UNIVERSITY OF TARTU
INSTITUTE OF ECOLOGY AND EARTH SCIENCES
DEPARTMENT OF GEOLOGY

Siim Ots

Magnetic susceptibility, its anisotropy, and magnetic carriers of Upper Devonian sedimentary rocks, Latvia

Master thesis

Supervisor: Jüri Plado

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1 Introduction

The Devonian time started 416 and ended 359 Ma ago. During that time, the Baltica Plate was located close to the equator, where it had collided with Laurentia, forming the Euamerica continent and the Caledonides. During the Devonian time the intensive erosion of Caledonides started, that caused the formation of large sandstone deposits. In the later stage of Devonian time the epeiric seas started to transgress, decreasing the detrital input and formation of carbonate sediments started (Kleesment et al., 2006).

The palaeogeographical position of Baltica during Devonian time is however determined with only two reliable primary palaeomagnetic poles with both being from 400 Ma (Smethurst and Khramov, 1992; Jelenska and Lewandowski, 1986). Therefore, any new data to specify the Apparent Polar Wander Path palaeogeographic location of Baltica in Devonian is desired.

During the Devonian time part of the Baltica Plate was covered with sea. In the Latvian territory shallow sea sediments with various content of iron bearing minerals accumulated. The ferromagnetic minerals of sedimentary rocks can be detrital, authigenic, and diagenetic. A detrital mineral can be completely transformed, and authigenic minerals can be formed which are not usually stable and therefore may evolve over time with diagenetic processes, further modifying the magnetic characteristics of sedimentary rock. Carbonate rocks have an extremely low content of ferromagnetic minerals. The most widespread minerals are magnetite of detrital origin and hematite of diagenetic transformation from magnetite. Under particular conditions goethite may be formed and preserved over time (Lanza and Meloni, 2006).

The purpose of current work is to describe the behaviour of the magnetic susceptibility (MS) in the available Upper Devonian sedimentary rock outcrops, since the value of MS gives the first indication of the rock forming minerals and the amount of magnetic minerals in it. It also shows the amount of detrital input either by wind or water (Matasova et al., 2001). To determine the remanence origin (either primary or secondary) of the studied samples, measurements of anisotropy of magnetic susceptibility (AMS) were carried out. From the AMS data it is also possible to determine the direction of paleo-flow directions which could better describe the sedimentary environment. To determine the magnetic remanence carriers, or more importantly the presence of primary magnetite that is suitable for palaeomagnetical studies, thermal demagnetization of the acquired induced remanent magnetization (IRM)

(Lowrie, 1990) was carried out. SEM and XRF studies were also carried out to further characterize the studied outcrops. In the end the potential of each outcrop to fit for palaeomagnetical studies was evaluated.

2 Geological setting

Latvia is situated in the territory of East European Platform, where the Baltic Syneclise, the Latvian Saddle and southern slope of the Baltic Shield meet, forming a dislocated block-type crystalline basement overlain by the sedimentary cover consisting of Upper Proterozoic, Palaeozoic, Mesozoic, and Cenozoic deposits (Figure 1), which vary in thickness from 300 – 600 m in north-eastern Latvia to 1900 m in the south-western part.

Deposits of the Devonian system occur all over Latvia (Figure 1), while the total thickness varies considerably from 820 m in south-western to about 100 m in northern Latvia. The Devonian surface dips towards the south and south-west. The deposits are formed by terrigenous (sandstone, siltstone, and shale) and carbonate rocks (limestone, dolomite, and marl).

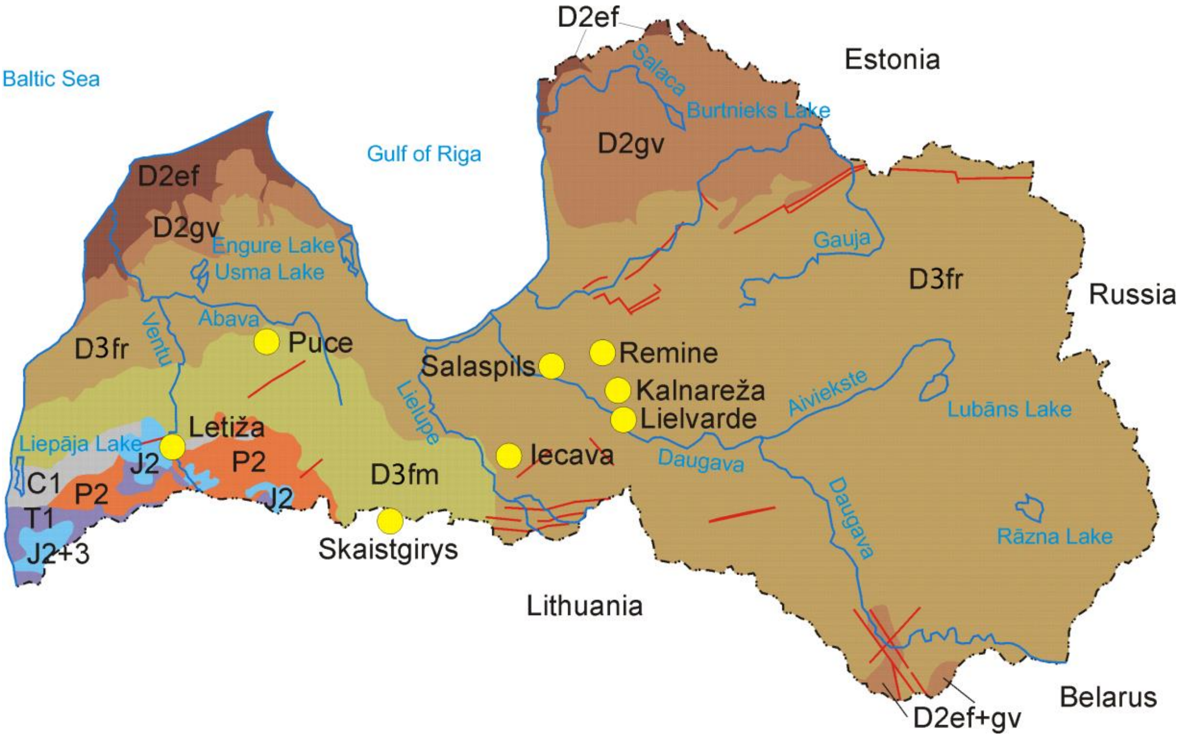
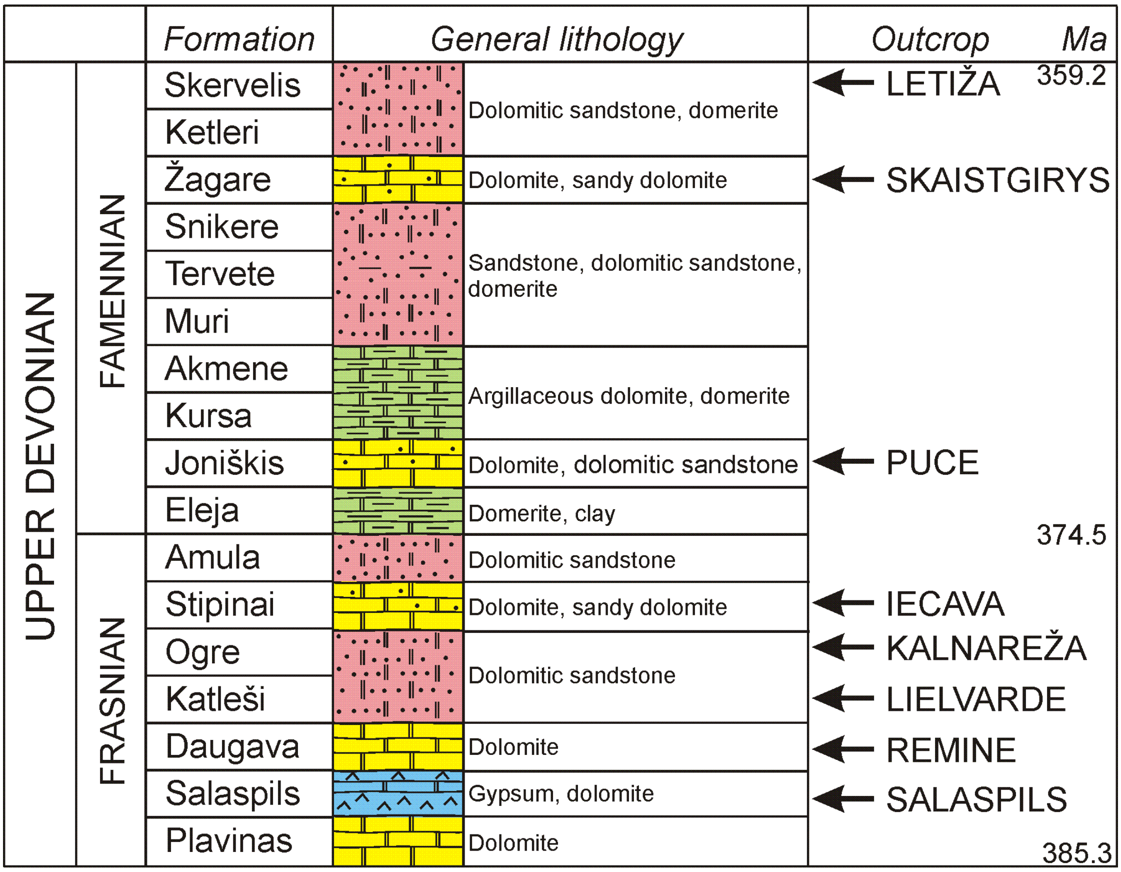


Figure 1. Geological bedrock map of Latvia and the locations for studied samples.

