

## AN AUTOMATIC DEVICE FOR IMBIBOMETRIC INVESTIGATION OF CLAYS

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Received 20. 2. 1978

*The imbibograph consists of (1) a sorption part, (2) an analytical balance adapted for an electrical weighing of liquid absorbed in the course of time, and (3) a recording system. The sorption part consists of two small glass vessels containing the liquid used (most often distilled water), connected with flexible tubing. In the first vessel, fixed on a holder outside the analytical balance, the liquid reaches up to a fritted glass which is overlain by a dense filter carrying a pressed sample. The second vessel, resting on the left pan of the balance, contains a sufficient reserve of feeding liquid. The imbibometric curve expresses the ratio sorption weight/time. The 200 mm range of the recorder scale may be adjusted for the required sensitivity between 20 mg and 5 grams, which permits the different imbibition rates and capacities for various substances to be followed. Each batch of clay materials (or any insoluble powder) is most often 0.500 g, the temperature of the liquid being  $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .*

*Examples of imbibition curves and a survey of imbibometric data of various clay minerals and clays from well known localities are presented.*

### INTRODUCTION

Imbibometry is a method for following the soaking up of a suitable liquid into an aggregate consisting of clay minerals and/or various powdered materials [KONTA, 3, 4]. What is being examined and evaluated is either the behaviour of a water drop and an ethylene glycol drop on evenly ground section of dry clay or the imbibition of a liquid into clay or other material in a sorption apparatus. FREUNDLICH, SCHMIDT and LINDAU [2] and FREUNDLICH, ENSLIN and LINDAU [1] used a sorption apparatus in which the sorption of water by powdered material was examined; according to their method the material studied is piled into the shape of a small cone on a porous even surface. KONTA [5] used dry clay cut into a solid prism whose base, of area  $100\text{ mm}^2$ , was flush with the bedding; he showed that for imbibometry it is most suitable to press powdered material — under constant conditions — into the shape of a small cylinder whose circular imbibition base is  $100\text{ mm}^2$ . The introduction of pressed cylinders instead of cones of a loose material, whose non-constant shape cannot be precisely defined, led to an improvement in the reproducibility of results. In addition, the application of Enslin's sorption apparatus required the attention of one worker observing and recording the decrease of the liquid in a pipette at certain time intervals. The authors of this paper endeavoured to design and construct an apparatus which would automatically and continuously record the soaking of a liquid with reliable precision and reproducibility.

An evaluation of the behaviour of a drop of liquid on a ground section of clay, or of the imbibometric curves of diverse powdered and pressed materials is useful in investigating some physical properties such as specific surface area,

swelling intensity, porosity, orientation of anisometric particles, influence of sorbed ions, relative geological age etc. [KONTA, 5, 6; KONTA and BOŠEVSKA, 8]. In many cases, imbibometry may be applied for a rapid identification of clay minerals or admixtures occurring in clays and soils. Imbibometry has also been a decisive method aiding the solution of problems in industrial production [KONTA, 6, 7].

## AN ELECTRONIC IMBIBOGRAPH BASED ON THE PRINCIPLE OF AN ANALYTICAL BALANCE

### The basic components of the apparatus

A basic part of the apparatus is an analytical balance adapted for electrically recording the weight of liquid sorbed in relation to time. The sorption part of the apparatus is composed of a small glass vessel *A* (fig. 1) fixed on a holder outside the analytical balance, with a horizontal fritted glass (*F*) to which the liquid reaches. The fritted glass is overlain by a wet dense filter on which is laid a pressed tablet (*C*) of the material studied. A thin flexible tube (*P*) connects the liquid in the sorption vessel with a reservoir of liquid in another glass-vessel (*B*) resting on the left pan of the balance. The circular reservoir vessel has a diameter of 50 mm so as to render the error due to falling liquid level as small as possible. The thin flexible tube of silicon rubber does not move during measurement and does not influence the result of measurement. The necessary height difference between the upper surface of the fritted glass and the level of the liquid in the vessel *B* is shown in fig. 1.

