

Aluminum Solutions instead of Ferric in Qualitative Testing for Acetate

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Introduction

A common test for acetate is the precipitation of basic ferric acetate in a boiling solution. A serious objection to this method is the necessity for a definite very faint acidity of the generally used ferric chloride solution. Ferric chloride solution, made from most preparations of ferric chloride, is usually too acid to give any precipitate with acetate. The reduction of this acidity with ammonium or sodium hydroxide must be just sufficient so that the solution will not hydrolyze upon boiling with precipitation of a basic salt. This "blank" is the critical part of the test. Students usually have trouble getting the ferric chloride of just the right degree of acidity. Ammonium ferric alum gives a solution that hydrolyzes and precipitates when water is added, hence it has no advantage over ferric chloride as, in its use acid must be added before using. These iron solutions cannot be made neutral to litmus because hydrolysis constantly supplies acid. The acetate solution being tested must be neutral, but this can be easily done with litmus as there is no troublesome hydrolysis.

With the properly prepared ferric chloride solution, the acetate test gives very satisfactory results with students in the qualitative laboratory. However, if the ferric solution is too acid the test is negative with or without acetate and if too low acidity it is positive all the time.

Review

The most complete research on this method was done by Curtman and Harris (1). They found that a blank must be run each time to make sure no precipitate would form in the absence of acetate. On the other hand, no precaution was suggested for avoiding the error due to too much acidity. The greater the acidity of the ferric solution the less is the sensitiveness of the test. A summary of their results are shown in this table:

Mg. Fe per cc used	50 cc final solution mg. acetate not giving test	200 cc final solution mg. acetate not giving test
5	15	20
10	30	40
25	50	70

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These data show that the sensitiveness is reduced by increasing the volume of the solution and by increasing in the quantity of ferric chloride added. No explanation is apparent for increased dilution reducing the sensitiveness. The poorer results with greater quantities of the ferric solution could have resulted from the greater total acid present in the larger volume of the "unneutralized" ferric chloride. Their conclusion is that the test is not sensitive enough for good qualitative analysis. Their low sensitiveness could have been due to too high an acidity of their iron solution.

Experimental

Certain possible advantages of aluminum—using the potassium alum instead of the ferric chloride—are apparent. This salt contains no free acid. Its solution does not require any neutralization and its solution does not give a precipitate when a "blank" is boiled. This makes possible an easily prepared uniform testing solution without any of the troublesome and uncertain manipulation of "blanks."

Results with alum are as follows:

Mg. of alum used	Mg. of Na acetate no test	Mg. of Na acetate giving test	Volume of final solution
10	5	10	50
25	5	10	50
10	—	2.5	100
25	2.5	5	100

Conclusion

These data show that aluminum is much more sensitive than the ferric—as much as 25 times in some of the cases. It is possible that if Curtman and Harris had more fully neutralized their ferric, the test would have been more sensitive and their conclusion would not have been that "the ferric test for acetate is not sensitive enough." Also, the results in this research shows that the test is more sensitive the greater the dilution which is contrary to the ferric results. Also, an increase in the quantity of alum used did not greatly affect the sensitiveness of the test while the ferric test was less good.

One less desirable feature of the test with aluminum is that the basic aluminum acetate is white and, when the quantity of precipitate is small, it is much more difficult to see than is the highly colored basic ferric acetate.

Literature Cited

1. Curtman and Harris. 1917. J. Amer. Chem. Soc. 39:1315.