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Soil Sampling Intercomparison Exercise by Selected Laboratories of the ALMERA Network

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The Agency's Statute was approved on 23 October 1956 by the Conference on the Statute of the IAEA held at United Nations Headquarters, New York; it entered into force on 29 July 1957. The Headquarters of the Agency are situated in Vienna. Its principal objective is "to accelerate and enlarge the contribution of atomic energy to peace, health and prosperity throughout the world".

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Soil Sampling Intercomparison Exercise by Selected Laboratories of the ALMERA Network

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FOREWORD

The IAEA's Seibersdorf Laboratories in Austria have the programmatic responsibility to provide assistance to Member State laboratories in maintaining and improving the reliability of analytical measurement results, both in radionuclide and trace element determinations. This is accomplished through the provision of reference materials of terrestrial origin, validated analytical procedures, training in the implementation of internal quality control, and through the evaluation of measurement performance by the organization of worldwide and regional interlaboratory comparison exercises.

The IAEA is mandated to support global radionuclide measurement systems related to accidental or intentional releases of radioactivity in the environment. To fulfil this obligation and ensure a reliable, worldwide, rapid and consistent response, the IAEA coordinates an international network of analytical laboratories for the measurement of environmental radioactivity (ALMERA). The network was established by the IAEA in 1995 and makes available to Member States a world-wide network of analytical laboratories capable of providing reliable and timely analysis of environmental samples in the event of an accidental or intentional release of radioactivity.

A primary requirement for the ALMERA members is participation in the IAEA interlaboratory comparison exercises, which are specifically organized for ALMERA on a regular basis. These exercises are designed to monitor and demonstrate the performance and analytical capabilities of the network members, and to identify gaps and problem areas where further development is needed.

In this framework, the IAEA organized a soil sampling intercomparison exercise (IAEA/SIE/01) for selected laboratories of the ALMERA network. The main objective of this exercise was to compare soil sampling procedures used by different participating laboratories.

The performance evaluation results of the interlaboratory comparison exercises performed in the framework of the ALMERA network are not the same for those laboratories which participate as ALMERA members.

The IAEA wishes to thank the participating laboratories to this intercomparison exercise and all the contributors to drafting and review of this report. The IAEA officer responsible for this publication was U. Sansone of the Agency's Laboratories, Seibersdorf, Austria.

CONTENTS

1. BACKGROUND

The soil sampling intercomparison exercise took place from 14 to 18 November 2005 in an agricultural area qualified as a reference site in the frame of the SOILSAMP international project, funded and coordinated by the Italian Environmental Protection Agency (ISPRA) and aimed at assessing the uncertainty associated with soil sampling in agricultural, semi-natural, urban and contaminated environments [1]. The term reference site was recently defined in the International Union of Pure and Applied Chemistry (IUPAC) Recommendations (2005), as "an area, one or more of whose element concentrations are well characterised in terms of spatial and temporal variability"[2]. The definition includes 'spatial and temporal variability' that replace the terms 'homogeneity and stability' used in the context of reference material. Homogeneity and stability are mandatory requirements for a reference material, as stated by the ISO Guide 35 [3], because the measurand is 'quantity intended to be measured' [4] such as a mass fraction of an element in a given matrix and the users should demonstrate the ability of a measurement method in obtaining a well defined quantity value. In the case of 'reference site', the measurand is represented by the mass fraction of an element in a sampling target including its spatial and temporal variability and the users are required to demonstrate the ability of their sampling strategy to obtain that quantity value.

The reference site is located in the north-eastern part of Italy (Pozzuolo del Friuli, Udine), in the research centre belonging to the Agenzia Regionale per lo Sviluppo Rurale del Friuli Venezia Giulia (ERSA). The reference site is characterised in terms of spatial/temporal variability of trace elements. The trace elements present at the reference site are of natural and anthropogenic origins.

Soil is the final receptor of trace elements, including radionuclides and organic pollutants dispersed in the environment [5]. In the long term after deposition, the behaviour of long-lived radionuclides in soil can be expected to be similar to that of some stable trace elements and the distribution of these trace elements in soil can simulate the distribution of radionuclides. Trace elements in soil, including radionuclides, are mostly associated with finer particle-size fractions [5, 6, 7, 8, 9, 10]. Positive correlation of most radionuclides was observed with metals: Fe, Zn and Mn with ^{226}Ra and ^{232}Th [11] while Sc is positively correlated with Fe (similar soil distribution pattern).Furthermore, sampling for radionuclides in the environment is not unlike sampling for other attributes of environmental media, as it was for the reference site characterization. In addition, soil sampling procedures for radionuclides derive from techniques used in agriculture and engineering [12]. For all these reasons, the reference site characterized in term of trace elements can be used to compare the soil sampling strategies developed for radionuclide investigations by the ALMERA laboratories.

On this basis, the radionuclides considered in planning the sampling exercise in the frame of the Soil Sampling Intercomparison Exercise (IAEA/SIE/01), were those that require radiochemical separation (^{90}Sr, ^{240}Pu, ^{241}Am, ^{238}U) and a test portion, defined as the "Quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample" [2], ranging from 10 to 50 g, depending on the activity concentration of the radionuclide.

2. OBJECTIVES OF INTERCOMPARISON EXERCISE

The objective of the exercise was to compare soil sampling strategy/pattern and sampling techniques utilized in the ALMERA network (Appendix I) in the case of the determination of several radionuclides mean values in an agricultural area of about 10,000 m^2. Sampling strategy/pattern is defined as "The result of the selection of the sampling points within a sampling site" and sampling techniques are defined as "All appropriate procedures and sampling devices used to obtain and describe samples of soil, either in the field or during transportation and in the laboratory" [2]. The sampling exercise was organized taking into account that ^{90}Sr, ^{240}Pu, ^{241}Am, ^{238}U could be the radionuclides to be considered, because their analysis require a test portion ranging from 10 to 50 g, depending on the activity concentration of the radionuclide.

3. PARTICIPANTS OF THE INTERCOMPARISON EXERCISE

Considering that collaborative field studies require considerable organizational efforts [15, 16], ten ALMERA Institutions were selected to participate in the sampling exercise. Experts from the IAEA, the Italian Environmental Protection Agency and ERSA provided assistance during the sampling exercise. In the following the participants to the soil sampling intercomparison exercise are reported:

Chang Byung-Uck	:	Korea Institute of Nuclear Safety (Republic of Korea)
Aguirre Jaime	:	Comision Nacional de Seguridad Nuclear y Salvaguardias (Mexico)
Al-Masri Mohammad S.	:	Atomic Energy Commission of Syria (Syrian Arab Republic)
Derkach Grygoriy	:	Ukrainian Hydrometeorological Institute (Ukraine)
Fathivand Khalili Mir Ali Asghar	:	Iranian Nuclear Regulatory Authority (Islamic Republic of Iran)
Grubel Stefan	:	NPP Mochovce (Slovakia)
Kanivets Volodymyr	:	Ukrainian Hydrometeorological Institute (Ukraine)
Korun Matjaz	:	Josef Stefan Institute (Slovenia)
Kozar-Logar Jasmina	:	Josef Stefan Institute (Slovenia)
Ladygiene Rima	:	Radiation Protection Centre of Lithuania (Lithuania)
Lee Dong-Myung	:	Korea Institute of Nuclear Safety (Republic of Korea)
Tarjan Sandor	:	National Food Investigation Centre (Hungary)
Tirollo Taddei Maria Helena	:	Brazilian Nuclear Energy Agency (Brazil)
Varga Beata	:	National Food Investigation Centre (Hungary)
Vasarab Julius	:	NPP Mochovce (Slovakia)

4. REFERENCE SITE CHARACTERIZATION

4.1. Site location and properties

The reference site is located at Pozzuolo del Friuli (North-Eastern Italy). Table 1 shows the main pedo-chemical properties of the site [14].

TABLE 1. PEDOLOGICAL AND CHEMICAL CHARACTERISTICS OF THE SAMPLING REFERENCE SITE

Soil parameters	**Mean**	**Median**	**Coefficient of variation (%)**
Sand (%)	37.6	37	12
Silt (%)	46.7	48	9
Clay (%)	15.7	16	16
Water content (%)	15.7	16.2	8
Particle size >2mm (%)	13	12	31
pH	7.7	7.7	0.8
Carbonate (%)	5.7	6	38
Organic carbon (%)	1.2	1.2	9
Total nitrogen (%)	0.1	0.1	8
Phosphorus Olsen (mg kg^{-1})	42.5	39	67
CEC (cmol(+) kg^{-1})	16.4	16.3	8
Exchangeable calcium(mg kg^{-1})	3319	3300	11
Exchangeable magnesium (mg kg^{-1})	544.7	550	16
Exchangeable potassium (mg kg^{-1})	78.4	74	25

The spatial variability (distribution) of element mass fractions was assessed, performing a *reference sampling,* as adapted from the sampling scheme proposed by Desaules et al. [13] and described in more details by Barbizzi et al [14]. The site (10,000 m^2) was subdivided into 100 sub-areas (cells), each of 10 m × 10 m (Figure 1). One hundred composite samples (pooling 25 increments each), one for each sub-area, were obtained.

The sampling was performed within the ploughed layer (0-40 cm), where the content of trace elements can be considered uniform. Furthermore, two cells were sampled again and the resulting 25 samples per cell were analyzed separately, in order to verify the spatial variability within-cell. Figure 1 shows the composite sample reported in the middle of the cells and the single samples collected for the reference sampling.

The reference sampling was carried out by a single sampling team.

4.2. Test samples collection and characterization

The test sample, defined as "sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis" [2] was obtained from the laboratory sample with the following procedure: i) mixing and stone hand-picking; ii) drying, iii) sieving, homogenizing and splitting (sample mass reducing) and iv) milling. A detailed description of the sample pre-treatment procedure is reported by Barbizzi et al. [14].

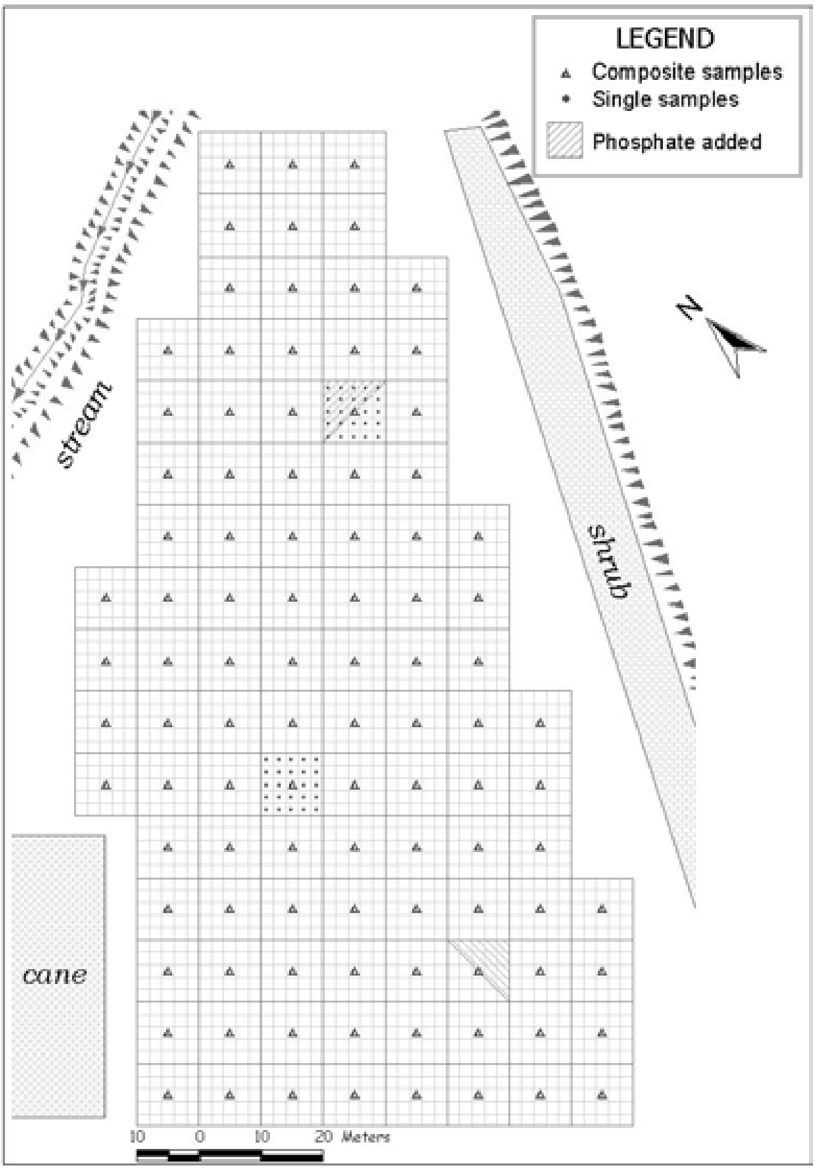

FIG. 1. Location of composite and single samples collected for the reference sampling

The k_0-method of instrumental neutron activation analysis **(**k_0-INAA) was used at the Jožef Stefan Institute, Ljubljana, Slovenia, for measurements of As, Fe, Sc, and Zn in the soil samples. These elements show a similar environmental behaviour of many radionuclides. More details about k_0-INAA and the relevant nuclear data are reported in Jaćimović et al. [17].

Test portions, defined as the quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample [2], of about 200 mg (one for each test sample) were measured. The test portions were sealed into pure polyethylene ampoules (SPRONK System, Lexmond, The Netherlands), irradiated, transferred to clean polyethylene vials and subsequently counted on calibrated coaxial HPGe detectors connected to a multi-channel analyzer (MCA). k_0-INAA quality control was performed by using the reference material IAEA Soil-7. k_0-INAA technique allows achieving high precision levels and requires little or no sample processing before the analysis. A single laboratory was responsible for all the analyses.

Normality of data distribution (coefficient of skewness, kurtosis, Kolmogorov-Smirnov test) of the characterization data was assessed using S-Plus 6 for Windows [18]. The reference value for each trace element was assessed using the mean and the standard deviation of the mean.

Table 2 reports the reference values for the elements used in this intercomparison with their expanded uncertainty (U). U is calculated as the experimental standard deviation of the mean values (100 composite sample collected) multiplied for a coverage factor k=2.

To the overall uncertainty contributes: spatial variability over the site, sampling (strategy, sampling device, sampler), sample preparation (from the primary sample to the test sample) and analysis.

TABLE 2. REFERENCE VALUE ASSIGNED TO EACH ANALYTE OF THE REFERENCE SAMPLING SITE

As	**Fe**	**Sc**	**Zn**
	mg kg^{-1}		
10.6 ± 0.2	25570 ± 565	8.6 ± 0.2	91.8 ± 2.1

4.3. Spatial and temporal variability

Figures 2-5 show the patterns of the spatial variability of the elements of interest. The dimensions of the dots are proportional to the mass fraction values of the elements. Y and X axes represent the position of each cell in the reference site (meters).

Temporal variability was assessed carrying out a new sampling in selected cells three years later the reference sampling. Sampling and measurement procedures were the same as those applied during reference site characterization.

Temporal variability of As, Sc, Fe and Zn are shown in Figure 6. The values of element mass fractions related to soil samples collected in different time (2001 and 2004) into 2 cells are in agreement according to the criterion by which the difference from two results is less than two times the combined standard uncertainty [19, 20]. The element mass fractions do not vary in a way to affect any comparison between samples collected in different time. Assuring a suitable temporal variability, sampling intercomparison exercises can be properly carried out after the reference site characterization.

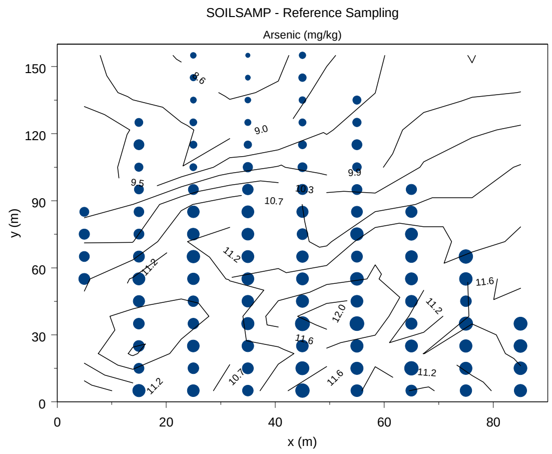

FIG. 2. Arsenic mass fraction in the composite samples of the reference sampling site

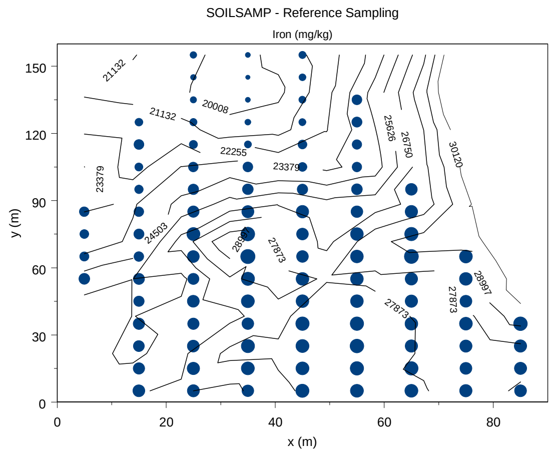

FIG. 3. Iron mass fraction in the composite samples of the reference sampling site

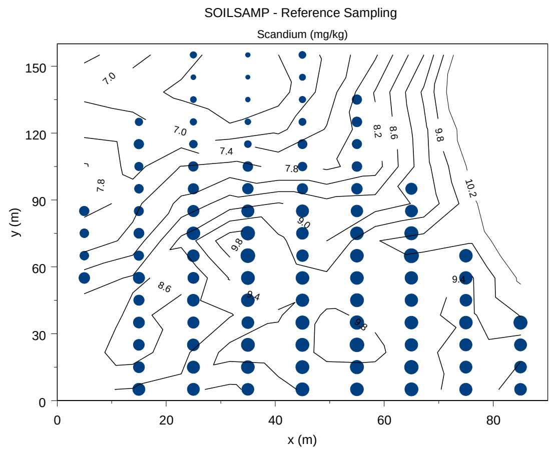

FIG. 4. Scandium mass fraction in the composite samples of the reference sampling site

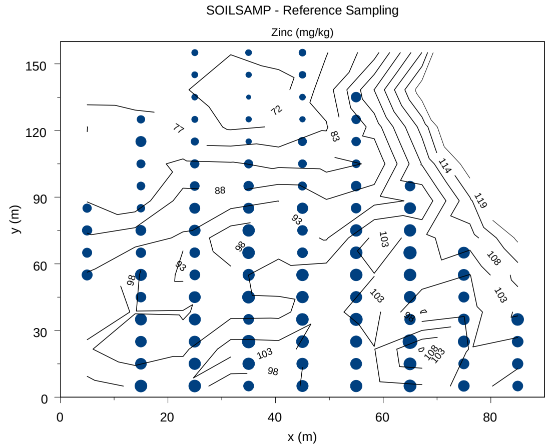

FIG. 5. Zinc mass fraction in the composite samples of the reference sampling site

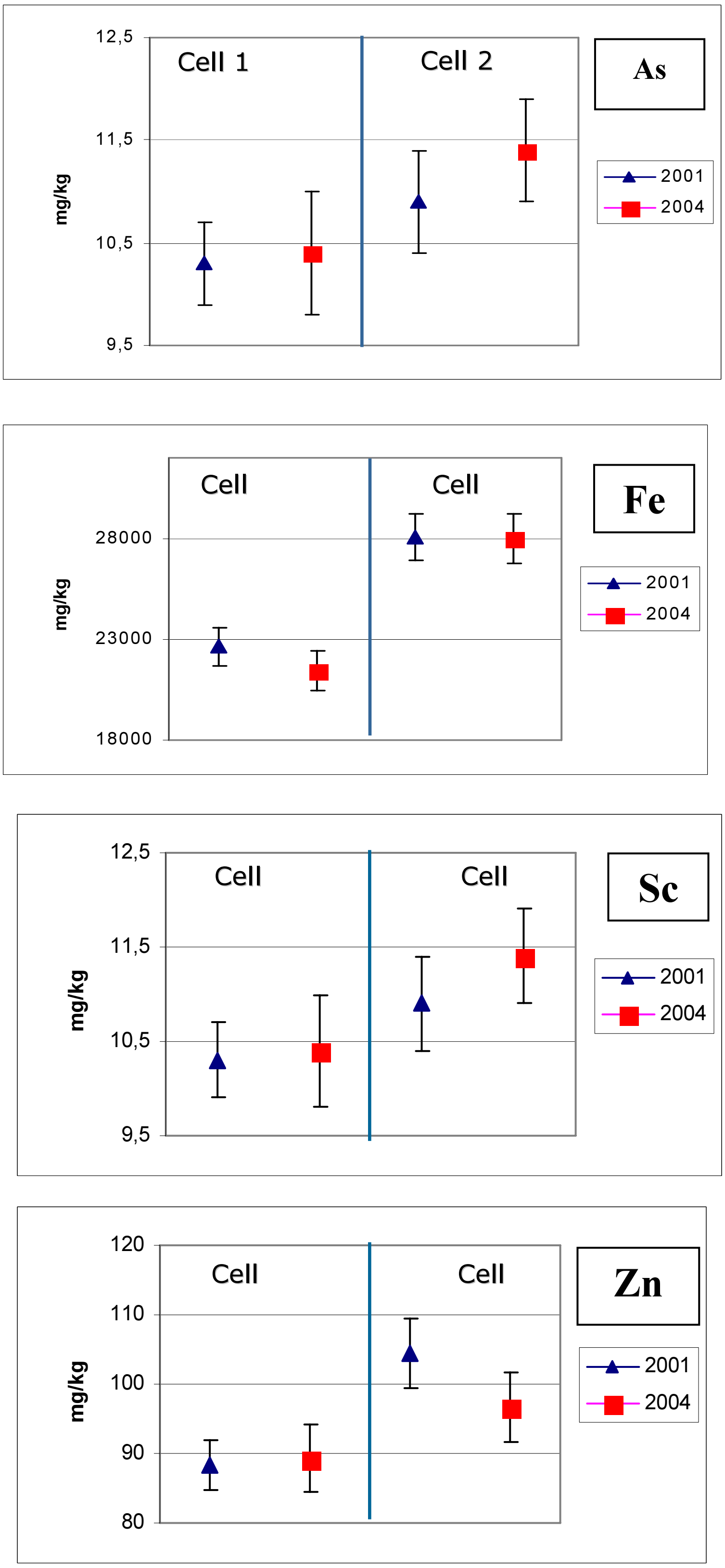

FIG. 6. As, Fe, Sc and Zn temporal variability of the reference site.

