Sodium tetraphenylboron, Na(C₆H₅)₄B, reacts with potassium ions to form a white insoluble compound. The equation for the reaction may be written as follows:

\[(\text{C}_6\text{H}_5)_4\text{B}^- + \text{K}^+ \rightarrow \text{K(C}_6\text{H}_5)_4\text{B}\]

Sodium tetraphenylboron was first prepared by Wittig and co-workers \((1, 2, 3)\) in 1949, and since that time a number of methods have been developed for use of this reagent in the quantitative determination of potassium. A comprehensive bibliography from 1949–58 is given by Barnard \((4)\) and Barnard and Buechl \((5)\).

A number of qualitative procedures are available using Na(C₆H₅)₄B for the detection of K⁺ by itself or in the presence of certain specific cations \((6, 7, 8, 9, 10)\). The only methods using Na(C₆H₅)₄B for the detection of K⁺ in general cation unknowns \((11, 12, 13)\) suggest testing for K⁺ in the “soluble cation” group. These procedures further specify either the prior removal of NH₄⁺, which also forms an insoluble compound with the reagent, or the distinction of K⁺ from NH₄⁺ by flame testing the precipitate. In this communication a method is described which uses Na(C₆H₅)₄B to test for K⁺ in original general cation unknown solutions. This test resulted from the observation that K(C₆H₅)₄B is insoluble in NaOH while NH₄(C₆H₅)₄B is soluble. The method, which eliminates all interfering ions by a one step treatment with NaOH, is specific for K⁺, and hence the need for flame testing the precipitate is eliminated.

The concentration of the reagent was found to be a critical factor in the practical success of the test, an unnecessarily high concentration resulting in sensitivity to traces of K⁺ in many common reagents. Most of the qualitative and quantitative procedures for the analysis of K⁺ using Na(C₆H₅)₄B recommend the use of a 1–4% solution of the reagent. \((3.4\% = 0.1 \text{ M})\) This is needlessly high for the detection of K⁺ in the concentrations \((0.1 \text{ M})\) normally found in student unknowns. The solubility product at 25°C of K(C₆H₅)₄B is \(3 \times 10^{-8}\) \((14)\). Using one drop of 0.1 M reagent in five drops of test solution, one could theoretically detect about \(10^{-6} \text{ M} \text{ K}^+\). Probably because of supersaturation, neutral solutions containing less than \(10^{-4} \text{ M} \text{ K}^+\) were found to give no observable precipitate with 0.1 M reagent. This is in qualitative agreement with the sensitivity determinations reported by Amin \((9)\), Crane \((10)\), and Geilmann and Gebauhr \((15)\). Concentrations of K⁺ of the order of \(10^{-4} \text{ M}\) are often found in student unknowns as the result of traces of K⁺ in reagents, or possibly from storage in soft glass reagent bottles. A number of Reagent Grade chemicals were found to give definite tests for K⁺ using 0.1 M reagent.

The optimum concentration of reagent for present purposes was found to be 0.005 M. A curdy precipitate is obtained by using a more concentrated reagent, but with the 0.005 M reagent the result is a finely divided precipitate which is slow to settle. The sensitivity of the 0.005 M reagent is about \(10^{-2} \text{ M} \text{ K}^+\) in neutral solution which is equivalent to a dilution limit of about \(1:2.5 \times 10^3\). The sensitivity appears to be considerably greater in NaOH of the concentration specified in the procedure below. Experience has shown that 0.005 M reagent will give a definite test for K⁺ when present in student unknowns at a concentration of 0.1 M, but will not detect the traces of K⁺ commonly found in such unknowns. A further advantage of
using these very dilute solutions of the reagent is that the cost is reduced to a negligible figure.

The reagent was prepared by dissolving in distilled water a weighed amount of solid Na(C₂H₄)₂B, which was purchased from the J. T. Baker Chemical Company. Some workers (16) advocate clarifying the reagent by addition of AlCl₃ which coagulates any K(C₂H₄)₂B which may result from impurities and allows it to be filtered easily and quickly. Since no turbidity was observed when 0.005 M solutions of the reagent were prepared, it was not felt necessary to add any AlCl₃. The reagent is unstable in acidic solutions (17). The addition of AlCl₃ makes the solution acidic by hydrolysis, and it is necessary to adjust the pH to about 8 for several weeks. Decomposition eventually occurs in the products. A strong odor of benzene is observed from solutions of the reagent which have partially or completely decomposed.

