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The Interference of Borate Ion

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Many conflicting statements have been made about the interfering effect of borate ion in the usual group separations of qualitative analysis; some text books state that borate should be removed because the alkaline earth borates are insoluble in ammonia solution\(^1\), and would therefore be precipitated with the Group III hydroxides, while other texts\(^2\) state that borate does not interfere because the ammonium chloride present prevents the precipitation of borates under the foregoing conditions. The reactions of various borates were therefore examined in order to determine whether or not any interference occurred. Initial experiments suggested that there should be no interference, but as the investigation developed it was found that serious interference could arise from a cause which has hitherto been overlooked. This probably accounts for the conflicting statements which have been made, for those texts which have always advocated the removal of borate have done so in the erroneous belief that the alkaline earth borates would precipitate, and they appear to have been unaware of the real source of the interference. Those texts which state that borate does not interfere presumably have found that alkaline earth borates do not precipitate in Group III (when Group III metals are absent) and have overlooked other ways in which the interference could arise.

Calcium borate was precipitated when ammonia solution was added to the neutral solution, but rapidly redissolved when ammonium chloride was added. When ammonium chloride was already present (as is normally the case), then calcium borate was not precipitated even in the presence

of Group III metal ions. However, when this experiment was repeated with the Group IV metals, it was found that cobalt, zinc, and manganese borates were very insoluble, and, as can be seen in Table I, a considerable excess of ammonium chloride was required to prevent precipitation when ammonia solution was added. Further to this, even when enough ammonium chloride had been added to prevent direct precipitation, when any of the Group III metals (Fe, Cr, Al) were present the Group IV borates were co-precipitated when the solution was made ammoniacal (see Table II). This was readily seen when Al and Co were present, for the colour of the aluminium hydroxide precipitate remained pink even after careful washing.

This effect was examined on a quantitative basis and it was found that when there was just enough ammonia present to precipitate the aluminium, and about 10 mg Co$^{2+}$ was present originally, then less than 1 mg remained in solution (determined as cobalt pyridine-thiocyanate complex).

An investigation carried out with $^{60}$Co confirmed that under these conditions less than 10 per cent of the initial activity remained in the solution.

Table III shows that when excess of ammonia solution was used much of the cobalt passed through to Group IV, presumably as an ammine, and this metal was then easily detectable by the normal methods for this group. This also applied to zinc. However, chromium also passed through, and enough cobalt was left behind to produce a black residue in Group III (probably cobalt oxide), after the peroxide/alkali treatment to separate iron from aluminium or chromium.

In view of these observations, experiments were carried out to find out how borate could best be removed before the cation analysis was carried out. Repeated evaporation with HCl did not prove very effective, but a quantitative investigation indicated that conversion to methyl borate eliminated most of the borate.

Experiments have shown that even in the hands of elementary students this method is both rapid and effective.

**Experimental Details**

**Solution used**

- **Metals [as chlorides]** 40 mg metal ion per ml
- **Borate solution** 20 mg borax per ml
- **Ammonia solution** 1 : 1 ammonia (.880)
- **Ammonium chloride** 20% w/v solution