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SUSCEPTOMETER IN SOIL MAGNETIC SUSCEPTIBILITY STUDIES

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Abstract

This work presents a method for measuring the magnetic susceptibility of soil samples based on interactions of magnetic particles contained in a tested sample with a weighed permanent magnet placed in the balance mechanical design. The MYA 2.4Y microbalance manufactured by Radwag Wagi Elektroniczne, Poland, was used to perform mass measurements. The weighing system was adjusted for mass indication using a certified mass standard, and for magnetic susceptibility indication using a certified magnetic susceptibility standard. The volume of each analysed soil sample was 3.93 cm^3 and was similar to the volume and the size of the magnetic susceptibility standard. The research was carried out for 10 soil samples with a magnetic susceptibility range varying from 20 to 1600×10^{-8} m^3kg^{-1}. The soil samples contained technogenic magnetic particles and particles of natural magnetite of geogenic origin. The study was performed for a field of 2 mT.

Keywords: magnetic susceptibility, soils, analysis, mass measurement.

1. Introduction

Numerous important types of soil contaminants are toxic, or *potentially toxic, trace elements* (PTEs). These elements (mainly metals) emitted into the air by ironworks, non-ferrous metal smelters, cement plants, power stations and coking plants, are deposited in topsoil then absorbed by plants, and finally enter the food chain. This transfer of pollutants poses a real environmental threat not only in the form of excessive *particular matter* (PM) [1], but also as absorption of heavy metals into the human food chain. It is one of many negative factors determining the quality of life and health of human populations living in industrialized areas [2].

The co-occurrence of soil magnetic and geochemical anomalies of anthropogenic origin has been confirmed by numerous researchers [3–9]. The use of soil in situ magnetometry, according to [10], enables accurate identification of sites of soil magnetic anomalies. These locations are characterised by a high probability of exceeding the permissible contents of certain metals and

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metalloids, and require preventive surveillance [11, 12]. Magnetic susceptibility tests can also be carried out under laboratory conditions where more detailed analysis of the material collected can be carried out. Such a test cycle includes a well-defined sampling process [13], which is particularly important in this type of research. It should be noted that accurate information on the current state of environmental pollution to heavy metals is essential for initiating and indicating corrective measures. They constitute the basis of well-functioning quality management systems [14] and one of essential elements of risk management [15].

In general terms, magnetic susceptibility defines the ability to change the magnetisation that results from an external magnetic field. Practically, it is an indicator of the degree of soil contamination, but also a critical value for the applicability of steel mass standards in legal metrology [16]. Regardless of the area in which magnetic susceptibility is used, it is a parameter that describes the magnetic property of matter. This value is directly proportional to the content of magnetic particles in the investigated sample [17].

The magnetic susceptibility (κ) is directly proportional to volume magnetisation (M) that is induced in the material, and inversely proportional to magnetic field strength (H) that induces this magnetisation. This relationship is presented in (1).

$$\kappa = \frac{M}{H}, \tag{1}$$

where: M – volumetric magnetisation of the medium [A/m], H – magnetic field strength [A/m].

The magnetic properties of loose samples can be defined by the mass magnetic susceptibility. In this case, accurate information on the mass and density of the test sample is required. Statistical analysis of the results can then take into account not only the geographical variation of the soil sampling sites, but also their varying density. The mass magnetic susceptibility (χ) is determined by the ratio of the volume magnetic susceptibility κ to the specific density (ρ) of the substance (2).

$$\chi = \frac{\kappa}{\rho} \quad \left[\frac{\mathrm{m}^3}{\mathrm{kg}}\right], \tag{2}$$

where: χ – specific magnetic susceptibility, κ – volume magnetic susceptibility [–], ρ – sample density [kg/m^3].

2. Magnetic susceptibility – test methods

Magnetic susceptibility measurements can be performed in situ, where apparent magnetic susceptibility is measured, or in the laboratory. Field magnetometer tests are important for assessing the impact of linear sources of pollution such as roads and point sources (industrial plants) on forest and urban ecosystems. The deposition of harmful elements (copper, zinc, cadmium, lead, chromium and nickel) in the environment is the result of the use of motor vehicles (combustion, abrasion, corrosion), but can also be the result of the use of post-industrial waste in the process of building road infrastructure.

The test of in situ magnetic susceptibility of forest areas with long-term undisturbed deposition of pollutants also allows for correct interpretation of the origin of pollution sources (anthropogenic or natural). Usually, MS2 series meters equipped with the MS2D probes are used in field studies.

The idea of measuring magnetic susceptibility of samples of solid or liquid structure is to determine the degree of interaction of magnetic particles contained in the sample structure with the measuring element, which is usually a system of induction coils supplied with a current of variable frequency of several or several thousand Hz. The value measured is the inductance of the coil, which depends on the number of magnetic particles and on the homogeneity of the

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test sample. Such a measuring system is used in accurate laboratory tests, *e.g.* the Bartington MS2 meter equipped with the MS2B dual frequency sensor (Fig. 1). The test sample is placed in a so-called measuring cell (Fig. 1 – item 2). This is important when assessing the degree of environmental degradation as a result of industrial activity.

