

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON

PROVISIONAL CERTIFICATE
OF ANALYSIS OF
STANDARD SAMPLE 154
TITANIUM OXIDE

	Percent
TiO ₂	98.7*

*Based on material dried at 105° C.

Composition.- The following percentages of constituents of Standard Sample 154 were obtained by chemical methods: loss on ignition -- 0.29; SiO₂ -- 0.69; Al₂O₃ -- 0.06; Fe₂O₃ -- 0.009; BaO -- 0.04; PbO -- 0.03; Sb₂O₅ -- 0.02; P₂O₅ -- 0.17. In addition, spectrographic analysis indicates the presence of B, Cu, Ca, Cb, Mg, and Sn in amounts probably less than 0.01 percent. These values have not been established with sufficient certainty to warrant certification and are given only as a matter of general information.

PROCEDURE FOR USE AS A STANDARD IN COLORIMETRY

Place 3 to 5 g of the sample, thoroughly mixed (by tumbling in the bottle) in a glass-stoppered weighing bottle and weigh accurately. Transfer approximately 1.014 g of the sample, without loss, to a 250-ml Erlenmeyer flask and reweigh. Without delay, transfer approximately 1.0 g of the sample to a clean, dry weighing bottle provided with a well-fitting ground glass stopper. Weigh accurately and determine the loss on drying for 2 hours at 105° C. Correct the weight of the first sample taken for the loss on drying. Add 10 g of (NH₄)₂SO₄ and 25 ml of H₂SO₄ (sp.gr. 1.84). Insert a short-stemmed glass funnel in the neck of the flask and heat cautiously to incipient boiling while rotating the flask over a free flame. Continue the heating until complete solution has been effected and no unattacked material remains on the wall of the flask. Cool and rapidly pour the solution into 450 ml of cool water which is vigorously stirred. Rinse the flask with diluted H₂SO₄ (5 + 95). Stir and let the solution and washings stand for 24 hours. Filter through a small retentive paper, or through a glass filter if the

insoluble residue is not to be examined.¹ Wash thoroughly with

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If operations have been properly performed, this residue should contain less than 0.1 mg TiO₂, representing 0.0001 mg per ml -- an amount of no moment in the standardization.

diluted H₂SO₄ (5 + 95). Transfer the solution without loss to a 1000-ml volumetric flask, dilute to the mark with diluted H₂SO₄ (5 + 95), and thoroughly mix. The weight of the sample, on a dry basis, multiplied by 0.000987 represents the amount of TiO₂ in gram per milliliter.

PROCEDURE FOR USE AS A STANDARD IN VOLUMETRIC ANALYSIS

Transfer to a 250-ml Erlenmeyer flask approximately 1.75 g of the standard, accurately weighed and corrected for loss on drying at 105° C as described in the Procedure for Use as a Standard in Colorimetry.² Add 20 g (NH₄)₂SO₄ and 25 ml of H₂SO₄ (sp.gr. 1.84).

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Alternately for control work a 0.3500-g sample can be used. In this case transfer the sample to a 200-ml Erlenmeyer flask and dissolve by heating with 10 g (NH₄)₂SO₄ and 10 ml of H₂SO₄ (sp.gr. 1.84). Cool and rapidly pour the solution into 175 ml of cool water which is vigorously stirred. Rinse the flask with diluted H₂SO₄ (5 + 95), dilute the solution to 200 ml and proceed with the reduction and titration as directed. By this procedure 98.69 and 98.67 percent of TiO₂ were obtained in duplicate determinations.

Insert a short-stemmed glass funnel in the neck of the flask and heat cautiously to incipient boiling while rotating the flask over a free flame. Continue heating until complete solution has been effected. Cool, pour the solution rapidly into 450 ml of cool water which is vigorously stirred. Rinse the flask with H₂SO₄ (5 + 95). Stir and let the solution and washings stand for 24 hours. Filter through a small retentive paper, wash thoroughly with diluted H₂SO₄ (5 + 95), and reserve the filtrate. Transfer the filter and insoluble residue to a small platinum crucible, ignite to destroy carbon and treat with HF and H₂SO₄ to remove silica. Fuse the non-volatile residue with a little Na₂CO₃, extract with water, filter and wash. Transfer the filter and insoluble residue to the crucible and heat to destroy carbon. Fuse with a little K₂S₂O₇, dissolve the melt in 10 ml of diluted H₂SO₄ (5 + 95) and add to the reserved filtrate. Transfer the solution without loss to a 1000-ml volumetric flask and add diluted H₂SO₄ (5 + 95) to make exactly 1000 ml at the temperature of calibration of the flask (usually 20° C). Mix thoroughly and transfer a 200-ml aliquot portion to a

suitable beaker. Heat the solution to $40^{\circ} \text{C} \pm 5^{\circ}$ and add 0.1 N solution of potassium permanganate to a pink coloration. Reduce the titanium in a Jones reductor in the following manner: (1) Transfer 30 ml of ferric sulfate solution (0.02 g iron per ml of diluted H_2SO_4 (1 + 10) and prepared preferably from pure ingot iron) to the receiver; (2) draw 50 ml of H_2SO_4 (5 + 95) through the reductor at a speed of approximately 100 ml per minute; (3) transfer the titanium solution to the reductor and draw through at the same speed; (4) wash the beaker and reductor with 100 ml of H_2SO_4 (5 + 95) and finally with 100 to 150 ml of water; and (5) close the cock on the reductor. Gradually release the suction, raise the reductor above the receiver, and rinse the exit tube of the reductor internally (by opening the cock on the reductor and allowing 15 to 20 ml of water to pass through) and externally with water. Titrate the solution with approximately 0.1 N potassium permanganate until within 3 to 5 ml of the endpoint (approximately 38 to 40 ml), add 10 ml of H_3PO_4 (85 percent) and continue the titration to a permanent faint pink coloration. Correct the volume of the permanganate used in the titration by deducting the amount required in a blank carried through all steps of the procedure. To obtain the TiO_2 titer of the solution, divide the weight of TiO_2 taken, $\left(\frac{\text{g TiO}_2 \text{ (dry basis)} \times 0.987}{5} \right)$ by the corrected volume of permanganate.

(Signed) LYMAN J. BRIGGS, Director.
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