UNIVERSITY OF DEBRECEN CENTRE OF AGRICULTURAL SCIENCES FACULTY OF AGRICULTURE

### DOCTORAL SCHOOL OF CROP PRODUCTION AND HORTICULTURE

Head of Doctoral School: **Prof Dr habil Zoltán Győri** DSc

PhD Thesis

## EXTENSIBILITY OF MEASUREMENT RESULTS OF POINT SAMPLES OF THE SOIL PROTECTION INFORMATION AND MONITORING SYSTEM (TIM)

Supervisors:

Prof Dr emeritus Jakab Loch DSc

**Dr habil Béla Kovács** Associate Professor, PhD

Submitted by:

Dr László Várallyai

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### **1. INTRODUCTION**

Analysing the relationship between agricultural production and the natural environment (soil, water and air) and the unfavourable reactions between them became an important question in the second half of the 20th century both in Hungary and across Europe. Impairment of the environment is a complex process, it affects all the three basic components of the biosphere (soil, water, air) always at the same time, but the level of the effect is different for each part. Soils are threatened by two basic dangers: the various soil degradation processes (water and wind erosion, acidification, salinisation, physical and biological degradation, unfavourable changes in the humus content and a decrease in the buffer capacity) as well as pollution. In spite of all these, quality, functionality and productivity of soils can be preserved and maintained.

Within the framework of my dissertation I introduce the ongoing actions for the protection of the environment and soil in the United States and some countries of Europe, with special emphasis on Hungary.

The Soil Protection Information and Monitoring System (TIM) was created in 1991 as one of the operating subsystem of the Environmental Protection Information and Monitoring System, but its real operation began only in 1992. The TIM is the essential basis of rational agrienvironmental management and at the same time is an integrated unit of the environmental diagnosis of soils. Its operation in Hungary is obligatory under EU regulations. In addition, TIM could play an important role in the conservation and protection of soils.

Parameters to be examined within the TIM were determined only once when the basic conditions were recorded (soil forming rock, soil type, morphological description of soil profile, GIS coordinates). Based on the temporal variability of different soil features, laboratory analyses must be repeated annually, over three or six years.

The diagnostic net consists of three different types of sampling areas with 1236 points:

- national master diagnostic net (I-points, agricultural lands)
- special (S-points, degraded areas). Within these, D-points represent areas exposed to wind erosion and R-points represent areas exposed to water erosion
- E-points represent diagnostic points in forest areas

Researchers of the University of Debrecen Centre of Agricultural Sciences Department of Food Science and Quality Assurance analysed by ICP-OE spectrometer the samples of the TIM provided by the Plant and Soil Protection Service for 45 elements. I analysed the figures of the samples provided for me by the TIM database. The objective of the analysis was the extensibility of the element content measured at a certain point in the environment of the diagnostic point. On the basis of the results, I developed a statistical database that is suitable for calculating the quantity of the elements to a certain level of precision at a certain place. In order to do so, only the GPS coordinates of the point to be measured have to be known. The objectives of my research work were as follows:

- To develop a new computer based program which is suitable for receiving diagnostic figures from text files, carrying out rounding and integrating continual figures into a matrix.
- My software should be suitable for determining the distance between the diagnostic points based on GPS coordinates and then to calculate the element content on the basis of certain points located nearest to the diagnostic point.
- The software should give the relative error and confidence interval for each diagnostic point and element.
- The new, internet-based software should make it possible to set up a database based on the results of the statistical analysis, as well as to develop a two-level authorisation system and query options.

### 2. BASIS AND METHOD OF RESEARCH

### 2.1. Measuring the samples of the Hungarian Soil Protection Information and Monitoring System (TIM)

Within the TIM samples I analysed those that were taken in 1998 at 314 points in four counties (Figure 1). I used an OPTIMA 3300 DV type inductive coupling plasma optical emission spectrometer (ICP-OES) for the analytical determination.



**Figure 1** TIM diagnostic points in four counties (Borsod-Abaúj-Zemplén, Hajdú-Bihar, Heves, Szabolcs-Szatmár-Bereg)

I usually applied 2-points, sometimes 1-point, background corrections in the background measurements of the examinations carried out with the ICP-OES instrument. For the calibration points I usually adapted non-linear, in some cases linear, calibration functions. I used only the no. 1 database at each diagnostic point, since I wanted to determine results for the upper soil level (0-300 mm). I used the wet fracturing method (mixture of concentrated HNO<sub>3</sub> and concentrated H<sub>2</sub>O<sub>2</sub>) for preparing the soil samples. I saved the data measured by the ICP-OES instrument into a text file format database with "prn" extension.