5. ORGANIZATION AND INTERCOMPARISON DESIGN

Each participant to the soil sampling exercise was asked to use their own strategy and procedures (sampling strategy/pattern, type of sampling design, sampling device, sampling depth, etc.) for the assessment of the mean values of trace elements (metals) on the reference site.

The limiting conditions given by the organizers to the laboratories were:

- 15 laboratory samples (laboratory sample is defined as *the sample or sub-sample sent to or received by the laboratory* [2]) as the maximum number of samples to be delivered to the IAEA for treatment and analysis;

- 1 liter as the maximum volume of each sample;

- 3 hours as the maximum time period to carry out the sampling.

The Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the IAEA's Seibersdorf Laboratory has established a web-based interface to the project database to collect information from the participants. Each participant provided:

- a description of the sampling devices used and their protocol applied during the intercomparison exercise (description of devices, number of samples, size of each sample, depth of sampling, sampling locations, etc.) (Appendix II);

- a description of its own methodology to prepare the Test Portion from each of the samples collected during the sampling exercise;

- their own methodology to estimate the mean value of several analytes in a sampling exercise (Appendix III).

At the end of the sampling exercise, a meeting with the sampling groups, the IAEA team and the Italian Environmental Protection Agency (ISPRA) experts took place at the Abdus Salam International Centre for Theoretical Physics (ICTP, Trieste, Italy) for final comments and considerations on the Soil Sampling Intercomparison Exercise (IAEA/SIE/01). During the sampling exercise, the sampling of each participant team was documented photographically and a short video was recorded for each procedure. The videos were processed on the spot and presented to the participants for discussion during the meeting. Each participant presented and commented on his/her procedure briefly while showing the video.

6. METHODS AND STRATEGIES APLLIED IN THE SAMPLING EXERCISE

During the soil sampling exercise the participant laboratories adopted strategies belonging to the following three main classes (Table 3):

- systematic by transects, triangular grid and diagonals;

- stratified random;

- non-systematic/irregular.

Table 3 reports also the sampling devices used during the exercise. All samplings were carried out within the ploughed layer (0-40 cm) at a sampling depth ranging from 5 to 25 cm. Table 4 reports the number of laboratories that have collected composite/single sample, the minimum and the maximum number of samples collected and the minimum and the maximum of increments collected for each composite sample. The weight of composite samples ranges from 850 to 2000 g, while the weight of single samples ranges from 50 to 1900 g (Figure 7). Some laboratories performed part of the sample pre-treatment in the field by stone hand-picking, sieving, coning-quartering, homogenization, reducing the volume, till a maximum sample volume of one litre (laboratory sample).

TABLE 3. FREQUENCY OF SAMPLING STRATEGIES AND SAMPLING DEVICES ADOPTED BY THE LABORATORIES

Sampling strategies	**Number of Laboratories**
Systematic	5
Stratified Random	2
Non-systematic - Irregular	3
Sampling devices	
Shovel-spatula-spade	5
Sample ring	1
Corer	4

TABLE 4. TYPE AND NUMBER OF SAMPLES COLLECTED BY THE LABORATORIES

	Type of sample	
	Composite	**Single**
Number of laboratories	5	5
Number of samples	9-14	13-15
Number of increments	4-10	-

The application of the different sampling strategies and techniques in the case of the sampling intercomparison exercise influences other operational aspects, such as the necessary number of the samplers (sampler is defined as *person or group of persons carrying out the sampling procedures at the sampling point)*[2] and the time spent for sampling (Figure 8).

Lab 1-Republic of Korea collected 10 composite samples with 10 increments each, leading to a total of 100 increments. Lab 2-Brazil carried out sieving, coning and quartering directly in the field. For these reasons, Lab 1 and Lab 2 spent more than 3 h in their sampling activities. In both cases, only one person carried out the sampling activity. Certainly the presence of two samplers would have reduced the time and the sampling effort.

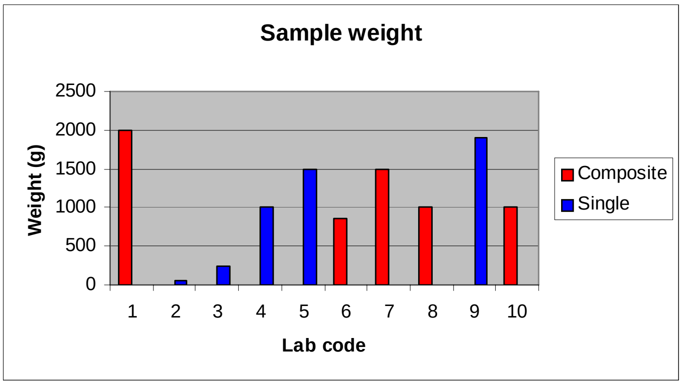

FIG.7. Average sample weight of the samples collected by the laboratories

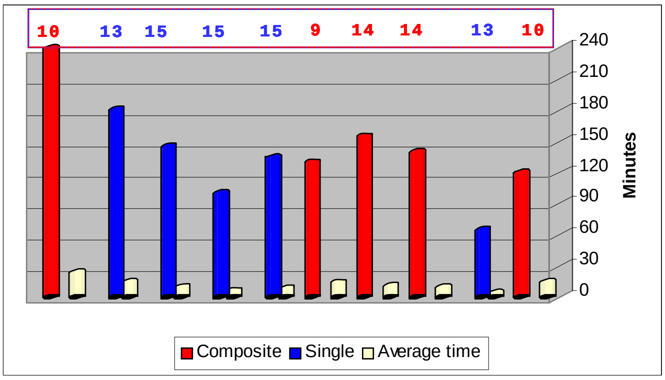

FIG. 8. Time of the sampling. The number associated to each bar represents the numbers of laboratory samples delivered by the laboratories to IAEA

Three others sampling teams (Lab 3-Slovenia, Lab 7-Mexico and Lab 8-Islamic Republic of Iran) needed most of the time available. In the first case, the time is associated to two particular phases of the sampling: i) the critical (and potentially dangerous) insertion of the heavy sampling corer into the soil hammering the devices on the top and ii) the accurate selection and cutting of the slices of soil from the sample core. For the Mexican sampler, the significant time requested is mainly due to the preliminary demarcation of the sample location by a circle and the high number of composite samples (14) collected. Similar reasons justify the time requested by the Iranian sampler. The fastest sampling teams were the Lab 9-Hungary and the Lab 4- Lithuania: the first composed by two persons and not adopting any sample pre-treatment in the field, the second, composed by a single sampler using a very simple sampling techniques.

Time and number of samples can obviously impact directly on the final cost of sampling and analysis. However, some logistical restrictions must be taken into account in order to better interpret the different operational aspects of the sampling procedures adopted. Some teams, mainly those coming from not-European institutions, were obliged to reduce the number of the samplers, so that their usual sampling procedures are a bit modified. The teams were composed at most by two persons and in also in this better condition some samplers declared having worked under a small stress and with time just sufficient. The worse cases of one-man team do not reflect the usually recommended practice for sampling in the field.

To rule out variability eventually caused by different soil sample preparation techniques and by different analytical laboratories, a single laboratory, the IAEA's Chemistry Unit, reduced the laboratory samples received from each participant to test samples. The test portions from each test sample were analysed by a single laboratory, the Atomic Energy Commission of Syria, using a technique (Instrumental neutron activation analysis - INAA) that can achieve high precision levels and requires little or no sample processing prior to analysis. IAEA Soil-7 reference material was used as quality control material for INAA measurements of As, Sc, and Zn in the soil samples. IAEA Soil-7 does not report the certified value for Fe, but only an informative value.

7. CRITERIA FOR EVALUATION OF RESULTS

The participants' data were evaluated according to ISO 13528:2005 and ISO/IEC 17025 [21, 22]. The performance of each participating institution was evaluated using the mean values of the measurands, assessed by the methodology provided by the participants, or in the case in which the participants did not report any suggestion for mean value assessment, using the arithmetic averages. The raw analytical results are reported in Appendix IV. In Appendix V the summary statistics (mean value, standard deviation and coefficient of variation) is included

Shapiro Wilk and Kolmogorov-Smirnov tests [23] were used to check the normality of the distribution of the mean values of all participating institutions. Grubbs's test was used to identify outliers. The results of the tests are summarized in Appendix VI.

According to ISO 13528:2005 [21], the participants sampling performance was evaluated through the bias and the ζ scoring method using the following equations:

$$D = X_{par} - X_{ref} \quad (1)$$

$$\zeta = \frac{X_{par} - X_{ref}}{\sqrt{u_{par}^2 + u_{ref}^2}} \quad (2)$$

Where:

D = is the difference between the assigned values and laboratory's mean value

X_{ref} = Reference value assigned to each measurand of the reference sampling site

X_{par} = Participant's mean value for each measurand

ζ= score of the participant

u_{ref}= Experimental standard uncertainty for each measurand assigned to the reference site, expressed as standard deviation of the mean

u_{par}= Participant's experimental standard uncertainty for each measurand

The critical values related to the parameters used for evaluating the laboratory's performance are shown in Tables 5 and 6. The robust standard deviation calculated for the interlaboratory comparison exercise is in Table 7.

TABLE 5. SCALE OF ACCEPTABILITY FOR BIAS (D). $\hat{\sigma}$ IS THE ROBUST STANDARD DEVIATION OF THE SOIL SAMPLING INTERLABORATORY COMPARISON

$\|D\| \leq 2\hat{\sigma}$	*Suitable strategy*
$2\hat{\sigma} < \|D\| \leq 3\hat{\sigma}$	*Warning/Questionable*
$\|D\| > 3\hat{\sigma}$	*Action*

TABLE 6. SCALE OF ACCEPTABILITY FOR SCORE (ζ)

$\|\zeta\| \leq 2$	*Suitable strategy*
$2 < \|\zeta\| \leq 3$	*Warning/Questionable*
$\|\zeta\| > 3$	*Action*

TABLE 7. ROBUST STANDARD DEVIATION ($\hat{\sigma}$) OF THE SOIL SAMPLING INTERLABORATORY EXERCISE

Measurand	**Robust Standard Deviation** (mg kg^{-1})
As	0.7
Fe	1565
Sc	0.4
Zn	5

8. RESULTS OF THE INTERCOMPARISON

In Figures 9-12 the graphs of the bias values attributed to each laboratory are shown. The bias values are assessed versus the $2\hat{\sigma}$ and 3 $\hat{\sigma}$ values with $\hat{\sigma}$ equals to the robust standard deviation of the intercomparison exercise. $\hat{\sigma}$ values have been calculated according to ISO 13528:2005, [21] Annex C-Algorithm A. The green and red lines represent respectively the $2\hat{\sigma}$ and $3\hat{\sigma}$ values. Absolute bias values are acceptable for values $\leq 2\hat{\sigma}$, while action signal, corresponding to sampling strategies to be corrected, is given by bias absolute value > $3\hat{\sigma}$.

In general, the resulted participants' mean values do not lead to critical bias values. At most 10% of the participants biases (Lab 9-Hungary for Fe, Lab 8-Islamic Republic of Iran for Sc and Lab 6-Ukraine for Zn) is higher than $3\hat{\sigma}$ and in the case of Lab 6-Ukraine this value is clearly associated to an outlier measurement result for zinc. 60-90% of the bias values are $\leq 2\hat{\sigma}$ corresponding to suitable sampling strategies. On the basis of these results significant differences between the laboratories' mean values, due to different sampling strategies and techniques used in the intercomparison exercise, are not identified

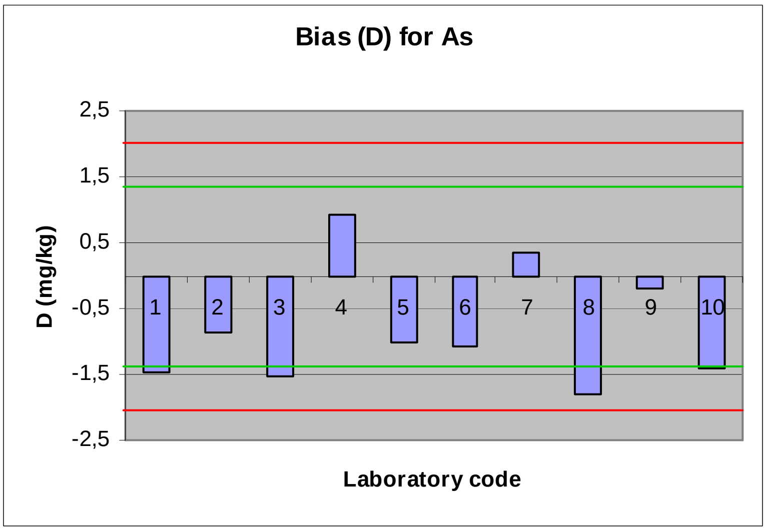

FIG. 9. Bias values (D) for arsenic.

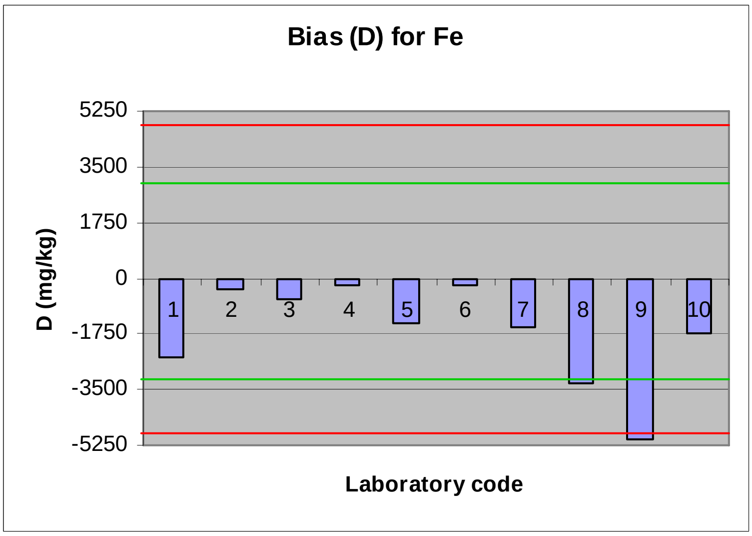

FIG. 10. Bias values (D) for iron.

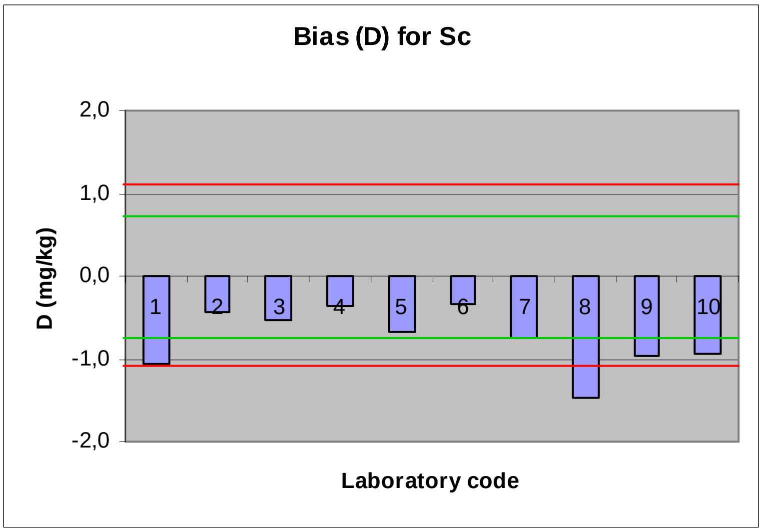

FIG. 11. Bias values (D) for scandium.

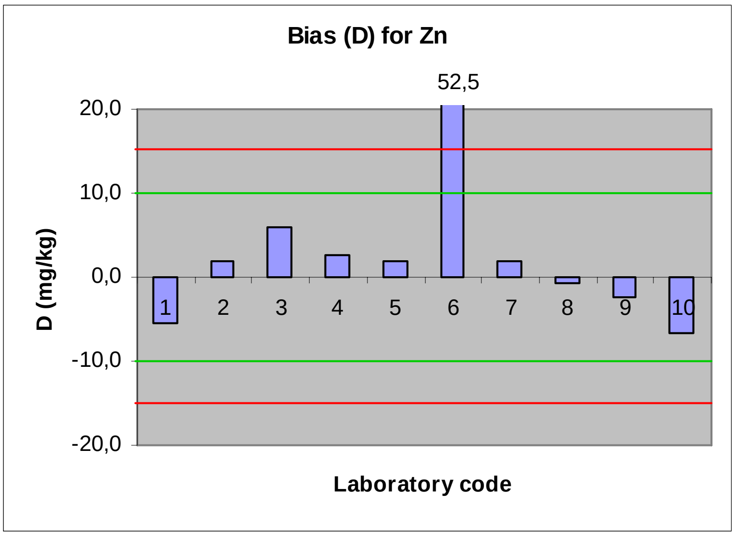

FIG. 12. Bias values (D) for zinc.

A confirmation of this behavior can be done comparing for the measurand of interest and for each laboratory the mean value calculated for the intercomparison exercise IAEA/SIE/01 and the corresponding mean value calculated simulating the same sampling strategies on the data set of the reference sampling. The comparison, in analogy with the criterion used for the assessment of the temporal variability (see the Chapter 'Description of the reference site used for the soil sampling intercomparison exercise') is done applying the criterion of Equation 3 by which the difference Δ_m of the laboratory's mean value (e.g. X_{Lab_1}) and the corresponding simulated laboratory's mean value (e.g. $X_{Lab\_1\_simulation}$) is less than two times the associated combined standard uncertainty $u\Delta_m$ (expanded uncertainty $U\Delta_m$) [19, 20]. $u\Delta_m$ is calculated for all the elements from the uncertainty of the laboratory's mean values and the uncertainty of the simulated value.

$$X_{Lab\_1} - X_{Lab\_1\_simulation} = \Delta_m < U\Delta_m \quad (3)$$

For As, Fe, Sc and Zn all the laboratories pass the criterion stated above, with the exception of Lab 6-Ukraine for Zn, due to an outlier value (Table 8). As applying different sampling strategies on the reference sampling data set the criterion is always respected by all the laboratories, this means that for this kind of reference site the sampling strategy chosen does not influence the final measurements results in terms of mean value.

Moreover, the figures show a general underestimation of the mean values obtained within the framework of IAEA/SEI/01 in respect of the assigned values. Considering the under control temporal variability previously shown, this behaviour, mostly marked for scandium, can be explained by a sampling which was randomly performed by the laboratories in different location in respect of the reference sampling. However, the underestimation observed does not lead in most of the cases bias values higher than $3\hat{\sigma}$.

TABLE 8. APPLICATION OF ISO GUIDE 33 [19] CRITERION FOR THE ASSESSMENT OF THE EQUIVALENCE OF THE SAMPLING STRATEGIES

Lab code	**Criterion ISO Guide 33**	**As**	**Fe**	**Sc**	**Zn**
1 (Republic of Korea)	Δ_m	1.1	2098	0.9	3
	$U\Delta_m$	3.3	11614	4.1	42
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
2 (Brazil)	Δ_m	0.9	285	0.1	-2
	$U\Delta_m$	3.3	8567	2.7	32
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
3 (Slovenia)	Δ_m	1.4	821	0.6	-4
	$U\Delta_m$	1.8	5870	2.2	19
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
4 (Lithuania)	Δ_m	-0.8	574	0.5	-2
	$U\Delta_m$	4.9	7414	2.7	26
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
5 (Slovakia)	Δ_m	1.0	1461	0.7	-3
	$U\Delta_m$	4.4	7037	2.6	23
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
6 (Ukraine)	Δ_m	1.1	-6	0.3	-51
	$U\Delta_m$	2.6	9099	3.3	41
	Δ_m - $U\Delta_m$	pass	pass	pass	failed
7 (Mexico)	Δ_m	-0.5	1841	0.9	-1
	$U\Delta_m$	2.6	7819	2.9	29
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
8 (Islamic Republic of Iran)	Δ_m	2.0	2997	1.4	-1
	$U\Delta_m$	3.1	6614	2.3	35
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
9 (Hungary)	Δ_m	0.2	5534	1.1	3
	$U\Delta_m$	3.4	12050	3.5	31
	Δ_m - $U\Delta_m$	pass	pass	pass	pass
10 (Syrian Araba Republic)	Δ_m	1.2	1693	0.9	5
	$U\Delta_m$	2.8	8899	3.2	31
	Δ_m - $U\Delta_m$	pass	pass	pass	pass

A second test for evaluating the laboratory's performance was applied on the basis of the ζ scores. The absolute values of ζ scores attributed to the laboratories are reported in the graphs of Figures 13-16. The green and red lines represent the critical limits of 2 and 3. For values ≤ 2 the participant's result associated to the sampling strategy adopted could be considered suitable for the objective stated; for value between 2 and ≤ 3 the participant's result could be questionable and for values > 3 the sampling strategy should need correction actions.