The Upper Devonian system is in the Baltic region separated into 17 formations (Figure 2), from which 8 – Salaspils (Salaspils quarry), Daugava (Remine quarry), Katleši (Lielvarde outcrop), Ogre (Kalnareža quarry), Stipinai (Iecava quarry), Joniškis (Puce outcrop), Žagare (Skaistgirys quarry), and Škervelis (Letiža outcrop), were studied in this thesis. The oldest of them is **Salaspils** formation, which is mainly characterized by clayey dolomites and dolomitic marls. The role of dolomitic marls and amount of gypsum increases westwards. Gypsum-rich deposits formed either in hypersaline lagoon or sabkha, due to considerable regression of the basin (Sorokin, 1978). The **Daugava** formation is dominated by dolomites (Brangulis et al., 1998). During Daugava time the epeiric sea was in wide transgression from the east (Sorokin, 1978). The **Katleši** formation is mainly characterized by sandstone and dolomite beds in the lower part and carbonate clay or dolomitic marl beds in the upper part. In central Latvia there are more dolomite and dolomitic marl interbeds. The epeiric sea continued to transgress from the east during Katleši time (Sorokin, 1981). Next consecutive formation is the **Ogre** formation, which is composed of sandstones with carbonate cement or silty clays and dolomitic marls (Sorokin, 1981). The Ogre basin differed



		Formation	*General lithology*	*Outcrop*	*Ma*
UPPER DEVONIAN	FAMENNIAN	Skervelis	Dolomitic sandstone, domerite	← LETIŽA	359.2
		Ketleri			
		Žagare	Dolomite, sandy dolomite	← SKAISTGIRYS	
		Snikere	Sandstone, dolomitic sandstone, domerite		
		Tervete			
		Muri			
		Akmene	Argillaceous dolomite, domerite		
		Kursa			
		Joniškis	Dolomite, dolomitic sandstone	← PUCE	
		Eleja	Domerite, clay		374.5
	FRASNIAN	Amula	Dolomitic sandstone		
		Stipinai	Dolomite, sandy dolomite	← IECAVA	
		Ogre	Dolomitic sandstone	← KALNAREŽA	
		Katleši		← LIELVARDE	
		Daugava	Dolomite	← REMINE	
		Salaspils	Gypsum, dolomite	← SALASPILS	
		Plavinas	Dolomite		385.3

Figure 2. Geological section of Upper Devonian in Latvia.

from other Frasnian basins by a large inflow of sand (Brangulis et al., 1998). During the Ogre time the basin likely advanced from the southwest (Sorokin 1978). The **Stipinai** formation mainly consists of alternating dolomite, clayey dolomite, and dolomitic marl, which in the northern part of its distribution area is characterized by sandstone and siltstone interlayers (Sorokin, 1981). The Stipinai time corresponds to the transgression of the sea which was open to the south-west (Brangulis et al., 1998). The **Joniškis** formation is in its northern part of the distribution area (southern Latvia) dominated by dolomites and the admixture of clayey and sandy material. During the Joniškis time open marine settings in wide areas dominated (Savvaitova, 1977). The **Žagare** formation is composed of sandy or fossiliferous dolomites and carbonate-rich sandy to clayey deposits in the northern distribution area (western Latvia). Open marine environment was dominant; however a considerable inflow of sand and clay is reported in the northern part of the basin (Savvaitova, 1977). The youngest of the Upper Devonian formations, the **Skervelis** formation, is mainly composed of cross-stratified sandstone with irregular dolomite (Gailite et al., 2000). The deposits of the lower part of Skervelis were likely to have formed in a shallow sea with considerable clastic inflow from the north (Stinkulis, 2004).

3 Sampling and methods

The samples were taken in 2011 by the employees of the University of Tartu (Jüri Plado, Ulla Preeden, and Leho Ainsaar) from 8 different locations. Samples were mainly drilled on site using a portative gasoline drill. From each outcrop 30 – 45 core samples were drilled. At Lielvarde, oriented blocks were taken which later were prepared into the standard specimens in the Solid Earth Geophysics laboratory at the University of Helsinki. Independent orientation of the cores or block samples was obtained by sun or magnetic compass. In total 538 specimens were prepared, each 2.5 cm in diameter and ~2.2cm in length.

Magnetic susceptibility and anisotropy of magnetic susceptibility were measured for all the specimens, from which 23 were picked later for Lowrie test (see below) and also some of the samples from each outcrop were studied with scanning electron microscope (SEM) and x-ray fluorescence (XRF). The magnetic susceptibility, SEM, and XRF studies were carried out at the University of Tartu. The AMS measurements and Lowrie test were done at the University of Helsinki.

3.1 Rock magnetic studies

3.1.1 Magnetic properties of materials

The extent to which a rock (material) can be magnetized when a magnetic field is applied is characterized by MS, which can be either positive or negative in value. Magnetic susceptibility is negative when the rock is dominated by **diamagnetic** minerals (quartz, halite, dolomite etc.) where in applied field electrons form a weak magnetic field, which is with opposite direction to the applied field. With **paramagnetic** minerals (pyroxene, biotite, clay-minerals etc.) the electrons are partially aligned with the external field and so they form a slightly positive magnetic field parallel to the applied field. The strongest MS values are shown with **ferromagnetic** minerals (iron, cobalt etc.), where the magnetic moments of the electrons form regions called domains, which results in a strong magnetic field that has the same direction as the applied field and after the outer field has been cancelled, the ferromagnetic minerals still preserve the direction of the applied field. This effect is called remanent magnetization that is the permanent magnetization of a material in the absence of an external magnetic field, and thus occurs only in materials which exhibit magnetic hysteresis (Hunt et al. 1995). Ferromagnetic materials subdivide to (i) true ferromagnetics (discussed above), (ii) antiferromagnetics, and (iii) ferrimagnetics.

In some materials the domains are divided into subdomains that are aligned in antiparallel directions, nearly cancelling out their moments. Such material is **antiferromagnetic** (hematite). The MS of an antiferromagnetic mineral is weak and positive – similar to paramagnetic substances, increasing with temperature and reaching the maximum value at the materials Curie temperature. The remanent magnetization on an antiferromagnetic substance is not possible unless there are no crystal defects.

When a material has subdomains that have antiparallel magnetic moments, but where in one direction the magnetic alignment is stronger or there are more subdomains of one type than the other, the magnetization is strong and positive and the material is **ferrimagnetic** (magnetite, maghemite, goethite, titanomagnetite etc.). Practically all magnetic minerals are ferrimagnetic.

The magnetic properties of ferro- and ferrimagnetics are temperature dependant. When heated to Curie temperature, the ferromagnetic becomes paramagnetic, because the distance between atoms becomes so distant due to heat that the formation of magnetic domains is blocked. After cooling, the substance becomes ferromagnetic again (Lillie, 1999; Telford et al., 1990; Lowrie, 1997).

Each ferromagnetic substance contains magnetic domains, each with its own magnetization direction that differs from the neighbouring domains. Mineral grains that contain many domains are called **multidomain** (MD) particles; grains containing only one domain are called **single-domain** (SD) particles. There is also an intermediate grain type called **pseudo-single-domain** (PSD) particle, which has more than one domain, but also possesses many of the properties of a SD grain (Evans and Heller, 2003). When a grain decreases to smaller size than a SD grain (<0.03 μm, varies with different magnetic minerals) it is still SD, but displays unique properties. The magnetization is strong but unstable due to thermal energies counteracting induced magnetization quickly after a magnetic field is removed. The behaviour is similar to paramagnetism, but with much greater susceptibility. Hence it is termed **supermaramagnetic** (SP) behaviour. The SP crystals are characterized by their response to susceptibilities measured at different frequencies and are detected by frequency-dependent measurements (Dearing, 1999).

3.1.2 Magnetic susceptibility

Magnetic susceptibility κ (or **volume susceptibility**) is defined by

$$\kappa = \frac{M}{H};$$

where M is the induced magnetization of the material and H is the inducing magnetic field. Since both M and H are measured in A/m (amperes per meter), volumetric susceptibility is dimensionless (SI). To obtain what is called the **mass susceptibility** (m^3/kg), we divide by the density,

$$\chi = \frac{\kappa}{\rho};$$

where ρ is the density (kg/m^3) of the material (Evans and Heller, 2003). Mass susceptibilities of different minerals are given in Table 1.

Table 1. Magnetic susceptibilities of selected rocks and minerals (according to Hunt et al. 1995).

Rock/Mineral	Mass χ (10^{-6} m^3/kg)
dolomite	-0.01 … 0.41
limestone	0.001 … 12
sandstone	0 … 9.31
clay	0.1 … 0.15
calcite	-0.003 … 0.014
gypsum	-0.005 … 0.013
pyrite	0.01 … 1
hematite	0.1 … 7.6
maghemite	400 … 500
magnetite	200 … 1100
titanomagnetite	25 … 120
goethite	0.26 … 2.8
iron	500

The magnetic susceptibility measurements were carried out using MS meter SM-100 (Figure 3). All the samples where weighted for the masses and then measured at three different frequencies – 1 kHz, 4 kHz and 16 kHz - and at field strength of 160 A/m. The relatively strong field strength of 160 A/m was chosen, because the measuring precision is better at higher fields (Hrouda and Pokorny, 2011). At least three measurements were carried out with each specimen, so that an average value could be taken. To measure, the specimens were put into an empty holder which was then lowered into the SM-100 for about five seconds, after what in about ten seconds the MS value was received. The values were copied to MS Excel where several parameters where calculated – frequency-dependent magnetic susceptibility (X_{FD}), standard deviation and the X_R parameter.

The frequency-dependent susceptibility (%) is expressed as a percentage loss of susceptibility and is calculated using the values of two frequencies,

$$X_{FD} = 100\,\frac{\chi\mathrm{LF}-\chi\mathrm{HF}}{\chi\mathrm{LF}};$$

Figure 3. Magnetic susceptibility meter SM-100 with some of the specimens.

where χ_{LF}, χ_{HF} are the susceptibilities at the low and high frequencies. The X_{FD} is usually very low (<5 %), rarely exceeding 15 %. The measuring accuracy has therefore to be high and the error in determination of the X_{FD} parameter should be <1 %. It has been shown that when the measured MS values are greater than 1×10^{-8} m^3/kg, the theoretical root-mean-square error in determining the X_{FD} parameter is better than 0.5 %. With MS values being $\chi < 1\times10^{-9}$ m^3/kg, the relative error is an order of magnitude higher than that required for reliable determination of the X_{FD} parameter (Hrouda and Pokony, 2011).

Samples where the SP minerals are present show slightly lower values when measured at high frequency, because the SP grains are frequency-dependent; samples without the SP minerals show near identical χ values at both frequencies (Dearing, 1999). The X_{FD} enables the amount of newly formed ultrafine SP grains to be assessed (Table 2) that can indicate remagnetization of minerals by chemical processes and therefore showing the possible event of secondary magnetization.

Table 2. Interpretation of X_{FD} values (Dearing, 1999).