### 2.2. Statistical boundary conditions

The statistical sample includes 314 points, if I include all of the diagnostic points (figures from four counties). The number of diagnostic points from agricultural areas only is 223. In practice both numbers of samples are considered as sufficient in statistical terms.

When determining the confidence interval I assumed that the basic population from which I took the samples had a normal distribution and I also knew the standard deviation (this was calculated before determining the confidence interval using a well known statistical method: equation 5). The object of the estimate is the prospective value of population, and the estimate function is the average of samples calculated from samples of identical distribution where the number of samples is "n".

From the point of view of realisation, the question is in which interval the prospective figure of the measured quantity will belong with the probability level of 95%. This interval is the confidence interval.

In most interval estimate methods – including the estimate of confidence interval – we look for a symmetric interval. This means that we look for a lower and upper line where the probability of being outside the interval is equal on both sides..

### 2.3. Conversion of the measured data by ICP-OES

The ICP-OES instrument averages the measured data and calculates the deviation for each element. The measured data have to be converted, rounded and placed into a table on the basis of a certain aspect for further processing. This process takes a long time in spite of the fact that a fixed Excel macro is available at the department. Processing of the databases takes at least one hour depending on the number of measured elements and samples.

I carried out the conversion, rounding and placing into table with Microsoft Visual Basic 6.0 developing system, since its objects and programming opportunities provided more possibilities than an Excel macro. I saved the figures received when running the program into an Excel worksheet in order to make further data processing easier.

# 2.4. Calculating distance, element content, relative error and confidence interval

After converting and rounding the measured data of the available TIM samples I carried out further calculations in order to determine the distance between the points, their element contents, their relative errors and confidence intervals. For the calculations I used the following connections:

The program calculates the distances by using the Pythagorean Theorem:

$$Z = \sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2} \tag{1}$$

where  $x_1$ ,  $y_1$  are the coordinates of the known point,  $x_2$ ,  $y_2$  are the coordinates of the

#### unknown point.

The software determines the calculated figure of chemical element content in case of 10 nearest neighbouring points by using the following connection:.

$$C_{x} = \frac{\frac{1/Z_{1} * C_{1} + \frac{1}{Z_{2}} * C_{2} + \dots + \frac{1}{Z_{10}} * C_{10}}{\frac{1/Z_{1} + \frac{1}{Z_{2}} + \dots + \frac{1}{Z_{10}}}$$
(2)

where  $Z_1$ ,  $Z_2$ ,... $Z_{10}$  are the distances of the known points correlated to the basis profile number,

 $C_1, C_2, \ldots C_{10}$  measured chemical element content in the known points.

The program calculates the percentage deviation – relative error – on the basis of the following formula:

$$Deviation = ABS\left(100 * \left(\frac{Sz - M}{M}\right)\right) [\%]$$
(3)

where ABS is the absolute value function

"Sz" means the element content calculated by me, "M" means the measured element content. The program invokes two Excel functions to calculate the confidence interval, one of them calculates the standard deviation ( $\sigma$ ), the other one calculates the confidence interval. To calculate the confidence interval, it calculates the standard deviation first ( $\sigma$ ):

To calculate the confidence interval, it calculates the standard deviation first ( $\sigma$ ):

$$\sigma = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \overline{x})^2}{n - 1}}$$
(4)

Formula of the confidence interval:

$$\overline{x} \pm 1,96\left(\frac{\sigma}{\sqrt{n}}\right)$$
 (5)

### 2.5. Relative error-neighbouring point functions

I presented the relative error figures calculated for the elements considering the different number of neighbouring points by diagrams.

I carried out two different kinds of analysis. In the first case I considered all the available points (314) from the four counties (all\_1level), while in the other case I examined only those points (223) that were located in agricultural areas (I\_marked\_1level). I used only the no. 1

database (1level), since I wanted to determine results only for the upper soil level (0-300 mm).

I determined the average relative errors from the entire available calculated database. When running the program and calculating the relative error, I considered the 1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30 nearest points around the selected points.