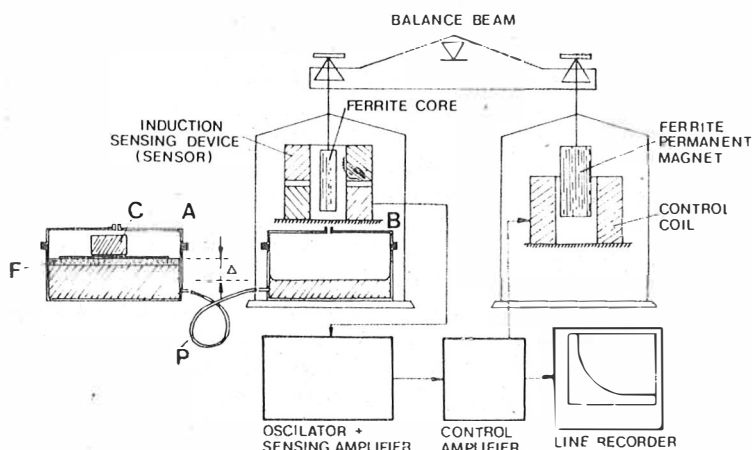


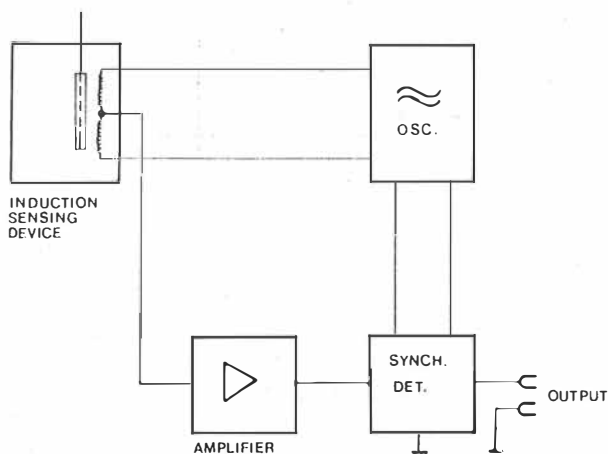
Fig. 1. Scheme of the imbibograph.

### Recording system

This system is based on the principle of balance beam deviation compensation; the beam remains practically stationary during the different loading of the balance plates. This secures a stable position of the liquid level in the

vessel *B* in relation to the vessel *A*, as well as a more linear dependence between the output voltage and the force acting on the stirrup and the influence of the thin tube *P* connecting both vessels is substantially decreased. The decreasing weight of the vessel *B* during the liquid's imbibition by the sample examined is continuously measured electrically and recorded by a line recorder. In this procedure a MEOPTA analytical balance was used, supplemented by a sensor, which followed the position of the balance beam, and by a compensation device.

The balance beam position sensor is located in the left damper of the analytical balance and consists of two parts (fig. 1): (1) a coil fixed on the firm lower part, (2) a ferrite core attached to the left stirrup of the balance. An oscillator and auxiliary electronic circuits (fig. 2) are located outside the balance box.



*Fig. 2. Schematic diagram of the sensing device.*

The resulting direct-current signal is fed into the control amplifier, whose set-up is very simple. It consists mainly of an integrated operational amplifier of the  $\mu A$  709 type with a power stage and damping circuit. A simplified diagram of the control amplifier is presented in fig. 3. Under the right-hand stirrup of the balance is attached a control coil into which a permanent ferrite magnet is inserted, extending to about half the coil's length. This secures a linear proportionality of the current flow in the coil to the load of the balance beam. When the load of the beam changes, the ferrite core's position changes slightly and the sensor delivers a signal to the control amplifier. The change of current in the coil causes a change in the strength of the magnetic field of the control coil, which acts upon the permanent magnet so as to induce the balance beam to return to its original position. In order to avoid any oscillation of the beam due to its delayed action, there must be a suitable damping device. The choice went to electric damping, produced by connecting a suitable circuit of negative feedback to the control amplifier (resistors  $R_2$ ,  $R_3$  and  $R_5$  and a condenser  $C_1$  — fig. 3).

