

Appendix 8D. Nuclear forensic analysis

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I. Introduction

Like many international treaties and national laws, the 1968 Treaty on the Non-Proliferation of Nuclear Weapons (Non-Proliferation Treaty, NPT) requires mechanisms to verify compliance.¹ The International Atomic Energy Agency (IAEA) has a specific system of safeguards to verify compliance with the treaty's prohibitions on the manufacture of a nuclear weapon by a non-nuclear weapon state. Individual states also have regulatory and law-enforcement agencies whose tasks are to enforce controls on the transfer of nuclear material and to prevent or prosecute the illicit trafficking of nuclear materials. Should the 1996 Comprehensive Nuclear Test-Ban Treaty (CTBT) come into force, the compliance verification mechanism currently being worked on by the Preparatory Commission for the Comprehensive Nuclear Test-Ban Treaty Organization (CTBTO Preparatory Commission, or CTBTO) would apply.² Other examples include verification of the proposed fissile material cut-off treaty.³

The practical implementation of these verification mechanisms requires the application of appropriate technologies; these verification mechanisms and technologies influence each other. On the one hand, the application of verification arrangements must rely on technology. As technology advances, it provides better means for verification arrangements to pursue their goals. On the other hand, the goals or working conditions of verification mechanisms may change with time, creating a demand for new technologies and even new scientific disciplines. Nuclear forensic analysis (or nuclear forensics) is an example of such a new discipline. Certain nuclear forensic techniques have been used for many years in isolated applications. However, the maturity and popularity of the technologies involved have recently increased to the point where nuclear forensics should be treated as a separate scientific discipline. This appendix explains its importance for the verification and law enforcement applications mentioned above.

Section II of this appendix outlines the definition, major features, applications and some technologies employed by nuclear forensics analysis. Section III gives some examples of the use of nuclear forensic analysis in specific cases. Section IV concludes by discussing the advantages and limitations of nuclear forensics.

II. The definitions, process and technologies of nuclear forensics

The terms 'nuclear forensic analysis' and 'nuclear forensics' were probably first coined in the context of combating nuclear smuggling, a problem that emerged in the

¹ For a summary of the NPT and a list of its 190 parties see annex A in this volume.

² For a summary and list of signatories of the CTBT, which has yet to enter into force, see annex A in this volume.

³ On the negotiation of a fissile material cut-off treaty see chapter 8, section V.

early 1990s.⁴ The investigations and prosecutions of the first such cases called for the development and application of techniques to analyse the nuclear materials involved in order to produce evidence for use in courts of law—hence the term ‘nuclear forensics’.

The *Oxford English Dictionary* defines ‘forensic’ as ‘Pertaining to, connected with, or used in courts of law; suitable or analogous to pleadings in court’.⁵ More broadly, the term is understood in the specialized literature as ‘the application of science to law’.⁶ Although such definitions probably refer mostly to national laws, they could be interpreted as including international laws, regulations and, in particular, treaties.

The IAEA defines *nuclear forensics* as ‘the analysis of intercepted illicit nuclear or radioactive material and any associated material to provide evidence for nuclear attribution’, where *attribution* refers to ‘the process of identifying the source of nuclear or radioactive material used in illegal activities, to determine the point of origin and routes of transit involving such material, and ultimately to contribute to the prosecution of those responsible’.⁷

These IAEA definitions are based on the work of the US Department of Energy’s National Laboratories community involved in combating nuclear smuggling.⁸ The definitions are used in the context of the IAEA’s work on nuclear security, which is separate from the IAEA’s safeguards activities.⁹ However, the analytical techniques used in the combating of illicit trafficking of nuclear materials have much greater potential and, in fact, have been extensively used for many years in other fields. In order to capture all possible applications of the techniques in question, this appendix uses the following broad definitions.¹⁰

Nuclear forensic analysis (nuclear forensics) is the analysis of a sample of nuclear or radioactive material and any associated information to provide evidence for determining the history of the sample material. Nuclear forensic analysis includes characterization, nuclear forensic interpretation and reconstruction.

Characterization is the determination of a sample’s characteristics. It typically involves an elemental analysis of the sample, most often including isotopic analysis of nuclear materials—uranium (U) or plutonium (Pu)—and selected minor constituents—e.g. lead. It also includes physical characterization, for example, measuring the key dimensions of solid samples or determining the particle size and shape distributions of powder samples.

⁴ Moody, K. J., Hutcheon, I. D. and Grant, P. M., *Nuclear Forensic Analysis* (CRC Press: Boca Raton, Fla., 2005), pp. vi–vii.

⁵ *The Oxford English Dictionary*, vol. IV (Oxford University Press: Oxford, 1978), p. F-438.

⁶ Saferstein, R., *Criminalistics: An Introduction to Forensic Science*, 4th edn (Prentice Hall: Englewood Cliffs, N.J., 1990), p. 1, quoted in Moody, Hutcheon and Grant (note 4), p. vi.

⁷ International Atomic Energy Agency (IAEA), *Nuclear Forensics Support: Reference Manual*, IAEA Nuclear Security Series no. 2, Technical Guidance (IAEA: Vienna, 2006), pp. 2–3. Most of the IAEA documents and publications cited here are available from the IAEA’s website, <<http://www.iaea.org/>>.

