



Designation: C1854 – 17

Standard Test Method for Determination of Hydrogen (total from all sources) in Mixed Oxide ((U, Pu)O₂) Sintered Pellets by the Inert Gas Fusion Technique Followed by Thermal Conductivity Measurement¹

This standard is issued under the fixed designation C1854; 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 hydrogen in nuclear-grade mixed oxides of uranium and plutonium ((U, Pu)O₂) sintered fuel pellets. This test method is an alternative to Test Method C698 for the determination of moisture in nuclear-grade sintered mixed oxide (MOX) fuel pellets. Test Method C698 describes the detection of moisture in mixed oxides using a coulometric, electrolytic moisture analyzer. Although the main source of H₂ in the fuel pellets is moisture, there could be other sources. The MOX pellet Specification C833 specifies a limit for hydrogen from all sources, not only moisture. The inert gas fusion followed by thermal conductivity detector specified in this test method allows for detection of hydrogen from all sources. Therefore, this test method can be used to determine the limit specified in C833.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

C698 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ((U, Pu)O₂)

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

3. Terminology

3.1 For definitions of terms used in this test method but not defined herein, refer to Terminology C859.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *MOX*—nuclear fuel composed of a mixture of uranium and plutonium oxides ((U, Pu)O₂).

3.2.2 *reference material*—material traceable to a reference material from a national standards body such as the U.S. National Institute for Standards and Technology (NIST) or equivalent.

3.2.3 *sintering*—to increase the bonding in a mass of powder or a compact by heating below the melting point of the main constituent.

3.3 *Acronyms:*

3.3.1 *LIMS*—Laboratory Information Management System

3.3.2 *TCD*—Thermal Conductivity Detector

4. Summary of Test Method

4.1 The method for the determination of total hydrogen (H₂) from all sources presented in this test method consists of fusion

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved June 1, 2017. Published June 2017. DOI: 10.1520/C1854-17.

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.

of the sample in an electrode impulse furnace in a stream of nitrogen (N₂) or argon (Ar) gas at a temperature sufficient to release all hydrogen in the sample. The stream of gas carries the released hydrogen through a series of filters to remove interfering impurities and the hydrogen is measured by TCD.

4.2 The specimen, a single MOX pellet weighing approximately 6 g, contained in a small single-use graphite crucible, is fused under a flowing nitrogen (N₂) or argon (Ar) atmosphere and at a temperature greater than 1770°C. Hydrogen present in the sample is released as molecular hydrogen into the flowing carrier gas. Other gases are also liberated into the carrier gas, such as carbon monoxide, and need to be removed before they reach the thermal conductivity detector as they interfere with the measurement. These sample impurities are swept by the carrier gas through a series of filters to remove dust and water and then the gases flow through a quartz tube filled with Schuetze reagent (see 8.3) and activated charcoal. The Schuetze reagent acts as an oxidizing agent to convert carbon monoxide (CO) to carbon dioxide (CO₂). The gas then flows through a molecular sieve to separate nitrogen from hydrogen by size, delaying the larger nitrogen molecule and allowing the hydrogen to reach the TCD before the nitrogen.

4.3 The carrier gas, now free of interfering species then flows through the measurement branch of the thermal conductivity cell which is where the quantitative detection of the hydrogen released from the sample takes place. The hydrogen concentration in the carrier gas is measured by the TCD which detects small changes in the thermal conductivity of the carrier gas containing the liberated hydrogen gas compared to the thermal conductivity of the carrier gas alone.

4.4 The detector signal plotted versus time is a function of the concentration of the H₂ in a carrier gas. The area below the curve (integral) corresponds to the total amount of hydrogen in the sample. The peak is integrated by the software and the concentration is calculated taking into account the calibration factor, the blank analysis and the sample weight. The calibration of the analyzer is made by means of a reference material. Blank values are obtained from analyzing the empty crucibles. The blank results are stored. The final sample result is corrected by the blank value and the results are expressed in µg hydrogen/g MOX.

5. Significance and Use

5.1 MOX is used as a nuclear-reactor fuel. This test method is designed to determine whether the hydrogen content of the pellets meet the requirements of fuel specification. Examples of these requirements are given in Specification C833. Other requirements may apply based on agreements between the supplier and the customer.

5.2 This method is suitable for all sintered MOX pellets containing up to 15 weight % PuO₂ when the UO₂ and PuO₂ meet the requirements of Specifications C753 and C757. The method uncertainty is related to the concentration of the hydrogen in the sample. At lower concentrations, the relative

uncertainty increases. At hydrogen contents close to the typical hydrogen content specification limit (1.3 µg hydrogen/g U + Pu metal); the combined relative uncertainty at the 95 % confidence level (k = 2) is approximately 30 %.

