Special reports:

At the IAEA Safeguards Analytical Laboratory, scientists and technicians determine the concentration and isotopic composition of safeguarded nuclear materials by analysing samples from nuclear facilities.

Safeguards analytical services: Their role in verification

The IAEA carries out independent measurements of nuclear materials subject to safeguards under agreements concluded between it and Member States. The measurements are part of inspection activities at nuclear facilities and help provide for a timely detection of potential diversions of significant quantities of nuclear material from peaceful nuclear activities.

Two types of techniques are employed to perform the measurements:

- Items of nuclear material are verified at the facility during an inspection using a *non-destructive technique* (NDT).
- Samples are collected during an inspection from an item of nuclear material and measured off the site using *destructive analysis* (DA).

Results of the Agency's measurements are reflected in the technical conclusions of its verification activities. Together with other safeguards activities, these measurements confirm the Agency's assurance that so far no significant quantities of materials have been diverted to undeclared applications.

The IAEA Department of Safeguards devotes about 7% of its budget to operate a comprehensive system of *safeguards analytical services* (SAS). It was set up to provide DA and assist in NDT measurements required by its inspection activities. These services have been in operation for more than 10 years.

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Analysis in Agency safeguards

In the Agency's measurement system, NDT and DA techniques are complementary:

- *Non-destructive techniques* are used for the verification of manufactured items and for detecting gross and partial defects on bulk materials. Measurements are performed with transportable equipment installed in the course of the inspection and then compared to standards of nuclear materials with similar physical and chemical properties. These techniques yield an immediate answer. However, suitable calibration standards are not always available to establish the accuracy of the results on the spot.
- *Destructive analysis* still yields the most precise and accurate results. However, delays introduced by transport of samples to analytical laboratories cannot yet ensure a timely detection through DA of abrupt diversions of sensitive materials, such as plutonium products or spent-fuel reprocessing solutions. Thus, DA particularly serves to determine small defects that would remain undetected by NDT and may accumulate over a long period into undeclared significant quantities. The DA technique, therefore, allows verification of the closing of the balance of nuclear material over a specific period and the amount of material unaccounted for (MUF). It also is needed to characterize the working reference materials.

Safeguards analytical services (SAS)

The Agency's safeguards analytical services form a worldwide system. Their tasks are distributed as follows:

The "resin bead" technique is used to take small but representative samples of spent fuel solutions.

A worldwide network of laboratories, expertise, and technical support

by S. Deron and U. Wenzel

• IAEA's *Department of Safeguards* is responsible for the co-ordination of the services, the management of the nuclear materials involved, and the evaluation of the analytical results.

• IAEA's *Department of Research and Isotopes*, with its *Safeguards Analytical Laboratory* (SAL), analyses the samples; characterizes reference materials; and assists in the design and testing of sampling procedures, in the operation of the Agency's Network of Analytical Laboratories (NWAL), and in the evaluation of the performance of the services.

• IAEA's *Network of Analytical Laboratories* is composed of laboratories designated by their respective State in reply to the Agency's request. The network does part of the analyses and assists the SAL in its various functions *(see map on page 26)*.

• The *Member States Support Programmes* provide assistance in the development of new techniques, their testing and implementation, and other specific services, such as supplying special source and reference materials.

(See "Improving technical support to IAEA safeguards", a related article in this edition.)

How and when services are used

Within the safeguards verification system, DA is requested in three sorts of instances:

• ***Direct verification measurements***. These constitute about 80% of the services requested.

• ***Post-calibration measurements***. These are done to analyse selected samples taken during inspections. The samples are taken from items used in the field as references to normalize NDT measurements.

• ***Certification measurements***. These are performed on samples of working reference materials especially prepared for the calibration of NDT or DA measurements.

In all instances, the DA measurement system involves a number of steps. These must be carried out according to well-qualified procedures and monitored carefully, so that results are accurate and timely. Services are arranged at each step of the system by a steering group.*

• *Sampling* is performed by the facility personnel in the presence of the Agency's inspector. The procedure for taking the samples, a very sensitive aspect of the system, is defined in consultation with the steering group, and entered into an agreement with the facility operator and the State.

