

NE M 14-2T

Supersedes NE M 14-2T

January 1984

nuclear STANDARD

FUEL AND CONTROL ASSEMBLY TAG GAS

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JANUARY 1986

U. S. DEPARTMENT OF ENERGY
NUCLEAR ENERGY PROGRAMS

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Westinghouse
Hanford Company
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Westinghouse Electric
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NE Standards Transmittal

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P. O. Box 1970
Richland, WA 99352

Hanford Engineering Development Laboratory

NE-M--14-2T-Rev.1/86

DE93 011211

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	NE M 14-2T	Fuel and Control Assembly Tag Gas. January 1986. Supersedes January 1984 issue of NE M 14-2T.

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FOREWORD

This standard supersedes the January 1984 issue of NE M 14-2T and incorporates those changes to that issue of the standard that were approved for publication in this revision. These changes are identified by the following marginal notations:

C	Change approved	1-14-86
D	Deletion approved	1-14-86
N	Addition approved	1-14-86

Editorial changes that were made during the preparation of this revision are not identified.

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FUEL AND CONTROL ASSEMBLY TAG GAS

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FUEL AND CONTROL ASSEMBLY TAG GAS

1. SCOPE

This standard establishes the requirements for tag gas to be used for locating failed fuel pins and control rod absorber pins in the reactor by the failure monitoring system. Tag gas shall consist of varying isotopic mixtures of xenon, krypton, or other gases as specified in the Ordering Data. 1D

2. BASIS OF PURCHASE

Orders for material under this standard shall include the following information (numbers in parenthesis refer to paragraphs in this standard):

1. Title, number and date of this standard.
2. Specify isotopically enriched and naturally occurring gases required (3.).
3. Specify blending procedure if other than reference process (4.1).
4. Specify required composition (4.2).
5. Specify gas cylinder pressure (5.2).
6. Specify volume of cylinders (5.3).
7. Specify type of cylinder valve (5.4).
8. Specify sampling requirements (6.2).
9. Specify amount of tag gas for archive sample (6.3).
10. Quality verification requirements (7.1).
11. Specify cylinder shipping containers (8.1).

3. TAG GAS CONSTITUENTS

Tag gas constituents, isotopically enriched and naturally occurring gases, shall be of such a quality that when blended, the resulting tag gas will meet the requirements for composition and purity specified in 4.2, 4.3, and 4.4. Tag gas constituents will be specified in the Ordering Data.

4. TAG GAS BLENDS

4.1 Blending. Tag gases may be prepared by blending isotopically enriched and naturally occurring gases in accordance with Appendix X1, or as otherwise specified in the Ordering Data.

4.2 Composition. The composition of each tag gas blend shall meet requirements specified in the Ordering Data. The tag gas composition shall be reported to the purchaser.

4.3 Purity. The blended tag gas; e.g., xenon and krypton mix, shall be 99.0% pure.

4.4 Impurities. Tag gas shall be analyzed for impurities. Individual impurities shall not exceed the maximum limits specified in Table 1. Total impurities shall be consistent with the purity requirement of 4.3. Other impurities found during analysis shall be reported to the purchaser.

Table 1. Individual Impurity Limits for Blended Tag Gas

Impurity	Mole Percent, Max
Hydrogen	0.5
Nitrogen	0.5
Carbon dioxide	0.5
Total hydrocarbons (includes halogenated hydrocarbons)	0.2

5. CONTAINERS

5.1 Tag gas shall be stored and shipped in containers that will ensure conformance to this standard upon receipt and storage at the designated destination. A description of containers shall be submitted to the purchaser for approval prior to use.

5.2 Pressure. Tag gas shall be delivered in compressed gas cylinders at the pressure specified in the Ordering Data.

5.3 Volume. Tag gas cylinder volume shall be as specified in the Ordering Data.

5.4 Container Valve. The cylinder valve shall meet requirements specified in the Ordering Data.

5.5 Cleanliness. Cylinders shall be clean and free of rust, oil, plastic, dirt, moisture, or other material that might contaminate the delivered product.

5.6 Leakage. Tag gas cylinders equipped with valves shall be helium leak tested before each use to $5 \times 10^{-6} \text{ cm}^3/\text{sec}$ [$5 \times 10^{-12} \text{ m}^3/\text{sec}$] (STP). The leak test procedure shall be submitted to the purchaser for approval prior to use.

