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# ANALYTICAL METHODS UNDER EMERGENCY CONDITIONS

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## 1 PURPOSES

### 1.1 Medical Treatment

An emergency condition is defined here as a situation in which results on internal radioactive contamination are needed quickly. The principal purpose for speed is to determine if medical treatment to increase the natural elimination rate is advisable. The goal is to minimize the damage due to internally deposited radionuclides, and the effectiveness of treatment generally decreases rapidly with time. If analytical results are not needed to decide on medical treatment, much of the urgency for speed disappears. If a large number of individuals are potentially contaminated, quick evaluations are useful in delineating those who require immediate attention. Negative results - a determination that medical treatment is not required - is also important to relieve anxiety and eliminate any unnecessary risk and expense associated with treatment.

### 1.2 Other Purposes

When a decision is reached without the help of analytical results that medical treatment is not needed, quick evaluation of potential uptake, deposition, and dose through analytical measurements may still be desirable. An overall assessment of the seriousness of the situation will

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be desired by the affected individuals, the facility management, governmental regulatory bodies, and the news media and public if press releases are distributed.

## 2 REQUIREMENTS FOR EMERGENCY EVALUATIONS

Quickly, as used in Section 1.1, means within a few hours to a few days after the emergency condition is recognized or after the sample is obtained. Analytical methods - in vivo or otherwise - are available that meet these time constraints if sufficient activity is present. A difficulty arises in deciding first on the minimum body content that must be measured quickly. Several other lectures in this series discuss medical treatment, dose assessment, and management of accident cases. For the purposes of this lecture, a criterion on sensitivity of detection is needed in order to discuss applicable analytical methods. Although several criteria are defensible, agreement is not universal and decisions in this area are partly subjective. As a starting point to evaluate the sensitivity needed for emergency work, a recommendation in ICRP Publication 6 (1) states that when total accidental intake exceeds the amount that results from an annual intake at the maximum levels given in ICRP Publication 2, the case shall be referred to competent medical authorities for appropriate action. An updated, somewhat analogous criterion would be to use the ALI (annual limit of intake) proposed more recently by the ICRP (2). The ALI is the quantity of radioactive material taken in that results in a committed dose equivalent of 5 rem for stochastic effects and 50 rem for non-stochastic effects. ALI values are

published in ICRP 30 for many radionuclides. Excretion rates that result from an intake equivalent to the ALI must be calculated from retention/elimination models. These calculations are beyond the scope of this lecture, but are covered in other lectures in this series. However, the ability of the analytical methods to detect levels of this order will be discussed. This discussion is not meant to imply that a decision to treat will be based on a dose of 5 rem. Such decisions are made on a case-by-case basis, and require a risk/benefit analysis. Considerable information on treatment is given in references (3) and (4).

### 3 METHODS

#### 3.1 In Vivo Measurements

This is the first method to be considered in an emergency, and should be used if it is applicable. Although this topic is the subject of a separate lecture, it will be discussed briefly as it applies to emergencies.

##### 3.1.1 Whole Body Counting

Whole body counting has the advantage of speed, accuracy, and good sensitivity for a large number of radionuclides. Counting times are short, less than 30 minutes, and a direct measurement of the desired quantity, the body radioactivity, is obtained. Even with the problems of calibrating a detection system for a radiation source as large as the human body and the problems of activity distribution in the body, direct counting provides the best measurement of body content available.

There are two principal limitations. A single measurement will frequently not distinguish between activity in the body and that on the skin and hair. Several measurements in different positions, and careful examination of gamma spectral shapes are helpful. The radiations obviously must leave the body, limiting the method to nuclides that emit moderately energetic gamma- or x-rays or to energetic beta-ray emitters, in which case the bremsstrahlung radiation is counted. The absorption of electromagnetic radiation by the body limits normal whole-body counting to the detection of gamma rays of energies greater than about 50 keV, although x-rays of much lower energy (such as the 17-keV x-rays emitted by the uranium daughter of plutonium-239) can be detected if the material is present near the surface or in other favorable body locations. The detectors most used are sodium iodide (thallium) crystals and lithium-drifted or hyperpure germanium diode detectors. The best detection system is a large sodium iodide crystal to measure the quantity and a large germanium crystal to identify the energies.