• *Transport* of the samples to the IAEA Laboratories in Seibersdorf, Austria, is initiated by the Agency inspector. The steering group conceived a model procedure for such transfers. It is based on international regulations and agreements regarding the Agency's Immunities and Privileges, and it greatly speeds up transfers when it can be enforced. The group also arranges the distribution of the samples to the SAL or to the NWAL after they arrive in Vienna.

* IAEA's safeguards analytical services are administered by a steering group composed of staff members of the Divisions of Development and Technical Support and Safeguards Evaluation of the Department of Safeguards, and of the Seibersdorf Laboratories of the Department of Research and Isotopes.

- *Analyses* are performed by the SAL and the NWAL with methods of internationally accepted standards of precision and accuracy *(see accompanying table)*. The results are reported to the Department of Safeguards. There, a statistical evaluation compares them with the results reported by the facility operators. This evaluation is needed to complete the Agency's Inspection Statement, which is addressed to the State concerned, and the *Safeguards Implementation Report*, which is submitted annually to the Board of Governors.
- *Quality assurance* is carried out to provide evidence on the quality of the analytical results produced by the SAL and the NWAL. Other elements of quality control are tracking of the samples and monitoring of the process, responsibilities of the steering group.
- *Disposal of sample residues*, also a task of the group, is achieved by returning them to the countries of origin, while analytical radioactive wastes are sent to facilities under contract to the Agency or a Member State.
- *Training* of new inspectors includes a presentation by the steering group of the requirements and procedures of the system, with a visit to the SAL. An important objective of the group in these sessions is to encourage personal contacts between inspectors, analysts, and statisticians.
- *Development of DA techniques* is a permanent task to maintain the quality of the Agency's measurements at necessary standards.

Assistance of Member States

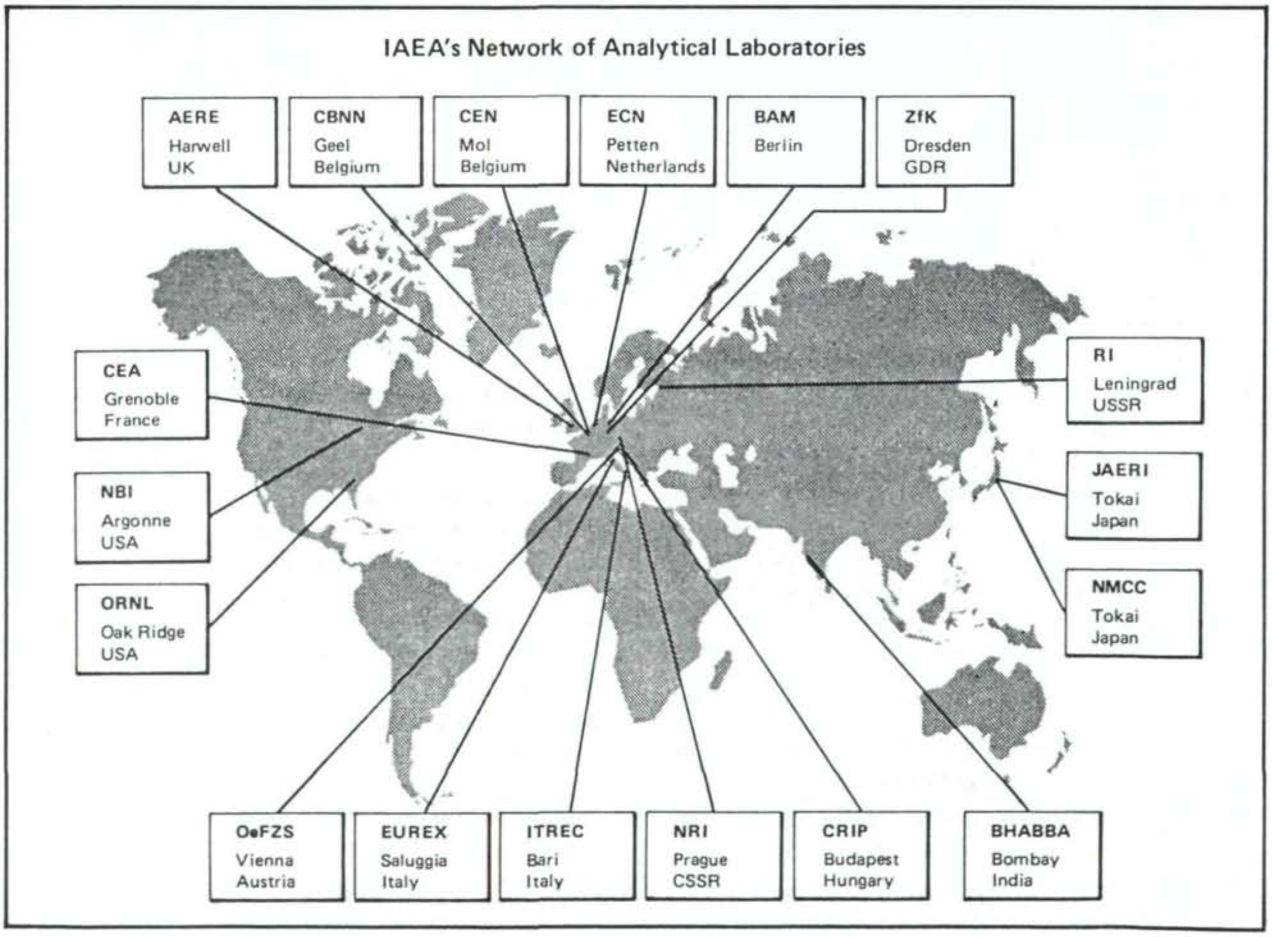
Throughout these efforts, the IAEA relies on the assistance of Member States, usually in the framework of support programmes. The assistance covers five areas:

- Adaptation of advances in analytical technology to safeguards applications (plutonium coulometry, amperometry, and isotope dilution mass spectrometry, for instance)
- Development of stationary analytical measurement stations for field use (for example, K-edge X-ray densitometer, combined XRF/K-edge instrument, quadrupole mass spectrometer, and optical spectrophotometry)
- Procurement of equipment, and special source and reference materials
- Establishment of the analytical quality assurance programme of the SAL and NWAL, with the help of analytical and statistician experts
- Study of the feasibility and design of Agency-operated analytical facilities at large plutonium fuel plants.

Performance of the system

Several indicators of the system's performance may be referenced.

- The *throughput of analyses* tripled over the last



Safeguards Analytical Laboratory (SAL): Isotopic abundance and element assay of sensitive nuclear materials

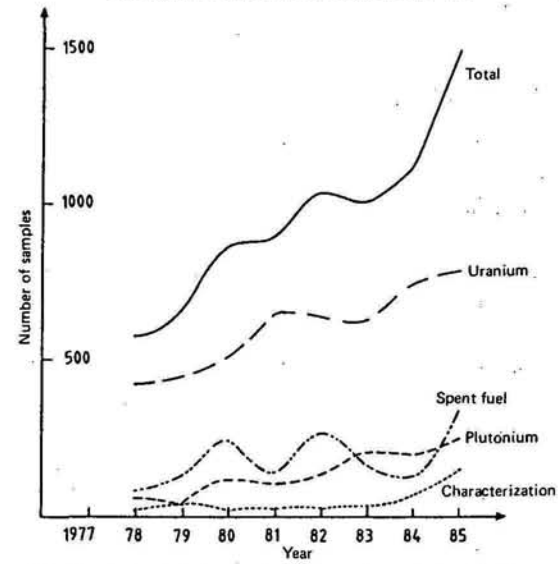
Isotope	Abundance level (weight %)	SAL typical performance		1983 ESARDA target values	
		Random	*Systematic*	*Random*	*Systematic*
U-235	90	0.03	0.03	0.05	0.03
Pu-238	0.3	2.0	2.0	2.0	2.0
	1.5	1.0	1.0	1.0	1.0
Pu-239	50–80	0.10	0.10	0.15	0.10
Pu-240	10–30	0.30	0.30	0.30	0.30
Pu-241	3	0.50	0.50	0.50	1.0
	15	0.30	0.30	0.50	0.30
Pu-242	1–5	0.50	0.50	0.50	0.50
Material type	**Element**				
U-oxide, sintered	U	0.02	0.05	0.05	0.10
Spent fuel input	U	0.50	0.50	0.50	0.50
	Pu	0.50	0.50	0.50	0.50
Pu samples (gram-size)	Pu	0.15	0.20	0.20	0.20
Pu or U/Pu nitrate	U	0.15	0.20	0.20	0.20
PuO_2-powder	Pu	0.15	0.20	0.20	0.20
MOX, FBR	Pu	0.15	0.20	0.30	0.20
	U	0.15	0.20	0.30	0.20
MOX, LWR	Pu	0.20	0.40	1.0	0.50
	U	0.15	0.20	0.30	0.20
Pu samples (milligram-size)	Pu	0.50	1.0	—	—
	U	0.50	0.50	—	—

