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ORGANIC MATTER MATURITY IN PERI-KLIPPEN FLYSCH OF THE INNER CARPATHIAN MTS. (EAST SLOVAKIA)

(Figs. 5, Pl. 1, Tab. 1)

A b s t r a c t s : On 30 samples of dark pelitic rocks from Šambron borehole the degree of thermal alteration of sedimentary organic matter was determined by vitrinite reflectance Rm_0 measurements. The method is briefly characterized including some own experience concerning microscopical measurements.

Based on statistically evaluated vitrinite mean reflectance Rm_0 the coalification stage was determined as equivalent to „coking coal" (Czechoslovak classification) which corresponds to the condensate and wet gas maturation level.

Higher reflectance values (which do not show considerable increase with depth) indicate higher diagenesis temperatures caused probably by deeper burial of the sedimentary layers before folding. The abnormal character of Rm_0/depth curve may be considered to be a result of the very complicated tectonic structure. A limited correlation between deviating Rm_0 values and more important faults may be inferred.

Р е з ю м е: Степень термической перемены рассеянного органического вещества была исследована в 30 образцах темных пеллитических пород из скважины Шамброн. Характеристика метода дополнена собственным опытом авторов. На основе статистической оценки микроскопической средней отражательной способности витринита R_0 определен коксовый этап углефикации органического вещества, соответствующий этапу „созревания" конденсата и жирного газа.

В общем повышенные величины отражательной способности R_0 свидетельствуют о повышенных температурах катагенеза вследствие более глубокого погружения осадков перед орогеном. Исключительный характер кривой зависимости R_0 от глубины можно считать следствием осложненной тектоники (обратной последовательности слоев в нижней части разреза скважины). В определенной мере можно наблюдать связь между отклоненными величинами R_0 и положением более значительных разломов.

Introduction

Dispersed organic matter (MOD)[1] in sedimentary rocks is a source of some important information about their facial origin and burial development. MOD is, therefore, being studied more carefully in recent years.

This paper reports the results of a microscopical study of MOD in reflected light on samples from Šambron borehole, East Slovakia. The results obtained should contribute to geological exploration of the region considering the degree of diagenesis (catagenesis) and thermal alteration of the rocks and of the included organic matter, occurrence of oil and gas, and to certain extent also the complicated tectonic structure.

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[1] MOD — abbreviation from French „matière organique dispersée".

Geological setting

The Šambron borehole is situated in the Peri-klippen area of the Inner Carpathian Flysch in Hromoš—Šambron anticline. According to the latest geomorphological division of the West Carpathian Mts. (M a z ú r — L u k n i š. 1980) it lies in Hromovec, subunit of the Spišsko-šarišské medzihorie Mts. The area is being systematically studied as a part of the State research programs: "West Carpathian deep structures exploration in bitumens occurrence" (1971—1980) and "Geological exploration of prospective regions in oil and gas deposits occurrence" (1981—1986).

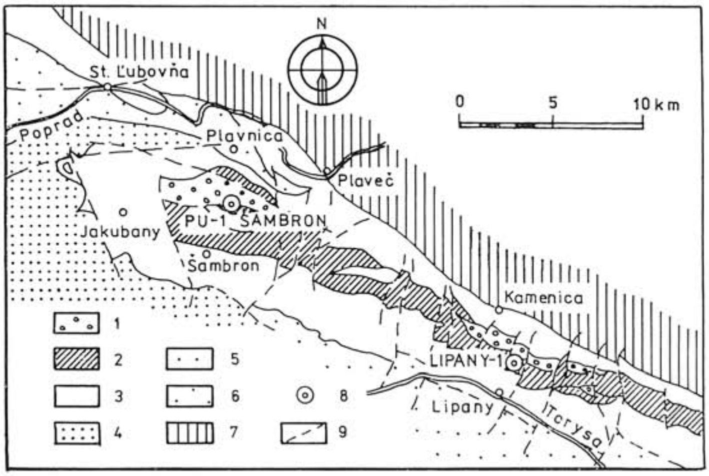


Fig. 1. Tectonic map of Hromoš—Šambron anticlinal zone with location of deep boreholes (adapted after R. M a r s c h a l k o, 1975).
Explanations: 1 — proximal flysch facies with conglomerates and sandstones, 2 — Šambron beds, 3 — claystone lithofacies, 4 — coarse flysch with graded sandstones, 5 — claystone-sandstone flysch, 6 — flysch with predominant sandstones, 7 — Klippen Belt and Magura Flysch, 8 — boreholes, 9 — faults.