Low $X_{FD\%}$	<2.0	Virtually no SP grains
Medium $X_{FD\%}$	2.0-10.0	Mixture of SP and coarser grains, or SP grains < 0.05 µm
High $X_{FD\%}$	10.0-14.0	Virtually all SP grains
Very high $X_{FD\%}$	>14.0	Erroneous measurement, anisotropy, weak sample or contamination

The X_R parameter is calculated using susceptibility measurements at three operating frequencies and is defined as:

$$X_R = \frac{\chi 1 - \chi 4}{\chi 4 - \chi 16}.$$

The χ_1, χ_4 and χ_{16} are MS values at 1, 4, and 16 kHz, respectively. The parameter itself is insensitive to dia-, paramagnetic fractions and helps to differentiate between wide and narrow size distributions of ferromagnetic particles (Hrouda, 2011) meaning that if the X_R parameter stays homogenous in a studied set of samples, the grain size deviation of non MD ferromagnetic particles is low. The higher X_R values could indicate finer grain size of non MD ferromagnetic particles.

3.1.3 Anisotropy of magnetic susceptibility

The AMS of rocks, which investigates the preferred orientation of magnetic minerals, contains information about both the grain susceptibilities and their orientation-distribution. A preferential orientation-distribution of mineral grains develops during various geological processes, such as water flow in sediments, magma flow in igneous rocks or ductile deformation in metamorphic rocks (Sagnotti, 2011).

The AMS is generally controlled by all minerals present in a rock. If the susceptibility is more than 5×10^{-6} m^3/kg the effects of para- and diamagnetic minerals are negligible and the AMS is controlled by the ferromagnetic fraction only. With susceptibility less than 5×10^{-7} m^3/kg the AMS is effectively controlled by the paramagnetic fraction and with MS less than 5×10^{-8} m^3/kg, even the effect of diamagnetic fraction cannot be neglected (Hrouda, 2010).

The output of AMS measurements is an ellipsoid of magnetic susceptibility (AMS ellipsoid, Figure 4) defined as the length and orientation of its three principal axes, $K_1 \geq K_2 \geq K_3$ – the three eigenvectors of the susceptibility tensor (Rochette et al., 1992). The K_1 defines the maximum susceptibility (K_{max}) or the direction where most of the magnetic grains are aligned. The K_2 is defined as the intermediate (K_{int}) and K_3 as minimum susceptibility (K_{min}).

When measured, each AMS eigenvector is determined with two uncertainty angles, which define the regions where each principal susceptibility direction lies with a probability of 95 %. AMS ellipsoid shapes are classified according to the relationships between the MS eigenvalues seen from Figure 5.

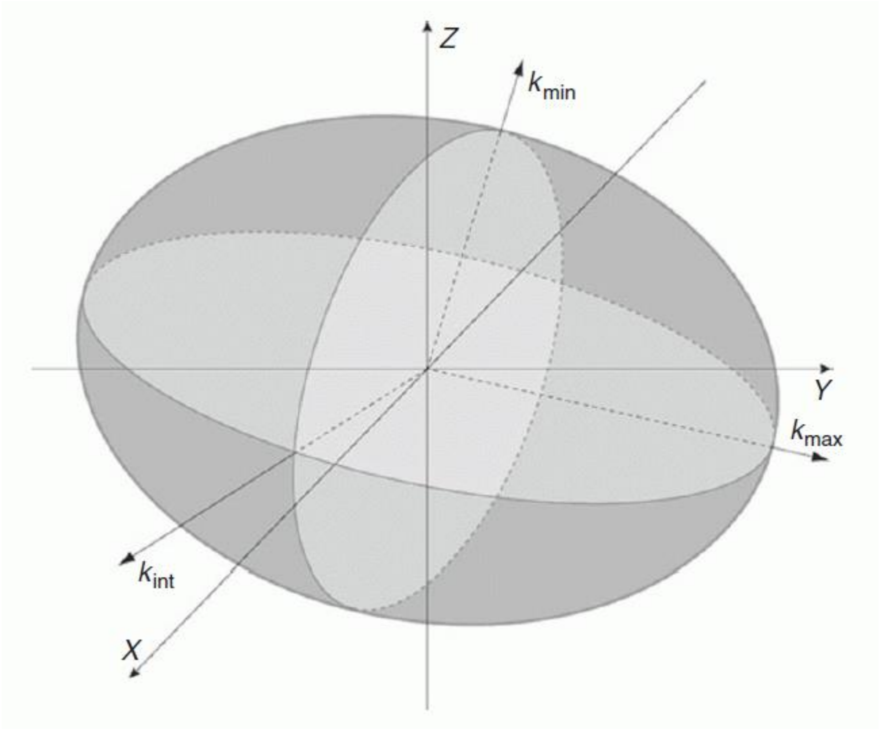


Figure 4. The AMS ellipsoid defined by K_{max}, K_{int} and K_{min} (from Sagnotti, 2011).

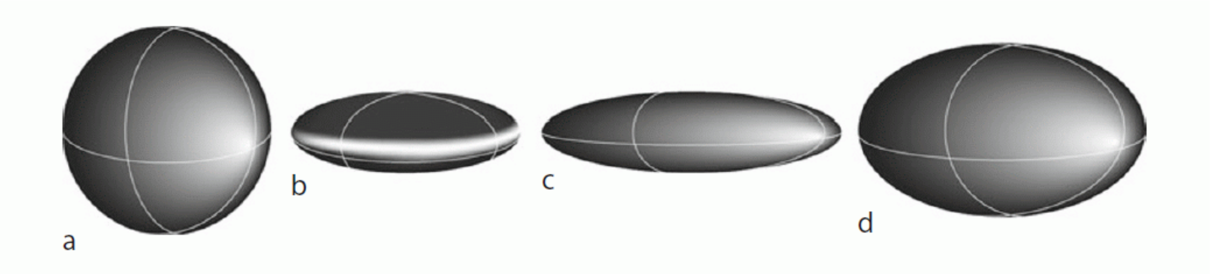


Figure 5. Shape of AMS ellipsoids: (a) $k_{max} \approx k_{int} \approx k_{min}$; isotropic susceptibility, the AMS ellipsoid is a sphere; (b) $k_{max} \approx k_{int} > k_{min}$; the AMS ellipsoid is an oblate shape; (c) $k_{max} \approx k_{int} < k_{min}$; the AMS ellipsoid has a prolate shape; (d) $k_{max} > k_{int} > k_{min}$; the AMS ellipsoid is triaxial (from Sagnotti, 2011).

After the measured data has been modified with the corrected strike and dip, taken at the outcrops for each sample, the shape of the AMS ellipsoid and the directions of the K_{max} and K_{min} eigenvectors can describe paleo-flow direction and post-sedimentary processes (Sagnotti, 2011; Liu et al., 2001). Mostly the sediments show K_{max} axes that are distributed within the depositional plane and K_{min} axes perpendicular to it, resulting in oblate magnetic ellipsoid (absent or very low flow of water). In moderate currents, the imbrication induces a slightly vertical offset of the mean K_{min} (pointing towards the direction of paleo-flow) and the clustering of the K_{max} axes in a direction antiparallel to the paleo-flow (Sagnotti, 2011).

The AMS measurements were carried out using the AGICO KLY-3S kappabridge (875 Hz, 300 A/m and precision of 2×10^{-8} SI), which allows the automatic measurement of susceptibility along three perpendicular axes while the specimen is rotating. Equal-area projections of the AMS ellipsoid were plotted using the anisotropy data browser Anisoft 4.2.

3.1.4 Lowrie test

The common ferromagnetic minerals have distinctive, characteristic coercivities and thermomagnetic properties (Table 3), which can be used to identify the magnetic carriers. Using stepwise thermal demagnetization of the acquired induced remanent magnetization the magnetic mineralogy can be interpreted (Lowrie, 1990).

At first, the natural remanent magnetization (magnetization that the rock has acquired during geological time and that exists without external field) of the selected specimens was destroyed by demagnetizing the specimens with strong fields. Then three successive saturation fields along three perpendicular directions were applied: 1.5 T in the z-direction (diagnostic for hard magnetic carriers), 0.4 T in the y-direction (diagnostic for medium

Table 3. Composition, maximum remanent coercivities and Curie temperatures (T_C) for some common ferromagnetic minerals (Lowrie, 1990; Lanza and Meloni, 2006).

Mineral	Composition	Max coercivity (T)	T_C (ºC)
Hematite	α-Fe_2O_3	1.5-5	675
Maghemite	γ-Fe_2O_3	0.3	~350
Magnetite	Fe_3O_4	0.3	580
Titanomagnetite	$Fe_{3-x}Ti_xO_4$	0.1-0.2	150-540
Pyrrhotite	Fe_7S_8	0.5-1	320
Goethite	α-FeOOH	>5	120

magnetic carriers), and 0.12 T in the x-direction (diagnostic for soft magnetic carriers). The soft IRM typically corresponds to magnetite, medium IRM to pyrrhotite, and hard IRM to hematite and/or goethite (Lowrie, 1990). The IRM magnetization was done at the Geophysics laboratory of the Geological Survey of Finland.

After that the specimens were thermally demagnetized step by step up to 680ºC. Argon gas was used to minimize the effect of remagnetization during heating. Between each step the MS (to determine mineralogical changes due to heating) and remanent magnetization were measured. For MS measurements SM-100 was used. The remanent magnetization was acquired using 2G-Enterprizes superconducting SQUID magnetometer. After the measurements the magnetization of each orthogonal axis was plotted against the temperature.

3.1.5 SEM and XRF

The SEM and XRF studies were carried out during the course of "Sedimentary petrology" in 2012 under the supervision of prof. Kalle Kirsimäe. One sample for each formation was prepared. Both studies can illustrate the chemical composition of the formations and the magnetic mineralogy of the specimens. XRF measurement was carried out using the fps-method and later the values were corrected with the calculated LOI (Loss on ignition) values.

4 Results

4.1 Magnetic susceptibility

The average MS values for all the studied outcrops is low (characteristic for sedimentary rocks; Table 1), varying from 2.5×10^{-9} m^3/kg up to 31.2×10^{-9} m^3/kg (according to measurements at 1 kHz; Table 4). The highest values were measured in the dolomitic sandstone of Puce specimens (42.6×10^{-9} m^3/kg) and the lowest in the dolomites of Salaspils (1.7×10^{-9} m^3/kg) (Figure 6). The MS values for Letiža, Skaistgirys, Iecava, Kalnareža, and Remine are similar to each other, varying the most in the sandy-clayey dolomites of Skaistgirys specimens ($\pm 5.5 \times 10^{-9}$ m^3/kg; Table 4). The values with lowest standard deviation ($\pm 1.9 \times 10^{-9}$ m^3/kg; Table 4) were measured in Letiža specimens.

Table 4. Average, standard deviation and median values of MS, X_{FD} (calculated with 1 and 16 kHz) and X_R of the studied outcrops.

