Designation: E1409 – 13

Standard Test Method for
Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion[1]

This standard is issued under the fixed designation E1409; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of oxygen in titanium and titanium alloys in mass fractions from 0.01 % to 0.5 % and the determination of nitrogen in titanium and titanium alloys in mass fractions from 0.003 % to 0.11 %.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 8.8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, refer to Terminology E135.

4. Summary of Test Method

4.1 This test method is intended for use with automated, commercially available, inert gas fusion analyzers. These analyzers typically measure both oxygen and nitrogen simultaneously or sequentially utilizing parallel measurement systems.

4.2 The test sample, plus flux, is fused in a graphite crucible under a flowing inert gas stream at a temperature sufficient to release oxygen and nitrogen. Oxygen combines with carbon to form carbon monoxide (CO) and nitrogen is released as N_2. Depending on instrument design, the CO may be oxidized to carbon dioxide (CO_2). The CO or CO_2, or both, are swept by the inert gas stream into either an infrared or thermal conductivity detector. The detector response generated by analysis of the test sample is compared to the response generated by analysis of reference materials and the result is displayed as percent oxygen. The nitrogen is swept by the inert gas stream into a thermal conductivity detector. The detector response generated by analysis of the test sample is compared to the response generated by analysis of reference materials and the result is displayed as percent nitrogen.

4.3 In a typical instrument for the determination of nitrogen, the sample gases are swept with inert gas through heated rare earth/copper oxide that converts CO to CO_2 and hydrogen (H_2) to water (H_2O). The CO_2 is absorbed on sodium hydroxide impregnated on clay, and the H_2O is removed with magnesium perchlorate. The nitrogen, as N_2, enters the measuring cell and the thermistor bridge output is integrated and processed to display percent nitrogen.

5. Significance and Use

5.1 This test method is primarily intended as a test for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory.

6. Interferences

6.1 The elements usually present in titanium and its alloys do not interfere but there is some evidence to suggest that low purity flux can cause some adsorption of the released oxygen.

7. Apparatus

7.1 *Instrument*—Fusion and measurement apparatus, automatic oxygen and nitrogen determinator consisting of an

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[1] E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf, Re.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

electrode furnace, provision for scrubbing impurities from analytical gas stream; infrared or thermal conductivity measurement system(s), or both, and auxiliary gas purification systems (Note 1).

NOTE 1—Several models of commercial oxygen and nitrogen determinators are available and presently in use by industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's instruction manual for operational details.

7.2 *Graphite Crucibles*—The crucibles must be made of high-purity graphite and be of the dimensions recommended by the instrument manufacturer.

7.3 *Flux*—Flux must be made of high-purity nickel. If nickel baskets are used, the dimensions must meet the requirements of the automatic sample drop, if present, on the instrument. (See Note 2.) Ultra high-purity nickel flux is commercially available and may eliminate the need to clean the flux before using it.

NOTE 2—In some instruments, nitrogen and oxygen are run sequentially and platinum is the required flux for nitrogen. High-purity platinum can be substituted for nickel in the same ratio of flux to sample.

7.4 *Tweezers or Crucible Tongs,* made of solvent and acid resistant material.

8. Reagents

8.1 *Acetone*—Low residue reagent grade or higher purity.

8.2 *Graphite Powder (optional)*—High-purity as specified by the instrument manufacturer.

8.3 *Inert Gas*—Use the purity and type specified by the instrument manufacturer.

8.4 *Magnesium Perchlorate, Anhydrous*[4]—Used in the instrument to absorb water. Use the purity specified by the instrument manufacturer.

8.5 *Nickel Flux Cleaning Solution*—An acid solution capable of removing surface contamination from the nickel flux. A solution made by combining 75 mL of acetic acid, 25 mL of HNO_3, and 2 mL of HCl has been found suitable for this purpose.

8.6 *Copper Oxide or Rare Earth/Copper Oxide*—Reagent used in some instruments to oxidize CO to CO_2 for detection. Use the purity specified by the instrument manufacturer.