6. Interferences

6.1 After the sample fusion, the carrier gas containing the carbon dioxide and other potentially interfering impurities (sulfur, water, and small particulate matter) pass through a series of filters and purifying reagents that remove these impurities from the carrier gas stream leaving only H₂ in the stream at the detector. If the MOX pellets are made from UO₂ and PuO₂ that meet the requirements of Specifications C753 and C757, all interferences are eliminated by the purification system.

6.2 The crucibles, if they contain hydrogen, will yield erroneously high results for the sample. The analytical method requires running blanks to correct for this potential interference in the calculation of the results (see 12.2).

6.3 The nitrogen (N₂) or argon (Ar) carrier gas could contain water and CO₂ and is filtered prior to injection in the sample combustion chamber to remove these potentially interfering components.

6.4 Weighing uncertainty of the samples is critical to the method. If the balance meets the specification in 7.1, is calibrated in accordance with manufacturer's guidance, and is checked by procedure, the potential for the balance to be a source of error is insignificant.

6.5 When using nitrogen gas with graphite crucible a secondary reaction occurs at high temperatures (>3000°C) where HCN is created, causing a bias that increases with temperature. Argon as carrier gas is used at higher temperatures to avoid this reaction.

7. Apparatus

7.1 *Analytical Balance*, with precision ±0.1 mg.

7.2 *Graphite Crucibles*—Use the crucibles of size recommended by the manufacturer of the instrument. Crucibles shall be composed of high purity graphite.

7.3 *Hydrogen analyzer*, consisting of an electrode impulse furnace suitable for operation at 1770 to 2200°C, a TCD for measuring H₂, a nitrogen or argon carrier gas injection system, auxiliary gas purification systems, and a chiller to cool the electrodes. The analyzer typically has an integrated data collection and analysis software system that allows for efficient collection and analysis of data.

7.4 *Tongs and Forceps*, for handling crucibles.

7.5 *Stainless Steel Scoops and Spatulas*, for handling pellets and reference materials.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without compromising the accuracy of the determination.

8.2 *Molecular Sieve (Zeolites of Silicon and Aluminum)*—Captures CO₂ from the carrier gas.

8.3 *Schuetze Reagent (I₂O₅ and SiO₂)*—Oxidizes CO to CO₂.

NOTE 1—Schuetze reagent is commercially available and is typically not prepared in-house. If the user of this test method wishes to prepare the Schuetze reagent in house, a procedure is given in [Appendix X1](#).

8.4 *Activated Coal or Sodium Hydroxide (NaOH)*—absorbs CO₂ before and after the CO and CO₂ conversion.

8.5 *Quartz Wool*, for dust traps and to plug the reagent tubes.

8.6 *Platinum Wire (if needed)*, for minimum furnace temperature verification, 1.0 mm diameter, 99.99 % trace metal basis.

8.7 *Rhodium Wire (if needed)*, for maximum furnace temperature verification, 1.0 mm diameter, 99.9 % trace metal basis.

8.8 *Nitrogen or Argon Carrier Gas*, ≥99.999 % purity, inlet pressure: 200 kPa or as specified by the equipment manufacturer.

8.9 *Magnesium Perchlorate (Mg(ClO₄)₂)*, removes H₂O.

9. Reference Materials

9.1 The calibration of the analyzer is made by measuring materials with hydrogen content in the range of concentration expected in the MOX pellet (~1.3 ppm) traceable to a reference material from a national standards body such as the U.S. National Institute for Standards and Technology (NIST) or equivalent. Suitable materials traceable to a reference material in steel matrices (steel pins, steel rings, steel granules, and steel powder) are available and have been found satisfactory. Matrix matched reference materials for MOX pellets are not available.

10. Precautions

10.1 Because of the toxicity of plutonium, all operations should be performed within an approved glove box fitted with appropriate filters to contain any small particle of plutonium. A detailed discussion of the necessary precautions is beyond the scope of this test method. Personnel involved in these analyses should be familiar with safe handling practices for radiologically controlled materials.

10.2 The furnace, sample tube and sample crucibles are heated to >1770°C. Care should be taken to avoid contacting

gloves with hot surfaces. Typically these hot surfaces are guarded and inaccessible during the heating process and therefore do not pose a risk to the operator.

10.3 Exercise appropriate caution when working with compressed gases.

10.4 This procedure uses hazardous chemicals. Use appropriate precautions for handling corrosives, oxidizers, and gases.

11. Preparation and Verification of Apparatus Prior to Sample Analysis

11.1 Turn on the analyzer and set the operating controls of the instrument system according to the operating instructions for the specific equipment used.