	Magnetic susceptibility (10^{-9} m^3/kg)				
Outcrop/Quarry	1kHz	4kHz	16kHz	$X_{FD(1,16)}$ (%)	X_R
Letiža	12.5±1.9; 12.04	12.4±1.8; 11.94	12.3±1.8; 11.83	1.7±0.4; 1.54	1.7±1,7; 1.28
Skaistgirys	10.8±5.5; 9.92	10.7±5.5; 9.74	10.6±5.5; 9.57	2.2±1.3; 1.91	1.5±0.9; 1.21
Puce	31.2±5.1; 32.05	31.0±5.0; 31.84	30.8±4.9; 31.75	1.2±0.4; 1.12	1.5±1.1; 0.76
Iecava	12.5±3.2; 11.67	12.3±3.2; 11.57	12.2±3.2; 11.43	3.0±1.2; 2.74	2.6±1.9; 2.84
Kalnareža	15.4±2.9; 16.16	15.1±2.9; 15.87	15.0±2.9; 15.72	2.8±0.6; 2.66	2.3±1.1; 1.91
Lielvarde	20.4±4.8; 19.61	20.2±4.9; 19.47	20.1±4.9; 19.36	1.2±0.4; 1.19	1.0±0.4; 1.02
Remine	11.1±5.2; 11.58	11.0±5.2; 11.50	10.9±5.1; 11.36	2.2±1.3; 1.78	1.2±0.5; 1.06
Salaspils	2.5±4.1; 2.05	2.2±3.8; 1.92	2.1±3.8; 1.84	16.0±10.8; 9.95	1.9±0.9; 1.93

All the average values for the frequency-dependency of MS stay low (≤3 %; Table 4) except for the Salaspils specimens, where the average calculated value is 16.0 % (Table 4) and the maximum value reaching almost 40 %. These high values occur cause of diamagnetic samples giving very low MS values (near the sensitivity of SM-100), and this leads to erroneous values when calculating X_{FD} (Table 2). From the rest of the samples the lowest calculated X_{FD} values are in Puce and Lielvarde samples (1.2 ±0.4 %; Table 4) and highest values occur in Iecava (3.0 ±1.2 %; Table 4). In Iecava and Skaistgirys samples the X_{FD} shows slightly higher values at the bottom of the studied samples than that of the higher layers (Figure 7). In Remine samples an upward increase of X_{FD} can be seen (Figure 7). Rest of the sections do not show any significant changes of the X_{FD}.

The X_R parameter, suggested by Hrouda (2011), shows average values from 1.0 in the Lielvarde up to 2.6 in the Iecava samples (Table 4). The standard deviation is highest in the Iecava samples (±1.9; Table 4) and lowest values were calculated for Lielvarde (±0.5; Table 4). Only notable change in X_R can be seen from Puce (increasing upward) and Iecava (increasing downward) (Figure 8). For Lielvarde and Remine samples the X_R shows the most homogenous values.

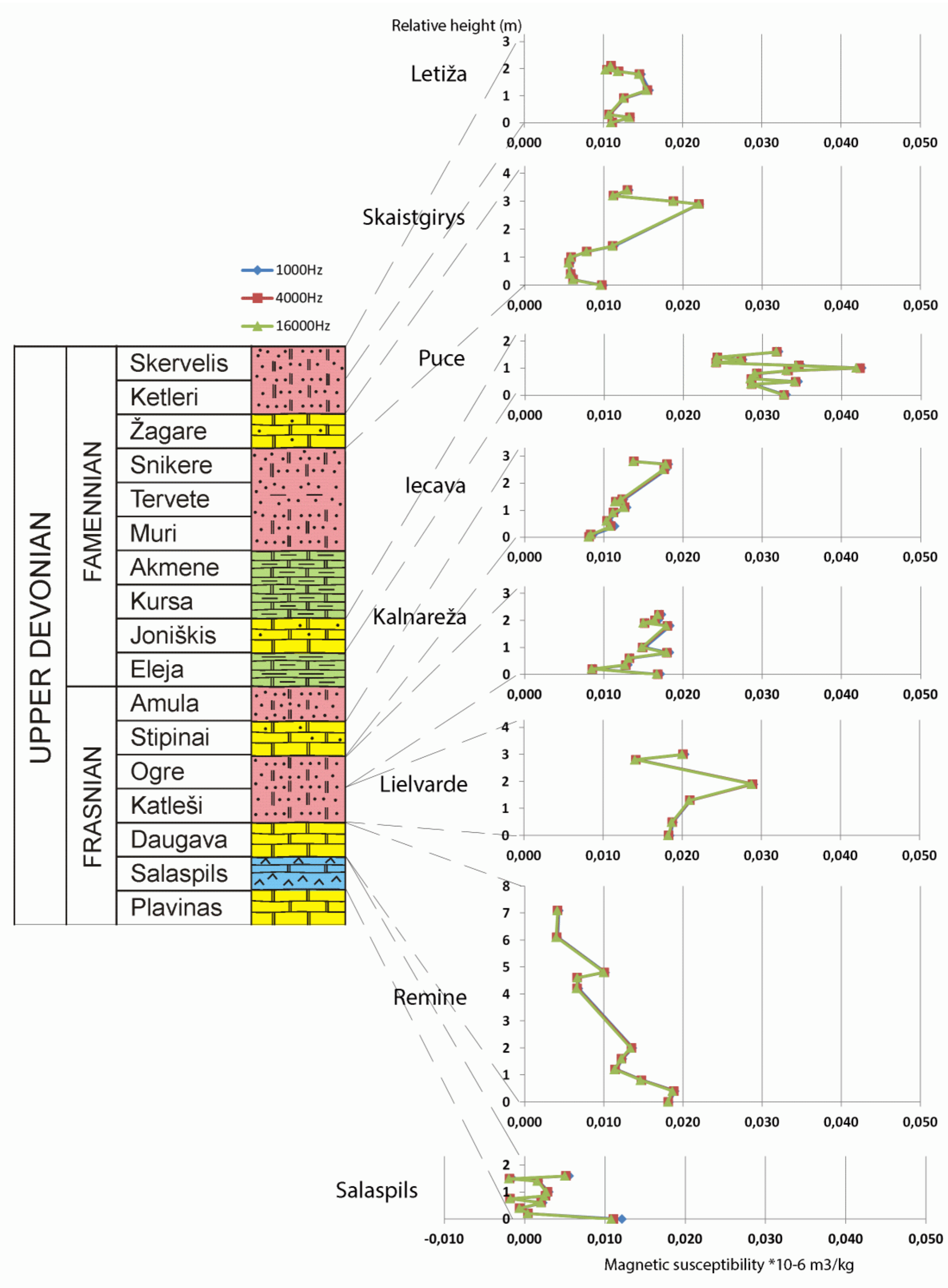


Figure 6. Magnetic susceptibility behaviour of studied outcrops.

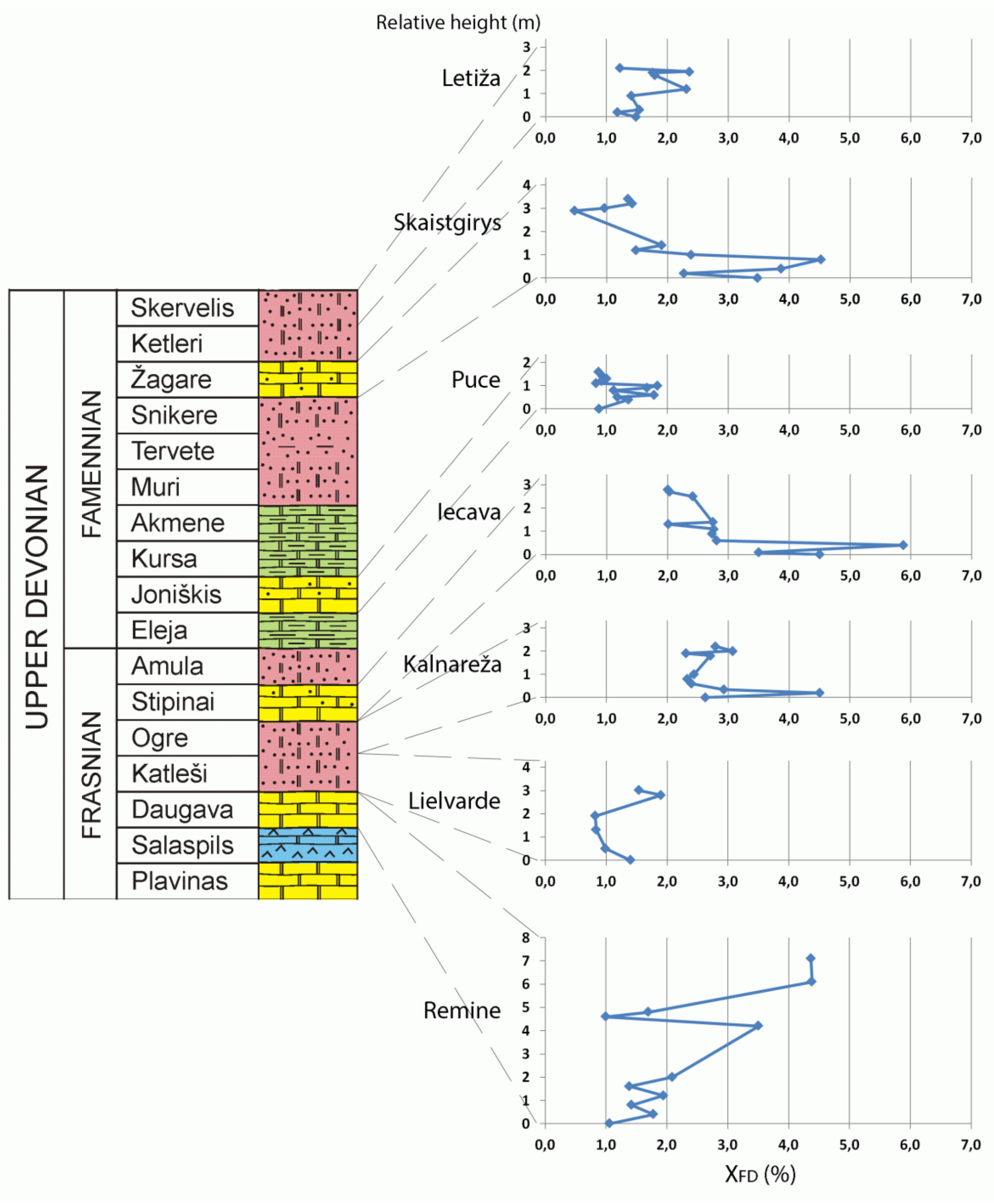


Figure 7. Frequency-dependent magnetic susceptibility values for studied outcrops.

