

JUL 23 1962

MASTER

**Analytical Method For
THE DETERMINATION OF HYDRAZINE IN
35% HYDRAZINE SOLUTIONS**

Presented by

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**UNITED KINGDOM ATOMIC ENERGY AUTHORITY
PRODUCTION GROUP**

**Headquarters, Risley, Warrington, Lancashire
1962**

Analytical Method For
THE DETERMINATION OF HYDRAZINE
IN 35% HYDRAZINE SOLUTIONS

SUMMARY

The hydrazine, in hydrochloric acid solution, is titrated with standard potassium iodate solution.

APPLICABILITY

For the inspection of bulk quantities of 35% w/v hydrazine solution, which is used as an oxygen scavenger in the Calder Works and Advanced Gas Cooled Reactor water systems.

HISTORY

This is the classical Andrews's method. See Andrews, L. W., J. Am. Chem. Soc., vol. 25, p.756 (1903). The method is described in standard text books of analysis.

SAFETY NOTE

'PRECAUTIONS IN HANDLING HYDRAZINE COMPOUNDS

Hydrazine and its compounds can be dangerous and must be handled only in accordance with an approved method.

Hydrazine and its salts, whether in solid, liquid or gaseous state or in concentrated solution, may explode violently in contact with oxidizing agents or when heated. They are poisonous and may cause dermatitis by contact with the skin. Oxidation may give rise to the very poisonous and explosive gas, hydrogen azide. The oxidation is catalysed, sometimes violently, even in dilute solutions, by metals such as platinum and by ions of metals such as cobalt, manganese and copper.

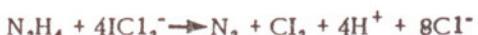
HYDRAZINE COMPOUNDS MUST NOT BE HEATED OR OXIDIZED EXCEPT IN DILUTE AQUEOUS SOLUTION IN A WELL VENTILATED FUME-CUPBOARD. ACCIDENTAL CONTAMINATION OF THE SKIN MUST BE DEALT WITH IMMEDIATELY BY WASHING THE AFFECTED PART WITH PLENTY OF WATER.

A. OUTLINE OF METHOD

The hydrazine is acidified with hydrochloric acid and titrated with standard potassium iodate solution. Free nitrogen and a chloride complex of iodine monochloride are produced.



In the presence of excess hydrazine, the iodine monochloride is reduced to free iodine



When all the hydrazine has been used up, the free iodine is titrated.



The end point is obtained by adding a small amount of carbon tetrachloride, in which the iodine released in the second reaction dissolves to give a violet solution. When the titration is complete all the iodine has been converted into iodine monochloride complex and the violet colour disappears.

B. REAGENTS REQUIRED

All reagents are analytical reagent quality.

1. Potassium Iodate Solution, Standard, 0.025M.
2. Hydrochloric Acid, 11M.
3. Carbon Tetrachloride.

C. SPECIAL APPARATUS REQUIRED

1. Iodine flasks, 250 ml, with well fitting ground glass stoppers.

Procedure	Notes
1. Pipette 25 ml of the sample into a 500 ml standard flask, make up to the mark with distilled water and mix thoroughly. Dilute 25 ml of this solution to 500 ml.	
2. Pipette 25 ml of the dilute hydrazine solution into a 250 ml iodine flask, add 50 ml of 11M hydrochloric acid and 10 ml of carbon tetrachloride.	

3. Titrate slowly with 0.025M potassium iodate solution, shaking vigorously between additions, until the purple coloration [Note (a)] just disappears from the carbon tetrachloride layer.

4. Calculate the hydrazine content of the sample from the expression:

$$\text{Hydrazine \% w/v} = 1.282T$$

where T = the titre in ml.

(a) The purple colour due to free iodine does not appear in the carbon tetrachloride until approximately 5 ml of iodate has been added. The intensity of the colour decreases as the end point is approached.