UDC 551.73+552.54+549.1 (474.2)
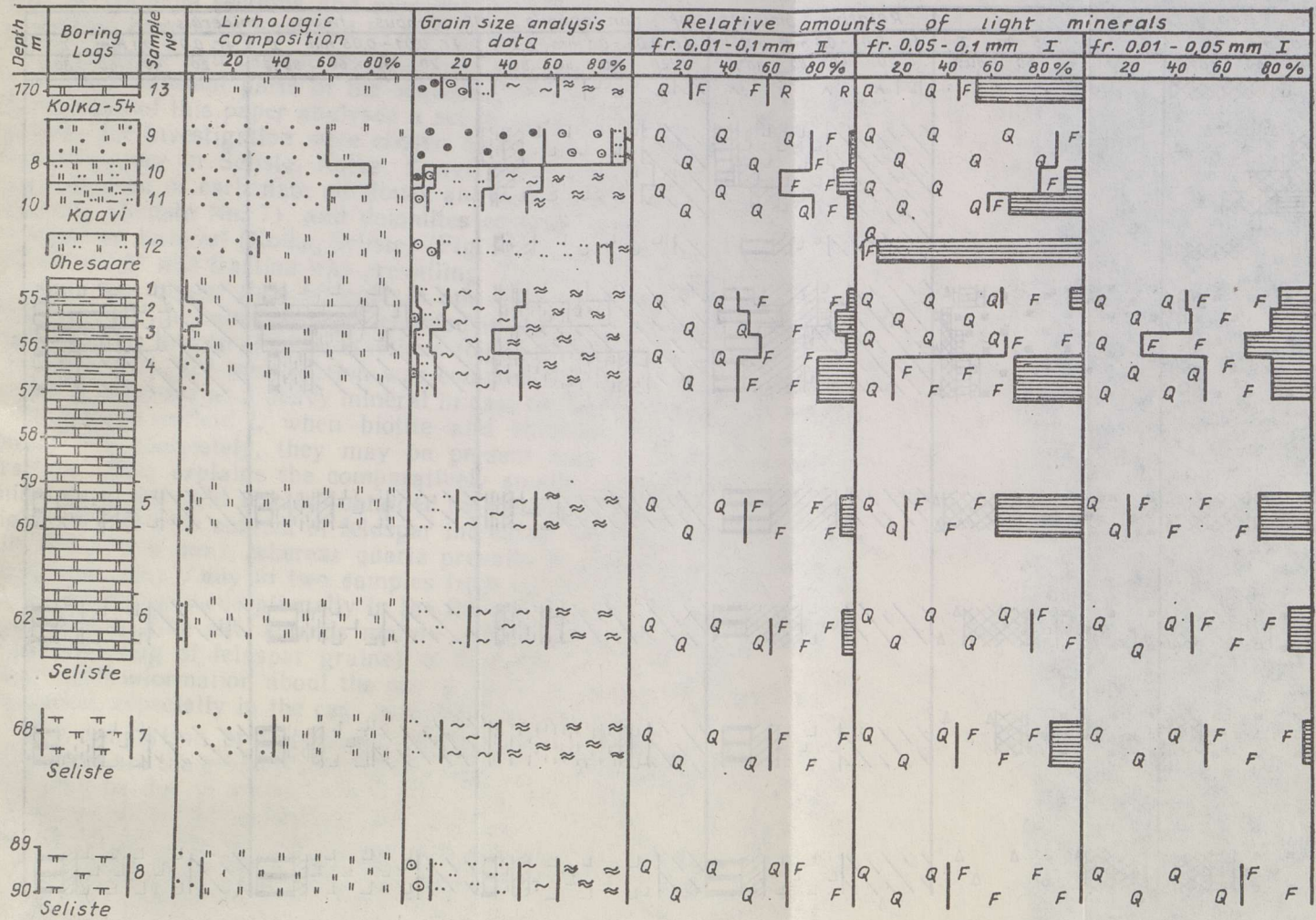
Erika JÜRGENSON * and *Anne KLEESMENT* *

