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FI9800010

STUK-A149

November 1997

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29 - 10

The conclusions presented in the report are those of the authors and do not necessarily represent the official position of STUK

ISBN 951-712-233-0
ISSN 0781-1705

Oy Edita Ab
Helsinki 1997

Sold by:
STUK - Radiation and Nuclear Safety Authority
P.O. Box 14 FIN-00881 Helsinki, Finland
Tel. +358 9 759881

TOIVONEN H, IKÄHEIMONEN T.K, LEPPÄNEN A, PÖLLÄNEN R, RANTAVAARA A, SAXEN R, LIKONEN J, ZILLIACUS R. Application of Various Laboratory Assay Techniques to the Verification of the Comprehensive Nuclear Test Ban Treaty. Analyses of samples from Kuwait and from AFTAC. Helsinki 1997, 17 pp.

ISBN 951-712-233-0
ISSN 0781-1705

Keywords Radioactive sample, air filter, laboratory assay techniques

ABSTRACT

Various laboratory assay techniques were applied to two particulate air filters from Kuwait and to one filter salted artificially. The monitoring system, run by the PIDC in Arlington, identified ^{137}Cs but no ^{134}Cs in the air samples. Long-term counting using a 100 % HPGe detector in laboratory did not reveal ^{134}Cs either. Upper limit of the activity ratio $^{134}\text{Cs}/^{137}\text{Cs}$ was estimated to be 0.015 which is below the expected average value of the Chernobyl fall-out (0.025). This finding may indicate that the Cs in the sample has other origin than Chernobyl fall-out. Radiochemical methods to purify Cs from the bulk material were investigated. However, because of low yield, the preliminary efforts failed to improve detection limits. The high-resolution gamma-spectrometry of the artificial sample (AFTAC) identified the following man-made radionuclides: $^{95}\text{Zr}/^{95}\text{Nb}$, ^{103}Ru , ^{137}Cs , $^{140}\text{Ba}/^{140}\text{La}$, ^{141}Ce , ^{144}Ce , ^{147}Nd . ^{241}Am was found in alpha spectrometry. The isotope ratios indicate that the sample is produced early in November 1996. The presence of Am shows that the material is most likely irradiated high-burnup uranium or plutonium containing transuranium elements before irradiation. Advantages of mass spectrometry were studied and the preliminary results are very promising. However, a separate programme for sample preparation should be launched.

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1 INTRODUCTION

A global network for radionuclide monitoring will be built for verification of the compliance of the Comprehensive Nuclear Test Ban Treaty (CTBT). During treaty negotiations it was foreseen that radionuclide monitoring stations alone are not sufficient tools for the verification. The monitoring system needs laboratory support, training, quality assurance programmes and in particular, high-level analysis capability to find conclusive evidence about the presence of radionuclides in the samples.

In the CTBT negotiations in Geneva the functions of the radionuclide laboratories were not defined. The Treaty and the Working Papers produced do not give specifications or requirements in detail. However, the laboratories must have state-of-the-art technology available to perform the most advanced analyses for the Technical Secretariat. To develop appropriate assay techniques a test was organised by the Prototype International Data Centre (PIDC).

The PIDC network detected airborne ^{137}Cs in Kuwait on 21-23 September 1996 at concentrations that are beyond normal variability. No ^{134}Cs was found. The laboratory studies should give further data to deduce whether the finding is resuspension of Chernobyl fallout or some other debris. Before shipment the filters were cut in four pieces. The samples were sent to different laboratories for a more detailed analysis; the STUK received two samples, coded as KW0348B and KW0349B.

Third sample, a salted filter, paper was prepared by the US Air Force Technical Applications Center (AFTAC). The shipment form gave approximate activities of the fission products generated by the reactor or other isotopes that were added to the solution to salt the filter (Table 1).

Table 1. Approximate activities given in the shipment form of the AFTAC filter.

