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.

Designation: C1316 – 08 (Reapproved 2017)

Standard Test Method for Nondestructive Assay of Nuclear Material in Scrap and Waste by Passive-Active Neutron Counting Using ^{252}Cf Shuffler[1]

This standard is issued under the fixed designation C1316; 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 nondestructive assay of scrap and waste items for U, Pu, or both, using a ^{252} Cf shuffler. Shuffler measurements have been applied to a variety of matrix materials in containers of up to several 100 L. Corrections are made for the effects of matrix material. Applications of this test method include measurements for safeguards, accountability, TRU, and U waste segregation, disposal, and process control purposes (**1**, **2**, **3**).[2]

1.1.1 This test method uses passive neutron coincidence counting (**4**) to measure the ^{240}Pu-effective mass. It has been used to assay items with total Pu contents between 0.03 g and 1000 g. It could be used to measure other spontaneously fissioning isotopes such as Cm and Cf. It specifically describes the approach used with shift register electronics; however, it can be adapted to other electronics.

1.1.2 This test method uses neutron irradiation with a moveable Cf source and counting of the delayed neutrons from the induced fissions to measure the ^{235}U equivalent fissile mass. It has been used to assay items with ^{235}U contents between 0.1 g and 1000 g. It could be used to assay other fissile and fissionable isotopes.

1.2 This test method requires knowledge of the relative isotopic composition (See Test Method C1030) of the special nuclear material to determine the mass of the different elements from the measurable quantities.

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

1.4 The techniques described in this test method have been applied to materials other than scrap and waste. These other applications are not addressed in this test method.

1.5 *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 precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

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

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1133 Test Method for Nondestructive Assay of Special Nuclear Material in Low-Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry

C1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

C1592 Guide for Nondestructive Assay Measurements

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.10 on Non Destructive Assay.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this test method.

[3] 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.

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C1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 *ANSI Documents:*

ANSI 15.20 Guide to Calibrating Nondestructive Assay Systems[4]

ANSI N15.36 Nondestructive Assay Measurement Control and Assurance[4]

3. Terminology

3.1 *Definitions*—Terms shall be defined in accordance with Terminology C1673.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *active mode, n*—determines total fissile mass in the assayed item through neutron interrogation and counting of the delayed neutrons from induced fissions.

4. Summary of Test Method

4.1 This test method consists of two distinct modes of operation: passive and active. The instrument that performs the active mode measurement is referred to as a shuffler due to the cyclic motion of the ^{252}Cf source. This test method usually relies on passive neutron coincidence counting to determine the Pu content of the item, and active neutron irradiation followed by delayed neutron counting to determine the U content.

4.1.1 *Passive Neutron Coincidence Counting Mode*—The even mass isotopes of Pu fission spontaneously. On average approximately 2.2 prompt neutrons are emitted per fission. The number of coincident fission neutrons detected by the instrument is correlated to the quantity of even mass isotopes of Pu. The total Pu mass is determined from the known isotopic ratios and the measured quantity of even mass isotopes. This test method refers specifically to the shift register coincidence counting electronics (see **(4)** and Test Method C1207).

4.1.2 *Active Neutron (Shuffler) Mode*—Fissions in ^{235}U, ^{239}Pu and other fissile nuclides can be induced by bombarding them with neutrons. Approximately 1 % of the neutrons emitted per fission are delayed in time, being emitted from the fission products over the time range from μs to several minutes after the fission event. Roberts et. al **(5)** were the first to observe delayed neutron emission. We now know that over 270 delayed neutron precursors contribute to the yield although the time behavior can be adequately described for most purposes using a few (six to eight) effective groups each with a characteristic time constant. The idea of detecting delayed neutrons for the analysis of ^{235}U has been attributed to Echo and Turk **(6)**. The active shuffler mode consists of several irradiate-count cycles, or shuffles, of the ^{252}Cf neutron source between the positions illustrated in Fig. 1. ^{252}Cf emits a fission neutron spectrum. During each shuffle, the ^{252}Cf source is moved close to the item for a short irradiation, then moved to a shielded position while the delayed neutrons are counted. The number of delayed neutrons detected is correlated with the quantity of fissile and fissionable material. The total U mass is determined from the known relative isotopic compostion and the measured quantity of ^{235}U equivalent **(7)**.