A COMPARISON OF TWO MINERALOGICAL METHODS APPLIED TO THE DETERMINATION OF THE TERRIGENOUS COMPONENT

Abstract. The terrigenous component of Estonian bedrock was analysed using two methods: method I for the Vendian, Cambrian, Ordovician, and Devonian rocks and method II (express) for the Silurian rocks. This paper compares the results of the two methods applied in parallel for the determination of the terrigenous component of the Silurian carbonate rocks. The most essential differences appear in the amounts of micaceous minerals, opaque heavies, and phosphate minerals. The similarity is the greatest in case of the amounts of allothigenous non-opaque minerals, which can be successfully used for the reconstruction of sedimentation conditions and the determination of stratigraphic boundaries.

Investigation of the terrigenous component of sedimentary (resp. carbonate) rocks is of essential help in solving problems of sedimentogenesis and lithogenesis, i. e. problems closely connected with the determination of stratigraphical boundaries and correlations. Examination of allothigenous minerals in the sand-silt fraction under the microscope with the aid of immersion liquids is of special importance. First investigations in this field were started in Estonia by Herbert Viiding about thirty years ago (Вийдинг, 1965). The method was developed on the basis of samples from the Devonian arenaceous sediments. The examined fraction 0.05—0.1 mm of terrigenous minerals was separated by sieving; next the grains with a diameter smaller than 0.01 mm were removed by washing. The 0.05—0.1 mm fraction was separated using bromoform and the light and heavy minerals were determined separately. The most valuable information was received from the allothigenous heavy mineral suite. The method was used for the research of Cambrian and Vendian clastic rocks, and also for the study of the insoluble residue from the Devonian and Ordovician carbonate rocks. The initial weight for a sample of carbonate rock was 1000 g, and it was crushed and dissolved in 10% acetic acid. Further the procedures with the insoluble residue were carried out as mentioned above. This method of the determination of the mineralogical composition of the insoluble residue is very labour- and time-consuming as the dissolution and separation are long-lasting processes. After washing out the fraction finer than 0.01 mm, the amount of the sample left for sieving is often only 50—100 g and consists mostly of aggregates and pyrite and Fe oxides as authigenous heavies. This was the reason why despite of a prolonged period very few Ordovician sections could be investigated using this method.

To increase the labour productivity and the number of studied samples, another method was elaborated for determining the terrigenous component in the insoluble residue of the Silurian carbonate rocks (Юргенсон, 1976). The diameter of the Silurian deepboring core is often only a few centimetres, and the weight of the samples taken for the analysis was 30—50 g. The samples were acidified in 3.5% hydrochloric acid. The size

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Depth m	Boring Logs	Sample №	Lithologic composition	Grain size analysis data	Relative amounts of light minerals		
			20 40 60 80%	20 40 60 80%	fr. 0.01-0.1 mm II	fr. 0.05-0.1 mm I	fr. 0.01-0.05 mm I
					20 40 60 80%	20 40 60 80%	20 40 60 80%
170	Kolka-54	13			Q F F R R	Q Q F	
	Kaavi	9			Q Q Q F	Q Q Q F	
8		10			Q Q F	Q Q F	
10		11			Q Q Q F	Q Q F	
	Ohesaare	12					
55		1			Q Q F	Q Q F	Q Q F F
		2					
56		3			Q Q F	Q Q F	Q F F
		4			Q Q F	Q Q F	Q Q F
57					Q F	Q F F F	
58							
59		5			Q Q F F	Q F F	Q F F
60					Q F	Q F	
62		6			Q Q Q F F	Q Q Q F	Q Q F F
	Seliste				Q Q F	Q Q F	Q F
68	Seliste	7			Q Q F F Q	Q F F	Q Q F F
					Q Q F	Q F	
89	Seliste	8			Q Q Q F	Q F F F	Q Q Q F
90					Q Q F	Q F F	Q Q F F

Fig. 1. The studied sections of Silurian rocks in the Kolka-54, Kaavi, Ohesaare, and Seliste borings with lithologic (terrigenous and carbonate material) and granulometric composition. Light minerals are represented in different fractions analysed by both methods (I, II).

