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REVIEW OF NDA TECHNOLOGY FOR ENRICHMENT PLANT SAFEGUARDS

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ABSTRACT

A review of nondestructive assay technology for enrichment plant safeguards is presented with emphasis on field experience. Actual and potential applications of this technology in domestic and international safeguards are described. Domestic applications of NDA emphasize verification of uranium enrichment in UF_6 storage cylinders and measurement of uranium in scrap and waste materials. International applications also would involve measurements to detect covert production of uranium enrichments above declared values. The state of development and application of standard techniques, e.g. gamma-ray and passive neutron assay of ^{235}U in UF_6 cylinders, are reviewed critically based on experience at the U. S. gaseous diffusion plants. Random driver measurements and segmented gamma scanning of wastes and trap materials are described. New instruments, such as portable intrinsic Ge detectors and enrichment monitors for gaseous UF_6 are identified.

INTRODUCTION

Enrichment plant NDA safeguards technology can be divided into three broad categories of instruments and methods: (1) the standard gamma-ray and passive neutron measurement methods; (2) the new large active or passive devices like the random driver (RD) and the segmented gamma scanner (SGS); and (3) the new generation of instruments and techniques currently being developed for international safeguards. The first group has been applied extensively to measurements of ^{235}U enrichment in UF_6 feed, product and tails cylinders, as well as in other uranium products. The use of high-resolution Ge detectors is a recent improvement in this group. The RD and the SGS in the second group have been known to the safeguards community for some time, but the sophistication of these devices has increased during the last 5 years. The third group includes a variety of new instruments (and modifications of current ones) in the developmental stage, which were designed for use in international safeguards.

This review appraises the current use and development of NDA for enrichment plants and related uranium handling facilities. We have drawn extensively from the direct experience of one of us whose realistic field results, as well as those of other authors, we use for our critical evaluation. We apologize to those whose work we have unintentionally omitted. Space limitations prevent us from illustrating this discussion with figures (many would be needed) of the new instruments of groups 2 and 3; further, the latter group could only be summarized. However, the figures will be shown, and more detailed descriptions given, during our paper presentation.**

STANDARD GAMMA-RAY AND PASSIVE NEUTRON MEASUREMENTS OF UF_6 IN CYLINDERS

These methods have been used for several years to verify enrichment in UF_6 product, tails and feed cylinders of various sizes. Their

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accuracy varies with the range of enrichments measured and the cylinder size. Product whose ^{235}U assay, is below 5% is considered low enriched uranium (LEU) while highly enriched uranium (HEU) contains more than 20% ^{235}U. Tails have concentrations less than 0.3%, and feed is generally natural uranium (0.711%). Better accuracies are obtained with higher assays.

Gamma-Ray Measurements. This assay technique is based on determining the net (peak minus background) 186 KeV ^{235}U gamma-ray intensity. This intensity is proportional to the assay, after correcting for attenuation by the cylinder wall. The wall thickness is usually measured ultrasonically. The direct proportionality is a consequence of the enrichment principle[1] which requires that no less than an "infinitely thick" layer of UF_6 (10-20 mm) be counted.

During the last five years gamma-ray measurements have been improved due to the increased use of stabilized electronics such as the SAM-II (second generation Stabilized Assay Meter, Eberline Instrument Corp., New Mexico)[2] and of high-resolution Ge detectors with multichannel analyzers (MCA). The SAM-II is portable but is incompatible with high resolution detectors. Because of its much better peak/background ratio, the high-resolution system (HRS) has advantages in solving two well-known problems of low-assay measurements. One problem is the interference of "heels" (UF_6, UO_2F_2, UF_4, UF_5) of different assays remaining from previous cylinder fills. The other is the deposition on cylinder walls of ^{234}Th and its daughter ^{234m}Pa which emits 1.001 MeV gamma rays. The HRS helps to establish if these interferences are heterogeneously distributed, which could bias the measurements.

