Thermobaric characteristics of fluid inclusions in subsalt rocks of the East of Precaspian synclise and the Pre-Ural of Aktobe

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Abstract. This article presents the results of study of fluid inclusions in calcite and quartz of the Devonian, Carboniferous and Permian strata of the eastern part of the Precaspian synclise and the Aktobe Preuralie of Pre-Ural trough. Were determined by microthermometry method the temperatures of melting and homogenization of fluids in crystals of rock and the types of hydrocarbons in inclusions. Interpretation of the measurement results made it possible to determine the most specific values of pressure and temperature, equal, respectively, 350-500 atm and 85°-130°C increasing up to 650 atm / 350°C and more towards the Urals. Accordingly, the geothermal gradient increases from west to east from 24°C/km to 40°C/km in the Aktobe Preuralie. The mineralization of fluid inclusions varies in the same direction from 10-12% to 3-6% of the weight equivalents of NaCl. These results demonstrate three types of gradients: the lowest in the western inner and platform parts of the Precaspian basin (24°C/km), the average in the Pre-Ural trough and the highest in the eastern suture with the Ural folded system. Accordingly, there is an increase in the geothermal background in the Aktobe Preuralie of Pre-Ural trough to 40°C/km.

Keywords: fluid inclusions, microthermometry, melting point, homogenization temperature, Precaspian synclise, Aktobe Preuralie region.

1. Introduction

The purpose of the study is reconstruction of the evolution of pressure and temperature of Paleozoic sediments based on the study of fluid inclusions in rock crystals of the east of the Precaspian Depression and the Aktobe Preuralie (Lower carboniferous and Lower Permian divisions). We studied the fluid inclusions in calcite and quartz of Devonian, Carboniferous and Permian systems. The melting and homogenization temperatures of fluids in crystals were determined by microthermometry.

2. Methodology

The technique of microthermometry consists in measuring temperatures at the moment of phase change in a fluid inclusion during heating (thermometry) or freezing (cryometry). Melting point (Tf) H2O: allows to determine the salinity equivalent to the weight percent equivalents of NaCl in accordance with the data in the case of fluid inclusions devoid of vaporous parts. The homogenization temperature – Th corresponds to the minimum temperature of the formation of fluid inclusions [1,2,5,11].

Fluid inclusions (FI) are one- or two-phase fluids, sometimes containing a solid phase, enclosed in intracrystalline cavities at the normal temperature of the enclosing rocks. The sizes of fluid inclusions are mostly insignificant and do not exceed 20 microns (2-10 microns, as a rule).

The use of fluid inclusions in thermobarometry is based on two fundamental hypotheses [1-5].

- Fluid inclusions are witnesses of fluids present during the primary growth or crystallization of the mineral.
- Fluid inclusions are a chemically inert fluid enclosed in a hermetically sealed cavity; this determines the constancy of the physico-chemical characteristics of fluids from the moment of their formation.

However, these conditions apply mainly to quartz. In cases of brittle minerals (calcite, anhydrite) natural decrepitation or disequilibrium of density often occurs if FI are subjected to thermobaric conditions exceeding those under which they were formed.

3. Results

3.1. Technique of study

The technique of microthermometry consists in measuring the of phase changes that occur in a fluid inclusion during freezing or heating. The used device consists of an optical microscope in transmitted light, equipped with a microthermometric plate Linkam.

Melting point (Tf) H2O: allows to estimate the salinity similar to the weight percentage equivalent of NaCl in accordance with the data in the case of fluid inclusions devoid of vaporous parts. The boiling point (homogenization-Th; total) corresponds to the minimum temperature of the formation of fluid inclusions. Homogenization (fluid, gas or critical) of organic constituents is an important parameter used to calculate the density of organic (hydrocarbon) fluids.
3.2. Features of fluid inclusions

The following types of inclusions were observed:
- Two-phase fluid (aqueous) inclusions (Lw): inclusions with a dominant aqueous phase (most often > 80%) and whose general homogenization was carried out in the fluid phase.
- Monophasic fluid (aqueous) inclusions (Mw): inclusions with a dominant aqueous phase (100%) and the total homogenization of which could not be determined, because these inclusions, which are monophasic at normal temperature, remain them despite attempts to form a gas ball at low temperatures. The Th of such inclusions can be assumed and low temperature conditions (< 80°C) are typical for them.

