ЦИК core research centre

V.I.SHPILMAN RESEARCH AND ANALYTICAL CENTRE FOR THE RATIONAL USE OF THE SUBSOIL

Lithological and mineralogical studies

- Layer-by-layer lithological core description
- Grain size analysis
- Petrographic analysis of thin sections
- Electronic raster microscopy
- Microtomography
- Determination of mineral composition by X-ray diffraction (XRD) method
- Thermal analysis
- X-ray fluorescent analysis (XRFA)
- Determination of microelement composition by ISP-AES method
- Determination of ultra-microelement composition by ISPMS method

Lithological and petrographic studies

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The core description – lithological characteristic of rocks

While core studying the complete layer-by-layer description is carried out (the layers having significantly uniform lithological structure are visually identified, thickness of each layer is measured, rock identification, color, density, structural and textural features, straticules, lenses, inclusions, pore spaces, organic chemistry, signs of oil saturation, difference of this layer from previous, contact is defined)

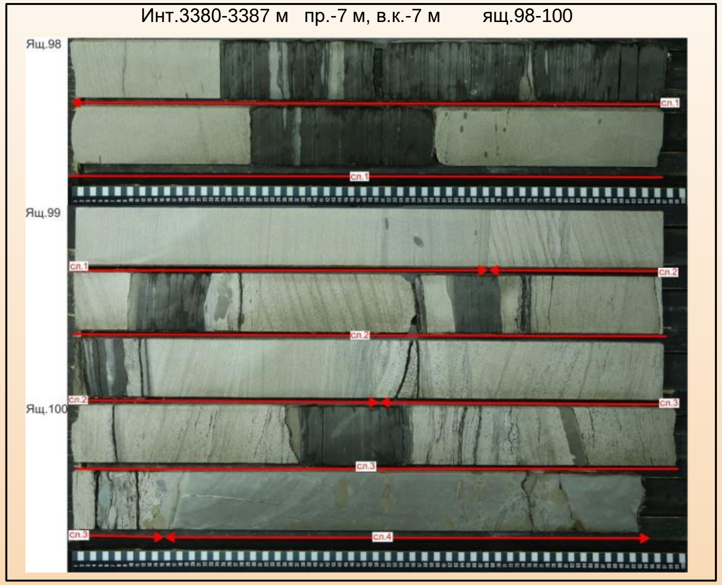
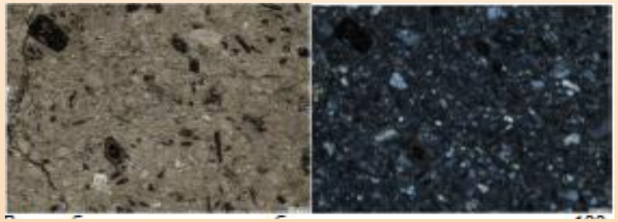


Photo of core in boxes, arrows indicate the identified layers

The layer (depth in core: 2 - 1.77 m) is light gray sandstone, coarse-grained, medium-grained, on clay cement, thin beds of rock are uniform, with horizontal, inclined, undulating lamination. The lamination is caused by alternation of straticules of uneven-grained sandstone and very thin alluviation of carbonaceous - argillaceous material (photo 82, photo 83). The grain sizes increase to the middle of the layer, and decrease from the middle of the layer up to the end. At depths of 37 cm, 89 cm, 1.29 m and 1.34 m from the layer top four intercalations (12 cm, 8 cm, 1.5 cm and 2.5 cm respectively) dark gray, argillaceous siltstone, sideritized intercalations, with horizontal minute folding caused by thin alluviation of lighter silty sand material. Four intercalations of the thin alluviation of carbonaceous material passing with places into lenses are defined on the layer (photo 82). At the depth of 51 cm from the layer top the intercalation with inclusions of coarse pebble of 5 cm thick is observed. Rounded pebble, sideritized, the size is from 5x8 mm to 1.5 x 4.5 cm. At the depth of 1.31 m from the layer top three pebbles of argillaceous and sideritized rocks, gray, gray blue and brown color, by the size ~ 1x1.5 cm are observed. Hydrocarbons are odorless.Contact with an underlying layer is accurate, undulating.

Photo 82. 1.03 m from layer top - coarse-grained sandstone with straticules and lenses of carbonaceous material.

Photo (depth of core: 3554 m - 3 m from layer top). Gray with a greenish tinge (basalt?), dense, hard, massive, fractured, with spots, fragments of more dark main rock



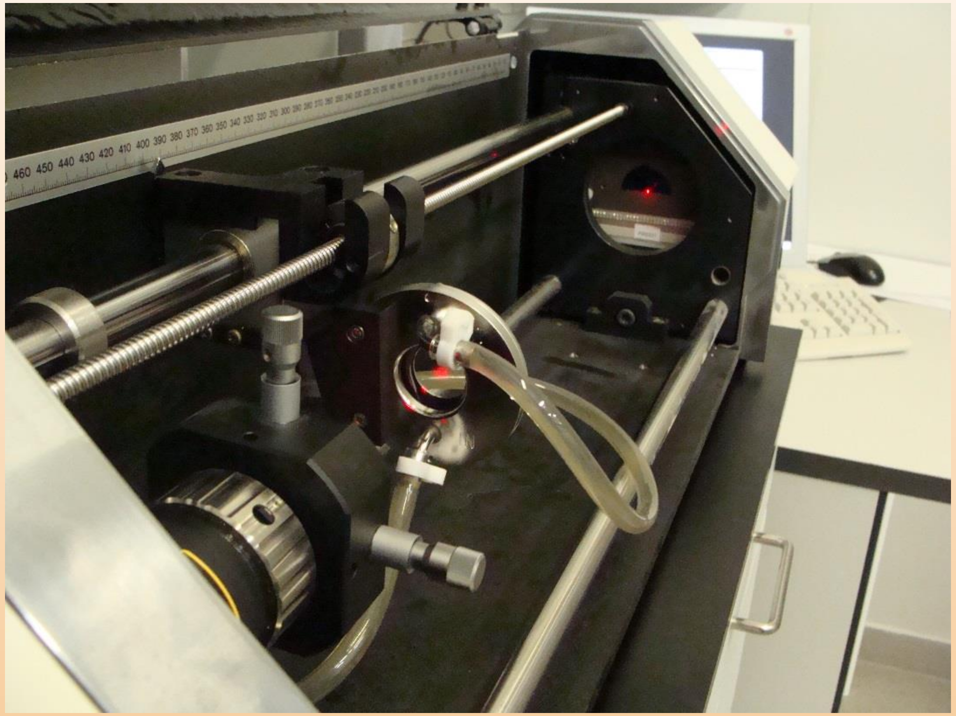
View a) without analyzer, view b) - with analyzer, 100x optical zoom
Thin section: the depth of core: 3,555.2 m - Carbonatized, chloritized, porphyritic trachyandezibazalt, with microlitic structure, intersalate texture fragments of groundmass, with massive texture, slightly fractured. Single dendrite and convulated streaks of chlorite - carbonate structure are presented.
Extended, slightly dendrite, convulated cracks are presented by thin aggregate mix of argillaceous and ore minerals.