11.2 *Verification of the Gases*—Carrier gas flow and system pressure are two essential parameters that must be controlled to ensure satisfactory performance of the instrument. Most analyzers are equipped with pressure regulation and electronic flow control.

11.2.1 Ensure that the regulator valve is set to the correct value for the nitrogen line per manufacturers' recommendations.

11.3 *Verification of the Reagents*—Change instrument column packing and reagents as recommended by manufacturer.

11.3.1 The molecular sieve is usually changed or regenerated after eight hours of continuous analysis, but exact change out times vary based on analyzer model and use.

11.3.2 The Schuetze reagent and activated carbon should be replaced approximately 40 % of the color of the Schuetze reagent changes from lemon yellow to pink or brown.

11.3.3 The quartz wool should be replaced when visible particulate matter is observed.

11.3.4 At regular intervals and at each replacement of the reagents, the O-rings at the reagent tube holders must be inspected for wear and tear and replaced when appropriate.

11.4 *Verification of the Furnace Temperature*—In some cases, the furnace is designed with an automatic temperature sensor or pyrometer. In these cases, temperature verification can easily be performed per the manufacturer's instructions. In other cases where automatic temperature sensors are not available, it is necessary to use pure metal standards with known melting points to verify furnace temperature above 1770°C and well below 2200°C. Metals such as platinum and rhodium can be used. For example, if platinum melts, and rhodium does not melt, the verification is successful. If the sample is heated above 2200°C, significant amounts of carbon dioxide can be released as a result of the reduction of UO₂ by the graphite crucible which could interfere with the detection of the hydrogen by the TCD. Therefore avoiding temperatures near 2200°C is important.

11.5 *Cleaning the Analyzer*—It is typically recommended that furnace cleaning should be performed after approximately 20 samples have been analyzed. Clean the furnace according to the operating instructions for the specific equipment used. Cleaning typically includes cleaning of the furnace chamber, the top electrode insert, the front end of the quartz rod and the

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

O-ring on the electrode base. A small amount of silicone grease should be applied to the O-ring.

11.6 *Performing a Leak Test*—A blank should be run after the analyzer has been cleaned and after replacement of any reagent tubes. This blank run serves as a leak test.

11.7 *General Verifications*—Depending on the type of analyzer, other parameters that could require verification prior to sample analysis include the voltage supply, current and temperature of the TCD and the cooling water flowrate of the furnace electrodes.

11.8 *Verifications of Molecular Sieve*—When two peaks with little to no separation appear in the analysis plot, it is time to replace the molecular sieve. The time separating the nitrogen and hydrogen peak will vary depending on packing material, instrument, and method parameters.

12. Calibration of the Instrument

12.1 The calibration range and number of reference materials analyzed will depend upon the instrument used and the expected range of hydrogen in the samples being analyzed. Two to four blank measurements and two to four reference materials are recommended for performing a calibration. A one point calibration in the range of expected hydrogen content in the MOX pellet is sufficient for this method.

12.2 Using a pair of tongs, load a new crucible into the furnace and start a blank analysis according to the manufacturers' recommended operating conditions. The blank result is recorded automatically in the analyzer software.

12.3 Repeat 12.2 two to four times. It is recommended to check inter-sample blank results for statistical consistency prior to using the data for calibration purposes. If the values obtained do not show statistical correlation, they should not be used for calibration of the analyzer and may be indicative of analyzer malfunction.

12.4 Using a spatula or a pair of tongs, weigh approximately 1 g of reference material to the nearest 0.1 mg and record the weight of the reference material in the analyzer.

12.5 Using a spatula or pair of tongs, load the crucible into the furnace and start the analysis according to the manufacturers' recommended operating conditions.⁴ The result is typically automatically corrected for the blank content and the result is recorded automatically in the analyzer software. If the analyzer does not have this functionality, the analyst must manually subtract the blank from the result. In both cases the data is collected and stored for use in step 12.7.

12.6 Repeat step 12.5 two to four times. It is recommended to check inter-sample reference material results for statistical consistency prior to using the data for calibration purposes. If the values obtained do not show statistical correlation, they should not be used for calibration of the analyzer and may be indicative of analyzer malfunction.

⁴ Typically an analysis sequence begins with an outgas step where the sample chamber is purged of air. The analyzer will then prompt the operator for introduction of the sample or reference standard. Most analyzers allow for addition of the sample through a port consisting of an isolation valve that automatically inserts the sample into the furnace chamber without introducing air into the chamber.

