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APPLICATION OF THE KATIEKS K CATION-EXCHANGE COLUMN TO DETERMINING SODIUM, AMMONIUM, AND POTASSIUM IONS IN GROUND WATER SAMPLES

Introduction

To determine sodium, potassium, and ammonium ions in ground water samples traditional methods are usually applied [1]. Their main drawback is the need to use different procedures for sodium and potassium ions (flame photometry) and for ammonium ions (colorimetry).

Recently, ion chromatography (IC) has become a widely-used analytical method for the determination of several cations in water [2].

In this paper, the chromatographic performance of the Katieks K cation-exchange column ("Ecos", Estonia) was assessed in terms of the capacity factor, efficiency, resolution, and linear calibration range. The results were compared with those obtained with the BT IV KA column (Biotronik, Germany), a commercially available one for routine analysis. The purpose of the present work was to check the applicability of the Katieks K column to determining these cations in ground water samples.

Experimental

Instrumentation. The IC system consisted of the following elements: a single piston pump of an amino acid analyzer AAA 881 ("Mikrotecna", Czechoslovakia); an injector i 1000 ("Inkrom", Estonia) with a 0.1 ml sample loop; separator columns Katieks K ("Ecos", Estonia) and BT IV KA (Biotronik, Germany); a conductivity detector JD-1 (Special Design Office, Estonian Academy of Sciences), and a 10 mV input recorder.

Reagents and procedures. All the reagents used were of analytical grade ("Reakhim", USSR). The standard solutions of cations were prepared by dissolving the weighed amounts of nitrate salts in degassed distilled water.

When the Katieks K column was used, the flow rate of the eluent (nitric, tartaric or citric acid) was 1.0 ml/min and its concentration 1 mM. For the BT IV KA column the values (for nitric acid) were 1.5 ml/min and 2 mM. To measure the capacity factors, efficiency, and resolution the test solution containing 5 ppm sodium, 5 ppm ammonium, and 10 ppm potassium ions was used. For the Katieks K column the test solution contained also 1 ppm lithium ions.

The linearity of calibration for the Katieks K column was studied using standard sodium solutions at concentrations from 0.1 to 100 ppm.

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A comparative test of calibration linearity for both the columns was performed using standard sodium, ammonium, and potassium solutions in the concentration range of 0–100 ppm.

The chromatograms were evaluated by measuring the peak heights.

Results and discussion

Performance characteristics. The test solutions were injected into the chromatograph to evaluate column performance using nitric, tartaric, and citric acid eluents. The chromatograms obtained were used to calculate the capacity factors (k'), column efficiency ($HETP$, using half-height method), and resolution (R). All the values obtained in this study were measured according to [3]. The results are listed in Table 1.

The resolution values for the Katiex K column show that the separation of cations is sufficient for their simultaneous determination under the conditions used. The resolution values obtained for the BT IV KA column were too high (see Table 1) for the optimum routine conditions of analysis ($R \gg 1$). The worst resolved peak pair differed from column to column so that sodium and ammonium peaks were less resolved on the Katiex K column, whereas ammonium and potassium were less resolved on the BT IV KA column.

Table 1

Performance characteristics for the separation of a cation test solution.
Columns: 1 — Katiex K ($t_0=0.72$ min); 2 — BT IV KA ($t_0=0.40$ min)

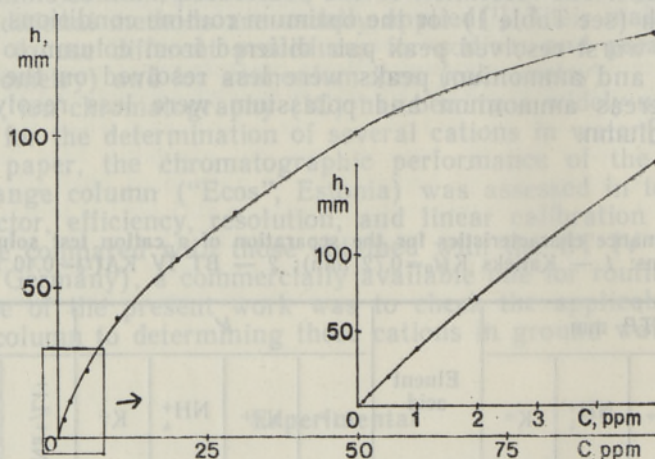
Columns	$HETP$, mm				Eluent acid	k'				R		
	Li ⁺	Na ⁺	NH ₄ ⁺	K ⁺		Li ⁺	Na ⁺	NH ₄ ⁺	K ⁺	Na ⁺ /Li ⁺	NH ₄ ⁺ /Na ⁺	K ⁺ /NH ₄ ⁺
1	0.24	0.21	0.17	0.15	Nitric	5.15	6.85	8.24	11.44	1.30	0.95	1.94
					Tartaric	7.72	9.97	11.93	16.32	1.28	1.01	1.93
					Citric	8.64	10.90	13.18	18.54	1.27	1.04	1.90
2	—	0.22	0.20	0.19	Nitric	—	16.40	27.20	37.10	—	2.25	1.39

Calibration linearity. A preliminary experiment to check calibration linearity for the Katiex K column was carried out using standard sodium solutions. The peak heights (h) of the sodium ion were plotted vs. sodium ion concentration (C). As the Figure shows, the plot obtained was non-linear. This indicates that the Katiex K column was easily overloaded even at low concentrations. A linear range was observed at concentrations below 2 ppm.