The geology of the region has been characterized by C h m e l í k (in B u d a y et al., 1967), M a r s c h a l k o (1975), C h m e l í k — L e š k o (1967), N e m č o k et al. (1977), N e m č o k — R u d i n e c (1979), and others.

The location of Šambron borehole and the geological setting of the surrounding area is shown in Fig. 1. The exposed sequences belong to the epimiogeosynclinal development of Inner Carpathians of Late Eocene. From among them the Šambron beds, claystone and claystone-sandstone lithofacies are estimated as possible source rocks. Potential reservoir properties are expected at the deeper basal conglomerate which in this area does not reach the surface (C h m e l í k — L e š k o, 1976).

The comprehensive exploration of the structural borehole Šambron has been presented by N e m č o k et al. (1977). The borehole section is represented by the lower part of Šambron beds with intraformational thin beds of conglomerates and slip conglomerates. The Šambron beds comprise dark claystones or siltstones alternating in flysch development with gray sandstones, both Late Lutetian to Early Priabonian. The simplified lithological log is shown in Fig. 5.

Method

Vitrinite reflectance measurements are being used increasingly among modern oil and natural gas prospecting methods. This coal petrological method has been applied to the study of dispersed organic matter (MOD) in clayey, sandy and calcareous rocks (Teichmüller, 1971; Ammosov — Gorshkov, 1971; Bostick, 1974; and others).

The bituminous coal maceral vitrinite (or brown coal huminite) is a microscopically distinguishable particle of organic matter characteristic by continuous change of its chemical and physical properties during coalification. As the essential factors of organic matter transformation are temperature and time, vitrinite is sometimes accepted as "thermometer" of the sedimentary rocks alterations during diagenesis and anchimetamorphism.

Vitrinite reflectance in oil R_0 is being widely used as a coal rank parameter (in Czechoslovakia according to Standard ČSN 44 1346, 1971).

Since coalification and petroleum genesis depend on the same diagenetic factors, each stage of both MOD and petroleum maturation can be matched with a particular rank stage of coaly particles in the rocks (Teichmüller, 1971) (Fig. 2).

Reflectance measurement using microscope with photometer is a non-destructive method which is not time-consuming and does not require complicated preparation of samples. The reliability of the results is, however, strongly influenced by correct selection of suitable microscopical particles of MOD for reflectance measurements. This must be based on good coal petrological knowledge and experience of the microscope operator. Problems arising from the correct constituent selection are analysed, e. g. by Müller (in Strnad — Müller, 1978 and 1980; in Dvořák et al., 1981).

In this work for reflectance measurements 30 core samples of dark claystones and siltstones from borehole Šambron were taken. The polished surface specimens ware prepared according to internationally accepted criteria. To compare the quality of the results a few specimens were pararelly prepared without vacuum mould-casting into a synthetic resin.

The measurements were carried out in the laboratory of Geological Survey in Brno using microscope-photometer MPV—2, Leitz Wetzlar under following condidions: non-polarized monochromatic light (546 nm), oil objective 50 x, refractive index of oil n = 1.514, glass prism standard of reflectance 1.24 % R_0.

In each specimen from 30 depths 50 values of mean reflectance Rm_0, measured on vitrinitic constituents (Pl. 1a, b), were taken, the arithmetic mean $\overline{Rm_0}$, standard deviation and 95 % confidence interval of the mean were calculated. Using t-test a statistically significant difference of two $\overline{Rm_0}$ was found. Results obtained are listed in table 1. The Rm_0/depth curve in the borehole section is shown in Fig. 5.