Nuclide	Activity (mBq)	Nuclide	Activity (mBq)
^7Be	77	^{241}Am	0.51
^{46}Sc	0.38	^{239}Pu	0.16
^{48}V	0.15	^{238}Pu	4.5×10^{-3}
^{44}Ti	0.14	^{240}Pu	0.038
Mixed fission		^{241}Pu	0.49
products	190×10^3	^{242}Pu	5.6×10^{-6}

2 METHODS

2.1 Gamma spectrometry

The air samples from Kuwait and a blank filter were prepared for gamma spectrometry by compressing them with a hydraulic press to a cylindrical form of 42 mm in diameter and 4-5 mm in height. The samples were counted with HPGe detectors of 99.8% and 40.0% relative efficiencies. Sample self-absorption and true coincidence correction were taken into account in the analyses of the spectra. To reduce minimum detectable concentration (MDC) of ^{134}Cs for obtaining a low minimum detectable activity ratio of $^{134}\text{Cs}/^{137}\text{Cs}$, the two compressed samples were wrapped in thin plastics and placed one on top of the other without a beaker for a counting period of 3.7 d (99.8% HPGe).

It transpired that the salted filter paper by AFTAC is not suitable for volume reduction. In compressing the blank filter some oil or other liquid (about 10 % of weight) escaped out of the material. Therefore, half of the filter was folded by hand into a cylindrical beaker of 74 mm in diameter and the sample was counted with the 99.8 % HPGe detector. The blank filter sample was treated similarly. The salted filter sample was also counted using an n-type HPGe detector of 40 % relative efficiency.

2.2 Autoradiography

Autoradiography is useful for searching, identifying and isolating radioactive particles from different surfaces. Radioactivity is revealed by blackening of photographic emulsion which is placed in close contact with the particles. Blackening is mainly caused by beta particles. An individual radioactive particle may be identified on the film as a circular black spot considerably larger than the beta-active particle itself. Size of the black spot depends on the exposure time, activity of the particle, nuclides in the particle and exposure geometry (Pöllänen et al. 1996).

The compressed bulk materials of the Kuwait filters are not suitable for autoradiography. The powder should be dispersed on a wide area before exposure. This is difficult and prone to contamination and loss of material. Thus, no autoradiograms were prepared. The AFTAC filters were not compressed; they were easy to prepare for autoradiography. One quarter of the salted and blank filter was placed in close contact with BioMax MR X-ray film for 18 hours.

2.3 Caesium chemistry

Detection limit of ^{134}Cs can be improved by radiochemistry. Separation of Cs from the compressed filter matrix improves counting geometry and reduces the amount of disturbing natural nuclides.

Cs carrier (20 mg) was added to the Kuwait sample KW0349B. The filter was wet ashed with nitric and hydrochloric acids. A small undissolved residue was separated from the solution. Cs was coprecipitated by adding 1 g of ammoniummolybdo-phosphate (AMP) to the solution. The AMP precipitation was separated and measured by gamma spectrometer. The remaining solution was completely evaporated and the dry residue was measured, too.

2.4 Alpha and beta spectrometry

Presence of transuranium elements in the AFTAC filter paper was investigated. Half of the filter was treated by wet ashing and acid leaching with strong nitric and hydrochloric acids. Tracers of ^{242}Pu and ^{243}Am were added before ashing. Pu isotopes were separated using anion exchange; this process produced also Am and Cm fractions (Taipale and Tuomainen 1984). The Pu fraction was divided in two equal parts. After Pu electrodeposition on a stainless steel disc, the sample was counted by an alpha spectrometer. For measuring ^{241}Pu , the other half of the Pu fraction was counted by a liquid scintillation counter (Quantulus) with pulse shape analyzer for alpha and beta spectra (Ikäheimonen 1997).

Am and Cm fractions were purified with several precipitations and ion exchanges before electrodeposition and counting with an alpha spectrometer.

2.5 Mass spectrometry

Long-lived radionuclides suit well for the analysis by a mass spectrometer. Half of the AFTAC filter (the same sample that was in gamma spectrometry) was treated in a microwave oven with nitric acid. The samples were diluted and 10 ng/ml of Bi was added to the solutions as an internal standard. The samples were analysed by using VG Plasma Quad 2+ ICP-MS.