A comparative test of calibration linearity for both the columns was performed using standard sodium, ammonium, and potassium solutions. Ion peak height (h) was plotted vs. ion concentration (C), and the intercept and the slope for the calibration equation were calculated using a simple linear regression. The results listed in Table 2 show that as expected on the basis of a preliminary experiment the calibration plots for the Katiex K column were obtained with low correlation coefficients of linear regression. The high intercept values indicated that the calibration plots curved when the concentration of ions increased and the calculated linear plots for these ions did not cross the zero of the coordinates. For the BT IV KA column good correlation coefficients of linear regression

Linear regression data (main \pm std. dev.) for h plotted against C in the range 0–100 ppm

Ion	Katieks K			BT IV KA		
	Intercept	Slope	Correlation coefficient	Intercept	Slope	Correlation coefficient
Na ⁺	13.22 \pm 3.46	2.80 \pm 0.26	0.9754	0.76 \pm 0.85	2.16 \pm 0.06	0.9987
NH ₄ ⁺	9.05 \pm 2.61	2.17 \pm 0.12	0.9907	0.91 \pm 0.23	0.87 \pm 0.01	0.9998
K ⁺	6.04 \pm 2.20	1.40 \pm 0.06	0.9940	2.06 \pm 0.37	0.41 \pm 0.01	0.9990



Sodium ion calibration plot for the Katieks K column.

were obtained and the low intercept values indicated that the calibration plots passed the zero of the coordinates closely. So, when the BT IV KA column was used the linear calibration range was much wider than that of the Katieks K column.

Applications. To study the applicability of the Katieks K column to ground water analysis, five different samples taken from the Lahemaa National Park were analysed by measuring the content of sodium, ammonium, and potassium ions using IC and conventional methods (flame photometry, colorimetry). No statistically significant differences between these methods were observed. The IC detection limit ($S/N=3$) was 0.2 ppm for sodium and ammonium, and 0.5 ppm for potassium.

In conclusion, the Katieks K cation-exchange column is applicable to simultaneous routine determination of sodium, ammonium, and potassium ions in water samples; calibration linearity is satisfactory at rather low concentrations ($C < 10$ ppm) of these cations.

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REFERENCES

1. Лурье Ю. Ю. Унифицированные методы анализа вод. Москва, 1973, 113, 245, 248.
2. Smith, F. C., Chang, R. C. The Practice of Ion Chromatography. John Wiley & Sons, New York; Chichester, 1983, 32, 81.
3. Snyder, L. R., Kirkland, J. J. Introduction to Modern Liquid Chromatography. John Wiley & Sons, New York; Chichester, 1979, 22—34.

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KATIOONIVAHETUSKOLONNI «KATIEKS K» KASUTAMINE NAATRIUM-, AMMOONIUM- JA KAALIUMIOONIDE MÄÄRAMISEKS PÕHJAVEE PROOVIDES

Et rakendada naatrium-, ammonium- ja kaaliumioonide ionkromatograafilist määramist praktilistes analüüsid, uuriti katioonivahetuskolonni «Katieks K» («Ecos», Eesti) kromatograafilisi omadusi. Selleks mõõdeti nimetatud katioonide ja liitiumiooni retentsiooniparameetrid, kasutades eluendina 1 mM lämmastik-, viin- ja sidrunhapet. Saavutatud lahutusvõime osutus küllaldaseks, et määrata neid katioone koosinemisel. Kolonni «Katieks K» kalibratsiooni lineaarsust on uuritud vahemikus 0—100 ppm ja võrreldud tulemust BT IV KA rutiinanalüüsi kolonni («Biotronik», Saksamaa) vastava näitajaga. Kolonni «Katieks K» puhul saadi rahuldav kalibratsiooni lineaarsus ionide kontsentratsioonil alla 10 ppm, kusjuures BT IV KA kolonnil oli vastav näitaja hea kogu uuritud diapasoonis (0—100 ppm). On leitud, et kolonniga «Katieks K» tehtud põhjavee ionkromatograafilise analüüsi tulemused langevad hästi kokku traditsiooniliste meetodite (leekfotomeetria, kolorimeetria) abil saadutega. Avastamispiir kolonni «Katieks K» kasutamisel varieerus 0,2 ppm-st naatriumil ja ammoniumil kuni 0,5 ppm-ni kaaliumil.

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ИСПОЛЬЗОВАНИЕ КАТИОНООБМЕННОЙ КОЛОНКИ «КАТИЕКС К» ДЛЯ ОПРЕДЕЛЕНИЯ ИОНОВ НАТРИЯ, АММОНИЯ И КАЛИЯ В ПРОБАХ ПОДЗЕМНЫХ ВОД

Проведено исследование хроматографических свойств катионообменной колонки «Катиекс К» («Экос», Эстония) в целях внедрения ионохроматографических методов определения ионов натрия, аммония и калия в аналитическую практику. Измерены параметры удерживания перечисленных катионов, а также иона лития с использованием в качестве элюента 1 mM растворов азотной, винной и лимонной кислот. Селективность разделения смеси катионов признана хорошей. Исследована линейность калибровочного графика для колонки «Катиекс К» в диапазоне концентраций 0—100 млн⁻¹, результаты сравнены с полученными для колонки BT IV KA («Biotronik», Германия). Для нашей колонки этот параметр был удовлетворительным до 10 млн⁻¹, для BT IV KA — до 100 млн⁻¹. Установлено, что ионохроматографический анализ с применением колонки «Катиекс К» сравним по точности с классическими методами (пламенная фотометрия, колориметрия). Пределы обнаружения для ионов натрия и аммония составляют 0,2 млн⁻¹, для ионов калия — 0,5 млн⁻¹.