Proc. Estonian Acad. Sci. Chem., 1998, **47**, 1, 30–34
https://doi.org/10.3176/chem.1998.1.04

USE OF ELUENTS OF VANILLIC ACID AND N-METHYLDIETHANOLAMINE IN NONSUPPRESSED ION CHROMATOGRAPHY

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Received 8 October 1997

VANILLIINHAPPE/N-METÜÜLDIETANOOLAMIINI KUI ELUENDI KASUTAMINE ÜHEKOLONNILISES IOONKROMATOGRAAFIAS. Irina JAKOVLEVA, Ülo HALDNA ja Mihkel KALJURAND

Key words: nonsuppressed ion chromatography, vanillic acid, N-methyldiethanolamine, eluent, conductivity detector.

INTRODUCTION

The technique of ion chromatography has gained wide acceptance for the analysis of inorganic anions in a variety of aqueous matrices [1]. The technique involves separation of anions of interest on a low-capacity ion-exchange column. Typically a conductivity detector is used. If the single-column version of ion chromatography is used, the analysis time is, as a rule, rather short, but the eluent used should separate the anions well, have an acceptable low background conductance, and have no system peaks on the chromatograms disturbing the peaks of the anions to be determined.

To fulfil these requirements simultaneously, careful choice of the eluent is an important step in this technique. This is also a reason why the choice of the eluent for single-column chromatography has been rather thoroughly studied [1].

The eluent made of vanillic acid and N-methyldiethanolamine has been recommended for use in nonsuppressed ion chromatography. In this work we report the application of eluents of this composition to two analytical columns.

EXPERIMENTAL

A model IVK-11 ion chromatograph with a conductometric detector JD-1, both from the Design Office of the Estonian Academy of Sciences, Tallinn, was used. The analytical columns were (1) column A (4 × 250 mm) packed with HIKS-1 resin anion exchanger [—CH_2—CH—C_6H_4—$CN(CH_3)_3$]$_n$ (Khimifil Inc., Tallinn), capacity 0.0189 mequiv./cm^3, and (2) column B (3 × 150 mm) packed with Anieks-N10 resin anion exchanger formed by treating the epoxidized methacrylic matrix (Separon) with trimethylamine (Anieks-N10 resin from Ecos, Tallinn), capacity 0.0233 mequiv./cm^3. No suppressor devices were used. The eluents were made from reagent grade chemicals. The concentrations of vanillic acid and N-methyldiethanolamine were varied in the range from 2 to 5 mM. The eluent flow rate was 1–3 ml/min and the loop volume used was 0.221 or 0.430 ml.

RESULTS AND DISCUSSION

A typical chromatogram obtained using the vanillic acid/N-methyldiethanolamine eluent is presented in Fig. 1 with column A. The same eluent was used for column B to record the chromatogram shown in Fig. 2. This eluent is suitable for the separation of Cl^-, NO_3^-, and SO_4^{2-} anions from their mixtures. For comparison the performance of analytical column A used to get the chromatogram in Fig. 1 was studied applying a suppressed system with a carbonate eluent (see Fig. 3). Therefore, the differences between Figs. 1 and 3 are related mainly to the different eluents applied.

Eluents with different ratios, a, of the concentration of vanillic acid to the concentration of N-methyldiethanolamine were tested on the two columns A and B. The ratio a was varied between 0.5 and 5. The capacity factors $k = (t_R - t_0)/t_0$ for the above-mentioned anions vs the ratio a are presented in Figs. 4 and 5. These figures show that the retention time increases with increasing vanillic acid concentration. We observed an important fact that these eluents showed only one system peak on the chromatograms if $1 < a < 2$ and the system peak was located at the beginning of the chromatogram for both columns.

The eluent system studied did not give a system peak disturbing the analysis. Also the conductivity background of the eluents tested was acceptable for single-column ion chromatography (see Fig. 6).

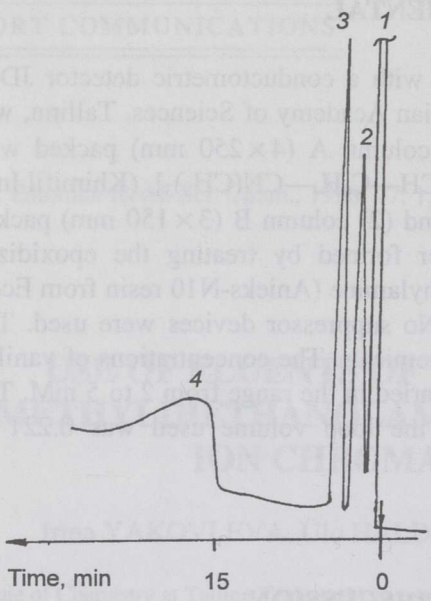

Fig. 1. Analytical column A, chromatogram of a mixture. *1*, system peak; *2*, Cl^-; *3*, NO_3^-; *4*, SO_4^{2-}. Eluent: 4.0 mM vanillic acid/4.0 mM N-methyldiethanolamine; flow rate 3.0 ml/min; backpressure 30 kG/cm^2; loop 0.430 ml. Detector: 0.1 mS full scale, 1/100.