4.2 Either corrections are made for the effects of neutron absorbers and moderators in the matrix, or a matrix-specific calibration is used. The effect that needs correction is the increase or decrease in the specific neutron signal caused by the matrix.

4.3 Corrections are made for deadtime, neutron background, and the Cf source decay.

4.4 The active mode also induces fissions in Pu if it is present in the assay item. The passive measurement of Pu can be used to correct the active measurement of ^{235}U effective for the presence of Pu.

4.5 Calibrations are generally based on measurements of well documented reference materials **(8)** and may be extended by calculation **(9-11)**. The method includes measurement control tests to verify reliable and stable performance of the instrument.

5. Significance and Use

5.1 This test method is used to determine the U and Pu content of scrap and waste in containers. Active measurement times have typically been 100 to 1000 s. Passive measurement times have typically been 400 s to several hours. The following limits may be further restricted depending upon specific matrix, calibration material, criticality safety, or counting equipment considerations.

5.1.1 The passive measurement has been applied to benign matrices in 208 L drums with Pu content ranging from 30 mg to 1 kg.

5.1.2 The active measurement has been applied to waste drums with ^{235}U content ranging from about 100 mg to 1 kg.

5.2 This test method can be used to demonstrate compliance with the radioactivity levels specified in safeguards, waste, disposal, and environmental regulations (for example, see NRC regulatory guides 5.11, 5.53, DOE Order 5820.2a, and 10CFR61 sections 61.55 and sections 61.56, 40CFR191, and DOE/WIPP-069).

5.3 This test method could be used to detect diversion attempts that use shielding to encapsulate nuclear material.

5.4 The bias of the measurement results is related to the item size and density, the homogeneity and composition of the matrix, and the quantity and distribution of the nuclear material. The precision of the measurement results is related to the quantity of nuclear material and the count time of the measurement.

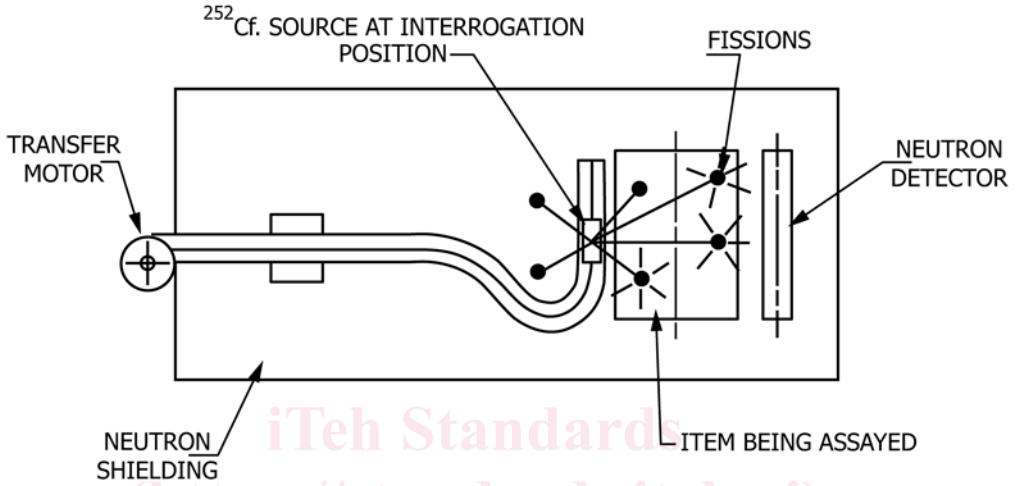
5.4.1 For both the matrix-specific and the matrix-correction approaches, the method assumes the calibration materials match the items to be measured with respect to the homogeneity and composition of the matrix, the neutron moderator and absorber content, and the quantity of nuclear material, to the extent they affect the measurement.