Table 1

Mean vitrinite reflectance in rocks of Šambron borehole Explanations: d — depth in m; Rm_o % — mean reflectance in oil (arithmetic mean of 50 measurements); 95 % c.i. — confidence interval of the mean

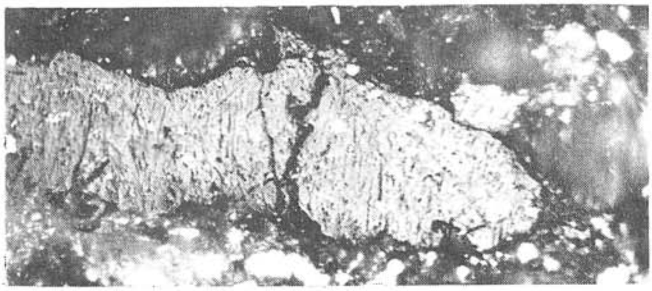
n	d [m]	Rm_o %	95 % c.i.	n	d [m]	Rm_o %	95 % c.i.
1	14	1.28	1.24—1.32	16	761	1.23	1.15—1.31
2	16	1.18	1.14—1.22	17	781	1.34	1.28—1.40
3	49	1.12	1.06—1.18	18	938	1.57	1.49—1.65
4	83	1.37	1.29—1.45	19	956	1.76	1.67—1.85
5	108	1.38	1.30—1.46	20	1041	1.22	1.14—1.30
6	162	1.25	1.17—1.33	21	1086	1.58	1.52—1.64
7	233	1.29	1.20—1.38	22	1150	1.42	1.35—1.49
8	280	1.11	1.03—1.19	23	1227	1.58	1.52—1.64
9	312	1.18	1.10—1.26	24	1288	1.57	1.50—1.64
10	350	1.35	1.28—1.42	25	1351	1.53	1.47—1.59
11	414	1.39	1.32—1.46	26	1450	1.41	1.35—1.47
12	537	1.49	1.43—1.55	27	1776	1.75	1.68—1.82
13	646	1.60	1.52—1.68	28	1985	1.57	1.51—1.63
14	677	1.60	1.53—1.67	29	1992	1.45	1.39—1.51
15	727	1.34	1.27—1.41	30	2001	1.55	1.47—1.63

Some methodical findings

The vitrinite reflectance determination in sediments is despite its evident advantages bound with certain specific problems. In our study we tried to work out a system how to diminish the inaccuracies and thus to contribute to the reliability and reproducibility of the method.

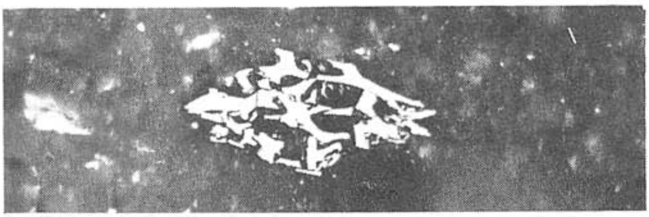
Comparing the results obtained from polished specimens prepared by use of vacuum casting into synthetic resin and those without the latter showed up to double scatter of Rm_o values because of the lesser specimen compactness which results in uneven surface with grinding and polishing imperfections.

The crucial problem is the correct identification and selection of MOD constituents suitable for reflectance measurements. It is the maceral group of vitrinite (Pl. 1 a, b left) which is to be distinguished from exinites (liptinites) and inertinites (Pl. 1 b right, c, d). Reflectograms of these essential groups based on a)



b)

c)



d)

Plate 1

Macerals of dispersed organic matter in Šambron beds claystones from Šambron borehole (Rm_o — mean reflectance in oil), 500 x

Fig. a) — vitrinite (gelificated organic matter) 1.12 % Rm_o
Fig. b) — left: vitrinite, 1.80 % Rm_o; right: inertinite — fusinite with visible cell walls of plant, 3.42 % Rm_o.
Fig. c) — inertinite — fusinite with „bogen“ or „star“ structure (fractured cell walls) 4.18 % Rm_o.
Fig. d) — inertite — sclerotinite (fungal remains) 3.09 % Rm_o.
(Photo Francŭ).

measurements of claystone specimen from depth of 727 m is in Fig. 3. It is the most difficult task to determine the boundary between the group of vitrinite and inertinite because of existence of intermediate constituents (e. g. semivitrinite, semifusinite). In Fig. 3 these are represented by reflectance range of about 1.9—2.7 % Rm_0.