The magnitude of the current through the control coil is taken as the voltage across the serial resistor  $R_1$  (fig. 3). This voltage is measured and registered by

a ZEISS line recorder. The 200 mm range of the recorder scale may be varied by resistors  $R_1$  changes at amounts from 20 mg to 5 g of the liquid sorbed; this is important in investigating substances of very different imbibometric capacities.

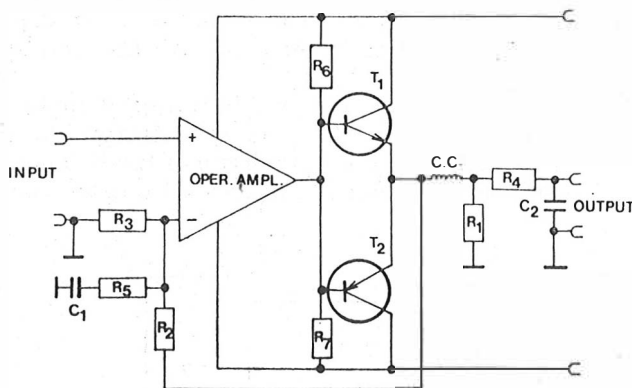


Fig. 3. Schematic diagram of the control amplifier.

### Measuring procedure

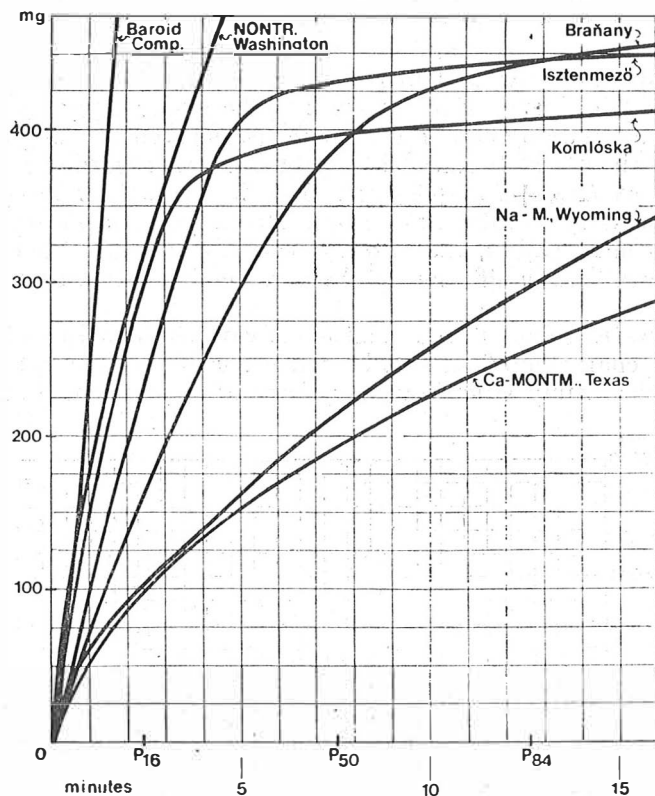
Before commencing imbibometric recording it is necessary to check whether the temperature of the liquid used is  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ . This secures a nearly constant viscosity and surface tension of the liquid. First, the system of balance and recorder is switched on and the zero time is recorded. The pressed cylinder is laid on a filter paper resting on the fritted glass of the sorption vessel. The reservoir vessel  $B$  contains an amount of liquid many times exceeding the amount of liquid sorbed. If before the end of the analysis the sample has soaked up an amount of the liquid whose weight exceeds the chosen range of the recorder scale indicated in grams (from 0.02 g through 0.05 g, 0.1 g, 0.2 g, 0.5 g, 1 g, 2 g to 5 g), a certain accurately measured amount of the liquid of temperature  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$  and weighed with accuracy of 0.001 g may be added to the reservoir vessel  $B$ , so that the recording stylus can return to the registration paper and record the further imbibition of the liquid as desired.