6. INSPECTION REQUIREMENTS

6.1 Tag gas blends shall be inspected for conformance with requirements for each gas ordered.

6.2 Sampling. Tag gas shall be sampled as specified in the Ordering Data.

6.3 Archive Samples. Archive samples of tag gas blends shall be provided as specified in the Ordering Data. The archive samples shall meet all requirements of this standard and Ordering Data and shall be identified with type, tag identification and purchase order number. The archive samples shall be delivered to the purchaser with the gas.

6.4 Reference Test Method. Tag gas isotopic composition, purity and impurities shall be determined by use of a gas mass spectrometer. Reference test methods in Appendixes X2 and X3, respectively are the preferred methods. Other analytical methods may be substituted for the reference test method provided that equivalent results have been demonstrated; i.e., the alternate method has been qualified and provides a measurement error not greater than that typical of the reference method, and that the method shall have been approved by the purchaser prior to implementation.

7. QUALITY ASSURANCE REQUIREMENTS

7.1 Quality Assurance Program Requirements. The supplier shall plan, establish, implement, and maintain a documented quality assurance program that fulfills the ANSI/ASME NQA-1 requirements specified in the Ordering Data.

7.2 Quality Assurance Documentation. The supplier is responsible for the preparation of all quality assurance documentation specified in this standard and in the Ordering Data. Records of all tests and examinations shall be kept complete and available to the purchaser. Quality verification records shall include:

1. The identity of each tag gas shown on the inspection or test and other quality status records.
2. Reports showing the results of all inspections and tests required by this standard.
3. Other data which show the quality status of the tag gas.

Quality verification records shall be furnished with the delivered material.

7.3 Inspection or Test Level. Acceptability of a tag gas shall be determined by tests on representative samples taken in accordance with 6.2. Tag gas shall be accepted only when all specified requirements are met.

7.4 Measuring and Test Equipment Environmental Controls. Measuring and test equipment and measurement standards shall be calibrated and utilized in an environment controlled to the extent necessary to assure continued measurements of required accuracy, giving due consideration to temperature, humidity, vibration, cleanliness, and other controllable factors affecting precision measurement. When applicable, compensating corrections shall be applied to calibration results obtained in an environment which departs from standard conditions.

7.5 Tolerance Limit Interpretation. For purposes of determining conformance with isotopic requirements and impurity limits of this standard and Ordering Data, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.

8. PREPARATION FOR DELIVERY

8.1 Shipping Containers. Tag gas cylinders shall be shipped in containers of the size specified in the Ordering Data. The containers shall comply with ICC regulations and other state and local regulations pertaining to the shipment of the product.

8.2 Identification. Each tag gas cylinder and its shipping container shall identify the type of gas being shipped, along with the pressure, the tag number, the number of this standard, the purchase order number, and the supplier lot number.

9. REFERENCE DOCUMENTS

The following documents are a part of this standard to the extent specified in Sections 3 through 8. The issue of a document in effect on the date of the invitation to bid, including any amendments or other published changes also in effect on that date, shall apply unless otherwise specified. Where this standard appears to conflict with the

requirements of a reference document, such conflict shall be brought to the attention of the purchaser for resolution.

9.1 American National Standards (ANSI).

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities

9.2 American Society for Testing and Materials (ASTM) Standards.

ASTM E 29 Recommended Practices for Indicating Which Places of Figures are to be Considered Significant in Specified Limiting Values

APPENDIX X1-NON-MANDATORY

TAG GAS BLENDING

X1.1 SUMMARY

Natural and isotopically enriched gases of the same element are blended to desired isotopic compositions by cylinder rotation. In this method, two or more gases are combined in a cylinder and then the cylinder is rotated to obtain complete isotopic mixing of gases.

X1.2 APPARATUS

A gas blending apparatus can be used having the components listed below (see Fig. X1). The apparatus is designed to be operated under vacuum from 10^{-7} torr (1.3×10^{-5} Pa) to atmospheric pressure. Gases to be blended are added to the apparatus from pressurized gas cylinders and once added to the blending apparatus, they are handled under less than atmospheric pressure. Pressures above atmospheric pressure are not encountered again until the blended gas is sealed in the storage cylinder at room temperature.