The ζ score takes into account the standard uncertainty of the participants' results and of the assigned value. The score is used when uncertainty of the assigned value is not calculated using the results reported by the participants, as in the case of IAEA/SIE/01 sampling intercomparison exercise. At most 40% of the ζ scores (As) exceed the critical value of 3. 60-90% of the values are ≤ 2 (suitable strategy). The higher percentage of acceptable scores is observed for Zn (80%) while Fe, Sc, and As show comparable percentage of scores ≤ 2 and in the range 2-3. These results confirm what has been observed evaluating the bias values.

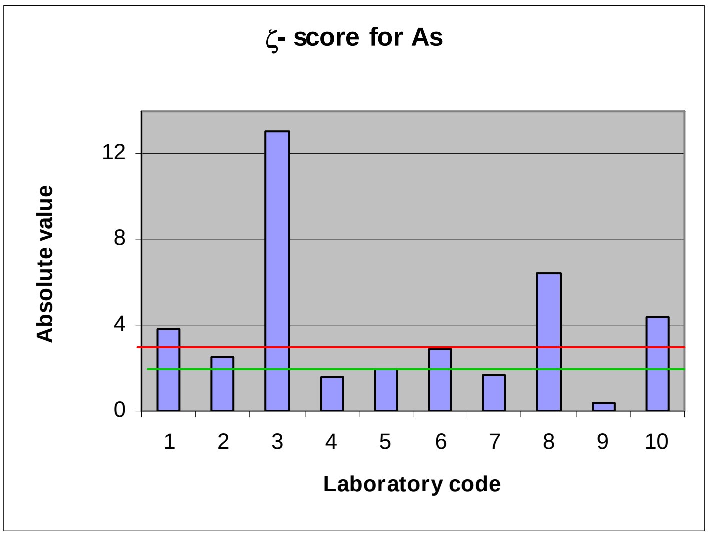

FIG. 13. Evaluation of the laboratories performance (ζ-score values) for arsenic.

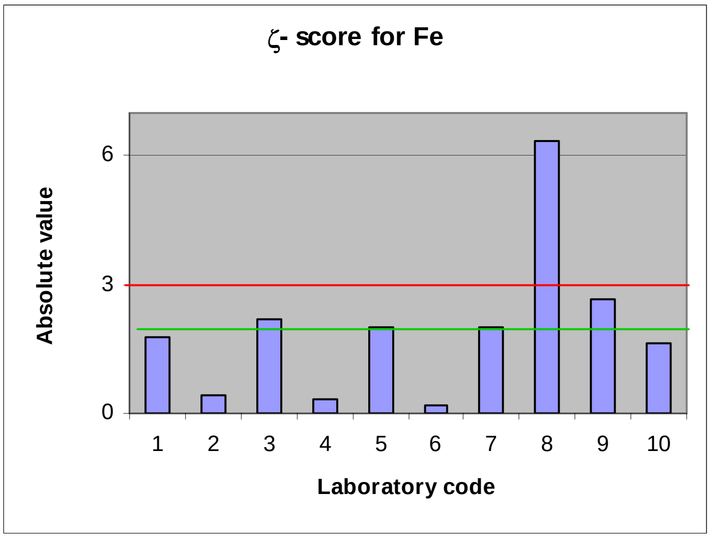

FIG. 14. Evaluation of the laboratories performance (ζ-score values) for iron.

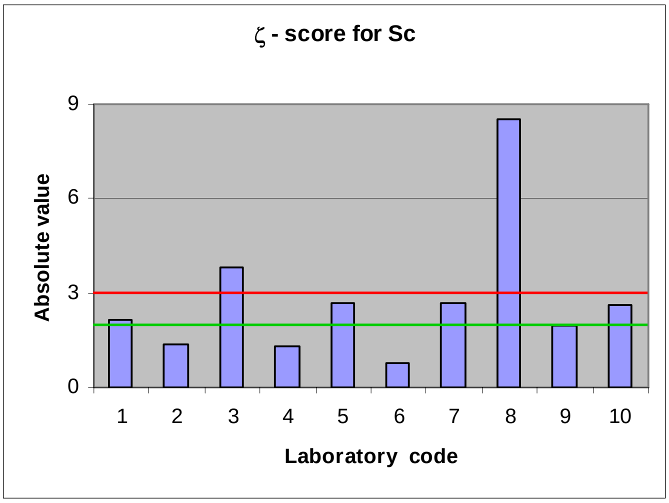

Fig. 15. Evaluation of the laboratories performance (ζ-score values) for scandium.

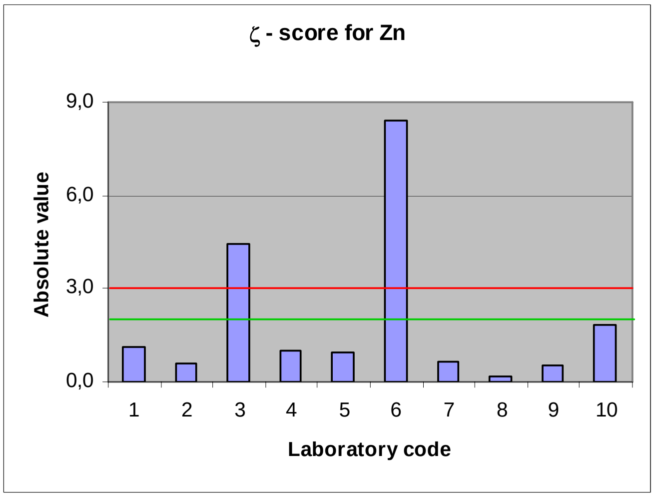

FIG. 16. Evaluation of the laboratories performance (ζ-score values) for zinc.

On the basis of these results, in a homogeneous agricultural area (within the ploughed layer) the sampling strategies chosen by the laboratories can be considered comparable.As sampling of top soil in arable and ploughed land is relatively easy leading to comparable results between different sampling procedures, operational aspects of the different sampling procedures become of interest:

- type of samples;
- number of samples/increments;
- time dedicated;
- effort.

In Table 9 a summary of the main aspects of the sampling procedures adopted by each laboratory is shown. The qualitative synthetic evaluation of the operational aspects of the sampling strategies and techniques, summarized by operational remarks, takes into account the number of samples and increments collected, the time consumed on the field and the estimated effort.

TABLE 9: SUMMARY OF THE SAMPLING PROCEDURES AND OPERATIONAL REMARKS. THE HIGHER NUMBER OF ASTERISKS (*) THE BETTER THE JUDGMENT AND THE OPERATIONAL PERFORMANCE

(1) the number of samples collected is qualitatively evaluated: *** (1-5 samples), ** (6-10 samples), * (11-15 samples)
(2) the number of increments pooled into each composite sample is qualitatively evaluated: *** (1-5 increments), ** (6-10 increments), * (≥ 11 increments)
(3) *** (≤ 90 minutes); ** (91-180 minutes); * (> 180 minutes). The time was corrected by expert judgment considering each team composed by two samplers
(4) The asterisks (*) represent an average of the qualitative judgments concerning number of samples/increments, time consumed and effort.

	Laboratories Code	Sampling pattern	Sampling devices	Type of sample	Sample pre-treatment in the field	Number of laboratory samples (1)	Number of increments (2)	Time (3)	Effort	Operational Remarks (4)
1.	Republic of Korea *(1 sampler)*	Non systematic *Zig-zag*	Sampling rings	Composite	No	**	**	*	**	**
2.	Brazil *(1 sampler)*	Systematic	Shovel / Spatula	Single	Yes	*	-	**	*	*
3.	Slovenia *(2 samplers)*	Systematic *Random (vertical)*	Corer	Single	No	*	-	**	*	*
4.	Lithuania *(1 sampler)*	Non Systematic	Spatola / Spade	Single	No	*	-	***	**	**
5.	Slovakia *(2 samplers)*	Systematic	Shovel	Single	Yes	*	-	**	**	**
6.	Ukraine *(2 samplers)*	Stratified random	Corer	Composite	No	**	***	**	*	**
7.	Mexico *(1 sampler)*	Systematic	Corer	Composite	Yes	*	**	**	***	**
8.	Islamic Republic of Iran *(1 sampler)*	Systematic	Shovel	Composite	Yes	*	***	**	**	**
9.	Hungary *(2 samplers)*	Stratified random	Corer	Single	No	*	-	***	**	**
10.	Syrian Arab Republic *(1 sampler)*	Non Systematic	Spatula	Composite	No	**	***	**	***	***

9. CONCLUSIONS

Ten sampling teams, belonging to different international scientific institutions, participated in the IAEA/SIE/01 sampling intercomparison exercise in November 2005 [15, 16]. The laboratories were asked to apply their own sampling procedures, respecting some general and common rules fixed by the intercomparison organizer.

The above data show that the sampling teams elaborated different approaches to respond to the objective of the intercomparison. A wide set of different sampling procedures, in terms of sampling strategy/patterns, sampling devices, type and number of samples, sample pre-treatment in the field were chosen by the teams.

The most common sampling strategies are systematic and non-systematic, equally distributed between the laboratories (40% both), as well as the type of samples (50% of the laboratories delivered composite samples and 50% single samples).

According to the criterion of ISO 13258:2005 [21] for the assessment of the laboratories' performances, the strategies adopted by all the sampling teams were in general suitable for the purpose, not exceeding in most of the cases bias values of $2\hat{\sigma}$. Same conclusions can be done with reference to the ζ scores.

In general for all the elements the slight differences observed between the laboratories do not appear attributable to the different sampling strategies. The figures show that sampling of top soil in an arable, ploughed land is relatively easy leading to comparable results between different sampling procedures.

Nevertheless, the significant differences are mainly due to some operational aspects. Collecting high number of samples (composite or singles), using more complicated sampling devices or, in some cases, applying sample pre-treatment in the field lead to different time for performing the sampling, increasing also the total expected costs for sampling and analysis.

The general equivalence of the approaches proposed by the laboratories, in terms of agreement of their measurement results with the assigned values, seems justifying, in analogue situation (aim of the measurements, environmental condition, etc.) the use of simple sampling procedures, not including any sample pre-treatment in the field (mainly sieving). The results of the intercomparison confirm, on the basis of experimental data, what was probably intuitive.

The IAEA/SIE/01 intercomparison exercise suggests could be useful for the next future testing different sampling strategies in a more heterogeneous area, such as a semi-natural or a contaminated soil area, with the aim of finding and identifying artificial hot spots. The fundamental requirement of a reference material for chemical analysis, represented by its homogeneity, becomes the heterogeneity, in the case of a reference site used within the framework of sampling intercomparison exercise. The terms homogeneity and stability are replaced by spatial variability and temporal variability.

APPENDIX I. ALMERA NETWORK LABORATORIES (updated May 2009)

Argentina	Comision Nacional de Energia Atomica Laboratorio de Metrologia de Radioisotopos Centro Atomico Ezeiza Presbitero Juan Gonzales y Aragon nro 15 CP B1802AYA Partido de Ezeiza Provincia de Buenos Aires (Argentina)
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	Australian Nuclear Science and Technology Organization ANSTO – Environment New Illawarra Rd 2234, Lucas Heights Menai N.S.W. (Australia)
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	Universitaet Wien Waehringerstrasse 42 A-1090, Vienna (Austria)
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Belgium	Studiecentrum Voor Kernenergie Centre d'etude de l'Energie Nucleaire Boeretang 200 B-2400, Mol (Belgium)
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	Kozloduy Nuclear Power Plant Kozloduy, 3321 (Bulgaria)
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	Chinese Academy of Agricultural Sciences (CAAS) Institute of Agro-Environment and Sustainable Development ALMERA of CAAS No. 12 Zhongguancun South Street 100081 Beijing (China)
	China Institute of Atomic Energy P.O.Box 275-24 Beijing, 102413 (China)
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Cuba	Centro de Proteccion e Higiene De Las Radiaciones Calle 20 Entre 41 Y 47 Apartado Postal 6791 Havana, 11300 (Cuba)

	Centro de Isotopos Carretera la Rada Km 3 Guanabacoa, Apto 22 Havana (Cuba)
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Czech Republic	National Radiation Protection Institute Bartoškova 28 140 00 Praha 4 (Czech Republic)
Denmark	Risoe National Laboratory P.O. Box 49 Dk-4000, Roskilde (Denmark)
Egypt	National Center for Nuclear Safety & Radiation Control Atomic Energy Authority 3 Ahmed El-Zomor Street P.O. Box 7551, Nasr City Cairo, 11762 (Egypt)
Estonia	Estonian Radiation Protection Centre Kopli 76 Tallinn, Ee-10416 (Estonia)
Ethiopia	National Radiation Protection Authority P.O.Box 20486 Code 1000, Addis Ababa (Ethiopia)
Finland	Radiation & Nuclear Safety Authority (STUK) P.O. Box 14 Fin-00881, Helsinki (Finland)
France	IPSN/DPRE/SERNAT F-91191, Gif-Sur-Yvette (France)
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	Hungarian Agricultural Authority Food & Feed Safety Directorate Central Radioanalitycal Laboratory P. O. Box 1740 H-1465, Budapest, 94 (Hungary)
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	Tarapur Atomic Power Station OIC/Environmental Survey Laboratory Via Boisar TAPS Colony, TAPP(post) 401 504 Tarapur, Maharashtra (India)
	Bhabha Atomic Research Centre Radiation Standards Section Mumbai, 400085 (India)
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Islamic Republic of Iran	Atomic Energy Organization of Iran (AEOI) North Karegar Street P.O. Box 14155-4494 14374, Tehran (Iran)
Iraq	Iraqi Atomic Energy Commission P.O. Box 765, Tuwaitha Baghdad (Iraq)
Israel	Nuclear Research Center Negev (NCRN) Negev (Israel)

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	Centrale Elettronucleare di Trino So.G.I.N Laboratories Strade Statale 31 Bis 13039, Vercelli (Italy)
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Peru	Instituto Peruano de Energia Nuclear (IPEN) Direccion de Investigation y Desarrollo Av. Canada n. 1470 Lima 41 (Peru)
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	Nuclear Power Plant Mochovce Komenskeho 3 Sk-935 39, Levice (Slovakia)
	Institute of Preventive & Clinical Medicine Limbova 14 Sk-833 01, Bratislava (Slovakia)
Slovenia	Institute Jozef Stefan Jamova 39 P.P.3000 Si-1001, Ljubljana (Slovenia)
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Sweden	Swedish Radiation Protection Institute Solna Strandväg 96 S-171 16, Stockholm (Sweden)
Switzerland	Federal Office of Public Health 3, Chemin Du Musee Ch-1700, Fribourg (Switzerland)
Syrian Arab Republic	Atomic Energy Commission of Syria P.O. Box 6091 Damascus (Syrian Arab Republic)

Thailand	Office of Atomic Energy for Peace 16 Vibhavadi Rangsit Chatuchak Bangkok, 10900 (Thailand)
Tunisia	Centre National des Sciences et Technologies Nucleaires Pole Technologique Sidi Thabet, 2020 (Tunisia)
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	National Physical Laboratory Radioactivity Metrology Group F7-A7, Hamptom Road Middlesex, TW11 OLW (United Kingdom)
	Centre for Environment, Fisheries & Aquaculture Science Pakefield Road Lowestoft, Nr33 0HT Suffolk (United Kingdom)
	LGC Limited Queens Road Teddington, Middlesex, TW11 0LY (United Kingdom)
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	US Department of Energy (DOE) 850 Energy Drive Idaho Falls, ID 83402 (United States of America)
	Purdue University 1396 Physics Building West Lafayette, IN 47907-1396 (United States of America)
	University of California Lawrence Livermore National Laboratory 7000 East Avenue P.O. Box 5001 Livermore, CA 94551 (United States of America)
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Department of Nuclear Sciences and Applications
Marine Environnent Laboratory
4, Quai Antoine Premier
MC980000 Monaco (Monaco)

APPENDIX II. LABORATORY'S REPORTING FORMS AND SAMPLE LOCATIONS

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 1

Sampling Team Members: - Dr. Byung-Uck Chang

1.) Weather Conditions: Fog

2.) Temperature: 14 [C]

3.) Sampling Time: from 8:00:00 to 12:00:00 on 2005-11-16

4.) Description of the sampling device:

Simplified soil sample ring kit consist of
- supplemental soil sampler (stainless steel)
- 11 soil sample rings with PVC cover (100ml, stainless steel)
- rubber hammer, hand shovel, and polyester bag

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:

I designed the systematic zigzag sampling based on the geostatistic idea to obtain the average surface soil sample of each cell.The agricultural soil shows characteristic having particular direction by humans cultivation activity during long time. This systematic zigzag sampling is more effective than traditional systematic random sampling for average surface soil of agricultural area.
First of all, I selected 10 cells (12, 20, 35, 45, 59, 67, 80, 89, 93, and 100) by zigzag shape in the reference site to calculate the average concentration of selective radionuclide in the reference site.
In each cell, I placed the 10 capped sampling rings with systematic zigzag shape points.
And collected each points 100ml soil samples put into the polyester bag as one soil sample of each cell.
I used the only one sample ring per each cell to avoid cross-contamination. And I used other ten capped rings to mark only each zigzag point. Before using the sampling ring and hammering, surface vegetations were removed by hand shovel (about 2~5 cm depth).The total amount of sampled surface soil of each cell is about 2 kg. And the sampling depth of each point was about 3~15 cm.

6.) Your Notes:

I think this systematic composite method of surface soil sampling is applicable effective method to obtain the average concentration of radionuclide either normal times or emergency. If two or three person enforces, time that take in sampling may reduce.

Sample Details

Sample No: 1

1.) Mass: 2000.0 [g]

3.) Square No: 12

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.12

Sample No: 2

1.) Mass: 2000.0 [g]

3.) Square No: 20

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.20

Sample No: 3

1.) Mass: 2000.0 [g]

3.) Square No: 35

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.35

Sample No: 4

1.) Mass: 2000.0 [g]

3.) Square No: 45

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.45

Sample No: 5

1.) Mass: 2000.0 [g]

3.) Square No: 59

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.59

Sample Details continued...

Sample No: 6

1.) Mass: 2000.0 [g]

3.) Square No: 67

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.67

Sample No: 7

1.) Mass: 2000.0 [g]

3.) Square No: 80

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.80

Sample No: 8

1.) Mass: 2000.0 [g]

3.) Square No: 89

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.89

Sample No: 9

1.) Mass: 2000.0 [g]

3.) Square No: 93

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.93

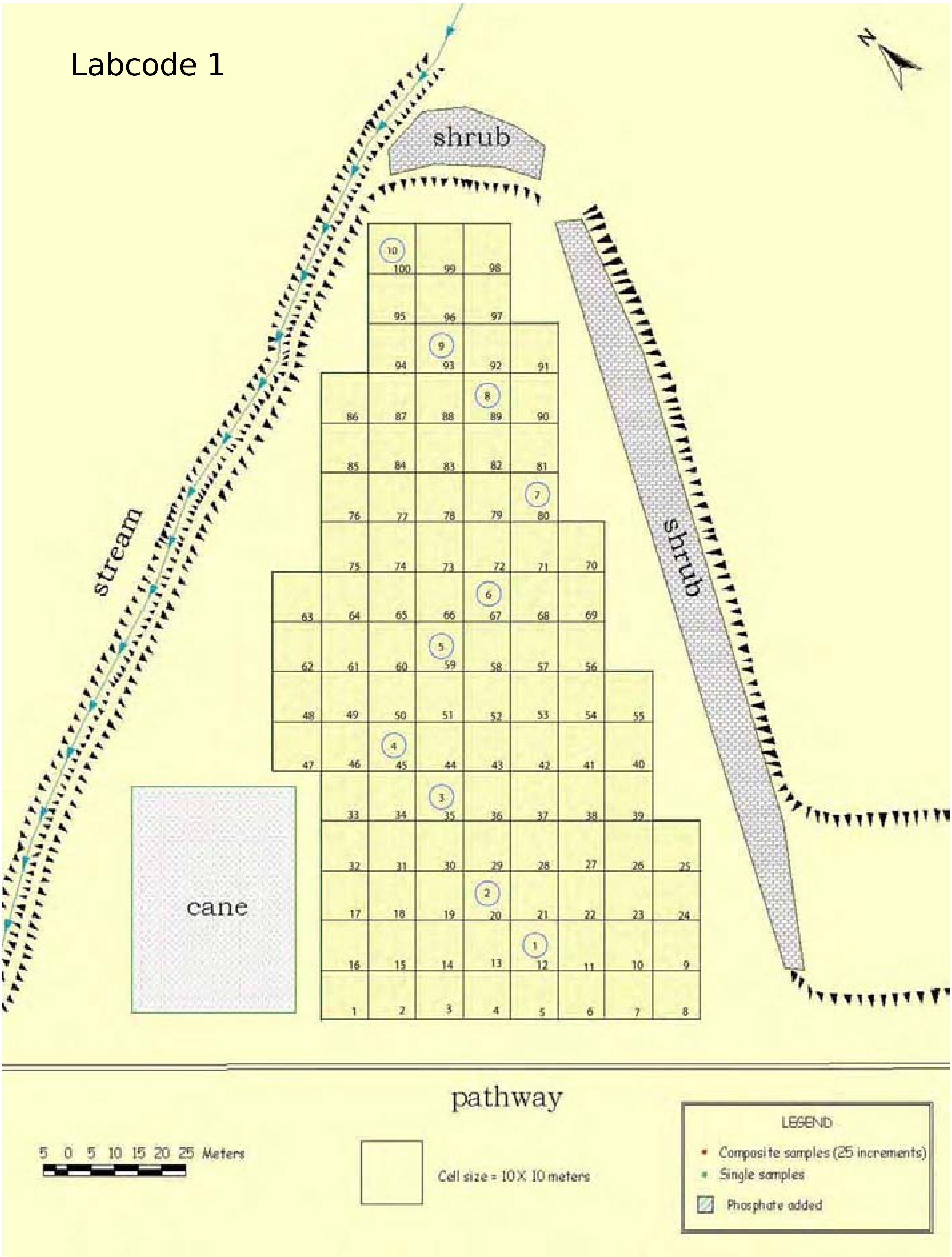
Sample No: 10

1.) Mass: 2000.0 [g]

3.) Square No: 100

2.) Sample Type: Composite

composite 10 systematic zigzag point samples with sample ring in the cell no.100


Labcode 1
shrub
stream
shrub
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 2

Sampling Team Members: - Ms. Maria Helena Tirollo Taddei

1.) Weather Conditions: Sun

2.) Temperature: 15 [C]

3.) Sampling Time: from 8:00:00 to 11:00:00 on 2005-11-17

4.) Description of the sampling device:

Shovel,spatula,sieve,gloves,absorbent towel,sieve <2mm, plastic bags, plastic sheet with a circular cut 30 cm diameter, tankard.