5.4.2 It is recommended that measurements be made on small containers of scrap and waste before they are combined in large containers. Special arrangement may be required to assay small containers to best effect in a large cavity general purpose shuffer.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

^{252}Cf. SHUFFLER MEASUREMENT PRINCIPLE

A ^{252}Cf. NEUTRON SOURCE IS USED TO INDUCE FISSIONS IN THE SAMPLE.



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DELAYED NEUTRONS ARE COUNTED WITH THE SOURCE STORED

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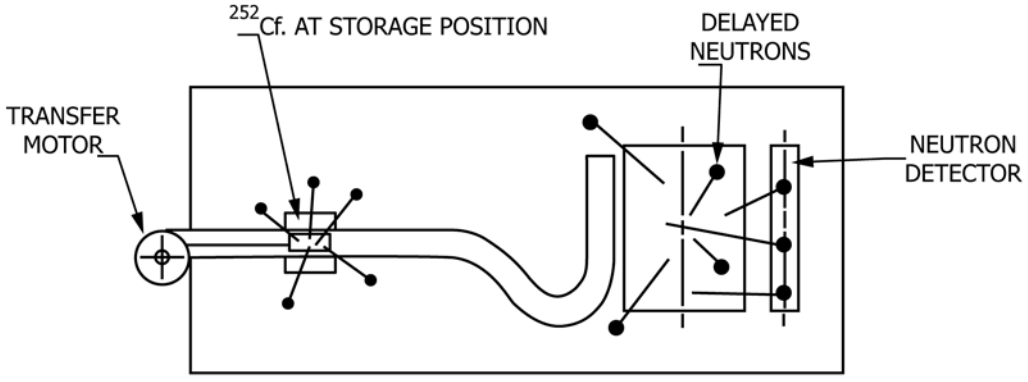

NOTE 1—The shuffler measurement consists of several cycles. Each cycle includes the movement of the ^{252}Cf source from the storage (or home) position to the irradiation position close to the item, irradiation of the item for a period of about 10 s, return of the source to the shield followed by a counting period of about 10 s. In obvious notation this cycle structure may be succinctly described by the four time periods involved (t_{in}, t_{irr}, t_{out}, t_{cnt}). Typically the one-way transit times are less than 1 s.

FIG. 1 Cf Shuffler Measurement Principle

5.4.3 It is recommended that measurements be made on containers with homogeneous contents. In general, heterogeneity in the distribution of nuclear material, neutron moderators, and neutron absorbers has the potential to cause biased results.

5.5 This test method requires that the relative isotopic compositions of the contributing elements are known.

5.6 This test method assumes that the distribution of the contributing isotopes is uniform throughout the container when the matrix affects neutron transport.

5.7 This test method assumes that lump affects are unimportant—that is to say that large quantities of special nuclear material are not concentrated in a small portion of the container.

5.8 For best results from the application of this test method, appropriate packaging of the items is required. Suitable training of the personnel who package the scrap and waste prior to measurement should be provided (for example, see ANSI 15.20, Guide C1009, Guide C1490, and Guide C1068 for training guidance). Sometimes site specific conditions and requirements may have greater bearing.

6. Interferences

6.1 Potential sources of measurement interference include unexpected nuclear material contributing to the active or passive neutron signal, self-shielding by lumps of fissile material, neutron self-multiplication, excessive quantities of absorbers or moderators in the matrix, heterogeneity of the matrix, and the non-uniformity of the nuclear material spatial distribution especially within a moderating matrix. In general, the greatest potential source of bias for active neutron measurement is heterogeneity of the nuclear material within a highly moderating matrix, while the greatest for passive neutron measurement is neutron moderation and absorption (**12**).

6.2 The techniques described in this test method cannot distinguish which isotope is generating the measured response. If more than one nuclide that produces a response is present, the relative abundances and relative specific responses of those nuclides must be known.