1. Cylinder (stainless steel) having a volume consistent with the amount of tag gas required per blend.
2. Capacitance manometer.¹
3. Dewar.
4. Header (stainless steel).
5. Ion gauge.
6. Leak valves
7. High vacuum pump.
8. Sampling cylinder (stainless steel).

¹A MKS Baratron manometer can be used.

9. Storage Cylinder (stainless steel) and valve. For a 2 L volume of gas at STP contained in a 75 mL cylinder at room temperature (25°C), the pressure of the gas in the cylinder will be about 430 psi (2.96 MPa).^{2,3}
10. Thermocouple gauge.
11. Thermometer.
12. Tubing (metal).
13. Vacuum pump, mechanical.
14. Valves and joints, assorted, that have been checked by a helium leak test.

X1.3 REAGENTS AND STANDARDS

1. Various gases, natural and enriched, specified according to the gas tags required.
2. Liquid nitrogen

X1.4 CALIBRATION

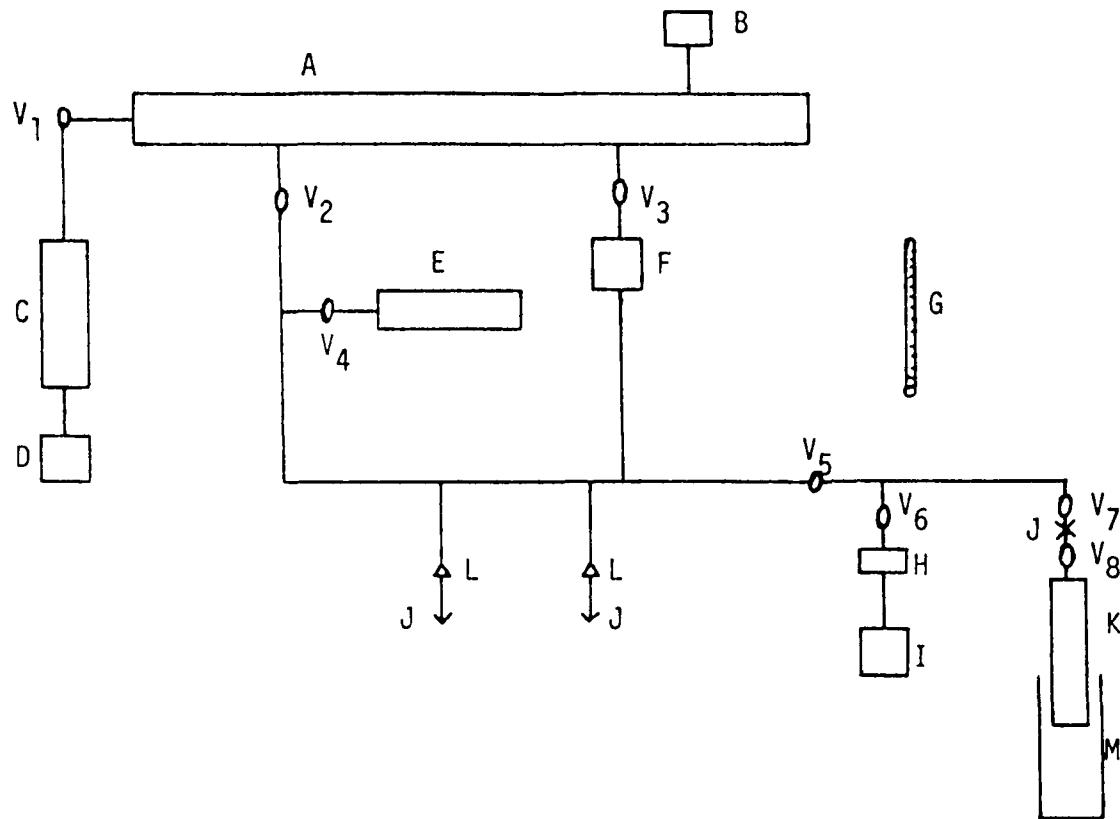
The blending volume of the gas blending apparatus shall be calibrated by introducing known volumes of a gas into the system and measuring the pressures with a capacitance manometer.