Secondary ion mass spectrometry (SIMS) is an analytical technique that is used to characterize the surface and region near surface ($\sim 30\mu\text{m}$) of solid samples. The technique uses a beam of energetic (1 - 30 keV) primary ions to sputter the sample surface, producing ionized secondary particles that are detected using a mass

spectrometer. The primary ions can be O_2^+ , Cs^+ , Xe^+ and Ga^+ . O_2^+ is typically used for the detection of electropositive species, Cs^+ for electronegative species and Ga^+ for improved lateral resolution. Most of the sputtered particles are neutral and only a small fraction is ionized. These sputtered secondary ions are extracted by electric fields and then energy and mass are analyzed with a mass spectrometer.

A mass spectrum consists of secondary ion intensities of the species detected as a function of mass. Three types of SIMS data can be obtained. (1) A mass spectrum is produced by sputtering a sample while the mass spectrometer scans the mass range. (2) Depth profiles are obtained if, as the sputtering continues, one or more masses are monitored sequentially by switching rapidly among masses. (3) SIMS can also be used in a mode of data collection that is analogous to SEM, but instead of obtaining images of the sample by using a focused electron beam, a focused ion beam is used for the bombardment; the image is then formed by secondary ions emitted from the sample.

Characteristic features of the SIMS technique are

- very high detection sensitivity (in the range of ppm to ppb)
- large dynamic range (concentrations)
- capability of detecting all elements
- very good depth and lateral resolution (~ 300nm).

The SIMS technique was tested in a preliminary study to utilize samples from Pu chemistry (see section 2.4). Very promising results were obtained for transuranium elements. However, the results were artifacts produced by Si in the sample and Pb in the plate for electrodeposition. These two elements form short-lived atom clusters that have masses between 235 - 245, producing an unknown large background. Thus, ultra pure reagents should be used in the Pu chemistry and the deposition plate should not contain Pb as impurity. In the present study it was not possible to develop the method in further detail.

3 RESULTS

3.1 Gamma spectrometry

^{134}Cs was not identified in the Kuwait samples (Table II). The minimum detectable activity ratio of $^{134}\text{Cs}/^{137}\text{Cs}$ for the combined samples of KW0348B and KW0349B is 0.015 (counting time 5313 min, reference time 22 September 1996 14:26). Figure 1 shows that this ratio corresponds to fuel burn-up of 6000 MWd tUO₂⁻¹ which is below the average burn-up Chernobyl fuel. Thus, these samples may contain ^{137}Cs that is not explained by the Chernobyl fall-out. The final conclusion could be drawn through purification of Cs utilizing methods of radiochemistry. However, because of low yield, no improvement was achieved in the present study (see section 3.3).

Table II. Activity concentration (or MDC) of different nuclides in Kuwait in September 1996. The confidence level of MDC's is approximately 95 % (estimated from 3 times background standard deviation).

KW0348B: reference time 22 September 1996 02:22, counting times 1012 and 3888 min, total air volume 3156 m³.

KW0349B: reference time 23 September 1996 02:30, counting times 1012 and 3890 min, total air volume 2956 m³.

Nuclide	KW0348B μBq/m ³	Error % (1 σ)	KW0349B μBq/m ³	Error % (1 σ)
^7Be	5800	3	6000	3
^{40}K	930	8	770	7
^{134}Cs	<2.2		<2.2	
^{137}Cs	62	5	83	5

