

Measurement report

1. Materials

Adsorbent mixture

Acetonitrile (Merck, Darmstadt, Germany)

Methanol (Fisher Scientific, Loughborough, UK)

Glyceryl-triacetate (Fluka Analytical, Steinheim, Germany)

Water (Millipore)

2. Aim of analysis

The aim of this analysis is determination the uptake capacity of an adsorbent mixture to methanol, acetonitrile.

3. Sample preparation

First two ATD tubes were filled with the adsorption mixture and then the weight of the tubes was measured with analytical scales. After then the tubes were attached to the evaporating arrangement, which can be seen in the Figure 1 and 2. After adequate amount of the solvent was placed in the evaporating bottle, the bottle of the measuring arrangement was placed in water bath (100 °C) and it was rinsed constantly with Nitrogen flow for 30 minutes. Then the contents of the tube were put in headspace vials and the adsorbent was suspended in 1 mL of glyceryl-triacetate to extract the analyte from the surface and then the vials were closed.

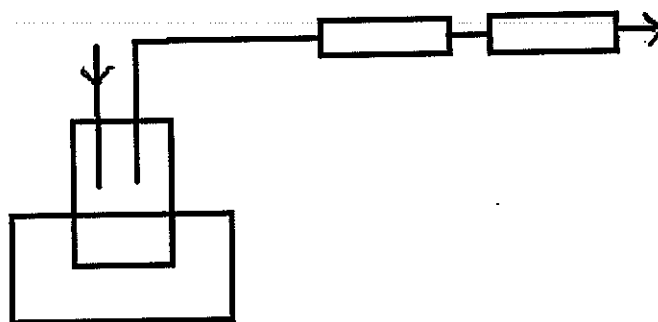


Figure 1 – Sematic ordeof the evaporating arrangement

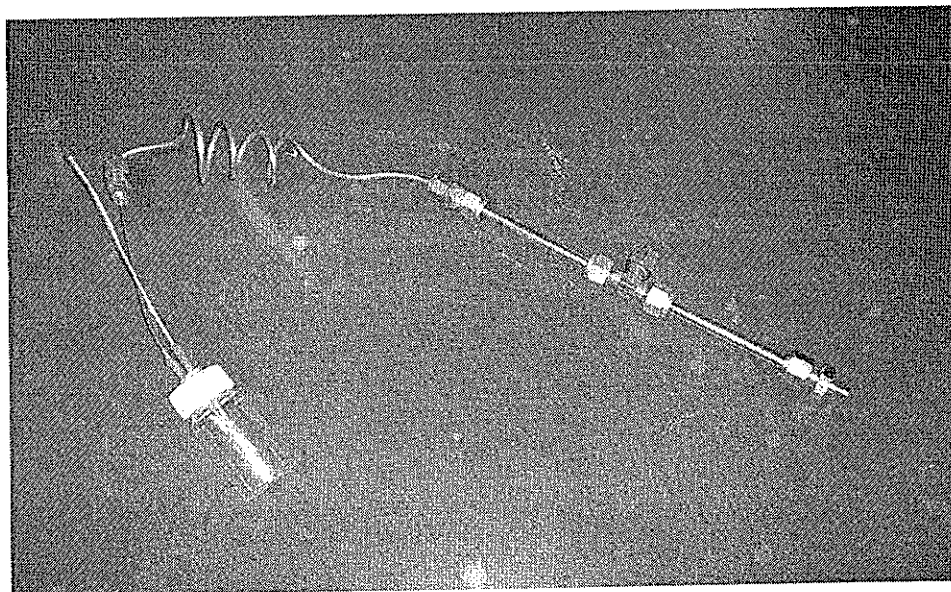


Figure 2 – Evaporating arrangement

4. HS-GC methods

Determination of acetonitrile:

Sample introduction: Perkin Elmer Headspace HS-40

Oven temperature: 90 °C

Needle temperature: 100 °C

Transfer line temperature 110 °C

Thermostating time: 15 min

Pressurization time: 1 min

Inject time: 0.04 min

Withdrawal time: 0.5 min

Gas chromatograph: Perkin Elmer Autosystem XL Gas chromatograph

Column: Phenomenex Zebron ZB-624, 60 m x 0,53 mm x 3 μ m

Oven program initial temperature: 50 °C

Hold time 1: 5 min

Ramp 1: 20 °C/min to 200 °C

Hold time 2: 2 min

Carrier gas: Nitrogen

Column pressure: 15 psi

Detector: FID, 200 °C

Nitrogen adsorption were measured at -196 °C with a Nova2000e (Quantachrome) computer controlled apparatus. The apparent surface area SBET was calculated using the Brunauer–Emmett–Teller (BET) model. Before measurement sample was outgassed for 24 h at 110 °C.