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:

Sampling pattern:see figure.
Sample weight:~50g.
Sieve: < 2 mm.
Each sample is taken through at 30 cm diameter circular cut in plastic sheet and at 20 cm depth.
The grass was removed and the sample was collected by shovel and deposited at the plastic sheet.
The stones and roots was removed and the homogeneization was by manual quartering.
The sample was sieved < 2mm ad put in identified plastic bags.
Finally each sample was measured with a tankard and put in the final vial.

6.) Your Notes:

Ideally the sampling must always to be with two persons.

Sample Details

Sample No: 1

1.) Mass: 50.0 [g]

3.) Square No: 15

2.) Sample Type: Single

Sample No: 2

1.) Mass: 50.0 [g]

3.) Square No: 45

2.) Sample Type: Single

Sample No: 3

1.) Mass: 50.0 [g]

3.) Square No: 74

2.) Sample Type: Single

Sample No: 4

1.) Mass: 50.0 [g]

3.) Square No: 94

2.) Sample Type: Single

Sample No: 5

1.) Mass: 50.0 [g]

3.) Square No: 98

2.) Sample Type: Single

Sample Details continued...

Sample No: 6

1.) Mass: 50.0 [g]

3.) Square No: 82

2.) Sample Type: Single

Sample No: 7

1.) Mass: 50.0 [g]

3.) Square No: 58

2.) Sample Type: Single

Sample No: 8

1.) Mass: 50.0 [g]

3.) Square No: 29

2.) Sample Type: Single

Sample No: 9

1.) Mass: None [g]

3.) Square No: None

2.) Sample Type:

Sample No: 10

1.) Mass: 50.0 [g]

3.) Square No: 25

2.) Sample Type: Single

Sample Details continued...

Sample No: 11

1.) Mass: 50.0 [g]

3.) Square No: 41

2.) Sample Type: Single

Sample No: 12

1.) Mass: 50.0 [g]

3.) Square No: 70

2.) Sample Type: Single

Sample No: 13

1.) Mass: 50.0 [g]

3.) Square No: 62

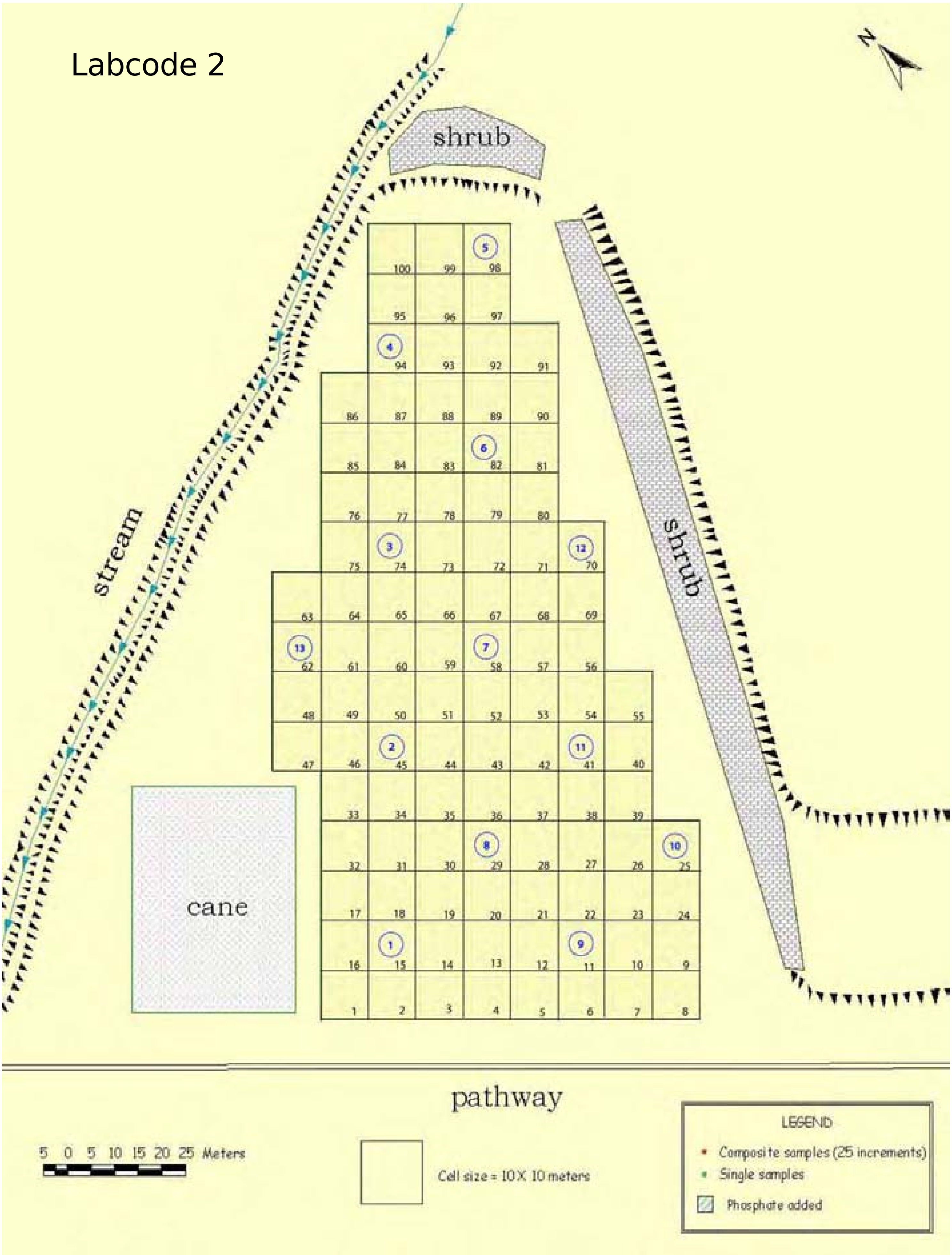
2.) Sample Type: Single

Sample No: 14

1.) Mass: None [g]

3.) Square No: None

2.) Sample Type: None


Labcode 2
shrub
stream
shrub
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 3

Sampling Team Members: - Dr. Matjaz Korun

- Dr. Jasmina Kozar-Logar

1.) Weather Conditions: Fog

2.) Temperature: 13.7 [C]

3.) Sampling Time: from 9:10:00 to 12:25:00 on 2005-11-16

4.) Description of the sampling device:

Corer with diameter of 8.7 cm. The corer can be opened from the side to anable the cutting of the core and removing the slices. The intrensing accuracy of the position of the cuts is 2 mm.
However, the actual accuracy is defined by the soil properties, the most important property is presence of the stones in the core and their size. The average accuracy attained under the circumstances at the sampling site is better than 0.5 cm.

5.) Sampling Strategy: other

Detailed Description of the Sampling Strategy:
The sampling was performed systematicaly in the horizontal plane and randomly in the vertical direction.
The sampling was performed near the eastern corner of the cells. The distance of sampling point from the corner varied between 20 and 60 cm, depending on the presence of the stones. In the vertical direction, the sampling depths were determined by generation random numbers. The range of the depths was between 0 and 20 cm at all locations except at the cell 61.

6.) Your Notes:

a.The detailed description of the soil sampling is in LMR-DN-07, which is attached as hard copy to the sampling sheets.
b.The methodology to prepare the Test portion is given in LMR-DN-07, chapter 11, Soil Sample preparation.
c.The description of the methodology to estimate the mean values is described in Appendix 1, attached to the sampling sheets.

Sample Details

Sample No: 1

1.) Mass: 100.0 [g]

3.) Square No: 71

2.) Sample Type: Single

depth: 3.5 - 6.5 cm
temp: 13 C, humidity: 76 %, time: 9:10
stones removed from the sample

Sample No: 2

1.) Mass: 250.0 [g]

3.) Square No: 78

2.) Sample Type: Single

depth: 8 - 12 cm
temp: 13.5 C, humidity: 77 %, time: 9:25
2nd attempt OK, stones removed

Sample No: 3

1.) Mass: 250.0 [g]

3.) Square No: 85

2.) Sample Type: Single

depth: 5 - 9 cm
time: 9:45
stones removed

Sample No: 4

1.) Mass: 200.0 [g]

3.) Square No: 91

2.) Sample Type: Single

depth: 12 - 15.3 cm
time: 9:57
2nd attept OK (stones), stones removed

Sample No: 5

1.) Mass: 250.0 [g]

3.) Square No: 96

2.) Sample Type: Single

depth: 5 - 9 cm
temp: 14.0, humidity: 70 %, time: 10:15
stones removed

Sample Details continued...

Sample No: 6

1.) Mass: 250.0 [g]

3.) Square No: 61

2.) Sample Type: Single

depth:18 - 22 cm
temp: 13.2 C , humidity: 77 %, time: 10:35
2nd attempt OK, stones removed

Sample No: 7

1.) Mass: 200.0 [g]

3.) Square No: 61

2.) Sample Type: Single

depth: 0 - 4 cm
other date as sample 6
stones and roots removed

Sample No: 8

1.) Mass: 300.0 [g]

3.) Square No: 61

2.) Sample Type: Single

depth: 36.3 - 43 cm
other data as sample 6
stones removed

Sample No: 9

1.) Mass: 250.0 [g]

3.) Square No: 51

2.) Sample Type: Single

depth: 5 - 9 cm
time: 11:20
stones removed

Sample No: 10

1.) Mass: 250.0 [g]

3.) Square No: 42

2.) Sample Type: Single

depth: 1 - 5 cm
temp: 14.6 C, humidity: 71 %, time: 11:34
stones removed

Sample Details continued...

Sample No: 11

1.) Mass: 250.0 [g]

3.) Square No: 39

2.) Sample Type: Single

depth: 9 - 13
time: 11:45
stones removed

Sample No: 12

1.) Mass: 250.0 [g]

3.) Square No: 7

2.) Sample Type: Single

deph: 3 - 7 cm
time: 11:58
stones removed

Sample No: 13

1.) Mass: 250.0 [g]

3.) Square No: 12

2.) Sample Type: Single

depth: 6 - 10 cm
time: 12:06
stones removed

Sample No: 14

1.) Mass: 300.0 [g]

3.) Square No: 19

2.) Sample Type: Single

depth: 6 -10 cm
temp: 14.3 C, humidity: 71 %, time: 12:15
no stones in the sample

Sample No: 15

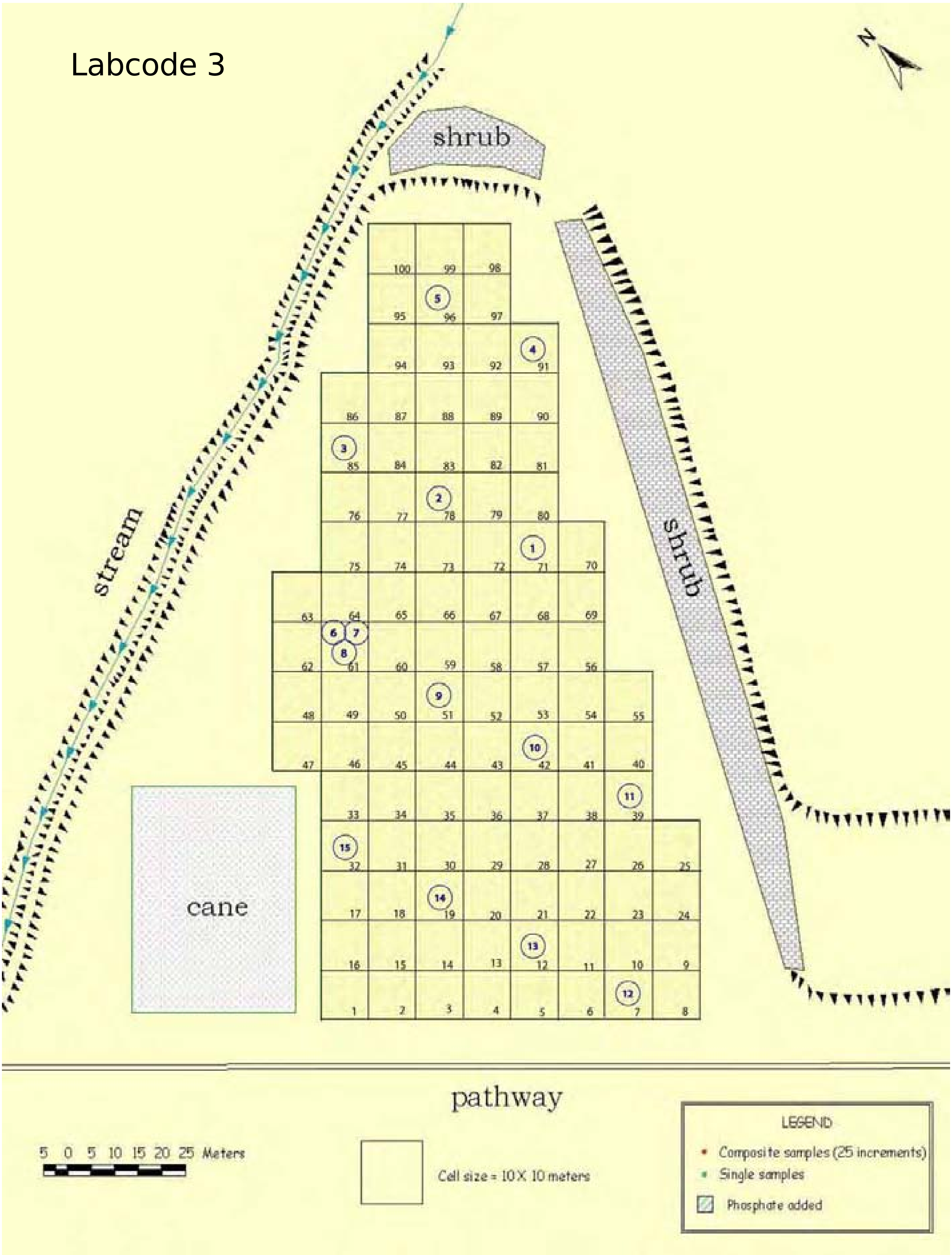
1.) Mass: 200.0 [g]

3.) Square No: 32

2.) Sample Type: Single

depth: 0 - 4 cm
time: 12:25
2nd attempt OK, stones removed

Labcode 3



shrub
shrub
stream
cane
pathway
100 99 98
95 96 97
94 93 92 91
86 87 88 89 90
85 84 83 82 81
76 77 78 79 80
75 74 73 72 71 70
63 64 65 66 67 68 69
62 61 60 59 58 57 56
48 49 50 51 52 53 54 55
47 46 45 44 43 42 41 40
33 34 35 36 37 38 39
32 31 30 29 28 27 26 25
17 18 19 20 21 22 23 24
16 15 14 13 12 11 10 9
1 2 3 4 5 6 7 8
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 4

Sampling Team Members: - Ms. Rima Ladygiene

1.) Weather Conditions: Fog

2.) Temperature: 12 [C]

3.) Sampling Time: from 9:55:00 to 11:35:00 on 2005-11-16

4.) Description of the sampling device:

Digging by spade
Determination of surface of sampling using ruler set on the spade
Cutting of the plants of the surface of the sampling up to 2 cm fom the ground
Determination of depth of the sampling using ruler
Cleaning of the sampling device after each of the sample using water and papier towel

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:

Sampling of 15 single samples was made in 15 sub-areas selected in the field that was devided to almost equal areas.
Sampling was made in the middle of each sub-area, in the middle of the square indicated in the sampling sheet.
Sampling surface of each of sample was 15cmx15cm, sampling depth 5 cm. Weight of the each sample was approximatelly 1 kg (not weighted at field), volume - 1 litter.
Sample was taken after cutting the plants up to 2 cm from the ground.
Sampling surface was measured using simple ruler on the spade set before the sampling.
Sample contains all materials available at sampling area to the depth up to 5 cm including plants, small stones etc.
Sample was taken directly to the plastic box prepared for the sampling.
No sieving was made at sampling field.

6.) Your Notes:

There were no problems related to the sampling.
Weather conditions were excelent, no rain, soil was not very dry and not very hard.
The field was appropriate to do sampling using spade and one man power.
Only for one of the samples the big stone disturbed the taking sample, and the sample was taken in the place close to that.

Sample Details

Sample No: 1

1.) Mass: 1000.0 [g]

3.) Square No: 10

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 2

1.) Mass: 1000.0 [g]

3.) Square No: 12

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 3

1.) Mass: 1000.0 [g]

3.) Square No: 15

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 4

1.) Mass: 1000.0 [g]

3.) Square No: 27

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 5

1.) Mass: 1000.0 [g]

3.) Square No: 30

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample Details continued...

Sample No: 6

1.) Mass: 1000.0 [g]

3.) Square No: 41

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 7

1.) Mass: 1000.0 [g]

3.) Square No: 43

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 8

1.) Mass: 1000.0 [g]

3.) Square No: 46

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 9

1.) Mass: 1000.0 [g]

3.) Square No: 57

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 10

1.) Mass: 1000.0 [g]

3.) Square No: 60

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample Details continued...

Sample No: 11

1.) Mass: 1000.0 [g]

3.) Square No: 71

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 12

1.) Mass: 1000.0 [g]

3.) Square No: 74

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 13

1.) Mass: 1000.0 [g]

3.) Square No: 83

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 14

1.) Mass: 1000.0 [g]

3.) Square No: 91

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Sample No: 15

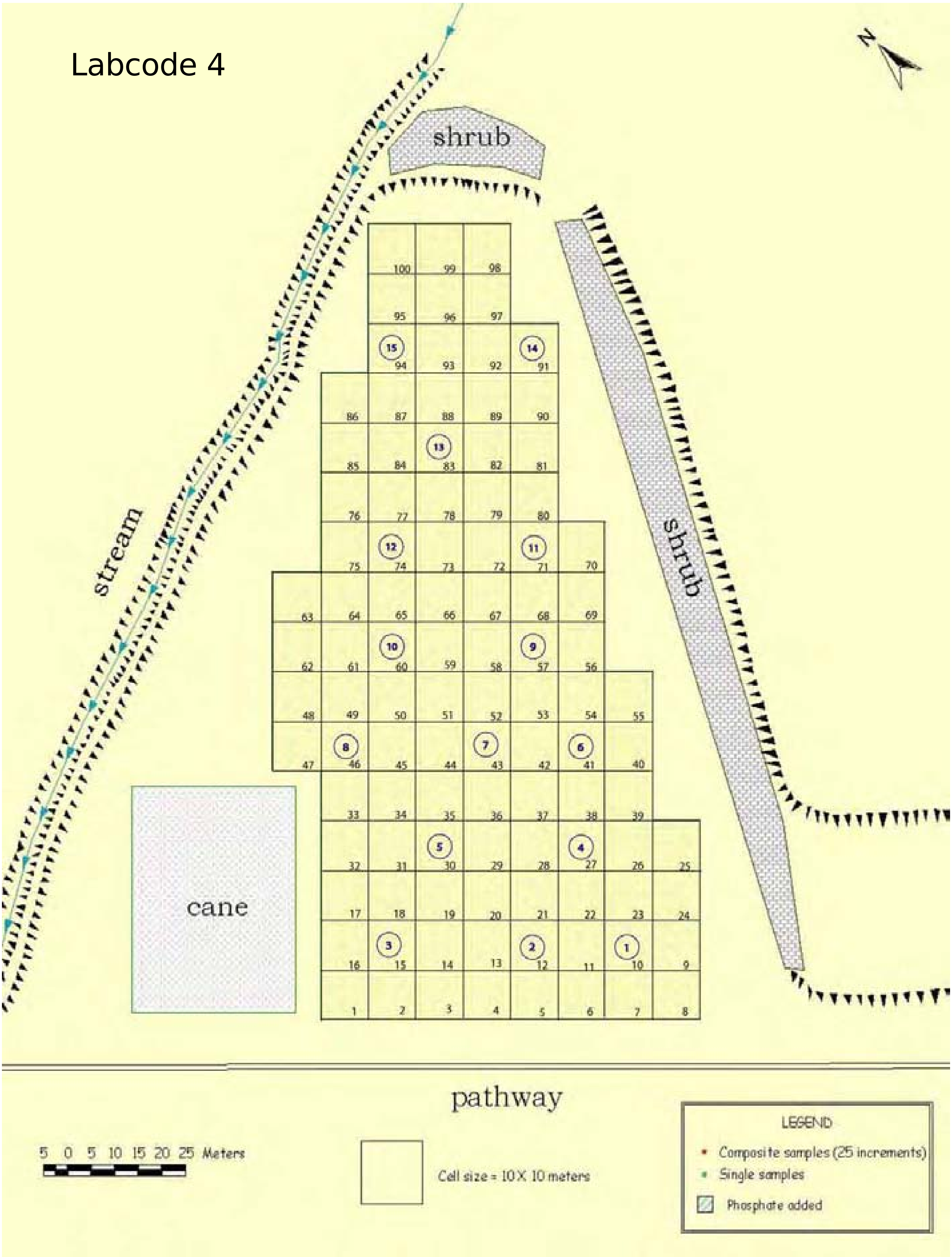
1.) Mass: 1000.0 [g]

3.) Square No: 94

2.) Sample Type: Single

sampling surface 15cmx15cm
sampling depth 5 cm

Labcode 4


shrub
stream
shrub
cane
pathway
100 99 98
95 96 97
15 14
94 93 92 91
86 87 88 89 90
13
85 84 83 82 81
76 77 78 79 80
12 11
75 74 73 72 71 70
63 64 65 66 67 68 69
10 9
62 61 60 59 58 57 56
48 49 50 51 52 53 54 55
8 7 6
47 46 45 44 43 42 41 40
33 34 35 36 37 38 39
5 4
32 31 30 29 28 27 26 25
17 18 19 20 21 22 23 24
3 2 1
16 15 14 13 12 11 10 9
1 2 3 4 5 6 7 8
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 5

Sampling Team Members: - Mr. Julius Vasarab

- Dr. Stefan Grubel

1.) Weather Conditions: Fog

2.) Temperature: 13.5 [C]

3.) Sampling Time: from 8:30:08 to 10:45:00 on 2005-11-16

4.) Description of the sampling device:

We used carbon steel shovel. Dimensions of this shovel was: 100 mm x 200 mm. With this sampling devices we can to sample in various depth: 0-20mm, 20-50mm, 0-50mm, 50-70mm, 70-100 mm,50-100 mm.
Soil (top of soil) was carefully cleans from the grassy vegetation.
After this we made small hole (app. 150 mm) with perpendicular cut and fixed slat at the margin of this cut. We put shovel on down rails and inserted shovel to the end of slat with the impact of hammer. We taked away soil sample and inserted into plastic bag.
After this we weighted the sample, removed biger stones and weighted again.

