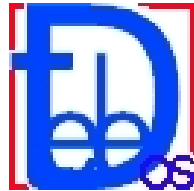**The IAEA-CU-2007-03 world wide open proficiency test on the determination of radionuclides in soil, spinach and water**

**Laboratory's Final Report**
**Laboratory Code: 146 (CuNo: 13949)**
**Total Pages (with cover): 11**

# Reporting Form  F-01

The IAEA-CU-2007-03 World-wide open proficiency test

## Address Information, Laboratory No. 146

Sample requested by: Laboratory No. 146 (13949)

*MR. CHRISTOS ATH. MARAMATHAS*
*NUCLEAR TECHNOLOGY &*
*ENVIRONMENTAL LABORATORY*
*teleDOS LTD*
*APOSTOLOU PAULOU 102*
*GR-20100, KORINTHOS*
*GREECE*

*Tel: +30 697 4867477*
*Fax: +30 2741 084349*
*e-mail: cmaramat[a.t.]teledos.eu*

**Name of Analyst:**     ..... Christos Ath. Maramathas.

**Name of Supervisor:**   ..... There is no Supervisor.

**Collaborators:**       ..... John N. Sarris.

**Please sign and send back to:**

**Abdulghani Shakhashiro**
**Reference Materials Group**
Chemistry Unit, Agency's Laboratories Seibersdorf
A-2444 Seibersdorf - Austria
Tel: + 43 1 2600 28226
Fax: + 43 1 2600 7 28226
Email: a.shakhashiro@iaea.org

**Signature:**          ................................................

**Date:**              ..... Wednesday, 20 February 2008.

I confirm that I checked the data printed on this form.
I agree, that this data will be used for the proficiency test evaluation.

# Reporting Form  F-02

The IAEA-CU-2007-03 World-wide open proficiency test

# Data Sheet, Laboratory No. 146 Sample: IAEA-330

*Activity concentrations [Bq/kg d.m.] for soil and spinach and [Bq/kg] for water (background corrected) on reference date: 15th of October 2007

Table of Results

| Analyte | Activity (*) [Bq/kg d.m.] | Unc (**) [Bq/kg d.m.] |
|---|---|---|
| Sr-90 | | |
| U-234 | | |
| Pu-238 | | |
| U-238 | <11.75 | 0.03 |
| Pu-239+240 | | |
| Am-241 | <21.45 | 0.05 |
| K-40 | 1213.33 | 95.41 |
| Cs-137 | 1164.43 | 86.85 |

Counting date: *2008-02-08*, Counting time [s]: *70000*
Sample mass [g]: *88.52*, Moisture content: *0.24*
Software: *Genie 2000 Basic Spectroscopy (Canberra), FitzPeaks HPGe Gamma Analysis, Microsoft Office Excel*
Efficiency calibration: *Multinuclide standard*
Applied corrections: *moisture content, decay correction, self attenuation, background correction, others: dead time correction*
Sample preparation: *Our laboratory uses a standard procedure for solid pulverised samples' gamma radioactivity measurements. The containers that are used for the handling of the (near 100 gr) samples have a maximum volume capacity of 110 ml and their structural material consists of acid resistant plastic, which is (gamma ray) transparent. The inner diameter of the containers is about 50 mm. Due to their airtight closing and in combination with silicon glue soldering of their lid after the filling, they have the limitation of a single use. Their advantage is that they have a zero radiation background, so they do not increase the measurement's uncertainty*

*and the system's lower limits of detection. The volume of the sample's powder (pressed in these containers if there is a need to reach the 1 gr/ml density), which is used for our standard measurement procedure, is approximately 100 ml\*\*\*. According to this, in order to maintain this volume and to repeat the counting of the same material several times (for better statistics), it is necessary the division of the main sample into many aliquots.*

*For the project in question: Initially it was determined that from the main sample it will be produced only one aliquot of volume about 100 ml. According to this, one blank container was weighed. Afterwards, the aliquot was added to the container, and once again it was weighed in order to quantify the gross weight. The net (wet) mass of the aliquot derived from the subtraction of the mass of the container from the gross mass. Finally, silicon glue was added up to the volume of 110 ml of the container and then it was firmly sealed. After the spectrum's collection the base of the container was cut and the 60% of the sample was extracted and distributed randomly to three aliquots. Their net (wet) mass was measured in the same manner as mentioned above and finally they were dried at 90 oC until constant weight (approximately for 30 hours). Afterwards, the dry aliquots weighed again to estimate their dry mass and lastly the wet to dry mass conversion factor. An electronic laboratory microbalance (Sartorius / accuracy 1E-02gr) was used for the weighing.*