With heavy shielding, equivalent to about 15 cm of iron, and large sodium iodide detectors, about 20 cm x 10 cm in diameter, approximately 1 nCi of radium-226 or its equivalent in gamma-ray activity can be detected. This sensitivity is adequate to detect less than the ALI for most nuclides that emit gamma-rays greater than 50 keV in reasonable abundance. However, following an accident such elaborate equipment is not necessary for satisfactory whole-body counting of many nuclides. Quantities of the order of 1 Ci of gamma emitters can be detected with a moderately sized crystal, 10 cm x 10 cm, without the use of any shielding. This makes is

possible to improvise a whole-body counter for many conceivable emergency situations. To improve the sensitivity over that obtained with no shielding, it is relatively simple and inexpensive to shield the crystal with lead bricks and perhaps provide some additional shielding with iron plates or concrete walls and floors.

Table 3.1-1 lists some common nuclides that can be satisfactorily detected by whole-body counting in less than 30 minutes. The two beta emitters, strontium-90-yttrium-90 and phosphorus-32 are best determined by bremsstrahlung counting with a sodium iodide crystal a few millimeters thick to obtain the optimum efficiency to background ratio. The detection limit for energetic beta emitters is about 10 nCi with good shielding.

### 3.1.2 Organ Counting

The organs of primary interest are the lungs (or chest cavity), the liver, and the thyroid, the first because many intakes are through inhalation (particularly of insoluble material), the liver because colloidal particles (the transuranium elements tend to behave as colloids at body pH's unless complexed) concentrate in the liver, and the thyroid because iodine concentrates in that organ.

The measurement of bremsstrahlung or energetic gamma-rays in the chest cavity can be done by the same methods used for whole-body counting of these emitters. Of special concern are the heavy elements that emit only alpha particles, x-rays, and low-energy beta particles. The most sensitive detector for measuring the x-rays emitted in the decay of plutonium-239 (about 17 keV) is the phoswich scintillation system. A thin

TABLE 3.1-1

Annual Limits of Intake and Whole Body Counting  
Detection Limits for Selected Nuclides

Nuclide	ALI (nCi)	Detection Limit (nCi)
$^{32}\text{P}$	$2.7 \times 10^5$	50
$^{57}\text{Co}$	$5.4 \times 10^5$	10-50
$^{58}\text{Co}$	$8.1 \times 10^5$	10
$^{60}\text{Co}$	$2.7 \times 10^4$	1-5
$^{90}\text{Sr}$	$5.4 \times 10^6$	10
$^{137}\text{Cs}$	$6.8 \times 10^4$	1.5-10
$^{239}\text{Pu}, ^{241}\text{Am}$	5.4	0.4 nCi (liver)

Note: The ALI values were taken from reference (2).  
The detection limits were taken from reference (4).

sodium iodide crystal (a few mm thick) is cemented to a thicker cesium iodide crystal (10-20 mm thick), which is coupled to a phototube. Ionizing radiation that produces scintillations detected by both crystals is rejected electronically on the basis of pulse shape discrimination. This anti-coincidence shielding reduces the background in the sodium iodide x-ray detector significantly. Two 5-inch diameter phoswich detectors in an iron-shielded room will detect about 35 nCi of pure plutonium-239, if the chest thickness averages 28 mm (5). This is satisfactory for emergency measurements, although better sensitivity would be desirable. Improved sensitivity may be obtained in the future with arrays of large diameter germanium or lithium-drifted silicon detectors. The phoswich detector is also sensitive to 0.3 nCi of americium-241 and 0.2 nCi of uranium-235. Gas (xenon) filled proportional counters are 3 to 4 times less sensitive than the phoswich crystals, but give better resolution.

The same detectors can be used to measure actinides in the liver by placing them over the right side of the abdomen, followed by a background count over the left side (5). Thyroid counting for radioiodines with sodium iodide is common and has adequate sensitivity. Additional information on these and other methods is given in reference (3).