### 2.6. Applied software

I considered the following aspects when addressing the task:

- Firstly a reliable web server is needed since it is a web-based solution. On the basis of users' statistics Apache is the most suitable for this.
- A stable relational spreadsheet is also needed which will handle data and also provide data for the query system. Since I aimed to apply standard solutions, I needed an SQL-based system and finally chose MySQL.
- A kind of programming language is also needed for the realisation in which the clientside and server-side programmes will be prepared. From the programming language options I chose Javascript and PHP, which are the most commonly used and with which I am familiar.

# 2.7. Database management and preparation of a dynamic website in order to calculate the unknown element content at a certain point

The analyses, based on the results of the above mentioned executing of programmes, made it possible to arrange the new results into a database. My objective was to prepare a website for the users which together only with the GPS coordinates of the certain point could provide an estimate of the element content for the measured elements at points where no samples had been taken and thus no measuring could have been carried out. The relative error and confidence interval for each element content figure have to be provided of course.

I applied the MySQL spreadsheet to prepare the database, since I want to allow access to the newly developed database via internet technology.

I filled up the data to the data tables mainly from the Excel tables created when running the programme called "Minta" (SAMPLE).

Data in internet-based databases must be properly protected. The program can provide access for two types of users at present: *the database administrator* who is authorised to do everything in connection with the database, and *the user* who is authorised to make queries only on the basis of GPS coordinates in connection with the selected element content of the certain sample.

I inserted a general query module into the program with which optional queries can be made from the database.

### 2.8. Structure of the available "TIM database"

Modules of the TIM program package – for which the program language is FOXPRO – are the following:

• Database filling and maintenance module

It provides the opportunity to enter data (soil physical, soil chemical, underground water, etc.), modify data, delete single or grouped data and extend the database.

• Query module

This module helps the preparation of statistics describing the database at national, regional and county level, as well as on the diagnostical site level.

• Mapping module

This module ensures data transmission towards the ARCView and ARC/INFO geographical information systems.

• Service module

This module ensures menu-lead archiving and transmission of data under optionally selected query conditions in Dbase format to other users.

Data tables available for us from the total TIM database are the following:

- GPS data table
- HELYSZIN (LOCATION) data table
- TALAJTIPUS (SOIL TYPE) data table
- ALAPKO (BASIC ROCK) data table
- TIM data table

I used for my programme form the "GPS" data table the JEL (SIGN), SORSZAM (SERIAL NUMBER), MEGYEKOD (COUNTY CODE) (compound key) and the X and Y fields (GPS coordinates); from the "HELYSZIN" (LOCATION) data table I used the "JEL" (SIGN), "SORSZAM" (SERIAL NUMBER), "MEGYEKOD" (COUNTY CODE), "ALAPKO" (BASIC ROCK) and "TLJ\_TIP" (SOIL TYPE) fields; from the "TALAJTIPUS" (SOIL TYPE) and "ALAPKO" (BASIC ROCK) data tables I used the "NEV" (NAME) fields. I used the "TIM" data table to compare data measured by the ICP-OES instrument where possible.

### 3. RESULTS OF THE EXPERIMENTS AND THEIR EVALUATION

### 3.1. Conversion of the measured results of the ICP-OES

I developed new software that makes it possible to quickly arrange the ICP data into a matrix and to estimate the element content at a certain point on the basis of the data of the neighbouring points. There are some parts within the program that had been developed especially for processing TIM data (calculation of distance, element content, standard deviation and confidence interval), but there also some parts (rounding, matrix preparation) which can be also used to evaluate optional samples.

One diagnostic data line often includes hundreds of samples with 45 measured elements per sample. However among the measured elements I obtained a concentration value above the demonstration line only in the case of 21 samples. Originally, processing of data lines was carried by a fixed Excel macro, but with even this method it took at least one hour depending on the number of measured elements and samples. By using the program developed within the framework of my PhD dissertation the same process takes only a few minutes.

### 3.2. The developed method for estimating element content

I developed a method to estimate the element content. During the process I chose an optional point that will be considered as unknown in the process (in Figure 2, I marked this point with "U"). I have the measured results for the unknown point, but I did not count with them, I proceeded as if I did not have those measured results.

Around the unknown points I determined the nearest ten points (this number can be changed in the program). I marked these nearest points in Figure 2 with "K".