8.7 *Sodium Hydroxide on Clay*[5]—Reagent used in some instruments to absorb CO_2. Use a purity specified by the instrument manufacturer.

8.8 *Titanium Sample Pickle Solution*—Three parts 30 % hydrogen peroxide (H_2O_2) and 1 part 48 % HF. Other pickle solutions may be substituted if there are data supporting the effectiveness of the solution on removing contaminants. For example, substituting concentrated HNO_3 for 30 % H_2O_2 has been found effective (see Note 3). (**Warning**—HF causes serious burns that may not be immediately painful; refer to the paragraph about HF in the Hazards Section of Practices E50.)

NOTE 3—In 2004, alternative sample preparation procedures (Section 12) were tested by seven laboratories. Three laboratories processed the sample materials by pickling their samples in HF-H_2O_2 (8.8). Two laboratories utilized the HF-HNO_3 alternative pickle solution (8.8). Two laboratories utilized abrasion (in this case diamond saw and shear) in accordance with 12.2. The prepared samples were distributed among the laboratories for analysis. Six laboratories analyzed these samples in random order under a single operator, single-day, single calibration sample run. The results of this testing are given in Tables X1.1 and X2.1 for oxygen and nitrogen, respectively. In both cases, the analysis of variance (ANOVA) indicates that there is no significant difference at the 95 % level of confidence for either oxygen or nitrogen due to the preparation technique.

9. Hazards

9.1 Use care when handling hot crucibles and operating furnaces to avoid personal injury by either burn or electrical shock.

9.2 For precautions to be observed in the use of HF and other reagents in this test method, refer to Practices E50.

10. Preparation of Apparatus

10.1 Assemble the apparatus as recommended by the manufacturer. Make the required power, gas, and water connections. Turn on the instrument and allow sufficient time to stabilize the equipment.

10.2 Change the chemical reagents and filters as required. Test the furnace and analyzer to ensure the absence of leaks (Note 4). A minimum of two test runs using a sample as directed in 14.3 and 14.4 is recommended to condition the newly changed filters. This should be done before attempting to calibrate the system or to determine the value of the blank.

NOTE 4—Typical leak checks should be 0.0 mm Hg to 1.5 mm Hg. The maximum allowable leak check should follow the manufacturer's recommendation.

11. Nickel Flux Preparation

11.1 Ultra high-purity nickel is commercially available that does not require the nickel cleaning procedure below. Its sufficiency must be verified by satisfactory blank determinations. If ultra high-purity nickel is not used, the nickel must be cleaned to remove contamination (11.2).

11.2 Immerse the flux in freshly prepared nickel flux cleaning solution for 50 s to 60 s, then rinse in running water for 2 min to 3 min. Pour flux onto paper towels to remove excess water. Place flux in sealable glass container, rinse with acetone and decant. To prevent new oxidation from forming, the flux may be stored under fresh acetone until used. (See Note 5.)

NOTE 5—The fluxing agent must be of proper size to be introduced through the sample drop mechanism and into the graphite crucible.

12. Sample Preparation

12.1 Remove the surface of the sample either mechanically (12.2) or chemically (12.3). Start with a sample of sufficient size that the final sample after surface removal will be between 0.100 g and 0.150 g.

12.2 To mechanically remove the sample surface, abrade with a clean file or similar abrasive device to remove contamination. Other methods, such as shearing, saw cutting, or

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[4] Known commercially as Anhydrone.

[5] Known commercially as Ascarite II.

turning down on a lathe, may be employed for reducing sample size and removing the surface of the sample. Regardless of the method used, the sample must not be allowed to overheat, as this will adversely affect the results of the analysis. Indications that the sample has overheated while being worked may include discoloration of the metal or the sample becoming too hot to handle without tools. Rinse the sample in acetone and air dry. Weight to ±0.001 g. Proceed to 12.4.

12.3 To chemically remove surface contamination, follow 12.3.1 and 12.3.2.

12.3.1 Leach the test sample in the titanium sample pickle solution. (**Warning—**See 8.8.) (see Note 3) until the surface is clean. This will normally require approximately 5 s from the time of the initial vigorous reaction between the sample and the solution.

