Science Together

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Determination and quantification of acrylic acid derivatives

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SUMMARY

We are constantly exposed to acrylic monomers as part of our everyday lives. Diverse forms can be found at home, at work, on the street, or at the supermarket. End products based on acrylic monomers are utilized in many products from paints and lacquers to adhesives, water treatment products, and plastics to detergents, or textile fibers. In this application, four common acrylic acid derivatives were quantified with the AZURA® HPLC Plus system.

INTRODUCTION

Acrylate monomers used to form acrylate polymers are based on the structure of acrylic acid or are derivatives of it. Acrylic acid and some acrylate oligomers and monomers can affect human health as eye and skin irritants. Residual monomers might be exposed to consumers and that is why the content of residual monomers in acrylic polymers needs to be examined. Methyl methacrylate, 2-hydroxyethyl methacrylate, ethylhexyl acrylate, and isobornyl acrylate are examples of acrylic acid derivatives and were determined in this application.

Additional Information

Determination and quantification of acrylic acid derivatives

RESULTS

A mixed standard of the four acrylate monomers was used to determine a calibration with the following concentrations for each compound: 0.001 mg/mL, 0.002 mg/mL, 0.004 mg/mL, 0.01 mg/mL, and 0.02 mg/mL. The four detected peaks are baseline separated. **Fig 1** shows the chromatogram of the acrylate mix standard at a concentration of 0.01 mg/mL. For all compounds the limit of detection (LOD, S/N=3) and the limit of quantification (LOQ, S/N=10) were calculated based on the measurement of the lowest calibration concentration. **Tab 1** displays a summary of the determined quantification results.

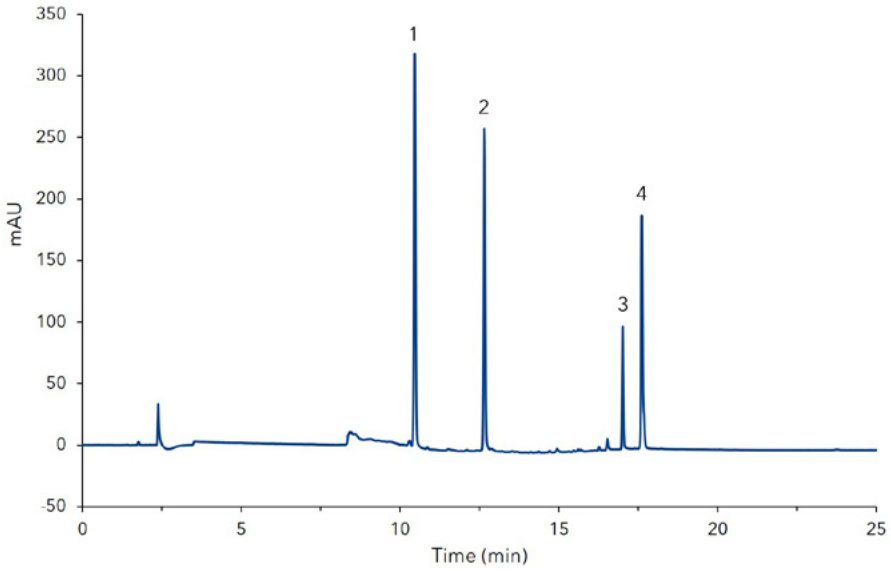


Fig. 1 Measurement of standard mix (0.01 mg/mL); 1) 2-Hydroxyethyl methacrylate, 2) Methyl methacrylate, 3) Ethylhexyl acrylate, 4) Isobornyl acrylate

