

results of analyses with more exhaustive washings and attentions other than routine.

### Discussion

The proposed method for the determination of titanium in steel is of special advantage for analysis of steels of the 5 per cent chromium-0.6 per cent molybdenum type. This alloy dissolves slowly in strong hydrochloric acid, but rapidly in mixed hydrochloric-nitric acids. The test solution is free of color, while with the cupferron method chromium, as carbide, remains with the titanium cupferron precipitate. A disadvantage is the fact that all the titanium may not be contained in the filtrate from the chromium trioxide (0 to 0.02 per cent).

In the case of the (18-8) steels containing titanium, the advantage of speed in solution of the steel is no longer marked. Some nickel will be found in the titanium filtrate, but this error is easily overcome by adding nickel sulfate to the test solutions, or standard, previous to peroxidation.

When the alloying elements columbium, tungsten, molybdenum, chromium, and nickel are also present, difficulties are encountered. When the cupferron separation was used (Method A, Table II) considerable titanium was left in the residues. Method A does not use an oxidizing acid attack. Method B involves an oxidizing acid attack, a perchloric acid attack, and a water extraction. Method C is proposed for titanium-chromium-molybdenum steels, but with a double extraction. Of the three procedures used, the best seems to be Method A, unless the sample will not respond to hydrochloric acid attack. Method B is unusually long, while Method C may require three or more filtrations.

In a columbium steel, the columbium carbide is resistant to attack of aqua regia and fuming perchloric acid (3); and if a titanium-columbium steel is similarly treated most of the titanium is to be found in the filtrate (Method B); but if titanium and columbium salts are fumed with perchloric acid (1) a separation cannot be made. The apparent deduction is that columbium carbide is an insoluble entity and has

no solvent action on titanium (whatever the latter's chemical state may be).

In contrast, tungstic and molybdic oxides can be expected to retain small amounts of titanium. For this reason, one must expect to determine titanium in these alloyed steels by recovering the titanium in a soluble and in an insoluble portion of the sample, as indicated in Table II.

### Proposed Procedure for Titanium in Chromium-Molybdenum-Nickel Steels

Transfer 1 gram of steel to a 150-cc. beaker, add 15 cc. of mixed acids (300 cc. of hydrochloric acid, 100 cc. of nitric acid, 400 cc. of water), and warm to complete solution. Add 30 cc. of 70 to 72 per cent (technical) perchloric acid, heat till red chromium trioxide appears, and several minutes more, and cool.

Prepare a wash solution by heating perchloric acid in a small beaker until heavy fumes appear. Cool.

Use a bell-jar type suction or a filtering desiccator apparatus. Filter through a Gooch crucible with an asbestos pad, or better, a Pyrex M filtering crucible. Wash with five or six 5-cc. portions of wash solution, using a small glass rod to agitate the precipitate. Catch the filtrate and washings in a 150-cc. beaker.

Evaporate the filtrate to 10 to 15 cc., cool, add 25 cc. of water, and boil out chlorine. Dilute with 5 per cent sulfuric acid solution. Add 5 cc. of 3 per cent peroxide, and compare with a standard titanium solution.

**STANDARD.** Bureau of Standards No. 121 (0.394 per cent titanium) may be prepared as above, except that the insoluble chromium trioxide residue is redissolved in water, fumed in perchloric acid, and cooled, and the washings are added to the first filtrate. Dilute to 197 cc. with 5 per cent sulfuric acid.

Vanadium, if present, accompanies titanium. Decolorize the yellow titanium solution with hydrofluoric acid. A residual brown color indicates vanadium.

### Literature Cited

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## Removal of Adsorbents from Chromatographic Tubes

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THE contents of a developed chromatographic tube are usually removed by laying the tube horizontally on a piece of paper and extruding the contents from the top of the tube by pressing the bottom of the tube with a wooden stick or the like (1, 2). The method, however, involves considerable skill, since the material in the tube must be neither too dry nor too wet to slip out easily.

The following method has been found particularly effective and has reduced the breakage of chromatographic tubes to a negligible point in undergraduate courses.

After development of the chromatogram the solvent is allowed just to disappear from above the surface of the tube and suction is discontinued. The tube is placed horizontally on a table with paper to receive the contents of the tube. A rubber stopper of a size to fit the bottom of the chromatographic tube is attached to a compressed gas line and by means of this stopper gas pressure is applied gently and periodically to the bottom of the tube. The flow of gas through the tube not only presses against the solid contents of the tube but also dries the adsorbent to such a state that the contents slip out easily.

The pressure must be regulated either by a valve in the gas line or by placing the rubber stopper sideways in the bottom of the tube thus permitting part of the gas to leak through the space between the stopper and the tube. There is first a period of drying and packing the adsorbent, which normally requires greater pressure than the second period of extrusion. Consequently the pressure is applied intermittently for short periods, noting the results of each successive application. The column will finally start to move, often in segments. The pressure and the time of application must be decreased and the process is continued until the contents are pressed out of the tube. If the pressure is not controlled either by a valve or by periodic application of the rubber stopper for short times to the bottom of the tube, the adsorbent will shoot out of the tube across the laboratory. If the adsorbed material is sensitive to oxygen, the gas may be oxygen-free nitrogen or carbon dioxide.

### Literature Cited

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