Example of the layer-by-layer macrodescription of rock and the thin section description of rock

Lithological and petrographic studies

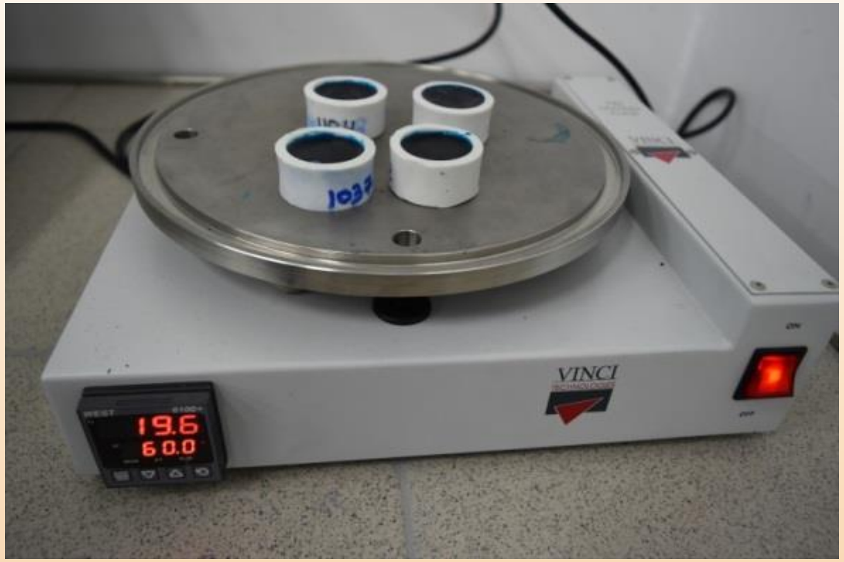
Lithological characteristic of rocks Grain size distribution

The grading analysis is made using traditional sieve method. Crushed, hydrochloric acidized, the dried-up tests washed from clay fraction, dissipate on set sieves made Fritch, or are investigated on the laser analyzer Fritch Analisette 22.



Lithological and petrographic studies

Petrographic thin section preparation



Petrographic thin section preparation

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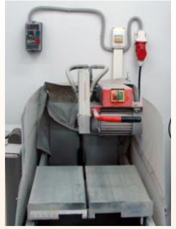
Phase 1

Core sampling for petrographic thin section preparation of 20x30 mm in size or Ø of 30 mm, is made using specialized cutting machines.

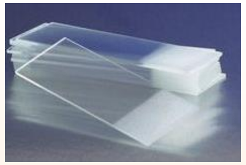
The samples are linked to the depth of sampling and marked.

A visual inspection of the sample, cementation by epoxy resin, preliminary and thin processing on abrasives of different granularity are then produced.



Phase 2

The glass surface is processed using the cutting machine. Buehler Petro-Thin used for fixing a sample



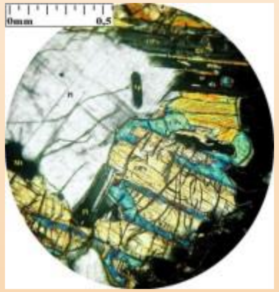
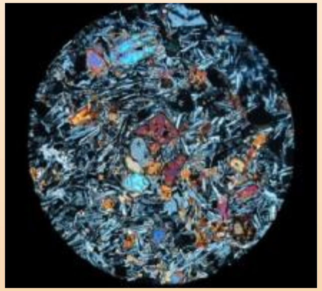
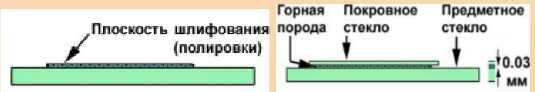

Fixing a sample to the glass is produced by epoxy resin and it is a critical phase of thin section preparation. Before the beginning of operation, epoxy resin is processed by vacuum for air elimination.

Hardening of epoxy resin is 12 hours.


Phase 3

The sample is cut off from the glass slide on the Buehler Petro-Thin machine and polish to thickness of 30-50 microns.


The final polishing of the thin section is carried out manually on glass with an abrasive until done; the required thickness and surface finish are controlled using a microscope.

If necessary epoxy resin is colored by colored matter, a coating glass is fixed on the thin section.