Legend

- limestone
- dolomite
- marl
- domerite
- arenitic domerite
- clay
- siltic clay
- silt stone with dolom. cem.
- sandstone
- sandstone with dolom. cem.
- siltic sandstone
- carbonate component
- terrigenous component
- fr. > 0.1 mm
- fr. 0.05-0.1 mm
- fr. 0.01-0.05 mm
- fr. 0.002-0.01 mm
- fr. < 0.002 mm
- heavies in fr. 0.01-0.05 mm I
- heavies in fr. 0.05-0.1 mm I
- heavies in fr. 0.01-0.1 mm II
- Q Q quartz
- R R chalcedony
- F F feldspar
- An An anhydrite
- mica
- pyrite
- Fe-(hydr)-oxides
- ilmenite + magnetite
- leucoxene
- Sf Sf sphalerite
- △ △ non-opaque allothig. heaves
- zircon
- L L garnet
- tourmaline
- rutile a.a. Ti-minerals
- apatite
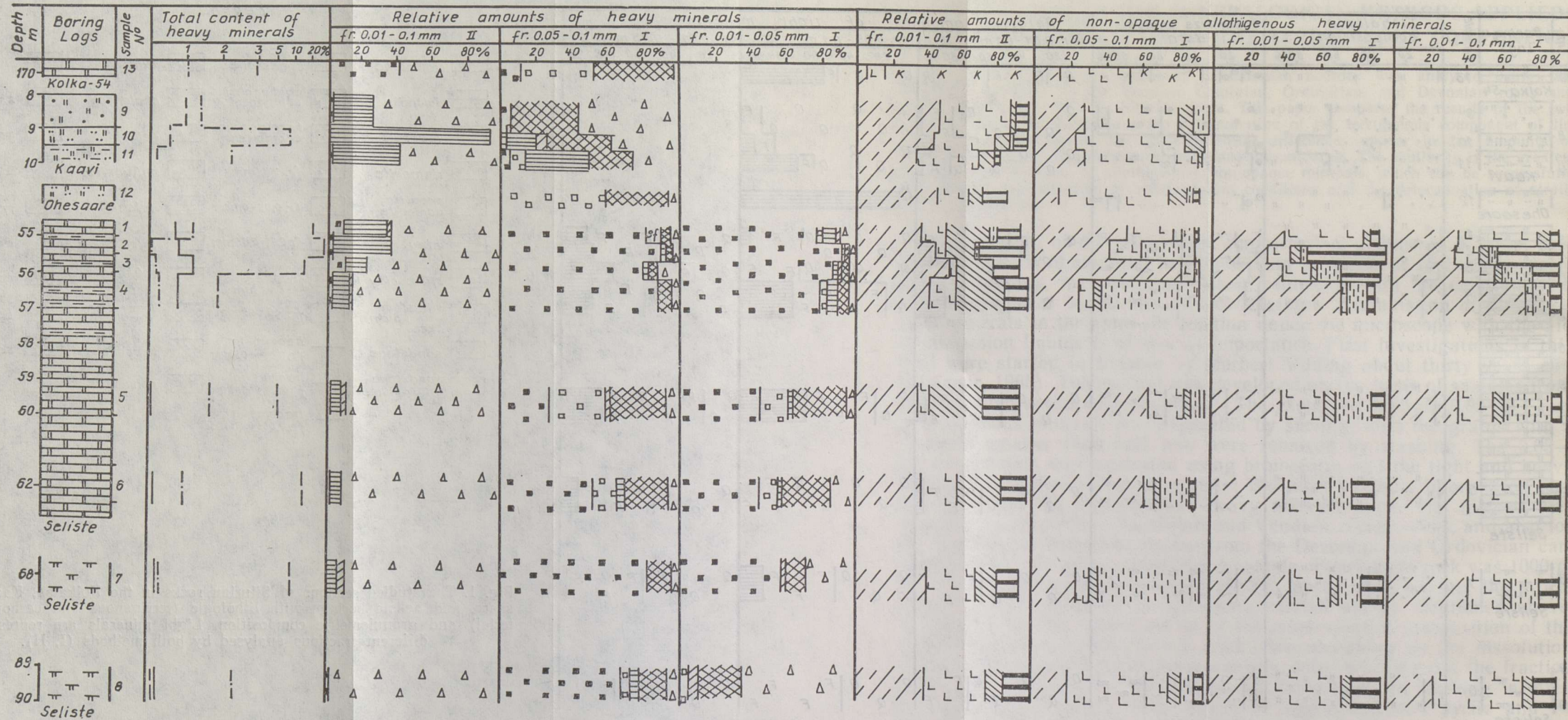- K K corundum
- Ba Ba baryte
- Ts Ts celestine
- varia