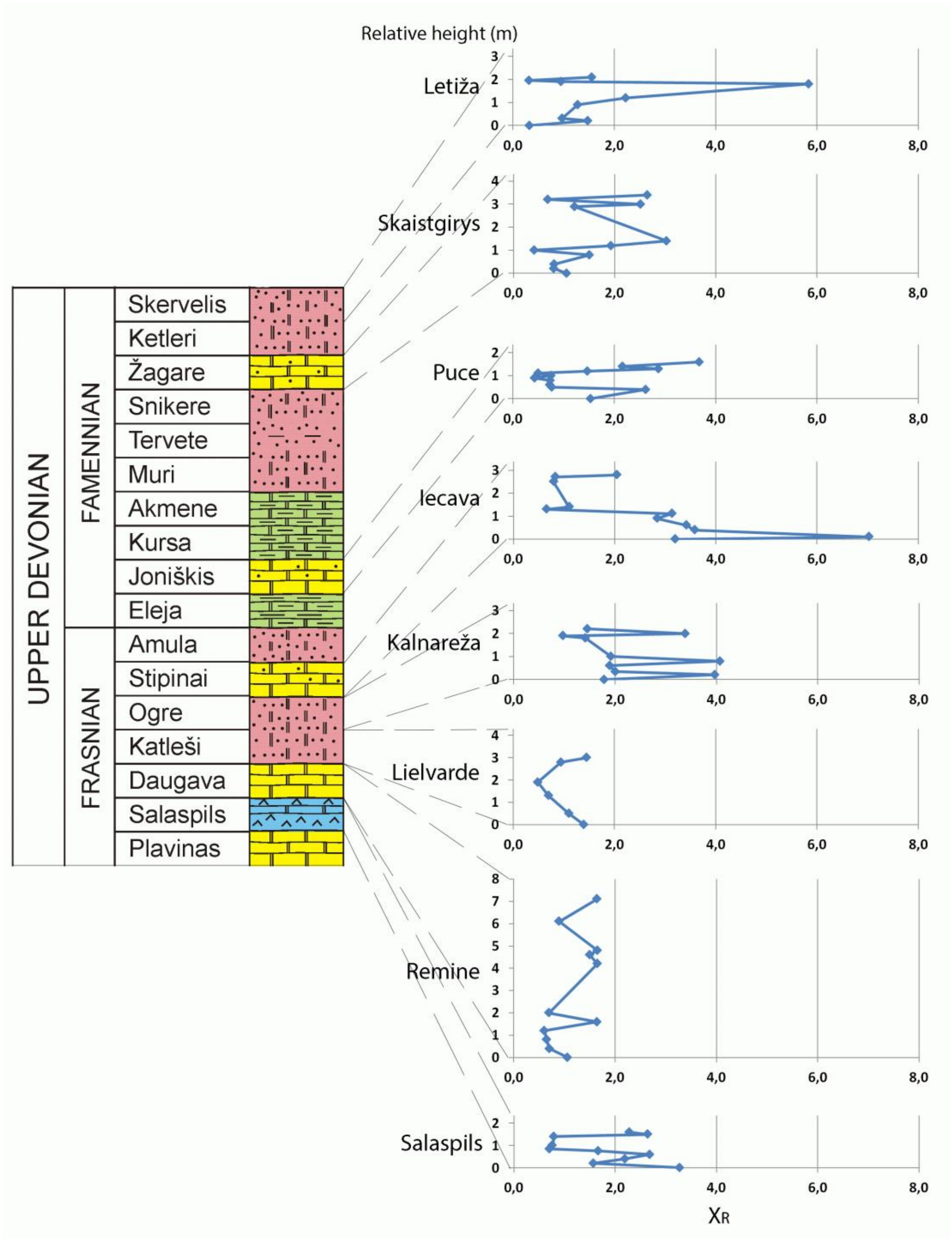


Figure 8. Values of the X_R parameter for studied outcrops.

4.2 AMS

All studied locations but Lielvarde showed either a prolate or oblate shape of the ellipsoid. When determining the direction of the paleo-flow the conditions described by Liu (2001) and Sagnotti (2011) are considered. The AMS of Letiža (Figure 9a) shows a triaxial ellipsoid with more oblate shape. The K_{min} being tilted to the NNW direction shows a possible paleo-flow from SSE to NNW. The Skaistgirys ellipsoid (Figure 9b) has the same shape as in Letiža, only here the vertical offset of the K_{min} mean value shows a paleo-flow from SW to NE. For the next two – Puce and Iecava (Figure 9c; d), the K_{max} axes seem to be perpendicular to the depositional plane and K_{min} and K_{int} scattered in the bedding plane, indicating a prolate shape ellipsoid. The Kalnareža AMS ellipsoid (Figure 9e) shows the most oblate shaped ellipsoid from the studied locations, where the K_{min} is perpendicular to the bedding plane and K_{max} scattered within it. The AMS ellipsoid for Lielvarde (Figure 9f) does not show any distinguishable shape and cannot be interpreted. In Remine (Figure 9g), the triaxial, more oblate shaped ellipsoid can be seen again. A possible paleo-flow direction from SEE to NWW can be interpreted. The ellipsoid from the Salaspils (Figure 9h) shows an oblate shape, with a possible paleo-flow from SWW to NEE.

4.3 Identification of magnetic minerals (Lowrie test)

All of the samples have low IRM values (from 10^{-8} to 10^{-6} A/m) indicating low content of magnetic minerals and because of that most of the samples did not give any valuable data. Thermal demagnetization diagrams of the 3 axes IRM for specimens from five locations are presented in Figure 10.

The samples from Puce outcrop (Figure 10a) show unblocking of hematite at 680ºC on broad range of coercivities. From soft fraction at 350ºC and medium fraction at 550ºC possible titanomagnetite and magnetite can be seen. From the hard fraction of Letiža samples (Figure 10b; c), unblocking temperatures at 120 and 680ºC show the presence of goethite and hematite. Medium and soft fractions show unblocking of magnetite at 580ºC and possibly maghemite at 360ºC. The samples from Kalnareža outcrop (Figure 10d) show nearly identical thermal demagnetization of three component IRM. The soft fraction show unblocking of titanomagnetite or maghemite at 330 to 360ºC. Soft and medium coercivity fractions both show an unblocking of magnetite at 550ºC. The samples from Skaistgirys outcrop (Figure 10e) show unblocking of some minor magnetite at 580ºC and also the dominance of hematite on all fractions. Samples from Iecava (Figure 10f) also show dominance of hematite on all fractions. The soft fraction shows unblocking of titanomagnetite at 300 to 400ºC. At 550ºC, the medium coercivity fraction shows unblocking of magnetite. In Remine (not illustrated) only goethite could be distinguished from one sample. No magnetic carriers from Salaspils samples could be distinguished.

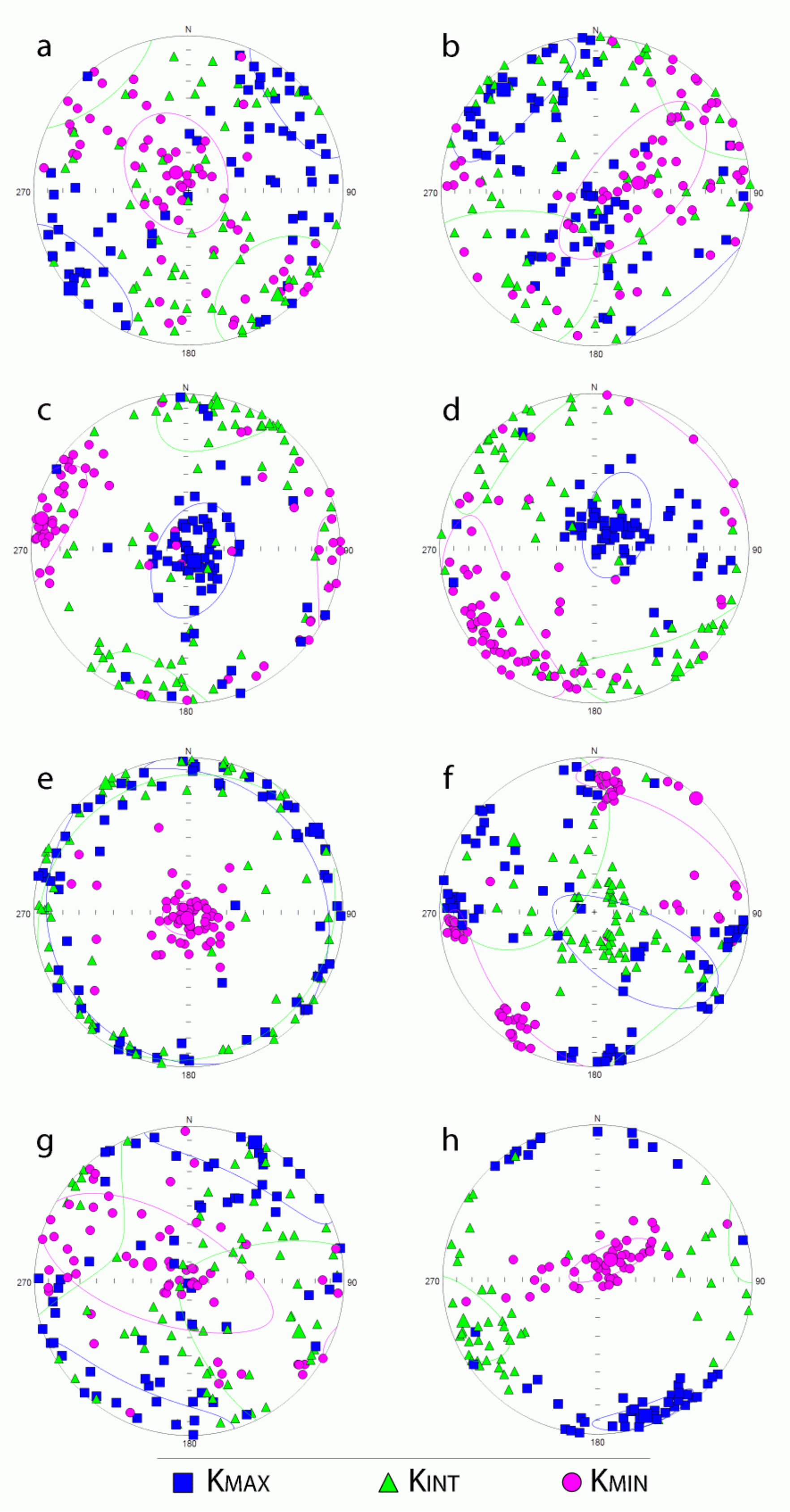


Figure 9. Equal area projection of AMS principal axes with 95% confidence ellipses of measured samples at a) Letiža; b) Skaistgirys; c) Puce; d) Iecava; e) Kalnareža; f) Lielvarde; g) Remine and h) Salaspils.