Table 1 lists typical gamma-ray measurements that have been reported recently. Series G-1 consisted of 40 5A plus 16 8A and 12A cylinders,[3] measured with a 50 mm x 127 mm NaI(Tℓ) detector, with a 76 mm Pb-W collimator, covered by a 0.75 mm Cd foil to filter U x-rays. The cylinder wall thickness (4.8-6.4mm) was measured with a portable ultrasonic Branson Model 104 gauge; all the equipment was mounted on a cart and could be moved to the cylinders for measurement. Two "infinitely thick" U_3O_8 standards (19.5 and 93.51% ^{235}U) were used to determine the calibration constants,[2] and a known UF_6 cylinder bearing an assay of 50.3% was used to verify those constants. Series G-2 to G-6 in Table 1 were measured in the Federal Republic of Germany, under IAEA contract.[10] The detector of the low-resolution system (LRS) used for G-2 had a 50 mm Pb collimator. Two cylinders (43.30% and 93.20% ^{235}U) were selected as standards for calibration. In the HRS used for series G-3 to G-6, the Silena MCA had linear background subtraction capability and the thickness gauge was an ultrasonic Krauterkramer DM-1 with 0.2 mm accuracy. A 30cc Princeton Gamma Tech (PGT) detector was used for G-3 and a 56cc detector was used for the LEU series G-4 to G-6. A PGT detector with a 50 mm Pb collimator was used for series G-7 at British Nuclear Fuel Ltd. The ultrasonic thickness gauge was Baugh and Weedon, Model PA1050. A 1 km coiled coaxial cable was used between the detector amplifier output and the MCA. This aspect might be interesting for international safeguards, to transmit information outside a security fence.

The percent standard deviations $\sigma\%$ of the relative differences in Table 1 were calculated as:

$$\sigma\% = \sqrt{\frac{\sum_i (D_i - \bar{D})^2}{n-1}}; \qquad \bar{D} = \frac{1}{n}\sum_i D_i; \qquad D_i = 100\,(I_{T,i} - I_{M,i})/I_{T,i}; \qquad (1)$$

where I_T,i and I_M,i are the tag assay (determined by mass spectrometry) and the NDA-measured assay, respectively, for cylinder i; D_i is the percent relative difference for that cylinder, and $\bar{D}$ is the average percent relative difference. The σ% values in the table show that the HRS gives slightly more precise results than the LRS; however, this improvement is not substantial and should be weighed against the higher cost, longer counting time, and need for liquid nitrogen in the HRS. As expected, HRS results were improved when a collimator was used or when the uranium was in equilibrium with its daughters ("old" UF_6), so that the ratio of gamma-ray intensities 186 Kev/1001 KeV was proportional to the ^{235}U assay.

Passive Neutron Measurements. This technique is based on measuring neutrons produced in uranium fluorides (UF_4, UF_6) from the nuclear reaction $^{19}F(\alpha,n)^{22}Na$. This reaction is caused by α-particles mainly from the decay of ^{234}U, which is enriched along with the ^{235}U;[2] the ratio $^{235}U/^{234}U$ for LEU varies only 5-10% among different enrichment plants.[10] During the last 5 years these measurements have been made with the SNAP (Shielded Neutron Assay Probe) and the SAM-I or SAM-II; progress in this area is marked by evaluations of various counting geometries, as Table 1 shows for series N-1 to N-11. For the Oak Ridge (N-2, N-6 to N-8) and the Portsmouth (N-4, N-5) series, the single standard cylinder calibration method was used. Series N-2 and N-8 were run simultaneously, on the same cylinders; the poor precision of the "SNAP-on-top" (of cylinder) method was found to be due to marked dependence on cylinder fill height. Many cylinders had significant amounts of "heels" (see above) heterogeneously distributed over their inside walls; this effect reduced the precision of these measurements. Series N-10 and N-11 in Table 1 were measured with the cylinders lifted from the ground in a location isolated from other cylinders. Although precision is good for these conditions, series N-4 to N-6 show that the customary in situ measurement approach (average results at two positions)[2] givescomparable results, without transportation complications. As expected, Table 1 shows that gamma measurements are generally more precise than neutron counting. More importantly, both methods can provide satisfactory verification results and appear to be independent of cylinder size and enrichment.