3.3. Distribution of different types of inclusions

More than 70 thinsections were studied on various samples, dissected by streaks or which could contain transparent neoformations of minerals. Visible fluid inclusions were found in only 17 thinsections, of which only a part could be studied due to their small size.

Within the eastern part of the Precaspian syncline monophase inclusions were most often observed in Permian sediments (Koumsai, Zhussa, Baizharyk, Keuktobe, Akkuduk) and very rarely in deeper series (terrigenous strata III at Karnak). Two-phase fluid inclusions were most often observed in Devonian series (Kumai 811, 813).

Within the Aktobe Preuralie the rock samples from the Dombay Hills area (Lower Carboniferous) and the Alexandrovka section (Lower Perm) were studied.

3.4. Microthermal characteristics

The percentage of the fluid phase varies from 85 to 95%. Most of the data were obtained for calcite, which is a brittle mineral. The diagram of the dependence of the melting temperature (Tf) on the homogenization temperature (Th) (Figure 1) allows to compare the data on microthermometry.

![Figure 1. Diagram of the Melting temperature (Tf) versus the Homogenization temperature (Th) of rock samples of Precaspian syncline. Studied structures and fields: Loktyb-Loktybai, Koum.-Koumsai, Kam.-Karnak, Keukt.-Keukto](image)

3.5. Two-phase water inclusions (Lw)

The melting point of water inclusions varies in large ranges from -38°C to +2°C. The largest number of values varies within -14/5°C. The salinity of water inclusions is quite representative for the most common diagenetic fluids (10-12% weight equivalents of NaCl).

The homogenization temperature of aqueous inclusions varies from +60 to +190°C with a maximum in the area of +92/+130°C (30 measurements) and +60/+72°C (23 measurements). The first maximum is typical for sedimentary basins that have experienced hydrocarbon-producing diagenesis (Th in the range of 100-130°C) [13].

Table 1 shows the results of micro-thermometric analysis of fluid inclusions of the Pre-Ural trough of limestones of geological outcrops of the Dombar hills of the Lower Carboniferous division and the Alexandrovsky section of the Lower Permian division. The general homogenization is carried out in the fluid phase. The homogenization temperature varies on average between +78°C and +242°C with a maximum around +90°/+110°C.

Heterogeneity of filling, coloring in normal light, different melting and homogenization temperatures allow us to assume the presence of post-trap formation phenomena (opening, volatilization).

Two-phase water inclusions (Lw) of limestones of the Dombar Hills area (C1).

Table 1. Microthermometric data of fluid inclusions of the Aktobe Preuralie of Pre-Ural trough

<table>
<thead>
<tr>
<th>Area</th>
<th>Age</th>
<th>Type of inclusions</th>
<th>Tf (min/max/ mode) °C</th>
<th>Th (min/max/ mode) °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dombar Hills</td>
<td>C1</td>
<td>Lw</td>
<td>(17)* 3/-7,5 mode (6) -6,6/-7,5 mode (6) -3/-3,6</td>
<td>(17) 76/106 mode (16) 92/106</td>
</tr>
<tr>
<td>Alexandrovka section</td>
<td>P1</td>
<td>Lw</td>
<td>(13) 0/-3,3 mode (4) -0,9/-1,2</td>
<td>(13) 90/255 mode (4) 225/240</td>
</tr>
</tbody>
</table>

Note: (17)* – number of analyses

![Figure 2. Histograms of Lw-type fluid inclusions of the Dombar Hills sample: a) Melting point (Tf); b) Homogenization temperature (Th)](image)
This area is located to the west of the formations of the Ural folded system, on the site of its maximum overhang to the west, in the zone of linear folding, which is part of the Aktobe Urals region. In the geological structure of this area take part the sediments from the Lower Carboniferous to the Upper Permian. The most elevated sections of the relief are composed of limestones, structurally lying in the core part of the synclinal fold and related by age to the Upper Viséen, Serpukhovian.

