Gas Chromatographic Analysis of Fluid Inclusions

Sukune Takenouchi*

Abstract: Major gas compositions of fluid inclusions in quartz and anhydrite from epithermal gold deposits and active geothermal systems were determined by the combination of a vacuum system and gas chromatography. A special gas sampler was designed for collection of gases released from fluid inclusions in a vacuum system and for transfer of them to a gas chromatograph. A ball-crushing method or decrepitation method was applied to release inclusion fluids and it was concluded that the decrepitation method was preferable to the crushing method in gas analysis of quartz, because small amounts of N₂ and CH₄ were generated in case of crushing of quartz. Gas concentrations of fluid inclusions from epithermal gold deposits and geothermal systems were generally less than 2 in molality (moles/kg of water). Major gas species are CO₂, CH₄, and N₂, and CO₂ predominates over CH₄ and N₂. H₂ concentrations of fluids were calculated from CO₂ and CH₄ concentrations obtained by analysis assuming that the fluids were the H₂O-CO₂-CH₄-H₂ system in boiling condition. H₂ concentrations of inclusion fluids calculated on the assumption that the systems were not controlled with carbon, agree fairly well with the analyzed ones of the present geothermal fluids. H₂ concentrations of fluid inclusions from epithermal gold deposits are in the order of 10⁻⁷ in mole fraction, and those of the present geothermal fluids are about 1 x 10⁻⁶ but those of fluid inclusions are about 1 x 10⁻³.

1. Introduction

Several types of technique are available for analysis of gases in fluid inclusions.

The first one is the absorption method with a small crushing apparatus set on a microscope stage. A fluid inclusion is crushed in an oil between two glass plates under a microscope (Roedder, 1970). Ratio of the volume of a gas bubble released into oil to that of a bubble in fluid inclusion will be calculated from the diameter of bubble on both of the condition. When the released gas bubble is brought into contact with a proper reagent solution, which absorbs a specified gas, volume of the absorbed gas is known from the decrease of volume of the bubble (Yermakov, 1965). The accuracy of this method is low because of the uncertainty of volume estimation under a microscope, but the method is sensitive to check on the presence of compressed gases in a small fluid inclusion.

The second one is the mass spectrometry. Gases released from fluid inclusions in a vacuum system are promptly analyzed with a mass spectrometer. The method is qualified for analysis of minor amounts of gases as it is sensitive, but needs a troublesome calibration for quantitative analysis. A computerized quadrupole mass spectrometer would be handy for this purpose (Barker and Smith, 1986; Norman and Sawkins, 1987).

The third one is the gas chromatography. This method is simpler in the operation and cheaper in the price of equipment than the mass spectrometry. Fluid inclusions contain much of water as compared to gases. As water hinders the function of gas chromatography, it is desirable to separate water from gases in advance.

The above-mentioned three techniques were studied in former times especially in the glass work industry in order to know the causes of the formation of small bubbles in glass (Bryan and Neerman, 1962; Clarke and Cable, 1967; Mairlot and Girard, 1967; Makino, 1971, 1974). The same methods are applicable to gas analysis of fluid inclusions, although fluids released from inclusions contain a large quantity of water.

In the fluid inclusion study, it is necessary to obtain data from a portion of sample as small as
possible in order to correlate the data with growth zones of host crystals which represent stages of hydrothermal activity. Combination of gas chromatograph and mass spectrometer would be favorable to the inclusion gas analysis because of the small volume of gases to be analyzed.

Besides these techniques, laser-excited Raman spectroscopy is useful for the nondestructive analysis of a single inclusion but it is limited to polynuclear species in gases, liquids and solids (ROSASCO et al., 1975). The Raman spectroscopy is especially valuable in analysis of sulfur species such as $\text{SO}_4^{2-}$, $\text{HSO}_4^-$, $\text{H}_2\text{~S}$ and $\text{HS}$ (ROSASCO and ROEDDER, 1979).