During our study it prooved more effective as a first step to carry out an orientational identification of MOD macerals occurring in the studied rocks. The macerals should be distinguished first visually according to their microscopic features and the reflectances Rm_0 should be recorded for each maceral group separately. Using of obtained reflectograms may improve the operator's ability of further classification of individual MOD particles to be measured and finally of the representative Rm_0 range delimitation for arithmetic mean calculations.

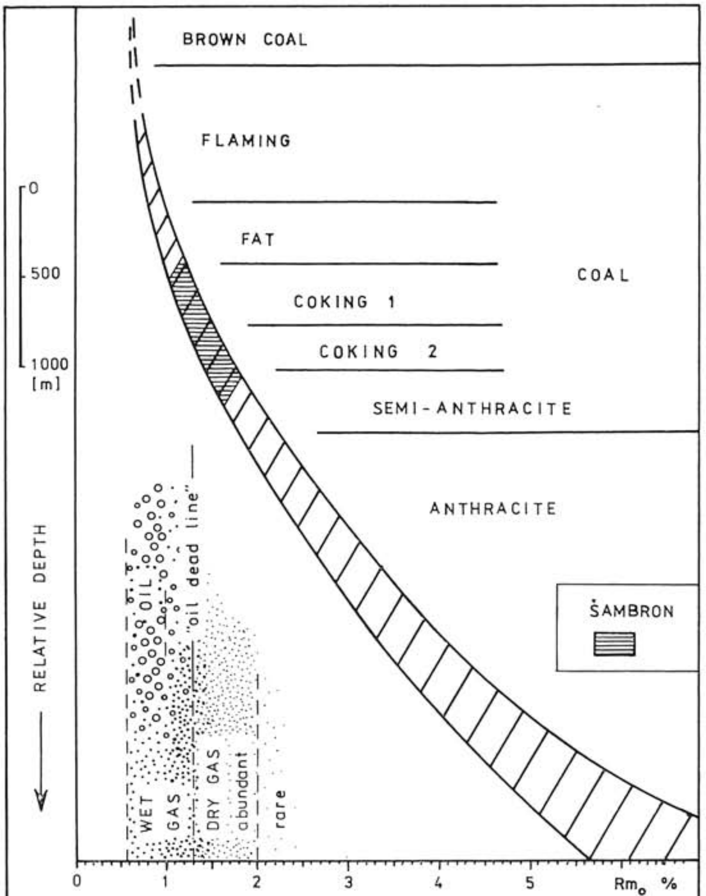


Fig. 2. Mean vitrinite reflectance (Rm_0) increase with relative depth and the respective zones of occurrence of oil and natural gas deposits (adapted after M. Teichmüller in E. Stach et al., 1975), and the corresponding coalification stages according to Czechoslovak classification (after G. Weiss, 1980 b). Hatched area represents Šambron borehole.

The described steps helped to solve the constituent selection in shales from Šambron borehole. Yet, it is necessary to admit than in case of other rocks with different MOD association using such steps will not lead to correct results automatically. In higher metamorphosed organic matter the reflectance anisotropy of vitrinite is observed and at the same time the difference between Rm_o of vitrinite and inertinite diminishes and the constituent selection becomes therefore more difficult (M ü l l e r in S t r n a d — M ü l l e r, 1980).

The insufficient number of MOD particles in specimens tempts often to measure MOD grains with defected surface. The influence of surface inhomogeneities in scanning area of the photometer on the obtained results was examined. It was found out that tiny sulphide inclusions, or on the contrary, narrow ruptures, scratches or holes of about 0.5 μm (i. e. at the limit of the operator's visual

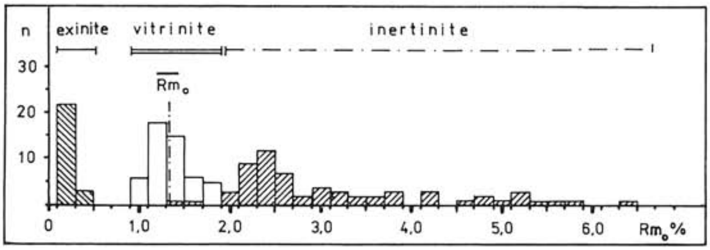


Fig. 3. Reflectograms of the three essential maceral groups of *MOD* in claystone specimen from Šambron borehole (depth 727 m).
Explanations: number of measurements: exinites — 25, vitrinite — 50, inertinite — 60. The dot-and-dash line marks the arithmetic mean of Rm_o for the vitrinite group.

resolution using objective 50 ×) may cause the alsolute deviation of the reflectance measured up to $\Delta Rm_o = \pm 0.40$ % from the value measured on the same particle but in homogenous part. For comparison, resolution of the used photometer is 0.01 % R_o. It is therefore obligatory to measure exclusively the homogenous surface area of vitrinite grains.