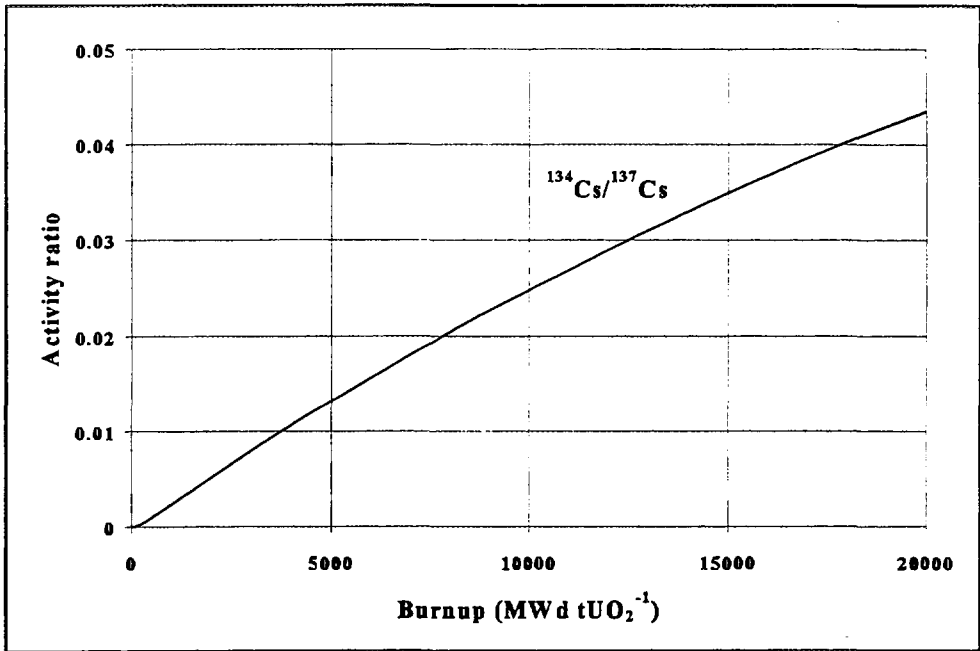


Figure 1. Activity ratio of Cs isotopes of the Chernobyl fuel 10 years and 5 months after the accident. The calculation was performed with ORIGEN2 and OTUS software (Pöllänen et al. 1995). The average burn-up of Chernobyl fuel was 10000 MWd tUO₂⁻¹.

The high-resolution gamma spectrometry revealed the presence of several fission products in the AFTAC sample (Table III). The isotope ratios imply that the material is fresh debris and would thus definitely belong to group 5 in the present PIDC classification.

ORIGEN2 calculations (OTUS) were performed for low-burnup uranium fuel although the irradiated material is most likely old high-burnup uranium or plutonium. Otherwise there would not be Am in measurable quantities (see section 3.4). It is known that Zr and Ce remain always fixed to the carrier matrix (Toivonen et al. 1988); the measurement and the calculations for low-burn-up fuel are consistent.

The ratio ⁹⁵Zr/⁹⁵Nb can be used to estimate the age of the material. The data in Table IV show that the irradiation occurred most likely sometimes between 28 Oct - 5 Nov 1996 which is in good agreement with the information given by AFTAC.

Table III. Activities (or MDA at approximately 95 % confidence level, estimated from 3 times background standard deviation) of different nuclides in the AFTAC filter. Reference time 3 Nov 1996 11:07, counting time 5308 min, start of measurement 23 Dec 1996 13:07. The activities relative to ^{95}Zr , given in the last column, are calculated for low-enriched (1.8 %) uranium fuel at low burn-up (50 MWd tUO₂⁻¹). Zr/Ce isotope ratios are almost similar in fissions from uranium and plutonium. The sample did not contain ^{131}I . Most likely the volatile elements have escaped during the sample manufacture.

Nuclide	Activity or MDA Bq/sample	Error % (1 σ)	Measured activity ratio	Activity ratio in uranium fuel
^{95}Zr	3.77	4	1	1
^{103}Ru	0.75	6	0.20	0.82
^{137}Cs	0.031	22	0.0082	0.0057
^{140}Ba	28.3	6	7.5	4.5
^{141}Ce	6.4	6	1.7	1.6
^{144}Ce	0.70	8	0.19	0.19
^{147}Nd	7.8	14	2.1	1.9
^7Be	<0.3			
^{44}Ti	<0.01			
^{46}Sc	<0.02			
^{48}V	<0.2			
^{241}Am	<0.02			<< 10 ⁻¹⁰

Table IV. Activity ratio of $^{95}\text{Zr}/^{95}\text{Nb}$ for the AFTAC filter.

Reference time	Activity Ratio	Error % (1 σ)
23 Dec 1996 13:07	1.29	3
03 Jan 1997 13:46	1.02	2

3.2 Autoradiography

Blackened areas and small black spots were observed in the autoradiography film of the AFTAC sample (Fig. 2). To ensure, that blackening was caused by radioactive materials, a piece of blank filter was exposed, too. Blackened areas were discovered although they were slightly different and not as black as the blobs of the salted filter. Chemical reactions, perhaps due to presence of organic solvents in the filter, must have occurred between the film and the filter. Similar effects are not observed when glass-fibre filters (such as Whatman GF) are used in autoradiography. The AFTAC filter material is not appropriate for isolation and identification of radioactive particles by autoradiography.