⁸ Kristo, M. J. et. al., ‘Model action plan for nuclear forensics and nuclear attribution’, UCRL-TR-202675, US Department of Energy, Lawrence Livermore National Laboratory, 5 Mar. 2004, <http://www.osti.gov/energycitations/product.biblio.jsp?osti_id=15009803>.

⁹ IAEA, ‘Nuclear security’, <<http://www-ns.iaea.org/security/>>.

¹⁰ These definitions of ‘nuclear forensic analysis’, ‘characterization’, ‘nuclear forensic interpretation’ and ‘reconstruction’ were developed on the basis of the IAEA definitions (note 7) in close cooperation with Dr James Acton of King’s College London.

Nuclear forensic interpretation is the process of correlating the characteristics of the sample with information on known methods of material production and handling to produce endogenic information about a sample.¹¹

Reconstruction is the process of combining the endogenic information obtained by nuclear forensic interpretation with exogenic information to determine as full a history as possible of the nuclear or radioactive material or an event. This phase is called *attribution* in the narrower contexts of investigations of illicit nuclear materials trafficking and nuclear terrorism events.

Usually there are specific features that interest a researcher in the material's history, such as its origin and producer, point of diversion, age, routes of transit and planned end use. The goal of nuclear forensics—to reconstruct the history of the material or an event—makes it a technique of choice in a number of applications. The specific application defines what is required to be found out from the material in the sample. For example, investigators of a nuclear smuggling case would want to determine the source of the material, at which point it was diverted from legitimate uses, what its possible illegitimate use could be, and so on. Investigators of a nuclear or radiological terrorism incident would look for the material's origin in order to ensure a correctly targeted response. IAEA safeguards inspectors may want to know if the isotopic composition and production date of sample material gathered from a state's nuclear facilities correspond to the state's declared inventory. The CTBTO collects air samples in order to verify the nuclear nature of suspected explosions. Verification procedures of a fissile material cut-off treaty, if and when negotiated, would probably include determination of the age of nuclear materials and might include some kind of environmental sampling to ensure that production of new nuclear materials subject to the treaty does not continue.

Analytical processes and technologies

Following the way in which the IAEA analyses the samples collected in the framework of its safeguards activities both illustrates the process of nuclear forensic analysis and allows the most popular technologies involved to be described.¹²

There are two types of samples: samples of nuclear materials and environmental samples (such as swipes from various surfaces of equipment or buildings, or some volume of air, water, sediments, vegetation, soil or biota). The IAEA operates a nuclear material analysis system consisting of two distinct networks of analytical laboratories (NWAL), a Network of Analytical Laboratories for Nuclear Samples and a Network of Analytical Laboratories for Environmental Samples (with 13 active laboratories in seven member states).¹³ These laboratories, which are nominated by

¹¹ Endogenic information is information derived from the analysis of the sample material and interpretation of the resulting data. In contrast, exogenic information—e.g. archive material or historical databases—is external to the analysis of the material and interpretation of the results of that analysis. IAEA (note 7), p. 31; and Mayer, K., Wallenius, M. and Ray, I., 'Tracing the origin of diverted or stolen nuclear material through nuclear forensic investigations', eds R. Avenhaus et al., *Verifying Treaty Compliance: Limiting Weapons of Mass Destruction and Monitoring Kyoto Protocol Provisions* (Springer: Heidelberg, 2006), p. 402.

¹² On the use of nuclear forensics for IAEA safeguards see also Fedchenko, V., 'Weapons of mass analysis', *Jane's Intelligence Review*, vol. 19, no. 11 (Nov. 2007), pp. 48–51.

¹³ Information on the number of active laboratories was provided by Dr Klaus Mayer. For the formal list, which may also include laboratories that are temporarily uninvolved in the network, see IAEA,

IAEA member states, provide the IAEA with highly specialized measurement capabilities which it could not afford to establish for itself. The IAEA inspectors collect 600–1200 samples every year. In 2006, for example, 756 nuclear samples were collected, 760 were analysed and 1664 verification results were generated. An additional 492 environmental samples were processed as described below.¹⁴

The first step in sample analysis is characterization. Once collected, environmental samples are shipped to the Clean Laboratory for Safeguards of the IAEA's Safeguards Analytical Laboratory (SAL) in Seibersdorf, Austria. These samples typically contain six cotton swipes, four of which are archived for reference purposes and two of which are analysed.¹⁵ The samples are assigned code numbers to conceal their origin before being screened at SAL for the presence of radioactive isotopes by high-resolution gamma spectrometry (HRGS) and for the presence of uranium and plutonium by X-ray fluorescence analysis (XRF).¹⁶ On the basis of the screening results and according to the IAEA inspectors' requirements, the IAEA identifies methods and laboratories for further sample analysis. These subsequent measurements are conducted by either bulk or particle analysis techniques.

The IAEA Department of Safeguards requests 200–400 bulk analyses of environmental samples each year, which are conducted by about seven members of the NWAL (other than SAL). No information concerning origin is attached to any sample, so bias from laboratory personnel is normally ruled out. Traditionally, bulk analyses are conducted by various mass spectrometry methods, most importantly thermal ionization mass spectrometry (TIMS) and inductively coupled plasma mass spectrometry (ICP-MS).¹⁷

'Safeguards Analytical Laboratory: IAEA Network of Analytical Laboratories for Safeguards', <<http://www.iaea.org/OurWork/ST/NA/NAAL/sal/salCLnwal.php>>.