Surface are: 561m²/g

Determination of methanol:

Sample introduction: Perkin Elmer Headspace HS-40

Oven temperature: 75 °C

Needle temperature: 100 °C

Transfer line temperature 110 °C

Thermostating time: 15 min

Pressurization time: 1 min

Inject time: 0.04 min

Withdrawal time: 0.5 min

Gas chromatograph: Perkin Elmer Autosystem XL Gas chromatograph

Column: Phenomenex Zebron ZB-624, 60 m x 0,53 mm x 3 μ m

Oven program initial temperature: 50 °C

Hold time 1: 5 min

Ramp 1: 20 °C/min to 200 °C

Hold time 2: 2 min

Carrier gas: Nitrogen

Column pressure: 15 psi

Detector: FID, 200 °C

5. Results

Adsorption properties of the measured adsorbent to acetonitrile:

Weight of the adsorbent (g)		Volume of the acetonitrile (µl)	Weight of the acetonitrile (mg)	Area (µVs)	
1. tube	2. tube			1. tube	2. tube
0,4833	0,4525	100	78,6	523140	201
0,2822	0,2794	200	157,2	1673382	529
0,3064	0,3560	400	314,4	3249665	90958
0,2572	0,2183	600	471,6	3922472	1231701
0,4456	0,5113	700	550,2	3859732	107016

Table 1

Adsorption properties of the measured adsorbent to methanol:

Weight of the adsorbent (g)		Volume of the methanol (µl)	Weight of the methanol (mg)	Area (µVs)	
1. tube	2. tube			1. tube	2. tube
0,4207	0,2951	100	79,2	115764	7
0,4432	0,3065	300	237,5	814534	205
0,3289	0,4383	400	316,7	1064699	37477
0,4918	0,5548	500	395,9	1048309	135

Table 2

Analyst:

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Enikő Sipkó
PhD student

Controller:

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head of laboratory

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Appendix

Figure 1: Chromatogram of methanol

Figure 2: Chromatogram of acetonitrile

Figure 1: Chromatogram of methanol

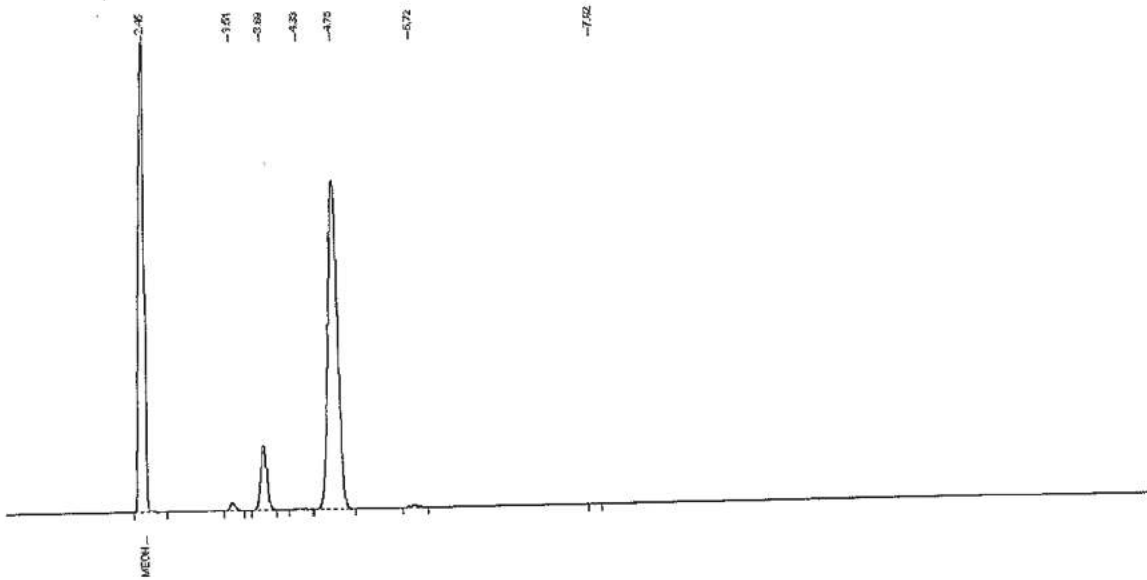


Figure 2: Chromatogram of acetonitrile