Fig. 2. The content of heavy minerals in the insoluble residue of Silurian carbonate rocks. Comparative data of relative amounts of heavy minerals in different fractions are represented for both methods. See legend in Fig. 1.

distribution was determined by pipetting and washing; that of the fractions with a diameter bigger than 0.1 mm by sieving. The terrigenous material in the Silurian carbonate rocks consists mainly of clay and silt fractions and it contains fine sand only in a few cases.

Because of the fineness of the analysed material no separation of the heavy fraction occurred in bromoform. Heavy and light mineral grains were counted in parallel under the microscope. The percentage of light and heavy fractions was calculated from the sum of the grains counted (500—600). The mineral identification was carried out using immersion liquids like in case of the first-mentioned method. About 1300 samples from 52 borehole sections and numerous outcrops of the Silurian carbonate rocks have been analysed by this method (Юргенсон, 1988).

To compare and use the results of the analyses received by methods I and II for different parts of the sequence of bedrock in the Baltic area, the authors of this paper analysed a set of samples by both methods. The sections for investigation were chosen at the level of the Silurian-Devonian boundary in Seliste, Kolka, Kaavi, and Ohesaare boreholes. They are composed of carbonate siltstones and sandstones (Kaavi, Ohesaare), domerite (Seliste No. 7), and dolomites containing different amounts of terrigenous material (Kolka, Seliste) (Fig. 1). In most of the studied samples the <0.01 mm fraction was prevailing. The percentage of the sand-silt fraction was bigger than 50 only in one third of the analysed samples. By method I, the minerals were counted in 0.01—0.05 mm and 0.05—0.1 mm fractions and in the two fractions together, while by method II only the whole 0.01—0.1 mm fraction was counted. Differently from method I, biotite is regarded as a heavy mineral in case method II is applied, whereas in case of method I, when biotite and chlorite do not separate in bromoform completely, they may be present both in light and heavy fractions. This explains the comparatively smaller amount of micaceous minerals in the light fraction obtained by method II (Fig. 1). Among the light minerals, the content of feldspar increases relative to quartz in fine silt (0.01—0.05 mm), whereas quartz prevails in the coarse silt fraction (0.05—0.1 mm). Only in two samples from the Seliste core (Nos. 4 and 8) quartz prevails exceptionally in the fine silt fraction. The quartz: feldspar ratio in fine silt depends mainly on the mechanical differentiation (light crushing of feldspar grains) of the sediment, and thus does not yield much information about the source material and conditions of sedimentation, especially in the case of authigenic feldspar.

The comparison of the results got by the two methods shows that differences are the greatest in the total amount of heavy minerals (Fig. 2). This may be due to errors and differences in counting as well as difficulties faced in the determination of opaque minerals in the fine silt fraction. Partly the differences can be explained by the greater solubility of Fe oxides (hematite) in hydrochloride acid. The differences in the results of the analyses seem to decrease with the increasing content of terrigenous material in rocks.

The content of non-opaque allothigenous heavy minerals is somewhat bigger in the fine silt fraction than in the coarse silt fraction (Fig. 2). The difference is the most notable in sample No. 8 from the Seliste borehole. In dolomites and domerites the opaque minerals are concentrated in the coarse silt fraction, while in clastic rocks the situation is the opposite.

