica gel as the non-polar stationary phase, the individual components are retained in the order of their increasing hydrophobic nature. A UV detector records every change in the polar mobile phase that is caused by the eluted components. A TEST RUN must be performed at the beginning of each new type of analysis and when varying the data acquisition rate (SEMP PERIOD). If the mobile phase contains acids, change the eluant once the analysis is completed. Rinse the pump, column and other wetted parts with a 75:25 acetonitrile/water mixture for approx. 10 minutes.

Chromatograms

Calibration standard: 0.180 mg MAS/50 ml water

Methacryl amide



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