¹⁴ Schmitzer, C. et al., 'The Safeguards Analytical Laboratory and the future of nuclear materials analysis for the IAEA', European Safeguards Research and Development Association (ESARDA), *29th Annual Meeting: Symposium on Safeguards and Nuclear Material Management* (Office for Official Publications of the European Communities: Luxembourg, 2007), p. 1.

¹⁵ Bevaart, L., Donohue, D. and Fuhr, W., 'Future requirements for the analysis of environmental samples and the evaluation of the results', ESARDA (note 14), p. 2.

¹⁶ HRGS is a technique making use of the fact that most radioactive isotopes emit characteristic gamma rays, thus determining the energy and count rate of gamma rays emitted by the material may provide information on its isotopic contents. HRGS is capable of detecting as little as 5 micrograms of uranium, down to tens of nanograms of plutonium, and in some cases, of estimating uranium enrichment. A microgram (μg) is 1×10^{-6} grams; a nanogram (ng) is 1×10^{-9} g.

XRF exploits the fact that, if X-rays bombard a material, they can expel electrons from the inner orbitals of the atoms. Electrons at higher orbitals will 'fall' into the vacant places in lower orbitals and emit X-rays in the process. The energy of such X-rays is characteristic to the element emitting it, and the count rate is proportional to the amount of the element present. The XRF system installed at SAL is reported to have a detection limit for uranium on a swipe of 35 ng per square centimetre. Bevaart, Donohue and Fuhr (note 15), pp. 2–3; and Piksaikin, V. M., Pshakin, G. M. and Roshchenko, V. A., 'Review of methods and instruments for determining undeclared nuclear materials and activities', *Science and Global Security*, vol. 14, no. 1 (Jan.–Apr. 2006), pp. 49–72.

¹⁷ Mass spectrometric techniques utilize the difference in masses of nuclides. The atoms contained in a sample are transformed into ions, separated by an electromagnetic field and counted according to their mass and charge. Mass spectrometric methods differ in the way the sample material is ionized (e.g. thermal ionization or ionization by plasma) and in the type of mass analyser. For TIMS, the entire swipe is ashed and dissolved in acid, then uranium and plutonium are chemically separated, placed on a metallic filament and ionized by heating. For ICP-MS the sample is also dissolved and chemically purified, then it is nebulized in a spray chamber and aspirated into an argon plasma. Moody, Hutcheon and Grant (note 4), pp. 350–54.

TIMS analysis conducted in SAL reportedly allows for detection limits of 70 femtograms of plutonium-239 and 1 nanogram of natural uranium.¹⁸ Much better TIMS uranium detection limits, also down to the femtogram range, have been reported elsewhere.¹⁹ In 2007 SAL was in the process of acquiring ICP-MS equipment, which would allow for less stringent chemical separation procedures and thus decrease the time needed for analysis. ICP-MS detection limits are generally comparable to or better than those of TIMS.²⁰

Some samples contain just a few useful particles and so cannot be analysed in bulk. The IAEA normally issues 500–800 requests for analysis of particles in environmental samples each year. Of these, about 40 per cent are analysed using secondary ion mass spectrometry (SIMS) and about 60 per cent using fission track thermal ionization mass spectrometry (FT-TIMS).²¹ SIMS is usually used for uranium isotope measurements, while FT-TIMS is used for both uranium and plutonium measurements. FT-TIMS has a lower detection limit, in a pico- and femtogram range, compared with a nano- and picogram range for SIMS.²² For this reason, in 2007 the IAEA explored the possibilities of installing more FT-TIMS or equivalent capacity, such as ultra-high sensitivity SIMS (UHS-SIMS).²³ Much can be learned by examining the particle visually using optical or electron microscopy, and thus collecting information on its morphology.²⁴

Once the characterization of the sample is finished, the IAEA interprets the information produced. For instance, the information on the isotopic composition of plutonium in a collected sample could be used to calculate the date when it was separated from the spent fuel or otherwise chemically purified. As another example, the details of a uranium particle's morphology can yield information on the temperature at which it was formed and thus indicate the production process. All information analysed at this second step is endogenic. Sometimes certain parameters obtained during the characterization and interpretation processes can be combined into a 'nuclear fingerprint'—the combination characteristic for the mode of production of the material.²⁵

During the third step—reconstruction—the endogenic information is fed into a broader analysis, which employs all relevant data that is available. In order to reconstruct the history of the material or the facility under consideration, the IAEA Department of Safeguards can use satellite imagery analysis, open source information ana-

¹⁸ Bevaart, Donohue and Fuhr (note 15), p. 4. A femtogram (fg) is 1×10^{-15} g.

¹⁹ Pikaikin, Pshakin and Roshchenko (note 16), p. 71.

²⁰ Bevaart, Donohue and Fuhr (note 15), p. 4; and Moody, Hutcheon and Grant (note 4), p. 357.

²¹ In SIMS the individual particles in the sample are found and bombarded by a high-energy, finely focused primary ion beam, usually O_2^+ , Cs^+ , or O^- . The beam penetrates a few nanometers into the particle and causes secondary ions to be 'sputtered' out, making them available for separation according to their mass and analysis.