The distance between the known points (K) and the point considered as unknown (U) can be calculated by using the Pythagorean Theorem (equation 1):

When I determined the distance between all the ten known and "unknown" points, I had ten distance data  $z_1, z_2, z_3, \dots, z_{10}$ , from which with linear estimation by using equation 2, concentration ( $c_x$ ) of the certain element can be estimated even in those places where there is no TIM diagnostic point.



Figure 2

K = Known diagnostic points (x1,y1: GPS coordinates of the given place)
U = diagnostic point to be determined (unknown)
(x2,y2: GPS coordinates of the "unknown" place )

If I calculate the concentration  $(c_x)$  of the certain element for the unknown point, I can compare it with the measured data by ICP-OES spectrometer for the certain element and I can determine the relative standard deviation (equation 3).

These steps have to be done first for the same point regarding the other elements which quantities exceed the demonstration line. After that, these steps have to be done again for each available TIM diagnostic point, considering that they are the unknown points in the experiment. After this, I will have the concentration value and its relative standard deviation for each measurable element above the demonstration line for each diagnostic point.

I applied two Excel functions to calculate the confidence interval, one of them (equation 4) calculates the standard deviation ( $\sigma$ ) and the other calculates the confidence interval (equation 5).

I visualised the received data on Excel worksheets in each case to make further data processing easier.

# 3.3. Comparison of the average values of relative error received from diagnostic points marked with I and (I-, E-, R-, S-)

On the basis of the calculations carried out by my program and the figures prepared for certain elements, I reached the following conclusion in the case of the major proportion of examined elements. The average value of relative error is higher (67% as an average) in the case of all the nearest neighbouring points (1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30) if I consider all types of diagnostic points (marked with I-, E-, R- and S) than (42% as an average) if I consider only the diagnostic points marked with I (agricultural lands). The reason for that is that in the first case extreme data coming from diagnostic points on polluted areas increase the value of relative error. Sodium and Strontium are exceptions to this conclusion (the order changes only above ten neighbouring points) as for some diagnostic points marked with I, I obtained higher values than if I would have considered all types of diagnostic points.

# 3.4. Percentage deviation of relative errors at diagnostical points marked with I- and (I-, E-, R-, S)

In the following experiment series I studied the deviation of average relative errors for each diagnostic point and for those marked with I and denoted the differences between them in as percentages. I discovered that the elements can be ranged into four zones ranging 15-15% each (Table 1).

1-15%	16 - 30%	31 - 45%	46-60%
Al	K	Ca	Pb
Fe	Mn	Sr	
Na	Ni	Ti	
Mg	Cu		
Ba	В		
Р	Zn		
S	Cr		
V	Со		
Y			

 Table 1. Difference between the average values of relative error of certain elements in percentage for each diagnostic point and for those marked with I

It is obvious from the results introduced in point 3.3. that the relative error is smaller than if I consider samples marked with I (agricultural areas) only, thus I examined hereafter only these.

# 3.5. Determining the number of nearest neighbouring points to be considered in the case of certain elements

I examined hereinafter different numbers of nearest neighbouring points (1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30). Determining the minimums of the average values of relative error for elements (21) measured above the demonstration line, on the basis of the prepared figures, they can be arranged into three groups (Table 2).

3 point	5 point	10 point
К	Al	Na
Р	Fe	
Sr	Са	
Ni	Mg	
Cu	Mn	
В	S	
Со	Ba	
Ti	Cr	
	V	
	Pb	
	Y	
	Zn	

Table 2. Minimums of the average values of relative error for certain elements

It means that in case of a sample originating from a genuinely unknown diagnostic point, for elements in the first column 3, for elements in the second column 5, while for Sodium 10 nearest neighboring points have to be considered in order to estimate the element content with the smallest error.

# 3.6. Further reduction of the averages of relative error with mathematical method

Since after the first examinations I had the average values of relative errors for the certain elements in each calculated nearest diagnostic point, I investigated whether these values could be further reduced and the confidence interval of calculated element content thus improved using any mathematical method. For this, I examined the values of relative error for 1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30 nearest diagnostic points in the case of each profile, then selected and recorded these values. I averaged the received minimum values for the profiles and found that in comparison with the previous results the value of relative error can be decreased by an average of 40% or so. In Table 3 the minimums of the average value of relative error and the average error values that had been reduced by the method introduced by myself are shown. The difference of 40% (44% as an average) for the benefit of the latter method is evident from the table.