In fractures of limestone samples were observed both monophase and two-phase fluid inclusions, healed with calcite. Melting temperatures of water inclusions vary from -3°C to -7.5°C (Figures 2, 4). Three modes of values are intended within -6.6/-7.5°C (6 measurements), -3/-3.6°C (6 measurements) this indicates the circulation of different flows of diagenetic fluids with different mineralization. The salinity of water inclusions is representative for diagenetic fluids of relatively weak and medium salinity (11.2% of NaCl weight equivalents).

The homogenization temperature of aqueous inclusions varies from +78°C to +106°C with highs in the area +92/+106°C (16 measurements).

These temperature ranges are typical for sedimentary basins that have experienced hydrocarbon producing diagenesis at the level of the main oil and gas formation zone (Th are around 150°C).

Two-phase water inclusions (Lw) of the Alexandrovka section limestones (P1). In the area of the village of Alexandrovka, the deposits of the Permian system are confined to the western side of the Alexander anticline fold and lie on the Artinskian stromatolite, wavy-layered limestones overlain by organogenic-cephalopod limestones composing Mount Zheltu. The development of rocks of the Kungur also takes place along the frame of the Zhilyanska brachiactineline fold. Moreover, in the central part of its western wing, two types of sections of the Kungurian stage are distinguished: without reef limestones and with reef algal limestones forming two reef massifs in the area of the village Akzhahr (abandoned limestone quarry).

The sample is represented by limestone, light yellow, massive, biomicrite with halite content. Fluid inclusions are distributed mainly in fractures.

In the limestone sample, both monophase and two-phase fluid inclusions were observed in fractures healed with calcite. The melting temperatures of aqueous inclusions vary from 0°C to -3.3°C. One mode of values within -0.9/-1.2°C (4 measurements) is planned, which shows a relatively homogeneous composition of aqueous fluids (Figures 3, 4). The salinity of water inclusions is representative for diagenetic fluids of relatively weak salinity (5.3% of NaCl weight equivalents).

The homogenization temperature of water inclusions varies from +91 to +242°C with maxima in the area of +225/+240°C (4 measurements).

### 3.6. Data interpretation

The obtained homogenization temperatures are distributed between two geothermal gradients of 10° and 30° ± 5°C/km for samples of the Precaspian basin and up to 37°C/km for the Aktobe Preuralie. However, the Th data correspond only to a minimal assessment of the conditions for the formation of fluid inclusions.

Diagram of the dependence of the homogenization temperature (Th) on the depth (Figure 5).

It should be noted that the interpretation of data on aqueous fluids is complicated if the gas phase contains not only H2O, but also methane CH4. However, the available data of results of rock samples of Precaspian syncline allow us to draw the following conclusions:

homogenization temperatures of the rocks of the East of Precaspian syncline are distributed between two thermal gradients from 10 to 30° + 5°C /km (Figure 4). The value of this gradient can be assumed in the range of 37°C in connection with the study of samples taken on the surface.

For the rock samples of Pre-Ural trough, the percentage of the fluid phase varies from 85 to 95%. Most of the data were obtained from calcite. The diagram of the dependence of the melting temperature (Tf) on the homogenization temperature (Th) allows us to compare the microthermometry data from the samples of the Dombar Hills and the Alexandrovka section (Figure 4).
The graph based on the samples of the Dombar hills and the Alexandrovka section (Figure 8) shows, in general, a mixture of fluids of different salinity with medium and relatively high homogenization temperatures (+78/+242°C). In the samples of Dombar hills, at relatively equal homogenization temperatures of about +100°C, they have different melting temperatures. In the samples of the section Alexandrovka Th increases from +91°C to 240°C and have at the same time low melting points.

In general, for these samples of the Pre-Ural deflection, the melting point of aqueous inclusions varies in small ranges from -7.7°C to -0.4°C. The largest number of values varies within -1/-4°C and -6/-8°C. The salinity of water inclusions is quite representative for the most common diagenetic fluids (8.2% of NaCl weight equivalents).

The homogenization temperature of the water inclusions of the Pre-Ural trough varies from +78°C to +242°C with a maximum in the area of +96/+106°C (8 measurements) and +225/+240°C (4 measurements). The first maximum is typical for sedimentary basins that have experienced hydrocarbon-producing diagenesis (Th in the region of 100-130°C). The second maximum is typical for the main gas formation zone (Th in the region of 180-250°C).

a) Reconstruction of the Pressure-Temperature condition

Considering the data of salinity and density obtained by microthermometry, were constructed averaged isochores for the diagram of the pressure versus temperature using the literature data [10]. Together with the isochores, added a curve of the twophase state (gas bubble) of hydrocarbon fluids with the composition closest to those determined by infrared spectrometry, as well as geothermal gradients for hydrostatic and lithostatic pressures were plotted on the graph (Figures 6-8).

b) Conditions for the formation of water inclusions.