### 3.1.3 Wound Monitoring

A frequent route of intake of radioactive material is through the broken skin. These accidents occur primarily in chemical laboratories and involve the fingers and hands. Explosions of glass containers and other



cuts with contaminated glassware are common causes. Since this is a special case of whole body and organ counting, the same techniques can be used. The area to be counted is so much smaller, however, that small diameter detectors are used to reduce the background and to allow better localization of the contamination. Of special interest is the detection of the actinide elements, particularly plutonium, in wounds. For this purpose, a thin sodium iodide crystal (a few mm thick) is commonly used to measure the x-rays, and is useful for the energy region from 10-100 keV. The sensitivity for plutonium-239 is approximately 0.2 nCi; for americium-241, the sensitivity is about 0.05 nCi. Since low-energy radiation is being measured, the sensitivity decreases with the depth of the material beneath the skin. If beta and gamma-ray emitters are present, the background is elevated and the sensitivity becomes proportionately poorer. A phoswich will reduce this background. Small area silicon and germanium diode detectors are also useful, and give much better resolution. Cadmium telluride diode detectors have the advantages of good efficiency and resolution, low operating voltages, room temperature operation, and low cost. A small detector of 10 mm<sup>2</sup> diameter is useful for locating small areas of contamination on the skin. Several wound monitors intended primarily for plutonium have been described in the literature (6-8).

### 3.2 Excretion and Other Sample Analyses

In most cases of suspected large intakes, it is important to collect excretions and other types of samples, such as nose swipes, nose blows, blood, surface smears, and air samples. The types and timing of sample collection is primarily the concern of the health and safety personnel,

the physicians, and the health physicists who evaluate the accident. The analyst, however, should be consulted on areas such as sample preservation, proper identification, labeling, and containers, and must be provided with information about the accident so he can perform the correct analysis. These factors are of particular concern if a large number of samples are collected. References (3) and (4) contain valuable information on these topics.

An important factor is cross-contamination of samples, reagents, glassware, and equipment. One fairly radioactive sample processed in an analytical laboratory together with normal samples can contaminate and give false high results for several months. Suspected high-activity samples must be screened and handled separately.

### 3.2.1 Sample Treatment

Sample treatment depends on the analytical method to be used. If gamma-ray spectrometry is applicable, the sample need only be treated to make it homogeneous and of a definite size for which the counting efficiency is known. For example, urine should be acidified with hydrochloric acid to prevent precipitates from forming and fecal samples can be compacted to a known size.

For chemical separations, treatments must be appropriately short so the entire procedure can be completed within the desired time. If samples require evaporation or the destruction of organic matter, much time is saved if the samples are relatively small compared to the sizes used for normal operations. Urine sample volumes should be less than 200 ml, pre-

ferably 50-100 ml; fecal samples, 10-20 g (wet weight), blood volumes about 10 ml. Such sizes are satisfactory since high sensitivity is not needed. Many emergency analytical methods for urine can be performed directly on the unashed sample, and volumes up to about 75 ml can be conveniently and rapidly handled in standard laboratory glassware and equipment. Urine samples can be ashed, if necessary, as described in lecture 17 on "Analytical Methods for Radionuclides in Liquid and Solid Media." Fecal samples must be ashed. Since drying and high temperature ignition, followed by dissolution of the ash is time consuming, such samples should be ashed by several treatments with concentrated nitric acid and strong hydrogen peroxide, or by the hydroxyl oxidation procedure of Sansoni (9). Blood samples are similarly treated. Other materials, such as nose swipes, air filters, surface smears, usually have a small amount of organic materials and inorganic residue. These can be rapidly ashed by either chemical or high temperature oxidation.

### 3.2.3 Rapid Chemical Separations

If alpha- or beta-particle activity must be detected, simple and rapid chemical separations are required. Chemical methods that can be completed in less than two hours are probably limited to blood volumes less than about 15 ml, urine volumes less than about 100 ml, and fecal samples less than about 15 g.

