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RDT Standard

FUEL AND CONTROL ASSEMBLY TAG GAS

OCTOBER 1972

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United States Atomic Energy Commission

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FUEL AND CONTROL ASSEMBLY TAG GAS

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DIVISION OF REACTOR DEVELOPMENT AND TECHNOLOGYDate October 1972Page 1 of 17

FUEL AND CONTROL ASSEMBLY TAG GAS

Material furnished to this standard shall be produced in accordance with the requirements included herein and with supplemental requirements as specified in the Ordering Data.

1. SCOPE

This standard establishes the requirements for tag gas to be used for locating failed driver 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, neon, or other gases as specified in the Ordering Data.

2. DEFINITIONS

2.1 Purchaser. A person or corporation who purchases services or material in accordance with this standard.

2.2 RFQ. Request for quotation.

2.3 Supplier. A contractor or subcontractor actually performing the services or producing the contracted-for material.

3. ORDERING DATA CHECK LIST

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

1. Tag Gas. Specify tag gas required. (5.1)
2. Isotopic Content. Specify isotopic content of tag gas required. (5.1.1)
3. Naturally Occurring Gas. Specify naturally occurring gas required. (5.2)
4. Purity. If naturally occurring gases other than those listed in 5.1 are ordered, specify purity. (5.1.2)
5. Impurities. If naturally occurring gases other than those listed in 5.2 are ordered, specify impurity levels. (5.1.3)
6. Blending. Specify blending procedure if other than reference process. (6.1)
7. Composition. Specify required composition (6.2)

-
8. Pressure. Specify gas pressure in gas cylinders. (7.3)
 9. Volume. Specify volume of cylinders. (7.4)
 10. Container Valve. Specify type of cylinder valve. (7.5)
 11. Sampling. Specify sampling requirements. (8.2)
 12. Quality Assurance Program Requirements. Specify any additional quality assurance requirements. (9.1)
 13. Responsibility. Specify any additional tests and examinations required. (9.4)
 14. Shipping Containers. Specify cylinder shipping containers. (10.1)

4. MANUFACTURE

4.1 Lotting. A description of the lotting procedures shall be submitted to the purchaser for approval.

5. GAS TAG CONSTITUENTS

5.1 Tag Gas Isotope. This standard lists requirements for tag gases of the nominal isotopes listed in Table 1. Other tag gases may be specified in the Ordering Data.

TABLE 1 - TAG GAS ISOTOPES

<u>Xenon</u>	<u>Krypton</u>	<u>Neon</u>
124	78	20
126		21
128		22

5.1.1 Isotopic Content. The isotopic content of the selected tag gas, or gases, shall conform to the requirements of the Ordering Data. A mass spectrometric analysis of each tag gas ordered shall be furnished to the purchaser.

5.1.2 Purity. The purity of the selected tag gas shall be 99.0 mole percent, minimum.

5.1.3 Impurities. The levels of individual impurities in the selected tag gases shall not exceed the maximum limits shown in Table 2. Other impurities found during analysis shall be reported to the purchaser. Total impurities shall be consistent with the purity limit of 5.1.2.

TABLE 2 - IMPURITY LIMITS FOR TAG GAS ISOTOPES

<u>Impurity</u>	<u>Mole Percent, Max.</u>
Hydrogen	0.5
Nitrogen	0.5
Carbon Dioxide	0.5
Total Hydrocarbons (includes halogenated hydrocarbons)	0.2
Water	0.5

5.2 Naturally Occurring Gas. Naturally occurring gases for blending with selected gases for gas tags shall conform to the following requirements and Table 3. Other gas may be specified in the Ordering Data.

5.2.1 Purity. The purity of the naturally occurring gases for preparation of gas tags shall be 99.95 volume percent or as specified in the Ordering Data.

5.2.2 Impurities. The levels of individual impurities in naturally occurring gas for preparation of gas tags shall be as shown in Table 3 or as specified in the Ordering Data. Total impurities shall be consistent with the purity limit of 5.2.1.