12.3.2 Immediately remove the reacting test sample and rinse it twice with water and once with acetone and allow to air dry. Weigh to ±0.001 g.

12.4 All subsequent operations on the test sample and flux must be done without introducing contamination to either. Use only clean tweezers or crucible tongs and never let the test sample or flux contact the analyst's skin. In the event this does happen, rinse the sample and nickel with acetone and air dry before analysis.

13. Calibration

13.1 *Reference Materials*—Select only titanium or titanium alloy reference materials such that the high point on the calibration curves will represent an amount of oxygen and nitrogen that is approximately equal to or greater than the amount expected in the samples. The accuracy of the test method is dependent upon the accuracy of the methods used to certify the oxygen and nitrogen values of the reference materials, as well as upon their homogeneity. Thus, wherever possible, reference materials used to confirm instrument calibration should be traceable to certified reference materials from a national or international metrological institute.

13.2 *Gas Dosing*—Automatic and manual gas dosing, recommended by some manufacturers, can be used to calibrate the instrument, but instrument response must be verified by calibration with titanium reference materials because of the fusion characteristics of the furnace/sample combination.

13.3 *Initial Adjustment of Measurement System (that is, "warm-up")*—Place a titanium material (not necessarily a titanium reference material) with nickel flux into an outgassed graphite crucible containing graphite powder (optional, see Note 6). Proceed as directed in 14.3 and 14.4. Repeat in duplicate. (Outgassing is accomplished automatically either as part of the continuous analysis cycle used with the automatic sample drop, or as the first step in a two-stage cycle associated with the manual addition of the sample to the crucible.)

NOTE 6—The use of graphite powder is optional. In some instruments the addition of graphite powder (0.01 g to 1.0 g depending on crucible size and style) is designed to optimize furnace performance and facilitate the release of nitrogen from the test sample. Refer to the instrument manufacturer's instructions for recommended graphite powder additions (Note 1). If graphite powder is used, it must be employed consistently for blanks, samples, and reference materials.

13.4 *Determination of Blank*—Proceed as directed in 14.2 and 14.3 with a graphite crucible containing graphite powder (Note 1 and Note 6) and analyze the nickel flux without a sample. Determine the average blank of three to five individual runs and enter this value into the appropriate mechanism of the analyzer. If each individual result is within 5 µg of the average, the blank is acceptable. Alternatively, a maximum value may be used. Values of 0.0005 % for oxygen and 0.00007 % for nitrogen have been found adequate. Higher limits may be appropriate, particularly for reporting results that are not near the lower end of the scope. If other values are used, data showing that they are acceptable must be on file. Problems with inconsistent or high blank values must be corrected before the analysis can be continued. If the unit does not have provision for automatic blank compensation, then the blank value must be manually subtracted from the total result prior to any other calculation. Refer to the manufacturer's instructions for proper blanking procedures (Note 6).

13.5 *Calibration*—Follow the calibration procedure recommended by the manufacturer using titanium reference materials.

13.5.1 For each non-zero calibration point, weigh a titanium reference material to the nearest milligram, place it with nickel flux into an outgassed graphite crucible containing graphite powder if appropriate (Note 6).

13.5.2 Proceed as directed in 14.3 and 14.4.

13.5.3 Repeat 13.5.1 and 13.5.2. Analyze three to five specimens of each titanium reference material. For each reference material used to calibrate, calculate the average of these results, and compare the average to the certified value for the reference material. Adjust the instrument output to match the certified value unless the average already agrees with the certified value within the range of the uncertainty given on the certificate. (Note 7.)

NOTE 7—Some instruments have expanded computer capabilities that allow multi-point calibration which may improve the accuracy of the calibration over the single point calibration as tested in the current interlaboratory study (ILS). Either calibration type may be used.

13.5.4 Confirm the calibration by analyzing another specimen of the reference material after the calibration procedure is complete. The result should agree with the certified value within a suitable confidence interval (Note 8). If the result agrees with the certified value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Alternatively, if the certified value falls within an interval calculated as described in Eq 1, the calibration is acceptable.