Table 3.2-1 gives the minimum detectable amounts of a number of important nuclides that can be determined in excretions in 1 to 2 hours. These values were determined for the following conditions. Samples and

TABLE 3.2-1

## Minimum Detectable Amounts by Excretion Analysis

Nuclide	Direct Counting		Chemical Separation
	Urine (pCi/1400 ml)	Feces (pCi/sample)	Urine (pCi/1500 ml)*
$^3\text{H}$	$6.7 \times 10^4$		
$^{14}\text{C}$ , $^{35}\text{S}$ (total)	$1.8 \times 10^4$		
$^{35}\text{S}$ (sulfate)	-	-	$1.4 \times 10^3$
$^{32}\text{P}$ , $^{90}\text{Sr}$	-	-	35
$^{45}\text{Ca}$	-	-	150
$^{99}\text{Tc}$ , $^{147}\text{Pm}$	-	-	70
$^{59}\text{Fe}$	200	40	-
$^{60}\text{Co}$	100	20	-
$^{65}\text{Zn}$	180	35	-
$^{95}\text{Zr}$ ( $^{95}\text{Nb}$ )	250	50	-
$^{106}\text{Ru}$ ( $^{106}\text{Rh}$ )	125	25	-
$^{127\text{m}}\text{Te}$	500	100	-
$^{131}\text{I}$	150	30	-
$^{137}\text{Cs}$	300	60	-
$^{144}\text{Ce}$	450	90	-
$^{226}\text{Ra}$	950	190	7
Actinide Alpha Emitters, $^{210}\text{Po}$	-	-	7
$^{241}\text{Am}$	300	60	7

backgrounds are counted for 15 minutes; the minimum detectable amount is then taken as three times the standard deviation of a 15-minute background. Direct counting of tritium, carbon-14, and sulphur-35 is done on 1.0 ml samples in a standard liquid scintillation counter. Direct counting of the other nuclides is done with a solid 10 cm x 10 cm diameter lead-shielded sodium iodide crystal using 250 ml of urine. Alpha counting of separated activity is performed in a 2 windowless counter with a background of 0.05 count/min. Beta counting of separated activity is performed in a 2-in diameter, gas-flow, windowed ( $1 \text{ mg/cm}^2$ ), shielded counter with a background of 1.2 count/min. The separations are performed on 50 ml of urine, and the daily urinary output is assumed to be 1400 ml/day. The sensitivity of blood analysis by chemical separation would be similar, per sample, as for urine, while the sensitivity by direct counting would be proportional to sample size.

The conditions given above are not difficult to obtain, and the sensitivities meet the criterion of detecting one ALI. If necessary, there are several ways of improving the sensitivity, some requiring additional time. The sensitivity of gamma-ray counting can be improved by evaporating urine samples to obtain better geometry, by counting in a well crystal, or by counting with a large germanium diode detector. Chemical separations can be performed on larger samples, which will improve the sensitivity in proportion to the increase in sample size. Backgrounds can be decreased by a factor of 2 or 3 with better counting equipment, and counting times can be increased.

As has been mentioned earlier, whole-body counting is the preferred method for determining intakes and body burdens. There is, however, an important group of long-lived alpha emitters and low-energy pure beta emitters, including the bone seekers, for which whole-body counting is not possible or is marginal. It is useful, therefore, to consider what type of excretion sampling should be done for these nuclides in emergencies.

Experience has demonstrated the value of fecal sampling for certain types of intakes. When material is inhaled in a form not readily soluble in biological fluids, elimination will be primarily by the feces. However, several days of samples are required to evaluate the intake. Quantitative interpretation of fecal elimination in terms of lung deposition cannot be expected shortly after intake, although fecal elimination is a valuable qualitative index of exposure. Soluble forms of trace elements may be eliminated in the feces when they are inhaled in a biologically insoluble matrix. Radium is excreted almost entirely in the feces regardless of manner, form, or time of intake. In such a case, urinalysis is not as sensitive as fecal analysis, but since the feces will include radium coming directly from the lung and the skeleton as well as currently ingested radium, interpretation of fecal excretion will be difficult.

Significant urinary excretion can occur soon after the inhalation of presumably insoluble materials. This is illustrated by two cases of accidental inhalation at Argonne National Laboratory of small quantities of irradiated metal dust. In one case the elimination of cobalt-60 was 50 times greater in the urine than in the feces less than one day after in-

take. Maximum fecal excretion occurred between 1.5 and 3 days after intake. In the second case, involving cyclotron-irradiated material, daily urinary and fecal excretion of cobalt-57, cobalt-58, and zinc-65 were of the same order of magnitude. This indicates that urine sampling can be useful shortly after inhalation of poorly soluble material. Since some time is required for material to move from the lung to and through the GI tract, urine may contain the first evidence of intake.