In FT-TIMS the particles are removed from the swipe, attached to a fission-track plastic (Lexan), and irradiated in a nuclear reactor with thermal neutrons. Fissile isotopes fission during the irradiation and leave tracks in the plastic, which permits the particles containing them to be located under an optical microscope. These particles are then picked out, loaded onto a heating filament and then ionized and analysed by a TIMS instrument. Bevaart, Donohue and Fuhr (note 15), p. 4; and Moody, Hutcheon and Grant (note 4), pp. 354–56.

²² Pikaikin, Pshakin and Roshchenko (note 16), pp. 71–72. A picogram (pg) is 1×10^{-12} g.

²³ Bevaart, Donohue and Fuhr (note 15), p. 5.

²⁴ US Congress, Office of Technology Assessment, *Environmental Monitoring for Nuclear Safeguards*, OTA-BP-ISS-168 (Government Printing Office: Washington, DC, Sep. 1995), p. 26.

²⁵ Mayer, K. et al., 'Recent advances in nuclear forensic science', ESARDA (note 14), pp. 1–2.

lysis, data on the facility design and associated nuclear trade, as well as other information provided by the member states. At this stage the nuclear fingerprint can be compared against the IAEA's nuclear fingerprint database and other sets of nuclear fingerprints collected elsewhere. The process may be iterative; that is, the results of the reconstruction process may call for additional measurements of the collected samples or for the collection of new samples.

III. Examples of applications of nuclear forensic analysis

The first applications of nuclear forensic analysis took place during and in the aftermath of World War II as the United States and its allies investigated first the German and then the Soviet nuclear programmes. Nuclear forensic techniques were expanded and refined in the subsequent decades as more states developed nuclear capabilities. The entry into force of the NPT created a much greater need for nuclear forensic techniques as the IAEA implemented its comprehensive safeguards agreements (CSAs), most recently in the cases of Iran and North Korea. Demand for nuclear forensic analysis began to grow from the early 1990s partly due to the emergence of nuclear material smuggling and the need to investigate and prosecute such cases.

Examples of the use of nuclear forensics in these varying cases are given below. However, although nuclear smuggling is by far the best known application of nuclear forensics, in both the scientific and popular literature,²⁶ the examples below focus on less publicized applications.

Analysis of airborne debris to verify nuclear reactor operation²⁷

The demand for what have since been named nuclear forensic analysis techniques was probably first recognized and formulated by the head of the British–US Manhattan Project, Brigadier General Leslie R. Groves, in 1943. Finding that the information produced by the US intelligence community was not sufficient to provide an adequate picture of the German nuclear weapon programme, Groves introduced the innovative concept of radiological intelligence.²⁸ He assigned Luis W. Alvarez, a future Nobel laureate for physics, to develop a method for detecting operating nuclear reactors on German territory.²⁹

Alvarez's method involved detecting the radioactive gases that reactors emit during their normal operation, in particular the radioactive isotope xenon-133 (Xe-133). It is generated at a high rate during fission of uranium-235, uranium-238 and plutonium-239, which means that it is produced in significant quantities by any reactor. Xenon is a noble gas, so it escapes a reactor in detectable quantities instead of chemically reacting with other elements. The half-life of Xe-133 is 5.243 days, which means that it does not appear in the atmosphere naturally. It is also relatively easy to separate

²⁶ Moody, Hutcheon and Grant (note 4), pp. 401–20; and e.g. Clancy, T., *The Sum of All Fears* (Putnam: New York, 1991).

²⁷ The term debris can be applied to all sizes of particles resulting from a nuclear reaction, including gases.

²⁸ Ziegler, C. A. and Jacobson, D., *Spying without Spies: Origins of America's Secret Nuclear Surveillance System* (Praeger Publishers: Westport, Conn., 1995), pp. 3–9. Groves is also famous for leading the Alsos mission. See Hart, J. D., 'The ALSOS mission, 1943–1945: a secret U.S. scientific intelligence unit', *International Journal of Intelligence*, vol. 18, no. 3 (Oct. 2005).

²⁹ Alvarez, L. W., *Alvarez: Adventures of a Physicist* (Basic Books: New York, 1989), pp. 119–22.

from the nitrogen and oxygen in the air. All these qualities make Xe-133 perfect for detection as a 'signature' of an operating nuclear facility.³⁰

A xenon-detection system consisting of air-sampling equipment and a ground-based laboratory for sample analysis was developed by the summer of 1944. A few Douglas A-26 Invader aircraft collected air samples over Germany in the autumn of 1944, but no Xe-133 was found.³¹ This confirmed that Germany did not have any nuclear reactors in operation and initiated a new form of intelligence gathering.