In comparing the two methods, attention was focused on non-opaque heavy minerals which are quite diagnostic of the terrigenous material in carbonate rocks. Zircon, garnet, tourmaline, and rutile are the main minerals delivering information since they exist in sufficient quantities in most carbonate rocks. Comparing the results obtained by applying the

Table I

Heavy non-opaque mineral composition of the sand-silt fraction of insoluble residue counted by the different methods

Sample No.	Section; depth, m	Method	Zircon	Garnet	Tourmaline	Staurolite	Kyanite	Apatite	Corundum	Rutile	Titanite	Anatase	Monazite	Hornblende	Pyroxene	Epidote
1	Seliste 54.5—55.0	II	38.1	12.3	22.2	—	3.6	2.9	—	7.9	3.6	—	—	5.0	2.9	1.5
		I	51.8	28.0	2.7	0.3	—	8.0	0.3	2.4	—	—	—	4.1	2.1	0.3
2	Seliste 55.0—55.3	II	42.0	5.4	21.7	—	2.6	2.6	0.7	17.2	5.1	—	—	1.1	1.6	—
		I	41.8	18.0	1.1	—	0.4	30.8	0.6	0.9	0.4	—	0.6	4.8	0.2	0.4
3	Seliste 55.8—55.9	II	44.8	7.3	22.8	—	2.4	1.5	0.9	12.7	2.9	—	—	0.9	3.8	—
		I	84.9	6.5	2.0	—	0.2	2.3	—	2.3	1.0	0.2	0.2	0.4	—	—
4	Seliste 56.7—56.8	II	38.7	14.4	27.7	—	—	2.9	—	11.5	1.3	1.2	—	—	2.3	—
		I	26.7	9.1	4.9	0.6	—	54.5	2.6	—	—	0.6	—	3.0	1.2	—
5	Seliste 59.6—59.7	II	35.9	5.7	29.0	—	2.4	5.2	0.7	15.7	2.4	0.6	—	2.0	3.1	—
		I	66.6	19.6	2.6	0.7	—	0.5	—	2.6	—	—	—	2.0	—	—
6	Seliste 62.0—62.3	II	39.1	20.7	23.5	—	—	—	0.5	8.6	3.8	1.9	0.5	—	1.9	—
		I	64.1	6.8	4.4	—	1.6	15.9	—	2.9	—	—	—	4.4	0.5	0.7
7	Seliste 68.3	II	52.9	23.4	8.2	—	—	0.9	0.5	11.0	0.4	0.4	0.2	0.4	0.8	—
		I	26.0	8.4	3.8	—	—	58.8	1.4	0.8	—	—	—	1.6	0.2	—
8	Seliste 89.5	II	34.0	44.2	9.9	0.2	—	—	—	8.5	1.3	0.2	—	—	1.2	—
		I	22.5	57.6	5.7	—	—	5.7	—	1.4	5.7	—	—	—	—	—
9	Kaavi 8.6	II	42.6	41.7	6.9	0.8	—	—	—	6.5	0.4	—	0.6	0.8	—	—
		I	28.6	52.9	7.8	—	—	8.3	—	1.2	0.7	—	—	0.2	—	—
10	Kaavi 9.0	II	34.7	39.5	12.6	0.2	—	—	0.6	11.0	0.6	0.1	—	0.3	0.4	0.3
		I	23.3	61.6	11.1	0.2	—	0.6	0.2	1.0	0.6	0.6	—	0.4	0.6	0.2
11	Kaavi 9.6	II	33.4	41.5	16.4	—	—	0.8	—	5.2	2.0	—	—	0.3	—	—
		I	28.4	64.4	1.6	1.0	—	—	2.1	1.2	0.4	—	0.2	1.6	1.0	0.2
12	Ohesaare	II	45.4	17.9	8.4	—	—	2.9	—	14.7	6.3	1.0	—	3.2	—	—
		I	14.3	62.7	8.6	—	—	—	—	2.9	—	—	—	2.9	5.7	—
13	Kolka 167.8	II	3.7	10.5	—	—	—	—	84.3	—	—	—	—	1.0	—	0.5
		I	14.6	39.5	—	4.2	—	2.1	37.5	—	—	—	2.1	—	—	—