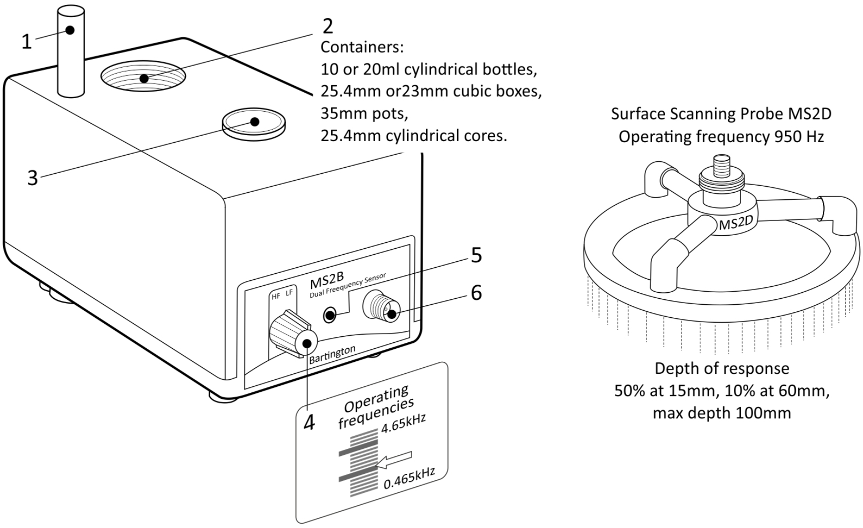

Fig. 1. Bartington MS2B dual frequency magnetic susceptibility sensor: 1 – sample insertion mechanism & height adjustment screw; 2 – sample cavity; 3 – flat bladed adjuster tool; 4 – operating frequency selector; 5 – HF calibration screw; 6 – TNC connector for connection with the MS2 meter.

A similar principle is used in measuring devices called Kappameters, where oscillations of an oscillator dependent on an induction coil with which magnetic particles contained in the sample are measured. When the sample is free of magnetic particles, the oscillator's vibrations depend only on the magnetic permeability of the air. The measurement yields an accurate value of magnetic susceptibility, assuming thermal stability of the oscillator system.

The shift of frequency is converted to magnetic susceptibility. With the latest version of the MFK1-FA Multi-Function Kappabridge, the measurements of κ can be carried out at three measuring frequencies: 976 Hz, 3904 Hz and 15616 Hz.

Since during in situ field measurements of magnetic susceptibility fast measurement is expected, the assessment only concerns the concentration of ferrimagnetic materials (MS2D probe, Fig. 1). Such a test does not allow the identification of ferrimagnetic grains in the vicinity of a superparamagnetic/stable single domain transition, as in the MS2B gauge. It should be noted, however, that magnetic susceptibility is only a parameter to assess the quantitative content of magnetic particles in a sample and, possibly, the proportion of superparamagnetic grains. Interpretation concerning the rock weathering process, the study of sediments or PM sources require the study of the entire soil core and more advanced magnetic measurements, *e.g.* magnetic hysteresis parameters.

3. Frequency dependence of magnetic susceptibility

High magnetic susceptibility is an effect of the presence of ferrimagnetic particles such as magnetite, maghemite (geogenic or anthropogenic origin) or superparamagnetic grains (of pedogenic or biogenic origin) in the tested sample. The origin of the magnetic signal can be estimated by testing the same sample at two different frequencies. The value of the magnetic susceptibility frequency factor (χ_{fd}) is expressed by (3).

$$\chi_{fd} = \frac{\kappa_{lf} - \kappa_{hf}}{\kappa_{lf}} \cdot 100\%, \tag{3}$$

where: κ_{lf} – volume magnetic susceptibility measured at low frequency, κ_{hf} – volume magnetic susceptibility measured at high frequency.

The value of the magnetic susceptibility frequency factor determines the percentage decrease of magnetic susceptibility when measuring susceptibility at different frequencies [18]. If the χ_{fd} factor value is greater than 5%, it means that superparamagnetic grains (usually of natural origin) are present in the soil in considerable amount. A value below 5% suggests that the proportion of superparamagnetic grains is small and that the magnetic signal in the tested sample comes from *multi-domain* (MD) or *single-domain* (SD) magnetite. A very low χ_{fd} value (< 2%) is characteristic for technogenic MD magnetic particles, but also for natural MD magnetite derived from weathering of the bedrock [19, 20].

4. Material and methodology

Because it was impossible to obtain standards with known magnetic susceptibility for soil material ten archival soil samples were selected for testing. Their magnetic susceptibility had previously been measured by means of the most commonly used measuring equipment, the MS2B Bartington system with measurements carried out at the frequencies of 470 and 4700 Hz, and the MFK1-FA Kappabridge by AGICO – at the frequencies of 976 and 15 616 Hz. Soil samples were taken from regions with different industrial and urban dust deposition rates, containing both technogenic magnetic particles and particles of natural magnetite of geogenic origin (Table 1).

Table 1. Geographic location of soil samples, position in the soil profile and description of a potential source of a magnetic signal.