Analysis of airborne debris to verify nuclear weapon tests

Similar equipment designed to filter out airborne radioactive particles was later mounted on Boeing WB-29 aircraft and used to detect the first Soviet nuclear weapon tests. The first such test, designated RDS-1 in the Soviet Union, was conducted on 29 August 1949 at the Semipalatinsk test site, which is now within the territory of Kazakhstan. By the spring of 1949 the US radiological intelligence agency, AFOAT-1 (Air Force, deputy chief of staff for Operations, Atomic Energy Office, Section 1), had established routine airborne dust collection flights along two routes—from Fairbanks, Alaska, to the North Pole and from Fairbanks to Yokota, Japan—in order to analyse air masses travelling eastward from Soviet territory.³²

On 3 September 1949 the WB-29 aircraft returning to Fairbanks from Japan collected the first traces of radioactive particles, which were presumably carried to the Pacific from the Semipalatinsk nuclear test site in an air mass. During the following days an all-out effort was made to collect as many samples as possible. Since the air mass containing particles had moved on over the territory of North America to the northern regions of the Atlantic, the USA also enlisted the help of the British atomic energy authorities and the British Royal Air Force.³³

Analysis of the particles collected revealed the presence of fission products, mostly isotopes of barium, cerium, iodine and molybdenum. The radioactive isotopes all had the same age, indicating that their probable origin was a bomb explosion rather than a nuclear reactor accident. Also, the fission product yield curve was more consistent with the fission of plutonium than of highly enriched uranium (HEU), so the US scientists guessed that the Soviet nuclear weapon was plutonium-based and was therefore an implosion-type bomb.³⁴ In addition, US scientists tested the particles for traces of neptunium-237, an isotope produced from U-238 in nuclear reactions involving energetic neutrons. The test allowed the conclusion that the RDS-1 bomb probably had a layer of natural uranium as a tamper and reflector.³⁵

³⁰ Saey, P. R. J., 'Ultra-low-level measurements of argon, krypton and radioxenon for treaty verification purposes', *ESARDA Bulletin*, no. 36 (July 2007), p. 44; and Kalinowski, M. B. et al., 'Environmental sample analysis', eds Avenhaus et al. (note 11), pp. 376–77.

³¹ Ziegler and Jacobson (note 28), pp. 7–8.

³² Ziegler and Jacobson (note 28), p. 201.

³³ Ziegler and Jacobson (note 28), pp. 204–11.

³⁴ The fission product yield curve, sometimes referred to as the 'Mae West curve' due to its characteristic 2-peak form, is a graph of the mass or mole yield of fission products against their atomic number. Its shape depends on the fissile isotope and the energy of the neutrons inducing fission. See e.g. Saey (note 30), p. 43.

³⁵ A tamper is a layer of a dense material that surrounds the fissile material in a nuclear weapon. The tamper lengthens the short time for which the material holds together under the extreme pressures of the explosion and thereby increases the efficiency of the weapon by increasing the proportion of the fissile material that undergoes fission. A neutron reflector is a layer of material immediately surrounding the

Analysing the known meteorological data, meteorologists made backward projections of the trajectories of the air masses. The calculated age of the radioactive isotopes in the samples gave an estimate of the time of the event: sometime between 26 and 29 August 1949. This provided the cut-off time at which to stop the backward projection of air mass trajectories and thus defined, accurately but not precisely, the area where the test was conducted.³⁶

It is important to note that all this information was obtained using the radiochemical analysis methods available at the time. Contemporary analysis methodologies and equipment are reported to be much more advanced and sensitive and to be capable of providing more data on a weapon's design, yield and other parameters.³⁷ The same or similar techniques could also be employed to provide information on the origin of the nuclear material used in a nuclear explosive device set off in an act of terrorism. For instance, post-explosion analysis of the fission products may provide estimates of the pre-explosion isotopic content of the fuel.³⁸ This, in turn, may provide a nuclear fingerprint which can be used to identify the source of the material and, perhaps, to ensure correctly targeted retribution.

The successful detection of the RDS-1 test spurred the rapid development of the USA's global nuclear explosion monitoring infrastructure. The monitoring is performed by the US Atomic Energy Detection System (USAEDS), operated by AFOAT-1's successor, the US Air Force Technical Applications Center (AFTAC). In addition to national intelligence activities, AFTAC monitors compliance with the 1963 Partial Test-Ban Treaty (PTBT), the 1974 Threshold Test-Ban Treaty and the 1976 Peaceful Nuclear Explosions Treaty.³⁹ In 1998 USAEDS detected and confirmed the nuclear nature of explosions in India and Pakistan.

USAEDS was also involved in investigating the so-called Vela incident in 1979. On 21 September 1979 AFTAC personnel conducting a routine readout of the Vela 6911 monitoring satellite received sensor readings very similar to the 'double flash' that is characteristic of an atmospheric nuclear weapon test.⁴⁰ The US Government launched an extensive investigation, including a massive air sampling operation by AFTAC aircraft and the collection of environmental samples by Central Intelligence Agency (CIA) personnel. No traces of radioactive debris relevant to the event were found.⁴¹

Following North Korea's announcement in October 2006 that it had conducted an underground nuclear test, the US Air Force dispatched its WC-135W Constant Phoenix atmospheric collection aircraft, which is normally used for verification of the PTBT. It collected useful samples starting from two days after the event. Based on the analysis of collected radioactive debris, AFTAC was able to verify to US national

fissile material which reflects neutrons back to the core and thus reduces the critical mass of the missile material and increases the weapon's efficiency.

³⁶ Ziegler and Jacobson (note 28), pp. 204–11.

³⁷ Moody, Hutcheon and Grant (note 4), pp. 203–205.

³⁸ Moody, Hutcheon and Grant (note 4), p. 205.

³⁹ For summaries of these treaties and lists of their parties see annex A in this volume.