MOD maturation level

The mean value of vitrinite reflectance for the entire borehole is $\overline{Rm_o} = 1.42$ %. Rm_o values of individual samples lie between 1.11 % (depth d = 280 m) and 1.76 % (d = 956 m) (Fig. 2, 4 and 5). The statistically significant difference of two sample values is about $\Delta Rm_o = 0.10$ % (significance level $\alpha = 0.05$, mean standard deviation 0.25 %).

The reflectance range obtained is characteristic of medium to higher levels of organic metamorphosis. The coalification degree corresponds, according to Czechoslovak technological classification (ČSN 44 1346; W e i s s, 1980 b), to "coking coal" rank (Fig. 2) (according to ASTM medium to low volatile bituminous coal).

It is generally accepted that hydrocarbon deposits occur between certain depth boundaries determined by degree of thermal alteration of the rocks (V a s s o y e v i c h et al., 1969). Thus, "birth line" and "dead line" first for oil, then for condensate and wet gas and finaly for dry gas (methane) respectively are used. Current opinion suggests that the "dead line" for important economic petroleum occurrences lies at 1.3 % Rm_o, while most oil deposits are found where the mean vitrinite reflectance does not exceed 1.0 % Rm_o (Teichmüller in Stach et al., 1975).

For better illustration the MOD and/or petroleum maturation diagrams using vitrinite reflectance scales are shown in Fig. 2, 4 and 5 bottom.

The Rm_o range of Šambron borehole corresponds to more advanced thermal alteration of rocks and MOD maturation, namely, to formation of condensate and wet gas. The average $\overline{Rm_o} = 1.42$ % of the entire borehole lies behind the mentioned "oil dead line".

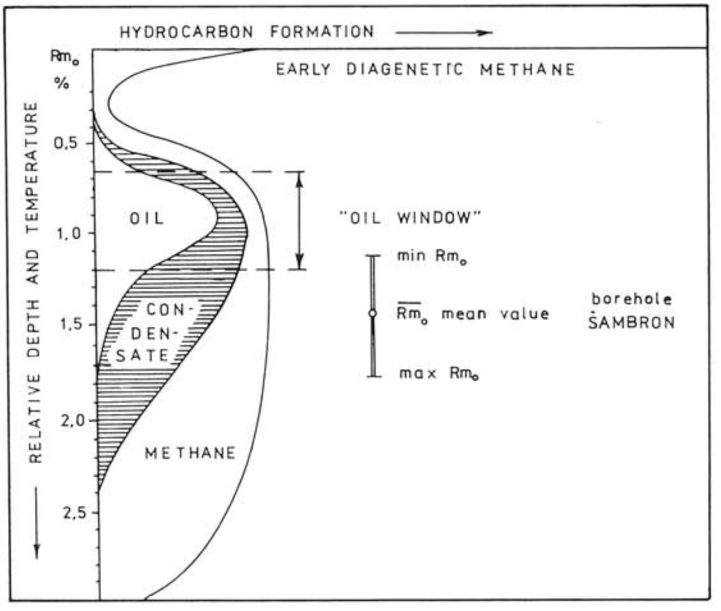


Fig. 4. Zones of occurrence of oil and natural gas deposits using mean vitrinite reflectance (Rm_0) scale (adapted after N. B. Vassoyevich, 1975). The Rm_0 range and arithmetic mean $\overline{Rm_0}$ representing the entire Šambron borehole see in the right side.

According to the MOD maturation level it is possible to admit condensate and wet gas occurrence in the rocks studied. Yet, the vitrinite reflectance determination does not give any quantitative data on hydrocarbon generation or deposits formation. For such estimations the chemical analyses are necessary.