TABLE 3 - IMPURITY LIMITS FOR NATURALLY OCCURRING GASES (MOLE PERCENT)

	<u>Krypton</u>	<u>Neon</u>	<u>Xenon</u>	<u>Other Gases*</u>
Argon	<0.0004	<0.0001	<0.0005	*
Helium	-	<0.0050	-	*
Hydrogen	<0.0005	<0.0005	<0.0005	*
Carbon Dioxide	-	<0.00005	-	*
Oxygen	<0.0004	<0.0001	<0.0005	*
Krypton	-	-	<0.0050	*
Xenon	<0.0025	-	-	*
Nitrogen	<0.0025	<0.0001	<0.0010	*
Hydrocarbons (includes halogenated hydrocarbons)	<0.0025	<0.00005	<0.0010	*
Water	<0.0001	<0.0001	<0.0001	*

*As specified in the Ordering Data

6. TAG GAS BLENDS

6.1 Blending. Tag gases may be prepared by blending gases in accordance with Appendix A, unless otherwise specified in the Ordering Data.

6.2 Composition. The composition of each tag gas blend shall meet requirements specified in the Ordering Data.

6.3 Impurities. The levels of individual impurities in blended tag gas shall not exceed the maximum limits shown in Table 4. Total impurities shall not exceed 1.0 mole percent. Other impurities found during analysis shall be reported to the purchaser.

TABLE 4 - IMPURITY LIMITS FOR BLENDED TAG GAS

<u>Impurity</u>	<u>Mole Percent, Max.</u>
Hydrogen	0.5
Nitrogen	0.5
Carbon Dioxide	0.5
Total Hydrocarbons (includes halogenated hydrocarbons)	0.2
Water	0.5

7. CONTAINERS

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

7.2 Particulate Matter. Tag gas shall be filtered through a 10 micron filter at the time it is loaded into cylinders. The filter shall be capable of removing 98 percent of 10-micron particles and 100 percent of particles larger than 25 microns. Maximum pore diameter and permeability of rigid porous filters shall be determined by ASTM E 128.

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

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

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

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

product. The cylinder cleaning procedures shall have been approved by the purchaser prior to use.

7.7 Leakage. The maximum leak rate of loaded gas cylinders shall be 5×10^{-6} cc/sec (STP). The leak test procedure shall be submitted to the purchaser for approval prior to use.

8. INSPECTION REQUIREMENTS

8.1 Tag gas constituents and blends shall be inspected for conformance with requirements for each gas ordered.

8.2 Sampling. Tag gas constituents and blends shall be sampled as specified in the Ordering Data.

8.3 Archive Samples. Unless otherwise specified, at least 20 ml of tag gas blended to meet the requirements of Section 6 shall be provided for an archive sample. 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.

8.4 Reference Test Method. Tag gas isotopic composition, purity and impurities shall be tested by use of a gas mass spectrometer in accordance with Appendices B and C, respectively. 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.

9. QUALITY ASSURANCE REQUIREMENTS

9.1 Quality Assurance Program Requirements. The tag gas isotopic and gas blending (excludes the supplier of naturally occurring gases of 5.2) supplier shall plan, establish, implement, and maintain a documented quality assurance program that fulfills all requirements of RDT F 2-4 and any additional quality assurance requirements in the Ordering Data.

9.2 Quality Assurance Documentation. The supplier, with the exception of the supplier of naturally occurring gases of 5.2, is responsible for the preparation of all quality assurance documentation required by RDT F 2-4. The documents identified in Table 5 shall be submitted to the purchaser for the applicable purpose at the indicated time. The granting of any process or procedural approvals by the purchaser shall not be construed to relieve the supplier in any way or to any extent from the full responsibility for delivering items conforming with all requirements of this standard.

TABLE 5 - DOCUMENTATION SUBMITTAL REQUIREMENTS FOR CONFORMANCE WITH RDT F 2-4

Item	Description	Reference Paragraph of RDT F 2-4	Submit to Purchaser for		Time of Submittal to Purchaser
			Approval	Information	
1	Quality Verification Plan and Sampling Plans	2.1.1 2.1.2	X		With Response to RFQ
2	Inspection and Test Procedure	3.1	X		30 Days Prior to Use
3	Test Equipment Calibra- tion and Control Plan	6.0	X		With Response to RFQ
4	Control of Nonconforming Items	7.0	X		With Response to RFQ
5	Document Control	3.2	X		With Response to RFQ
6	Identification and Marking	4.0	X		With Response to RFQ
7	Audit Reports	10.0		X	As Generated
8	Corrective Action Reports	8.0	X		As Generated
9	Inspection and Test Results to include Certifications	5.5		X	With Delivery of the Product