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Lithological and petrographic studies

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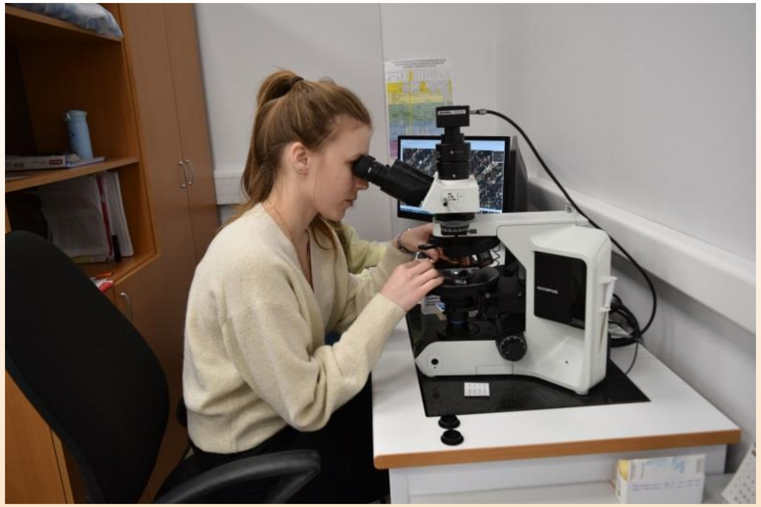
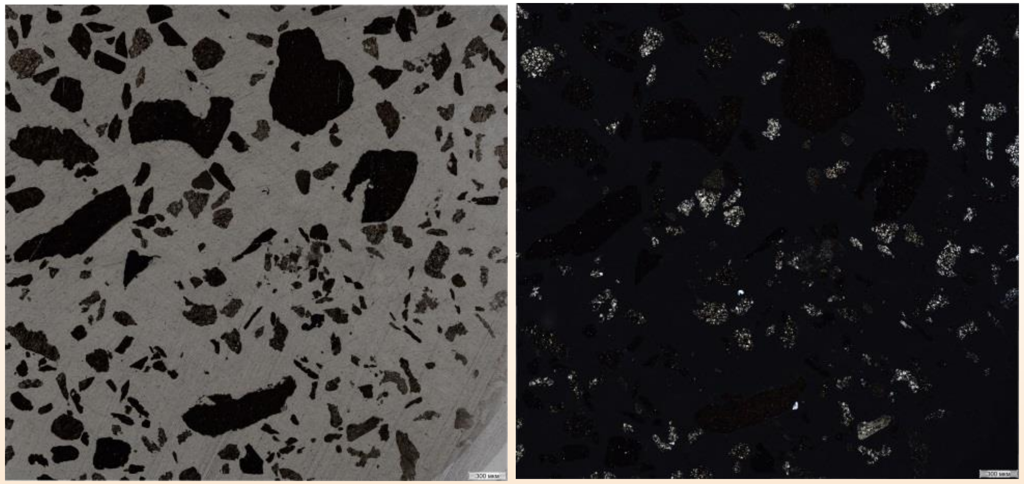
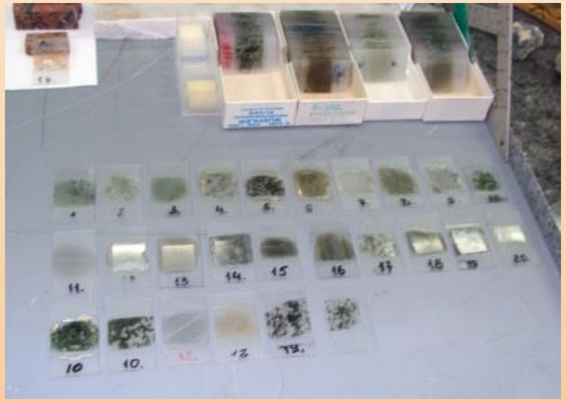
Petrographic thin section description

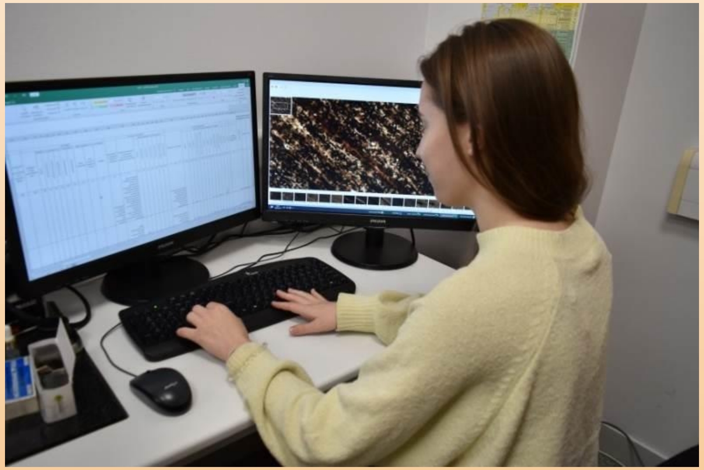


Petrographic thin section is a rock slide of 0.02-0.03 mm. The majority of minerals at such thickness of a cut are transparent that allows to study them by means of polarization microscope (mineral composition of rock, optical characteristics of minerals, their shapes, the nature of chemical interaction, etc.)



Photos of thin section at parallel and crossed Nicol.



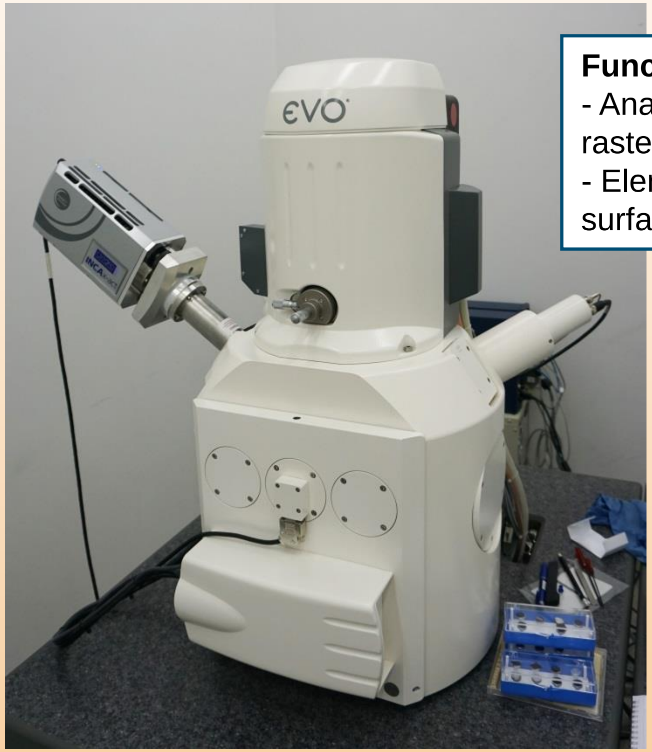


Studying material and chemical core composition

Electron microscopic investigation

Equipment

suitable for electron microscopic investigation:



Function:
- Analysis of sample surface with raster pattern
- Elemental mapping of sample surface

EVO 50 (Carl Zeiss, Germany) Microscope equipped with Inca Energy 350 energy dispersive spectrometer (Oxford Instruments, Great Britain)

Suitable for sample preparation:

Function:
Precision finish of core sample surface for subsequent analysis using scanning electron microscopy.