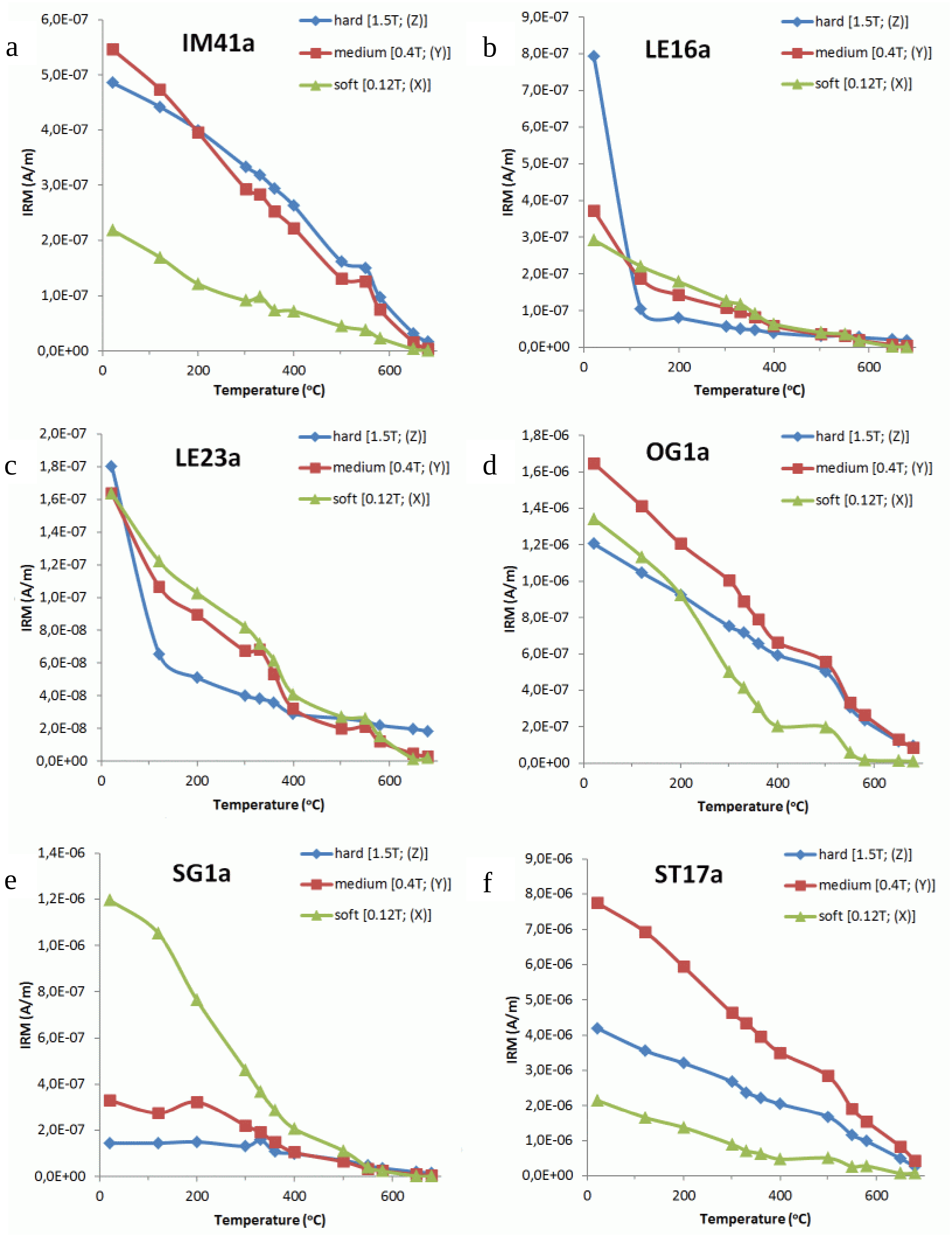


Figure 10. Thermal magnetization of a three-component IRM produced according to Lowrie (1990), for samples taken from Puce (a), Letiža (b; c), Kalnareža (d), Skaistgirys (e) and Iecava (f).

After a first magnetization at 1.5 T, a mobilization of part of the grains is possible with the second magnetization at 0.4 T, causing hematite to show both on high and medium coercivity axes (Lehman et al., 1996). Signal of hematite on low coercivity fraction curves may also be due to presence of large MD grains or indicates heat-caused mineralogical changes during the treatment (Dunlop and Özdemir, 1997).

4.4 SEM and XRF measurements

Figure 11 illustrates the low content of iron bearing minerals in all samples observed with SEM. Most of the samples show rather heterogeneous matrix of ferromagnetic grains. The XRF measurements are illustrated in Table 5. It can be seen that Iecava has the only pure dolomite content. Salaspils has the most sulphur (7.65 mass%) which is probably due to the content of gypsum and also pyrite. Letiža, Puce, and Remine all have similar CaO and MgO values and all show some detrital input with sand and clay mixture (7.2 – 12.8 mass% SiO_2 and 1.2 – 3.2 mass% Al_2O_3). When Fe_2O_3 is considered then Puce has the highest value (~1.4 mass%) for the three named outcrops. Next two very similar in composition outcrops are Skaistgirys and Kalnareža, showing large detrital content (~40 mass% SiO_2), meaning these are dolomitic sandstones. The highest detrital content can be seen in Lielvarde, where the amount of SiO_2 is 57 mass% and Al_2O_3 5.1 mass%, both being the highest from all the outcrops. Also the content of Fe_2O_3 (1.5 mass%) and TiO_2 (0.26 mass%) are the highest from all the samples measured.

Table 5. Chemical data accuired from the XRF measurements. Values show mass%.

Location (Specimen)	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	K_2O	P_2O_5	S_{total}	Balance
Letiža (LE1-3)	7.25	1.20	0.08	0.92	0.07	29.71	18.69	0.48	0.01	0.04	41.34
Skaistgirys (SG14-17)	40.69	1.68	0.22	1.35	0.07	20.49	9.57	1.35	0.02	0.03	24.29
Puce (IM41-45)	12.85	3.21	0.15	1.39	0.09	23.28	18.61	1.39	0.09	0.03	38.60
Iecava (ST17-19)	0.73	0.16	0.02	0.87	0.09	28.81	19.08	0.10	0.01	0.02	49.81
Kalnareža (OG1-3)	43.04	1.66	0.08	1.07	0.06	19.90	8.86	1.11	0.29	0.02	23.56
Lielvarde (LV1-3)	57.31	5.10	0.26	1.50	0.06	12.40	5.35	2.26	0.58	0.02	14.74
Remine (RE7-9)	12.78	3.25	0.14	0.95	0.07	25.83	13.26	1.28	0.02	0.02	42.25
Salaspils (SL4-6)	0.58	0.18	0.00	0.39	0.03	30.81	7.87	0.07	0.01	7.65	52.37

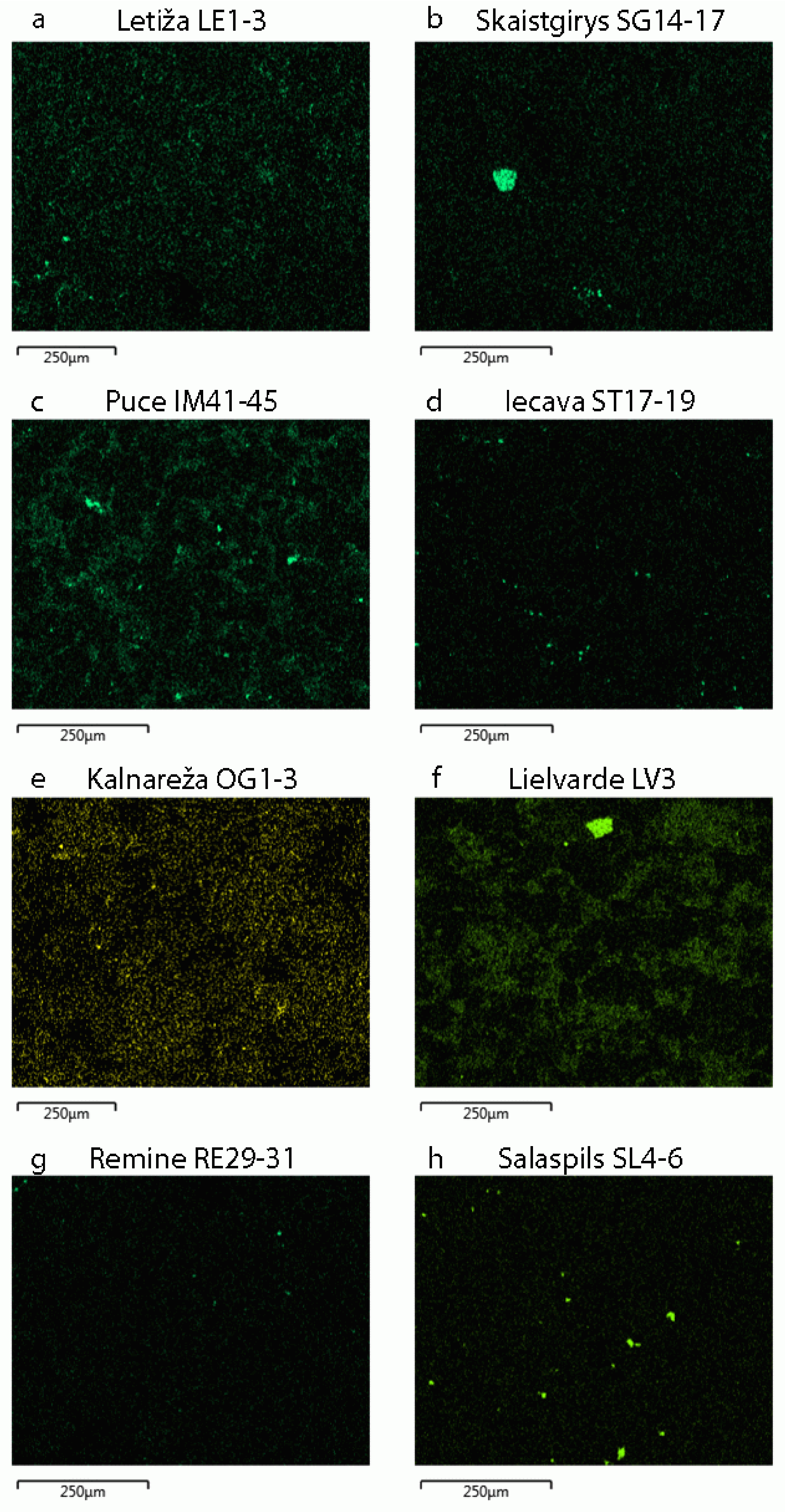


Figure 11. Images of iron bearing minerals taken with SEM for all locations. Intensity of colour shows the quantity of iron in minerals.