U = Uranium MOX = Mixed plutonium-uranium oxide
Pu = Plutonium FBR = Fast breeder reactor LWR = Light-water reactor
ESARDA = European Safeguards Analytical Research and Development Association
Statistical note: Expected measurement performance expressed in relative per cent random and systematic error standard deviation.

decade. *(See accompanying chart, which shows the growth in the implementation of DA verifications.)*

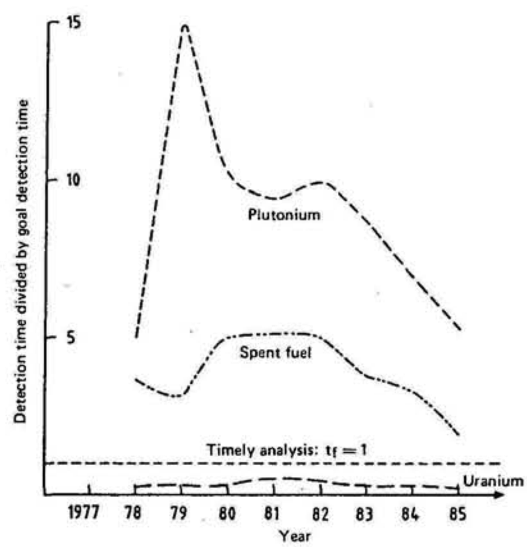
- The *response time* also is improving rapidly. Over the last 5 years, delays were reduced both in transport and analysis of samples, despite a rapidly growing throughput. An exercise, conducted at a spent-fuel reprocessing plant throughout its 1985 campaign, demonstrated that destructive analysis of input and product materials can be reported in a timely manner within 30 days after sampling, if the model procedure for the transfer of the samples is enforced. Off-site destructive analysis could even be applied for the detection of gross and partial defects. *(See accompanying chart.)*
- The *quality of the analyses* is comparable to or better than the analytical standards set by a worldwide group of experts under the auspices of the European Safeguards Analytical Research and Development Association (ESARDA). *(See table.)* The differences between the results from operators and the IAEA generally decreased over the years. Plutonium analyses of spent-fuel solutions are a significant example of the

Throughput of inspection samples, 1977–85

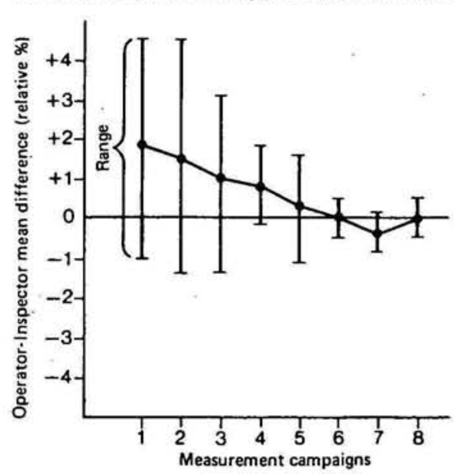


Note: Chart refers to implementation of destructive analysis verifications.

Detection time using destructive analysis



Performance of plutonium analysis in spent-fuel samples



progress made in the accountability of particularly sensitive fissile materials and their verification. *(See chart on page 27.)*

Trends and prospects

The Department of Safeguards expects SAS to double the throughput of inspection samples up to 1990. To accommodate this, the SAS structure is expected to be extended by about 25%. In addition, performance will be improved by employing new techniques (such as the use of quadrupole tracer techniques, frequent consultations with experienced NWAL representatives, and seeking agreement with relevant authorities in Member States and with facility operators on rapid sample transfers).