SEM Mill sample Model 1060 Ion Etching System (Fischione Instruments, USA)

The Raster Electronic Microscopy (REM) is based on interaction of an electron beam (electronic sonde) with a sample surface. Electrons of the sonde interact with sample material and generate signals of various physical nature which are used for synchronous raster pattern on the monitor screen

Studying material and chemical core composition

core research centre

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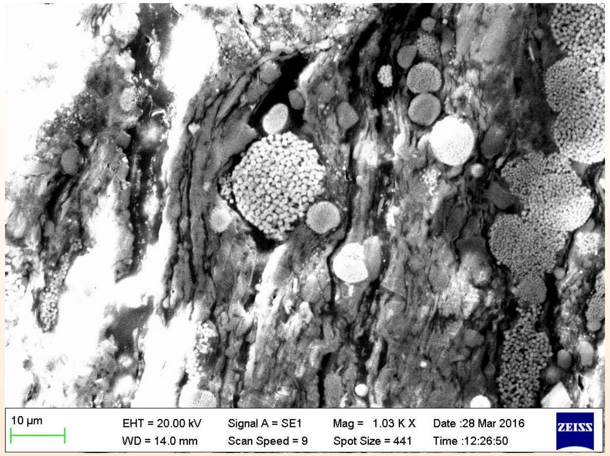
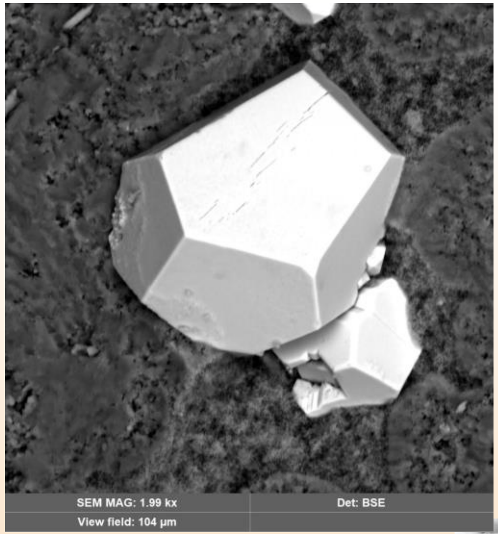


the Bazhenov suite sample
(1000x optical zoom)

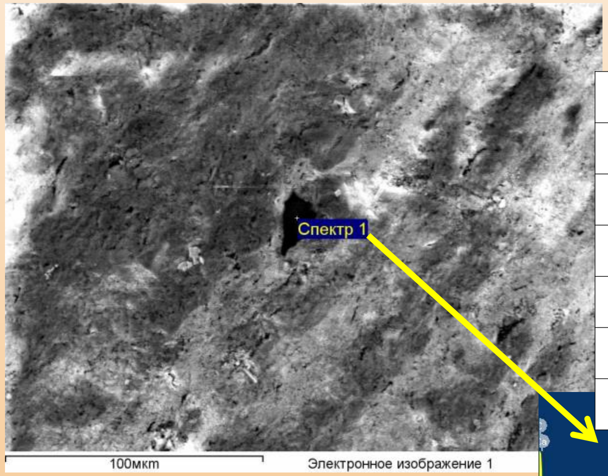


Pyrite crystals in siliceous rock

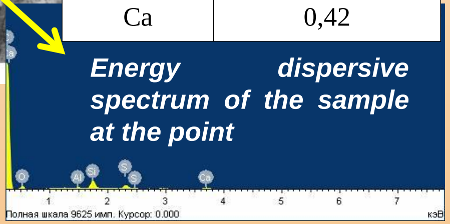
Electron microscopic investigation

Application:

- description of porosity structure
- studying texture of core surface
- identification of phases forming microinclusions in minerals
- elemental mapping of sample surface
- identifications of mineralogical and geochemical characteristics of deposits of oil source rocks