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:
Our first sampling point was in square No:2. This point was as randomly point with the distances (23m and 8m).
We used triangular grid sampling and we used the procedure from EPA and other documents.
We used Visual Sampling Plan software for the grid design. You can see it on original picture which we atached in paper form.
Used depth of sampling: 0-50 mm

6.) Your Notes:

We have been sampled bigger volume of the sample on this exercise (volume of our shovel).
Volume of the container was just sufficient for us.
Time in sampling was also just sufficient for our sampling technique a we worked under a small stress.

Sample Details

Sample No: 1

1.) Mass: 1250.0 [g]

3.) Square No: 2

2.) Sample Type: Single

Detailed position: 23m, 8m
GPS position: N: 45 59.355, E: 13 11.444
Orig. mass: 2400 g

Sample No: 2

1.) Mass: 1650.0 [g]

3.) Square No: 5

2.) Sample Type: Single

Detailed position: 51m, 8m
GPS position: N: 35 55.342, E: 13 11.452
Orig. mass: 1650 g

Sample No: 3

1.) Mass: 1550.0 [g]

3.) Square No: 7

2.) Sample Type: Single

Detailed position: 79m, 8m
GPS position: N: 45 59.336, E: 13 11.479
Orig. mass: 1550 g

Sample No: 4

1.) Mass: 1950.0 [g]

3.) Square No: 27

2.) Sample Type:

Detailed position: 65m, 32m
GPS position: N: 35 59.351, E: 13 11.482
Orig. mass: 2050 g

Sample No: 5

1.) Mass: 1800.0 [g]

3.) Square No: 30

2.) Sample Type: Single

Detailed position: 37m, 32m
GPS position: N: 45 59.357, E: 13 11.462
Orig. mass: 1950 g

Sample Details continued...

Sample No: 6

1.) Mass: 1500.0 [g]

3.) Square No: 40

2.) Sample Type: Single

Detailed position: 79m, 56m
GPS position: N: 45 53.356, E: 13 11.499
Orig. mass: 1500 g

Sample No: 7

1.) Mass: 1450.0 [g]

3.) Square No: 42

2.) Sample Type: Single

Detailed position: 51m, 56m
GPS position: N: 45 59.364, E: 13 11.484
Orig. mass: 1500 g

Sample No: 8

1.) Mass: 1300.0 [g]

3.) Square No: 45

2.) Sample Type: Single

Detailed position: 23m, 56m
GPS position: N: 45 59.374, E: 13 11.467
Orig. mass: 1650 g

Sample No: 9

1.) Mass: 1450.0 [g]

3.) Square No: 63

2.) Sample Type: Single

Detailed position: 5m, 56m
GPS position: N: 45 59.390, E: 13 11.468
Orig. mass: 1600 g

Sample No: 10

1.) Mass: 1450.0 [g]

3.) Square No: 66

2.) Sample Type: Single

Detailed position: 37m, 81m
GPS position: N: 45 59.382, E: 13 11.484
Orig. mass: 2400 g

Sample Details continued...

Sample No: 11

1.) Mass: 1250.0 [g]

3.) Square No: 69

2.) Sample Type: Single

Detailed position: 65m, 81m
GPS position: N: 45 59.374, E: 13 11.508
Orig. mass: 2050 g

Sample No: 12

1.) Mass: 1400.0 [g]

3.) Square No: 77

2.) Sample Type: Single

Detailed position: 23m, 105m
GPS position: N: 45 59.397, E: 13 11.488
Orig. mass: 1750 g

Sample No: 13

1.) Mass: 1650.0 [g]

3.) Square No: 80

2.) Sample Type: Single

Detailed position: 51m, 105m
GPS position: N: 45 59.387, E: 13 11.508
Orig. mass: 2000 g

Sample No: 14

1.) Mass: 1500.0 [g]

3.) Square No: 88

2.) Sample Type: Single

Detailed position: 37m, 129m
GPS position: N: 45 59.403, E: 13 11.506
Orig. mass: 1650 g

Sample No: 15

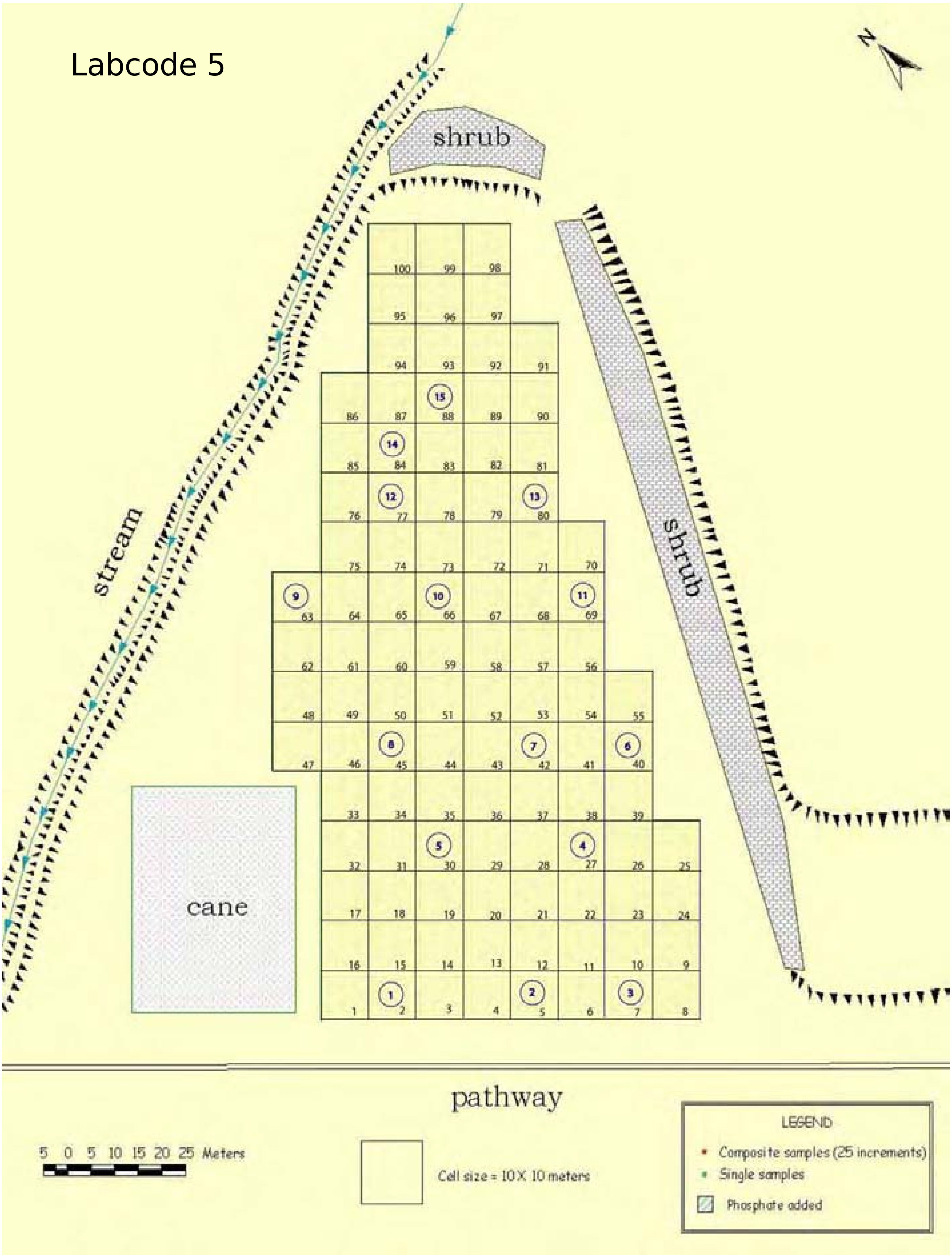
1.) Mass: 1900.0 [g]

3.) Square No: 100

2.) Sample Type: Single

Detailed position: 23m, 153m
GPS position: N: 45 59.416, E: 13 11.510
Orig. mass: 2000 g

Labcode 5



N
shrub
stream
shrub
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 6

Sampling Team Members: - Mr. Grygoriy Derkach

- Mr. Volodymyr Kanivets

1.) Weather Conditions: Sun

2.) Temperature: None [C]

3.) Sampling Time: from 8:30:00 to 10:40:00 on 2005-11-17

4.) Description of the sampling device:

Type of soil sampler is cylindrical soil corer. The length of sampling cylinder is 25 cm, inside diameter is 5.2 cm (2'), sampling area is 21.2 cm2. Cylindrical tube is manufactured by the firm NIBCOÃ‚Â® Inc. (USA) for drinking water supply from the material PVC-U. Sampling by tube was tested on semi-natural and agricultural soils in Ukraine and demonstrates simplicity in use and good quality of sampled cores.

5.) Sampling Strategy: non systematic

Detailed Description of the Sampling Strategy:
Pls. see Attachment 1

6.) Your Notes:

Pls. see Attachment 2

Sample Details

Sample No: 1

1.) Mass: 850.0 [g]

3.) Square No: 92

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
All increments were taken in the left upper corner of the square

Sample No: 2

1.) Mass: 850.0 [g]

3.) Square No: 85

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
One by one increments were taken in adjacent cornes of squares 85, 84, 77 and 76

Sample No: 3

1.) Mass: 850.0 [g]

3.) Square No: 79

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
Two by two increments were taken in squares 79 and 80

Sample No: 4

1.) Mass: 850.0 [g]

3.) Square No: 62

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
One by one increments were taken in adjacent squares 62, 61, 48 and 49

Sample No: 5

1.) Mass: 850.0 [g]

3.) Square No: 51

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
Two by two increments were taken in squares 51 and 52

Sample Details continued...

Sample No: 6

1.) Mass: 850.0 [g]

3.) Square No: 41

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
Two by two increments were taken in squares 41 and 40

Sample No: 7

1.) Mass: 850.0 [g]

3.) Square No: 17

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
Two by two increments were taken in squares 17 and 18

Sample No: 8

1.) Mass: 850.0 [g]

3.) Square No: 20

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
Two by two increments were taken in squares 20 and 21

Sample No: 9

1.) Mass: 850.0 [g]

3.) Square No: 23

2.) Sample Type: Composite

Sampling area is 85 cm2 (21.2 cm2 x 4);
All increments were taken in the right lower corner of the square

Attachment 1, Detailed Description of the Sampling Strategy

Sampling pattern

Reference site, selected for IAEA/SIE/01 has complicated shape, therefore we divided it on 3 sub-areas, roughly identical by shape and equal by size. Inside each sub-area it is selected 3 sampling points (stations), located in random order (random sampling). In each sampling point the composite sample has been taken.

Composite sample consists of 4 increments, sampled in corners of square by the size 2x2 m. Such distance between increments is enough for guarantee of independence in contamination of separate cores taken beside (Khomutinin et al., 2001).

Increment is represented by the core 10 cm by length and 5,2 cm by diameter (2').The size of increment is 210 cm3. Volume of 1 laboratory (composite) sample is 840 cm3 (210 m3 x 4).

Prevention of cross-contamination

9 sampling cylinders were prepared for the sampling exercise (1 per sampling point).

Cylinders were washed in consecutive order by detergents, 1 mol/l and distilled water. Each cylinder was packed hermetically into the separate polyethylene bags.

Methodology for preparation of Test portion

1. To weigh laboratory samples and to record wet weight

2. To dry samples in vent oven by the temperature 105 [C] to constant weight (air-dry state).

Note: for determination of the chemical forms of radionuclide it is necessary to dry sample by the temperature not higher than 40 [C].

3. To pound samples manually using wooden or porcelain pestle.

4. To screen sample through the sieve 1 mm mesh for removing of course sand, gravel, alien inclusions (stones, roots, twigs etc.). At first it is suitable to screen samples through the sieve 4-5 mm, then through the sieve 1 mm.

Note: radionuclides and some other contaminants is associated mainly with clayey minerals. For increase the comparability of contamination level in different samples it is useful, in some cases, to remove completely the particles with the diameter >0.1 mm (sand material). According to the numerous experimental data, this point (0.1 mm) on the granulometrical scale is the relative boundary between the material with high and low sorption properties.

5. If necessary, to dry and weigh sample again after sieving.

6. To homogenize sample manually. If the sample is large in volume it is necessary to use mechanical device for homogenization.

7. To reduce the sample size for the receiving of sample by the necessary volume. For this aim it is necessary to use the method of coning and quartering.

Note: For gamma-spectroscopy the following volumes for the test samples are used in UHMI: 2, 100, 500 and 1000 cm3 (depends from sample activity and gamma-spectrometer sensitivity). For radiochemical analysis the sample volume is 10-50 cm3.

Attachment 2, Notes

Number of samples

Quantity of samples, that are necessary for estimation of average (median) value of radionuclide concentration, is

determined by the expression, proposed by the Ukrainian Research Institute of Agricultural Radioecology

(Yu.Khomutinin et al., 2001) for ingradient fields:

1) for the sampling of single sample

2) for the sampling of composite sample

where is relative error of median (average value) value (0.1);

is quantile of normal distribution of a level of confidence (1.645);

m is quantity of increments (4);

is dispersion (variability) of logarithm of the soil contamination level between sampling points (0.3);

is dispersion (variability) of logarithm of the soil contamination level caused by inhomogeneity of contamination in sample volume (0.03);

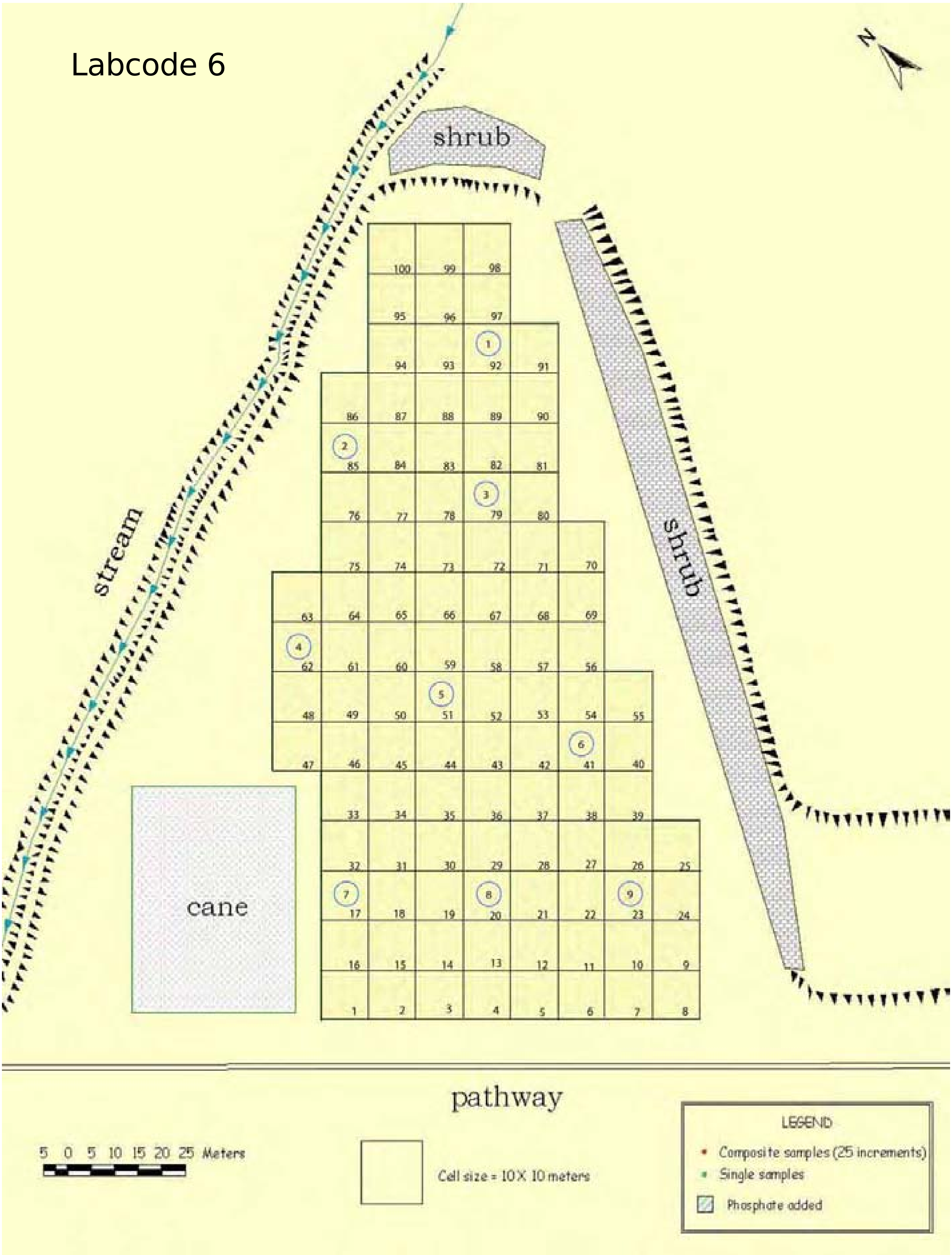
is dispersion (variability) of logarithm of the soil contamination level caused by the instrumental error (0.025);

no is the quantity of test portions, taken for measurements from the laboratory sample (1).

We have not data about variability of contamination in reference site. Therefore we took the value of (0.3), obtained for ingredient fields, contaminated by 137Cs.

For estimation of median value of contamination of the whole reference site with the relative error of 10% (P=0.95) it is necessary to take 8-9 laboratory composite soil samples (4 increments in each sampling point).

Labcode 6



N
shrub
stream
shrub
cane
pathway
100 99 98
95 96 97
94 93 92 91
86 87 88 89 90
85 84 83 82 81
76 77 78 79 80
75 74 73 72 71 70
63 64 65 66 67 68 69
62 61 60 59 58 57 56
48 49 50 51 52 53 54 55
47 46 45 44 43 42 41 40
33 34 35 36 37 38 39
32 31 30 29 28 27 26 25
17 18 19 20 21 22 23 24
16 15 14 13 12 11 10 9
1 2 3 4 5 6 7 8
1 2 3 4 5 6 7 8 9
5 0 5 10 15 20 25 Meters
Cell size = 10X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 7

Sampling Team Members: - Mr. Jaime Aguirre Gomez

1.) Weather Conditions: Sun

2.) Temperature: 8 [C]

3.) Sampling Time: from 8:15:00 to 10:50:00 on 2005-11-17

4.) Description of the sampling device:

SAMPLING DEVICES:
Shovel, Spade
Tray
Soil Markers
Coring tool
container Labelled (identification code)

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:

SAMPLING:
1. The reference site contains 100 cells, using the following formula I got the cell to be sampled: 100 cells/ 15 possible samples = aprox 7
It means that samples were collected each 7 cells, to obtain a total of 14 composite samples.
Cells sampled were: 7, 14, 21, 28, 35, 42, 49, 56, 63, 70, 77, 84, 91 and 98.
2. Once the site of sampling was chosen, demarcate it with a circle whose radius is 3.5 meter.
3. Demarcate 2 points, A is in the center of a circle and point B will be moved each 45 degree to obtain 8 simple samples at the same cell.
4.Cut vegetation until soil level, if necessary.
5. Obtain 8 nucleus of 7.5 cm diameter and 5 cm depth and produce a composite sample, in order to get a total area sampled of 4948 cm 2 which includes 8 nucleus per cell per 14 cells, (354 cm 2 per cell)
6.Register respective data of the soil sample, according with the type of sample, number, date, and facility.
7. Blend the 8 nucleus to obtain a composite sample, store 1 dm3 aprox in container labelled.
8.Transport the samples to the Laboratory.
9. In our procedure cleanliness, dry and sift of samples is performed at the laboratory after the sampling.

