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A Selective Reagent for the Removal of Chromate, Dichromate, Nitrate, Perchlorate, and Dibasic Phosphate from Methanol Solutions

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TECHNICAL NOTE

A Selective Reagent for the Removal of Chromate, Dichromate, Nitrate, Perchlorate, and Dibasic Phosphate from Methanol Solutions

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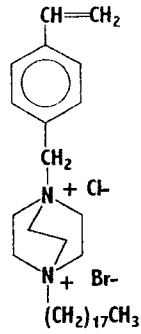
ABSTRACT

N-4-Vinylbenzyl-*N'*-octadecyl-1,4-diazabicyclo[2.2.2]octane diammonium salt was synthesized and tested to determine its ability to selectively precipitate anions. Fifty-one common anions were tested. The reagent reacted most readily with the monovalent oxyanions of Groups VIIB and VIA, and the divalent oxyanions of Group VIB.

INTRODUCTION

The primary purpose of this research was to prepare a reagent to selectively remove inorganic phosphate from solution. Such a reagent has been found. It is also suitable for the removal of chromate, dichromate, nitrate, and perchlorate.

Phosphate production is a major world industry. It is a vital component in the manufacture of fertilizers and cleaning products (1). Phosphate is used in soap because of its dispersing powers in aqueous suspensions of insoluble solids (2). It also softens washwater by complexing with metal cations, especially calcium and magnesium ions, which are characteristic of hard water. The ultimate destination for this and other chemicals are wastewater streams that eventually find their way to lakes or other water bodies. Phosphate is the limiting nutrient for algal growth; consequently unprecedented growth and anoxification of these water bodies is the final



[1]

FIG. 1 The compound synthesized and tested: *N*-4-vinylbenzyl-*N'*-octadecyl-1,4-diazabicyclo[2.2.2]octane diammonium salt.

result (3–6). The economic impact is obvious: death of game fish, and the loss of tourism to these water bodies due to the production of reeds that interfere with boating and yield unpleasant odors.

A reagent with high specificity for phosphate would be useful to prevent the environmental problems stated above. If that reagent also removed such ions as nitrate and chromate, which have harmful effects on human populations, it would be exceptionally useful. This research describes the behavior of just such a compound, *N*-4-vinylbenzyl-*N'*-octadecyl-1,4-diazabicyclo[2.2.2]octane diammonium salt. Figure 1 shows the structure of the prepared compound that was characterized and tested.

EXPERIMENTAL

All reagents used in testing were reagent grade and used as received. Solutions were prepared from the following: lithium fluoride, picric acid, and stannous titanate; potassium or sodium salts of acetate, aluminate, arsenate, arsenite, bicarbonate, bismuthate, bisulfate, bisulfite, borate, bromate, bromide, carbonate, chlorate, chloride, chromate, citrate, cyanate, cyanide, dichromate, dithionate, dithionite, ferricyanide, ferrocyanide, tetrafluoroborate, hydroxide, iodate, iodide, metaborate, molybdate, nitrate, nitrite, nitroferricyanide, oxalate, perchlorate, periodate, persulfate, selenate, selenite, silicate, sulfate, sulfite, thiocyanate, thiosulfate, tungstate, vanadate, tribasic phosphate, dibasic phosphate, and monobasic phosphate. All water was distilled and deionized.

The synthesized compounds were characterized with a Bruker WM-400 MHz NMR Spectrometer, a Hewlett-Packard model 5859A Mass Spectrometer, and a Perkin-Elmer model 1310 IR Spectrometer.

Synthetic Procedures

The synthetic schemes used and details of each step, as well as all spectra, were presented by Kopchinski (7). All products were spectroscopically characterized. The overall yield of the two-step synthesis was ca. 65.8%.

Analytical Tests

A 0.100-M solution of [1] was prepared by dissolving 5.979 g in 100 mL methanol. A solution (0.1 M) of each anion was prepared using a 1:1 mixture of water and methanol. An anion solution (10 mL) was placed in a test tube, and the reagent solution (2 mL) added. Observations were immediately made on any reactions and again after 2 and 24 hours.

TABLE 1
Anion Tests with *N*-4-Vinylbenzyl-*N'*-octadecyl-1,4-diazabicyclo[2.2.2]octane Diammonium Salt

No Reaction

Acetate
Bismuthate
Borate

Precipitate That Dissolved over Time

Bisulfite	Cyanate	Sulfate
Bromate	Molybdate	Tungstate
Bromide	Oxalate	Phosphate (HPO_4)
Carbonate	Selenate	Phosphate (H_2PO_4)
		Chloride

Formation of a Cloudy Solution

Aluminate	Cyanate	Selenite	Hydroxide
Arsenite	Cyanide	Sulfate	Metaborate
Bicarbonate	Nitrite	Titanate	Fluoride

Formation of a Precipitate

Bisulfate	Fluoroborate	Persulfate
Chlorate	Hydrosulfite	Picrate
Chromate	Nitrate	Silicate
Dichromate	Nitroferricyanide	Thiocyanate
Dithionate	Oxalate	Metavanadate
Ferricyanide	Perchlorate	Phosphate (HPO_4)
Ferrocyanide	Periodate	Iodate
		Iodide

RESULTS AND DISCUSSION

The results of the reactions between each reagent and the anions are summarized in Table 1. Fifty-one anions were tested with the reagent. In general, one of four results was observed: no reaction, formation of a precipitate that remained at 24 hours, formation of a precipitate that dissolved, or the formation of a cloudy solution.

The precipitates that formed were typically white, crystalline, and easy to filter and recover. IR spectra taken of the precipitates confirmed that complexation with [1] had occurred.

CONCLUSIONS

1. The reagent under investigation, [1], generally selectively precipitates the monovalent oxyanions of Groups VIIB and VIA, and the divalent oxyanions of Group VIB.
2. These selected anions are usually tetrahedral in shape.
3. Those anions that were going to react did so very quickly.

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