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## Determination of Nitrogen in Titanium Nitride

A simple, reliable method has been developed for the quantitative determination of nitrogen in titanium nitride (TiN). The procedure involves dissolution of TiN in 10M hydrofluoric acid containing an oxidant; the released nitrogen is determined as ammonia.

Interest in TiN as a refractory material has led to considerable investigation of its chemical and physical properties. Because TiN often varies widely from stoichiometry, the need of a simple chemical method for accurate determination of its nitrogen is apparent. Standard techniques such as the Dumas or Kjeldahl method give poor results.

This work is an extension of earlier investigations of the chemical reactions of nitrides. One difficulty encountered during the analysis of TiN is its inertness to many common laboratory reagents. The material is not readily dissolved by aqueous acids such as  $H_2SO_4$ , HCl,  $HNO_3$ , and hot 10M HF, and there is little or no reaction with aqueous 4M NaOH at 100°C. On the other hand a 10M HF solution containing a suitable oxidizer readily dissolves TiN, converting the nitrogen quantitatively to  $NH_4^+$ .

Selection of a proper oxidizing agent is of prime importance to the method. In terms of accuracy and convenience the best oxidizers are ferric chloride, potassium iodate, and potassium dichromate. The resultant  $NH_4^+$  is determined by standard volumetric titration. With use of these oxidizers the results for titanium and for nitrogen deviate less than 0.1% from those for a sample known to be stoichiometric TiN.

### Note:

Requests for further information may be directed to:  
Technology Utilization Officer  
Lewis Research Center  
21000 Brookpark Road  
Cleveland, Ohio 44135  
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No patent action is contemplated by NASA.  
Source: W. H. Philipp and J. E. Tetzlaff  
Lewis Research Center  
(LEW-11046)

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