2. Method of Analysis

Combination of a vacuum system for extraction of fluids from fluid inclusions and a gas chromatograph was used in the present study.

2.1 Sample preparation

Hand picked samples were crushed and sieved to a range of size between 1 mm and 0.25 mm for ball-crushing method and between 9 and 32 mesh for decrepitation method. In case of quartz, sized grains of samples were soaked in aqua regia for 24 hours in order to remove a small amount of carbonates or sulfide minerals, and cleaned with the electrolysis-cleaning method (ROEDDER, 1963) for 24 hours after washing with running tap-water for 2 hours. About two or three grams of sample was generally used for inclusion gas analysis with decrepitation method.

2.2 Vacuum system for extraction

Layout of the vacuum system is almost similar to that of the stable isotope study of $\text{H}_2\text{O}$. It consists of a vacuum crushing apparatus or decrepitation furnace, water trap, Zn-metal reducer, Pirani gauge, Toepler pump, MacLeod manometer, gas sampler, oil diffusion pump and rotary vacuum pump (Fig.1). For release of fluids from fluid inclusions, either ball-crushing method or decrepitation method was used.

Design of the vacuum ball-crusher, which consists of a pyrex glass head with a greaseless teflon stopcock, stainless steel vessel and an alumina ball, is the same as that reported by Kita (1981) except that the vessel is made of stainless steel instead of pyrex glass ( ROSASCO and ROEDDER, 1979).

![Fig.1 Vacuum system to extract inclusion fluids.](image)

![Fig.2 Design of the vacuum ball-crusher (after Kita(1981)).](image)
container were heated at 110 °C in a vacuum for 2 hours, before extraction of inclusion fluids. Gases in fluids released either by the ball-crushing or decrepitation method, were separated from water with a trap cooled by a mixture of dry ice and alcohol. Gases were collected to the MacLeod manometer with the Toepler pump and sent to a U-shaped gas sampler after measurement of the volume and pressure. The U-shaped gas sampler set, which was set on top of the MacLeod manometer, was specially designed for use in the gas chromatography. The sampler containing collected gases was transferred and inserted on the carrier gas capillary tube of the gas chromatograph with the aid of a sampler holder, which was designed to make exchange of a gas sampler easier (Fig.3).

2.3 Gas chromatography
The gas chromatograph (Shimadzu GC-3BT) is an ordinary type having a thermal conductivity detector with a set of parallel columns (Fig. 4). One of the columns (2.5 m in length) contains Molecular sieve 5A and the other consists of a series of Shimalite Q (0.5 m in length) and Porapak Q (1.5 m in length) columns. All are made of Shimadzu. These columns were kept at 60 °C in a thermobath. Helium was used as carrier gas with a flow rate of 80 ml/min. A gas mixture in a flow of carrier gas was split into the two columns and the former column retarded the flow of gases and released (N₂+O₂) mixed gas, CH₄ and CO₂, successively, and the latter retained gases longer and separated them into O₂, N₂ and CH₄ (Fig.5). Quantitative analysis of major gases in fluid inclusions such as CO₂, CH₄ and N₂, could be done with the former column when O₂/N₂ ratio was obtained from the result of the latter column. The output of detector was recorded on a chart and gas composition was calculated based on areas of peaks (Fig. 6). The sensitivity of detector varies according to gas species because of the difference of thermal conductivity of gases. Calibration factor for a gas is 1.15 for N₂, 1.20 for O₂ and 1.34 for CH₄, when the sensitivity for CO₂ is taken as standard. Thermal conductivity detector is insensitive to H₂ when helium is used as carrier gas. When Argon is used instead of helium, the sensitivity for H₂ is improved but those for the other gases decrease appreciably (Fig. 5). Relative error of gas chromatography is estimated to be less than 10% for CO₂, but it reaches as high as 60% for CH₄ and N₂ in some cases as the amounts of these two
gas components are generally much less compared to that of CO₂.