*\*\*\* The final geometry of the aliquots - subsamples is cylindrical, with diameter 50 mm, height 51 mm and the discoid base of the solid powderfull cylinder, during the measurement, is coaxial to detector's active window at a distance approximately 5mm above the detector's active volume. The Standard with which was accomplished the system's efficiency calibration, has the same cylindrical geometry as the subsamples, contains known activities (with their given uncertainties) of Mn-54, Co-57, Co-60, Cs-134, Cs-137, Eu-152 and Am-241 distributed (homogenized) in 1.0 +- 0.1 gr/ml mineral matrix, and its measurement done at the same standard conditions with those used for the measurement of the spinach and soil aliquots. The total activity of the calibration's Standard was comparable with this of the samples'. To avoid detector's contamination, all the containers were cleaned intently before their content's gamma-spectrum collection.*

Separation procedure: *Our Laboratory has not the capability to analyse the samples using the Alpha/Beta Analysis Techniques yet. There is no Separation Procedure due to only Gamma Analysis of the sample.*

Info: *View the spectrums and other data of this Proficiency Test in our site:* http://www.teledos.gr/PT/iaea_cu_2007_03

**Details for Sr-90:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: ; Detection efficiency: ; Background: ; Limit of detection:

**Details for U-234:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: ; Detection efficiency: ; Background: ; Limit of detection:

**Details for Pu-238:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: ; Detection efficiency: ; Background: ; Limit of detection:

**Details for U-238:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: HPGe; Detection efficiency: ; Background: ; Limit of detection:

**Details for Pu-239+240:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: ; Detection efficiency: ; Background: ; Limit of detection:

**Details for Am-241:** Name of tracer: ; Activity added: ; Chemical yield: ; Detector type: HPGe; Detection efficiency: ; Background: ; Limit of detection:

\*\* Combined uncertainty [±1s] is expressed as the square root of the sum of variances of all conceivable sources of uncertainty including the uncertainty of dry mass determination, evaluated using the law of propagation of uncertainty. The combined uncertainty must be expressed in the same unit as the activity.

d.m. : dry mass

# Reporting Form  F-02

The IAEA-CU-2007-03 World-wide open proficiency test

## Data Sheet, Laboratory No. 146 Sample: IAEA-444

*Activity concentrations [Bq/kg d.m.] for soil and spinach and [Bq/kg] for water (background corrected) on reference date: 15th of October 2007

Table of Results

| Analyte | Activity (*) [Bq/kg d.m.] | Unc (**) [Bq/kg d.m.] |
|---|---|---|
| Mn-54 | 63.51 | 4.94 |
| Co-60 | 82.50 | 6.14 |
| Zn-65 | 38.25 | 4.55 |
| Cd-109 | 813.10 | 94.78 |
| Cs-134 | 57.53 | 4.39 |
| Cs-137 | 67.04 | 5.26 |
| Pb-210 | 53.42 | 7.06 |
| Am-241 | 55.47 | 7.36 |

Counting date: *2008-01-28*, Counting time [s]: *70000*
Sample mass [g]: *112.47*, Moisture content: *2.80*
Software: *Genie 2000 Basic Spectroscopy (Canberra), FitzPeaks HPGe Gamma Analysis, Microsoft Office Excel*
Efficiency calibration: *Multinuclide standard*
Applied corrections: *moisture content, decay correction, self attenuation, background correction, others: dead time correction*
Sample preparation: *Our laboratory uses a standard procedure for solid pulverised samples' gamma radioactivity measurements. The containers that are used for the handling of the (near 100 gr) samples have a maximum volume capacity of 110 ml and their structural material consists of acid resistant plastic, which is (gamma ray) transparent. The inner diameter of the containers is about 50 mm. Due to their airtight closing and in combination with silicon glue soldering of their lid after the filling, they have the limitation of a single use. Their advantage is that they have a zero radiation background, so they do not increase the measurement's uncertainty*

*and the system's lower limits of detection. The volume of the sample's powder (pressed in these containers if there is a need to reach the 1 gr/ml density), which is used for our standard measurement procedure, is approximately 100 ml\*\*\*. According to this, in order to maintain this volume and to repeat the counting of the same material several times (for better statistics), it is necessary the division of the main sample into many aliquots.*