The conclusions presented are in fair accordance with the drilling oil shows. In the Šambron borehole in fault zones at the depths of 536 to 545 m, partly also at 629 to 631 m, gas and resin showings were detected. The gas (methane) has been detected also in the pores of surrounding rocks (Nemčok et al., 1977). In the neighbouring Lipany 1 borehole, about 15 km to ESE (Fig. 1), gas with condensate has been detected.

Thermal history and tectonical development

Evaluating the reflectance profile (Fig. 5) we may point out the certain specific features. The vitrinite reflectance reaches higher values already at shallow depths. It means, the thermal alteration of MOD is of relatively advanced level at these depths, where the present temperature does not reach the initial boundary of coalification or bituminisation alterations. It is, therefore, possible to conclude that the sediments were lifted from rather deep levels and that the present degree of alteration had been reached overwhelmingly before the upheaval of the Šambron beds.

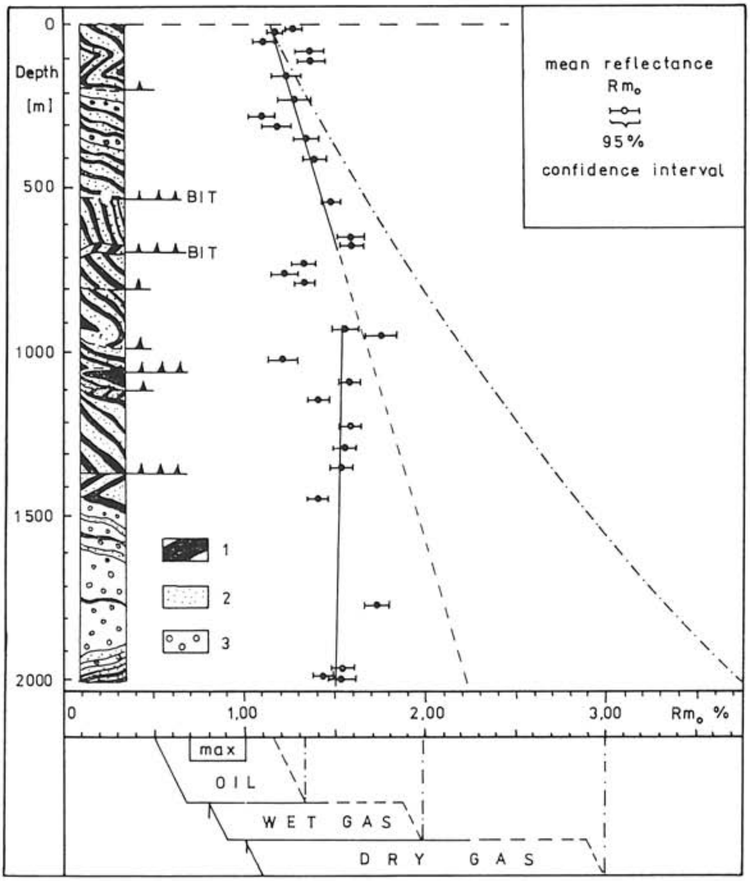


Fig. 5. Vitrinite mean reflectance Rm_0 plotted against depth in Šambron borehole. Left side — lithological log with more important faults and hydrocarbon shows (BIT) (adapted after J. Nemčok et al., 1977). Dot-and-dash curve is the regular Rm_0 increase with relative depth after M. Teichmüller (in E. Stach et al., 1975; see also Fig. 2). Bottom — essential stages of formation and thermal degradation of hydrocarbons with the respective „dead lines" using the same Rm_0 scale (adapted after W. G. Dow, 1977).

Explanations: 1 — claystone, siltstone; 2 — sandstone; 3 — conglomerate.

The reflectance/depth curve representing Šambron borehole does not coincide with the Rm_0 increase with relative depth in horizontal series after Teichmüller (in Stach et al., 1975) (Fig. 2 and 5 dot-and-dash). A comparison of both of them show a certain resemblance in the upper part of the section (0 to 700 m), while in the lower part the reflectance does not increase and the values vary about 1.50 % Rm_0. The difference in character of Rm_0/depth relationship in the upper and in the lower part becomes more obvious after plotting two regression lines for specimen sets from depths of 14 to 677 m and 938 to 2001 m.