For two-phase inclusions, the most feature the salinity of 10-12% of NaCl weight equivalents and a sufficiently high variability of Th from a very low +60°C at Kumsai, (5638 m) to an increased +130°C. After applying isochores 65°C, 115°C to the graph, geothermal gradients (30°C/km) were also applied in lithostatic and hydrostatic modes (Figure 5).

The intersection of the isochore ‘115°C’ with a hydrostatic gradient of 30°C/km, obtained from hydrocarbon inclusions of Loktybai 14G, gives pressure/temperature parameters equal to 350 atm, 130°C. The geothermal gradient in the lithostatic mode cannot be considered, because the intersection point gives an unrealistic pressure value.

Isochora ‘65°C’ corresponds to the Kumsai 5638m sample. Interpretation of the pressure-temperature pair is not easy; since two main hypotheses are possible.

1) The fluids were formed in the traps before the deflection which is observed now. In this case, the intersection of isochores with a hydrostatic gradient (30°C/km) leads to low pressure and temperature values (180 atm, 70°C), on the contrary, the intersection with a lithostatic gradient (30°C/km) corresponds to P/T values of 500 atm and 85°C. A pressure of 500 atm corresponds to a lithostatic depth of about 2 km.

2) The fluids were formed at depths equivalent to modern depths. And possible the following two pairs of extreme P-T values: 92°C, 560 atm corresponding to a hydrostatic gradient of 12°C/km and 120°C, 1300 atm corresponding to a lithostatic gradient of 18°C/km (Figure 7).

15°C
65°C
115°C
Hydrostatic gradient
30°C/km
Lithostatic gradient
10°C/km

Figure 6. Graph of the Pressure-Temperature relationship with isochores of aqueous inclusions (Th = 65°C for Kumsai-811; averaged isochore Th = 115°C for other samples) and geothermal gradients (30°C/km) in hydrostatic and lithostatic modes

148°C
139°C
173°C
Hydrostatic gradient
12°C/km
Lithostatic gradient
54°C/km

Figure 8. Pressure-temperature diagram with isochoric intersections of water inclusions Lw (Th = 100°C, 130°C, 175°C and 285°C with temperature gradients (24°C/km) in hydrostatic and lithostatic modes

Figure 7. Geothermal gradients in hydrostatic and lithostatic modes correlated with the isochore of water inclusion (Th = 65°C) for the depth of trap formation, equivalent to the present depth (5638 m)
After applying these temperatures to the isochore graph of the rock samples of Aktobe Preuralie, geothermal gradients (36°C/km) were also applied in lithostatic and hydrostatic modes, determined by hydrocarbon inclusions of Loktybai samples [13]. The intersection of the "100°C" isochore with a hydrostatic gradient of 36°C/km gives P/T parameters equal to 260 atm, 114°C. The isochore of homogenization temperatures "220°C" and a hydrostatic gradient of 36°C/km are the parameters of P/T equal to 650 atm, 272°C. The intersection of the "100°C" isochore with a lithostatic gradient of 36°C/km gives P/T parameters equal to 690 atm, 130°C. The intersection of the geothermal gradient "220°C" with the lithostatic gradient gives inflated pressure values of 2100 atm, therefore, cannot be considered. Thus, the interpretation of the results of measurements of samples of the Precaspian basin allowed us to determine the most characteristic values of pressure and temperature equal, respectively, 300-500 atm and 80°-130°C with a geothermal gradient of about 24°C/km. Similar P/T pairs are determined by the homogenization temperatures of 90-110°C samples of the Aktobe Preuralie. However, for homogenization temperatures of 225-240°C. The reconstruction of the P/T pair for this zone shows an increase in paleotemperature values up to 270°C and paleopressure values up to 650-700 atm, which corresponds to geothermal gradients of 37°C/km.