*For the project in question: Initially it was determined that from the main sample they will be produced a) one aliquot of volume about 100 ml for gamma analysis and b) four random aliquots for wet to dry mass conversion factor estimation. According to this, five blank containers were weighed. Afterwards, the aliquots were added to the containers, and once again they were weighed in order to quantify their gross weight. The net (wet) mass of the aliquots derived from the subtraction of the mass of each container from the respective gross mass. Finally, for gamma analysis' aliquot, silicon glue was added up to the volume of 110 ml of the container and then it was firmly sealed. The other aliquots were dried at 90 oC until constant weight (approximately for 30 hours). Afterwards, the dry aliquots weighed again to estimate their dry mass and lastly the wet to dry mass conversion factor. An electronic laboratory microbalance (Sartorius / accuracy 1E-02gr) was used for the weighing.*

*\*\*\* The final geometry of the aliquots - subsamples is cylindrical, with diameter 50 mm, height 51 mm and the discoid base of the solid powderfull cylinder, during the measurement, is coaxial to detector's active window at a distance approximately 5mm above the detector's active volume. The Standard with which was accomplished the system's efficiency calibration, has the same cylindrical geometry as the subsamples, contains known activities (with their given uncertainties) of Mn-54, Co-57, Co-60, Cs-134, Cs-137, Eu-152 and Am-241 distributed (homogenized) in 1.0 +- 0.1 gr/ml mineral matrix, and its measurement done at the same standard conditions with those used for the measurement of the spinach and soil aliquots. The total activity of the calibration's Standard was comparable with this of the samples'. To avoid detector's contamination, all the containers were cleaned intently before their content's gamma-spectrum collection.*

Separation procedure: *Our Laboratory has not the capability to analyse the samples using the Alpha/Beta Analysis Techniques yet. There is no Separation Procedure due to only Gamma Analysis of the sample.*

Info: *View the spectrums and other data of this Proficiency Test in our site:* http://www.teledos.gr/PT/iaea_cu_2007_03

\*\* Combined uncertainty [±1s] is expressed as the square root of the sum of variances of all conceivable sources of uncertainty including the uncertainty of dry mass determination, evaluated using the law of propagation of uncertainty. The combined uncertainty must be expressed in the same unit as the activity.

d.m. : dry mass

# Reporting Form  F-02

The IAEA-CU-2007-03 World-wide open proficiency test

## Data Sheet, Laboratory No. 146 Sample: IAEA-445

*Activity concentrations [Bq/kg d.m.] for soil and spinach and [Bq/kg] for water (background corrected) on reference date: 15th of October 2007

Table of Results

| Analyte | Activity (*)<br>[Bq/kg d.m.] | Unc (**)<br>[Bq/kg d.m.] |
|---------|------------------------------|--------------------------|
| Mn-54   | 5.99                         | 0.80                     |
| Co-60   | 7.22                         | 0.92                     |
| Zn-65   | 19.82                        | 2.64                     |
| Cd-109  | <43.78                       | 0.04                     |
| Cs-134  | 6.09                         | 0.81                     |
| Cs-137  | 7.31                         | 0.97                     |
| Pb-210  | <59.76                       | 0.06                     |
| Am-241  | <10.57                       | 0.01                     |

Counting date: *2008-02-11*, Counting time [s]: *70000*
Sample mass [g]: *100*, Moisture content:
Software: *Genie 2000 Basic Spectroscopy (Canberra), FitzPeaks HPGe Gamma Analysis, Microsoft Office Excel*
Efficiency calibration: *Multinuclide standard*
Applied corrections: *decay correction, background correction, others: dead time correction*
Sample preparation: *Our laboratory uses a standard procedure for liquid solutions' gamma radioactivity measurements. The containers that are used for the handling of the (near 100 gr) samples have a maximum volume capacity of 110 ml and their structural material consists of acid resistant plastic, which is (gamma ray) transparent. The inner diameter of the containers is about 50 mm. Due to their airtight closing and in combination with silicon glue soldering of their lid after the filling, they have the limitation of a single use. Their advantage is that they have a zero radiation background, so they do not increase the measurement's uncertainty and the system's lower*

*limits of detection. The volume of the liquid sample, which is used for our standard measurement procedure, is approximately 100 ml\*\*\*. According to this, in order to maintain this volume and to repeat the counting of the same material several times (for better statistics), it is necessary the division of the main sample into many aliquots.*

*For the project in question: Initially, it was desirable to produce three aliquots of volume about 100 ml from the main sample. According to this, three blank containers were weighed. Afterwards, the aliquots were added to the containers, and once again they were weighed in order to quantify the gross weight. The net mass of each aliquot derived from the subtraction of the mass of the container from the gross mass. Finally, silicon glue was added on the containers' lids surfaces and then they were firmly sealed. An electronic laboratory microbalance (Sartorius / accuracy 1E-02gr) was used for the weighing.*