The numerical difference in vitrinite reflectance between the deepest specimens (1.55 % Rm_0, d = 2001 m) and those from subsurface depths (1.18 % Rm_0, d = 16 m) is quite small: $\Delta Rm_0 = 0.37$ % (statistically significant difference of 2 samples is 0.10 % ΔRm_0, calculated with $\alpha = 0.05$). Teichmüller (in E. Stach et al., 1975) presents for relative depth increase of 2000 m a Rm_0 increase from 1.20 % to 3.75 %. A similar value is obtained by extrapolation of the same Rm_0/depth curve based on measurements in the Upper Silesian basin (Weiss, 1980 a).

The following explanation may be presented. In the upper part of the borehole section the vitrinite reflectance (i. e. coalification or thermal alteration of MOD) is considered as normal and regular, as in case of normal strata sequence. The rocks in the middle of the section (depths of 700 to 1300 m) seem to be effected by the strongest diagenesis (catagenesis), Rm_0 reach highest values. At depths of 1300 (1000) to 2000 m (probably even deeper) the strata sequence seems not normal, either because of scaled displacement of the layers or because of their reversed position.

These conclusions would support the hypothesis stating that the Šambron borehole penetrated a recumbent fold with the core at about the middle of the borehole section (Nemčok et al., 1977).

The reflectance values at depths of 727, 761, 781 and 1041 m do not coincide with the general trend. One possible explanation is that their outlaying positions result from displacements of the respective layers along the faults at 700 and 800 m, eventually also at 1040 and 1080 m. Deducing these conclusions we proceeded from the findings of Hacquebard (1975) and Damberger et al. (1964 in Stach et al., 1975) who described the discontinuity of the coalification curves by sudden jumps due to faults and thrusts.

Conclusions

The dispersed organic matter (MOD) in rocks of Šambron beds from the Šambron borehole has been studied microscopically in reflected light. For mean vitrinite reflectance measurements in non-polarized light the following rules may be recommended:

1. Use specimens prepared by vacuum casting into synthetic resin.
2. As a first step perform an orientational classification of the existing MOD macerals on selected specimens from the top, middle and bottom of the profile, or of the series of strata.
3. Use reflectograms to delimit the vitrinite reflectance representative range. Distinguish the maceral groups first visually, record Rm_0 values separately for

each group. The obtained reflectograms help to find the approximate boundaries exinite/vitrinite/inertinite.

4. Reflectance of MOD constituents of uncertain maceral classification should not be included in the representative Rm_0 data set for statistical calculations.

5. As representative the "low gray" vitrinites should be measured (Bostick, 1974).

6. The scanning area of photometer should always be placed upon a homogeneous part of surface of MOD particle.

The coalification stage of dispersed organic matter in the studied Šambron beds, determined by vitrinite reflectance measurements, attained the rank of coking coal according to Czechoslovak classification (corresponds with medium to low volatile bituminous coal according to ASTM).

From the point of view of the petroleum maturity the MOD in the studied potential source rocks reached the maturity level of condensate and wet gas formation. These conclusions coincide with the drilling shows: in the Šambron borehole resins and methane were detected. In the Lipany 1 borehole, 15 km to ESE, which penetrated Šambron beds to underlaying base conglomerate, gas and condensate were indicated. For quantitative estimation of hydrocarbon potential chemical analyses are necessary.

Relatively high vitrinite reflectance Rm_0 values even in subsurface samples and its low increase with depth indicate that the coalification or MOD maturation temperatures were quite high, corresponding to bigger depths of burial. The present degree of thermal alteration was reached predominantly before the upheaval and folding of the sediments.

The mean reflectance increase with relative depth in horizontal or gently dipping sequences after Teichmüller (in Stach et al., 1975) has been compared with the same relationship in the Šambron borehole. The layers in the lower part of the borehole section do not seem to be in normal position. The indications are that the highest Rm_0 values occur at the middle depths due to inverted sequence of strata at about depths of 1300 to 2000 m (probably even deeper). Such interpretation would coincide with the hypothesis of Nemčok et al. (1977) stating that the Šambron borehole penetrated a recumbent fold with core at the depth of about 800 to 1200 m. Some Rm_0 values outlaying from the general trend show a limited correlation to more important faults.

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