NOTE 8—The procedure outlined in the original version of 13.5.4 required the test result to "fall within the maximum allowable limit of the standard" typically interpreted as the range defined by the certified value ± its associated uncertainty. The original version was utilized in the generation of the data in this method's Precision and Bias statements (Section 16). The current method for confirming the standardization is statistically rigorous and should be used in general practice. As an option, the laboratory may obtain an estimate of s from a control chart maintained as part of their quality control program. If the control chart contains a large number of measurements, t may be set equal to 2 % at the 95 % confidence interval. At its discretion, the laboratory may choose to set a smaller range for the acceptable test result.

TABLE 1 Statistical Information—Oxygen in Titanium

Test Material ID	Certified (Wt. %)	ILT Analyzed Mean (Wt. %)	Diff. (%)	Published Uncertainty (Wt. %)	Minimum SD (S_m, Practice E1601)	Reproducibility SD (S_R, Practice E1601)	Reproducibility Index (R, Practice E1601)	$R_{rel\ \%}$
GBW–02603[A]	0.119	0.115	0.004	0.005	0.0027	0.0043	0.012	10.4
GBW–02604[A]	0.273	0.274	-0.001	0.008	0.0038	0.0054	0.015	5.5
BCR No. 24[B]	0.0608	0.0632	-0.002	0.0023	0.0026	0.0047	0.013	20.8
BCR No. 59[B]	0.175	0.179	-0.004	0.007	0.0073	0.0074	0.021	11.6
LECO 501–653[C] Lot 290–103	0.044	0.044	0	0.004	0.0028	0.0032	0.009	20.4
TIMET Material B[D]	0.239	0.238	0.001	0.009	0.0044	0.0083	0.023	9.8
LECO 501–664[C] Lot 390–113–1	0.154	0.153	0.001	0.006	0.0037	0.0051	0.014	9.2
TIMET HTL 80[E]	(0.0080)	0.0090	...	...	0.00091	0.0017	0.0048	53.3
CEZUS LH[F]	(0.35)	0.3309	...	...	0.0029	0.0092	0.026	7.8
ATI Allvac C092[G]	NA	0.503	...	...	0.0112	0.0131	0.037	7.3

[A] Certified Reference Material, People's Republic of China.

[B] Certified Reference Material, Community Bureau of Reference, Commission of the European Communities.

[C] Calibration samples, Leco Corporation.

[D] TIMET, Henderson Technical Laboratory (vacuum fusion technique).

[E] TIMET, Henderson Technical Laboratory (Electrorefined Ti, Estimated by Mass Spectrometry), not certified.

[F] CEZUS, Internal reference material, not certified.

[G] ATI Allvac, Monroe, NC Laboratory (Ti-Nb alloy), not certified.

TABLE 2 Statistical Information-Nitrogen in Titanium

Test Material ID	Certified (Wt. %)	ILT Analyzed Mean (Wt. %)	Diff. (%)	Published Uncertainty (Wt. %)	Minimum SD (S_m, Practice E1601)	Reproducibility SD (S_R, Practice E1601)	Reproducibility Index (R, Practice E1601)	$R_{rel\ \%}$
LECO 501–653[A] Lot 290–103	0.002	0.0031	–0.0011	0.001	0.0009	0.0012	0.0033	106
BCR No. 24[B]	0.0117	0.0101	0.0016	0.0013	0.0014	0.0019	0.0053	52.7
BCR No. 59[B]	0.0172	0.0167	0.0005	0.0027	0.0020	0.0025	0.0071	42.6
TIMET[C] B–10377	0.107	0.116	–0.009	0.006	0.0047	0.014	0.039	34.0
TIMET HTL 80[D]	(<0.0010)	–0.00008	...	...	0.00020	0.00062	0.0017	...

[A] Calibration sample, Leco Corporation.

[B] Certified Reference Material, Community Bureau of Reference, Commission of the European Communities.

[C] TIMET, Henderson Technical Laboratory, Nitrogen content determined by Kjeldahl distillation-titration method.