*\*\*\* The final geometry of the aliquots - subsamples is cylindrical, with diameter 50 mm, height 51 mm and the discoid base of the liquid cylinder, during the measurement, is coaxial to detector's active window at a distance approximately 5mm above the detector's active volume. The Standard Solution with which was accomplished the system's efficiency calibration, has the same cylindrical geometry as the subsamples, contains known activities (with their given uncertainties) of Mn-54, Co-57, Co-60, Cs-134, Cs-137, Eu-152 and Am-241 distributed (homogenized) in demineralised water, and its measurement done at the same standard conditions with those used for the measurement of the sample's water aliquots. The total activity of the calibration's Standard was comparable with this of the samples'. To avoid detector's contamination, all the containers were cleaned intently before their content's gamma-spectrum collection.*
Separation procedure: *Our Laboratory has not the capability to analyse the samples using the Alpha/Beta Analysis Techniques yet. There is no Separation Procedure due to only Gamma Analysis of the sample.*

Info: *View the spectrums and other data of this Proficiency Test in our site:* http://www.teledos.gr/PT/iaea_cu_2007_03

\*\* Combined uncertainty [±1s] is expressed as the square root of the sum of variances of all conceivable sources of uncertainty including the uncertainty of dry mass determination, evaluated using the law of propagation of uncertainty. The combined uncertainty must be expressed in the same unit as the activity.

d.m. : dry mass

# Reporting Form  F-03

The IAEA-CU-2007-03 World-wide open proficiency test

## Quality Control Procedure and Combined Uncertainty Estimation, Laboratory No. 146

1.) Quality Control Procedure:

Our laboratory is performing a continuous control of measurement quality and of measurement systems' characteristics. To suit the purposes of these controls, one (2 uCi) Co-60 Point Source and one 3Dimensional low activity mixed nuclide (radioactive) standard source, are used at constant conditions for all QC measurements (constant source's geometry and same distance from detector's window every time). The last (3D) standard consists of Co-60, Cs-137, Eu-152, Pb-210 and Am-241 distributed (homogenized) in 1.0 +- 0.2 gr/ml (extra low background) epoxy matrix at a volume of 100.7 +- 0.5 ml. The aforesaid controls and their respective frequency are as follows:

Monthly (QC 3D standard's and background's spectrum collection live time = 75000 seconds. Co-60 Point Source's spectrum collection live time = 5000 seconds at a distance 25 cm from detector window):
   a. Peaks' Position check for the 3D standard at the following occurring photon energies (in keV): 46.54 , 59.54 , 121.78 , 244.70 , 344.28 , 661.66 , 778.90 , 867.39 , 964.13 , 1112.12 , 1173.24 , 1332.50 , 1408.01 .
   b. Energy Resolution (FWHM) and Peak Shape check for the 3D standard at the above photon energies. The Total Count Rate is < 850 counts/s, the shaping time for all measurements is 2 us and for the FWHM at all peaks stands that FWHM > 4 channels.
   c. Peak to Compton ratio check for the 3D standard at the photon energies: 661.66 keV (of Cs-

137) and 1332.50 keV (of Co-60). The first Peak to Compton ratio is obtained by dividing the height of the 661.66 peak by the average Compton Plateau between 430 and 460 keV, and the second by dividing the height of the 1332.50 peak by the average Compton Plateau between 1036 and 1096 keV.
   d. Decay corrected count rates' check for the above Energy Peaks of the 3D standard.
   e. Check using the Decay Corrected Activities for the content radioisotopes in the 3D standard (obtained) from its Full (Spectrum) Analysis' Report. The analysis is performed by the software and the subroutines, routinely used in our laboratory for all the samples. The efficiency calibration that is needed for this control is performing half-yearly by another similar standard.
   f. HPGe Relative Efficiency (r.eff) to NaJ(Tl) check. The result for this control is given by the equation: r.eff=N/(e*T*A) , where N = Measured number of counts in 1332.50 keV Full Energy Peak of Co-60 Point Source, e = 1.2E-03 (Efficiency of NaJ detector), T = 5000 s (spectrum's collection live time), A = Activity of the source.
   g. Total count rate check using Background's Spectrum. The energy threshold for this control is at 39 - 40 keV.
   h. Peaks' Position check for the 63.29 and 92.50 keV peaks of natural Th-234 (in secular equilibrium with the U-238 of HPGe detector's shielding) and the 1460.83 keV peak of natural K-40, which are occurring in every Background's Spectrum.