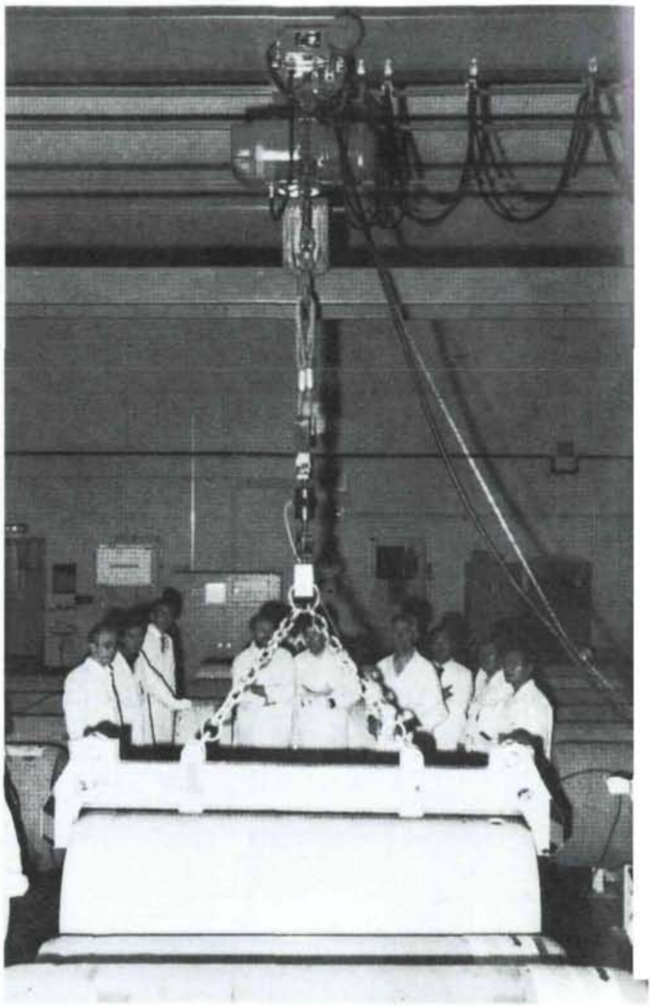
After 1990, large plutonium-processing and uranium-enrichment plants that are now under design or construction will be commissioned. Due to their throughput, even small defects would rapidly accumulate into significant quantities. Their detection within the safeguards goals will require a fast acquisition of analytical results with sufficient accuracy. To this end, the SAS is involved in investigating alternatives to the off-site destructive analysis that is currently used. These alternatives are:

- Analytical measurements carried out at the plant by Agency inspectors with installed instrumentation.
- Operation of analytical laboratories at the facility that are manned by Agency staff.

Such approaches would significantly change the nature of required analytical services. SAS will be called upon for training of inspectors and analysts; investigating and evaluating the capability of new methods and adapting them to specific facility conditions; and providing calibration, software, and maintenance of installed instrumentation. In addition, SAS will operate a quality assurance programme to provide evidence on the quality of the analytical results obtained from on-site measurements.

On-site measurement stations and laboratories would require acceptance by Agency Member States concerned. SAS is, therefore, also strengthening its endeavours to serve as a back-up solution for the alternatives above. As pointed out in this article, the trends in performance of SAS indicate that such an approach appears to be feasible without adversely affecting the quality of the safeguards verification system.

After 10 years of operation, SAS, despite its complexity, is well-mastered. It stays alert, however, to changing needs and is sufficiently flexible to adapt readily to new approaches. The IAEA can count on the tremendous resources and know-how available through the collaboration of more than 20 national or international laboratories, numerous plant laboratories, and the multiple contacts established with national authorities and transporters.



Seen in use, the load-cell-based weighing system checks the weight (approximately three tonnes) of a uranium hexafluoride cylinder.

The inspector's workhorse for gamma-spectrometric measurements is the portable mini-MCA.