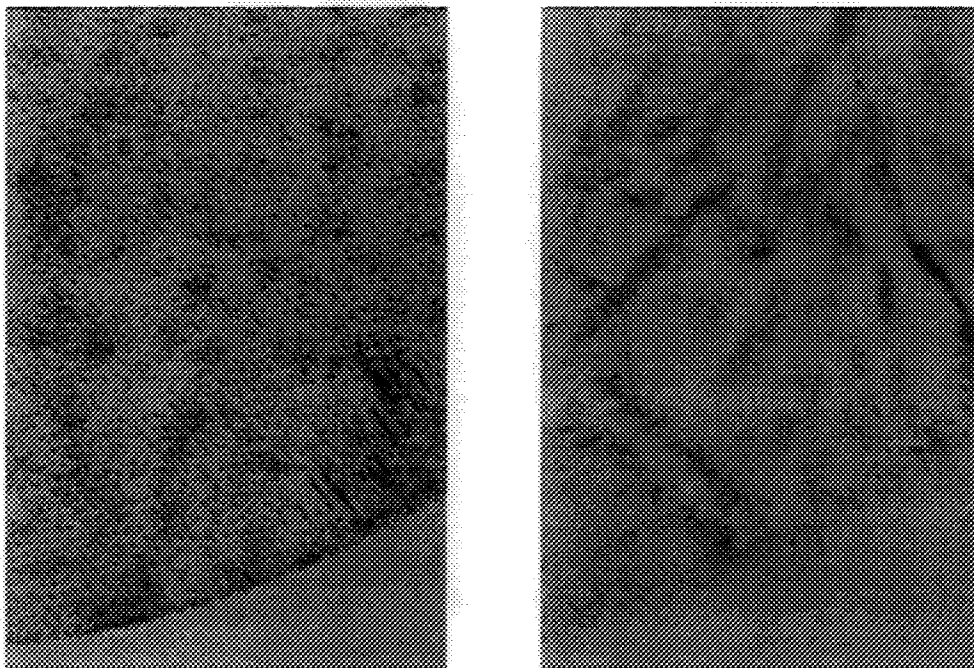


Figure 2. Autoradiograms of the salted filter (left) and the blank filter (right). The exposure time was 18 h.

3.3 Caesium chemistry

The counting efficiency in gamma spectrometry is ideal when Cs forms a thin homogeneous precipitation. In addition, removal of potassium (and ^{40}K) improves detection limit by decreasing the background. The original plan was to separate $^{137+134}\text{Cs}$ with Cs carrier from the sample matrix with AMP precipitation, and, if necessary, to consider an additional separation of Cs from K using Bi iodide and hexachloroplatinate precipitations. Separation of radiocaesium with AMP was tested.

Decomposing the filter with an oxidizing mineral acid HNO_3 resulted in a clear liquid. However, it contained some organic compound which complicated further separations of Cs. Only a small fraction of Cs (13%) was coprecipitated with AMP (Table V). The evaporation residue of the filtrate contained most of the caesium. Thus, the detection limit of ^{134}Cs was not improved as compared with the original result (Table II).

Table V. Activity (Bq) or detection limit (95 % confidence level) of gamma-emitting radionuclides in the original compressed filter KW0349B, in AMP precipitation and in the filtrate.

Sample form	^{7}Be (Bq)	Error % (1σ)	^{40}K (Bq)	Error % (1σ)	^{134}Cs (Bq)	^{137}Cs (Bq)	Error % (1σ)
Compressed filter, ¹	17.7	3	7.5	7	<0.014	0.24	5
AMP ²	-		-		-	0.03	13
Filtrate ³	5.7	4	3.5	7	-	0.21	7

¹⁾ - No ^{40}K background subtraction

²⁾ AMP = ammoniummolybdophosphate precipitation, measured in a geometry with diameter 21 mm

³⁾ Measured as dried evaporation residue in a geometry with diameter 73 mm, height 22 mm and volume 92 cm³.