In most samples and for common purposes the imbibition is usually followed for 15 minutes, while at other times imbibition may be prolonged to 8 hours and in some cases to several days. When the recording is intended to be long-term it is possible either to slow the course of the paper or after a normal recording during the first 15 minutes of soaking the apparatus may be switched off and then switched on a suitable predetermined intervals.

### Pressing

About 1.5 g of each representative sample is powdered in the dry state in an agate or porcelain crucible, laid on a watch-glass and put in a desiccator above a layer of granulated Ginovci bentonite having a high montmorillonite content and capability of intensive sorption. After 24 hours of maturing in the desiccator, a batch of exactly 0.500 g of the powdered sample is pressed in

a cylindrical mould at a pressure of 3 MPa (30 kp/cm<sup>2</sup>) for a standard time of 5 minutes or under other constant conditions. The pressing operation is arranged so as to produce uniform bulk density throughout the whole sample. The base of the pressed cylinder is 100 mm<sup>2</sup>.



*Fig. 4. Imbibometric curves of several smectite materials with indication of chosen percentiles.*

#### IMBIBOMETRIC CURVES OF CLAY MATERIALS AND VALUES CHARACTERIZING THESE CURVES

The imbibometric behaviour may be investigated on any kind of powdered material which is insoluble in the liquid used. Clay minerals in unconsolidated aggregates are ideal for the purpose of imbibometry, even when minute grains and particles of non-clay minerals are present. The size and shape of the crystals in such aggregates, the crystallochemical composition, liability of some minerals to swelling, the nature of the ions sorbed, the character of flocculation and coagulation or aggregation may influence significantly the shape of imbibometric curves if the investigation is carried out under the other constant working conditions. When the clay material does not contain swelling

minerals or when they are accessory only, then during the initial 15 minutes of sorption, the course of an imbibometric curve is the result of penetration of water (or other suitable liquid) into pores in dependence on the size and shape of the crystals and their arrangement in the aggregate pressed under constant conditions [KONTA, 7]. For instance, KONTA [7] has shown that imbibometric curves may be correlated with dry strength and with the apparent viscosity of washed kaolins. In the Karlovy Vary washed kaolins the dry strength values increase with decreasing imbibition, while the apparent viscosity values of thickened suspensions rise with increasing imbibition, as shown by the imbibometric curves. The degree of imbibition may be most simply expressed as the average of three suitably chosen time percentiles given in milligrams of water soaked, when the initial 15 minutes of imbibition are regarded as 100 per cent of time, i.e. as  $(P_{16} + P_{50} + P_{84}) : 3$ . But in evaluating an imbibometric curve it is necessary to take into account not only the mean measure but all the three percentile values, and also the resulting value for the chosen 100 % imbibition time. The significance of these values is evident from figs 4 and 5 and table I.

Fig. 4 illustrates a short-term imbibition of various clays with a high smectite content. The character of an imbibometric curve depends not only on the crystallochemical composition, size and shape of crystals but, in montmorillo-