Some rapid chemical methods for urine samples are summarized in Table 3.2-2. They were developed for the separation of nuclides that cannot be detected by gamma-ray counting, but they can be used for chemically similar nuclides regardless of mode of decay. These analyses can be completed in 1 to 2 hours if urine volumes of 100 ml or less are used. Blood and fecal samples can be analyzed by the same methods after first oxidizing the organic matter. High-temperature oxidation should be avoided since dissolution of trace elements such as plutonium may be very slow after this treatment.

All of the methods given use precipitation, co-precipitation, or solvent extraction separations since they can be done rapidly. A separation consisting of one precipitation only is probably the optimum method, provided the precipitation and carrying are complete in a few minutes, since the precipitate can be separated and counted quickly. Ion exchange separations generally require more time than precipitation or solvent extraction.

TABLE 3.2-2

## Chemical Methods for Emergency Analyses

Element	Procedure	Reference
Barium, calcium, strontium, rare earths	Precipitate calcium oxalate or phosphate	11
Phosphorus	Precipitate calcium phosphate	11
Barium, radium, sulfur (as sulfate)	Precipitate barium sulfate from acid solution	11
Americium, plutonium	Adjust pH to 9. Filter through membrane filter	10
Americium, curium, plutonium, thorium, promethium, and other rare earths	Precipitate rare-earth fluoride from acid solution	11
Technetium	Precipitate copper sulfide from acid solution	11
Uranium, plutonium, thorium, transplutonium elements	Extract with solution of organic phosphate ester; count extract directly in liquid scintillation counter, or evaporate to dryness and count	11
Uranium	Measure fluorescence with fluorophotometer	12



In addition to the time required for a single analysis, consideration must also be given to the analysis of a large number of samples in a short elapsed time. The methods are designed so that one worker can analyze several samples simultaneously and with simple and readily available equipment in little more than is required for one sample.

An important consideration in the analysis of complex organic materials for traces of radionuclides is the chemical form of the desired radionuclide. Some elements can be excreted in urine in forms which do not undergo the expected chemical reactions, possibly because they are complexed or colloidal. In lecture 18 the incomplete coprecipitation of plutonium with an alkaline earth phosphate was discussed. The plutonium in such samples could be returned to a form that coprecipitates with the phosphate by acidifying and heating the urine prior to precipitation. It is to be expected that such problems will be encountered primarily with trace concentrations of elements that have transition-element electronic structures and large oxidation numbers. With elements that do not form strong complexes in acid solution or do not hydrolyze readily, such as strontium, or are present in macroscopic amounts, such as calcium, this behavior does not occur. Although every separation method should be tested with urine containing naturally-excreted radionuclides, such material is not readily available. Of the methods reported in Table 3.2-2, the co-precipitation of plutonium with cerium as the rare earth fluoride was found to be satisfactory with urine containing excreted plutonium from experimental animals. Presumably other tri- and tetra-valent lanthanides and actinides will behave similarly.

Calcium oxalate can be precipitated from urine adjusted to pH 4 by adding a dilute acetic acid solution of ammonium oxalate. After a digestion period of 15 minutes, the precipitate is centrifuged, washed with dilute ammonium hydroxide and acetone, dried, and counted. The acetone removes organic matter that sometimes precipitates. At least 80% of radiostrontium is carried from normal urine without the addition of strontium carrier. However, a few milligrams of an alkaline-earth or rare-earth carrier ion can be added to insure a minimum carrier concentration.

The precipitation of the alkaline-earth phosphates should be made from a hot solution by first adding phosphoric acid and then making the solution alkaline with concentrated ammonium hydroxide. After a 30 minute digestion period, the precipitate is centrifuged and treated as given for the oxalate.