two methods (Table 1, Fig. 2), we must consider that the amount of apatite is modest in the samples dissolved in hydrochloric acid where it is prone to go into solution and to decompose. Certain differences are notable in the content of tourmaline and rutile as well. In the samples treated by method I, rutile concentrates mainly in the 0.01—0.05 mm fraction (Fig. 2), and its total content (fr. 0.01—0.1 mm) is considerably smaller than in the corresponding fraction received from the samples by method II. Analogous differences have been established in the content of tourmaline. These differences may be due to the shape and habitus of rutile and tourmaline grains affecting the results of sieving. We cannot measure the "pure" size of a grain by sieving, as the result is affected by both size and shape.

Table 2

Average diameter $(\bar{x})$ and median (Me) of the grains of zircon (120 measurings in mm in one sample) and garnet (90), and average length $(\bar{y})$ of tourmaline (80) and rutile (80) grains

Location and depth, m	Zircon		Garnet		Tourmaline		Rutile	
	$\bar{x}$	Me	$\bar{x}$	Me	$\bar{y}$	Me	$\bar{y}$	Me
Seliste 54.5	0.036	0.033	0.068	0.080	0.040	0.036	0.045	0.042
„ 56.7	0.026	0.025	0.038	0.038	0.040	0.038	0.035	0.033
„ 62.0	0.035	0.034	0.042	0.040	0.039	0.036	0.039	0.038
„ 69.5	0.037	0.035	0.046	0.048	0.044	0.043	0.036	0.036
„ 238.5	0.038	0.038	0.044	0.042	0.051	0.050	0.036	0.036
„ 342.1	0.045	0.048	0.051	0.049	0.046	0.048	0.043	0.044
Kaavi 8.9—9.6	0.050	0.050	0.070	0.070	0.055	0.056	0.050	0.050
Ohesaare	0.030	0.030	0.040	0.040	0.040	0.040	0.040	0.040

A special measurement was carried out with the four main minerals in samples from different depths under the microscope (Table 2). In the terrigenous material of carbonate rocks, zircon and garnet are mostly well-rounded with an average diameter 0.026—0.050 mm for zircon and 0.038—0.07 mm for garnet. Tourmaline and rutile are subrounded preserving still their elongated crystal form. They may behave differently at sieving because the ratio of their length and diameter is about 2—4 : 1, the average length of tourmaline grains being 0.04—0.051 mm and that of rutile 0.035—0.05 mm. Thus, some grains of rutile and tourmaline may be lost at sieving, and some tourmaline may stay in the light fraction at separation in bromoform.

The similarity between the results of the two methods is greater when the content of the terrigenous component in a sample is higher like in the case of carbonate siltstones of the Kaavi boring (Fig. 2). It is clear that the results received by method II cannot be compared with the results for 0.05—0.1 mm fraction of the first method. Neither can we compare the contents, but only the tendency of the amounts of minerals toward increasing or decreasing.

