

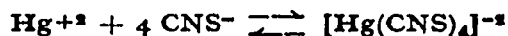
# MERCUROUS NITRATE AS A REDUCTIMETRIC REAGENT VI. THE DETERMINATION OF MERCUROUS MERCURY

by

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It was suggested in a previous paper in this series<sup>1</sup> that the scope of mercurous nitrate as a titrimetric reagent might be extended by using it in conjunction with a standard solution of ferric alum. Substances which reduce ferric iron might then be determined indirectly by treating with an excess of the standard ferric alum solution, and back titrating with standard mercurous nitrate. We have since used this procedure successfully for the determination of mercurous mercury, and the titration is unaffected by excessive amounts of mercuric mercury. Care must be taken however, to ensure that a sufficient excess of thiocyanate is present, since mercuric ions react as follows:



and if there is insufficient thiocyanate left after satisfying the requirements of the reaction



indistinct and drifting end-points are obtained.

As an example, 15 ml of 40 per cent ammonium thiocyanate solution are sufficient for the titration of 25 ml of a 0.1 *M* mercurous solution (0.5 g  $\text{Hg}^{+2}$ ) containing 1 g of mercuric mercury and treated with 30 ml of a 0.1 *M* solution of ferric alum.

In the preliminary tests the conditions of the general procedure recommended in previous papers were adhered to as closely as possible, *i.e.* the unknown mercurous solution was added dropwise to a known excess of 0.1 *M* ferric alum containing sufficient thiocyanate ions, and the ferric iron unconsumed in the reaction was then titrated with the standard mercurous nitrate solution. Good results were obtained by this procedure, but it would be inconvenient in practice. Accordingly, a further series of tests was undertaken in which the standard ferric alum solution was added from a burette to the solution of the mercurous salts, followed by the addition of the ammonium thiocyanate. The excess of ferric iron was then titrated by the standard procedure. Satisfactory results were obtained, some of which are included in Table I.

*Reference p. 475.*

TABLE I  
THE DETERMINATION OF MERCUROUS MERCURY

ml of 0.1N solution		Mercuric Nitrate added
Actual concentration of Hg <sup>+</sup>	Found	
28.00	28.00	—
28.00	28.00	1 g
25.00	24.98	—
25.00	24.96	1 g
21.00	21.02	—
20.00	20.00	1 g
15.00	15.00	—
15.00	15.00	1 g
10.00	10.03	—
10.00	10.00	1 g
5.00	5.00	—
5.00	5.00	1 g

## EXPERIMENTAL

*Solutions*

- 0.1M Mercurous nitrate reagent (also used for unknown Hg<sup>+</sup> solutions)
- 0.1M Ferric alum in 0.2N nitric acid
- 10 per cent Mercuric nitrate solution in 0.8N nitric acid
- 40 per cent Ammonium thiocyanate in water.

*Method*

A measured excess of ferric alum solution sufficient to give a back titre of 5-10 ml, was added from a burette to the test solution of mercurous mercury contained in a conical flask, followed by an excess of 40 per cent ammonium thiocyanate from a measuring cylinder. The blood red solution was then titrated with standard mercurous nitrate, shaking the flask thoroughly throughout the titration, and observing the usual precaution of dropwise addition near the end-point.

## SUMMARY

Mercurous mercury can be determined indirectly by allowing the test solution to react with an excess of standard ferric alum and back-titrating the unconsumed ferric iron with standard mercurous nitrate. The titration can be carried out in the presence of appreciable quantities of mercuric mercury.

## RÉSUMÉ

Le mercure (I) peut être dosé indirectement par addition d'une solution standard d'alun ferrique en excès. On détermine le fer (III) en excès par une solution titrée de nitrate de mercure (I). On peut effectuer ce dosage en présence de quantités appréciables de mercure (II).

## ZUSAMMENFASSUNG

Es ist möglich, Quecksilber (I) auf indirektem Wege zu bestimmen, indem man die zu untersuchende Lösung mit Überschuss Eisen (III)-Alaun reagieren lässt und nachher die unverbrauchte Menge Eisen (III)-Ionen mit standardisierter Mercuronitratlösung zurücktitriert. Die Titration kann in Anwesenheit erheblicher Mengen Quecksilber (II) durchgeführt werden.

## REFERENCE

- 1 R. BELCHER AND T. S. WEST, *Anal. Chim. Acta*, 5 (1951) 360.

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