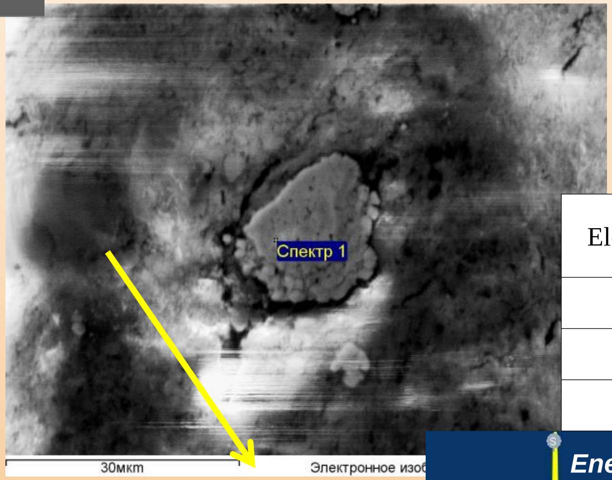


Element	Contents, %
C	**89,07**
O	8,03
Al	0,14
Si	1,45
S	0,90
Ca	0,42

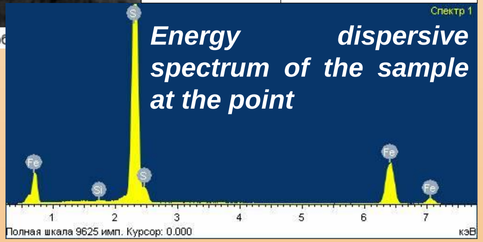


Energy dispersive spectrum of the sample at the point

Organic matter fills one of the pore space

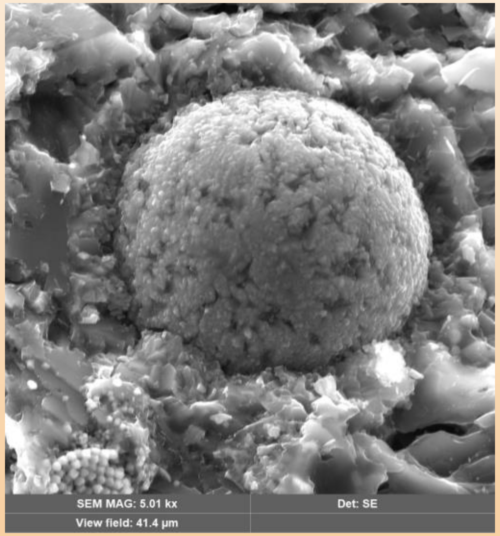


Element	Contents, %
Si	0,20
S	**58,49**
Fe	**41,31**



Energy dispersive spectrum of the sample at the point

Globular pyrite formed as bacteria waste product



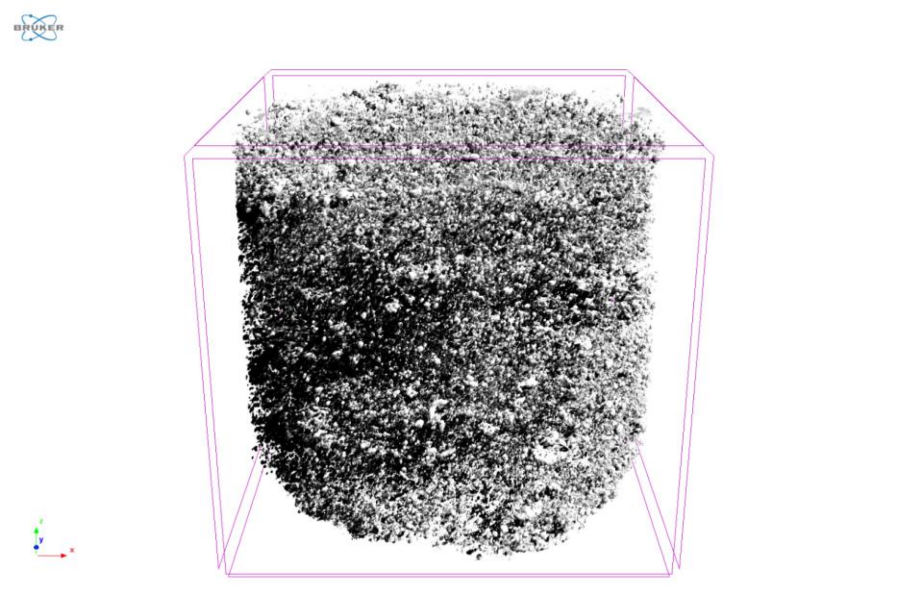
Calcishera filled with calcite in dolomite rock

Studying material and chemical core composition

Tomographic studies

SkyScan 2214 X-ray Nanotomograph (BRUKER, Belgium)

Function:
It opens possibilities for 3D imaging and exact accurate modeling of geological materials in oil and gas exploration



Core sample of the Bazhenov suite

Studying rocks using X-ray method is based on difference in density of rock, mineral inclusions, pore spaces and fractures, and reservoir fluids filled them. X-ray radiation, passing through rock, loses power in proportion to its density and is registered by matrix element of receiver, forming the pixel image

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Studying material and chemical core composition

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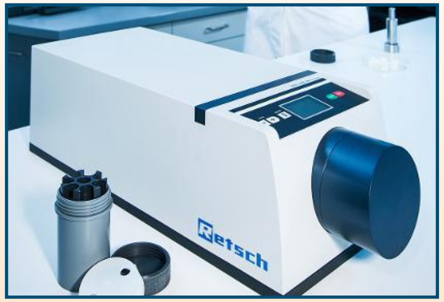
Determination of mineral composition of rock samples by X-ray diffraction (XRD) method

ARL X'TRA Diffractometer (Thermo Fisher Scientific, Switzerland)

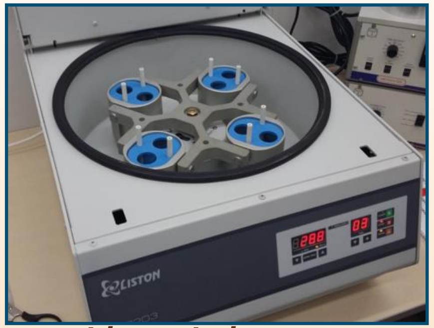
The measuring part consisting of X-ray tube, the automatic feeder and two detectors

Function:
Determination of mineral composition of powder test and oriented clay fraction preparation

- suitable for clay fraction preparation:



XRD-Mill McCrone Vibration Mill of *(Retsch, Germany)*



Liston Laboratory Centrifuge *(Liston, Russia)*

EKPS-10 Muffle Furnace *(Russia)*

SNOL Drying Oven *(SNOL, Lithuania)*

Studying material and chemical core composition

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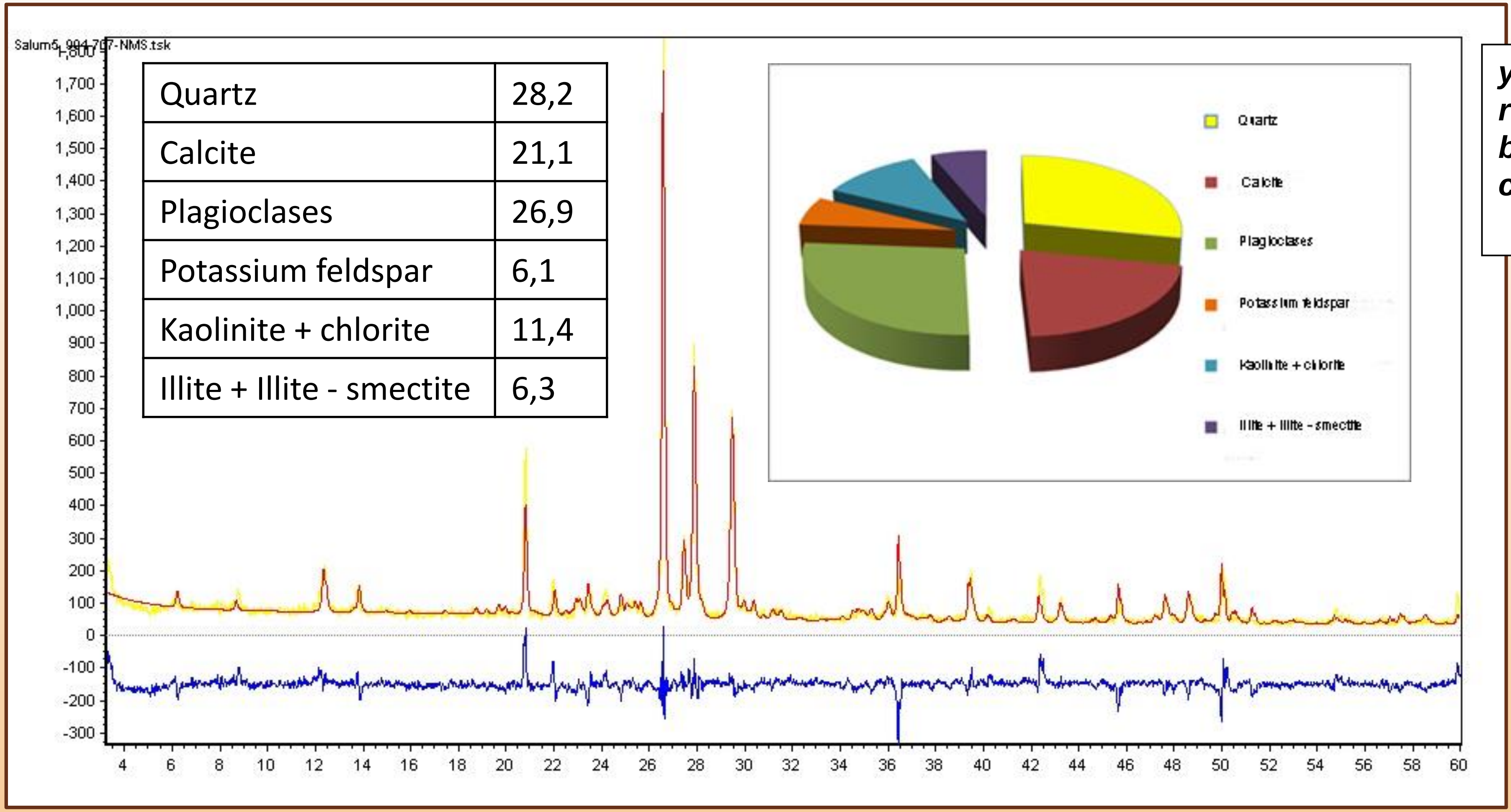
Determination of mineral composition of rock samples by method of X-ray diffraction using the Rietveld method



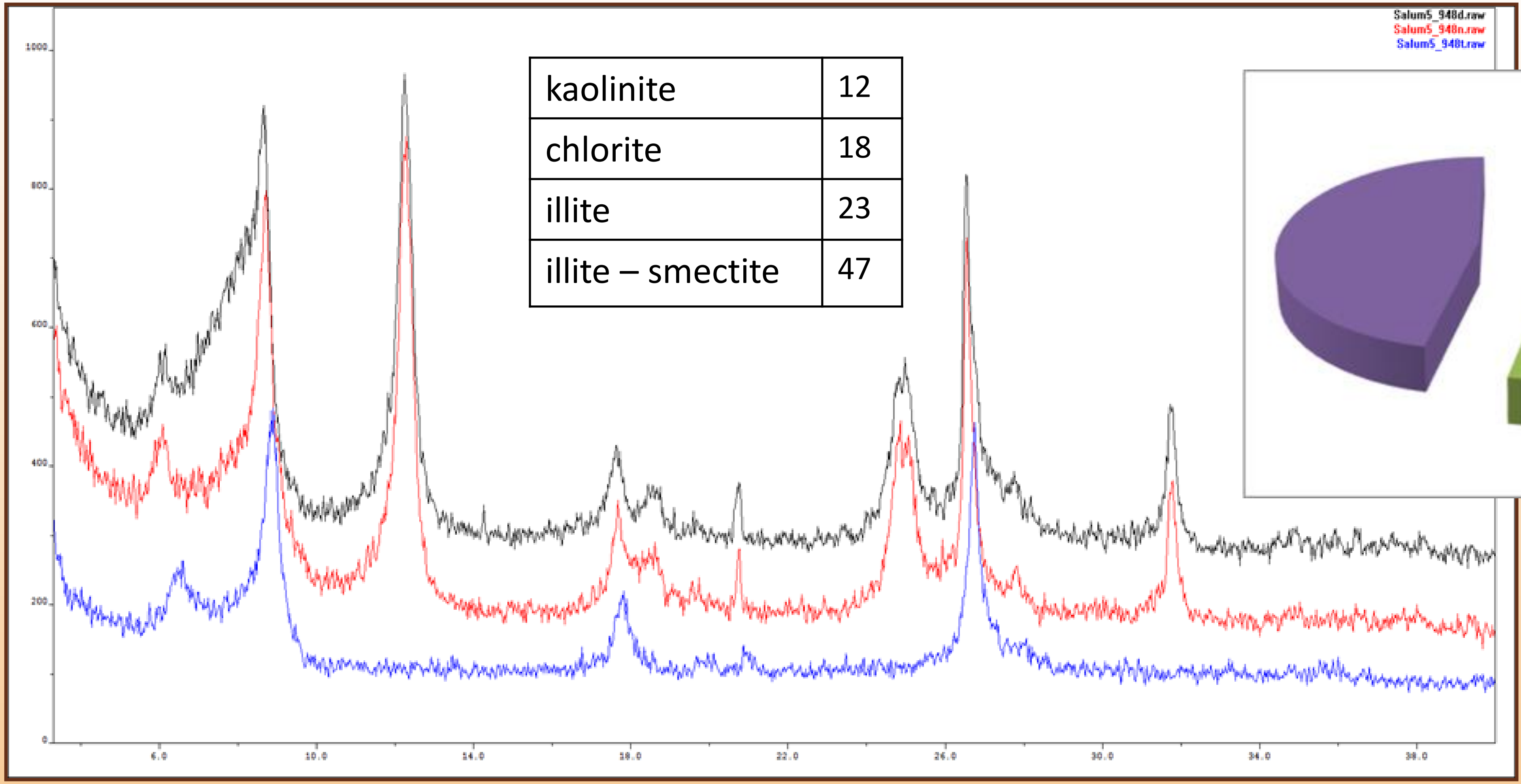
Quartz	28,2
Calcite	21,1
Plagioclases	26,9
Potassium feldspar	6,1
Kaolinite + chlorite	11,4
Illite + Illite - smectite	6,3