3.4 Alpha and beta spectrometry

The presence of Pu could not be shown in the AFTAC sample (Table VI). Am, however, was clearly above the brink of detection. An initial counting (5 d) revealed a weak signal. The measurement was repeated using a long counting time (10 d).

Table VI. Activity or detection limits (95 % confidence level) of transuranium elements for the AFTAC filter paper. Half of the original sample was used for the radiochemical analyses (1/4 for alpha spectrometry and 1/4 for beta spectrometry). The presence of Am was clearly shown after a long counting period.

Nuclide	Counting time, min	Activity or detection limit (mBq per total sample)
$^{239+240}\text{Pu}$	8700	< 0.24
^{238}Pu	8700	< 0.32
^{241}Am	15468	0.80 Error 29 % (1 σ)
^{241}Pu	600	< 12

3.5 Mass spectrometry

The concentrations of the elements determined were below the detection limit of the ICP-MS method. The detection limits for different radionuclides in sample solutions and in total samples (100 ml) are given in Table VII. The detection limits using quadrupole ICP-MS with a Mistral® nebulizer for the measured solution are 8 fg/ml-0.1 pg/ml depending on the definition of detection limit.

Table VII. Detection limits (95 % confidence level) in the ICP-MS for KW0348B and for half of the AFTAC filter.

Nuclide	pg/ml	mBq/ml	pg/sample	mBq/sample
^{99}Tc	1	0.63	100	63
$^{129}\text{I}^*$	10	0.01	1000	1
^{237}Np	0.4	0.01	40	1
$^{238}\text{U}^{**}$	0.4	0.00004	40	0.004
^{239}Pu	0.5	1	50	100
^{242}Pu	0.5	0.1	50	10

* iodine was probably lost in the sample treatment

** for the AFTAC filter; the Kuwait filters contain natural uranium 0.2 $\mu\text{g/g}$

4 CONCLUSIONS

The laboratory studies of samples produced by the global air sampling network for the CTBT verification may provide information that is crucial for the interpretation of the origin of the radioactive material. Our present studies have to be considered preliminary because some of the assay techniques were applied for the first time to the analysis of air filters. Also very limited budget was available for the analyses. However, some important conclusions can be made.

- Gamma spectrometry in laboratory, using counting times of several days, can improve detection limits considerably as compared to routine procedures in the CTBT network. Particular emphasis has to be placed on analysis method and on nuclide library used by the software. Coincidence correction and self-absorption correction have to be performed to get reliable activity ratios.
- Essential reduction of the detection limits in the gamma spectrometry is possible by purifying the sample. Separation of Cs from the filter matrix was applied in the present study to a sample from Kuwait. The filter material contained some unknown compound that interfered with the purification processes. The result was a low yield for Cs and consequently, the detection limits were not improved. The separation method works well for glass fibre filters used by STUK (yield 90 %). The filter matrix should not contain organic or inorganic compounds that make the laboratory analyses more complicated and, the chemical procedure itself must be well tested in advance for the filter material used by the sampling station.
- The use of autoradiography was not easy or fruitful in the analyses of the samples. The Kuwait filters were compressed, i.e they consist of bulk material that is difficult to handle. The background AFTAC filter was darkened considerably in a short-term exposure, thus making particle detection and isolation impossible. Glass fibre filters routinely used in the air sampling programme of STUK do not induce extra blackening. The filter material must be carefully chosen not only for particle collection but also for later laboratory analyses.
- The alpha spectrometry of transuranium elements gave very low detection limits for plutonium isotopes (0.1 mBq that is equivalent to 0.2 fg of ^{238}Pu or 0.04 pg of ^{239}Pu). ^{241}Am was detected at a level of 0.8 mBq in alpha spectrometry whereas the detection limit in gamma spectrometry is much higher (20 mBq).