X1.5 BLENDING PROCEDURE

The gases are blended by rotating the storage cylinder slowly (~2 rps) for several hours (usually four or more). The total volume of blended gas and enrichment required will be specified in the Ordering Data. When each item of equipment is first mentioned in this procedure, it is identified by a letter that refers to Fig. X1.

²A Whitey gas cylinder, Catalog No. HDF 4-75-304, has been used for this purpose. This cylinder has a 75 mL volume, is fabricated from type 304 stainless steel, and is designed for pressures up to 1800 psig (12.4 MPa). (Whitey Research Tool Co., Emeryville, California.)

³A NUPRO valve, Catalog No. 55-4H2, has been used for this purpose. This valve is fabricated from type 316 stainless steel and is rated for pressures up to 1000 psi (6.89 MPa). (NUPRO Company, Cleveland, Ohio.)



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A	Header	F	Capacitance Manometer	K	Storage Cylinder
B	Ion Gauge	G	Thermometer	L	Leak Valves
C	Oil Diffusion Pump	H	Thermocouple Gauge	M	Dewar
D	Vacuum Pump	I	Vacuum Pump		
E	Blending Cylinder	J	Joints	V	Valves

Fig. XI. Tag Gas Blending Apparatus.

a. Calculate the milliliters of each gas needed to give the specified volume and enrichment required.

The isotopic ratio of each gas must be known and it should be verified by mass spectrometry (Appendix X2).

b. Calculate the pressure of each gas individually and of the combined gases when the volumes calculated at step (a) are introduced into the blending volume of the apparatus at room temperature.

c. Attach each gas cylinder containing a gas to be blended to a leak valve (L).

d. Evacuate the entire apparatus to at least 10^{-7} torr (1.3×10^{-5} Pa) with valves V₁ through V₈ and leak valves open.

The gas cylinder valves must be closed.

e. Adjust the capacitance manometer (F) to zero.

f. Close valve V₂ to the header (A), valve V₆, and valves V₇ and V₈ to the storage cylinder (K).

g. Close each leak valve and open each gas cylinder valve.

h. Bleed gas through the appropriate leak valve from one of the gas cylinders until the pressure calculated for that gas at step (b) is reached.

Usually the first gas introduced is the one requiring the lowest pressure.

i. Repeat step (h) for each of the gases remaining until the combined pressure calculated at step (b) is reached.

j. Open the valves V₇ and V₈ to the storage cylinder and pump the gas mixture into that cylinder by cooling with a Dewar (M) containing liquid nitrogen.

The manometer will indicate when the gas has liquefied or solidified.

k. Close valves V₇ and V₈; remove the storage cylinder.

l. Mix the gas by rotating the storage cylinder at about one or two revolutions per second for at least 4 h.

m. Sample the gas at room temperature and determine the isotopic ratios by mass spectrometry.

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If the required isotopic ratio has not been obtained, proceed to step (n).

n. Calculate the additional amounts of gases required to adjust the isotopic ratio of the mixture to the required value.

o. Add those calculated amounts, blend again, and check the isotopic ratio again by following steps (h) through (m).

The blending process is now completed and the tag gas is ready for use. Tag gas shall be removed from the storage cylinder only at temperatures greater than 15°C.

X1.6 PRECISION

The precision of the isotopic ratio measurement by mass spectrometry (Appendix X2) limits the determination of blending precision.

APPENDIX X2-NON-MANDATORY

TAG GAS ISOTOPIC RATIO MEASUREMENT

X2.1 APPLICATION

This analytical method applies to gases such as xenon and krypton and gas mixtures used to tag fuel and control assemblies.

X2.2 SUMMARY

The isotopic ratios of natural, enriched, and isotopically blended gases are measured by electron bombardment gas mass spectrometry.

X2.3 INTERFERENCES

Interferences are not expected unless residual gas from a previous sample of differing isotopic composition is left in the system.

X2.4 APPARATUS

Mass spectrometers are available commercially that meet or exceed the following requirements:

1. Sample inlet, static mode, viscous or molecular leak.
2. Source, electron bombardment ionization.
3. Analyzer (and vacuum system) capable of producing a resolving power of at least 150. Resolving power is defined as $M/\Delta M$, where ΔM is the width, in atomic mass units, of a peak at mass M at 5% of its height.
4. Detector, Faraday cup, electron multiplier, or Faraday cage and cup dual detector system.