6.2.1 *Active Mode*—The unidentified presence of other fissionable nuclides will increase the delayed neutron count rate, causing an overestimation of the ^{235}U content. For example, a calibration based on highly enriched U will cause biased results if the unknowns actually contain low-enriched U due to the potential difference in the fractional contribution arising from the fast fission in ^{238}U (**13**, **14**).

6.2.2 *Passive Mode*—The unidentified presence of other spontaneous fission nuclides, such as Cm and Cf, will increase the coincident neutron rates, causing an overestimation of the Pu content. The active mode measurement of Pu is generally not sensitive to this source of bias (although counting precision may be affected) because the masses of concern are so small and present a comparatively tiny induced fission signal.

6.3 Lumps of nuclear material can exhibit self-shielding or multiplication. This effect is often larger for moderating (hydrogenous) matrices.

6.3.1 *Active Mode (Self-Shielding)*—The nuclear material on the surface of the lump shields the inside of the lump from the interrogating neutrons (**15**, **16**).

6.3.2 *Passive Mode (Multiplication)*—Neutrons originating in the lump induce fissions in the same lump which boosts the specific coincident rate.

6.4 Moderators in the matrix can cause a bias in the measurement results, unless a correction is made or an appropriate matrix specific calibration is used. The magnitude and direction of this bias depend on the quantity of moderator present, the distribution of the fissile material, and the size of the item (**2**, **17**).

6.4.1 Although moderation is the greatest potential source of bias for passive measurements, the passive method is generally less susceptible to the presence of moderator than the active method.

6.4.2 The presence of absorbers in the matrix can cause bias if there is sufficient moderator present. The moderator slows fast neutrons which can then be captured more effectively by the absorbers.

6.4.3 The instrument produces a nonuniform response across the container, the severity varying with the concentration of hydrogen in the matrix. A source at the center of the container can produce either a higher or lower response than the same source located at the surface of the container depending on the item and instrument design.

6.5 Background neutron count rates from cosmic ray-induced spallation can degrade the measurement sensitivity (detection limit) and the measurement precision for small masses (**18**, **19**).

6.6 High-background count rates mask the instrument response to small quantities of special nuclear material for both the active and passive modes (**20-22**).

6.7 High gamma dose rates eminating from the item (>10 mSv h^{-1} of penetrating radiation) may cause pile-up and break-down in the ^{3}He-filled proportional neutron detectors (**23**). Care should be taken to ensure the item is within the acceptable range of the instrument.

6.8 Certain other elements may produce delayed neutrons following (fast) neutron irradiation (**24**).

7. Apparatus

7.1 The apparatus used in this test method can be obtained commercially. Specific applications may require customized designs to cope with (for example) container sizes, container weights, activity levels, integration into the facility (**23**, **25-28**). The following description is one possible design. Fig. 2 is a cutaway illustration of a shuffler to measure 208 L drums. In this design, the ^{252}Cf source storage shield is positioned on top of the measurement chamber. This design weighs approximately 8000 kg, and is 3 m high and 2 m in diameter.

7.2 *Counting Assembly*—see Fig. 3.

7.2.1 The neutron detectors are ^{3}He-filled cylindrical proportional counters embedded in polyethylene, located around the item in a near 4π geometry. The detection efficiency for neutrons of fission energy should be above about 15 %. Larger