Despite the acceptable precision of series N-1, passive neutron measurements of HEU assay have been largely unsuccessful due to the variation of the ratio of $^{235}U/^{234}U$ over the enrichment range.[2] Furthermore, UF_6 produced from reactor returns may contain ^{232}U whose specific neutron output is 10^4 times that of ^{234}U. To alleviate this situation, a 4¶ neutron detector was developed[12] which consists of 14 ^3He counters embedded in a polyethylene annulus, designed to measure ^{234}U in UF_6 sample cylinders. Twenty-four samples with ^{234}U concentrations ranging from 0.001 to 1.0% were analyzed. Five 1S cylinders containing enrichments of 80.0 to 97.7% ^{235}U gave a σ% value (Equation 1) of ±0.5% for the ^{234}U measurements, while 19 2S cylinders with 0.3 - 4.0% ^{235}U gave a ± 2.5% standard deviation. Thus passive neutron measurements of ^{235}U assay in HEU cylinders would be feasible if the $^{235}U/^{234}U$ ratio for the corresponding batch were determined before hand by using this ^{234}U method in connection with a gamma measurement of ^{235}U.

Table 1

RECENT STANDARD GAMMA-RAY AND PASSIVE NEUTRON MEASUREMENTS OF ASSAY IN UF_6 CYLINDERS

Series No.	Plant	No. of Cylinders	Cylinder Type[a]	Enrichment Range (%)	Method	Counting Time (min.)	± σ%	Reference
G-1	GAT,Portsmouth,US	56	5A,8A,12A	5.00-93.15	SAM II + Collimated NaI (Tℓ)	1	2.2	3
G-2	NUKEM,Hanau,FRG	12	5A	43.30-93.20	"	1	2.7	10
G-3	"	14	5A	43.30-93.20	Silena MCA + Intrinsic Ge	10	2.7	10
G-4	"	22	30A,30B	0.711-4.096	"	5-10	8.0	10
G-5	"	6	30A,30B	0.711-4.096	Ditto + collimator	10	3.0	10
G-6	"	8	30A,30B	ditto;"old" UF_6	Ditto, no collimator	10	3.2	10
G-7	BNF Ltd., UK	130	30B	1.80-3.25	ND600 MCA + Intrinsic Ge	5	0.8-2.2	11
N-1	NUKEM,Hanau,FRG	8	5A	50.07-93.20	SNAP at 0.25 m	10	11.0	10
N-2	UCC-ND, Oak Ridge	33	48X,48Y	1.70-3.10	SNAP on contact[b]	1	15.5	4
N-3	NUKEM, Hanau, FRG	8	30A,30B	0.711-4.28	"	2	12.4	10
N-4	GAT,Portsmouth	89	48X,48Y	0.711	"	1	7.8-11.9	5
N-5	"	36	30B	1.7-5.10	"	1	6.0	5
N-6	UCC-ND, Oak Ridge	23	48X	1.80-3.45	"	1	6.7	4
N-7	"	20	30B	1.52-3.20	"	1	11.6	4
N-8	"	33	48X,48Y	1.70-3.10	SNAP on top	1	23.9	4
N-9	NUKEM,Hanau, FRG	8	30A,30B	0.711-4.28	SNAP at L/2[c]	2	14.4	10
N-10	"	9	30A,30B	0.711-4.28	SNAP 0.4 m under	2	7.0	10
N-11	"	20	30A,30B	0.711-4.28	SNAP 1 m under	2	8.7	10

(a) Types 5A, 8A and 12A are 5" (127mm), 8" (203mm), 12" (305mm) cylinders with maximum capacities of 25, 116, 209 Kg UF_6 respectively. Types 30A-B, 48X, 48Y are 2 1/2-ton and 14-ton cylinders, respectively.
(b) Average of 4 o'clock and 8 o'clock positions.[2]
(c) SNAP facing center of cylinder side, at 1/2-cylinder-length distance