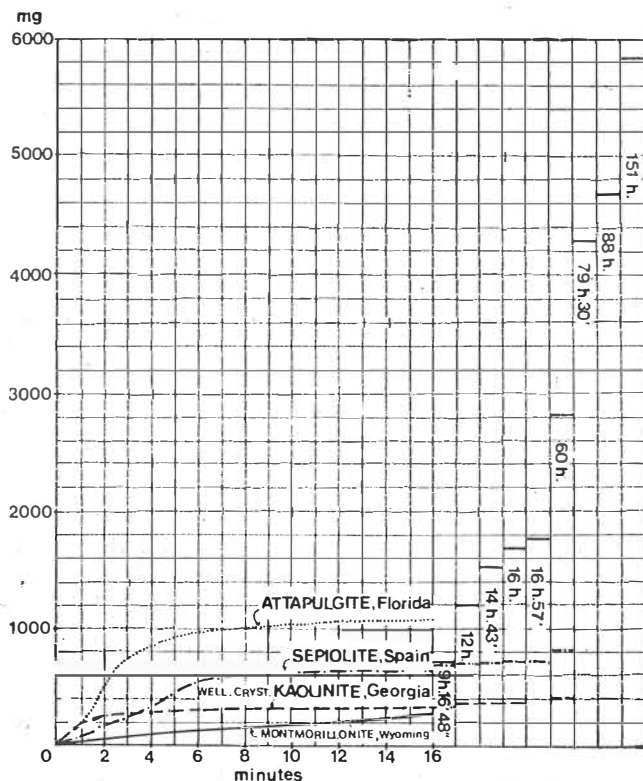
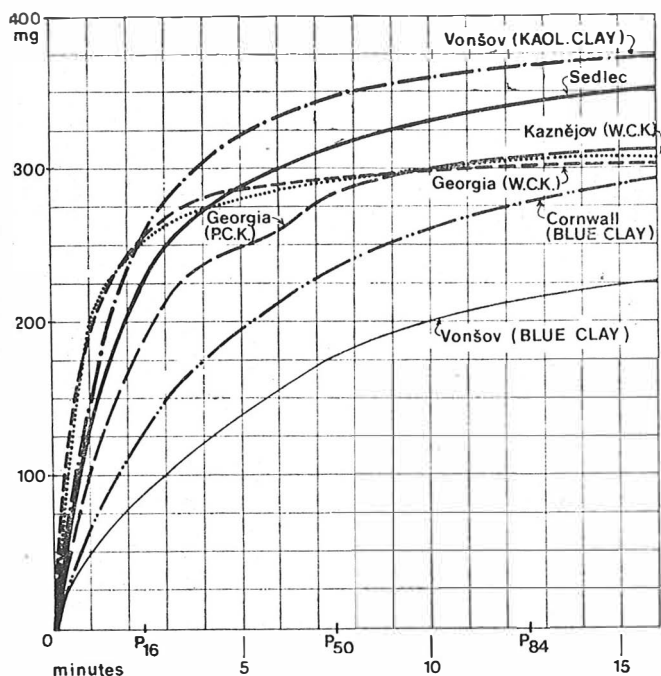


Fig. 5. Imbibometric curves of short period sorption and marks for long period sorption of different clay materials.



*Fig. 6. Imbibometric curves of clay materials rich in kaolinite with indication of chosen percentiles.*

nites, especially on the nature of sorbed cations which are hydrated to different degrees during penetration of water. For this reason, the short-term (15 minute soaking) imbibometric curves of montmorillonite materials are so different, irrespective of the influence of some further mineral admixtures.

Fig. 5 shows the shapes of imbibometric curves at an eight times wider range of mg values of soaked water; the time range on the horizontal axis remains the same.

In clays where kaolinite prevails and in washed kaolins in which kaolinite is crystallized to different degrees and where the distribution of pores also is non-uniform, the imbibometric curves display a more uniform course (fig. 6). In most cases the differences between the curves may be interpreted by the fact that the finer the material, the slower is its soaking up of water during the initial few minutes of sorption. Kaolins and clays containing coarser kaolinite crystals, such as those from Georgia or Kaznějov, or non readily disintegrable aggregates of a clay mass or those with a somewhat coarser clastic admixture display a more rapid imbibition rate, especially at the beginning of sorption. Blue bonding clays containing kaolinite pM and a considerable proportion of mixed illite-montmorillonite clay material are characterized by slow initial sorption.

The presentation of a great number of imbibometric curves expressing sorption after a short-term and an optimally chosen long-term soaking would greatly extend this paper, so the curves were evaluated and their characteristic values are given in table I.

Table I.