12.7 The data collected in steps 12.2 – 12.6 and stored in the analyzer is used for establishing the calibration curve of the instrument according to the manufacturers' operating instructions. Calibration coefficients are normally automatically calculated by the analyzer software and stored in the analyzer software.

12.8 Verify the calibration using the reference material measurements. The measurements should agree with the reference value within a suitable confidence interval. If the result agrees with the reference material value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Also, if the measured value falls within an established control chart limit, the calibration is acceptable. If not, repeat steps 12.2 – 12.8.

NOTE 2—Recalibration frequency will depend upon the type of instrument used. As a minimum, recalibration is required when critical instrument components are changed, or when control chart data indicates that the instrument is failing to meet performance criteria.

13. Procedure

13.1 *Pre-Sample Analysis Blank Measurement*—A blank measurement is performed prior to sample analysis in order to check that the background hydrogen signal is not above an acceptable limit. The signal from the blank is saved and automatically subtracted from the sample (MOX) signal to take into account hydrogen in the crucible and carrier gas.

13.1.1 Using a pair of tongs, load the crucible into the furnace and start a blank analysis according to the manufacturers' recommended operating conditions. The blank result is recorded automatically in the analyzer software.

13.1.2 The blank result should not be higher than the detection limit established for the analyzer as described in Guide C1068. If the result is below the detection limit, proceed to step 13.2. If the limit is exceeded, the furnace should be cleaned and another blank should be run. If the blank is still not low enough, the filtering reagents may need to be changed.

13.2 *Pre-Sample Calibration Verification*—An analysis of a reference material is performed prior to sample analysis in order to verify that the calibration and the stored calibration coefficients in the analyzer software are still valid.

13.2.1 Using a spatula or a pair of tongs, weigh approximately 1 g of reference material to the nearest 0.1 mg and record the weight of the reference material in the analyzer.

13.2.2 Using a pair of tongs, load the crucible into the furnace and start the analysis according to the manufacturers' recommended operating conditions. The result is typically automatically corrected for the blank content and the result is recorded automatically in the analyzer software. If the analyzer does not have this functionality, the analyst must manually subtract the blank from the result.

13.2.3 The calibration coefficient is valid if the result of the reference material analysis agrees with the reference value within the combined uncertainty. Also, if the measured value falls within an established control chart limit, the calibration is acceptable and the operator can proceed to step 13.3. If not, repeat steps 12.1 – 12.6.

13.3 Sample Analysis—An analysis is performed on an entire MOX pellet due to the low levels of hydrogen expected, and the need for sufficient sample to obtain a clear signal.

13.3.1 Using a spatula or a pair of tongs, weigh one pellet to the nearest 0.1 mg and record the weight in the analyzer.

13.3.2 Using a pair of tongs, load the crucible into the furnace and start the analysis according to the manufacturers' recommended operating conditions. The result is automatically corrected for the blank content and the result is recorded automatically in the analyzer software.

13.3.3 Repeat steps **13.3.1** and **13.3.2** as many times as required in order to gather sufficient replicate data. It is recommended to check replicate sample results for statistical consistency. If the values obtained do not show statistical agreement, it may be indicative of analyzer or process malfunction, but statistical consistency is not a prerequisite for reporting results.

NOTE 3—The number of replicates required per sample or lot will depend on the instrument used, the operating conditions, the client requirements, and the data quality objectives. Typically five samples are measured so that an average of the five analyses can be calculated and reported for a sample or lot result.

NOTE 4—The number of sample or lots that can be performed prior to steps **13.4** and **13.5** depend on the instrument used and the operating conditions. Typically, ten samples can be analyzed before checking analyzer calibration via the analysis of a blank and a reference material.

13.4 Post Sample Analysis Blank Measurement—A blank measurement is performed after sample analysis in order to ensure that the background hydrogen signal is not above an acceptable limit.

13.4.1 Using a pair of tongs, load the new crucible into the furnace and start a blank analysis according to the manufacturers' recommended operating conditions. The blank result is recorded automatically in the analyzer software.

13.4.2 The blank result should not be higher than the limit of detection established for the analyzer. If the result is below the limit of detection established for the analyzer, proceed to step **13.5**. If the limit is exceeded, the furnace should be cleaned and another blank should be run. If the blank is still not low enough, the results obtained in step **13.3** cannot be reported.

13.5 Post-Sample Calibration Verification—An analysis of a reference material is performed after sample analysis in order to verify that the calibration of the instrument and the stored calibration coefficients in the analyzer software are still valid. The calibration coefficients are valid if the result of the reference material agrees with the reference value within the combined uncertainty or if the measured value falls within an established control chart limit.