No	Sample location	GPS coordinates	Depth	Soil horizon	Potential source of magnetic signal	Distance from the source
R 1	Mo I Rana Nordland county (northern Norway)	66.3146 N 14.2664 E	5–10 cm	mineral	iron- and steelworks	5 km
R 2	Strzelce Opolskie (Opole Province, southern Poland)	50.5355 N 18.2991 E	5–11 cm	organic	cement plant	1 km
R 3	Orzesze (Silesian Province, southern Poland)	50.1241 N 18.8076 E	3–12 cm	organic	steelworks and power plant	2 km
R 4	Dąbrowa Górnicza (Silesian Province, southern Poland)	50.3445 N 19.3199 E	0–7 cm	organic	steelworks	0.5 km
R 5	Ozimek (Opole Province, southern Poland)	50.6744 N 18.2152 E	15–25 cm	anthropogenic horizon	old ironworks	0.5 km
R 6	Dzięgielów (Silesian Province, southern Poland)	49.7229 N 18,7188 E	2–11 cm	organic	ironworks	5.5 km
R 7	Ozimek (Opole Province, southern Poland)	50.6740 N 18.2144 E	7–16 cm	anthropogenic horizon	old ironworks	0.5 km
R 8	Lyngdal (Agder county, southern Norway)	58.2376 N 7.1680 E	20–25 cm	mineral	natural (geogenic)	0 km
R 9	Lyngdal (Agder county, southern Norway)	58.2376 N 7.1696 E	5–10 cm	mineral	natural (geogenic)	0 km
R 10	Lyngdal (Agder county, southern Norway)	58.23746 N 7.16917 E	7–13 cm	mineral	natural (geogenic)	0 km

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5. Gravimetric method for magnetic susceptibility testing – working principle

The idea of the gravimetric method was to use the gravitational interaction force that occurs when determining the mass of a sample, and the magnetic interaction force that occurs between the magnetic particles in the sample structure, and the magnet placed in the structure, all of this in a single measuring instrument. This approach makes it possible to determine the volume magnetic susceptibility and the specific (mass) magnetic susceptibility in a single measurement cycle. A diagram of such a measuring system is presented in Fig. 2.

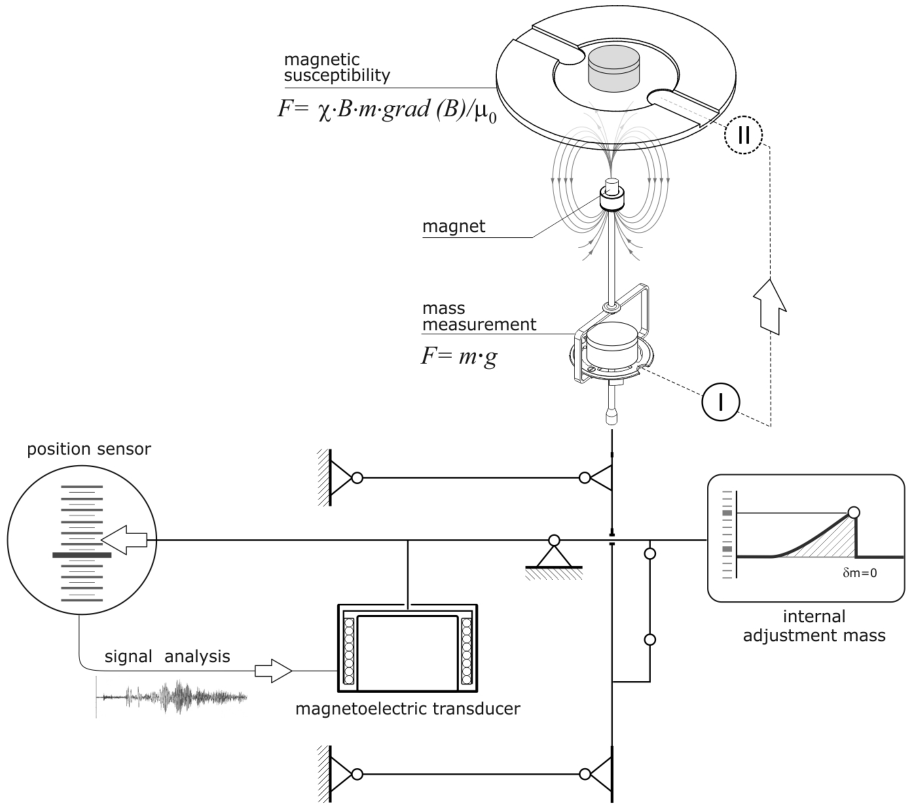

Fig. 2. Technical scheme of MYA 4.Y microbalance with equipment for magnetic susceptibility measurements.

"The idea of balance-based susceptometer for soil samples and operation guide is available on RADWAG website [21]. The aim of present paper is to make profound metrological verification of the method and the device".

The principle of measuring the mass of a sample is the same as for most electronic balances – the force ($F = mg$) of gravity is measured. The internal measuring resolution of the weighing system was over 50 million elementary units, the elementary unit for mass measurement was 1 µg. The accuracy of the measurement was guaranteed by an internal automatic adjustment.