X2.5 REAGENTS AND STANDARDS

1. Krypton, research grade¹ or equivalent, natural isotopic composition.
2. Xenon, research grade¹ or equivalent, natural isotopic composition.

¹Matheson Gas Data Book. The Matheson Co., Inc., East Rutherford, N. J., 4th Ed., 1966.

X2.6 CALIBRATION

Calibration standards shall be high purity natural krypton, xenon, or other appropriate natural gases. Periodic attention must be given to bias and to counting dead time when an ion-counting technique is used. These factors are determined when the mass spectrometer is first used and they should be checked whenever a calibration result is obtained that significantly deviates from the accepted value. They should be checked at periodic intervals.

X2.7 ANALYSIS²

a. Introduce sample into the ion source of the mass spectrometer through the appropriate inlet system.

b. Scan the isotopic spectrum repeatedly in both directions until at least five scans have been recorded.

X2.8 CALCULATION

Calculate the desired isotopic ratios. The calculation procedure is governed by the type of instrument used.

X2.9 PRECISION

Precision depends significantly upon the particular instrument used. Most instruments can provide a precision of at least 0.5% relative standard deviation.³ A relative standard deviation of 0.1% or less can be obtained using instruments equipped with a dual viscous inlet and a dual detector system.

²Detailed instructions for operating the mass spectrometer depend upon the particular instrument used. Consult the manufacturer's operation manual for such instructions.

³The relative standard deviation is the standard deviation of a series of replicate results expressed as a percent of that series' average.

APPENDIX X3-NON-MANDATORY

TAG GAS PURITY ANALYSIS

X3.1 APPLICATION

This analytical method applies to gases such as xenon and krypton and gas mixtures used to tag fuel and control assemblies. The method is sensitive to at least 0.1 mole percent for impurity gases.

X3.2 SUMMARY

The purity of natural, enriched, and isotopically blended gases is measured by ion bombardment, gas mass spectrometry.

X3.3 INTERFERENCES

Interferences are not expected unless residual gas from a previous sample is left in the system.

X3.4 APPARATUS

Mass spectrometers are available commercially that meet or exceed the following requirements:

1. Sample inlet, viscous or molecular leak.
2. Source, electron bombardment ionization.
3. Analyzer and vacuum system capable of producing a resolving power of at least 150. Resolving power is defined as $M/\Delta M$, where ΔM is the width, in atomic mass units, of a peak at mass M at 5% of its height.
4. Detector, Faraday cup or electron multiplier.

X3.5 REAGENTS AND STANDARDS

The purity of all gases used as standards shall be Research Grade¹ or equivalent.

1. Carbon dioxide.
2. Hydrogen.
3. Krypton, natural isotopic composition.

¹Matheson Gas Data Book. The Matheson Co., Inc., East Rutherford, N. J., 4th Ed., 1966.

4. Nitrogen.

5. Xenon, natural isotopic composition.

X3.6 CALIBRATION

Sensitivity factors shall be determined for carbon dioxide, hydrogen, natural krypton, nitrogen, natural xenon, and any other impurity gases of concern. Purified portions of the above gases shall be used to determine the sensitivity factors and to redetermine them periodically. When the sum of the partial pressures deviate from the measured total pressure by more than 15%, the calibration factors should be checked.

X3.7 ANALYSIS²

a. Introduce sample into the ion source of the mass spectrometer through the appropriate inlet system.

b. Scan the mass spectrum from below hydrogen to above xenon.

X3.8 CALCULATION

The calculation procedure is governed by the type of mass spectrometer used.

a. Identify each peak in the spectrum.

b. Calculate the mole percent of each impurity gas using the appropriate sensitivity factors.

c. Normalize to volume or mole percent.

X3.9 PRECISION

Precision depends significantly upon the particular instrument used. Most instruments can provide a precision of at least 1% relative standard deviation.³

²Detailed instructions for operating the mass spectrometer depend upon the particular instrument used. Consult the manufacturer's operation manual for such instructions.

³The relative standard deviation is the standard deviation of a series of replicate results expressed as a percent of that series' average.