Values obtained from imbibometric curves of various clay materials after 15 minutes and 8 hours (and long-term) of imbibition using distilled water at  $20 \pm 2^\circ \text{C}$  respectively. The weight of each sample being 0.500 g

Sample	15 minutes imbibition				Water absorbed in 15 minutes, 100 % of the time (= $P_{100}$ )	8 hours imbibition				Water absorbed in 8 hours, 100 % of the time (= $P_{100}$ )	56 hours
	$P_{16}$	$P_{50}$	$P_{84}$	$\frac{P_{16} + P_{50} + P_{84}}{3}$		$P_{16}$	$P_{50}$	$P_{84}$	$\frac{P_{16} + P_{50} + P_{84}}{3}$		
in milligrams											
Na-montmorillonite, Wyoming	98	214	295	202	343						
Bentonite, Komlóská, Hungary	299	397	409	368	413						
Montmorillonite clay, Istenmező, Hungary	224	434	447	368	450						
Bentonite, Braňany, Bohemia	280	417	442	380	451						
Nontonite, Washington	280	600	680	520	690						
Na-bentonite, Ginovec, Yugoslavia	110	190	240	180	270	720	1 240	1 600	1 187	1 750	3 310
Synthetic mica-montmorillonite, Baroid Co.	1 250	1 640	1 710	1 533	1 730						
Fuller's earth (blue), Woburn	255	680	880	605	910	1 030	1 100	1 155	1 095	1 170	
Ca-montmorillonite, Plum, Texas	148	245	299	231	322	418	453	476	449	488	
Attapulgit, Florida	240	455	580	425	660	1 290	1 610	1 690	1 530	1 720	28 hrs 1930
Sepiolite, Spain	190	580	630	467	640	690	690	710	697	720	28 hrs 810



Illite clay, Füzerradvány, Hungary	200	348	403	317	424	504	531	553	529	564	839
Illite (very well cryst.), Marbleheat, Wisconsin	101	184	238	174	270	499	687	805	664	852	
London clay (illite)	148	286	356	263	381	450	474	484	469	488	
Light blue ball clay, Bovey Basin, Newton Abbot	152	234	240	209	243	256	270	288	271	297	
Dark blue ball clay, Bovey Basin, Newton Abbot	191	275	298	255	304	323	338	355	339	365	
Black ball clay, Bovey Basin, Newton Abbot	173	286	322	260	330	350	362	371	361	377	
Blue clay, Wareham, Cornwall	125	234	278	212	289						
Blue clay, Vonšov, Bohemia	92	182	218	164	229	294	320	339	318	348	
Washed kaolin (well cryst.), Georgia	251	293	302	282	304						
Washed kaolin (poorly cryst.), Georgia	178	250	272	233	277	293	310	328	310	336	549
Washed kaolin, Sedlec Ia, Bohemia	200	258	273	244	277	314	337	364	338	371	(28 hrs) 480
Washed kaolin, Zarow, Poland	184	264	292	247	304	344	354	360	353	362	
Washed kaolin, Kaznějov, Bohemia	252	294	306	312	284						

## CONCLUSION

The imbibograph, an apparatus for automatically recording the amount of water or other liquid soaked up in relation to time by a suitably prepared clay material or any other kind of insoluble powdered material yields reliable reproducible results. Clay minerals, clay rocks and other materials closely related to them represent systems displaying characteristic physical and physico-chemical properties which are primarily due to the crystal structure of the mass particles present, their surface energy, the various ions bound by electric forces on their surface and in interlayer spaces, as well as to the size and shape of crystals and their state of aggregation. During imbibition of water into a pressed aggregate, free pores between crystals are filled, ions on the surface as well as in interlayer spaces in some clay minerals are hydrated, and sorption of individual water molecules takes place; all this may lead to swelling and to enormously intensive soaking of the liquid phase. An important prerequisite for optimal interpretation of imbibometric curves is knowledge of the material composition of the samples studied, obtained for instance by X-ray and microscopic studies, and the theory of their physical and chemical properties.