Element	Minimum of the relative error values	Averages of the minimums of the selected profile number	Decrease in percentage
Al	32	19	41
Fe	19	11	42
Ca	85	42	51
Na	57	30	47
K	43	25	42
Mg	22	13	41
Ti	69	36	48
Р	37	20	46
Mn	41	24	41
S	35	21	40
Ba	34	21	38
Cr	46	16	65
Sr	47	29	38

Table 3. Minimums of the relative error values of certain elements, averages of the minimums of the selected profile number and their ratio in percentage

Element	Minimum of the relative error values	Averages of the minimums of the selected profile number	Decrease in percentage
Ni	39	20	49
V	43	26	40
Cu	55	31	44
В	28	15	46
Co	43	26	40
Pb	50	29	42
Y	27	17	37
Zn	34	19	44

Continuation of Table 3

Together with the newly reached decrease with 40%, the relative error can be reduced to 23% as an average.

### 3.7. The Internet-based program

I developed a newer internet-based program that makes it possible to reach the objectives through arranging the results of analyses into a database and developing an authorisation system. When developing the newer software, my objective was to create a dynamic website for users which together with the GPS coordinates only of the certain point could provide the estimated element content for the measured chemical elements also in such points where no samples had been taken and thus measured data were not available. The relative error and the confidence interval must be provided for each element content value, since they orientate the users into which range the quantity of the certain chemical element at the given diagnostic point can fall.

I provided access to the database through internet-based technology. With the help of the database-based server-side script language (PHP) I planned and developed the user interface, through which the user can communicate with the program.

Through the authorisation system, the authorisation level of each user can easily be determined. In case of database administrator authorisation, almost any kind of query can be made in connection with the database through the general query module.

#### **NEW SCIENTIFIC RESULTS**

- 1. I developed new software that makes it possible to receive measured data from a text file and, after the necessary rounding, arranging the linear data into a matrix. These processes result in a considerable saving of time in comparison with the previous processing method with a built-in Excel macro. The program ensures the realisation of the final objective.
- 2. When statistically processing the measured data of TIM soils I discovered that the average values of relative error is higher in the case of all the nearest neighbouring points (1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30) if I considered all types of diagnostic points (marked with I-, E-, R- and S) than if I considered only the diagnostic points marked with I (agricultural lands). Sodium and Strontium are the only exceptions to this conclusion. It follows that other points which reduce the homogeneity of the aggregation should not be taken into consideration.
- When studying the standard deviation of average relative errors of TIM samples for all types of diagnostic points and for those marked with I, and denoting the differences between them in percentage, I discovered that the elements can be ranged into four zones ranging 15-15% each.
   1-15 %: Al, Fe, Na, Mg, Ba, P, S, V, Y

16-30 %: K, Mn, Ni, Cu, B, Zn, Cr, Co 31-45 %: Ca, Sr, Ti

- 46-60 %: Pb
- 4. I discovered when studying different numbers (1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30 in order) of nearest neighbouring diagnostic points within the aggregation, the minimums of the relative error values are different. Elements can be ranged into three groups depending on how many nearest neighbouring diagnostic points were considered when I received the minimum value.

3 neighbouring diagnostical points: K, P, Sr, Ni, Cu, B, Co, Ti

5 neighbouring diagnostical points: Al, Fe, Ca, Mg, Mn, S, Ba, Cr, V, Pb, Y, Zn

10 neighbouring diagnostical points: Na

- 5. I examined the received values of relative error for the nearest diagnostic point (1, 2, 3, 4, 5, 6, 7, 10, 15, 20, 25 and 30) in the case of each diagnostic point. I averaged the received minimum values for the profiles and found that in comparison with the previous results the value of relative error can be decreased by a further 40% as an average (to 23% as an average).
- 6. I developed a newer internet-based program that makes it possible to reach the objectives by arranging the results of analyses into a database and developing an authorisation system. The statistical-based database is suitable to estimate the chemical element content of the sample at a certain point with a certain precision and for this only the GPS corrdinates of the point are needed. When developing the database I used the results introduced in point 4 of the present thesis.

### Practical applicability of the program

Based on available data, the developed program makes it possible to estimate element content at a certain diagnostic point with some statistical errors; it can also be applied in analysing effects of environmental pollution.

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