Tab. 1 LOD and LOQ of acrylic monomers

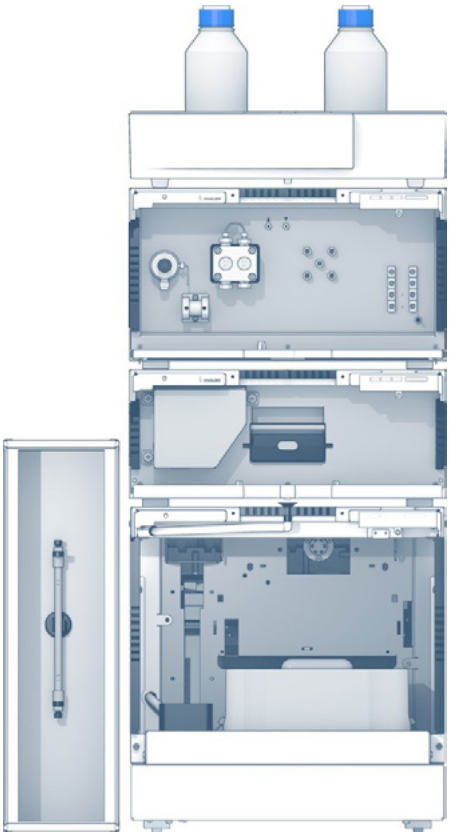
Substance	LOD (µg/mL)	LOQ (µg/mL)
2-Hydroxyethyl methacrylate	0.022	0.07
Methyl methacrylate	0.032	0.11
Ethylhexyl acrylate	0.075	0.25
Isobornyl acrylate	0.042	0.14

MATERIALS AND METHODS

All standards were provided by the Fraunhofer-Institut für Fertigungstechnik und Angewandte Materialforschung IFAM [2]. For this application an AZURA analytical system was used which consisted of an AZURA P 6.1L quaternary LPG pump, an AZURA DAD 2.1L diode array detector, an AZURA CT 2.1 column thermostat and an AZURA AS 6.1L autosampler. The flow was set to 1 mL/min at a column temperature of 40 °C. The detection wavelength was set to 210 nm. The sampling rate was set to 1 Hz and the time constant to 0.2 s. 10 µl of the standards were injected. The column with the dimensions 150 x 4.6 mm ID with precolumn was filled with Eurospher II 100-3 C18 silica.

CONCLUSION

The developed gradient consisted of two different elution steps. The first gradient step from water to acetonitrile separates the acrylic monomers. In the second step from acetonitrile to tetrahydrofuran, polyacrylates potentially present in the polyacrylate matrix can be eluted/washed from the column. These two steps are useful when both polar and non-polar acrylates are to be separated. Furthermore, this simplifies the sample preparation which in the end leads to a reduced analysis time.



REFERENCES

[1] http://www.acrylicmonomers.basf.com/portal/8/en/dt.jsp?page=basf_acrylic_monomers

[2] Fraunhofer-Institut für Fertigungstechnik und Angewandte Materialforschung IFAM

ADDITIONAL MATERIALS AND METHODS

Tab. A1 Method parameters

Eluent A	Water + 0.1 % phosphoric acid			
Eluent B	Acetonitrile			
Eluent C	Tetrahydrofuran			
Gradient	Time (min)	% A	% B	% C
	0	100	0	0
	5	100	0	0
	15	0	100	0
	25	0	100	0
	28	0	0	100
	38	0	0	100
	41	0	100	0
	51	0	100	0
	51.1	100	0	0
	60	100	0	0
Flow rate	1 mL/min	Run time	60 min	
Column temperature	40 °C	Injection mode	Partial loop	
Injection volume	10 µL	Data rate	1 Hz	
Detection wavelength	210 nm	Time constant	0.2 s	

Tab. A2 System configuration & data

Instrument	Description	Article No.
Pump	AZURA® P6.1L, LPG 10 mL	APH34EA
Autosampler	AZURA® AS 6.1L	AAA00AA
Detector	AZURA® DAD 2.1L	ADC01
Flow cell	PressureProof Cartridge 10mm, 10µL	AMC38
Column	Eurospher II 100-3 C18, Vertex Plus Column 150 x 4.6 mm ID with precolumn	15VE181E2G
Thermostat	AZURA® CT 2.1	A05852
Software	ClarityChrom 7.2	A1670-11

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