13.5.1 Using a spatula or a pair of tongs, weigh approximately 1 g of reference material to the nearest 0.1 mg and record the weight of the reference material in the analyzer.

13.5.2 Using a pair of tongs, load the crucible into the furnace and start the analysis according to the manufacturers' recommended operating conditions. The result is typically automatically corrected for the blank content and the result is recorded automatically in the analyzer software. If the analyzer does not have this functionality, the analyst must manually subtract the blank from the result.

13.5.3 The calibration coefficient is valid if the result of the reference material agrees with the reference material value within the uncertainty provided on the certificate of analysis. Also, if the measured value falls within an established control chart limit, the calibration is acceptable. If not, repeat steps **13.5.1** to **13.5.2**. If the result agrees with the reference material value within the uncertainty provided on the certificate of analysis or if the measured value falls within an established control chart limit, the calibration is acceptable. If not, the results obtained in step **13.3** cannot be reported.

NOTE 5—The following parameters should be checked during the analysis and are often automatically monitored by the analyzer software: carrier gas pressure when the furnace is opening, gas pressure during leak test, TCD reference gas flow during analysis, and purge gas flow (during the outgassing of the empty graphite crucible).

14. Calculation

14.1 Calculate the hydrogen content for each replicate as follows (this calculation is typically performed by the analyzer software):

$$[H] = \frac{(H_s - H_b)}{W} \quad (1)$$

where:

$[H]$ = micrograms of hydrogen per gram sample (MOX),
 H_s = micrograms of hydrogen in sample (MOX),
 H_b = micrograms of hydrogen in a blank run, and
 W = grams of sample (MOX).

If multiple replicates are measured, the average content of hydrogen $[H]_a$ is calculated as,

$$[H]_a = \frac{[H]_{(n)} + [H]_{(n-1)} + [H]_{(n-2)} + \dots}{n} \quad (2)$$

Where $[H]_a$ is calculated either by the analyzer software or independently by an operator or LIMS system.

14.2 For samples requiring hydrogen results expressed as μg hydrogen per g U + Pu, convert results to uranium plus plutonium metal basis as follows:

$$[H_m] = \frac{[H]}{f} \text{ or } \frac{[H]_a}{f} \quad (3)$$

where:

$[H_m]$ = micrograms of hydrogen per gram of U + Pu in sample (MOX), and
 f = mass fraction of U + Pu metal to UO_2 + PuO_2 in sample (MOX).

15. Precision and Bias

15.1 Within the different stages of the nuclear fuel cycle, many challenges lead to the inability to perform interlaboratory studies for precision and bias. These challenges may include variability of matrices of material tested, lack of suitable reference materials, limited laboratories performing testing, shipment of materials to be tested, and regulatory constraints. Because of this, each individual laboratory utilizing these test methods should establish their own precision and bias limits as part of their quality assurance program. Guidance can be found in Guide **C1068**.

15.2 *Precision*—The repeatability of the method as a relative standard deviation (1σ) as determined by a single laboratory using non-matrix matched reference material traceable to a reference material has been determined to be approximately 15 % at approximately 1 ppm H_2 . The reproducibility of this test method is not provided at this time because very few laboratories perform this analysis and because of the issues stated at the beginning of this section. As a result, it is not likely that reproducibility data will be available in the future.

15.3 *Bias*—The bias of the test method cannot be tested reliably because of the lack of suitable matrix matched reference materials. The bias observed using non-matrix matched reference material traceable to a reference material is approximately 6 % at approximately 1 ppm.

16. Keywords

16.1 hydrogen content; impurity content; mixed oxide; MOX; plutonium oxide; uranium oxide

APPENDIX

(Nonmandatory Information)

X1. PREPARATION OF SCHUETZE OXIDIZING REAGENT

X1.1 Dissolve 50 g of iodine pentoxide or 53 g of iodic acid in 250 mL of water.

X1.2 Add the solution to 500 g of coarse (6- to 16-mesh) silica gel and mix well. Dry at approximately 150°C for 18 to 19 h.

X1.3 Cool to room temperature, add 100 mL of 18M H_2SO_4 , mix well, vent the container frequently, and store

overnight in a closed container.

X1.4 Pack the mixture into a glass, 2000 mL volumetric flask, flush the tube with dry argon at 250 to 300 cm^3/min while heating the mixture to approximately 220°C for 2 h.

X1.5 Turn off the heat but continue the argon flow until the reagent is cool.

X1.6 Store the reagent in a capped glass bottle under argon.

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