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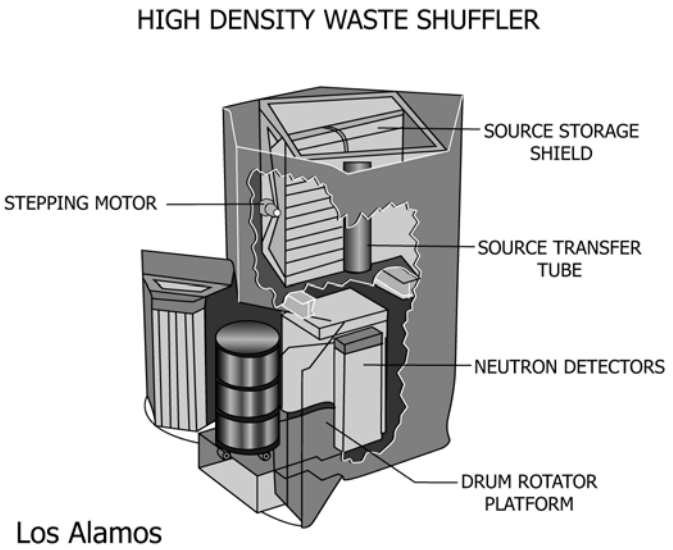
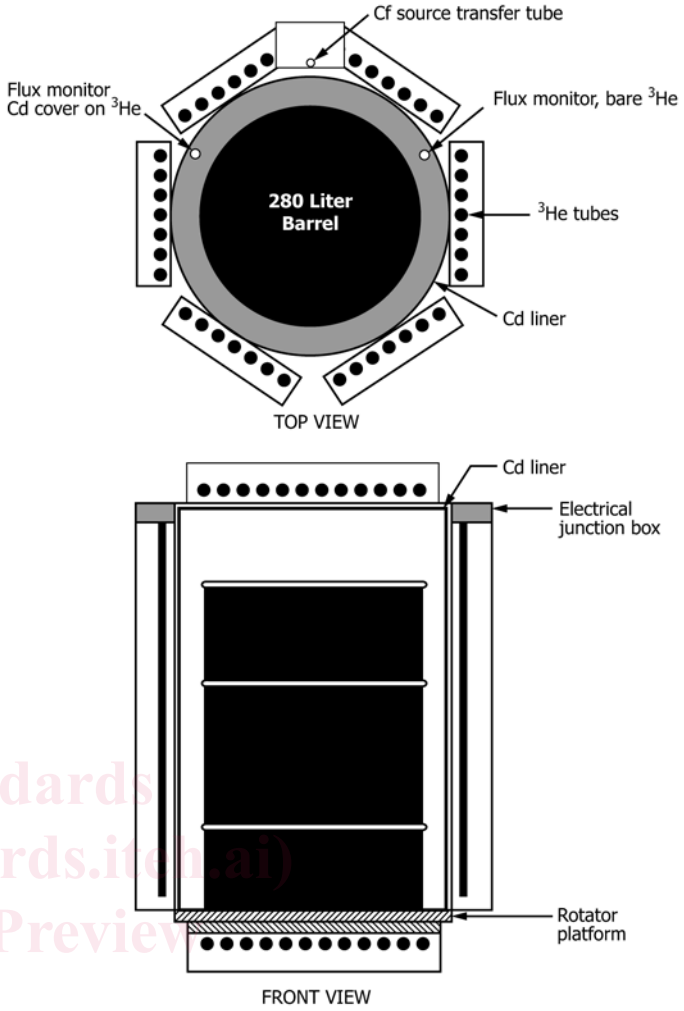


NOTE 1—A sketch of a shuffler designed to assay 208-L drums. The source storage shield is a 2000-kg, 1.2-m cube that resides close to the measurement chamber. In this design it is on top of the measurement chamber. This configuration reduces the footprint of the instrument and may reduce the cosmic ray induced background somewhat. Other configurations are also in common use. The stepping motor drives the Cf source through the source transfer (or guide) tube between the storage position and the irradiation position inside the measurement chamber.

FIG. 2 Shuffler for 208-L Drums of Waste



NOTE 1—The front and top views of the measurement chamber shown in Fig. 2 are shown here in greater detail. The 208 L drum sits on a rotating platform above the bottom detector bank. Six side banks surround the item, with the Cf source transfer tube at the back. The two flux monitors are placed at the rear of the item chamber.

FIG. 3 Shuffler Detector Bank Diagram

detection efficiencies generally provide better precision and lower detection limits for a given count time subject to cycle time, source coupling and other operational parameters. The counter detection efficiency should vary less than 10 % over the item volume with no item present.