[D] TIMET, Henderson Technical Laboratory (Electrorefined Ti, Estimated by Mass Spectrometry), not certified.

$$p = t \cdot \left(1 + \frac{1}{\sqrt{n}} \right) \cdot s \qquad (1)$$

where:

p = one-half the prediction interval,
n = number of replicates used in 13.5.3,
t = student's I chosen for the 95 % confidence level for n replicate measurements (for example: $t = 4.30$ when $n = 3$, 3.18 when $n = 4$, 2.78 when $n = 5$), and
s = standard deviation of n replicates in 13.5.3 (Note 9).

NOTE 9—Here, s should be comparable to S_m, the repeatability standard deviation, given in Table 1. If s » S_m, there is evidence that the repeatability of the particular instrument is not acceptable for use with this test method. The user should determine and correct the cause, and repeat 13.5.1 through 13.5.3.

13.5.5 Confirm calibration linearity by analyzing a mid-range (Note 10) titanium reference material each for oxygen and nitrogen (a single reference material may be used if a suitable reference material is available for both analytes), using the limits stated on the certified value as an acceptance range. Alternatively, analyze at least three specimens of a mid-range (Note 10) titanium reference material for each analyte. Calculate the average and standard deviation(s) of these results. In the absence of bias among the reference materials, the average result for this reference material should agree with the certified value within a prediction interval defined by the repeatability of the measurement system at the mid-range of the calibration (Note 11). This prediction interval may be calculated using Eq 1 and the s and n values for the mid-range reference material. If the prediction interval does not encompass the certified value, determine and correct the cause and repeat 13.5.1 through 13.5.5 (Note 12).

NOTE 10—Commercially available reference materials are not always available at the compositions required to have a true mid-point check. The mid-range material must have a value that is above the limit of detection, but below that of the high calibration point, preferably as close to the mid-point of the calibration curve as possible.

NOTE 11—Typically, repeatability standard deviation is a function of the mass fraction of the analyte. Compare the values labeled *ILT Analyzed Mean* in Tables 1 and 2 with the values for *Minimum SD* (S_m) to see a typical trend for laboratories using this test method. If your results are not comparable, investigate and correct the cause.

NOTE 12—The presence of bias between the reference material used in 13.5.1 and the reference material used in 13.5.5 may cause the calibration to appear to be non-linear. This cannot be corrected by making adjustments to the instrument.

14. Procedure

14.1 Start with an instrument that has been prepared and calibrated as directed in Sections 10 and 13.

14.2 Transfer a 0.100 g to 0.150 g titanium test sample with nickel flux to the sample drop port. The weight of nickel should exceed the weight of sample by at least a factor of 7(Note 13.) The weight tolerance of the titanium test sample must be ±1 mg.

NOTE 13—Research has demonstrated that the effective ratio of flux to titanium is 7:1 to 17:1.[6] Nickel baskets are commonly manufactured in 1.0 g and 1.5 g sizes with the 1.0 g size in common usage among the titanium producers and users and the weight utilized in this method's ILS.

14.3 Place the crucible containing graphite powder (Note 6) on the furnace pedestal, raise the mechanism, and start the analysis cycle. Refer to the instrument manufacturer's instructions for the specific instrument model regarding operation, entry of sample weight, and blank value.

14.4 One or more continuing calibration verifications must be performed prior to and upon completion of a period of continuous operation, and throughout this period with a predetermined minimum frequency to be established by each individual test facility. The acceptance range for the verification material may be the limits stated on the certified value for the reference material, or may be calculated using Eq 1 and the s and n values for multiple analyses of the verification material. If a continuing calibration verification indicates an out of calibration condition, stop analysis. Results must be supported by acceptable preceding and subsequent verifications to be reported.

14.5 It is the responsibility of the user to document the frequency of blank determination (13.4), routine calibration and confirmation (13.5.1 – 13.5.4) and linearity confirmation (13.5.5), and the conditions under which blank determination or recalibration, or both, beyond this frequency is required (examples may include changing reagents, beginning use of a new batch of crucibles, changing gas cylinders, or a personnel shift change).