6.) Your Notes:

Sample Details

Sample No: 1

1.) Mass: 1500.0 [g]

3.) Square No: 7

2.) Sample Type: Composite

Sample No: 2

1.) Mass: 1500.0 [g]

3.) Square No: 14

2.) Sample Type: Composite

Sample No: 3

1.) Mass: 1500.0 [g]

3.) Square No: 21

2.) Sample Type: Composite

Sample No: 4

1.) Mass: 1500.0 [g]

3.) Square No: 28

2.) Sample Type: Composite

Sample No: 5

1.) Mass: 1500.0 [g]

3.) Square No: 35

2.) Sample Type: Composite

Sample Details continued...

Sample No: 6

1.) Mass: 1500.0 [g]

3.) Square No: 42

2.) Sample Type: Composite

Sample No: 7

1.) Mass: 1500.0 [g]

3.) Square No: 49

2.) Sample Type: Composite

Sample No: 8

1.) Mass: 1500.0 [g]

3.) Square No: 56

2.) Sample Type: Composite

Sample No: 9

1.) Mass: 1500.0 [g]

3.) Square No: 63

2.) Sample Type: Composite

Sample No: 10

1.) Mass: 1500.0 [g]

3.) Square No: 70

2.) Sample Type: Composite

Sample Details continued...

Sample No: 11

1.) Mass: 1500.0 [g]

3.) Square No: 77

2.) Sample Type: Composite

Sample No: 12

1.) Mass: 1500.0 [g]

3.) Square No: 84

2.) Sample Type: Composite

Sample No: 13

1.) Mass: 1500.0 [g]

3.) Square No: 91

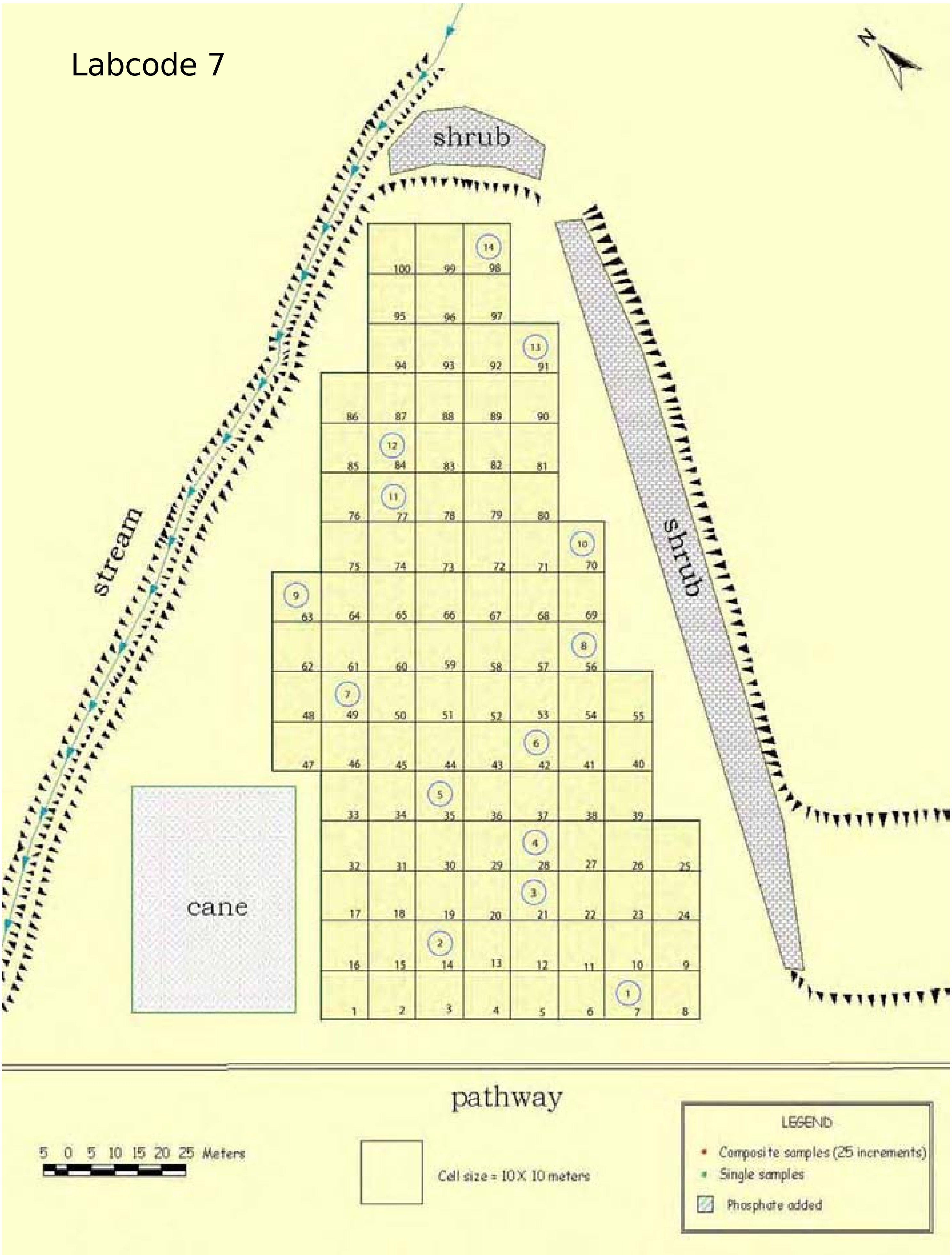
2.) Sample Type: Composite

Sample No: 14

1.) Mass: 1500.0 [g]

3.) Square No: 98

2.) Sample Type: Composite


Labcode 7
shrub
stream
shrub
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 8

Sampling Team Members: - Mr. Ali Asghar Fathivand Khalili

1.) Weather Conditions: Sun

2.) Temperature: 15 [C]

3.) Sampling Time: from 8:15:00 to 10:35:00 on 2005-11-17

4.) Description of the sampling device:

1.SHOVEL
2.Fork
3.SEIVE
4.METER
5.PLASTIC CONTAIER
6.LABELS
7.Maeker
8.Sample container

5.) Sampling Strategy: systematic

Detailed Description of the Sampling Strategy:

The reference site(10000 meter square)was flat and was sundivided in to 100 sub-areas(10x10 m)each.
Each sub-area was labeled(from 1 to 100).
14 sub-areas which cover whole the filed(sub-areas number 1,4,7,26,29,32,49,52,55,72,75,86,89 and 98) were choosen for soil sampling.From each sub-area one composit surface soil sample was taken as follows:
One square meter in the middle of each sub-area was labeld.Any surface vegetation and ston were removed.
Five surface soil sub sampls,up to 5 Cm in depth(1 from each corner and one from the middle of labeled one square meter)were taken.
Then the sub samples were processed to give one composit sample for each sub-area.
After sampling of each sub-area,the sampling devises were cleaned.
Finally each composit sample was put in labled(coded) container.

6.) Your Notes:

Sample Details

Sample No: 1

1.) Mass: 1000.0 [g]

3.) Square No: 1

2.) Sample Type: Composite

A composit sample from
five sub samples

Sample No: 2

1.) Mass: 1000.0 [g]

3.) Square No: 4

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 3

1.) Mass: 1000.0 [g]

3.) Square No: 7

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 4

1.) Mass: 1000.0 [g]

3.) Square No: 26

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 5

1.) Mass: 1000.0 [g]

3.) Square No: 29

2.) Sample Type: Composite

A composit sample from five sub samples

Sample Details continued...

Sample No: 6

1.) Mass: 1000.0 [g]

3.) Square No: 32

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 7

1.) Mass: 1000.0 [g]

3.) Square No: 49

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 8

1.) Mass: 1000.0 [g]

3.) Square No: 52

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 9

1.) Mass: 1000.0 [g]

3.) Square No: 55

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 10

1.) Mass: 1000.0 [g]

3.) Square No: 72

2.) Sample Type: Composite

A composit sample from five sub samples

Sample Details continued...

Sample No: 11

1.) Mass: 1000.0 [g]

3.) Square No: 75

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 12

1.) Mass: 1000.0 [g]

3.) Square No: 86

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 13

1.) Mass: 1000.0 [g]

3.) Square No: 89

2.) Sample Type: Composite

A composit sample from five sub samples

Sample No: 14

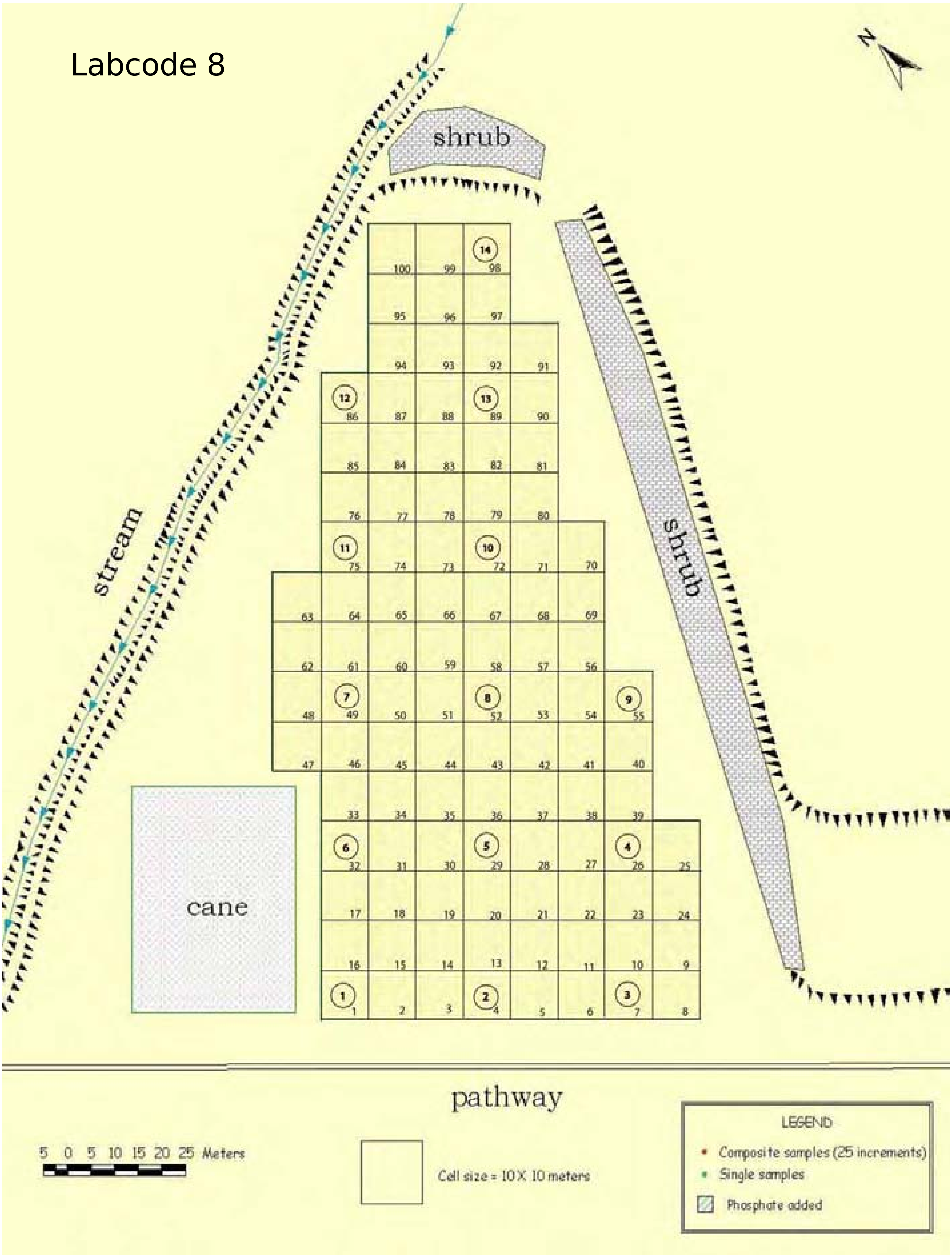
1.) Mass: 1000.0 [g]

3.) Square No: 98

2.) Sample Type: Composite

A composit sample from five sub samples

Labcode 8



shrub
stream
shrub
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 9

Sampling Team Members: - Dr. Sandor Tarjan

- Ms. Beata Varga

1.) Weather Conditions: Fog

2.) Temperature: 8 [C]

3.) Sampling Time: from 9:58:00 to 11:03:00 on 2005-11-16

4.) Description of the sampling device:

Auger made of stainless steel with cap, hammered into the soil.
Inner plastic tube: diameter - 85mm
length - 225mm

5.) Sampling Strategy: stratified

Detailed Description of the Sampling Strategy:
Sampling site was divided into 7 parts and subareas were selected randomly from each part: 13, 17, 34, 51, 60, 81, 94

The core sample from 51 subarea was sliced into 2,5cm layers.

6.) Your Notes:

Single samples were taken approximately 0-15cm depth.
Number of samples 13, empty sample-holders No 11 and No 15.

Sample Details

Sample No: 1

1.) Mass: 1900.0 [g]

3.) Square No: 13

2.) Sample Type: Single

Sample No: 2

1.) Mass: 1900.0 [g]

3.) Square No: 17

2.) Sample Type: Single

Sample No: 3

1.) Mass: 1900.0 [g]

3.) Square No: 34

2.) Sample Type: Single

Sample No: 4

1.) Mass: 142.2 [g]

3.) Square No: 51

2.) Sample Type: other
core sample, layer 0-2.5cm

Sample No: 5

1.) Mass: 261.3 [g]

3.) Square No: 51

2.) Sample Type: other
core sample, layer 2.5-5cm

Sample Details continued...

Sample No: 6

1.) Mass: 263.3 [g]

3.) Square No: 51

2.) Sample Type: other

core sample, layer 5-7.5cm

Sample No: 7

1.) Mass: 291.9 [g]

3.) Square No: 51

2.) Sample Type: other

core sample, layer 7.5-10cm

Sample No: 8

1.) Mass: 280.6 [g]

3.) Square No: 51

2.) Sample Type: other

core sample, layer 10-12.5cm

Sample No: 9

1.) Mass: 161.6 [g]

3.) Square No: 51

2.) Sample Type: other

core sample, layer 12.5-15cm

Sample No: 10

1.) Mass: 189.2 [g]

3.) Square No: 51

2.) Sample Type: other

core sample, layer 15-17.5cm

Sample Details continued...

Sample No: 11

1.) Mass: None [g]

3.) Square No: None

2.) Sample Type:
EMPTY

Sample No: 12

1.) Mass: 1900.0 [g]

3.) Square No: 60

2.) Sample Type: Single

Sample No: 13

1.) Mass: 1900.0 [g]

3.) Square No: 81

2.) Sample Type: Single

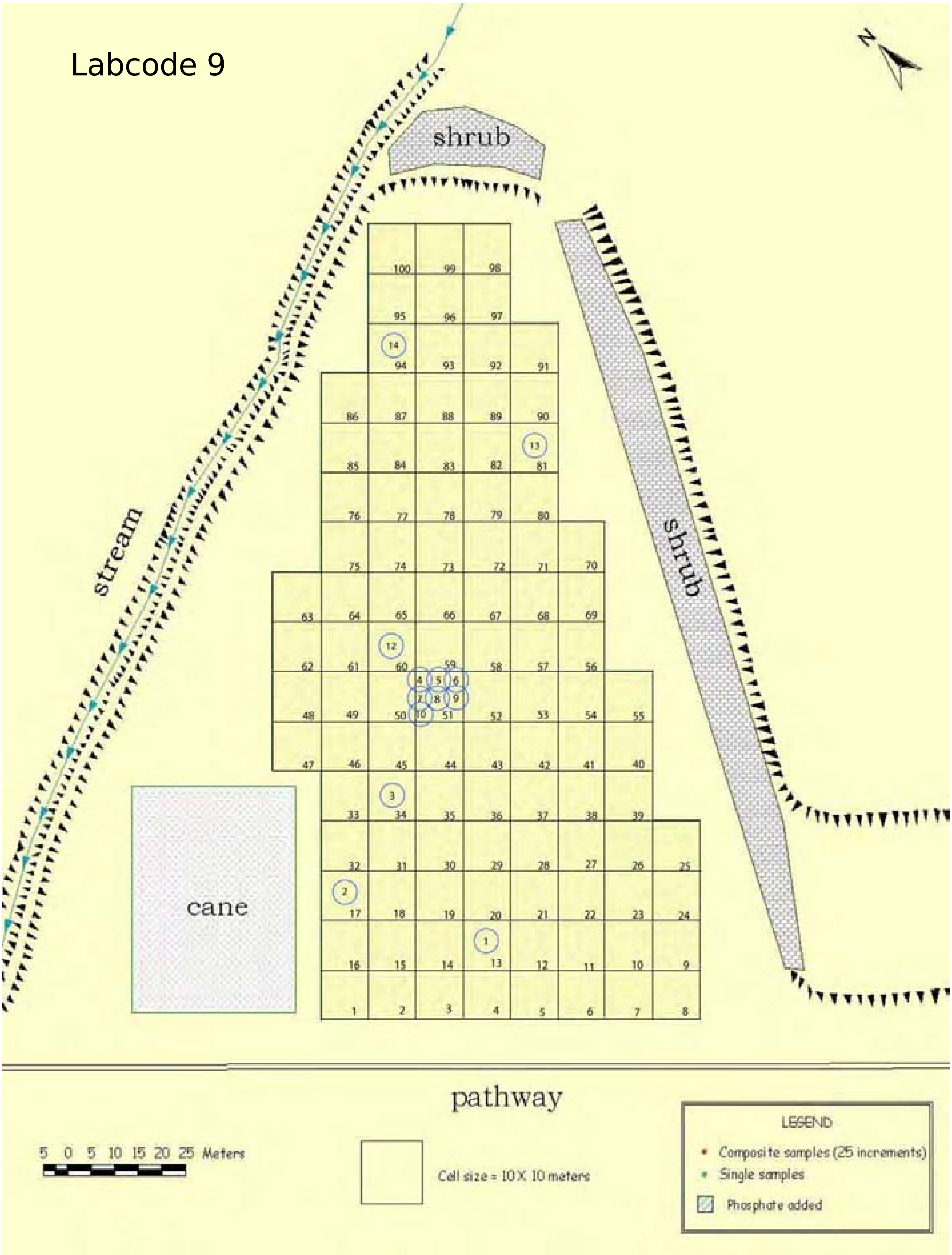
Sample No: 14

1.) Mass: 1900.0 [g]

3.) Square No: 94

2.) Sample Type: Single

Labcode 9



shrub
shrub
stream
cane
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

Reporting Form

IAEA/SIE/01: ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE 2005

Site: Pozzuolo del Friuli, Udine, Italy

Lab-Code: 10

Sampling Team Members: - Dr. Mohammnad S. Al-Masri

1.) Weather Conditions: Sun

2.) Temperature: 13 [C]

3.) Sampling Time: from 8:30:00 to 10:30:00 on 2005-11-17

4.) Description of the sampling device:

Scoop
Knife
Plastic bags

5.) Sampling Strategy: non systematic

Detailed Description of the Sampling Strategy:

The samples are taken fom flat area, open terrain with a minmum of earthworm and rodent and from an area of about 1 m2 taking five samples to a depth of 10 cm; composite sample (four samples from the corner and one from the center, around 250 g each). The samples are mixed togther in a bucket and one Kg subsample is taken for measurement. Stones and vegetation are removed from the sample and weighted for subsequent calcuations.

6.) Your Notes:

Sample Details

Sample No: 1

1.) Mass: 1000.0 [g]

3.) Square No: 73

2.) Sample Type: Composite

Sample No: 2

1.) Mass: 1000.0 [g]

3.) Square No: 87

2.) Sample Type: Composite

Sample No: 3

1.) Mass: 1000.0 [g]

3.) Square No: 77

2.) Sample Type: Composite

Sample No: 4

1.) Mass: 1000.0 [g]

3.) Square No: 92

2.) Sample Type: Composite

Sample No: 5

1.) Mass: None [g]

3.) Square No: 68

2.) Sample Type: Composite

Sample Details continued...