Evidently, zircon has a maximum in the Seliste boring at the depth 55.8—55.9 m, while garnet is represented abundantly at the depth of 89.5 m and in the samples of the Kaavi boring. Rutile is relatively abundant in the Seliste boring at the depth 55.2—55.3 m. The content of corundum has a maximum in the sample of the Kolka-54 boring. The presence of some accessory minerals is significant, too. In the upper part of the Seliste section (54.5—59.7 m), a notable appearance of kyanite and pyroxene (Table 1) occurs.
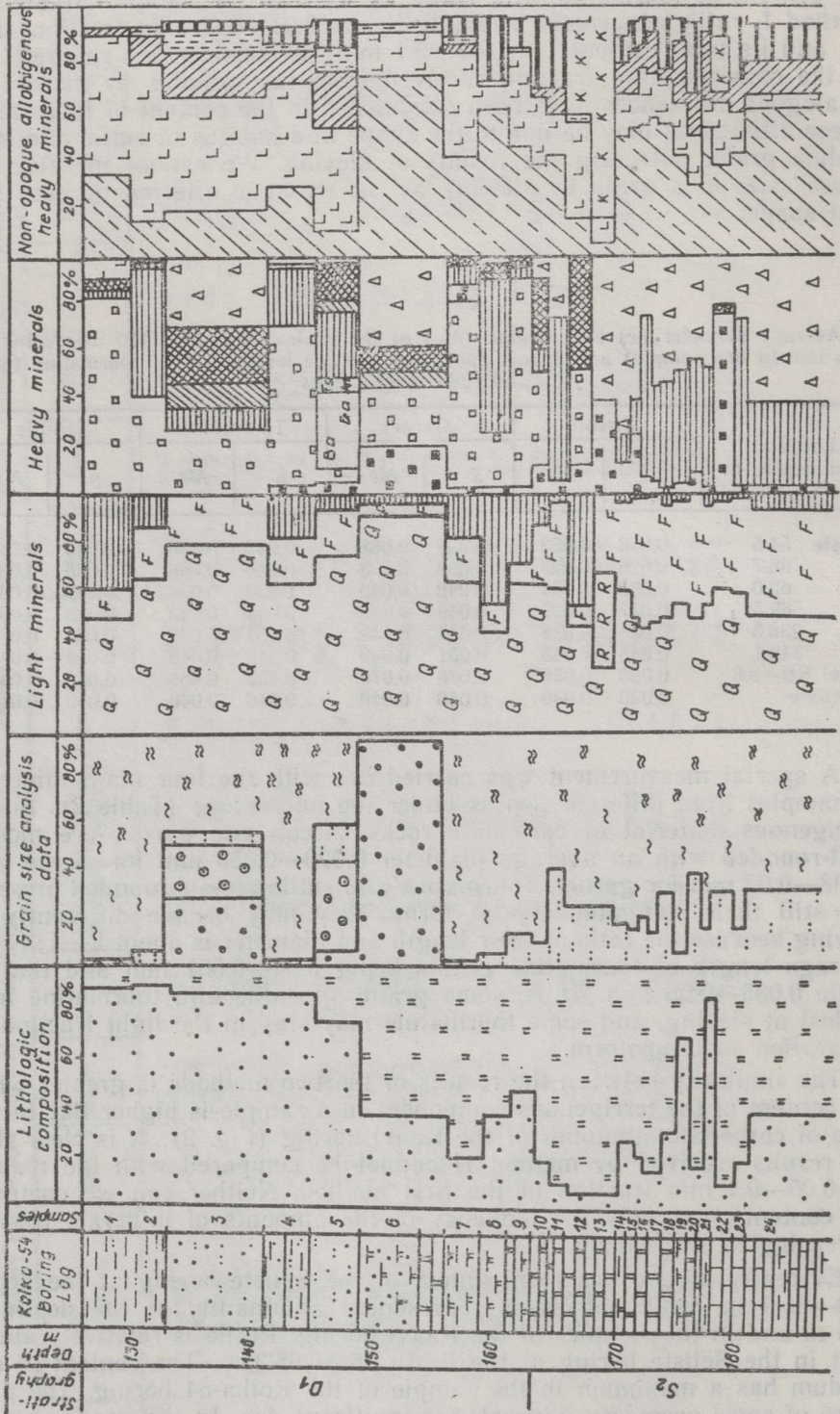
Fig. 3. Lithologic, granulometric, and mineralogic composition of the sequence of the Kolka-54 boring. Samples 1—10 and 12 were investigated by method I and samples 11 and 13—24 by method II.