- Preliminary studies utilizing mass spectrometry gave detection limits about 50 pg for the transuranium elements (ICP-MS). The Kuwait filters seem to contain natural uranium (0.2 μg per gram). This has of course a remarkable adverse affect in U trace analyses. The SIMS techniques applied to samples used in alpha spectrometry turned to be extremely promising in transuranium analyses. However, new sample preparation techniques need to be developed.

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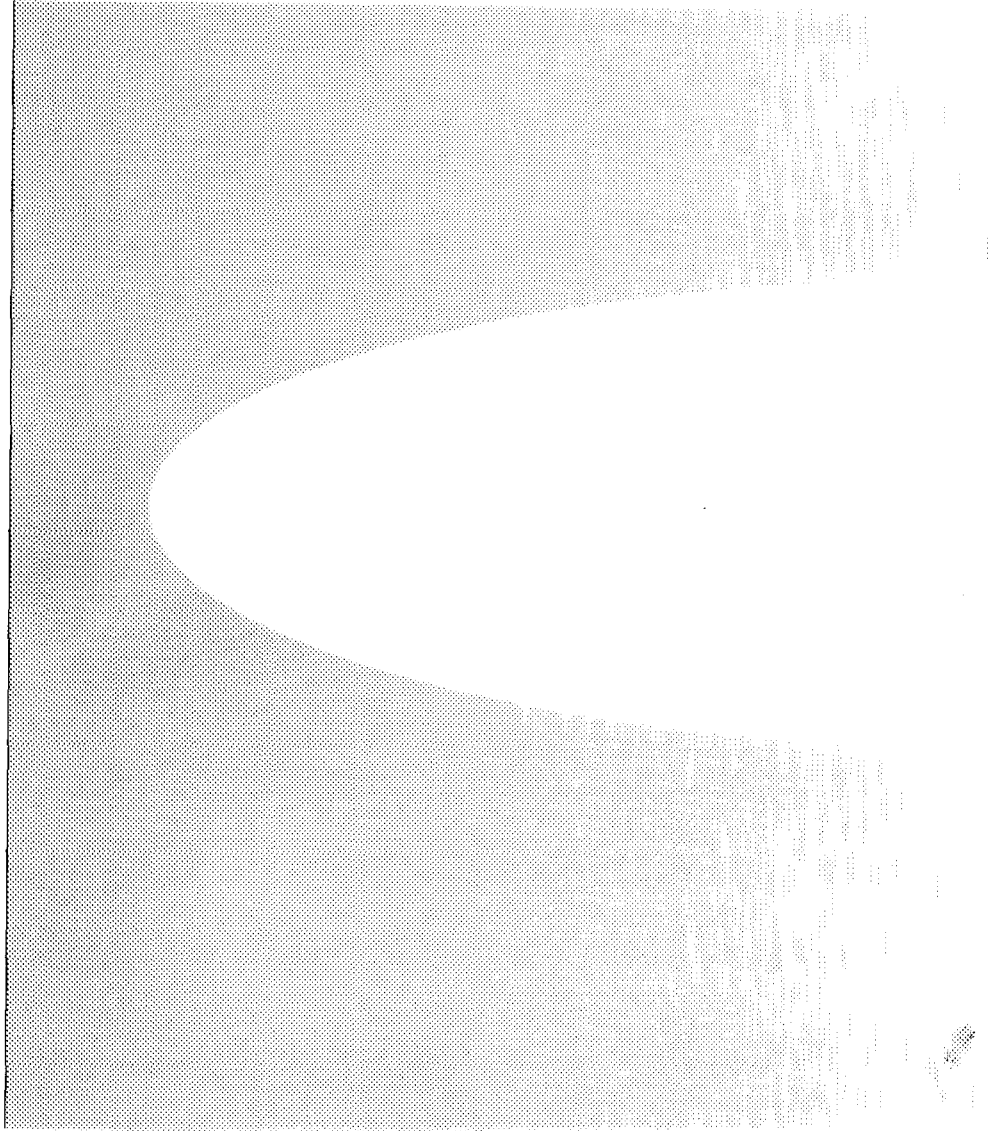
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STUK • A 149 Application of various laboratory assay techniques to the verification of the comprehensive nuclear test ban treaty



ISBN 951-712-233-0
ISSN 0781-1705

Oy Edita Ab, Helsinki 1997