The magnetic susceptibility of the sample was measured when the sample was placed on a fixed platform (II). Below the platform was the source of the magnetic field – a magnet with a dipole moment of 0.0025 Am^2. When magnetic particles were present in the sample structure, a change in the indication of the weighing system was recorded as a result of magnetic interaction between the magnet and the sample. Taking into account the geometric dimensions of the sample, its distance from the magnet and the indication of the measuring system, the volume and mass-specific magnetic susceptibilities were calculated.

6. Magnetic susceptibility test kit design

The design of the magnetic susceptibility test kit allows for testing changes in mass and magnetic susceptibility of soil samples placed in containers with an external diameter of 40 mm, at a volume of 3.93 cm^3. It is also possible to measure the magnetic susceptibility of a medium absorbed through filters with a maximum diameter of 47 mm as well as test other samples with solid or liquid structures to detect the presence of magnetic particles. Geometrical dimensions of the measuring system are presented in Fig. 3.

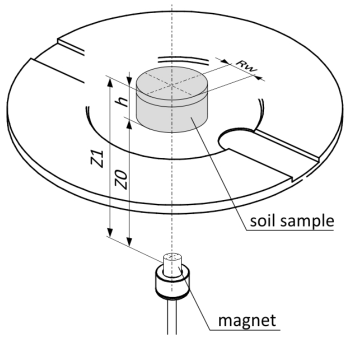

Fig. 3. Dimensions of the measuring system. $Z0$ – distance from the center of the magnet to the bottom of the sample, $Z1$ – distance from the top of sample to the center of the magnet, h – sample height, Rw – radius of the soil sample.

Figure 4 contains a graphic visualization of the separating elements of the magnetic susceptibility test kit. The kit is used in the mechanical design of the MYA 21.4Y microbalance by Radwag Wagi Elektroniczne, Poland.

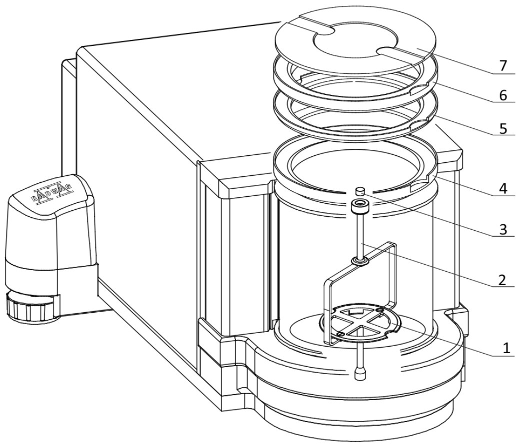

Fig. 4. Technical scheme of the set for magnetic susceptibility measurements. 1 – microbalance dish for mass measurement of analyzed samples; 2 – magnet holder; 3 – magnet; 4 – lower locating ring; 5 – spacer ring 1; 6 – spacer ring 2; 7 – upper cover of the weighing chamber, platform for magnetic susceptibility measurement.

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7. Test procedure – sample preparation

The sample shall be mixed to produce a homogeneous mixture before being placed in the container. For samples of variable texture, where homogeneity is difficult to achieve, it is recommended that tests are carried out for a series of samples. The mixed sample shall be placed in a plastic container. Some excess of the sample portion can be light compressed (Fig. 5) and tightly closed in the vessel. A correctly prepared sample should fill the entire volume of the container.

Fig. 5. Sample in a measuring dish.

8. Test procedure – mass measurement

After installing the magnetic susceptibility test kit (Fig. 4), perform the adjustment of the balance indication. Load the weighing pan with the sample (Fig. 6) and read the weighing result.

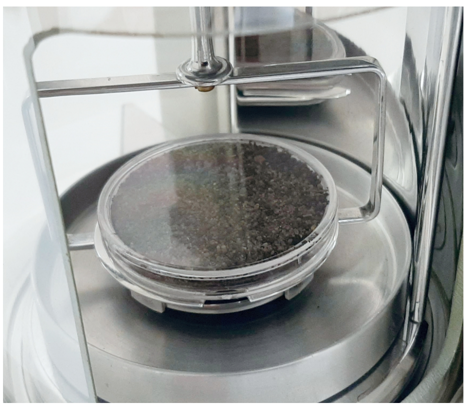

Fig. 6. Soil sample during the weighing process.

Based on the measurement of the sample net mass and its known volume of 3.93 cm^3, the density of the test sample shall be calculated using (4). It will be essential for the calculation of the mass-specific susceptibility following (2).

$$\rho = \frac{m}{V} \quad \left[\frac{\text{kg}}{\text{m}^3}\right], \tag{4}$$

where: ρ – sample density, m – sample mass, V – sample volume.

