The potentiometric titration of aluminum and gallium with tetraphenylborate*

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Potentiometrische Titration von Aluminium und Gallium mit Tetraphenylborat

Various cations have previously been determined as their oxonium complexes by potentiometric titration vs. tetraphenylborate (TPB). This includes the alkaline earth metals [1], the rare earth elements [2], and lead [2, 3]. Some of these elements can, in fact, be sequentially estimated [1, 2]. With the exception of lead, these elements are in group IIA or IIIB of the periodic table of the elements.

We now report that aluminum and gallium, in group IIIA of the periodic table, can similarly be determined. The oxonium complexes were formed by addition of polyethylene glycol (PEG) of average molecular weight 20,000, and were potentiometrically titrated with a 0.05 mol/l TPB solution.

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Emf's were monitored with a coated-graphite sensor and a double-junction reference electrode. The detailed experimental conditions were previously reported [2]. Note that the equivalent weights of Al and Ga in this titration are one third of the atomic weights. These elements can be estimated only over a fairly narrow pH range: gallium from pH 1.55 to 1.75, and aluminum from pH 2.3 to 3.0, with an optimum at pH 2.65. 1 mg of aluminum was determined with a standard deviation of 0.003 mg for 6 replicates; 3 mg of gallium were determined with a standard deviation of 0.020 mg for 7 replicates. The endpoint inflection for gallium was not as steep as for aluminum which accounts for the larger standard deviation in its recovery.

Aluminum plus gallium yielded only a single inflection for the sum of the elements; this was found also for barium plus gallium. Indium did not yield an inflection at all. This is similar to the potentiometric titration vs. cetylpyridinium chloride which was applicable only to gallium, and not to indium [4].

References

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Ion-exchange high-performance liquid chromatographic determination of leukotrienes after pre-column derivatisation with o-phthalaldehyde

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Ionen-Austauscher-HPLC-Bestimmung von Leukotrienen nach Vorsäulen-Derivatisierung mit o-Phthalaldehyd

Introduction

Cysteinyl leukotrienes LTC₄, LTD₄ and LTE₄ are endogenous mediators in anaphylactic and inflammatory reactions [1, 2]. The increasing interest in the biochemistry of the leukotrienes has necessitated the development of sensitive methods for the determination of these metabolites. Reversed-phase high-performance liquid chromatography (HPLC) with detection by UV, radioactivity and fluorescence are the most widely used chromatographic methods for the determination of leukotrienes [3–7]. We have developed a HPLC method for the determination of LTC₄, LTD₄, and LTE₄ using an ion-exchange column and pre-column derivatization with o-phthalaldehyde.

Experimental

Reagents and materials. Cysteinyl leukotrienes LTC₄, LTD₄, and LTE₄ were purchased from Paesel (Frankfurt, FRG) and used as received. o-Phthalaldehyde (OPA) and ammonium acetate were obtained from Fluka (Neu-Ulm, FRG). 2-Mercaptoethanol was purchased from Schuchardt (München, FRG). Methanol for HPLC and all other chemicals were from Merck (Darmstadt, FRG). All reagents were of the highest quality available and were stored as recommended. The column (200 mm × 4.6 mm I.D.) packed with an ion-exchange material, 100-5NH₂ Nucleosil, was obtained from Macherey-Nagel (Düren, FRG).

Chromatography. The HPLC system consisted of a Model 2156 (LKB, Sweden) microprocessor-controlled HPLC pump with heads and capillaries made of titanium. Samples were loaded with a Rheodyne high-pressure sampling injection valve provided with a 200 µl sample loop. Column effluent was monitored with a Model RF-530 (Shimadzu, Japan) variable wavelength fluorescent detector. The cell volume was 12 µl. The wavelengths were set to emission 450 nm and to excitation 338 nm. Chromatogram recordings and all calculations were performed on a Model C-R3A integrator (Shimadzu, Japan). The mobile phase consisted of ammonium acetate-acetic acid-methanol mixture. The composition of solvent A was methanol:water (4:1, v/v) and solvent B was prepared as follows: 154 g ammonium acetate, 122 ml water and 378 ml acetic acid were mixed and 200 ml of the resulting solution was added to 800 ml of solvent B [8]. The flow rate was 0.8 ml/min. The OPA derivatization reagent