yellow line – experiment,
red line – calculation (theory),
blue line – the difference of the calculation and the experiment

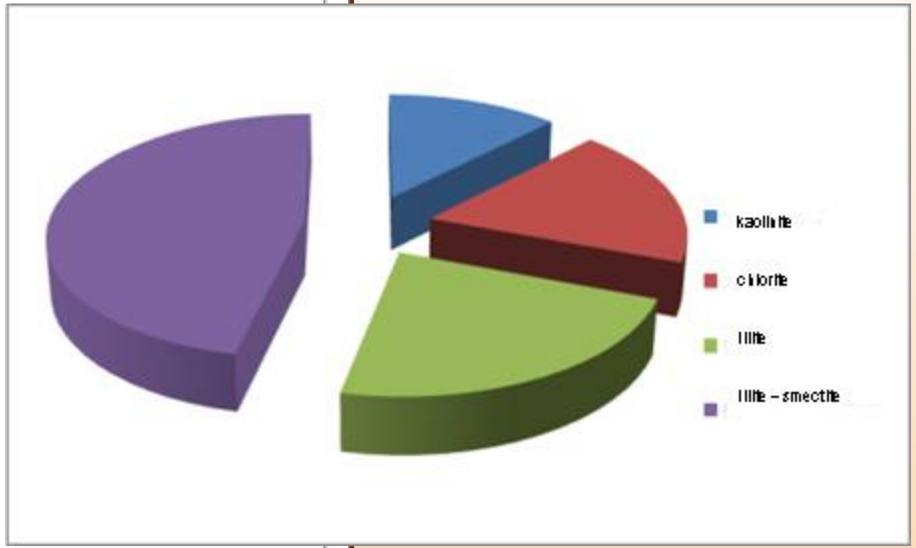
Example of X-ray diffraction pattern for disorienting sample

Studying material and chemical core composition

Determination of mineral composition of rock samples by X-ray diffraction (XRD) method



kaolinite	12
chlorite	18
illite	23
illite – smectite	47



Air-dry sample

Saturated sample

Heat treated sample

Superimposed X-ray diffraction pattern of the oriented samples

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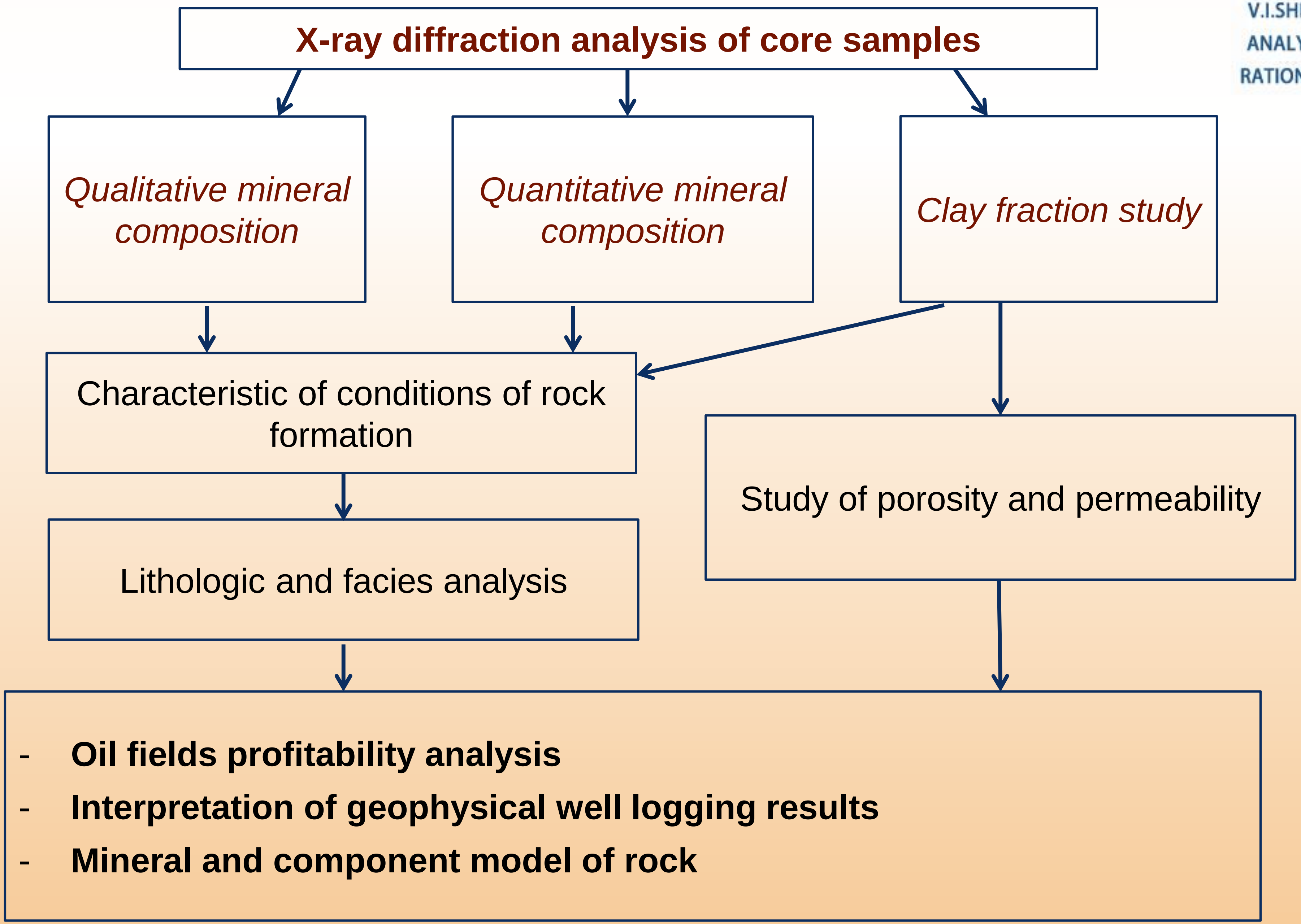