9. Magnetic susceptibility adjustment and measurement of magnetic susceptibility of samples

The adjustment was carried out with a cylindrical magnetic susceptibility standard No. 10090420: $d = 40$ mm, $h = 1.5$ mm, HE210 steel, by Häfner Gewichte GmbH. The certified magnetic susceptibility value was $397 \times 10^{-5} \pm 40 \times 10^{-5}$. The adjustment of magnetic susceptibility of the measuring system was achieved by introducing an adjustment factor. The task of the adjustment factor was to correct the indication of the magnetic susceptibility of the standard in such a way that the corrected magnetic susceptibility would be the same as the value included in the calibration certificate. The measurement precision of magnetic susceptibility of the standard, expressed by the standard deviation, was 702×10^{-7} with expanded uncertainty of $U = 152 \times 10^{-6}$. The adjustment error, measured as the difference between the certified value and the average susceptibility value obtained during the susceptibility standard test, was 4.60×10^{-5}. The magnetic field during testing was 2mT, measured at the base of the standard with an LZ-641H gaussmeter made by Enes.

Prior to the magnetic susceptibility test, the test parameters such as the distance between the sample and the magnet, and the geometric dimensions of the sample are be determined. These values are set in the microbalance menu:
- Distance: 20 mm,
- Sample height: 3.13 mm,
- Sample diameter: 40 mm.

Selection of a specific distance between the sample and the magnet is realized by means of a suitable spacer ring (Fig. 4). This results in altering the value of the magnetic field that interacts with the magnetic particles in the sample structure. The determination of magnetic susceptibility takes place automatically when the soil sample is placed on the upper platform of the kit (Item 7, Fig. 4). As a result of the interaction of the magnetic particles of the sample with the magnet (Item 3, Fig. 4), a mass indication is obtained which is used in the calculation of the volume and mass magnetic susceptibility (Fig. 7).

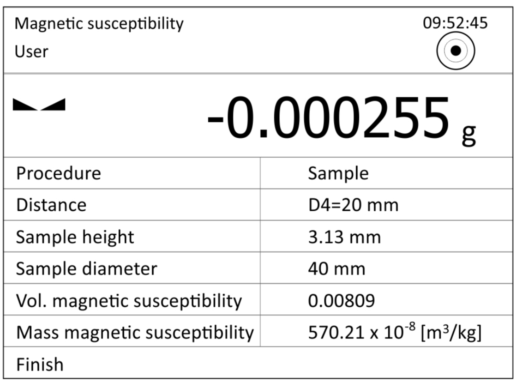

Fig. 7. Balance display during the measurement of magnetic susceptibility.

The volume magnetic susceptibility was calculated according to the OIML recommendations concerning testing weights [16]. Relation (5), given below, is valid for cylindrical samples provided that geometrical corrections are made.

$$\kappa = \frac{F_a}{I_a \cdot F_{\max} - 0.4F_a}, \tag{5}$$

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where: κ – volume magnetic susceptibility, F_a – force of sample/magnet interaction, I_a – geometric correction factor, $F_{\max}$ – maximum force of sample/magnet interaction.

$$F_{\max} = \frac{3\mu_0}{64\pi} \times \frac{m_d^2}{Z_o^4}, \tag{6}$$

where: μ_0 – magnetic permeability of vacuum, m_d – magnet dipole moment, Z_0 – distance from the magnet centre to the sample bottom.

The mass-specific magnetic susceptibility was calculated in accordance with (3).

10. Test results

The tests were carried out for 10 soil samples, whose magnetic susceptibility in the first stage was determined using the MS2B Bartington and MFK1-FA Kappabridge AGICO equipment (Table 2).

Table 2. Magnetic susceptibility of soil samples measured at two frequencies by two different instruments: Bartington MS2B and MFK1-FA Multi-Function Kappabridge.

Sample no.	**Sample mass (g)**	κ **(×10^{-5})**	**Bartington MS2 B**			**Agico MFK1**		
			χ_{LF}	χ_{HF}	χ_{fd}	χ_{LF}	χ_{HF}	χ_{fd}
			×10^{-8} m^3/kg		**%**	**×10^{-8} m^3/kg**		**%**
			470 Hz	**4700 Hz**		**976 Hz**	**15616 Hz**	
R 1	9.9	25.68	19.00	18.83	0.9	20.828	19.938	4.3
R 2	10.17	58.24	43.50	42.50	2.3	44.588	42.462	4.8
R 3	11.58	150.60	104.8	103.33	1.4	109.28	104.98	3.9
R 4	10.22	175.83	128.0	127.67	0.3	132.98	128.78	3.2
R 5	12.56	513.69	315.5	304.83	3.4	334.96	317.90	5.1
R 6	10.39	564.12	415.0	393.67	5.1	422.80	389.98	7.8
R 7	11.16	760.99	531.7	518.33	2.5	545.36	522.22	4.2
R 8	11.55	1470.42	1031.3	1019.2	1.2	1060.6	1029.0	3.0
R 9	10.09	1398.36	1105.3	1103.2	0.2	1144.0	1123.0	1.8
R 10	10.99	2316.90	1603.0	1589.2	0.9	1665.4	1627.0	2.3

These values were used at a later stage as a reference for the magnetic susceptibility results obtained with the use of the weighing method. A view of the sample during testing is presented in Fig. 8.

