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MAINTAINING CONTINUITY OF KNOWLEDGE ON SAFEGUARDS SAMPLES *

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1. ABSTRACT

The conclusions of the vulnerability test on VOPAN [1] (Verification of Operator's Analysis) as conducted at Safeguards Analytical Laboratory (ASA) at Seibersdorf, Austria in October 1990 and documented in STR-266, indicate that "whenever samples are taken for safeguards purposes extreme care must be taken to ensure that they have not been interfered with during the sample taking, transportation, storage or sample preparation process."

Indeed there exist a number of possibilities to alter the content of a safeguards sample vial from the moment of sampling up to the arrival of the treated (or untreated) sample at SAL. The time lapse between these two events can range from a few days up to months.

The sample history over this period can be subdivided into three main sub-periods: i) the period from when the sampling activities are commenced up to the treatment in the operator's laboratory, ii) during treatment of samples in the operator's laboratory, and finally, iii) the period between that treatment and the arrival of the sample at SAL.

A combined effort between the Agency and the United States Support Program to the Agency (POTAS) has resulted in two active tasks and one proposed task to investigate improving the maintainance of continuity of knowledge on safeguards samples during the entire period of their existence.

This paper describes the use of the Sample Vial Secure Container (SVSC) [2], of the Authenticated Secure Container System (ASCS) [3], and of the Secure Container for Storage and Transportation of samples (SCST) [4] to guarantee that a representative portion of the solution sample will be received at SAL.

2. INTRODUCTION

Wherever safeguards samples are analyzed and by whatever procedure this is accomplished, extreme care must be taken that the samples taken are representative of the bulk amount of material sampled and that they remain representative of that material. The most complex procedure for sampling is encountered in reprocessing plants where most of the sampling activities are performed remotely and the sample treatment is performed in hot cells. Therefore the authors will describe in this paper the entire procedure of sampling a tank solution and the typical sample handling in a reprocessing plant. It is possible to identify in these procedures several sequences which are identical or similar to those used in the sampling of bulk liquids or solids in other types of nuclear facilities. The entire sampling procedure can be subdivided into three main sub-periods which will be considered in detail in the next paragraphs. The relevant safeguards measures undertaken during each of these periods are subsequently discussed.

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DISCLAIMER

3. DESCRIPTION OF THE SAMPLING PROCEDURE

3.1 PREPARATORY ACTIVITIES AND SAMPLING

Before a sample from the solution in a tank is drawn that solution must be homogenized. This can be achieved by a variety of means such as mechanical stirring, recirculation by pump or by operating an air sparging system. The operator considers the solution to be homogeneous if the same mixing time as set during plant commissioning tests is achieved. In modern reprocessing plants, equipped with multiple in-tank density measurement systems, the satisfactory homogenization can be verified by taking a series of density readings. The solution is assumed to be homogeneous if consecutive density readings are matching within prefixed limits. Many factors influence this homogenization operation, but are out of the scope of this paper, though they can be consulted in reference [1].

The next step to achieve in providing representative samples consists of rinsing the sampling line with the tank liquid. Again the optimum procedure should have been determined experimentally during the commissioning stage of the plant.

An additional improvement could be achieved if an in-line density measurement possibility existed, permitting the comparison of the densities as measured in the tank with the density measured in-line in the sampling system. In plants which do not have this equipment this check is performed by comparing the densities as measured in the tank with the density as measured on the sample(s) in the laboratory.

At the moment when the tank is ready to be sampled a certain number of sample vials are uniquely identified by both the operator and the inspector and introduced into the sample blister; a hot cell with heavy lead shielding and operated by means of manipulators. The operator will take the required number of samples for operational purposes followed by a number of samples for accountancy and safeguards purposes. The procedure described above represents the operations for sampling Input solutions. Similar but slightly modified procedures are followed for other types of solutions.

3.2 SAMPLE PACKING, TRANSPORTATION TO AND ARRIVAL IN THE OPERATOR'S LABORATORY

As the samples contain very high radioactive or toxic material they are not normally transported by hand to the laboratory, a pneumatic sample transport system is used. For that purpose the samples are packed into a container (commonly called a "rabbit") before introduction to the pneumatic system. The purpose of the rabbit is to minimize contamination of the pneumatic transport system and damage to the sample vial, and to provide a seal onto the transport piping. These rabbits are very similar in the different plants but do vary slightly in shape and dimensions. The transportation time varies with the length of the transport line between the sample blister and the laboratory but is in the order of only a few seconds. Design Information Verification (DIV) must guarantee that no additional connections exist on this transport line. Reverifications would be performed to detect possible subsequent modifications.

3.3 SAMPLE COLLECTION, DISTRIBUTION AND TREATMENT IN THE OPERATOR'S LABORATORY

Once the sample arrives in the operator's sample collection cell it will either be directly transferred to the hot cell where the treatment will be performed, or it remains in the collection cell awaiting the time for treatment and will only then be transferred to the treatment cell. This last situation could last for many hours.

To avoid possible contamination of the treatment cell it is the practice in some facilities to replace the original rabbit by a new one freshly introduced into the collection cell.