6 Discussion

For all the outcrops, the measured MS values stayed very low (average of 15 × 10^{-9} m^3/kg for all the samples) correlating with the values of sedimentary rocks (Table 1). From the Figure 12 a possible correlation between X_{FD} and MS values can be seen where bigger X_{FD} values show lower MS values. This could be due to the fact that with low values, due to weak samples, the calculation of X_{FD} could be inaccurate (Table 2) and as a result slightly bigger values of X_{FD} could be expected.

The Letiža outcrop represented the Skervelis formation, which mainly consists of dolomitic sandstone. The MS values measured in Letiža had the smallest deviation from all the outcrops (±1.9 × 10^{-9} m^3/kg; Table 4) with an average value of 12.5 × 10^{-9} m^3/kg (Table 4), meaning that the average detrital input in the studied interval stayed the same. The calculated average X_{FD} value was 1.7 % and also showed no significant variations (Figure 7), meaning there is probably only very few SP particles present in the samples. This indicates that the possibility of any chemical remagnetizations is very low. When we look at the AMS data (Figure 9a), we can see that the K_{min} axes are perpendicular to the bedding plane while the K_{max} and K_{int} are scattered in it. This means the original sedimentary fabric is still preserved and the ferromagnetic minerals may have primary magnetization component. With the magnetic carriers being goethite, hematite, magnetite, and maghemite, the magnetite fraction could possibly carry the primary component needed for palaeomagnetical studies. Other ferromagnetic minerals are probably of secondary origin (Plado et al., 2010).

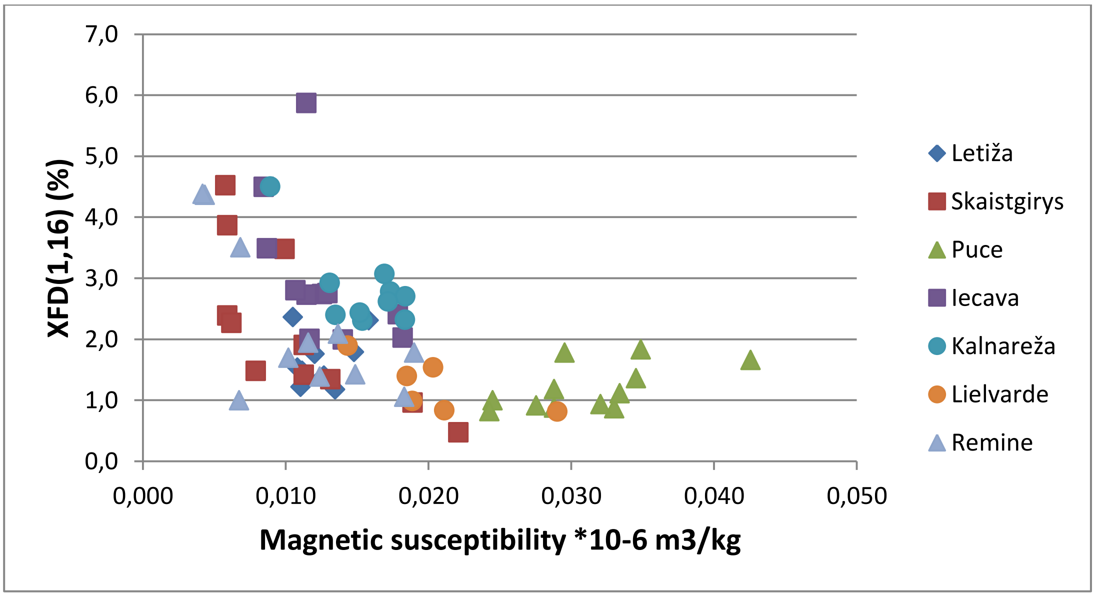


Figure 12. The frequency-dependent susceptibility ($X_{FD)}$ versus magnetic susceptibility (at 1 kHz) for studied outcrops.

The discussed paleo-flow from SE to NW coincides well with previous studies that showed a shallow sea environment with clastic inflow from the north (Stingulis, 2004). This could mean that the AMS ellipsoid is indicating the direction of marine currents coming from the south direction.

The Skaistgirys samples were taken from the Žagare formation, which is characterized by dolomite or sandy dolomite. In the studied specimens, the sandy content can be actually fairly high (40.7 mass%; Table 5). The MS showed lower values than most of the outcrops (Table 4), but the deviation was the highest (±5.5 × 10^{-9} m^3/kg; Table 4). A slight increase of MS from bottom to top can be seen from Figure 6, meaning that the detrital input could have increased in the upper part. The X_{FD} values showing higher values on the bottom part (Figure 7) could possibly be because of too low MS values and thus the X_{FD} showing larger per cent of SP particles. Overall low X_{FD} value of 2.2 % (Table 4) means that there is probably very low content of SP particles and no chemical remagnetizations have taken place. The AMS ellipsoid (Figure 9b) showed the same sedimentary fabric as that of Letiža, showing possible primary magnetization. The magnetic carriers being magnetite and hematite (Figure 10e) means, that magnetite possibly carries the primary magnetization and hematite is of secondary origin. The same magnetic carriers have previously been determined from the same quarry by Katinas and Nawrocki (2004). The direction of paleo-flow showing SW to NE orientation possibly shows the direction of waves coming from south, since the coastline was supposedly to the north (Savvaitova, 1977).

The Puce outcrop represented the Joniškis formation, characterized by sandy or clayey dolomites (studied samples had high content of clay; Al_2O_3 3.2 mass%; Table 5). The MS values were the highest from all the outcrops, being 31.2 × 10^{-9} m^3/kg in average (Table 4). The higher MS value than that of other outcrops could possibly indicate a larger amount of ferromagnetic minerals in the samples. The X_{FD} values calculated for Puce were the lowest from all outcrops, 1.2 % in average (Table 4), and the deviation was also one of the lowest (0.4 %; Table 4). This indicates that there is possibly no SP particles present. When we look at the AMS ellipsoid of Puce outcrop (Figure 9c), we can see that the K_{max} axes are perpendicular to the bedding plane. This means that possibly there has been some flow of liquids through the sediments (Sagnotti, 2011) and therefore the ferromagnetic minerals likely carry secondary magnetization. The magnetic carriers were found to be hematite and (titano-) magnetite, but here both are probably carrying the secondary magnetization and are not good for palaeomagnetic studies. No paleo-flow direction can be detected with Puce’s AMS ellipsoid because of the inverse shape of it.

The Iecava outcrop studied the Stipinai formation with its dolomites. The samples taken from Iecava had low content of terrigenous material in them (SiO_2 0.7 mass%; Table 5). The MS average value of 12.5×10^{-9} m^3/kg (Table 4) is typical of dolomites. Both the X_{FD} (Figure 7) and X_R (Figure 8) showing larger values at the lower part of the studied section could indicate presence of fine grained ferromagnetic minerals and possibly some SP minerals, which is indication for chemical remagnetization of carbonates. Hints of chemical changes to the ferromagnetic minerals can also be seen from the SEM data (Figure 11d), where the larger iron bearing grains have formed in the pores (possibly pyrite). The possibility of secondary magnetization can also be seen from the AMS ellipsoid (Figure 9d), which has the same inverse prolate shape as was with the Puce outcrop. This means that hematite and magnetite, which were identified as main magnetic carriers, are carrying the secondary magnetization component and are not suitable for palaeomagnetic studies. As with the Puce’s AMS ellipsoid, the inverse shape of the ellipsoid in Iecava does not show the possible paleo-flow direction.

The Kalnareža outcrop represent the Ogre formation with its dolomitized sandstones. The high content of terrigenous material can be seen from XRF data (SiO_2 43 mass%; Table 5). The MS average value is 15.4×10^{-9} m^3/kg (Table 4) and shows little change throughout the studied section (Figure 6). The average X_{FD} of 2.8 % (Table 4) means the possibility of SP mineral grains in the samples is quite low, indicating no chemical changes with the magnetic minerals. This fact is also supported by the shape of AMS ellipsoid being perfectly oblate (Figure 9e), meaning that the original sedimentary fabric has been preserved through time. So from the detected magnetic carriers magnetite could possibly carry the primary magnetic remanence and hematite and maghemite are of secondary origin. No paleo-flow direction could be detected from the AMS ellipsoid, because the K_{min} axes align right in the middle of the equal area projection and have no vertical offset to indicate a direction. This would mean that the rock formed in an area with no flow or very weak currents (Sagnotti, 2011).

The Lielvarde outcrop samples were taken from Katleši formation, are represented by clayey dolomitized sandstone. The studied samples had the most terrigenous material from all the outcrops (SiO_2 57.3 mass% and Al_2O_3 5.1 mass%; Table 5). With so much clastic input, one would expect high MS values, but here the values stayed near that of the average from all the outcrops, being 20.4 × 10^{-9} m^3/kg (Table 4). Calculated average X_{FD} and X_R (Table 4) values were the lowest for Lielvarde from all the outcrops, showing little variation (Figure 7; 8). This could indicate that the ferromagnetic fraction consists mainly of coarser ferromagnetic grains and no SP minerals are present. The anomalous shape (K_{int} being perpendicular to the bedding plane) of the Lielvarde AMS ellipsoid (Figure 9f) could possibly be explained by the susceptibility being carried by iron-bearing carbonates, tourmaline, cordierite, goethite, and SD magnetite (Rochette et al. 1992). To know better, Lowrie test should be conducted with the samples, as Lielvarde samples were not included in Lowrie test in current study. Also, new samples should be taken from the outcrop for remeasurements, to rule out any possible occasional error with current set of samples.