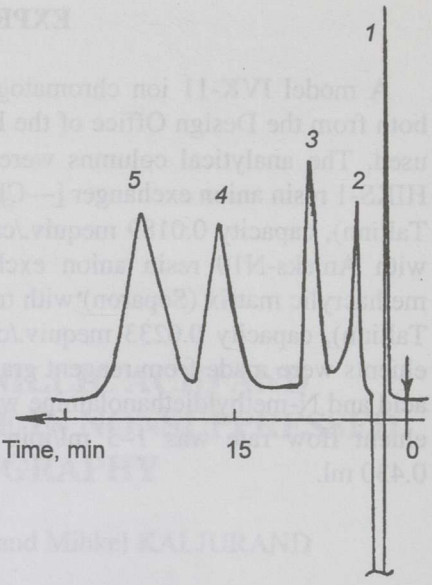

Fig. 2. Analytical column B, chromatogram of a mixture. *1*, system peak; *2*, F^-; *3*, Cl^-; *4*, Br^-; *5*, NO_3^-. Eluent: 5.0 mM vanillic acid/4.0 mM N-methyldiethanolamine; flow rate 0.6 ml/min; backpressure 55 kG/cm^2; loop 0.221 ml. Detector: 1 mS full scale, 1/40.

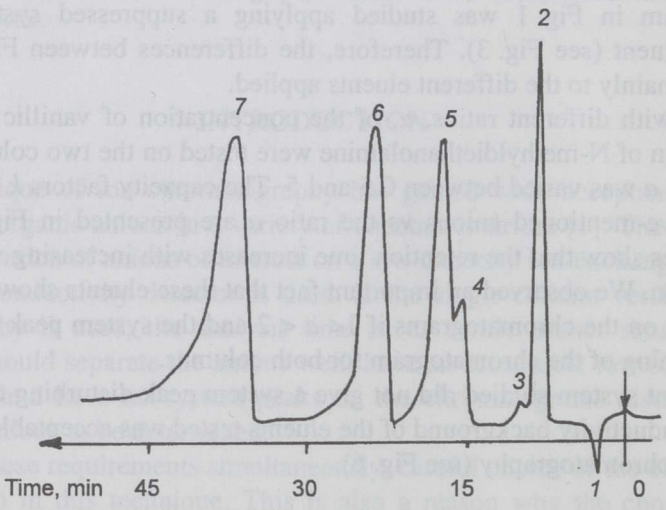

Fig. 3. Analytical column A, chromatogram of a mixture. *1*, system peak; *2*, Cl^-; *3*, NO_2^-; *4*, Br^-; *5*, NO_3^-; *6*, $H_2PO_4^-$; *7*, SO_4^{2-} in a suppressed system. Eluent: 3.0 mM Na_2CO_3; the suppressor column used was packed with DOWEX WX50 (4×250 mm); flow rate 1.5 ml/min; backpressure 20 kG/cm^2; loop 0.430 ml. Detector: 0.1 mS full scale, 1/20.

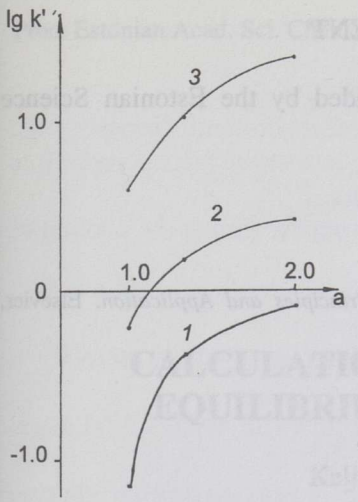

Fig. 4. The capacity factor $k' = (t_R - t_0)/t_0$ for column A. *1*, Cl^-; *2*, NO_3^-; *3*, SO_4^{2-}.

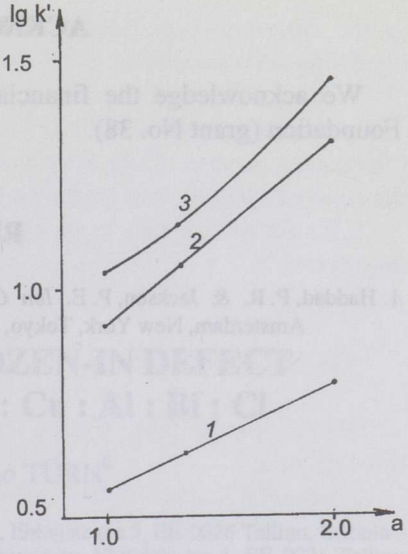

Fig. 5. The capacity factor $k' = (t_R - t_0)/t_0$ for column B. *1*, Cl^-; *2*, NO_3^-; *3*, SO_4^{2-}.

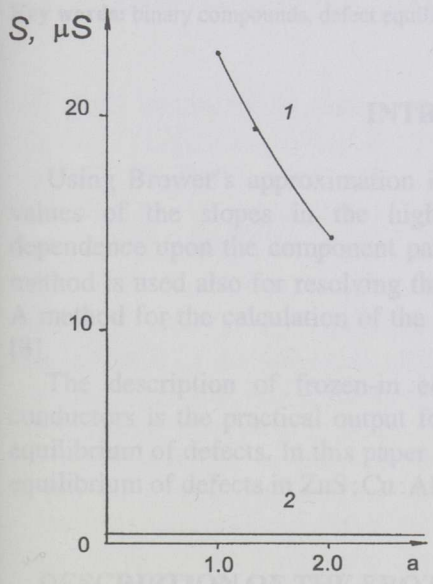

Fig. 6. The background conductivity (*S*) of eluents (*1*) vs the ratio of the concentration of vanillic acid to the concentration of N-methyldiethanolamine (*a*). The background conductivity of distilled water is shown by *2*. The cell constant $k = 7\ cm^{-1}$; the concentration of vanillic acid was 4.0 mM.

ACKNOWLEDGEMENT

We acknowledge the financial support provided by the Estonian Science Foundation (grant No. 38).

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