



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 154b

Titanium Dioxide

This Standard Reference Material (SRM) is in the form of fine powder, certified primarily for application in the paint and ceramic industries.

<u>Constituent</u>	<u>Percent by Weight^a</u>	<u>Uncertainty^c</u>
Titanium Dioxide (TiO ₂)	99.74 ^b	0.05

^aBased on material dried at 110°C for two hours.

^bThe value given in this certificate is based on the following analytical data:

^cThe uncertainty figure represents the 95% confidence interval of the mean of all accepted values.

NIST

<u>Method</u>	<u>Average</u>	<u>Standard Deviation¹</u>	<u>Number of Determinations</u>
Controlled-potential coulometric (0.2 g samples)	99.73	0.05	9
Volumetric (0.35 g samples)	99.71	0.03	10 ²

Cooperators³

Volumetric (Analyst A) ⁴	99.75	0.04	3
Volumetric (ASTM D1394)	99.78	0.05	3

¹Of single determinations.

²Two discrepant results were omitted.

³Results from one cooperating laboratory were deemed significantly high and have been omitted.

⁴Results from Analyst B at the same laboratory were deemed significantly low and have been omitted.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate value or any technical data presented in this certificate.

Gaithersburg, MD 20899
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(Revision of Certificate dated 5-16-73)

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(over)

SUGGESTED METHOD FOR THE DETERMINATION OF SILICON IN FERROSILICON ALLOYS

Fusion of ferrosilicon alloys in platinum with sodium carbonate and nitre though an excellent method for refined analysis is not as satisfactory for general purposes as is fusion with sodium peroxide in a pure iron crucible. Such a fusion, followed by dehydration with hydrochloric acid, is more convenient and gives accurate results if carefully made.

Procedure.—1. Transfer 0.5000 g (use 0.4000 g for 90 per cent silicon) of the finely powdered sample (150 mesh) to a 50 cc pure iron crucible made from No. 20 gauge (0.032-inch thickness) ingot iron, add approximately 13 g of sodium peroxide, mix thoroughly with a platinum or iron rod and carefully clean the rod of adhering particles by scraping with another rod. Cover the mixture with a layer of about 2 g additional peroxide. Heat the crucible and contents on a hot plate for 5 to 10 minutes to expel any water in the peroxide which would cause splattering in the subsequent fusion. Carefully fuse over a low flame by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly. When the fusion is molten, rotate the crucible carefully to stir up any unattacked particles on the bottom or sides, the crucible and contents being maintained at a low red heat. Just before completion of the fusion, which requires only three or four minutes, increase the temperature to bright redness for a minute. If the reaction has proceeded too violently, due to too rapid heating, use of insufficient sodium peroxide, or to lack of thorough mixing, appreciable loss will occur and the work should be repeated.

2. Cover the crucible and allow it to cool until the fusion has solidified, and tap the bottom of the crucible, while still warm, several times on an iron plate, which will loosen the fused mass in a solid cake. When the crucible and cake have completely cooled, transfer the cake to a large (275 cc) gold or preferably platinum dish. Cover the dish with a clock glass and cautiously add 50 cc of cold water. When the reaction ceases wash the small amount of adhering matter in the crucible into the dish with a little water. Cool the solution and carefully add an excess of hydrochloric acid. Evaporate to dryness, preferably on a steam bath, but do not bake at a temperature above 110° C. If platinum or gold dishes are not available for solution of the fused cake, the latter can be disintegrated with water in a pure nickel dish and the contents then transferred to a porcelain dish containing sufficient hydrochloric acid to provide an excess of acid. It is not desirable to dissolve the fusions directly in porcelain because of the action of the alkaline solution on the porcelain.

3. When the residue is dry, allow the dish to cool, add 10 cc of hydrochloric acid (sp. gr. 1.13), cover the dish, and heat gently for a few minutes. Dilute with 100 cc of hot water, digest for a few minutes, and filter through an 11 cm paper. Wash about eight times with hot 5 per cent hydrochloric acid, twice with warm 5 per cent sulphuric acid and then a few times with hot water. Reserve paper and residue.

4. Evaporate the filtrate to dryness and bake the covered casserole or dish at 110° C. for one hour. Heating at a higher temperature is unnecessary and also disadvantageous. Cool, add 10 cc of hydrochloric acid (1-1), digest on the steam bath for 10 minutes, add 100 cc of warm water, filter immediately, and wash about eight times with cold 1 per cent hydrochloric acid, once with cold 5 per cent sulphuric acid and then a few times with cold water.

5. Place the paper and silica from the *second* dehydration in a large partially covered platinum crucible and char the paper without inflaming. Add the paper containing the silica from the first dehydration, char as before, and then ignite carefully until the carbon has been burned off. Great care should be exercised in igniting the papers, as the current of air produced by a burning filter is sufficient to carry finely divided silica out of the crucible. When the carbon of the filter paper has been completely burned, gradually heat the tightly covered crucible to the full heat of a blast lamp and continue the heating at 1,050 to 1,110° C. for 25 minutes. Cool in a desiccator, weigh, and check for constant weight by additional heating for 10 minutes at the above temperature. Add sufficient sulphuric acid (1-1) to moisten the silica and then 15 to 20 cc of pure hydrofluoric acid (48 per cent). Evaporate to dryness, ignite, and weigh. The loss represents silica which contains 46.76 per cent silicon. A blank, not omitting the same amount of hydrofluoric acid, should be carried through all stages of the operation.

The silica remaining in the filtrate after the second dehydration is practically negligible, averaging about 0.0006 to 0.0010 g after dehydrations with hydrochloric acid and 0.0010 to 0.0015 g after dehydrations with sulphuric acid.