Due to the character of the test method that uses weighing device as an indicator of magnetic susceptibility, the main sources of potential error were specified in the first stage of the research.

a. *Sample height*
   The height of the sample is equal to the inner height of the container. However, due to the loose structure of the sample and the heterogeneity of the grains, it can be assumed that the maximum error in sample positioning (that can be observed by the operator) is about 2 mm. This results in an error in the determination of the magnetic susceptibility of approximately 2.3%.

b. *Sample diameter*
   The influence of a sample diameter error of approx. 2 mm on the volume magnetic susceptibility is 14%.

Fig. 8. Balance during the measurement of magnetic susceptibility.

c. Sample/magnet distance
A sample/magnet distance error of 2 mm results in an error in the determination of the volume magnetic susceptibility of 42% when the measured value is smaller than the real value, and 66% when the measured value is larger than the real value. The error magnitude in measuring the sample/magnet distance $Z0$ (Fig. 3) is influenced by the accuracy of measuring this value, but also by the unambiguous position of the magnet in the holder, and its position in space when the balance is restarted.

d. Dipolar moment of magnet
An inaccuracy of 0.0025 (Am^2) in determining the dipole moment of magnet results in an error of 4% in determining the volume magnetic susceptibility.

e. Sample magnetic heterogeneity i.e. The uniformity of magnetic particles distribution in the spatial structure of the sample.

11. Sample heterogeneity assessment

Potential errors included in points *a–d* may result from negligence at the stage of preparation for tests. Therefore, they should be minimized. The error resulting from sample heterogeneity is a priori unknown, however, its value can be estimated by further examination. The homogeneity degree of the magnetic particles distribution in the test sample is evaluated by testing the sample in the reference position and after rotating it by 180°. With regard to this, two test hypotheses can be formulated:

- Hypothesis 1: the sample is homogeneous,
- Hypothesis 2: the sample is heterogeneous.

Obtaining similar volume magnetic susceptibility results for the sample in the reference position and after its rotation by 180° supports the Hypothesis 1. Otherwise, the alternative one should be accepted. In the performed tests each sample was divided equally into two identical containers marked P1 and P2. It can therefore be concluded that the preparation of the sample

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for testing was the same for both of them. The results obtained during the tests are presented in Table 3. The studies were performed under a magnetic field of 2 mT measured with a kit presented in Fig. 4.

Table 3. Homogeneity of volume magnetic susceptibility for MYA 2.4Y measurements. P1 – container 1; P2 – container 2; P1R – container 1 rotated by 180°; P2R – container 2 rotated by 180°; x – no sample/magnet interaction.

Sample no.	κ ($\times 10^{-5}$)				$\bar{x}$	$\delta\kappa$ (%)			
	P1	P2	P1R	P2R		P1	P2	P1R	P2R
1	16	32	25	22	23.75	–32.63	34.74	5.26	–7.37
2	82	54	63	54	63.25	29.64	–14.62	–0.40	–14.62
3	161	161	142	146	152.5	5.57	5.57	–6.89	–4.26
4	199	190	190	164	185.75	7.13	2.29	2.29	–11.71
5	1010	475	x	437	x	x	x	x	x
6	697	659	695	573	656	6.25	0.46	5.95	–12.65
7	596	1009	383	809	699.25	–14.77	44.30	–45.23	15.70
8	1485	1408	1303	1283	1369.75	8.41	2.79	–4.87	–6.33
9	1689	1686	1523	1555	1613.25	4.70	4.51	–5.59	–3.61
10	2447	2528	2206	2235	2354	3.95	7.39	–6.29	–5.06

For each sample, the percentage error of volume magnetic susceptibility determination was evaluated with regard to the mean value. The largest discrepancies between the results were found for samples 1, 2 and 7. For sample 1 placed in containers P1 and P2, greater κ spread from the mean value was found (over 30 %) when the test was performed in the reference position. For the same sample tested after rotation by 180°, the discrepancy did not exceed 8 %. Similar relationships were found for sample 2, however, in this case, the best κ similarity to the mean value was obtained only for the sample placed in container P1 when the test was performed after rotating the container by 180°. For sample number 7, irrespective of the container number (P1, P2) and test position, significant discrepancies in κ results were obtained, ranging from 14.77% to 45.23%.

The distribution of magnetic particles in sample 5 placed in container P1 was extremely heterogeneous. The volume magnetic susceptibility result for the reference position was 1010, and no sample/magnet interaction was observed when this sample was tested in the rotated position (180°). From the obtained results it can be assumed that in this case the magnetic particles were located only in the lower part of the container P1. The test of sample 5 in container P2 in the reference position and after rotation gave results of 475 and 437 respectively, which allows us to conclude that the distribution of magnetic particles for sample 5 in container P2 was homogeneous.

For the other samples, the differences between κ values ranged from 2.29% (sample 4 placed in container P2) to 11.71% (sample 4 in container P2, test after rotation). Based on the obtained results it was concluded that the homogeneity of a sample is a random, individual characteristic. Hypothesis 1 is therefore true for samples 3, 4, 6, 8, 9 and 10. For the remaining samples, the alternative hypothesis should be accepted. The obtained results clearly show that for the research process the main source of measurement errors is heterogeneous distribution of magnetic particles in sample volume.