The trapped water was released after the completion of gas collection, and reduced to H₂ by passing on Zn-metal chips in the reducer at 400°C. The generated H₂ was collected into the MacLeod manometer and the pressure and volume was measured. The amount of released water could thus be calculated from the volume of H₂.

3. Gas Composition of Fluid Inclusion

3.1 Major gas composition

Major gases are generally CO₂, CH₄ and N₂. Although CO, H₂S, SO₂, H₂, O₂ or hydrocarbons other than CH₄ might be contained in fluid inclusions, those gases were not determined in the present study. In some cases, indistinct low peaks of O₂, hydrocarbons and H₂ were identified on gas chromatograms. When the peak of O₂ was observed, the corresponding amount of N₂ in N₂/O₂ ratio of air (3.73) was reduced from the measured amount of N₂, considering that a small amount of air had leaked into the vacuum system. Peaks for hydrocarbons were vague and ignored. Two low peaks for H₂ were occasionally recognized as shoulders of the first two peaks (O₂+N₂ and CH₄ peaks), when quartz samples were crushed. H₂ generation was probably because of a reaction between newly formed surface of quartz and water (KITA, et al., 1982).

3.2 Advantage and disadvantage of crushing and decrepitation method

Crushing method: Crushing efficiency of the vacuum ball-crusher was checked. Fused silica glass or transparent Brazilian quartz, which was crushed, burned at 1000°C, screened to the size between 1.0 mm and 0.25 mm and washed with acid and deionized water, was used for the crushing test. Crushing time was variable from 20 to 40 minutes. Fig. 7 shows some results of crushing tests. Crushing efficiency varied according to

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Fig. 6 Chromatograms of inclusion gases from the Nebazawa and Kasuga gold deposits. They are shown for ball-crushing, decrepitation at a higher temperature and at a lower temperature.
Fig. 7 Efficiency of the vacuum ball crushing.
Abbreviation; 1st S: stainless steel vessel, 2nd A: alu-
mmina ball, 2nd S: stainless steel ball, 3rd G: fused silica
glass, 3rd Q: quartz, last two figures: crushing time in
minutes.

sample species, kind of material of ball and crushing
time. The efficiency of an alumina ball was
much less than that of a stainless steel ball owing
to the difference of weight of ball (Fig.7, SAG-20,
SSG-20). In 20 minutes crushing, the finest part (smaller than 74 μm) were generally in a range from 50 to 60 wt. % of the crushed product in case of alumina ball (60 g in weight), but it occupied more than 70 wt.% in case of stainless steel ball (130 g in weight). The stainless steel ball, how-
ever, was not preferable for crushing of quartz because of the generation of N₂ and CH₄ during crushing, as it is discussed later. Extension of crushing time did not improve appreciably the crushing efficiency (Fig.7, SAG-20, SAG-30 and SAG-40). This is probably due to that coarser grains were buried in a mass of powder and not crushed furthermore when the crushed powder reached to a certain amount. The crushing method can not open all of fluid inclusions in a sample mass, especially when fluid inclusions are small.

Decrepitation method: Decrepitation method has some advantages compared with the crushing method. Those are 1) decrepitation apparatus is much simpler in design and much less possibility in leakage of air than ball-crusher, 2) the inside volume of the decrepitation tube is much less than that of the crushing vessel and this reduces the time necessary for recovery of gases with the Toepler pump, and 3) stepwise heating is available in decrepitation in order to collect separately the contents of primary and secondary inclusions in the same sample.

Disadvantage of decrepitation method are 1) released gases may change the composition owing to reactions with each other or with host minerals at high temperatures, and 2) gases generated by decomposition of host minerals such as sulfides, carbonates or hydrates may contaminate the gas composition of fluid inclusions. Accordingly, in case of decrepitation, a sample was heated up to about 500 °C within a few minutes and after that cooled down promptly to 110 °C and kept at the temperature during collection of gases in order to suppress the reaction between gases as low as possible.