The operation of sample treatment starts with the removal of the sample vial from the (old or new) rabbit. The rather complex treatment of samples at this stage normally involves aliquoting, spiking, weighing, dilution, heating, stirring, subsampling and drying. These activities require the use of pipettes, weighing devices, dilution liquids, ovens, additional sample vials and other glassware. They can be performed manually, or automatically using a computerized robotic system.

Sample treatment up to second aliquoting is normally performed in hot cells while the remainder of the treatment takes place in adjacent glove boxes.

The final step in the treatment consists of drying the safeguards samples. Depending on the amount of solution and the efficiency of the heating device this activity can take between 15 minutes and several hours.

3.4 SAMPLE, VIAL CLOSING, REMOVAL FROM GLOVE BOXES, STORAGE, PACKING AND TRANSPORTATION

Once the aliquots are dried the sample vials are capped and crimped, and possibly loaded into a shielding container and bagged out of the glove box system. The safeguard samples are then stored in a Safeguards safe awaiting the collection of a sufficient number of samples and/or the preparation of the necessary documentation for shipment. The storage time can vary from a couple of days up to several weeks. Some time before the shipment all the sample vials are removed from the safe and packed into the transport containers under Agency seal. The shipment is then organized by the operator so that the samples finally can be transported to the Safeguards Analytical Laboratory (SAL).

4. POSSIBILITIES TO ALTER THE SAMPLE VIAL CONTENT

Assuming that the delivery of a representative sample is available at the needle block in the sampling blister, many possibilities exist to alter the content of the sample vial. The following possibilities can be identified:

4.1 Addition of an analytically interfering material to the sample vial before taking the sample;

4.2 Diversion of the sample vial on its way to the collection cell and altering its content;

4.3 Altering its content upon arrival in the collection cell or hot cell before verification of arrival by the inspector;

4.4 Altering its content in the collection cell or hot cell whilst awaiting sample treatment;

4.5 Altering the sample or aliquots by manipulation of the glassware or reagents, or by changing the aliquot or dilution by manipulations on the weighing or aliquoting systems during or before sample treatment;

4.6 Altering the content of treated samples during drying operations (if not observed by the inspector) and/or during storage awaiting packaging for transportation (if stored in non-safeguards safe); and

4.7 Alterations during transportation, though that is not considered as a very plausible way.

Although some of these possibilities can be covered by inspector surveillance and/or by routine or random provision of the viais and glassware by the inspector and by random analyzing the reagents used during treatment, an enormous amount of inspector workload, often intensive, will be required to cover all the gaps, especiaily those described in 4.2, 4.3, 4.4, 4.6 and for completeness also 4.7.

5. RECENT IMPROVEMENTS

5.1 Maintaining continuity of knowledge on the content of sample vials during the period prior to treatment can be achieved by a recently developed containment system. A Sample Vial Secure Container (SVSC) similar to the operator's transfer rabbit has been deveioped by Sandia National Laboratories (SNL) under the US Program for Technological Assistance to the Agency (POTAS). This system is explained in greater detaii in reference [2].

it provides the advantage that the sampie remains enciosed during the entire period between sample taking and the commencement of treatment and any attempt to tamper with the container will readily be detected by the inspectors. As such any additional inspector workload or surveillance efforts become unnecessary during this period.

The system is identical in dimensions to the operator's rabbit but contrary to the operator's rabbit which only partially covers the sample vial, the SVSC totally covers the sample vial. This reduces greatly the possibility of contaminating the pneumatic transfer system by leaking vials whilst being transported. This system therefore presents an advantage to the operators. In addition the need for inspector's verification of sample arrival in the analytical laboratory is eliminated.

5.2 At the end of the sample treatment the Agency's aliquot must be dried and/or has to be stored over a certain time period in the glove box. This activity is observed by the inspector, either by human or video surveillance. This task is very time consuming for the inspector as well as for the accompanying State Inspector and operator.

For this and for other purposes Sandia National Laboratories have also developed the Authenticated Secure Container System (ASCS) in which the treated sample can be enclosed during drying if the container is adapted for this purpose and/or while stored awaiting closing and bagging out for shipping. Also, other safeguards items such as standard weights for checking the correct functioning of the scaie can be stored in the container.

The application of this additional container wiii also very much reduce the workload of the inspectors and the operator while guaranteeing continuity of knowiedge on the sample.

More details on this container is given in reference [3].

5.3 The final period in the iife of a sampie which involves much workload and could be the most time consuming to aii parties, is the post treatment period. Therefore Sandia National Laboratories have been requested to develop a third container which wiii function in a simiiar manner to the previously mentioned SVSC. This is the Secure Container for Storage and Transportation (SCST) of samples.

Once the sample is placed in such a container it provides full freedom to the inspectors and the operator because it avoids the cumbersome and time consuming observation of sample bagging out, placement into the safe, removal from the safe, packing and storage before transportation.

6. Conclusion

The implementation of the three containment systems described above in addition to the normal human surveillance during sampling and sample treatment, will guarantee the continuity of knowledge on the sample from the moment of sampling up to the arrival in the Agency's laboratory, and also will greatly reduce the Inspectors' and operator's workload.

7. References

[1] S. Deron (SAL), F. Franssen (SGOA2), P. Good (SGCP), A Trial of VOPAN Procedures, IAEA report STR-266, March 1991.

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[4] Report to be prepared.