It should be noted that samples 8, 9 and 10 contained particles of natural magnetite of geogenic origin, resulting in better uniformity of magnetic particles than in the other samples. In samples 1 to 7, the source of magnetic signal were technogenic magnetic particles from industrial dust deposition, which are characterised by low stoichiometry and high diversity (Magiera *et al.*, 2021a). Their number and distribution in the sample structure are random, which was confirmed by the measurement results obtained for samples no. 1, 2, 5 and 7.

The accuracy of the obtained volume magnetic susceptibility results (κ) was evaluated by comparing them with the susceptibility values measured using the MS2B sensor (Table 2). The evaluation concerned samples tested in the reference position as well as after being rotated by 180°. The results are summarized in Table 4.

Table 4. Accuracy of volume magnetic susceptibility for MYA 21.4Y measurements. H1 – homogenized sample; H2 – not homogenized sample: P1 – container 1; P2 – container 2; P1R – container 1 rotated by 180°; P2R – container 2 rotated by 180°.

Sample position	**$\delta\kappa$ (%)**									
	H2	**H2**	**H1**		**H2**	**H1**	**H2**	**H1**		
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
P1	–37.69	40.80	6.91	13.18	96.62	23.56	–21.68	0.99	20.78	5.62
P1R	–2.65	8.17	–5.71	8.06	–100.00	23.20	–49.67	–11.39	8.91	–4.79
P2	24.61	–7.28	6.91	8.06	–7.53	16.82	32.59	–4.25	20.57	9.11
P2R	–14.33	–7.28	–3.05	–6.73	–14.93	1.57	6.31	–12.75	11.20	–3.53

Based on the results presented in Table 4, there was no relationship between magnetic homogeneity of the samples and the measurement accuracy. It should be noted that there are quite clear differences in the test methods, *i.e.* the MS2B method "averages" the sample volumetrically, while the MYA weighing method is very sensitive to spatial distribution of magnetic particles in the sample. The unsatisfactory metrological accuracy for individual samples may therefore prove motivational for further improvement of the weighing method. Nevertheless, from the statistical point of view present correlation between results from the MYA weighing method, and the MS2B ones is sufficient (Fig. 9).

The magnetic susceptibility results of samples (1–10), tested in containers P1, P2 in the reference position and after rotation, reveal a clear linear correlation trend with the susceptibility results obtained using the MS2B method. The values of the r-Pearson coefficient (collected in Table 5) are noticeably higher than a critical value of 0.6319 for a confidence level of $\alpha = 0.05$ and $n = 8$ degrees of freedom, which proves the statistical significance of the presented correlations. Based on the R^2 determination coefficient value (being a measure of the quality of fitting model within least-squares method), it was found that more than 92% of the variation in the soil magnetic susceptibility obtained with the gravimetric method is in accordance with the corresponding variation observed with the MS2B equipment. Moreover, a linear regression coefficient (*i.e.* a slope of the straight line) takes value close to 1, which confirms the calibration correctness of the MYA gravimetric susceptometer. The values of r-Pearson correlation coefficient, R2 determination coefficient as well of the linear regression coefficient are presented in Table 5.

Table 5. Correlation coefficient for the tested samples.

	P1	**P2**	**P1R**	**P2R**
r-Pearson coefficient	0.97503	0.99157	0.96211	0.99305
R^2	0.95068	0.983211	0.925655	0.986152

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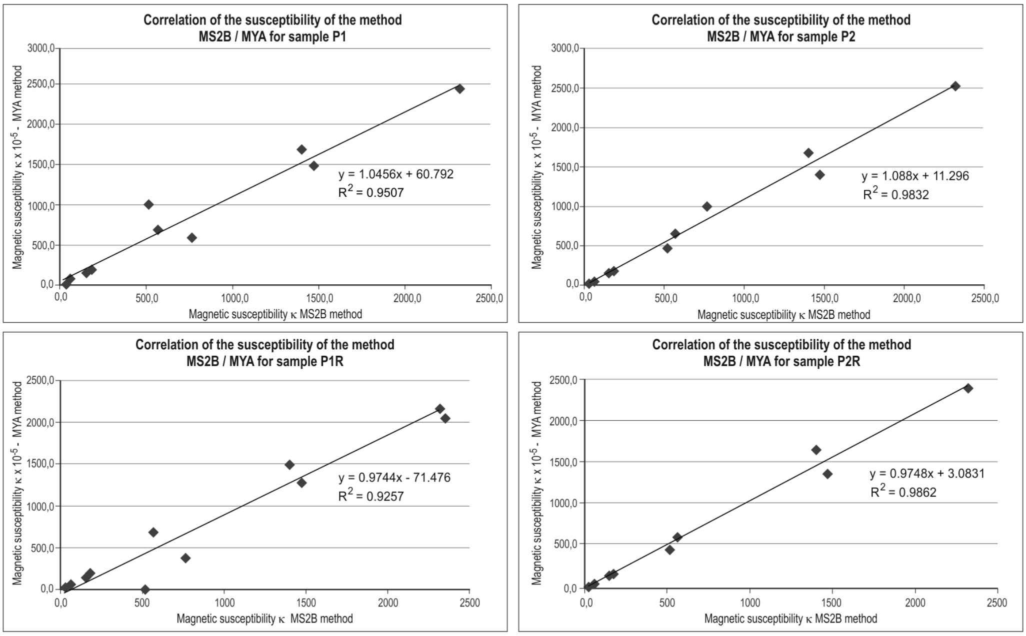

Fig. 9. Correlation between measurements of κ values using the MS2B Bartington sensor and the MYA weighting method.