The Remine outcrop is represented by the Daugava formation, which is composed by dolomites. The average MS value of 11.1 × 10^{-9} m^3/kg (Table 4) was one of the lowest in studied outcrops and showed decrease from bottom to top (Figure 6), meaning that the detrital input could have decreased in time. The X_{FD} showing opposite trend of increasing upwards (Figure 7) could possibly be because of low MS values giving erroneous X_{FD} values and not meaning an increase of SP minerals. It is also supported by the fact that the X_R parameter shows no distinctive change in its value throughout the section (Figure 8). With the AMS ellipsoid (Figure 9g) still showing the primary sedimentary fabric and X_{FD} average value being 2.2 % (Table 4), the magnetic minerals could be carrying primary magnetization. Since the thermal demagnetization of three component IRM however showed only presence of goethite (that does not carry any remanence), no palaeomagnetic studies can possibly give good results with Remine samples. The lack of iron bearing minerals can also be seen from the SEM study (Figure 11g). The AMS ellipsoid of Remine (Figure 9g) shows a paleo-flow direction of SEE to NWW. With the previous study indicating the transgression of the epeiric sea from the east during Daugava time (Sorokin, 1978), the NWW direction could describe the marine currents coming from the east.

The Salaspils outcrop studied the Salaspils formation, dominated by dolomites and gypsum-rich dolomites. The MS values were the lowest from all the outcrops, being 2.5 × 10^{-9} m^3/kg (Table 4) in average and giving negative values in some layers (Figure 6). This is due to the content of gypsum that is a diamagnetic mineral with negative MS values (Table 1). The high content of sulphur in one of the studied specimens (S_{total} 7.65 mass%; Table 5) is also due to gypsum content. The calculated X_{FD} values were high (Table 4) due to low MS values. Two specimens that were conducted to the Lowrie test did not give any indication of magnetic carriers being present in the samples. The iron bearing mineral grains visible from the SEM image (Figure 11h) are pyrite grains, since they also showed content of sulphur, but pyrite is of secondary origin and does not carry remanence (paramagnetic mineral). The fact that the Salaspils deposits formed in hypersaline lagoon or sabkha environment (Sorokin, 1978) would indicate no flow or very weak flow of water. Though when we look at the AMS ellipsoid (Figure 9h), it shows that the paleo-flow direction is possibly SWW to NEE. This probably indicates some inflow of water to the lagoon from the east, since from the Daugava time (and maybe at the end of Salaspils time) the sea started to transgress from the east (Sorokin, 1978).

All in all, from the eight studied outcrops three would fit for the palaeomagnetic studies – Letiža, Skaistgirys, and Kalnareža, since they show potential primary magnetization and have magnetite (or titanomagnetite) as one of the magnetic carriers. Although the MS values being very low indicates the scarcity of ferromagnetic minerals in them which could make the palaeomagnetic studies hard to carry out.

7 Conclusions

In current study, eight outcrops of Upper Devonian age were studied for their magnetic susceptibility, anisotropy of magnetic susceptibility, and magnetic remanence carriers, to determine their fitness for further palaeomagnetic studies. The concluding data, which can also be seen from Figure 13, is as follows:

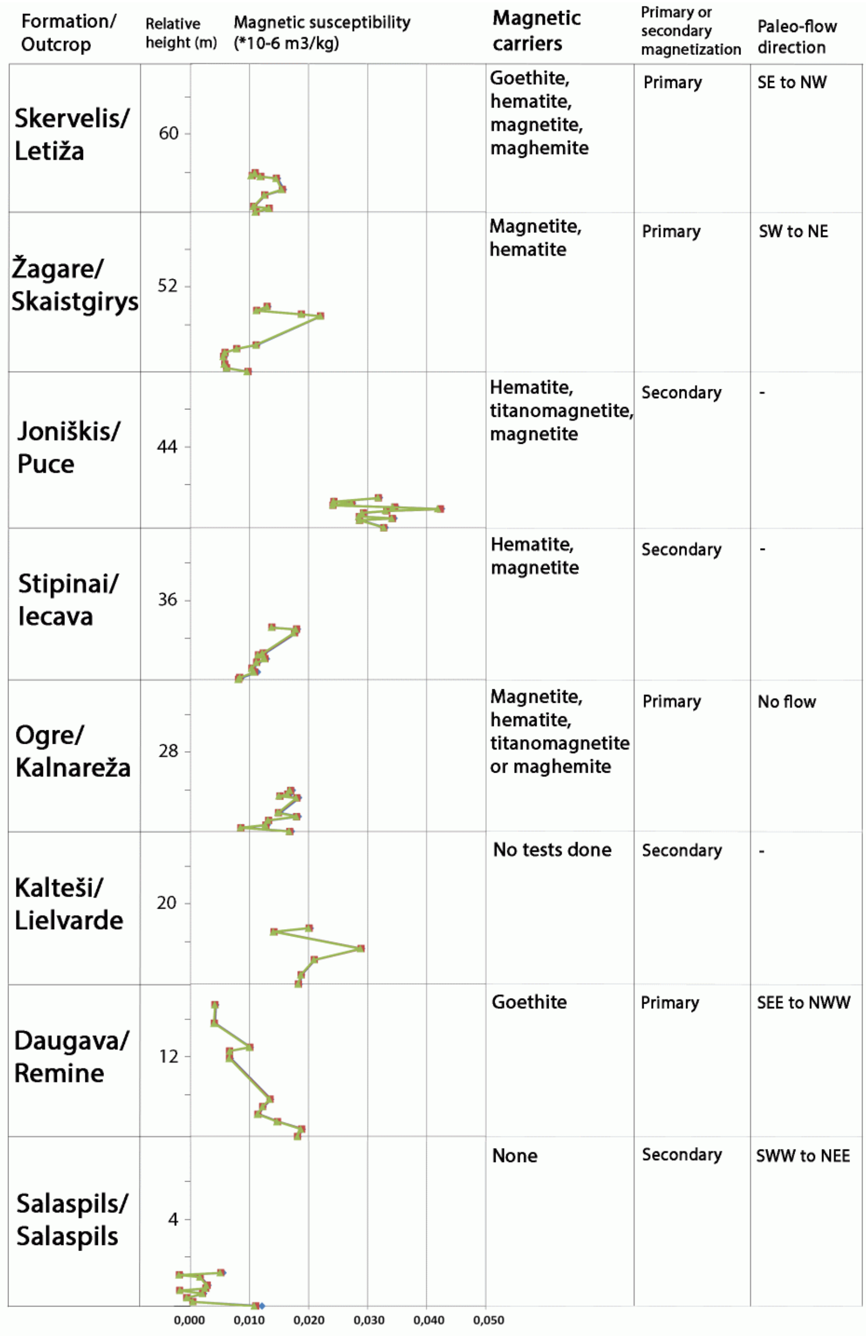
1) The MS for all the studied formations showed very low values, typical to sedimentary rocks. Distinguishable change in MS values within a studied section could be seen at Skaistgirys (increasing upwards) and Remine (decreasing upwards). These changes could indicate the increase or decrease of detrital input in time. The very low or negative values of MS in Salaspils are caused by the high gypsum content in the samples.

2) From the AMS data and X_{FD} calculations it can be said that the primary magnetization component could possibly be found from Letiža, Skaistgirys and Kalnareža with one of the magnetic carriers being magnetite.

3) The studied samples also had goethite, hematite and maghemite as magnetic carriers, but these minerals are of diagenetic origin and are of no interest for primary magnetization studies.

4) The possible paleo-flow directions found are SE-NW in Letiža, SW-NE in Skaistgirys, SEE-NWW in Remine and SWW-NEE in Salaspils.

To conclude, from the eight studied outcrops potentially Letiža, Skaistgirys and Kalnareža have the magnetite with primary magnetization component needed for palaeomagnetical studies, and further studies should focus on these outcrops.



Formation/ Outcrop	Relative height (m)	Magnetic carriers	Primary or secondary magnetization	Paleo-flow direction
Skervelis/ Letiža	60	Goethite, hematite, magnetite, maghemite	Primary	SE to NW
Žagare/ Skaistgirys	52	Magnetite, hematite	Primary	SW to NE
Joniškis/ Puce	44	Hematite, titanomagnetite, magnetite	Secondary	-
Stipinai/ Iecava	36	Hematite, magnetite	Secondary	-
Ogre/ Kalnareža	28	Magnetite, hematite, titanomagnetite or maghemite	Primary	No flow
Kalteši/ Lielvarde	20	No tests done	Secondary	-
Daugava/ Remine	12	Goethite	Primary	SEE to NWW
Salaspils/ Salaspils	4	None	Secondary	SWW to NEE

Figure 13. The concluding data for all the outcrops with their MS, magnetic carriers (according to Lowrie test), character of magnetization (according to X_{FD} and AMS) and detected paleo-flow direction.

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10 Summary in Estonian

Läti Ülem-Devoni settekivimite magnetiline vastuvõtlikkus, magnetilise vastuvõtlikkuse anisotroopia ja magnetismi kandjad

Siim Ots

Antud magistritöö eesmärk oli kaheksa Läti Ülem-Devoni paljandi magnetiliste omaduste mõõtmine selleks et iseloomustada nende magnetilise vastuvõtlikkuse (MV) iseloomu, hinnata sobivust paleomagnetilisteks uuringuteks ning määrata võimalikud paleovoolude suunad. Selleks teostati magnetilise vastuvõtlikkuse, magnetilise vastuvõtlikkuse anisotroopia (AMS), SEM ja XRF mõõtmised. Lisaks teostati osade proovidega kolmeteljelise indutseeritud magnetiseerituse termaalne demagnetiseerimine (Lowrie, 1990) selleks et määrata kivimites sisalduvad magnetismi kandjad.

Saadud tulemused näitavad, et MV väärtused vastavad tüüpilistele settekivimite väärtustele. Skaistgiryse paljandis võib täheldada MV suurenemist ülemistes uuritud kihtides ning Remines MV vähenemist, mis viitab detriitse materjali sissekande hulga muutusele. Salaspilsi väga madalad või negatiivsed MV väärtused tulenevad kivimis sisalduvast kipsist. Võimalikeks paleovoolude suundadeks määrati SE-NW Letižas, SW-NE Skaistgiryses, SEE-NWW Remines ja SWW-NEE Salaspilsis. Paleomagnetilisteks uuringuteks sobivad antud paljanditest kõige paremini Letiža, Skaistgiryse ja Kalnareža, kuna antud uuringud näitasid, et seal leidub magnetiiti, mis võib olla vajaliku primaarse magnetiseerituse komponendi kandjaks. Teised määratud magnetism kandjad, götiit, hematiit ja maghemiit, on sekundaarse päritoluga ning primaarse komponendi uuringutel ei oma tähtsust.

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