Studying material and chemical core composition

Thermal analysis

NETZSCH STA 449 F3 Jupiter® Synchronous Thermal Analyzer Mass Spectrometer Coupling QMS 403 Aeolos Quadro®

Function:
It allows carrying out measurements of weight and thermal effect changes, at temperatures between -150°C and 2400°C.

Function:
it is for the routine analysis of gases and, in particular, volatile decomposition products of thermal analysis

NETZSCH STA 449 F3 Synchronous Thermal Analyzer Jupiter® (NETZSCH Gerätebau GmbH, Germany)

QMS 403 Aeolos Quadro® Mass Spectrometer (NETZSCH Gerätebau GmbH, Germany) **coupling to the Synchronous Thermal Analysis**

Studying material and chemical core composition

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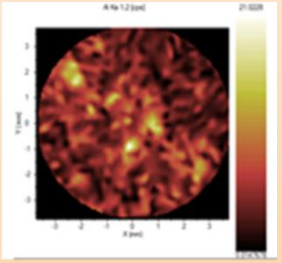
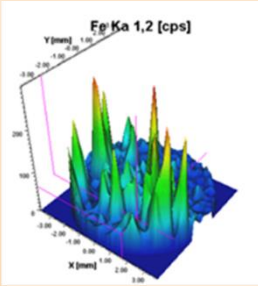
X-ray fluorescence (RPA) analysis of rocks

Purpose:
- Precision determination of element composition (from F to uranium U) of core, oil, produced water samples
- Elemental mapping of the surface of samples with the construction of 3D and 2D images:





ARL PERFORM'X 4200 X-ray Fluorescence Spectrometer
(Thermo Fisher Scientific, Switzerland)

Studying material and chemical core composition

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X-ray fluorescence analysis of rocks

Sample preparation equipment

Katanax X300 Fusion Furnace (Katanax ® inc., Canada)

Purpose:
Automated preparation of glass discs by fusing sample with flux for X-ray fluorescence analysis

Vaneox Automated Press (FLUXANA, Germany)

Purpose:
Automated pressing of powder samples into tablets for X-ray fluorescence analysis

Studying material and chemical core composition

Determination of microelement composition of rocks by atomic emission spectrometry with inductively coupled plasma

Microelement analysis of rocks is carried out on an atomic emission spectrometer with inductively coupled plasma iCAP 7000 DUO made by Thermo. The equipment package includes HotBlock Sample Decomposition System, Water Treatment System and iCAP 7000 DUO Acid Purification System

iCAP 7000 DUO

HotBlock Acid Decomposition System of Samples

Advantage: uniform heating in height during dissolution

High Water Treatment System

DST-1000Acid Purification System

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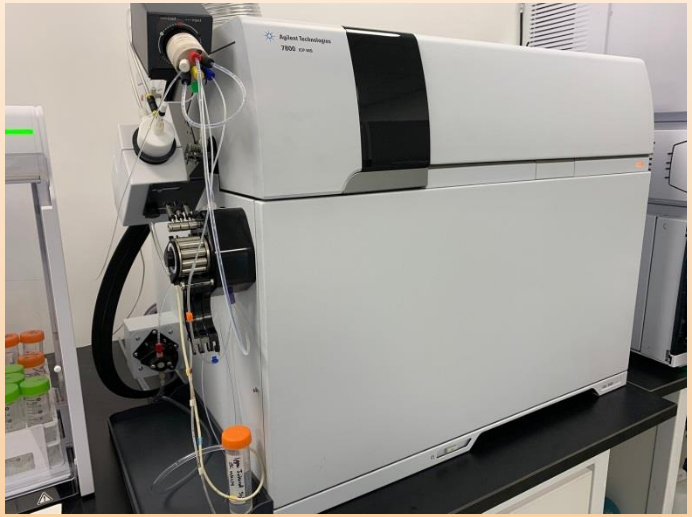
Determination of ultra-microelement composition of rocks by mass spectrometry with inductively coupled plasma

Ultra-microelement analysis is carried out using an inductively coupled plasma mass spectrometer

7800 ICP-MS made by Agilent with Multiwave PRO Microwave Sample Preparation System and ANTON PAAR Multiwave GO



7800 icp-ms, Agilent

Multiwawe PRO, Anton Paar

Multiwawe GO, Anton Paar

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Determination of element composition of core samples

Silicate analysis

Determination of impurity elements (micro- and ultra-microelements)

Elemental mapping with visualization of distribution of elements on inhomogeneous surface of rock sample

- Determination of rock composition change patterns
- Petrochemical characteristics of well logs

- **Geochemical mapping**
- **Identification of geological model of oil and gas reservoirs (formation, field, basin)**