

THE LANTHANUM NITRATE TEST FOR ACETATE IN INORGANIC QUALITATIVE ANALYSIS

Part II. Limits of Identification

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IN a previous communication which may be regarded as Part I, Neelakantam and Row¹ introduced the lanthanum nitrate test for acetate into systematic inorganic qualitative analysis. This test which is the best available for the detection of acetate has been in regular use in our laboratories and proved to be of immense value in routine work. It was occasionally found, however, that the test failed to detect acetate. Krüger and Tschirch² noted that a large excess of acetate itself tended to transform the blue colour normally obtained in the test into an yellowish brown but in our experiments in some cases there was no development of colour at all. Our experience of the test indicated that this difficulty was primarily due to a high acetate ion concentration—an upper limit of identification existed, and to some extent on the amount of ammonia added. The addition of a drop or two of ammonia after the customary “nearly but not completely discharged colour of iodine” stage frequently secured a positive result in those cases wherein a negative one was reported according to the usual procedure. The lower limits of identification had previously been reported upon by other investigators but the upper limit which is of considerable importance in routine qualitative analysis has not been determined. The present paper deals with this aspect.

EXPERIMENTAL

Solutions

Acetic Acid.—Solutions of acetic acid of known strength ranging from 8 N down to 0.005 N were prepared.

Lanthanum Nitrate.—A 5% aqueous solution was prepared using a B.D.H. sample.

Iodine.—A 0.1 N solution of iodine in potassium iodide was used.

Procedure.—Acetic acid solution (1.0 c.c.) was pipetted into a test-tube and treated with the lanthanum nitrate solution (1.0 c.c.). Enough drops

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of iodine solution were added to impart a distinct yellow colour to the solution and then dilute ammonia (2 N) was added dropwise until the yellow colour was nearly but not completely discharged. If no colour developed in the cold within a few minutes, the test-tube was placed in a boiling water-bath.

RESULTS

| Acetic acid (mg. per c.c.) | Colour | Remarks |
|-------------------------------|-----------------------------|--|
| 480 | Nil | None in the cold or on heating; none even on adding more ammonia. |
| 240 | Nil | None in the cold or on heating but on adding more ammonia a light purple tinge developed slowly in the cold but this was intensified very slightly on heating |
| 180 | Nil | do. |
| 120 | Light purple → blue | A light purple colour was immediately obtained; this turned purple within a minute and blue in about five minutes in the cold. Turbidity developed slowly in the cold and quickly in the hot |
| 60.0 | Deep purple → dark blue | A deep purple colour was immediately obtained and it turned dark blue rapidly in the cold. Turbidity developed slowly in the cold and quickly in the hot |
| 30.0 | do. | do. |
| 12.0 | Dark blue | A pure dark blue colour was immediately obtained in the cold. No turbidity in the cold. |
| 6.0 | Brownish purple → deep blue | At first a brownish purple which changed to pure deep blue rapidly in the cold. No turbidity in the cold. |
| 3.0 | Deep blue | Rapidly in the cold. No turbidity. |
| 2.0 | do. | do. |
| 1.5 | do. | Colour developed rapidly in the cold but the solution became turbid. |
| 1.2 | do. | do. |
| 1.0 | Blue | Developed slowly in the cold. Solution became turbid. |
| 0.87 | Greenish blue | do. |
| 0.75 | Dirty grey → deep blue | Gelatinous precipitate, light dirty grey in colour in the cold; on heating, deep blue |
| 0.67 | do. | do. |
| 0.60 | Nil → purple | Colour developed slowly on heating. |
| 0.55 | do. | do. |
| 0.50 | do. | do. |
| 0.46 | do. | do. |
| 0.43 | do. | do. |
| 0.40 | do. | do. |
| 0.38 | do. | do. |
| 0.35 | do. | do. |
| 0.33 | Purplish blue | do. |
| 0.32 | Bluish | do. |
| 0.30 | Light dirty blue | do. |
| 0.24 | Bluish grey | do. |
| 0.20 | Light bluish grey | do. |
| 0.17 | Nil | Cream coloured precipitate but no trace of purple or blue in the cold or on heating |

Upper limit of identification 120 mg. per c.c.

Lower limit of identification 0.2 mg. per c.c.

The results obtained show that there is an upper limit of identification. A definite blue colour is obtained with a maximum of 120 mg. per c.c. of acetic acid. The lower limit is 0.2 mg. per c.c. or 1 in 5,000 (Feigl,³ gave 1 in 2,000 for the micro-chemical test).

SUMMARY

1. An upper limit of identification exists for the lanthanum nitrate test for acetate.
2. A definite blue colour is obtained in the test for concentrations of acetic acid ranging from 120 mg. to 0.2 mg. per c.c.

REFERENCES

1. Neelakantam and Row .. *Proc. Ind. Acad. Sci.*, 1941, **13 A**, 194.
2. Krüger and Tschirch .. *Chem. Abs.*, 1930, 5656.
3. Feigl-Mathews .. *Spot Tests*, 1937, 299.