4. Conclusions

- Mineralization of water inclusions corresponds to 7-12% of NaCl weight equivalents.
- Based on the results of the interpretation of the pressure-temperature graphs for the two most common homogenization temperatures of the east of the Precaspian syncline and the lower values of similar temperatures of the Aktobe Preuralie (80-120°C) a reconstruction of the paleo-pressure / paleotemperature pair is constructed, which showing the following values - 350 atm/85°C and 500 atm/130°C. Some Devonian samples show even higher values of pressures (up to 600 atm) and temperatures (up to 200°C).
- According to the homogenization temperatures of the samples of the Aktobe Preuralie equal to 90-240°C, the reconstruction of the P/T pair for this zone shows an increase in paleotemperature values to 270°C and paleopressure values to 650-700 atm, which corresponds to geothermal gradients of about 37°C/km.
- High pressures can explain the relative poverty of fluid inclusions – opening and volatilization during calcite deformation.
- The studied melting temperatures of fluid inclusions indicate the circulation of water flows of various composition and mineralization. The presence of many single-phase water inclusions and very low homogenization temperatures (65°C) in the Precaspian basin indicates low temperature conditions for the formation of traps corresponding to a low degree of maturity of organic matter (vitrinite reflectivity <0.8). It may also indicate that the fluid inclusions were too susceptible to postshock phenomena (deformation, opening) for their current state to bring significant help to interpretation, i.e. the measured homogenization temperatures (~65°C) may be underestimated and do not reflect the true conditions.
- These results demonstrate three types of gradients: the lowest in the western inner and platform parts of the Precaspian basin (24°C/km), the average in the Pre-Ural trough and the highest in the eastern suture with the Ural folded system. Accordingly, there is an increase in the geothermal background in the Aktobe Preuralie of Pre-Ural trough to 37°C/km.
- Increase in homogenization temperatures from the Precaspian basin towards Pre-Ural trough and from older Lower Carboniferous deposits (samples of the Dombar hills formation) - +90/+110°C towards younger samples of the Alexandrovka formation of the Lower Perm age +220/+240°C is consistent with the geological evolution of the region. The time of the processes of subduction of the Ural Paleocean along the East European Plate and, accordingly, the increase in temperature regimes falls on the Devonian period.

The complex of data obtained shows rather peculiar conditions of diagenesis of the eastern part of the Precaspian basin, characterized by relatively weak temperatures. At the same time, flows of elevated temperatures took place in certain territories, the origin of which is apparently connected with the suture subduction zone of the Ural folded system.

References

Каспий маңы синеклизінің шығысы және Актюбе Орал өңірі тұзасты жыныстардан суylық қосындылардың палеотермобариялық режимдерін сипаттамасы

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Қолдану. Каспий маңы синеклизінің шығысы және Актюбе Орал өңірі тұзасты суылық қосындыларының палеотермобариялық режимдерін сипаттамасы.

Характеристика палеотермобариялық режимінің қосылымдарына, жыныстың косындыларының палеотермобариялық режимдерін сипаттамасы.

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Аннотация. Исследованы флюидные включения в кальците и кварце девонской, каменноугольной и пермской толщ восточной части Прикаспийской синеклизы и Актюбинского Приуралья Предуральского прогиба. Методом микротермометрии определены температуры плавления и гомогенизации флюидов в кристаллах горных пород. Интерпретация результатов измерений позволила определить наиболее характерные значения давления и температуры, равные, соответственно, 350-500 атм и 85°-130°С с увеличением вплоть до 650 атм/270°С в сторону Урала. Соответственно, геотермический градиент увеличивается с запада на восток от 24°С/км до 37°С/км в Актюбинском Приуралье. Минерализация водных включений изменяется в этом же направлении от 10-12 % до 3-6% весовых эквивалентов NaCl. Эти результаты демонстрируют три типа градиентов: наименьший в западной внутренней и платформенной частях Прикаспийской впадины (24°С/км), средний в Предуральском прогибе и наибольший в восточном шве с Уральской складчатой системой. Соответственно, происходит увеличение геотермического гоносы в Актюбинском Приуралье Предуральского прогиба до 40°С/км.

Ключевые слова: флюидные включения, микротермометрия, температура плавления, температура гомогенизации, Прикаспийская синеклиза, Актюбинское Приуралье.