Barium sulfate can be used to separate radiobarium, radium, or sulfur-35 present as sulfate. To precipitate the sulfate, excess barium ion is added to urine made about 0.1N in acid. Approximately 300 mg of barium per 50 ml of urine is sufficient, but the supernatant should be checked for completeness of precipitation because of the wide variation in the normal sulfate content of urine. To remove radiobarium and radium, a few milligrams of barium ion is added for each 50 ml of slightly acidified urine, and the barium is precipitated by the addition of excess sodium or ammonium sulfate solution. In either case, the precipitation is made from a hot solution, and the precipitate is digested for 30 minutes before it is centrifuged, washed, and transferred to a planchet for counting.

The precipitation of rare-earth fluorides will separate tri- and tetra-valent actinide elements as well as rare-earth radionuclides rapidly and in a form suitable for counting. Any rare earth can be used as a carrier, but since commercial rare-earth salts are frequently contaminated with thorium or actinium, each batch must be checked for radiochemical purity. The precipitation is carried out by adding hydrofluoric acid to urine previously made 2N in hydrochloric acid and containing 0.5 mg/ml of cerium carrier. A final hydrofluoric acid concentration of 1 to 1.5N is adequate. The precipitate is digested for a few minutes, centrifuged, washed, and transferred to a counting planchet for alpha or beta counting. Chemical recoveries are greater than 95%. The counting yield depends on the weight of carrier used and the characteristics of the radiations.

Technetium co-precipitates with copper sulfide from urine. The urine is made 0.5N in nitric acid, heated to 85 to 95°C, and 5 mg of copper(II) carrier and 100 mg of thioacetamide added per 50 ml of urine. After a 30-minute digestion period, the copper sulfide is separated and counted for beta activity. The chemical recovery is greater than 98%.

Uranium is separated from urine by extraction into di(2-ethylhexyl)-phosphoric acid (HDEHP) dissolved in benzene. The urine is made 0.1N in hydrochloric acid and contacted with one-tenth its volume of 0.2M HDEHP solution for 5 minutes. The extracted activity is washed with 4N hydrochloric acid and then returned to aqueous solution by contacting the organic phase with 12N hydrochloric acid for 5 minutes. The hydrochloric acid solution is evaporated to dryness and counted for alpha activity. Good phase separation must be obtained by centrifuging since

some foam forms at the interface. Plutonium(IV) and thorium also extract, but are not backextracted into concentrated hydrochloric acid. Plutonium(III) extracts, but is removed by the wash solution. The chemical recovery is about 70%. Other organic phosphates can also be used.

Although rapid radiometric methods for uranium are available, uranium is best determined in emergencies by measuring its fluorescence. With older instruments, in which the fluorescence in solid sodium fluoride is measured on a 0.2-ml sample, this method can detect in about 30 minutes about 4  $\mu\text{g}$  of uranium per 1400 ml of urine. This is equivalent to 3.4 pCi of natural uranium and to 340 pCi of enriched uranium if the enriched uranium contains about 1% uranium-234. Measurement of the fluorescence of the uranium-phosphate complex in solution after laser excitation is a factor of two faster and it is a factor of five more sensitive (12).

#### 4 INTERPRETATION OF RESULTS

Some discussion of this topic has been given earlier in this lecture. While this is not strictly in the province of those performing the analytical measurements, the analyst may be involved to some degree. At a minimum, he must be prepared to give the uncertainty associated with the measurement to assist in assessing the possible range in the body content.

The decision to treat (or not to treat) is a medical one, and will be made by physicians experienced in this area. A risk/benefit analysis will

be the basis for the decision, and no generalizations are possible because the benefit is long range and impossible to quantify (3).

The analyst will have a continuous responsibility for continued measurements, after the initial decision is made, to provide the results that measure the effectiveness of the treatment or to check on the validity of the decision not to treat.

## 5 SOURCES OF INFORMATION

The general sources of information on radiochemical analyses are the same as given in lecture 17. Unfortunately, there are no published collections of procedures of which the author is aware that specifically addresses the topic under discussion. Other laboratories may have in-house collections (as Argonne does), and may provide them if requested. Proceedings of two IAEA Symposia contain papers that are applicable, although the publications are relatively old. These are:

Personnel Dosimetry for Radiation Accidents, International Atomic Energy Agency, Vienna, 1965.

Rapid Methods for Measuring Radioactivity in the Environment, International Atomic Energy Agency, Vienna, 1971.

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