The presence of ^{232}U should be carefully monitored by gamma spectrometry[12] if the UF_6 sample contains recycled U; systematic errors of up to +20% in the ^{234}U neutron determination were caused by only 29-54 ppb of ^{232}U.[13]

NDA ASSAY MEASUREMENTS OF URANIUM METAL AND COMPOUNDS

Gamma-ray and passive neutron measurements also are used to determine assay in U metal and compounds other than UF_6. As an example, two series of analyses, which were made at the National Lead Corp., Ohio, provide comparative information between NDA equipment. The first one involved U metal ingots and tested a gamma LRS against a HRS. The second series tested the LRS against passive neutron counting with a SNAP in UF_4 measurements. Table II lists these results, as well as those for two U_3O_8 series.

Table II: Recent Typical NDA Measurements of U Metal and Compounds

U Form	Number	Enrichment %	Method	Counting Time (min.)	$\sigma\%$(a)	Ref.
Metal ingots	18	0.95-1.25	SAM II + NaI(Tℓ)	1	13.0	6
"	18	0.95-1.25	MCA + Intrinsic Ge	1.7	3.1	6
UF_4 (5 gal. cont.)	28	0.20-1.0	SAM II + NaI(Tℓ)	1	3.4	7
"	28	0.20-1.0	SNAP	1	5.4	7
U_3O_8 (containers)	28	<5	SAM II + NaI(Tℓ)	1	5.1	4
"	53	2.40-3.47	SAM II + NaI(Tℓ)	1	1.5	14

(a) Equation 1.

For these measurements, the single-standard calibration method was used for the HRS and the SNAP, while two standards were used to determine the calibration constants for the LRS. The LRS was found to be at its limit of applicability in the ingot measurements, due to large differences in decay-daughter background between new and old ingots.

ACTIVE AND PASSIVE MEASUREMENTS OF CONTAMINATED MATERIALS IN LARGE CONTAINERS

Instruments for active and passive measurements of uranium contaminated materials in large containers were developed in the early 1970's and are used extensively. Recently these instruments have been improved by computerized control and data handling. Examples of uses are measurements of skull oxide (U_3O_8), uranium contaminated trap materials (NaF, Al_2O_3) and wastes (tower and calciner ash) in large containers and combustible wastes.

Random Driver (RD). This device is based on coincidence counting of the fission neutrons and gamma rays generated when ^{235}U (or any other fissile nuclide) is bombarded by neutrons from (usually two) random neutron sources. Four large plastic scintillators with corresponding photomultipliers surround the large sample, which rests on a turntable. The scintillators are sensitive to both radiations, and are usually shielded with 12 to 50 mm of Pb from the gamma rays emitted by the neutron sources and the sample. The detectors constitute the 4 doors (adaptable to the sample size) of the RD, and form 4 sides of a pentagon whose 5th side holds the neutron sources. Neutrons are reflected towards the sample by steel or iron

shields in the back of the source holders. Low-density samples thermalize some of the source neutrons, which results in higher fission yields. This effect is corrected by monitoring the thermal neutrons with one or two ^{3}He proportional neutron counters. Depending on the density and total weight of the sample, two operation modes may be used. The "thermal active mode" gives high count rates, and is best used on small, lighter samples. For the "fast active mode" both sources and detectors are covered by Pb-B or boral slabs, which shield them from the sample. These shields absorb thermal neutrons, thus shifting the spectrum of interrogating neutrons and detected fission neutrons to higher energies; this results in better penetration of large, dense samples, but also lowers the count rate. Finally a "passive mode" is used in plutonium measurements.