7.2.2 The flux monitors are ^{3}He-filled proportional counters mounted on the inner walls of the measurement chamber and not embedded in polyethylene. One flux monitor is covered with Cd approximately 1 mm thick; the other is bare and responds predominantly to thermal neutrons. The Cd shields the so-called fast flux monitor from thermal neutrons; therefore, the two flux monitors can be compared in order to provide information about the neutron energy distribution emerging from the item when the Cf shuffler is brought up. Measured matrix corrections are functions of the fast and thermal flux monitor rates.

7.3 *Shielding*—The quantity of radiation shielding for the ^{252}Cf source is governed by personnel safety requirements although control of the background is also a consideration.

7.3.1 The measurement chamber is typically surrounded by 0.3 to 0.6 m of materials such as polyethylene and borated polyethylene to shield the operator during the ^{252}Cf irradiation.

7.3.2 The shield for the ^{252}Cf storage position is typically about 0.6 m thick (1.2-m cube), depending on the source strength, or the source is placed 1.8 m underground. Composite shields are more effective than polyethylene alone for large ^{252}Cf sources (**29**). The source home position may have a heavy-metal shield to reduce direct gamma dose. The composite shield concept should also takes into account secondary capture gamma-ray generation. If the source store is not directly mated to the measurement chamber, care should be taken in the routing of and shielding to the intervening guide tube so as to manage the time averaged dose rate in the vicinity.

7.4 *Electronics*—High count rate, commercially available nuclear electronics provide standard logic pulses from the ^{3}He-filled proportional counters. These pulses are typically processed by shift register coincidence electronics for the passive measurement, and by gated fast scalers or a multi-channel scaling system for the active measurement. Other correlated neutron counting electronics can be used, with appropriate changes to the data reduction equations.

7.5 *^{252}Cf Source Drive System*—The source is attached to a flexible drive cable that runs inside a guide tube. The source movement is controlled by stepping motors or an alternative method that offers precise timing, positioning, and computer control. During the active measurement, variations in the

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timing of the source transit, irradiation or counting portions of the shuffles cause variations in the measured response. Components should be selected to reduce this potential problem to negligible levels.

7.6 ^{252}Cf sources are commercially available and are usually replaced every few years (typically of the order of two half-lifes) subject to preserving desired active detection limits and precisions. The vendor should understand the safety issues and provide guidance in addressing them.

7.6.1 The source vendor should encapsulate the ^{252}Cf, securely attach the source drive cable, provide shielded shipping casks, and assist with the source installation and disposal.

7.6.2 The source vendor should be requested to provide documentation for the ruggedness and integrity of the source encapsulation and perform swipes to demonstrate that the outside of the source capsule is not contaminated.

7.7 Data acquisition and reduction, control of the source motion, and the diagnostic tests require interfacing the instrument to a computer as illustrated in Fig. 4. The computer and software normally are provided by the instrument vendor.