The following cations have been found to form precipitates with 0.005 M reagent: Ag⁺, Hg₂⁺, Hg⁺, and NH₄⁺. A number of other cations, which do not appear in the usual schemes of cation analysis, have been reported to form insoluble compounds with the reagent. These cations are Ti⁺ (19, 20, 21, 22), Ce⁺ (20), Cu⁺ (20), Rb⁺ (3), and Cs⁺ (3). Using 0.005 M reagent, no interference was found with Pb⁺, As⁺, Sb⁺, Sn⁺, Bi⁺, Cu⁺, Cd⁺, Co⁺, Ni⁺, Al⁺, Zn⁺, Fe⁺, Fe⁺, Mn⁺, Ca⁺, Ba⁺, Mg⁺, Sr⁺, Na⁺, and Li⁺, when the concentration of the above ions was 0.5 M. The following anions in concentrations of 0.5 M were found to give no interference using 0.005 M reagent: S²⁻, SO₃²⁻, SO₄²⁻, Cl⁻, Br⁻, IO⁻, NO₂⁻, NO₃⁻, C₂H₅O⁻, CO₂⁻, C₂O₄²⁻, AsO₃⁻, AsO₄³⁻, BO₃⁻, CN⁻, SCN⁻, CNO⁻, Fe(CN)₆⁻, Fe(CN)₆⁻, PO₄⁻, Cr₂O₇⁻, CrO₂⁻, MnO₂⁻, F⁻, Cl⁻, Br⁻, and I⁻. A number of organic alkaloids, amines, and quaternary ammonium salts have been reported to form precipitates with the reagent (10). Enough K⁺ is present in some soapy water to give a test with 0.005 M reagent.

In analysis for K⁺ in Group V, the only interference is from NH₄⁺. However, NH₄(C₂H₄)₂B can be distinguished from K(C₂H₄)₂B by the fact that the former is soluble in warm NaOH while the latter is insoluble. If precipitation with Na(C₂H₄)₂B is made in acid solution, and if NaOH then added to determine whether the precipitate is NH₄(C₂H₄)₂B or K(C₂H₄)₂B, Mg⁺ and traces of other cations will form insoluble hydroxides with NaOH and lead to false conclusions regarding the presence of K⁺. Therefore the most satisfactory procedure is to test for K⁺ on the original solution after first removing all interfering ions with NaOH. The decantate can then be tested for K⁺ by adding one or two drops of Na(C₂H₄)₂B. In strongly alkaline solution NH₄(C₂H₄)₂B does not precipitate, even when allowed to stand in a closed container for a period of several weeks. Concentrations of NH₄⁺ as great as 1.0 M do not interfere with the test for K⁺ using the procedure described below.

The following procedure has been tested by the 120 students in our sophomore course in qualitative analysis and found to be satisfactory.

To 5 drops of the original unknown solution add an equal volume of 3 N NaOH. Centrifuge and test for complete precipitation by adding one more drop of NaOH. Add two drops of 0.005 M Na(C₂H₄)₂B. If K⁺ is present a white precipitate forms immediately.

It has been found that 6 N NaOH prepared from a c.p. reagent gives a precipitate with 0.005 M Na(C₂H₄)₂B. Even with 3 N NaOH a very slight turbidity is sometimes observed, depending on the source of the NaOH. Since the NaOH is actually diluted to 1.5 N after the test for K⁺ is completed, the use of 3 N NaOH should cause no interference with the test. However in order to verify this assumption, it is recommended that a blank be run on the NaOH supplied for the test.

Using the above procedure, results for K⁺ in student unknowns have improved considerably. The test is far superior to both the cobaltinitrite test and the flame test, particularly if large amounts of sodium are present.

**Literature Cited**

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