The RD, patented in 1971, has proven its usefulness in numerous applications and has recently undergone important improvements. The earlier version had been used for verification of uranium measurements (yet unreported)[8,9] in 116 leached residues, 28 pressed briquettes, 43 trap material cans and 47 calciner ash containers: the corresponding σ% values (Equation 1) ranged from 4 to 18%. Three RD varieties are currently being used, tested and/or marketed: (a) a National Nuclear Corp. RD, (b) a LASL unit, and (c) an IRT Corp. instrument. The first of these uses two sources, each emitting $5x10^{5}$ n/s. It accomodates containers of 0.5 to 19L (1 pt to 5 gal.) and the number of coincidences (i.e., 2 detectors out of 4, or 3 of 4) can be dialed. One ^{3}He detector is optional. Systems like this have been used for a number of years at the International Atomic Energy Agency (IAEA), Union Carbide Corp.-Oak Ridge, and other plants.

The Los Alamos Scientific Lab. RD has been described and evaluated in several reports.[15-17] It features two $^{241}AmLi$ random sources and two ^{3}He counters for thermal neutron correction. Detection of fission gamma-rays depends strongly on the size, density and composition of the samples. These gammas are thus discriminated against by requiring the coincidence time window to open only within 6 and 46 nsec from the fission event. This is called time-of-flight (TOF) discrimination of fission gamma rays whose TOF sample/detectors is only 2-3 nsec. This RD was evaluated[15] in measurements of skull oxide (U_3O_8) and unleached solids in containers about 300mm ($\sim$12") high and 158mm ($\sim$6") in diameter; the σ% values were about 3% and 4-5% respectively (Equation 1). The study pointed out the influence of source position and of chamber temperature on the results.

The RD marketed by Instrumentation Research Technology (IRT) Corp., California, uses 4 neutron sources: two $^{241}AmLi$ sources with a total output of $6.25x10^{5}$ n/sec and two $^{241}AmBe$ sources with a total yield of $7.3x10^{5}$ n/sec. The sources are enclosed in W containers to reduce gamma background and can be positioned automatically. The neutron spectrum of the $^{241}AmLi$ sources (0.3 MeV average) is too low in energy to cause ^{238}U fissions, whereas the harder neutrons from the $^{241}AmBe$ sources are used to count ^{238}U fissions. When the $^{241}AmLi$ sources are used for ^{235}U determinations, coincidences of two events (within 45 nsec) is required; when the $^{241}AmBe$ sources are used for ^{238}U, coincidence of three events (within 45 nsec) is required. The sample table rotates at 6 rpm and is equipped with a load cell capable of weighing up to 16 kg. A ^{3}He detector is used for thermalization corrections, and a temperature probe is included in the system. Units of this RD are in use at several locations; two are being evaluated at Union Carbide Corp.-Oak Ridge and at Goodyear Atomic Corp.-Portsmouth, in measurements of high-density uranium compounds and waste materials (traps, ash, etc.). Calibration methods are being tested.

Segmented Gamma Scanner (SGS). The SGS measures ^{235}U content in low density materials using a transmission corrected gamma scan of the 186 KeV gamma of ^{235}U. The procedure used by the SGS consists in electronically segmenting the measured container into horizontal slices; for 19L (5 gal.) containers these slices are 25 mm thick, while for 200L (55 gal.) drums they are 76 mm thick. A thorough description and manual for the SGS has been recently published;[18] the commercial SGS is marketed by Canberra Industries Inc., Connecticut. The container being measured is slowly raised (or lowered) while the 186 KeV gamma ray of ^{235}U of each slice is measured by the SGS. Transmission correction is essential and it is automatically made by simultaneous counting of a ^{169}Y source across the container; interpolation between the transmissions of the two gamma rays of ^{169}Yb (177 KeV and 198 KeV) provide the correction for the 186 KeV gamma ray. The corrected ^{235}U values for each of the slices are combined by the computer to give an average final result. Use of a collimated Ge (Li) detector is essential for the SGS, in order to resolve the ^{169}Yb and ^{235}U gamma rays. Calibration of the SGS, as for the RD, should be made by using standards of a composition somewhat similar to that of the samples. Many SGSs are presently being used around the country. Two systems are currently being calibrated and evaluated at Union Carbide Corp.-Oak Ridge and at Goodyear Atomic Corp.-Portsmouth.