Sample No: 6

1.) Mass: 1000.0 [g]

3.) Square No: 56

2.) Sample Type: Composite

Sample No: 7

1.) Mass: 1000.0 [g]

3.) Square No: 52

2.) Sample Type: Composite

Sample No: 8

1.) Mass: 1000.0 [g]

3.) Square No: 35

2.) Sample Type: Composite

Sample No: 9

1.) Mass: 1000.0 [g]

3.) Square No: 31

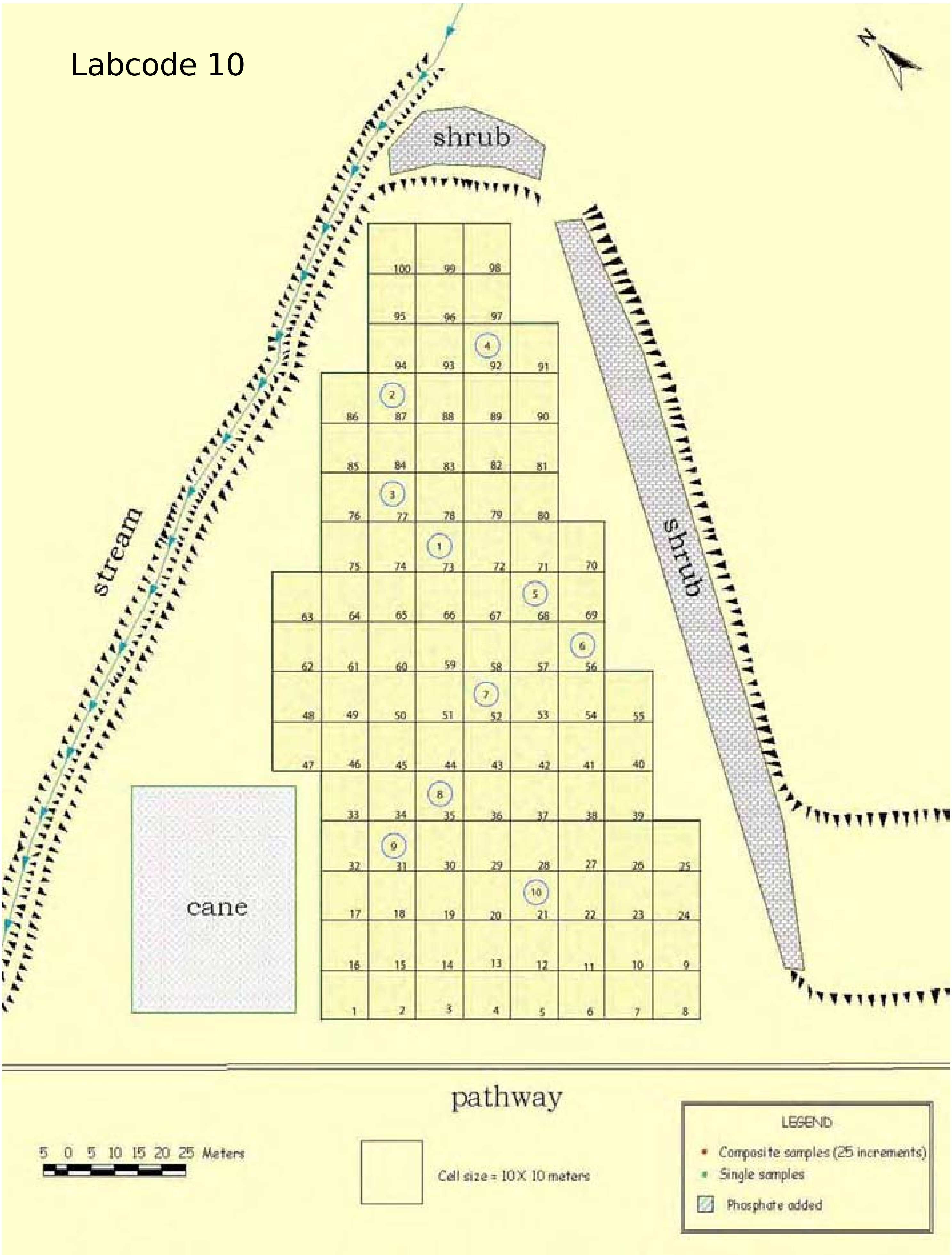
2.) Sample Type: Composite

Sample No: 10

1.) Mass: 1000.0 [g]

3.) Square No: 21

2.) Sample Type: Composite

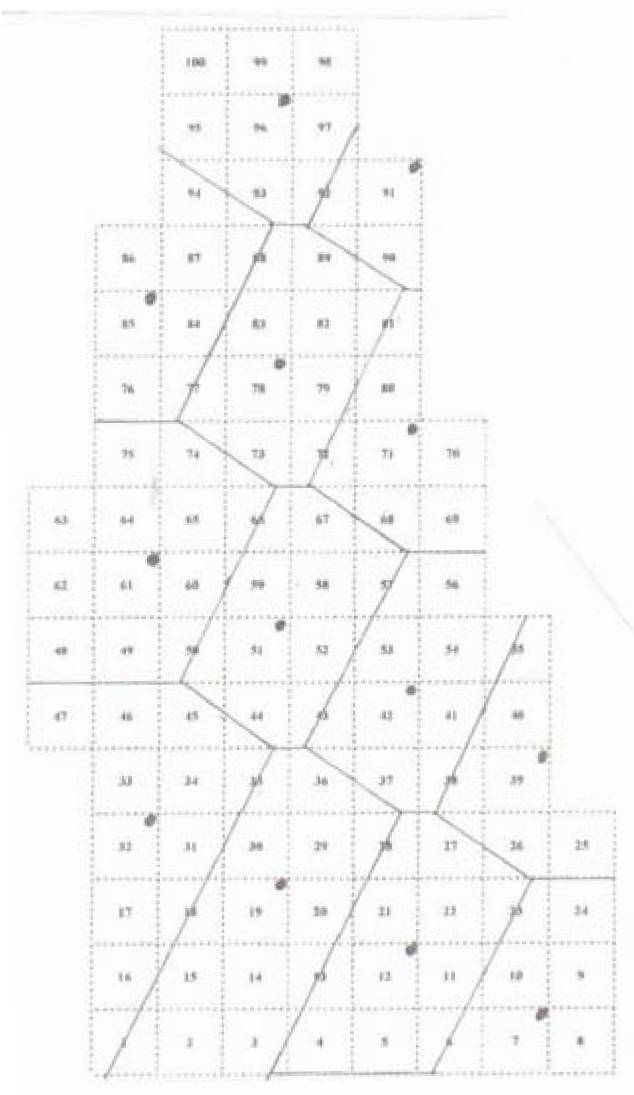

Labcode 10
N
stream
shrub
shrub
cane
100 99 98
95 96 97
94 93 92 91
86 87 88 89 90
85 84 83 82 81
76 77 78 79 80
75 74 73 72 71 70
63 64 65 66 67 68 69
62 61 60 59 58 57 56
48 49 50 51 52 53 54 55
47 46 45 44 43 42 41 40
33 34 35 36 37 38 39
32 31 30 29 28 27 26 25
17 18 19 20 21 22 23 24
16 15 14 13 12 11 10 9
1 2 3 4 5 6 7 8
1 2 3 4 5 6 7 8 9 10
pathway
5 0 5 10 15 20 25 Meters
Cell size = 10 X 10 meters
LEGEND
Composite samples (25 increments)
Single samples
Phosphate added

APPENDIX III. LABORATORY'S INSTRUCTION FOR CALCULATING MEAN VALUES

Laboratory code 3 – Slovenia

Methodology:

The mean value referring to the whole body sampled is obtained as the weighted average over the measurement results referring to the individual sampling locations. In case that no correlation exists between the spatial coordinates of the locations and the measurement results no model can be established and the weight of a specific measurement result is proportional to the fraction of the total area which is represented by the corresponding location. The cells which are represented by the sampling locations are presented in the figure below.



The uncertainty of the mean originates from two sources:

1. the uncertainties of the measurement results

2. the uncertainties of the weights.

Ad.1:

$$\overline{A} = \sum_{i=1}^{13} w_i A_i$$

w_i: weights $w_i=S_i/S$, where S_i is the area of the i-th cell, S the area of the reference site and A_i the individual measurement results referring to cell i.

$$\Delta_1^2 \overline{A} = \sum_{i,j=1}^{13} w_i w_j \Delta A_i \Delta A_j r_{ij}$$

ΔA_i: uncertainties of individual measurement results

r_{ij} : correlation matrix describing correlations among individual measurement results

Ad 2:

$$\overline{A} = \sum_{i=1}^{13} w_i A_i$$

$$\Delta_2^2 \overline{A} = \sum_{i,j=1}^{13} \Delta w_i \Delta w_j A_i A_j r'_{ij}$$

Δw_i : uncertainty of the weight wi

r'_{ij} : correlation matrix describing the correlation between the weights w_i

Δw_i are described by the uncertainties of the areas of the cells represented by individual locations. It is assumed that the positions of the borders separating the cells are uncertain for 0.5 m. Then the uncertainty of the weight w_i is

$$\Delta w_i = \frac{0.5m \cdot s_i}{S}$$

where s_i is the length of the borders, separating the cell i from the neighbouring cells.

The weights are correlated, since the displacement of a border increases the area of a cell in exactly the same amount as it decreases the area of the neighbour cell. For the specific sample pattern presented in Fig. 1 the correlation matrix is:

$$r'_{ij} = \begin{bmatrix}
1 & -1 & 0 & 0 & -1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
-1 & 1 & -1 & 0 & -1 & -1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & -1 & 1 & -1 & 0 & -1 & -1 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & -1 & 1 & 0 & 0 & -1 & -1 & 0 & 0 & 0 & 0 & 0 \\
-1 & -1 & 0 & 0 & 1 & -1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & -1 & -1 & 0 & -1 & 1 & -1 & 0 & -1 & 0 & 0 & 0 & 0 \\
0 & 0 & -1 & -1 & 0 & -1 & 1 & -1 & -1 & -1 & 0 & 0 & 0 \\
0 & 0 & 0 & -1 & 0 & 0 & -1 & 1 & 0 & -1 & -1 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & -1 & -1 & 0 & 1 & -1 & 0 & -1 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & -1 & -1 & -1 & 1 & -1 & -1 & -1 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & -1 & 0 & -1 & 1 & 0 & -1 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & -1 & -1 & 0 & 1 & -1 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & -1 & -1 & -1 & 1
\end{bmatrix}$$

In the matrix the sampling sites are sorted in the ascending order of the numbers of the squares where the sampling points are located.

Laboratory code 4 – Lithuania

Estimation of the mean value of the analyte – for the each measurement a result is given with total uncertainty, calculated taking into account all possible uncertainties and a confidence interval of 2σ. Estimation of uncertainties is a part of procedures of Quality manual of RPC. Detail descriptions of total uncertainty estimation are prepared for the gamma radionuclides, for strontium measurement in soil.

The mean value of the analyte is calculated using simple average value of all measuring values produced and calculating a confidence interval of population mean using Excel.

Laboratory code 5 – Slovakia

Estimation of the mean

For the estimation we used literature: Statistical Methods For Environmental Pollution Monitoring, author R.O. Gilbert.

In IAEA/SIE/01 soil sampling estimation we expect to collect app. 6000 g/each sample the soil at a given point (9 samples = 3 increments) or 2000g/each sample (6 samples = 1 increments) in time and space. They are too large weights and we will perhaps correct our soil sampling design while it is important estimate sub-sample app. 50g.

First stage in this estimation is collection soil samples mentioned above.

Second stage is to selected several aliquots from each environmental sample for measurement – sub-sampling. Sub-sampling introduces additionally uncertainty into estimates of means and totals because the entire sample mass is not measured.

In our case target population reference site is divisible into *N* primary (first stage) units, and the *i*-th primary unit is divisible into M_i sub-units. There is no requirement that the M_i is equal for all primary units.

In the IAEA/SIE/01 the estimation of the mean can be divided again into two ways:

1. *N (N=e.g. 15)* - only for each laboratory
2. $N=\Sigma A_i\ z_{ij}$, where $A_i=$ *i-th* participate laboratory, z_{ij} – number of collected primary samples of *i-th* participate laboratory

Situation will be complicated, perhaps that each participated laboratory submits primary samples of unequal size.

As you informed us sample treatment and trace elements determination will be done in a single laboratory.

We expect, that all *N* primary samples submit of each laboratory will be selected for measurement (*N*=*n*). From *n* primary samples will by selected *m* sub-samples, each of the equal size (perhaps *m=1*). Then the arithmetic mean of the *nm* measurements x_{ij} *is*:

$$\bar{\bar{x}} = \frac{1}{mn}\sum_{i=1}^{n}\sum_{j=1}^{m} x_{ij} = \frac{1}{n}\sum_{i=1}^{n}\bar{x}_i$$

where:

$$\bar{x}_i = \frac{1}{m}\sum_{j=1}^{m} x_{ij}$$

Estimate of variance among the *N* primary unit:

$$s_1^2 = \frac{1}{n-1}\sum_{i=1}^{n}\left(\bar{x}_i - \bar{\bar{x}}\right)^2$$

and estimate of variance among *M* subunits within primary units:

$$s_2^2 = \frac{1}{n(m-1)}\sum_{i=1}^{n}\sum_{j=1}^{m}\left(x_{ij} - \bar{x}_i\right)^2$$

Laboratory code 9 – Hungary

Estimation of the mean value: Gauss distribution of results is supposed. The best estimation of mean value of each analyte ($A_{(x)}$) is the following:

$$A_x = \frac{\sum_{i=1}^{7} \frac{X_i}{u_i^2}}{\sum_{i=1}^{7} \frac{1}{u_i^2}}$$

X result of the individual determination of given analyte,

i number of samples,

u uncertainty of individual result.

From the individual results of the core from 51 sub-area will be treated the same way (weighted mean) and it will be considered as one result.

The final result is $\mathbf{A_{(x)} \pm SD_{(x)}}$, where $SD_{(x)}$ is the standard deviation of analyte.

APPENDIX IV. ALMERA LABORATORIES' RESULTS

Lab Code	Sample Code	As mg kg^{-1} d.w			Fe mg kg^{-1} d.w			Sc mg kg^{-1} d.w			Zn mg kg^{-1} d.w		
1	1	10.1	±	0.2	28284	±	208	9.33	±	0.35	104	±	3
	2	10.5	±	0.2	29430	±	216	9.78	±	0.37	110	±	3
	3	10.4	±	0.2	27600	±	214	9.03	±	0.34	102	±	3
	4	9.8	±	0.2	23772	±	156	7.78	±	0.29	90	±	2
	5	9.5	±	0.2	23821	±	156	7.83	±	0.29	88	±	2
	6	9.1	±	0.2	22655	±	148	7.50	±	0.28	84	±	2
	7	8.5	±	0.2	21311	±	141	6.88	±	0.26	81	±	2
	8	8.4	±	0.2	19244	±	126	6.23	±	0.23	72	±	1
	9	7.8	±	0.1	16945	±	111	5.30	±	0.20	62	±	1
	10	6.9	±	0.1	17775	±	117	5.70	±	0.21	71	±	1

Lab Code	Sample Code	As mg kg^{-1} d.w			Fe mg kg^{-1} d.w			Sc mg kg^{-1} d.w			Zn mg kg^{-1} d.w		
2	1	10.0	±	0.2	26557	±	174	8.65	±	0.32	106	±	2
	2	11.6	±	0.2	28225	±	185	9.15	±	0.34	104	±	2
	3	8.8	±	0.2	23952	±	157	7.77	±	0.29	91	±	2
	4	7.7	±	0.1	20167	±	134	6.28	±	0.23	76	±	2
	5	8.3	±	0.2	21141	±	140	6.60	±	0.25	75	±	2
	6	8.3	±	0.2	21545	±	141	6.93	±	0.26	78	±	2
	7	9.4	±	0.2	24678	±	162	8.02	±	0.30	92	±	2
	8	10.9	±	0.2	29075	±	190	9.60	±	0.36	111	±	2
	9	10.9	±	0.2	27412	±	209	8.98	±	0.34	100	±	2
	10	10.3	±	0.2	27910	±	213	9.06	±	0.34	101	±	2
	11	10.5	±	0.2	28104	±	215	9.26	±	0.35	101	±	3
	12	10.2	±	0.2	26030	±	200	8.38	±	0.31	96	±	2

ALMERA LABORATORIES' RESULTS CONT.

Lab Code	Sample Code	As mg kg^{-1} d.w			Fe mg kg^{-1} d.w			Sc mg kg^{-1} d.w			Zn mg kg^{-1} d.w		
3	1	7.8	±	0.1	23745	±	157	7.57	±	0.28	98	±	2
	2	8.0	±	0.2	22074	±	146	7.06	±	0.26	90	±	2
	3	8.1	±	0.2	21919	±	144	6.90	±	0.26	96	±	2
	4	8.2	±	0.2	25757	±	169	8.26	±	0.31	114	±	2
	5	7.5	±	0.1	19651	±	129	6.19	±	0.23	81	±	2
	6	8.7	±	0.2	24716	±	162	8.00	±	0.30	94	±	2
	7	8.7	±	0.2	24004	±	157	7.77	±	0.29	96	±	2
	8	9.8	±	0.2	24934	±	164	8.05	±	0.30	84	±	2
	9	10.1	±	0.2	27958	±	202	9.13	±	0.34	105	±	3
	10	9.4	±	0.2	27268	±	207	8.98	±	0.34	102	±	3
	11	10.0	±	0.2	27576	±	205	8.74	±	0.33	103	±	3
	12	9.6	±	0.2	25365	±	189	8.17	±	0.31	94	±	3
	13	9.9	±	0.2	27284	±	196	9.07	±	0.34	104	±	3
	14	9.9	±	0.2	26961	±	208	8.89	±	0.33	105	±	3

Lab Code	Sample Code	As mg kg^{-1} d.w			Fe mg kg^{-1} d.w			Sc mg kg^{-1} d.w			Zn mg kg^{-1} d.w		
4	1	12.3	±	0.2	25794	±	169	8.41	±	0.31	94	±	2
	2	13.0	±	0.2	27816	±	185	9.12	±	0.34	103	±	2
	3	12.7	±	0.2	25991	±	174	8.48	±	0.32	102	±	2
	4	12.8	±	0.2	26718	±	176	8.82	±	0.33	99	±	2
	5	12.7	±	0.2	25964	±	172	8.50	±	0.32	98	±	2
	6	13.2	±	0.2	26895	±	178	8.91	±	0.33	98	±	2
	7	13.7	±	0.3	28073	±	186	9.15	±	0.34	99	±	2
	8	12.1	±	0.2	24046	±	159	7.80	±	0.29	95	±	2
	9	13.1	±	0.2	29204	±	193	9.62	±	0.36	107	±	2
	10	13.9	±	0.3	27910	±	184	9.05	±	0.34	97	±	2
	11	8.6	±	0.2	23355	±	180	7.59	±	0.28	90	±	2
	12	9.2	±	0.2	24405	±	188	7.98	±	0.30	95	±	2
	13	8.6	±	0.2	21355	±	165	6.79	±	0.25	79	±	2
	14	8.6	±	0.2	22316	±	171	7.07	±	0.26	81	±	2
	15	7.6	±	0.1	20252	±	157	6.44	±	0.24	78	±	2

ALMERA LABORATORIES' RESULTS CONT.

Lab Code	Sample Code	As (mg kg^{-1} d.w)			Fe (mg kg^{-1} d.w)			Sc (mg kg^{-1} d.w)			Zn (mg kg^{-1} d.w)		
5	1	12.3	±	0.5	22559	±	179	7.65	±	0.29	95	±	3
	2	12.5	±	0.5	25302	±	198	8.46	±	0.32	98	±	3
	3	12.6	±	0.6	24522	±	207	8.08	±	0.30	92	±	3
	4	9.6	±	0.2	27286	±	180	9.03	±	0.34	103	±	2
	5	9.7	±	0.2	26108	±	171	8.67	±	0.32	101	±	2
	6	9.7	±	0.2	25827	±	170	8.42	±	0.31	98	±	2
	7	9.3	±	0.2	25059	±	165	8.34	±	0.31	93	±	2
	8	10.0	±	0.2	24804	±	163	8.03	±	0.30	94	±	2
	9	8.6	±	0.2	21701	±	142	7.12	±	0.27	87	±	2
	10	9.7	±	0.2	26320	±	172	8.63	±	0.32	96	±	2
	11	9.7	±	0.2	27352	±	179	8.99	±	0.34	101	±	2
	12	8.6	±	0.2	23258	±	155	7.57	±	0.28	90	±	2
	13	8.8	±	0.2	22858	±	150	7.45	±	0.28	86	±	2
	14	5.0	±	0.1	19343	±	127	6.12	±	0.23	81	±	2
	15	7.1	±	0.1	20183	±	133	6.38	±	0.24	89	±	2

Lab Code	Sample Code	As (mg kg^{-1} d.w)			Fe (mg kg^{-1} d.w)			Sc (mg kg^{-1} d.w)			Zn (mg kg^{-1} d.w)		
6	1	7.8	±	0.1	19926	±	163	6.34	±	0.24	110	±	4
	2	8.4	±	0.2	22573	±	178	7.29	±	0.27	131	±	5
	3	9.0	±	0.2	*n.a.*			*n.a.*			*n.a.*		
	4	9.1	±	0.2	22399	±	156	7.31	±	0.27	136	±	5
	5	10.2	±	0.2	28694	±	206	9.39	±	0.35	159	±	6
	6	10.2	±	0.2	26842	±	206	8.87	±	0.33	153	±	6
	7	9.5	±	0.2	25376	±	193	8.27	±	0.31	148	±	6
	8	10.4	±	0.2	29126	±	210	9.69	±	0.36	163	±	6
	9	10.8	±	0.2	27934	±	226	8.98	±	0.34	154	±	4

ALMERA LABORATORIES' RESULTS CONT.

Lab Code	Sample Code	As			Fe			Sc			Zn		
		mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w		
7	1	10.8	±	0.5	24698	±	163	8.09	±	0.3	99	±	2
	2	11.0	±	0.5	26141	±	172	8.58	±	0.32	105	±	2
	3	11.8	±	0.5	27346	±	179	9.08	±	0.34	104	±	2
	4	11.7	±	0.5	27473	±	180	9.11	±	0.34	105	±	2
	5	10.7	±	0.5	25085	±	165	8.10	±	0.30	100	±	2
	6	10.7	±	0.5	25169	±	164	8.33	±	0.31	98	±	2
	7	10.0	±	0.4	22823	±	150	7.41	±	0.28	93	±	2
	8	12.2	±	0.5	26212	±	207	8.75	±	0.3	101	±	3
	9	10.9	±	0.5	20950	±	185	6.85	±	0.26	83	±	2
	10	11.1	±	0.5	23565	±	214	7.61	±	0.29	88	±	2
	11	10.7	±	0.5	21330	±	192	6.98	±	0.26	83	±	3
	12	10.1	±	0.4	25825	±	218	8.33	±	0.31	100	±	3
	13	9.9	±	0.4	21125	±	172	6.78	±	0.25	81	±	2
	14	11.1	±	0.5	18624	±	151	5.84	±	0.22	72	±	2

Lab Code	Sample Code	As			Fe			Sc			Zn		
		mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w		
8	1	10.5	±	0.2	24865	±	162	8.07	±	0.30	102	±	2
	2	10.0	±	0.2	26162	±	170	8.65	±	0.32	107	±	2
	3	10.3	±	0.2	26867	±	177	8.84	±	0.33	99	±	2
	4	10.2	±	0.2	27826	±	193	9.09	±	0.34	109	±	3
	5	10.6	±	0.2	28976	±	218	9.47	±	0.36	109	±	3
	6	9.6	±	0.2	25745	±	186	8.25	±	0.31	103	±	3
	7	9.5	±	0.2	24129	±	167	7.70	±	0.29	94	±	2
	8	10.2	±	0.2	28140	±	199	9.07	±	0.34	104	±	2
	9	10.0	±	0.2	27996	±	190	9.10	±	0.34	104	±	2
	10	10.2	±	0.2	24829	±	170	7.93	±	0.30	91	±	2
	11	8.5	±	0.2	21987	±	152	7.08	±	0.27	83	±	2
	12	9.0	±	0.2	22408	±	158	7.09	±	0.27	85	±	2
	13	8.6	±	0.2	21864	±	160	7.00	±	0.26	81	±	2
	14	7.4		0.1	20351		161	6.49		0.24	115		5

ALMERA LABORATORIES' RESULTS CONT.