9.3 Quality Records. The supplier shall submit a copy of final inspection data and test results to verify results of tests for conformance to this standard to the purchaser at the time of product delivery. Records for blending, showing complete processing, sequence of operation, chemical and isotopic analyses, shall be supplied to the purchaser. Documentation shall be readily available and traceable to individual tag gas cylinders.

9.4 Responsibility. Unless otherwise specified, the supplier shall be responsible for the performance of all tests and inspections required prior to submitting any tag gas to the purchaser for acceptance. The supplier shall determine and utilize his measurement and test capabilities to insure product compliance with requirements. The supplier may use either his own or any laboratory acceptable to the purchaser. Records of all additional tests and examinations specified in the Ordering Data shall be kept complete and available to the purchaser.

9.5 Inspection or Test Level. Acceptability of a lot shall be determined by lot qualification tests on representative samples taken in accordance with 8.2. Lots shall be accepted only when all specified requirements are met.

9.6 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.

9.7 Determination of Compliance and Data Recording for Inspection and Test.

9.7.1 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.

9.7.2 Equipment/Technique Accuracy. Unless otherwise required or approved by the purchaser, dimensional measurements shall be made with a device measuring in units no greater than 10 percent of the specified tolerance range. For limits specified as "min" or "max" use a device measuring in units to one decimal place beyond the character specified.

10. PREPARATION FOR DELIVERY

10.1 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.

10.2 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.

11. REFERENCE DOCUMENTS

The following documents are a part of this standard to the extent specified herein. The issue of a document in effect on the date of the invitation to bid, including any amendments also in effect on that date, shall apply unless otherwise specified. Where the standard appears to conflict with the requirements of a reference document, such conflict shall be brought to the attention of the purchaser for resolution.

11.1 RDT Standards

RDT F 2-4T Quality Verification Program Requirements

11.2 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
- ASTM E 128 Standard Method of Test for Maximum Pore Diameter and Perme-
ability of Rigid Porous Filters for Laboratory Use

APPENDIX A

TAG GAS BLENDING

A.1. SUMMARY

Natural and isotopically enriched gases of the same element are blended to desired isotopic compositions by cryogenic pumping. In this method, two or more gases are combined and then taken through sufficient cycles of liquefaction (or solidification) and evaporation to obtain complete isotopic mixing of gases.

A.2. APPARATUS

A gas blending apparatus can be used having the components listed below (see Figure 1). The apparatus is designed to be operated under vacuum from 10^{-7} Torr 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.

- a. Blending Cylinder (Stainless Steel). Having a volume consistent with the amount of tag gas required per blend.
- b. Capacitance Manometer.¹
- c. Dewar.
- d. Header (Stainless Steel).
- e. Ion Gauge.
- f. Leak Valves.
- g. Oil Diffusion Pump. With liquid nitrogen cold trap.
- h. Sampling Cylinder (Stainless Steel).
- i. Storage Cylinder (Stainless Steel) and Valve. For a 2-liter 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.^{2,3}

1. A MKS Baratron, Type 77 with a Type 774-1000 head, manometer can be used.

2. A 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 304 stainless steel, and is designed for pressures up to 1800 psig. (Whitey Research Tool Co., Emeryville, California).

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

- j. Thermocouple Gauge.
- k. Thermometer.
- l. Tubing (Stainless Steel).
- m. Vacuum Pump. Mechanical.
- n. Valves and Joints. Assorted valves and joints that have been checked by a helium leak test.

A.3. REAGENTS AND STANDARDS

- a. Various gases, natural and enriched, specified according to the gas tags required.
- b. Liquid Nitrogen

A.4. CALIBRATION

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

A.5. BLENDING PROCEDURE

This procedure applies only to the blending of natural and isotopically enriched gases of the same element.⁴ 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 Figure 1.

- a. Calculate the ml needed of each gas 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 B).
- 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 with valves V_1 through V_{10} and leak valves open.
The gas cylinder valves must be closed.

