

and 14 per cent of tungsten. Acid to dissolve the steel was then added and the analyses made as described above. The analytical results which we obtained in testing our method are given in the table below:

DETERMINATION OF TANTALUM IN THE PRESENCE OF CERTAIN OF THE CONSTITUENTS OF ALLOY STEELS  
50 and 100 cc. portions of ferro-tantalum solution added to 2 g. samples of steel

KIND OF STEEL	No.	PER CENT TANTALUM	
		Added	Found
Plain Carbon.....	1	1.05	1.06
	2	2.10	2.05
Chromium-Nickel Steel.....	1	1.05	1.03
	2	2.10	2.10
Chromium-Vanadium-Tungsten Steel.....	1	1.05	1.00
	2	1.05	1.09
	3	2.10	2.05
	4	2.10	2.27

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### NOTES ON THE ANALYSIS OF ALLOYS OF NICKEL AND ZIRCONIUM

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Received June 14, 1917

It recently became necessary in this laboratory to develop a convenient works-laboratory method for the analysis of an alloy of the following approximate composition:

Carbon.....	0.1 to 0.5 per cent	Aluminum....	0.5 to 10.0 per cent
Nickel.....	70.0 to 90.0 per cent	Zirconium....	2.0 to 18.0 per cent
Iron.....	2.0 to 6.0 per cent	Tungsten.....	0.5 to 10.0 per cent
Silicon.....	2.0 to 8.0 per cent	Manganese....	0.1 to 0.3 per cent

An outline of the method used is given in this paper.

Dissolve one gram of the material, preferably of a fineness to pass a 40 mesh sieve, in 40 cc. of hydrochloric acid to which 20 cc. of nitric acid have been added; then evaporate the solution with 20 cc. of sulfuric acid (sp. gr. 1.58) until  $\text{SO}_3$  fumes appear. On diluting with water the salts dissolve. Filtering separates nearly all of the tungsten as  $\text{WO}_3$  and the silicon as  $\text{SiO}_2$ , the zirconium appearing almost entirely in the filtrate.

Ignite the precipitate in a weighed platinum crucible. The first weight gives the weight of  $\text{SiO}_2$ ,  $\text{WO}_3$  and impurities. Then treat the residue with hydrofluoric and sulfuric acids to remove  $\text{SiO}_2$ , which is determined by the loss in weight: fuse the residue which now remains with sodium carbonate, leach with water and filter. The residue from leaching is generally very small and should be ignited in the original crucible and its weight subtracted from the weight of the crucible contents after the removal of  $\text{SiO}_2$ ; this gives the weight of  $\text{WO}_3$ . If extreme care is to be used in the determination of the tungsten, this residue should be fused with  $\text{KHSO}_4$ , leached in dilute  $\text{HCl}$  and precipitated with ammonia. Under these circumstances iron, aluminum and zirconium are precipitated free from sodium compounds which may contaminate the first residue. Upon ignition a corrected residue is obtained to be subtracted from the weight of impure  $\text{WO}_3$ . When such care is not necessary the residue may be fused with  $\text{KHSO}_4$  and leached with the filtrate from the  $\text{WO}_3$  precipitate.

By following the procedure as outlined above, the filtrate from the  $\text{WO}_3$  contains in solution all of the iron, nickel, aluminum and zirconium. The solution should be made up to a convenient volume, such as 200 cc., and one-half taken for the iron determination.

To determine the iron, make the solution alkaline with ammonia and boil or heat near the boiling point for a short time; filter and wash the precipitate. Transfer the bulk of the precipitate to a beaker and wash the paper by running through it a small amount of dilute sulfuric acid. Add enough sulfuric acid to the beaker to dissolve the precipitate and an excess equivalent to 10 cc. of acid (sp. gr. 1.58). Then pass the solution through a Jones reductor and determine the iron by titration with  $\text{KMnO}_4$ .

To the other half of the solution add ammonia in excess, and remove the nickel by electrolysis. The presence of this small amount of iron and aluminum does not seem to interfere, but in doubtful cases, the nickel may be dissolved in hydrochloric acid, evaporated with sulfuric acid, diluted and the iron and aluminum precipitated with ammonia; any iron and aluminum found in this way should be dissolved in a small amount of acid and added to the original electrolyte which now contains all of the iron, aluminum and zirconium. The ammoniacal solution of nickel is now free from the elements just named and is available for analysis by electrolysis or by the precipitation of its nickel content with dimethylglyoxime. In the event of using the latter procedure, one-tenth of the solution is sufficient for the purpose.

Boil the solution containing Al, Fe and Zr until nearly all of the ammonia has been driven off; then allow it to settle and filter, washing the precipitate with water. The precipitate contains Al, Fe and Zr. After transferring the precipitate to a beaker, dissolve in a minimum quantity of hydrochloric acid; then add solid  $\text{KOH}$  in excess to precipitate iron and zirconium, and leave aluminum in solution. Boil the mixture for one minute, allow to settle and filter, washing the precipitate with water. The filtrate contains the aluminum; make this solution acid with  $\text{HCl}$  and then barely ammoniacal; boil a few minutes, filter and wash. Then dissolve the precipitate in  $\text{HCl}$  and add 7 to 10 cc. of sulfuric acid (sp. gr. 1.58); evaporate the solution until fumes appear; cool, dilute and filter to remove  $\text{SiO}_2$ . Again add ammonia in slight excess and after brief boiling remove the  $\text{Al}(\text{OH})_3$  by filtration; ignite and weigh as  $\text{Al}_2\text{O}_3$ .

Dissolve the precipitate of iron and zirconium hydroxides which was separated from the aluminum in  $\text{HCl}$  (1 : 1) and re-precipitate with ammonia while boiling. The ignited precipitate contains  $\text{Fe}_2\text{O}_3$  and  $\text{ZrO}_2$ . Convert the iron found by titration into  $\text{Fe}_2\text{O}_3$  and subtract its weight from the weight of  $\text{Fe}_2\text{O}_3$  and  $\text{ZrO}_2$ . This gives the weight of  $\text{ZrO}_2$ .

If manganese is to be determined, weigh 1.5 g. of the material instead of 1 g.; then determine manganese in one-third of the filtrate from the tungstic oxide. For this purpose add ammonia and ammonium persulfate to the solution before boiling. Wash and dissolve the precipitate in nitric acid with the addition of a little sodium sulfite or hydrogen peroxide to aid solution. The manganese is then ready for oxidation by sodium bismuthate and determination by the various known methods.

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