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AUTOMATICKÝ PŘÍSTROJ PRO IMBIBOMETRICKÉ  
STUDIUM JÍLŮ

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Imbibograf se skládá ze (1) sorpční části, (2) analytických vah upravených pro elektrické vážení kapaliny vsáklé v časové závislosti, (3) zapisovacího systému. Sorpční část se skládá ze dvou malých skleněných nádob, obsahujících kapalinu (nejčastěji se užívá destilované vody), spojených pružnou hadičkou. V první nádobce, upevněné na držáku mimo prostor analytických vah, sahá kapalina až k fritě, pokryté hustým filtrem (modrá páska), na nějž se pokládá vylisovaný vzorek jílové hmoty. Druhá nádobka, spočívající na levé misce vah, obsahuje dostatečnou zásobu užitě kapaliny. Imbibometrická křivka vyjadřuje vztah váhy sorbované kapaliny k době vsakování. Šířka zápisu je 200 mm, avšak požadovanou citlivost lze upravit v rozsahu od 20 mg až do 5 gramů, což umožňuje sledovat různé imbibiční rychlosti a kapacity v závislosti na povaze studovaných látek. Navážka jílového materiálu (nebo jakéhokoliv nerozpustného prášku) je nejčastěji 0,500 g, teplota kapaliny během vsakování  $20 \pm 2^\circ\text{C}$ . Jsou uvedeny příklady imbibometrických křivek a přehled imbibometrických dat různých jílových minerálů a jílů ze známých zahraničních i československých lokalit.

- Obr. 1. Schéma imbibografu.  
 Obr. 2. Schematický diagram snímacího zařízení.  
 Obr. 3. Schématický diagram regulačního zesilovače.  
 Obr. 4. Imbibometrické křivky několika smektitových minerálů s uvedením zvolených percentil.  
 Obr. 5. Imbibometrické křivky krátkodobé sorpce a označení dlouhodobé sorpce různých jílových minerálů.  
 Obr. 6. Imbibometrické křivky jílových minerálů bohatých kaolinitem s uvedením zvolených percentil.

## АВТОМАТИЧЕСКИЙ ПРИБОР ДЛЯ ИМБИБОМЕТРИЧЕСКОГО ИССЛЕДОВАНИЯ ГЛИН

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Имбибограф состоит из: поглотительной части (1), аналитических весов (2), приспособленных для электрического взвешивания жидкости, впитывающейся в зависимости от времени, регистрирующей системы (3). Поглотительная часть состоит из двух небольших стеклянных сосудов, содержащих жидкость (чаще всего дистиллированную воду) в соединенных гибким шлангом. В первом сосуде, находящемся вне аналитических весов и укрепленном к держателю, уровень жидкости достигает фритты, покрытой плотным фильтром (синяя полоса), на который кладут прессованный образец массы глины. Вторым сосуд, находящийся на левой чашке весов, содержит достаточное количество примененной жидкости. Имбибометрическая кривая выражает отношение веса поглощаемой жидкости к времени впитывания. Ширина записи 200 мм, однако требуемую чувствительность можно получить в пределах от 20 мг до 5 г и таким образом дана возможность исследования разной скорости имбибиции и мощности в зависимости от характера исследуемых веществ. Доза глинистого материала (или любого нерастворимого порошка) чаще всего составляет 0,500 г, температура жидкости  $20 \pm 2^\circ\text{C}$ . Далее приводятся примеры имбибометрических кривых и в обзоре имбибометрические данные разных глинистых минералов и глины из чехословацких или известных зарубежных местонахождений.

- Рис. 1. Схема имбибографа.  
 Рис. 2. Схематическая диаграмма регистрирующей установки.  
 Рис. 3. Схематическая диаграмма регуляционного усилителя.  
 Рис. 4. Имбибометрические кривые нескольких смектитных минералов с подобранными перцентилами.  
 Рис. 5. Имбибометрические кривые кратковременной сорбции и продолжительной сорбции разных глинистых минералов.  
 Рис. 6. Имбибометрические кривые глинистых минералов, богатых каолинитом с подобранными перцентилами.