⁴⁰ The nature of a double flash produced by a nuclear explosion in the atmosphere is explained in Barasch, G. E., 'Light flash produced by an atmospheric nuclear explosion', LASL-79-84, Los Alamos Scientific Laboratory, Nov. 1979, <<http://www.gwu.edu/~nsarchiv/NSAEBB/NSAEBB190/>>.

⁴¹ Richelson, J. T., *Spying on the Bomb: American Nuclear Intelligence from Nazi Germany to Iran and North Korea* (W. W. Norton & Company: New York, 2006), pp. 288, 315.

authorities that ‘the event was nuclear in nature’.⁴² The governments of the USA and other states that had investigated North Korea’s claim and, later, the CTBTO independently concluded that the event had been a nuclear explosion.⁴³

The US Government did not provide details of the radioactive sample collection and analysis that followed North Korea’s announcement. A non-governmental researcher has concluded that the aircraft was probably able to collect only two fission products in detectable quantities: Xe-133 and Xe-135.⁴⁴ The same study concluded that the activity ratio of these two isotopes could be used to confirm that the test was nuclear, although the samples were probably not sufficient to ‘determine the fissile material used . . . , particularly if detected as much as two days after a test’.⁴⁵ Much less information could have been derived from collected debris in the case of the 2006 North Korea test than that of the 1949 RDS-1 test because the former explosion was underground, concealing almost all isotopes.

The same capacities employed by AFTAC in nuclear test verification are also used to help with the IAEA’s safeguards work. AFTAC is a member of the IAEA’s NWAL, specializing in analysing environmental samples.⁴⁶ As of 2007, AFTAC is one of three organizations in the world providing the IAEA with the capacity to analyse the isotopic composition of particles in swipe samples using one of the most sensitive techniques available, FT-TIMS.⁴⁷

Uranium particle analysis to verify Iran’s declaration to the IAEA

Media reports published in August 2002 prompted the IAEA to investigate the existence of undeclared uranium enrichment facilities in Iran.⁴⁸ During the visit of a high-level IAEA delegation to Iran in February 2003, Iranian authorities acknowledged the construction of two centrifuge enrichment plants at Natanz, the Pilot Fuel Enrichment Plant (PFEP) and the large Fuel Enrichment Plant (FEP), as well as the existence of a workshop of the Kalaye Electric Company (KEC) in Tehran used for production of centrifuge components. Iran stated that its enrichment programme was indigenous

⁴² US Air Force Intelligence, Surveillance and Reconnaissance Agency, ‘Air Force Technical Applications Center’, Fact sheet, June 2007, <<http://www.afisr.af.mil/library/factsheets/factsheet.asp?id=10309>>.

⁴³ US Office of the Director of National Intelligence, ‘Statement by the Office of the Director of National Intelligence on the North Korea nuclear test’, News release, 16 Oct. 2006, <<http://www.dni.gov/announcements/announcements.htm>>; Fedchenko, V. and Ferm Hellgren, R., ‘Nuclear explosions, 1945–2006’, *SIPRI Yearbook 2007: Armaments, Disarmament and International Security* (Oxford University Press: Oxford, 2007), pp. 552–57; and CTBTO, ‘The CTBT verification regime put to the test: the event in the DPRK on 9 October 2006’, Featured article, 4 Sep. 2007, <http://www.ctbto.org/press_centre/featured_articles/2007/2007_0409_dprk.htm>. Among the other states that investigated the event, Sweden collected and analysed air samples from South Korea. Swedish Defence Research Agency (FOI), ‘FOI found radioactive xenon following explosion in North Korea’, Press release, 19 Dec. 2006, <http://www.foi.se/FOI/Templates/NewsPage___5412.aspx>.

⁴⁴ Zhang, H., ‘Off-site air sampling analysis and North Korean nuclear test’, Proceedings of the Institute for Nuclear Materials Management 48th Annual Meeting, Tucson, Ariz., 8–12 July 2007, <<http://www.belfercenter.org/publication/17537/>>.

⁴⁵ Zhang (note 44), p. 6.

⁴⁶ IAEA (note 13).

⁴⁷ US Congress (note 24), p. 26; IAEA (note 13); and Bevaart, Donohue and Fuhr (note 15).

⁴⁸ For a detailed account of the disclosure of Iran’s pursuit of sensitive nuclear fuel cycle technologies see Kile, S. N., ‘Nuclear arms control and non-proliferation’, *SIPRI Yearbook 2004: Armaments, Disarmament and International Security* (Oxford University Press: Oxford, 2004), pp. 604–12.

and that no enrichment activities involving actual nuclear material were being conducted at those or other locations at that time.⁴⁹

That was a claim of a considerable significance because Iran—like any other non-nuclear weapon state party to the NPT which has in force a CSA with the IAEA—is required to declare any new nuclear facility before it commences operation and to provide the IAEA with specific information on its design.⁵⁰ States do so by completing a design information questionnaire (DIQ). The specific details of the DIQ submission are defined in an annex to the safeguards agreement that describes subsidiary arrangements. Such subsidiary arrangements are negotiated by the IAEA separately with each state.