⁴ Modification of this procedure will be required if gases of two or more elements are involved. The primary mechanism for mixing gases with significantly different boiling and melting points must be diffusion with time at room temperature.

A Header
 B Ion Gauge
 C Oil Diffusion Pump
 D Vacuum Pump
 E Blending Cylinder

F Capacitance Manometer
 G Thermometer
 H Thermocouple Gauge
 I Vacuum Pump
 J Joints

K Storage Cylinder
 L Leak Valves
 M Dewar
 N Sampling Cylinder
 V Valves

FIGURE 1 - TAG GAS BLENDING APPARATUS

-
- e. Adjust the capacitance manometer (F) to zero.
 - f. Close valve V_2 to the header (A), valve V_6 , valves V_9 and V_{10} to the sampling cylinder (N), and valves V_7 and V_8 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 V_7 and V_8 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. Warm the storage cylinder until the liquefied or solidified gas has evaporated by removing the Dewar.

The manometer will indicate when evaporation is completed.

- l. Replace the Dewar around the storage cylinder and liquefy or solidify the gas again as in step (j).
- m. Repeat steps (k) and (l) at least five times to insure complete isotopic mixing.

The degree of mixing is determined by analyzing replicate samples of the mixture following steps (n) through (p).

- n. Warm the storage cylinder to evaporate the liquified or solidified gas by removing the Dewar.

The manometer will indicate when evaporation is completed.

- o. Sample the gas using the sampling cylinder by opening valves V_9 and V_{10} .

- p. Close valves V_9 and V_{10} ; remove the sampling cylinder; determine the isotopic ratio by mass spectrometry (Appendix B).

If the required isotopic ratio has been obtained, proceed at step (s); otherwise, proceed at step (q).

- q. Calculate the additional amounts of gases required to adjust the isotopic ratio of the mixture to the required value.
- r. Add those calculated amounts, blend again, and check the isotopic ratio again by following steps (h) through (p).

- s. Liquefy or solidify the gas in the storage cylinder as in step (j); close valves V_7 and V_8 and remove the storage cylinder from the apparatus.

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

A.6. PRECISION

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

APPENDIX B

TAG GAS ISOTOPIC RATIO MEASUREMENT

B.1. APPLICATION

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

B.2. SUMMARY

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

B.3. INTERFERENCES

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

B.4. APPARATUS

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

- a. Sample inlet. Static mode, viscous or molecular leak.
- b. Source. Electron bombardment ionization.
- c. Analyzer. 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 percent of its height.
- d. Detector. Faraday cup, electron multiplier, or Faraday cage and cup dual detector system.

B.5. REAGENTS AND STANDARDS

- a. Krypton. Research grade¹ or equivalent, natural isotopic composition.
- b. Xenon. Research grade¹ or equivalent, natural isotopic composition.

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

B. 6. CALIBRATION

The mass spectrometer shall be calibrated each 8-hr shift in which isotopic ratios are measured. Calibration standards shall be purified 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 also be checked at periodic intervals of not greater than three months.

B.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 nine scans have been recorded.

B.8. CALCULATION

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

B.9. PRECISION

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

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

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

APPENDIX C

TAG GAS PURITY ANALYSIS

C.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.

C.2. SUMMARY

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

C.3. INTERFERENCES

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

C.4. APPARATUS

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

- a. Sample Inlet. Viscous or molecular leak.
- b. Source. Electron bombardment ionization.
- c. Analyzer. 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 percent of its height.
- d. Detector. Faraday cup or electron multiplier.

C.5. REAGENTS AND STANDARDS

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

- a. Carbon Dioxide.
- b. Hydrogen.

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

- c. Krypton. Natural isotopic composition.
- d. Nitrogen.
- e. Water.
- f. Xenon. Natural isotopic composition.

C.6. CALIBRATION

Sensitivity factors shall be determined for carbon dioxide, hydrogen, natural krypton, nitrogen, water, 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. At least one factor shall be checked during each 8-hr shift in which gas purity analyses are made. When a factor deviates significantly from the established value, all other sensitivity factors shall be redetermined.

C.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.

C.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.9. PRECISION

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

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

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