While sensitivity to gamma rays was minimized in the RD, the SGS is based on passive gamma-ray counting. Thus, while the RD is most suitable for high-density samples (because of the higher neutron penetrability) the SGS is preferred for ^{235}U measurements in low-Z matrices, such as trap materials (Al_2O_3, NaF) and burnable waste.

SYNOPSIS OF NEW-GENERATION INSTRUMENTS FOR INTERNATIONAL SAFEGUARDS IN URANIUM ENRICHMENT PLANTS

The requirements of international (IAEA) safeguards are different from those of domestic safeguards, and are reflected in new NDA instrumentation being developed for that purpose. One difference in uranium enrichment plants is the need for the IAEA inspector to verify ^{235}U concentrations in plant areas where his access may be restricted for proprietary reasons. Thus, a number of in-line and area monitors are being developed (or adapted) that will operate unattended in sealed, tamper-indicating enclosures, and that will transmit encrypted information to the inspector. For areas accessible to the IAEA inspector, improvements in field portable and transportable equipment are being reported. Instruments for enrichment safeguards are at different stages of development and testing and are listed in the following summary:

I. In-LINE MONITORS

A. *For Gas-Phase UF_6*

1. Continuous Assay Meter. Cylindrical chamber facing 127 mm (5") NaI(Tℓ) detector. Both ^{235}U assay and UF_6 density are measured; the latter by transmission of gammas from a ^{241}Am source. Under development at LASL.
2. Cyclic Cryogenic Meter. Gaseous UF_6 is successively frozen in a chamber for counting with a CdTe detector, and then sublimed. Assay is measured. Under development at Union Carbide Corp.-Oak Ridge.[20]

3. Pressed NaI Detector Clamp for UF_6 Pipe. Relative ^{235}U concentration is measured. To be developed at Union Carbide Corp.-Oak Ridge.[20]

B. *For Liquid UF_6:*

Sealed Tamper-Indicating Monitor with 5mm (2") NaI(Tℓ) Detector. Sealed container and encryption electronics[21] added to proven, ^{235}U assay monitor.[22] Adapted at Sandia Laboratories Albuquerque; to be tested at Union Carbide Corp.-Oak Ridge.

C. *For Utility Lines:*

1. Continuous NaI(Tℓ) Meter. In direct or by-pass water line. Under test at Sandia Laboratories.[23] Only for alarm purposes.
2. Ion Exchange Resin. In by-pass line; U compound is concentrated in resin and is periodically measured for alarm purposes. Under development at Sandia Laboratories.[23]

II. AREA MONITORS

1. Neutron Background Monitor. Eleven ^{3}He monitors imbedded in polyethylene. Background variations are measured in enrichment process buildings to detect high assays. Developed at LASL.[24]
2. Multihole Collimated Gamma Detector. Unusual relative ^{235}U assay variations may be detected. Developed at LASL.[25]

III. FIELD INSTRUMENTS

A. *Portable:*

1. Brookhaven (National Laboratory) Survey Assay Meter (B-SAM). Second-generation SAM which can use both NaI(Tℓ) and intrinsic Ge detectors. A Texas Instruments SR-51A calculator is included in the computing circuit.
2. Very Light (4.2Kg) Multichannel Analyzer. Both NaI(Tℓ) and intrinsic Ge detector types are usable. Standard MCA features and 1024 channels. Developed at LASL.[26]
3. Active Well Coincidence Counter. Assay is measured by active interrogation with a random (AmLi) neutron source. Developed at LASL.[27] Uses are similar to those of the RD, with which it has been compared.[28]

B. *Transportable:*

1. Precise Monitor for UF_6 Sample Cylinders. Assay is measured in 2S cylinders with 1% precision by Ge(Li) detector and MCA. Testing underway at Union Carbide Corp.-Oak Ridge.[29]

2. Trap Material Enrichment Meter. Alumina containers are measured by counting passive neutrons (see above) to "flag" HEU for alarm purposes. Proof-of-principle tests at LASL.[30]

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