Since 1976 all states have been required to complete a DIQ for any new installation no later than 180 days before the introduction of nuclear material to the facility. In the aftermath of the 1991 Gulf War the IAEA Board of Governors decided to change the subsidiary arrangements in subsequently negotiated CSAs so that the states would have to ‘provide design information to the Agency at the time of the decision to construct, or to authorize the construction of, any nuclear facility (i.e. well before construction actually begins) in order to create confidence in the peaceful purpose of the facility’.⁵¹ However, Iran did not accede to these new rules until 26 February 2003, after the existence of enrichment facilities had been discovered.⁵²

Thus, if no nuclear material had been introduced to those facilities before they were discovered, Iran had not committed an act of non-compliance with its CSA. If the material had been introduced, then the failure to declare such a facility would be in contravention of Iran’s CSA.⁵³

In order to determine whether nuclear material had been introduced into the facilities, the IAEA began to take environmental samples at the Natanz plants in March and at the Tehran workshop in August 2003.⁵⁴ The IAEA inspectors noted that there had been ‘considerable modification’ of the KEC site before they could take samples and that this ‘may impact on the accuracy of the environmental sampling and the Agency’s ability to verify Iran’s declarations’.⁵⁵ Despite the interference, samples revealed the presence of uranium particles at both sites that were not consistent with the material in the inventory declared by Iran to the IAEA.

In total, discoveries of particles of natural uranium, low-enriched uranium (LEU) and HEU particles of up to 70 per cent enrichment (with the majority of the HEU being enriched to 36–54 per cent of U-235) were reported by the IAEA. This pro-

⁴⁹ IAEA, Board of Governors, ‘Implementation of the NPT safeguards agreement in the Islamic Republic of Iran’, Report by the Director General, GOV/2003/40, 6 June 2003, p. 2.

⁵⁰ ‘Design information’ is defined by the IAEA as ‘information concerning nuclear material subject to safeguards . . . and the features of facilities relevant to safeguarding such material’. IAEA, *IAEA Safeguards Glossary*, International Nuclear Verification Series no. 3, 2001 edn (IAEA: Vienna, 2002), p. 26.

⁵¹ IAEA, Board of Governors, ‘Strengthening of agency safeguards: the provision and use of design information’, GOV/2554/Attachment 2/Rev.2, 1 Apr. 1992, p. 1. See also Hibbs, M., ‘Safeguards agreement required early completion of DIQ by Syria’, *Nuclear Fuel*, vol. 32, no. 23 (5 Nov. 2007), p. 9; and Schriefer, D., ‘The international level’, eds Avenhaus et al. (note 11), pp. 437, 452.

⁵² IAEA (note 49), p. 4.

⁵³ In fact, if Iran had intended to introduce nuclear material into the facilities within 180 days of their discovery, then, technically, Iran would have been in contravention of its CSA. However, there is no way in which such a supposition could be proved.

⁵⁴ IAEA, Board of Governors, ‘Implementation of the NPT Safeguards Agreement in the Islamic Republic of Iran’, Report by the Director General, GOV/2003/75, 10 Nov. 2003, annex 1, pp. 7–8.

⁵⁵ IAEA, Board of Governors, ‘Implementation of the NPT Safeguards Agreement in the Islamic Republic of Iran’, Report by the Director General, GOV/2003/63, 26 Aug. 2003, p. 7.

vided conclusive evidence of undeclared activity: either enriched uranium had been imported or enrichment experiments had taken place in Iran. Many of the LEU and HEU particles also had an elevated U-236 content, suggesting the use of uranium extracted from spent nuclear fuel. This again pointed to either unknown reprocessing activities or an import of enriched material.⁵⁶

Confronted with the evidence, Iran admitted its involvement in both undeclared domestic enrichment experiments and a covert international nuclear trade. In a letter of 21 October 2003 Iran admitted that, contrary to its earlier statements, it had conducted small-scale enrichment experiments between 1999 and 2002. These experiments achieved an enrichment level of no more than 1.2 per cent U-235.⁵⁷ More importantly, in August 2003 Iran officially admitted that it had in fact imported some centrifuge parts. It suggested that the HEU contamination originated from imported parts and identified Pakistan as a supplier.⁵⁸ Pakistan eventually agreed to hand over centrifuge components requested by the IAEA to allow comparison of uranium particles.⁵⁹ The IAEA received components on 21 May 2005, took swipe samples and analysed them at SAL. The results confirmed that most of the contamination was probably of Pakistani origin, as stated by Iran.⁶⁰

The experience in Iran has demonstrated that, although nuclear forensic techniques may be useful for safeguards implementation, they must be complemented with other sources of data, such as open source analysis, satellite imagery and information from IAEA member states. The discovery of enriched uranium in Iran was possible only after the IAEA learned about the Natanz plants from elsewhere.

Plutonium age determination to verify North Korea's initial declaration

North Korea acceded to the NPT in 1985 and, after a significant delay, signed a comprehensive safeguards agreement with the IAEA on 30 January 1992.⁶¹ As required by Article 62 of the CSA, on 4 May 1992 North Korea submitted to the IAEA 'an initial report on all nuclear material subject to safeguards'.⁶²

The report contained a declaration that North Korea had conducted a single experiment in March 1990 at the Radiochemical Laboratory in Yongbyon on separating less than 100 grams of plutonium from the damaged spent fuel rods removed from the

⁵⁶ IAEA, Board of Governors, 'Implementation of the NPT Safeguards Agreement in the Islamic Republic of Iran', Report by the Director General, GOV/2004/83, 15 Nov. 2004, p. 9.