Before (with the use of 3D standard) and after (with the use of background's spectrum) every samples'

set* measurement (QC 3D standard's spectrum collection live time = 10000 seconds):
    a. Peaks' Position check for the 3D standard as mentioned above.
    b. Energy Resolution (FWHM) and Peak Shape check for the 3D standard as mentioned above.
    c. Peak to Compton ratio check for the 3D standard as mentioned above.
    d. Total count rate check using Background's Spectrum as mentioned above.
    e. Peaks' Position check using Background's Spectrum as mentioned above.

Before every sample's measurement (only for long spectrum collection time per sample > 75000 seconds) (QC 3D standard's spectrum collection live time = 3000 seconds):
    a. Peaks' Position check for the 3D standard as mentioned above.
    b. Energy Resolution (FWHM) and Peak Shape check for the 3D standard as mentioned above.

From the spectrum of every measured sample:
    a. Position check for occurring single well known or expected peaks.
    b. Energy Resolution (FWHM) and Peak Shape check at the aforesaid peaks of the sample's spectrum.

Always after HPGe cooling periods or extraordinary circumstances like voltage supply failure (QC 3D standard's spectrum collection live time = 75000 seconds. Co-60 Point Source's spectrum collection live time = 5000 seconds at a distance 25 cm from detector window):
    a. Peaks' Position check for the 3D standard as mentioned above.
    b. Energy Resolution (FWHM) and Peak Shape check for the 3D standard as mentioned above.
    c. Peak to Compton ratio check for the 3D standard as mentioned above.
    d. Decay corrected count rates' check for the 3D standard as mentioned above.
    e. Check using the Decay Corrected Activities

for the content radioisotopes in the 3D standard as mentioned above.
    f. HPGe Relative Efficiency to NaJ(Tl) check as mentioned above.

The results of all the (periodical) checks are logged in our system's database and then are plotted in graphs to check system's stability through time (x-axis) (long term QC). In case of unexpected changes at these results / graphs for multiple times, further investigation is taking place.

* One samples' set measurement composed of i) a background's spectrum collection for 75000 seconds, ii) the spectrums' collection for a number (in accordance with a logical grouping philosophy) of samples that belong to a project and iii) the respective standards' spectrum collection for a suitable time which is similar to the samples' spectrum collection time. [e.g.: the three samples of this Proficiency Test, the three respective (with similar matrix and geometry) standards (should be used if the system was not calibrated) and the background's spectrum collection, constitute one samples' set.]
** According to the long term QC of teleDOS laboratory, for the last six months there is an increment at the total count rate of Background's Spectrum and at the energy resolution (FWHM) for all energies by a factor of 2.1 (approximately). The first increment caused by extraordinary electronic noise started after some power network problems and inserts to all spectrums a peak with a centroid at about 41 keV. This (nearly random) noise peak makes impossible the accurate measurement of radionuclides with photon emissions within the energy range 40 – 100 keV. Due to this, we expect some acceptance's problems with our results for Pb-210, Am-241, Cd-109 and U-238. We are trying to fix our power network problems to minimize the aforesaid noise. The second increment (FWHM*2.1) caused gradually through time and will be fixed by the HPGe detector's (high vacuum) regeneration in the near future.

## 2.) Uncertainty components, gamma analysis:

All the measurement results we extract from an analysis procedure are always provided with their combined standard uncertainty at one sigma level. This combined standard uncertainty is expressed as the square root of the sum of variances of all conceivable sources - components of uncertainty, evaluated using the law of propagation of uncertainty. The aforesaid uncertainty components about the measurements of this Proficiency Test, as for the other gamma analysis measurements are carried out in our laboratory, are as follows:

Sources of Uncertainty:

    a. Counting statistics for every useful peak (peak area measurement).
    b. Spectrum's background counting statistics for the above peaks (background area measurement).
    c. Environmental background counting statistics at the position of the above peaks (env. bkg. subtraction).
    d. Counting efficiency for the same area / position of the spectrum as above.
    e. Dead time.
    f. Half life.
    g. Decay factor.
    h. Photon emission probability.
    i. Multiplets deconvolution.
    j. Standard source activity.
    k. Source - Detector geometry (for the same sample's geometry and matrix there are only small variations of samples' volume).
    l. Sample's mass measurement.
    m. Sample's wet to dry mass conversion factor produced by sample's aliquots (wet and dry) mass measurements (only for solid samples).
    n. Sample aliquots' radionuclides heterogeneity contribution to the uncertainty (if given like the proficiency test in question).

## 3.) Uncertainty components, alpha/beta analysis:

Our Laboratory has not the capability to analyse the samples using the Alpha/Beta Analysis Techniques yet.

_____**End of Report**