

Separation of Organic Acids on a Hamilton Anion Micro Column using KOH Gradient Elution

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Abstract

A Hamilton Anion Micro column was used for the separation of organic acids using a potassium hydroxide (KOH) gradient. Beginning with a fast linear gradient the method was optimized to yield an acceptable separation of a mixture of typical organic and inorganic anion standards.

Introduction

In organic geochemistry the distribution, composition and transformation of organic material during the Earth's history is investigated. Molecular and microscopic fossils are the basis for reconstruction of prehistoric events and ecosystems. An important aspect of this research is the understanding of the formation processes of fossil fuel. Organic acids are important biomarkers which allow an inference on the organic precursors despite the molecular changes caused by e.g. the different conversion processes over the course of time. For positive identification of the organic acids a good separation of the different acid- and ion-species as well as a split-off from the matrix are essential. Ion chromatography is a convenient and fast technique for speciation of such components in a single analysis. A Hamilton Anion Fast column was used with a KOH gradient to evaluate the capabilities of this type of column for gradient separation.

Material & Methods

The separation of the sample was conducted on a Dionex ICS 3000 ion chromatograph equipped with a KOH eluent generator and suppressed conductivity detection. Data recording and processing was done with Dionex Chromeleon Software. A Hamilton Anion Micro ion exchange column with 2.1 x 250 mm PEEK housing and 5 µm Trimethylammonium (TMA)-modified PS-DVB particles was used for separation. The chemicals have been supplied by Merck KGaA (Darmstadt, Germany). The concentration of the ion standards is given in Table 1. The injection volume was 10 µL and the flow rate was 0.35 mL/min. The column temperature was 30°C for all experiments. The different gradients are shown in Table 2.

Anion Standard	Concentration [ppm]
Fluoride	2
Acetic acid	10
Formic acid	10
Chloride	5
Propionic acid	10

Table 1: Concentrations of injected species

Gradient No.	Time [min]	KOH Concentration [mM]
1	0	25
	13	100
	25	100
2	0	25
	20	100
	30	100

Table 2: Used KOH-gradient compositions

Anion Standard	Gradient 1	Gradient 2
	Retention Time [min]	Retention Time [min]
1. Fluoride	7.02	7.25
2. Acetic acid	10.75	11.25
3. Formic acid	11.15	11.75
4. Chloride	16.90	18.35
5. Propionic acid	18.75	19.80

Table 3: Retention times for different gradients

Results & Discussion

In Figure 1 the separation of the 5 anion standards with a fast linear gradient are shown (see Table 1, gradient 1).

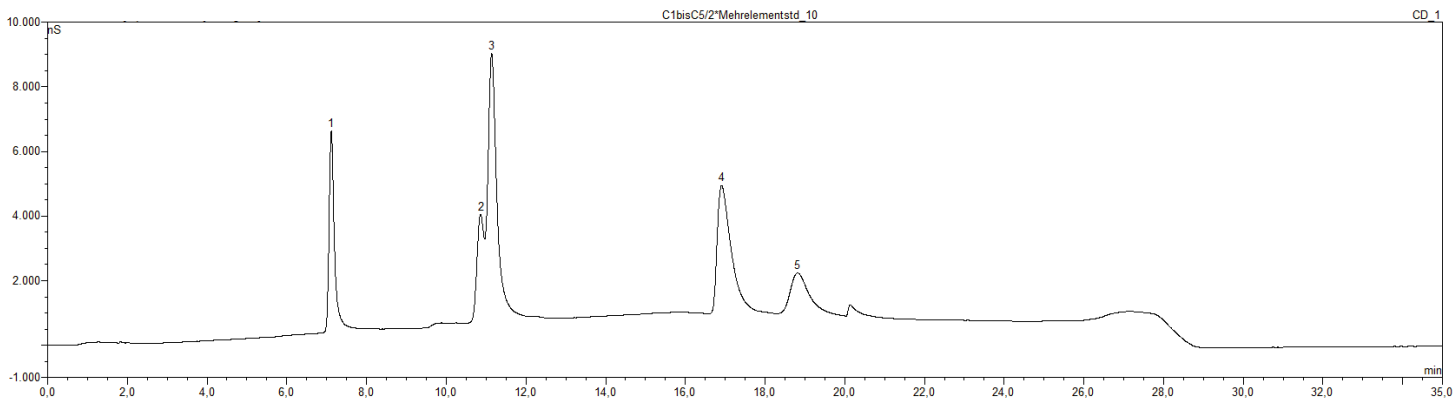


Figure 1: Separation of 5 anion and organic acids standards on a Hamilton Anion Micro column using gradient 1

Due to the rapid increase of the KOH concentration between 0 and 13 min the separation between the closely related organic acids acetic and formic acid is insufficient. To improve the separation, a shallower slope of the KOH gradient was used between 0 and 20 minutes (gradient 2 in Table 2). The resulting chromatogram is shown in Figure 2.

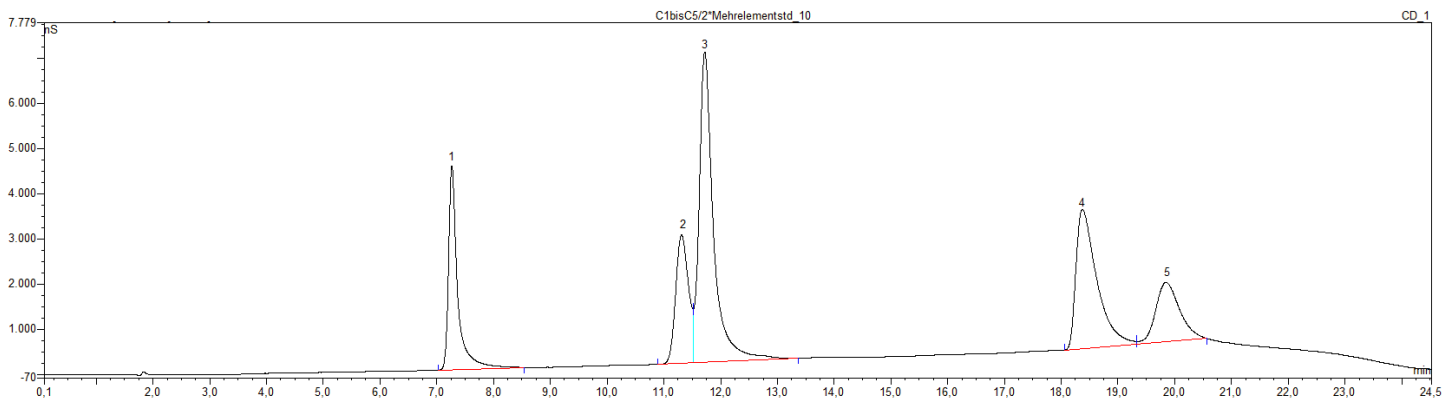


Figure 2: Separation of 5 anion and organic acids standards on a Hamilton Anion Micro column using gradient 2

A better separation between acetic acid and formic acid was achieved with a more shallow KOH gradient (gradient 2), making it possible to identify all of the shown organic and inorganic anions of an unknown sample by their retention times (Table 3).

Conclusion

The Hamilton Anion Micro column is a good choice when using a KOH gradient to separate closely related organic acids and anion standards. The results demonstrate that different ion exchange buffers such as KOH can be used with Hamilton Anion columns which greatly expands the applicability of these columns. The Anion Micro column with a 2.1 mm inside diameter allows lower flow rates that are required for ion chromatographs which are equipped with eluent generators.

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