15. Calculation

15.1 Refer to the manufacturer's instructions to ensure that all essential variables in the analysis have been addressed. Because most modern commercially available instruments calculate mass fraction directly, including corrections for blank and sample mass, manual calculations by the analyst are generally not required.

16. Precision and Bias[7]

16.1 *Precision*—Oxygen*:*

16.1.1 Eleven laboratories cooperated in testing Samples 1 through 7. The data obtained are presented in Table 1. The testing was performed in accordance with the provisions of Practice E173. The results of the testing are recalculated in accordance with Practice E1601, Plan A.

16.1.2 In August 2004, eight laboratories tested Samples 8 and 9 that were outside the oxygen content range of the original testing. The testing was performed in accordance with Practice E1601, Plan A and the results calculated accordingly. The resultant data are presented in Table 1. The scope of Practice E1601 reflects this additional testing. The lower scope is calculated as (2)(R) of the lowest analyzed material plus consideration of the 0.008 % material included in the August 2004 ILS.

16.1.3 In October 2007, six laboratories tested one additional sample for the element oxygen only. The testing was performed in accordance with E1601, Plan A and the results calculated accordingly. The resultant data have been appended to Table 1. As a result of this testing, the scope of this test method has been modified to reflect the new oxygen scope maximum of 0.5 %. Supporting data have been filed at ASTM Headquarters in Research Report RR:E01-1109.[8]

16.2 *Precision*—Nitrogen*:*

16.2.1 Twelve laboratories cooperated in testing Samples 1 through 4. The data obtained are presented in Table 2. The testing was performed in accordance to the provisions of Practice E173. The results of the testing are recalculated in accordance with Practice E1601, Plan A.

16.2.2 In August 2004, eight laboratories tested Sample 5 that was outside the nitrogen content range of the original testing. The testing was performed in accordance with Practice E1601, Plan A, and the results calculated accordingly. The resultant data are presented in Table 2. The scope of Practice E1601 reflects this additional testing. The lower scope is calculated as (2)(R) of the lowest analyzed material plus the consideration that a 0.002 % material was included in the original ILS and a <0.001 % material included in the August 2004 ILS.

16.3 *Bias*—The accuracy of this test method may be judged by comparing the results obtained from certified reference materials with their certified values.

17. Keywords

17.1 inert gas fusion; nitrogen; oxygen; titanium; titanium alloys

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[6] *Method of Analyzing Oxygen or Nitrogen Contained in Titanium Group Metal or Alloy Thereof*, Okamura, et al., U.S. Patent Number 4,673,655, June 16, 1987.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1002 for oxygen and Research Report RR:E01-1024 for nitrogen.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1109.

APPENDIXES

(Nonmandatory Information)

X1. PREPARATION ANOVA OXYGEN

X1.1 See Table X1.1.

TABLE X1.1 Preparation ANOVA Oxygen

Treatment Code	Lab Code Random Run Order	A	B	C	D	E	F
b[A]	2	0.08265	0.0822	0.08178	0.08371	0.08065	0.08413
b[A]	11	0.08317	0.08177	0.08045	0.08422	0.08320	0.08591
b[A]	20	0.08162	0.08936	0.08257	0.07965	0.08374	0.08455
e[A]	6	0.08055	0.08445	0.08096	0.08377	0.08292	0.08476
e[A]	13	0.08322	0.08559	0.08079	0.08457	0.08266	0.08498
e[A]	21	0.08103	0.08107	0.08004	0.07952	0.08258	0.08542
f[A]	1	0.08372	0.08219	0.08036	0.08226	0.08540	0.08507
f[A]	10	0.08372	0.08265	0.08147	0.08355	0.08388	0.08501
f[A]	17	0.08140	0.08686	0.08098	0.08340	0.08244	0.08362
a[B]	4	0.08109	0.08366	0.08200	0.08749	0.08360	0.08375
a[B]	7	0.08173	0.08202	0.08035	0.08428	0.08444	0.08378
a[B]	14	0.08033	0.08568	0.07878	0.08327	0.08300	0.08460
c[B]	12	0.08543	0.08221	0.07999	0.08271	0.08475	0.08899
c[B]	15	0.08023	0.09032	0.08237	0.08468	0.08240	0.08550
c[B]	19	0.08139	0.08889	0.08384	0.08243	0.08316	0.08575
d[C]	3	0.08224	0.08265	0.08275	0.08507	0.08448	0.08683
d[C]	8	0.08413	0.08271	0.08181	0.08509	0.08411	0.08488
d[C]	18	0.08215	0.08624	0.08233	0.08471	0.08544	0.08407
g[C]	5	0.08281	0.08621	0.08398	0.08696	0.08487	0.08499
g[C]	9	0.08437	0.08292	0.08370	0.08622	0.08397	0.08396
g[C]	13	0.08279	0.08962	0.08114	0.08333	0.08344	0.08682