The mass-specific magnetic susceptibility (χ) of the soil samples was determined using the MYA weighing method according to (3). The results are presented in Table 6. The accuracy of the measurements was assessed by comparing them to the specific magnetic susceptibility value obtained using the MS2B$-\chi$LF method (Table 2).

Table 6. Homogeneity of mass specific magnetic susceptibility for MYA 21.4Y measurements. P1 – container 1; P2 – container 2; P1R – container 1 rotated by 180°; P2R – container 2 rotated by 180°.

Sample position	$\chi\times10^{-8}$ m^3/kg									
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
P1	12.46	67.69	109.52	147.71	63.75	466.75	501.37	1224.03	1222.93	1846.09
P1R	19.93	52.07	96.63	140.68	0	441.22	322.43	1073.89	1102.48	1664.06
P2	26.3	40.34	112.48	153.61	298.39	548.89	620.43	992.85	1454.38	1777.42
P2R	18.41	40.34	101.45	133.12	274.48	477.48	497.14	905.01	1341.21	1571.46

It should be noted that the specific magnetic susceptibility depends on the volume susceptibility and the density of a sample. In this study, density was determined for each sample placed in containers P1 and P2. This approach does not allow a direct comparison between the results of mass-specific magnetic susceptibility for the samples placed in containers P1 and P2. The procedure requires the recalculation according to a predetermined density. However, also in this case, the discrepancies between the results obtained for the samples in containers P1 and P2 are the premise for sample heterogeneity. The measurement accuracy of the mass-specific magnetic susceptibility is presented in Table 7.

Table 7. Accuracy of volume magnetic susceptibility for MYA 21.4Y measurements. H1 – homogenized sample; H2 – not homogenized sample: P1 – container 1; P2 – container 2; P1R – container 1 rotated by 180°; P2R – container 2 rotated by 180°.

Sample position	$\delta\kappa$ (%)									
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
P1	–34.4	55.6	4.5	15.4	–79.8	12.5	–5.7	18.7	10.6	15.2
P1R	4.9	19.7	–7.8	9.9	–100	6.3	–39.4	4.1	–0.3	3.8
P2	38.4	–7.3	7.3	20.0	–5.4	32.3	16.7	–3.7	31.6	10.9
P2R	–3.1	–7.3	–3.2	4.0	–13.0	15.1	–6.5	–12.2	21.3	–2.0

The greatest differences in measurement accuracy were found for samples 1, 2, 5 and 7 which had previously been classified as having a heterogeneous distribution of magnetic particles. Accuracy deviations ranged from 34.4% (sample 1 in container P1) to 79.8% (sample 5 in container P1). In contrast, the most precise results were obtained for sample 3, regardless of the placement in container P1 or P2 and the position in which it was tested. The obtained accuracy error was respectively 4.5%, 7.8%, 7.3% and 3.2%. For samples 4 and 8 the accuracy deviation was about 20% and for samples 6 and 9 it was more than 30%, despite the fact that they were previously determined to be magnetically homogeneous. The determination accuracy of mass specific magnetic susceptibility of sample 10 ranged from 10.9% to 15.2%, with much smaller deviations recorded when the sample was tested after rotation by 180°.

It was assumed that samples were always tested in the reference position, so the best accuracy was obtained for sample no. 8 placed in container P2, measurement error –3.7%. In the other cases (marked with colour in the table), the discrepancies were maximum 15.4%. The susceptibility results for rotated samples are intended to confirm or exclude the effect of magnetic heterogeneity of the sample on the test result.

12. Summary and conclusions

The purpose of this study was to present a weighing method that can be used to test magnetic susceptibility of soil samples. Potential sources of errors that are associated with this method were pointed out. The research part aimed at presenting the results of volume magnetic susceptibility and mass-specific magnetic susceptibility for 10 soil samples. The accuracy of the measurements carried out and the correlation of the susceptibility results to those obtained using the MS2B Bartington susceptibility meter were evaluated. Based on the measurement results, it was found that homogeneity of distribution of magnetic particles in the sample volume is a crucial factor for the accuracy of measurements in the gravimetric method. Magnetic homogeneity can be expected for samples in which natural magnetite particles of geogenic origin predominate. Industrial and urban dusts containing mainly technogenic particles are magnetically heterogeneous which, however, does not exclude the possibility of obtaining a correct result. The ergonomics, simplicity and traceability of the weighing method allow it to be used for rapid magnetometric screening tests of diverse soil samples.

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