Taking into account the studied material, we can conclude that there is a characteristic relationship between the results of both methods. Considering the above-mentioned differences of the appearance and amount of different minerals (biotite, apatite, tourmaline, opaque minerals), we can successfully use both methods for the reconstruction of the geological circumstances. As an example, we can consider the sequence of the Kolka-54 boring at the Silurian-Devonian boundary, in which the samples of the Devonian beds (except one sample) were investigated mainly by method I (Fig. 3) and the Silurian beds by method II. The results give us an idea of the succession of the geological events at the end of the Caledonian phase of sedimentation. Taking into account the content of non-opaque allothigenous minerals, we can make for instance some provenance determinations. The appearance of corundum in the Upper Silurian beds indicates the continuation of nearshore sedimentation conditions also in the Lower Devonian. The mineralogical character of the beds at the stratigraphic boundary gives evidence of a steady sedimentation process. A notable change in the mineralogical composition indicating a possible break in sedimentation took place at the level between samples 5 and 6 (Fig. 3); in sample 6 the maximum of zircon and quartz content occurs. At this level, relatively coarse and well-weathered material is concentrated. Upwards in the younger sediments, the amount of tourmaline and apatite increases. In the beds immediately overlying those of sample 6, we can find typical minerals of the sedimentation break, such as baryte, coelestine, and anhydrite. The character of the Lower Devonian sedimentary rocks in the Baltic area indicates a shallow sedimentation basin where the sedimentation process was interrupted by frequent breaks.

We can conclude that both methods may give valuable information about the geological processes that occurred in the past. This investigation suggests that the results obtained are comparable. The choice of the analysis method depends on concrete circumstances. There exist numerous mineralogical analyses which wait for geological interpretation. A preliminary generalization of the mineralogical data of the Baltic bedrock has been given by Viiding and the present authors (Вийдинг, 1976; Юргенсон and Вийдинг, 1982; Вийдинг et al., 1983), but a more detailed treatment of the problem is still needed.

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TERRIGEENSE KOMPONENDI MINERAALSE KOOSSEISU MÄÄRANGUTE SÕLTUVUS METOODIKA VALIKUST

Terrigeense komponendi määranguid Eesti aluspõhja kivimeis on tehtud kahe metoo-
dika järgi (I, II) ja kõrvutatud tulemusi, mis saadi siluri karbonaatkivimite terrigeense
komponendi mineraloogilise koostise paralleelsel määramisel mõlema metoodikaga (tab.
1, joon. 1, 2). Põhierinevused ilmnesid vilkude, raskete opaaksete ja fosfaatsete mine-
raalide puhul. Lähedasemaid tulemusi andsid allotigeensete raskete läbipaistvate mine-
raalide määrangud. Kontrollimaks tera kuju mõju granulomeetrilisele koostisele mõõdeti
eraldi tsirkooni, granaadi, turmaliini ja rutiili teri mikroskoobis (tab. 2).

Saadud tulemuste kõrvutusest selgub, et mõlemat metoodikat on võimalik edukalt
kasutada kivimite settimistingimuste ja settebasseini arengu rekonstrueerimisel ning
stratigraafiliste piiride määramisel. Neid on võimalik kasutada ka eraldi teatud läbilõike
üksikute osade mineraloogilise koostise määramisel, kusjuures säilib muutuste pidevus
(joon. 3).

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СРАВНЕНИЕ ДВУХ МЕТОДИК ОПРЕДЕЛЕНИЯ МИНЕРАЛЬНОГО СОСТАВА ТЕРРИГЕННОГО КОМПОНЕНТА

Сравнены результаты параллельного определения минерального состава терри-
генного компонента силурийских карбонатных пород Эстонии двумя методиками
(табл. 1, рис. 1, 2). Основные различия выявлены в составе слюд, тяжелых непро-
зрачных и фосфатных минералов. Более близкие результаты получены для аллоти-
генных тяжелых прозрачных минералов. Под микроскопом проведены измерения
зерен циркона, граната, турмалина и рутила (табл. 2) в целях проверки влияния их
формы на гранулометрический состав. Установлено, что обе методики успешно
применимы для изучения условий осадконакопления и реконструкции развития
осадочного бассейна, а также для определения стратиграфических границ. Примени-
тельно к конкретному отрезку одного разреза каждая из методик дает сходную
картину изменения минерального состава во времени (рис. 3).