ANOVA Calculation:

Source of Variance	df	SS	MS	F	Significance at 95 % C.L.	% of Total Variance
Between Lab	5	0.01962	0.00393	12.2	Y	33.4
Between Preps	12	0.00549	0.00046	1.48	N	4.5
Error	108	0.03298	0.00031	...	...	62.1
Total	125	0.05809	...	...	...	...

[A] Laboratory B, E, F treatments b, e, f respectively: HF-H_2O_2.

[B] Laboratory A, C treatments a, c respectively: HF-HNO_3.

[C] Laboratory D, G treatments d, g respectively: Abrade.

X2. PREPARATION ANOVA NITROGEN

X2.1 See Table X2.1.

TABLE X2.1 Preparation ANOVA Nitrogen

Treatment Code	Lab Code Random Run Order	A	B	C	D	E	F
b[A]	2	0.00426	0.00526	0.00423	0.00520	0.00361	0.00335
b[A]	11	0.00454	0.00388	0.00469	0.00547	0.00362	0.00460
b[A]	20	0.00325	0.00470	0.00566	0.00466	0.00529	0.00414
e[A]	6	0.00399	0.00520	0.00409	0.00499	0.00555	0.00328
e[A]	13	0.00373	0.00461	0.00471	0.00531	0.00522	0.00404
e[A]	21	0.00332	0.00470	0.00423	0.00490	0.00493	0.00446
f[A]	1	0.00375	0.00542	0.00450	0.00491	0.00636	0.00426
f[A]	10	0.00367	0.00543	0.00453	0.00500	0.00552	0.00398
f[A]	17	0.00417	0.00444	0.00472	0.00425	0.00456	0.00489
a[B]	4	0.00399	0.00525	0.00432	0.00496	0.00510	0.00312
a[B]	7	0.00410	0.00476	0.00472	0.00497	0.00503	0.00404
a[B]	14	0.00352	0.00430	0.00486	0.00474	0.00517	0.00445
c[B]	12	0.00338	0.00505	0.00486	0.00498	0.00518	0.00414
c[B]	15	0.00366	0.00516	0.00542	0.00495	0.00416	0.00357
c[B]	19	0.00398	0.00523	0.00491	0.00557	0.00431	0.00479
d[C]	3	0.00457	0.00496	0.00537	0.00504	0.00436	0.00420
d[C]	8	0.00359	0.00541	0.00457	0.00512	0.00497	0.00416
d[C]	18	0.00345	0.00441	0.00478	0.00485	0.00564	0.00433
g[C]	5	0.00384	0.00465	0.00489	0.00612	0.00524	0.00387
g[C]	9	0.00446	0.00532	0.00449	0.00545	0.00551	0.00467
g[C]	16	0.00425	0.00531	0.00531	0.00529	0.00420	0.00468

ANOVA Calculation:

Source of Variance	df	SS	MS	F	Significance at 95 % C.L.	% of Total Variance
Between Lab	5	0.02514	0.00503	21.8	Y	49.1
Between Preps	12	0.00156	0.00013	0.54	N	0.0
Error	108	0.02612	0.00024	...	...	50.9
Total	125	0.05281	...	...	...	...

[A] Laboratory B, E, F treatments b, e, f respectively: HF-H_2O_2.
[B] Laboratory A, C treatments a, c respectively: HF-HNO_3.
[C] Laboratory D, G treatments d, g respectively: Abrade.