7.8 *Customized Design Issues:*

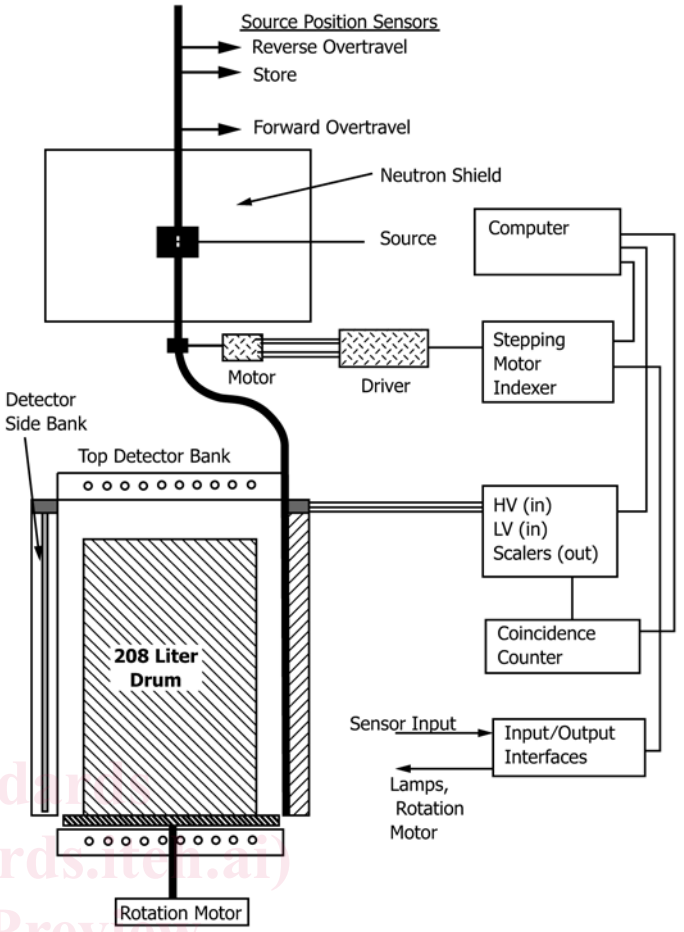
7.8.1 An initial ^{252}Cf source size of 550 µg is generally adequate for measurements of 208 L drums. Performance for a given source strength can be tailored to some considerable extent by adjusting the chamber design—in particular detection efficiency and source coupling play important roles.

7.8.2 It is recommended that the size of the measurement chamber be just slightly larger than the size of the items to be measured. If small items require measurement in a large measurement chamber, the items should generally be centered in the chamber. Coupling of the interrogation source to the item and of the item to the flux monitors may need special consideration and a container specific calibration will generally be needed.

7.8.3 During an active measurement of a large item, the item should be rotated and the Cf source should scan the vertical length of the item. Some designs use continuous rotation and scanning motion (**2**) while others acquire data using a series of discrete angular and source positions (**21**, **27**, **28**). Discrete scans can provide input for optional analysis algorithms (such as might provide coarse spatial corrections) or might be useful where a symmetric pattern of ^{3}He proportional counters can not be used (for example if the instrument is constrained by the interface to a hot cell).

7.8.4 The standard shuffler configuration assumes some hydrogenous and some metallic matrices will be measured. The interrogation-neutron energies are therefore kept high by not using spectrum tailoring materials between the Cf source and the item being measured and by using a steel reflector behind the Cf source (**1**, **2**). This configuration also includes lining the assay chamber with Cd, which prevents neutrons that are thermalized in the polyethylene of the detector banks from entering the measurement chamber. Thermal neutrons generally penetrate less deeply into the matrix and consequently spatial uncertainties will generally be higher if the matrix and special nuclear material distribution are not homogeneous. Thermal neutrons also are less pentrating into aggregates of special nuclear material. The down side of using a Cd liner, however, is that the sensitivity be over an order of magnitude poorer. The prospects and potential benefits of spectrum tailoring are discussed in (**30**). It should also be noted that some containers (for example, those with concrete liner or known to possess a particular waste characteristics) and some chambers (for example, those requiring significant Pb shielding to control the gamma-ray does rate on the ^{3}He proportional counters) introduce neutron transport peculiarities that should be considered as an integral part of the design process (**21**, **26**, **27**).

7.8.4.1 When it is assured that (*a*) lumps are not a significant problem and (*b*) the matrix is a weak moderator, a polyethylene sleeve can be placed around the assay item for the active mode measurement to reduce the energies of the interrogating neutrons, enhancing the fission rate, the



Shuffler Electronic Controls Diagram

NOTE 1—The electrical components and their connections are indicated. The Cf source is moved by the stepping motor and associated driver. Three source sensors are used to verify the source position. The detector signals are amplified and discriminated in junction boxes into which the ^{3}He-filled cylindrical proportional counters are fastened. The logic outputs of the discriminators are fed to scalers and a coincidence counting module. The computer controls the source and rotator and receives the results from the scalers and coincidence counter according to the strict timing sequence in use.

FIG. 4 Shuffler Electronic Controls Diagram

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