⁵⁷ IAEA (note 54), pp. 6–7.

⁵⁸ Kile, S. N., 'Nuclear arms control and non-proliferation', *SIPRI Yearbook 2005: Armaments, Disarmament and International Security* (Oxford University Press: Oxford, 2005), pp. 558–59.

⁵⁹ Bokhari, F., 'Pakistan may hand over nuclear centrifuges', *Financial Times*, 25 Mar. 2005; and 'Centrifuge parts sent to IAEA', *Dawn* (Karachi), 27 May 2005.

⁶⁰ IAEA, Board of Governors, 'Implementation of the NPT Safeguards Agreement in the Islamic Republic of Iran', Report by the Director General, GOV/2005/67, 2 Sep. 2005, p. 4.

⁶¹ The Agreement of 30 January 1992 between the Government of the Democratic People's Republic of Korea and the International Atomic Energy Agency for the Application of Safeguards in Connection with the Treaty on the Non-Proliferation of Nuclear Weapons entered into force on 10 Apr. 1992; its text is published in IAEA, INFCIRC/403, May 1992. See also Lockwood, D. and Wolfsthal, J. B., 'Nuclear weapon developments and proliferation', *SIPRI Yearbook 1993: World Armaments and Disarmament* (Oxford University Press: Oxford, 1993), p. 244.

⁶² An 'initial report' is defined by the IAEA as a document required under the CSA containing 'an official statement by the State on all nuclear material subject to safeguards', from which the IAEA 'establishes a unified inventory of all nuclear material . . . for the State'. IAEA (note 50), p. 94.

adjacent 25 megawatt-thermal gas-graphite reactor. In the summer of 1992 the IAEA conducted initial inspections in order to verify this and other declared information.

Inspectors took swipe samples from inside and outside glove boxes at the end of the reprocessing line at Yongbyon, where freshly separated plutonium is converted from liquid form into oxide compound. Inspectors also took samples of separated plutonium and the nuclear waste from which it was said to have been separated. The samples were sent to SAL and US laboratories to determine their elemental and isotopic composition. These data could then be used for calculating the 'age' of the material.⁶³

The 'age' of nuclear material is defined as the time elapsed since its last separation or latest chemical purification. Plutonium isotopes undergo various types of radioactive decay, producing so-called daughter nuclides. The greater is the age of the material, the more the 'parent' isotope decays, to be replaced by daughter nuclides. In other words, 'the disintegration of a radioactive (parent-) isotope and the build up of a corresponding amount of daughter nuclide serve as built-in chronometer'.⁶⁴ The ratios of some parent-daughter pairs can therefore be useful to calculate the material's age. For plutonium such pairs are: Pu-238-U-234, Pu-239-U-235, Pu-240-U-236 and Pu-241-americiam-241.

The age of the plutonium in the North Korean swipe samples should have been the time since it was separated from the spent fuel, which was declared as being slightly more than two years by the time of the analysis in the summer of 1992. The analysis of the plutonium decay products by the IAEA suggested that North Korea separated plutonium not once, as declared, but three times—in 1989, 1990 and 1991. The analysis could not determine the amount of separated plutonium that had been produced, but it did provide yet more evidence that the North Korean declaration to the IAEA was not entirely correct.⁶⁵

Age determination techniques could also form part of the verification processes of the proposed fissile material cut-off treaty. In this context, samples of both plutonium and uranium could be analysed in order to establish if they were produced before or after a certain cut-off date.

IV. Conclusions

Developments in technology offer continual improvements in the tools for verification and enforcement of national and global non-proliferation mechanisms. One of those tools, nuclear forensic analysis, was invented in its earliest form even before the first nuclear weapon was tested. For decades it developed in the context of cold war arms control treaty verification, until its utility for other important applications—such as nuclear smuggling investigations, IAEA safeguards and CTBT verification—was demonstrated during the 1990s. The anti-terrorism capability of nuclear forensics has been emphasized since the terrorist attacks of 11 September 2001 on the USA.

Nuclear forensic analysis is an impressive tool that is capable of extracting useful information from minute traces of material. It is most productive if used in con-

⁶³ Fischer, D., *History of the International Atomic Energy Agency: The First Forty Years* (IAEA: Vienna, 1997), p. 289; and Albright, D., 'North Korean plutonium production', *Science & Global Security*, vol. 5, no. 1 (Dec. 1994), pp. 66–67.

⁶⁴ Mayer, Wallenius and Ray (note 11), p. 401.

⁶⁵ Albright (note 63), p. 66.

nection with other techniques: nuclear weapon test analysis is more precise if examination of airborne radioactive debris is complemented with seismological, hydro-acoustic and infrasound monitoring; and environmental sampling is most useful for IAEA safeguards verification if complemented with input from open source and overhead imagery analysis and improved provision of design information.

There are also legal and political constraints. Nuclear forensics is most useful if applied quickly and as close to the event as possible. The denial of timely access to the location or the material can diminish accuracy or, in extreme cases, prevent useful information from being obtained by nuclear forensics. Although often designed specifically to deal with the lack of access, the technology is still limited by the external conditions determining when and how it can be applied. Thus, while of increasing importance, nuclear forensics remains just one tool in the verification and enforcement toolbox.