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A QUALITATIVE TEST FOR CYANIDE USING ION EXCHANGE RESIN-IMMOBILIZED REAGENTS

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ABSTRACT: A simple and sensitive test for cyanide ions based on the use of an ion exchange resin is described. The reagent for cyanide is appropriately attached to the ion exchange resin, and then several beads are used to perform the test. The test is quick, economical, requires only small samples of solution, and is relatively insensitive to the presence of certain other anions.

INTRODUCTION

Qualitative analysis was, and still is, a very important part of analytical chemistry; it is still important to know what is present in a sample as well as how much! Qualitative analysis is taught less frequently than perhaps it ought to be, but it still finds a place in some general chemistry courses as a way to teach clear observation and description and to demonstrate many common chemical techniques. Industry, however, frequently finds a use for clear, qualitative and semi-quanitative tests for certain inorganic chemical species, cyanide being one of them.

A review of the chemical literature shows that relatively few new qualitative chemical tests are being published, although many old ones are still being investigated and refined (Kjonaas, 1990). Qualitative inorganic tests are still valuable, being used in the food industry, for field geological testing, and for forensic work, to mention only three important fields. Occasionally, it is important to be able to detect extremely small quantities of materials, which could prove to be harmful: e.g., lead (drinking water limit 10 ppb) and cyanide, which might be present in tapioca pudding. A qualitative test was applied to Tylenol, when it was contaminated with cyanide several years ago (Beck, *et al.,* 1982).

Several tests exist for the detection of cyanides. Heating the sample with concentrated sulfuric acid evolves hydrogen cyanide gas, which possesses the odor of almonds. This test is not recommended! The use of silver nitrate solution is another test which could be used, but this test is fraught with difficulties for the inexperienced. An excellent review of methods for the detection and determination of cyanide exists which gives many useful references (Singh, *et al.,* 1986). None of the tests recommended in this review use ion exchange resins for improving the detection limits for any test for cyanide.

Ion exchange resins may be used to concentrate an analyte during both quantitative and qualitative analysis procedures. During a qualitative procedure, since the resin beads are generally small (< 1 mm in diameter), they may be used to work with small volumes of solutions. For example, only two or three beads of resin are normally required for about 0.05 mL of solution. The tests are frequently carried out with the use of a hand lens or low power microscope. Commonly, the resin beads are immersed in a drop of sample, agitated with a fine glass rod to assist in achieving equilibrium with the solution, and then exposed to a selective reagent for the analyte of interest. Appropriate choice of resin allows the investigation of both cations and anions, with concentration factors of over one thousand occasionally being achieved. This paper discusses the development and use of an ion exchange resin bead test for the cyanide anion.

EXPERIMENTAL PROCEDURE

Chemicals. All chemicals used for the experimental work were of analytical grade or better, and all solutions required were prepared using distilled of deionized water as the solvent. The resins used were commercially obtainable. They were washed carefully in deionized water, filtered, and stored, slightly damp, for use.

Equipment. The apparatus used was very simple, consisting of black and white porcelain spot plates, Pasteur pipettes, glass spatulas, and dropping bottles. All resin samples, prepared or otherwise, were stored in brown glass screw-capped bottles ready for use.

Preparation of the resin. The resin selected was Amberlite IRA-400, manufactured by Mallinkrodt, which is a strongly basic resin supplied in the chloride form. Strongly basic resins from other manufacturers would certainly work, but in choosing a resin, attention must be paid to its color and the contrast with the color of the test. A sample of this resin was soaked in a stirred solution of sodium chloride (~2.5 M) for about an hour to assure that it was totally converted to the chloride form. It was then rinsed carefully with deionized water until a sample of the washings showed only a slight haze when treated with acidified silver nitrate solution.

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1. **Palladium reagent.** Dissolve 80 mg of palladium (II) chloride (Aldrich) in 100 mL of deonized water and add 0.5 to 0.6 mL of concentrated hydrochloric acid. Store the final solution in a brown glass bottle. The solution is stable for at least three months.
2. **4-(2-pyridylazo)-resorcinol reagent (Aldrich).** Weigh out 25 mg of the solid powder and dissolve it in 100mL of deionized water. Store the solution in a brwn glass bottle. This solution is stable for three months.
3. **Buffer solution.** Dissolve 10.6 g of anhydrous sodium carbonate and 8.4 g of sodium bicarbonate in 100mL of deionized water. This solution should be prepared fresh once every two weeks. Store the buffer solution in a polymethylpentene bottle.
4. **Final impregnating solution.** Mix together 1 mL of the palladium reagent, 10 mL of the carbonate/bicarbonate buffer solution, and 12 mL of the 4-(2-pyridylazo) - resorcinol solution. Store this solution in a brown glass bottle. This solution should be remade every three days. In neutral or weakly acidic solution, 4-(2-pyridylazo) - resorcinol has a yellow color and reacts to form red chelates with a number af cations. Most of these chelates are destroyed by EDTA, and some are destroyed by cyanide. The use of the palladium complex provides the best and most sensitive test for cyanide, however.

Table 1. Color responses of treated resin to solutions containing no cyanide but concentrations of other anions.

Ion	Ion Concentration (ppm)	Resin color
Hydroxide	5000	Dark rose / violet
Iodide	600	Dark rose / violet
Permanganate	600	Dark rose / violet (after removal of the test solution)
Bromide	400	Dark rose / violet
Perchlorate (as an acid)	500	Green
Thiocyanate	350	Dark rose / violet
Nitrate (as an acid)	300	Green
Chloride	350	Dark rose / violet
Sulfate (as an acid)	< 50	Lemon yellow

5. **Conversion of the resin.** Two grams of resin (this will be found to be enough for many tests) are placed in a 50mL beaker equipped with a magnetic stirring bar and stirred for thirty minutes. Then, this solution is filtered, and the resin is air-dried and stored in a small brown glass bottle. The color of the treated resin should be dark rose/violet. Reaction of a resin sample with a cyanide-containing solution releases the 4-(2-pyridylazo)-resorcinol by producing a cyanide complex with the palladium, causing a colr change from rose/violet to yellow.

RESULTS

Mineral acids have the effect of changing the resin bead color from dark rose/violet to green. Since this has an adverse effect on the test, the pH of the test solution should be adjusted to between 6 and 8. Under these conditions, solutions containing 30 ppm of cyanide and varying concentrations of other ions were investigated to determine which ions would interfere with the test. Bromide, chloride, thiocyanate, sulfate, nitrate, and nitrite interfered with the test at concentrations over approximately 350 ppm. Sulfide caused the worst interference at a concentration over about 20 ppm. Under ideal conditions, the lowest concentration of cyanide to give a clear positive test was approximately 2 ppm. The response of the treated resin alone to various ions was also investigated. The results of this investigation are shown in Table 1.

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