Lab Code	Sample Code	As			Fe			Sc			Zn		
		mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w		
9	1	11.2	±	0.5	27406	±	180	9.11	±	0.34	100	±	2
	2	11.2	±	0.5	18813	±	126	8.29	±	0.31	100	±	2
	3	11.7	±	0.5	26671	±	175	8.67	±	0.32	97	±	2
	4	10.1	±	0.4	25569	±	169	8.41	±	0.31	98	±	2
	5	10.8	±	0.5	25966	±	170	8.50	±	0.32	93	±	2
	6	12.0	±	0.5	26704	±	175	8.77	±	0.33	95	±	2
	7	11.3	±	0.5	26454	±	173	8.70	±	0.3	95	±	2
	8	11.7	±	0.5	27219	±	179	8.89	±	0.33	97	±	2
	9	11.8	±	0.5	26924	±	176	8.81	±	0.33	94	±	2
	10	12.2	±	0.5	28603	±	188	9.37	±	0.35	104	±	2
	12	11.9	±	0.5	27144	±	179	8.90	±	0.33	103	±	2
	13	9.6	±	0.4	16441	±	109	7.20	±	0.27	87	±	2
	14	8.3	±	0.4	17932	±	118	5.61	±	0.21	70	±	1

Lab Code	Sample Code	As			Fe			Sc			Zn		
		mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w			mg kg^{-1} d.w		
10	1	8.9	±	0.2	21807	±	143	7.11	±	0.27	82	±	2
	2	7.6	±	0.1	18652	±	123	5.97	±	0.22	69	±	1
	3	8.2	±	0.2	20917	±	139	6.82	±	0.25	82	±	2
	4	7.9	±	0.1	19969	±	131	6.47	±	0.24	65	±	1
	5	9.5	±	0.2	26112	±	170	8.61	±	0.32	92	±	2
	6	10.0	±	0.2	27979	±	182	9.24	±	0.34	98	±	2
	7	9.8	±	0.2	26594	±	173	7.33	±	0.27	96	±	2
	8	9.3	±	0.2	24538	±	160	8.07	±	0.30	92	±	2
	9	9.6	±	0.2	24424	±	160	7.92	±	0.30	88	±	2
	10	10.6	±	0.2	27240	±	178	9.03	±	0.34	88	±	2

APPENDIX V. SUMMARY STATISTICS

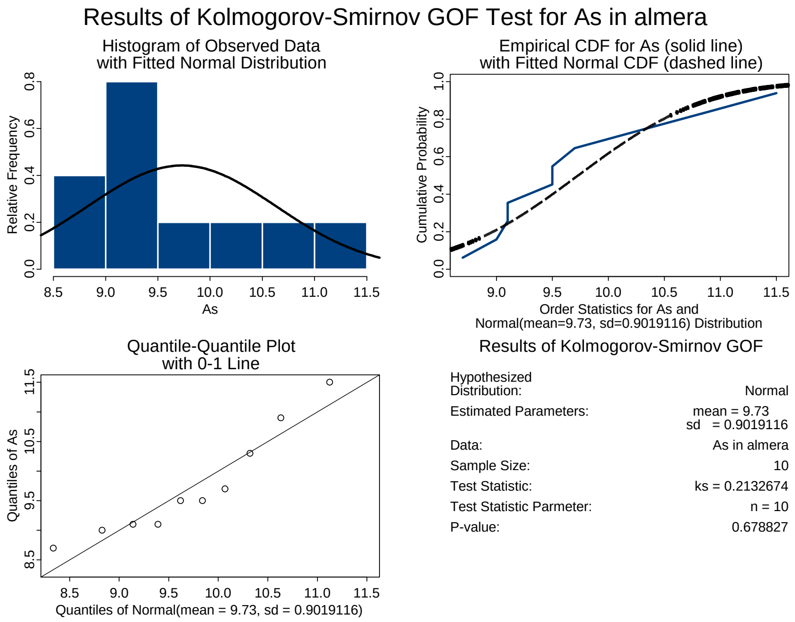
Lab Code	As			Fe			Sc			Zn		
	Mean value	SD mean	CV	Mean value	SD mean	CV	Mean value	SD mean	CV	Mean value	SD mean	CV
	mg kg^{-1}	mg kg^{-1}	%	mg kg^{-1}	mg kg^{-1}	%	mg kg^{-1}	mg kg^{-1}	%	mg kg^{-1}	mg kg^{-1}	%
1	9.1	0.4	13.0	23084	1385	19.0	7.54	0.5	20.3	86	5.0	18.2
2	9.7	0.3	12.2	25194	841	12.0	8.16	0.3	13.4	94	3.2	12.5
3 (*)	9.0	0.1	0.7	24929	61	0.2	8.08	0.08	1.1	98	0.8	0.9
4	11.5	0.6	19.6	25340	683	10.4	8.25	0.2	11.4	94	2.3	9.4
5	9.5	0.5	20.7	24166	632	10.1	7.93	0.2	11.1	94	1.6	6.8
6	9.5	0.4	10.7	25359	1197	13.3	8.27	0.4	14.3	144	6.2	12.1
7	10.9	0.2	6.0	24026	720	11.2	7.85	0.3	12.4	94	2.8	11.3
8	8.7	0.3	11.3	22288	433	7.3	7.12	0.1	7.2	91	3.8	15.5
9	10.3	0.5	12.7	20481	1891	24.4	7.64	0.5	16.4	90	4.3	12.7
10	9.1	0.3	10.7	23823	1037	13.8	7.66	0.3	14.35	85	3.5	13.0

Note (*) Mean value and standard deviation has been calculated by the laboratory itself on the basis of the raw analytical data communicated.

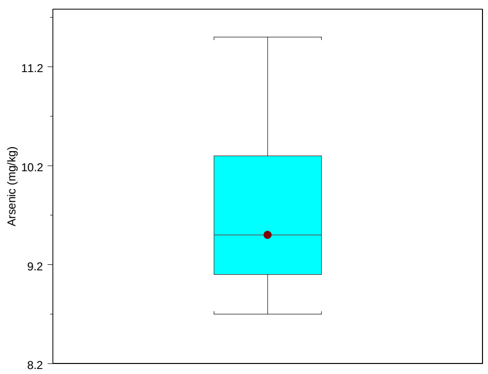
APPENDIX VI. SUMMARY OF NORMALITY TESTS & GRUBBS TEST

Measurand	**Normality tests**		**Grubbs test**
	Kolmogorov-Smirnov	**Shapiro-Wilk**	
As	Normal	Normal	-
Fe	Normal	Normal	-
Sc	Normal	Normal	-
Zn	Normal	Not normal	Outlier Lab code 6-*Ukraine*

Arsenic

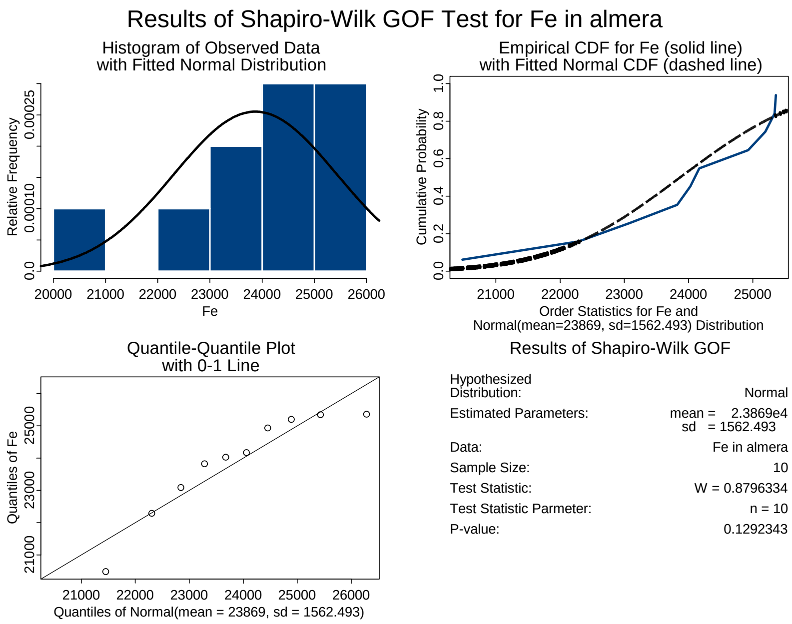


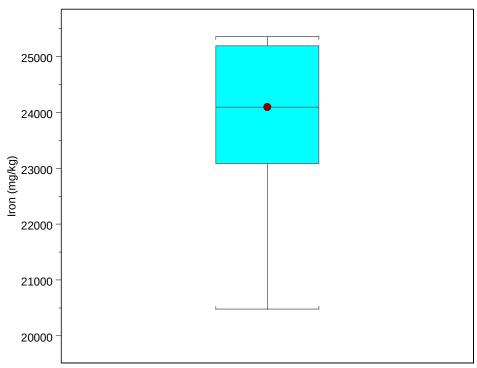
Results of Kolmogorov-Smirnov GOF Test for As in almera
Histogram of Observed Data with Fitted Normal Distribution
Relative Frequency
As
Empirical CDF for As (solid line) with Fitted Normal CDF (dashed line)
Cumulative Probability
Order Statistics for As and Normal(mean=9.73, sd=0.9019116) Distribution
Quantile-Quantile Plot with 0-1 Line
Quantiles of As
Quantiles of Normal(mean = 9.73, sd = 0.9019116)
Results of Kolmogorov-Smirnov GOF
Hypothesized Distribution: Normal
Estimated Parameters: mean = 9.73 sd = 0.9019116
Data: As in almera
Sample Size: 10
Test Statistic: ks = 0.2132674
Test Statistic Parmeter: n = 10
P-value: 0.678827



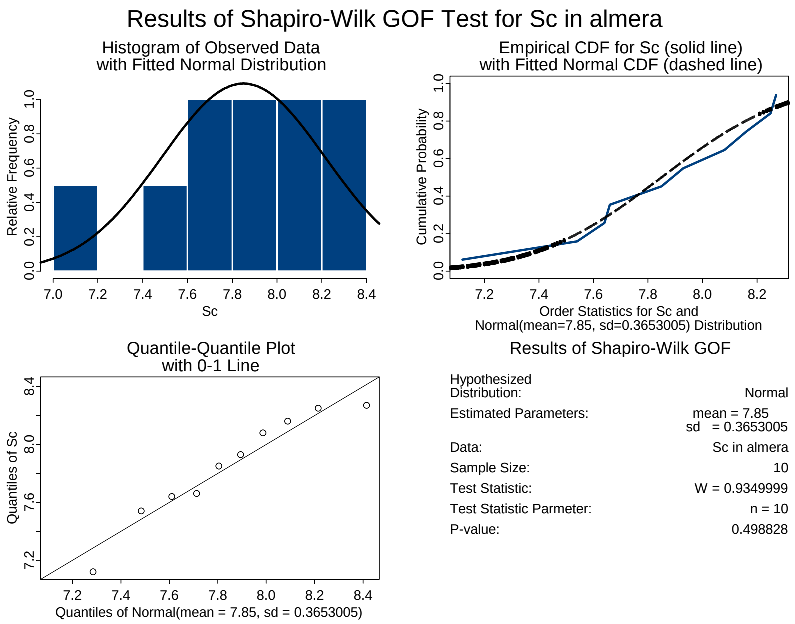
Arsenic (mg/kg)
11.2
10.2
9.2
8.2

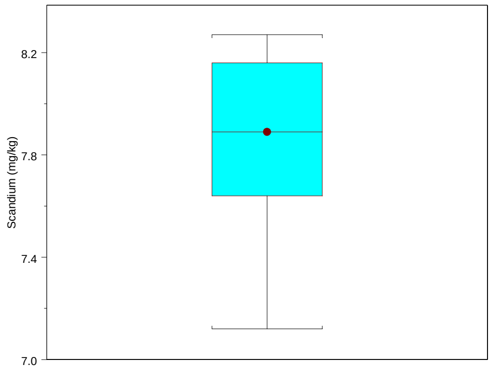
Iron


Results of Shapiro-Wilk GOF Test for Fe in almera
Histogram of Observed Data
with Fitted Normal Distribution
Relative Frequency
0.0
0.00010
0.00025
20000
21000
22000
23000
24000
25000
26000
Fe
Empirical CDF for Fe (solid line)
with Fitted Normal CDF (dashed line)
Cumulative Probability
0.0
0.2
0.4
0.6
0.8
1.0
21000
22000
23000
24000
25000
Order Statistics for Fe and
Normal(mean=23869, sd=1562.493) Distribution
Quantile-Quantile Plot
with 0-1 Line
Quantiles of Fe
21000
23000
25000
21000
22000
23000
24000
25000
26000
Quantiles of Normal(mean = 23869, sd = 1562.493)
Results of Shapiro-Wilk GOF
Hypothesized Distribution: Normal
Estimated Parameters: mean = 2.3869e4 sd = 1562.493
Data: Fe in almera
Sample Size: 10
Test Statistic: W = 0.8796334
Test Statistic Parmeter: n = 10
P-value: 0.1292343


Iron (mg/kg)
25000
24000
23000
22000
21000
20000

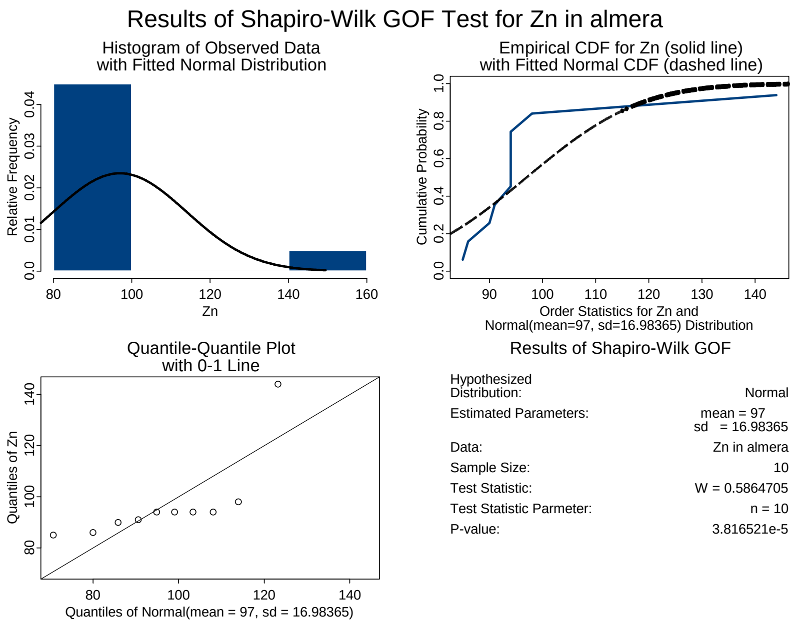
Scandium



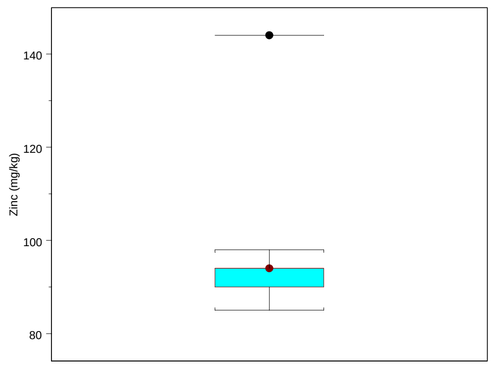


Grubbs test: No outlier

Zinc



Results of Shapiro-Wilk GOF Test for Zn in almera
Histogram of Observed Data
with Fitted Normal Distribution
Relative Frequency
0.0
0.01
0.02
0.03
0.04
80
100
120
140
160
Zn
Empirical CDF for Zn (solid line)
with Fitted Normal CDF (dashed line)
Cumulative Probability
0.0
0.2
0.4
0.6
0.8
1.0
90
100
110
120
130
140
Order Statistics for Zn and
Normal(mean=97, sd=16.98365) Distribution
Quantile-Quantile Plot
with 0-1 Line
Quantiles of Zn
80
100
120
140
Quantiles of Normal(mean = 97, sd = 16.98365)
Results of Shapiro-Wilk GOF
Hypothesized Distribution: Normal
Estimated Parameters: mean = 97 sd = 16.98365
Data: Zn in almera
Sample Size: 10
Test Statistic: W = 0.5864705
Test Statistic Parmeter: n = 10
P-value: 3.816521e-5



Zinc (mg/kg)
80
100
120
140

REFERENCES

[1]. DE ZORZI P, BELLI, M, BARBIZZI, S., MENEGON, S., DELUISA, A., A practical approach to assessment of sampling uncertainty. Accred.Qual Assurance, 7, 5, 182-18 (2002).

[2]. DE ZORZI, P., BARBIZZI, S., BELLI, M., CICERI, G., FAJGELJ, A., MOORE, D., SANSONE, U., AND VAN DER PERK, M., Terminology in soil sampling (IUPAC Recommendation 2005). Pure Appl. Chem., 77, 5, 827–841 (2005)

[3]. INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials-General and statistical principles for certification. ISO Guide 35, 2006.Geneva, Switzerland (2006).

[4]. INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, International vocabulary of metrology – Basic and general concepts and associated terms. ISO Guide 99, 3rd Edition, Geneva, Switzerland (2007).

[5]. COUGHTREY, P.J. AND THORNE, M.C., Radionuclide distribution and transport in terrestrial and aquatic ecosystems. A critical review of data. A.A, Balkema, Rotterdam (1983).

[6]. QIAN, J., SHAN, X., WANG, Z. AND TU, Q., Distribution and plant availability of heavy metals in different particle-size fractions of soil, The Science of the Total Environment 187, 131-141 (1996).

[7]. HAQUE, M.A., SUBRAMANIAN., Cu, Pb and Zn, pollution of soil environment. CRC Crit. Rev. Environ. Contr., 12, 13-90 (1982).

[8]. FOERSTNER, U., Trace metal analysis of polluted sediments, Part 1, Assessment of sources and intensities. Environ. Technol. Lett., 1, 494-505 (1980).

[9]. SPEZZANO P., Distribution of pre- and post-Chernobyl radiocaesium with particle size fractions of soil Journal of Environmental Radioactivity 83, 117-127 (2005).

[10]. LIVENS, F. R., BAXTER, M. S., Particle size and radionuclide levels in some West Cumbrian soils. The Science of the Total Environment 70, 1-17 (1988).

[11]. NAVAS, A., MACHÌN, J., SOTO, J., Mobility of natural radionuclides and selected major and trace elements along a soil toposequence in the central Spanish Pyrenees, Soil Science 170-9, 743-757 (2005).

[12]. HASL-300, THE PROCEDURES MANUAL OF THE ENVIRONMENTAL MEASUREMENTS LABORATORY 28th Edition, Section 2.4, Vol.1. US Department of Homeland Security, New York (1997).

[13]. DESAULES, A., SPRENGART, J., WAGNER, G., MUNTAU, H., THEOCHAROPOULOS, S., Description of the test area and reference sampling at Dornach. The Science of Total Environment 264, 17-26 (2001).

[14]. BARBIZZI, S., et al., Characterisation of reference site for quantifying uncertainties related to soil sampling. Environ. Pollut., 127, 131-135 (2004).

[15]. INTERNATIONAL ATOMIC ENERGY AGENCY. Report on the Second ALMERA coordination meeting and the ALMERA soil sampling intercomparison exercise – IAEA/SIE/01. IAEA/AL/164, IAEA, Vienna (2006) 36.

[16]. INTERNATIONAL ATOMIC ENERGY AGENCY. Report on the ALMERA Meeting 2006 at the Korea Institute of Nuclear Safety (KINS) – IAEA/AL/177, IAEA, Vienna (2007) 26.

[17]. JAĆIMOVIĆ, R, LAZARU, A., MIHAJLOVIĆ, D., ILIĆ, R., STAFILOV, T., Determination of major and trace elements in some minerals by k_0-instrumental neutron activation analysis. J. Radioanal. Nucl. Chem. 253, 427-434 (2002).

[18]. KALUZNY, S.P., VEGA, S.C., CARDOSO, T.P., SHELLY, A.A., S+Spatial Stats User's Manual for Windows and Unix. Springer, New York (1997).

[19]. INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Uses of certified reference material. ISO Guide 33, 2000, Geneva, Switzerland (2000).
[20]. LISINGER, T., Application Note 1 – Comparison of a measurement result with the certified value. ERM, European Reference Material (2005).
[21]. INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO 13528:2005 (E), Geneva, Switzerland (2005)
[22]. INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, General requirements for the competence of testing and calibration laboratories. ISO/IEC 17025, 2005, Geneva (2005).
[23]. MILLARD, S.P., NAGARAJ K. NEERCHAL; S.P.N., "Environmental Statistics with S-Plus", CRC Press, USA, (2001).

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