### Table 1 Generated amount of N₂ and CH₄ at crushing of fused silica glass in vacuum or in a CO₂ atmosphere by ball-crushing.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Gases/g glass (10⁻⁶mole)</th>
<th>Mole fraction</th>
<th>Generated amount 10⁻⁶mole/g/glass</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>N₂</td>
<td>CH₄</td>
<td>N₂</td>
</tr>
<tr>
<td>SAG-20(CO₂)</td>
<td>0.498</td>
<td>0.809</td>
<td>0.191</td>
</tr>
<tr>
<td>SAG-20(CO₂)</td>
<td>0.383</td>
<td>0.908</td>
<td>0.092</td>
</tr>
<tr>
<td>SAG-20(CO₂)</td>
<td>0.476</td>
<td>0.835</td>
<td>0.165</td>
</tr>
<tr>
<td>SAG-25(CO₂)</td>
<td>0.332</td>
<td>0.898</td>
<td>0.102</td>
</tr>
<tr>
<td>SAG-40(CO₂)</td>
<td>0.527</td>
<td>0.826</td>
<td>0.174</td>
</tr>
<tr>
<td>SSG-20</td>
<td>0.221</td>
<td>1.0</td>
<td>0</td>
</tr>
<tr>
<td>SSG-15(CO₂)</td>
<td>0.960</td>
<td>0.684</td>
<td>0.316</td>
</tr>
<tr>
<td>SAG-20 ave.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

SAG: Crushing of glass with an alumina ball in a stainless steel vessel. SSG: Crushing of glass with a stainless steel ball in a stainless steel vessel. Figures represent crushing time in minutes. (CO₂) means crushing in a CO₂ atmosphere.

In the preliminary stage of inclusion gas analy-
ysis, it was noticed that a fairly high gas concentra-
tion was obtained even though from a quartz sample containing few small fluid inclusions. Blank crushing tests with grains of fused silica glass in a vacuum yielded a certain amount of N₂ and when CO₂ existed in such blank tests a small amount of CH₄ was generated in addition to N₂ (KITA et al., 1982; ANDRAWES et al, 1984; KAZAHARA and MATSUO, 1985). The results of blank tests are shown in Table 1. It was found that 0.0084 cm³ (0.38x10⁻⁶ mole) of N₂ and 0.0016 cm³ (0.07x10⁻⁶ mole) of CH₄ per 1 gram of quartz were generated on an average in case of a normal crushing condition (crushing of 3 g sample for 20 minutes with an alumina ball). In the crushing method of quartz, therefore, these amounts were reduced from the measured amount of N₂ and
CH₄, respectively, as contamination during crushing. Because crushing in a stainless steel vessel with a stainless steel ball generated a more amount of N₂, the stainless steel ball was not used in the inclusion gas analysis of quartz, although the crushing efficiency was better.

4. Results of Analysis

Results of inclusion gas analysis of quartz from epithermal gold quartz veins and siliceous bodies, and ejected quartz from a Broadlands geothermal well, are shown in Table 2 and Figs. 8 and 9. In general, CO₂ is the most abundant gas species in fluid inclusions and H₂O occupies more than 95 mole % of inclusion fluids. Gas concentrations of hydrothermal fluids of epithermal gold deposits and geothermal areas are generally less than 2 in molality (moles/kg of water) and much dilute as compared with those of hypothermal or mesothermal tungsten or gold deposits, that range from 2 to 6 in general (TAKENOUCHI, 1991).

Separate collection of inclusion fluids at high and low temperature portions of decrepitation was tested on samples from the Yatani, Seigoshi and Nebazawa (Table 2 and Fig. 8). Based on the range of homogenization temperatures (Th) of...
In analysis of fluid inclusions of mineral deposits, it is difficult to know the chemistry of original fluids to check the results, as inclusion fluids are ancient fluids trapped in minerals. In case of active geothermal systems, however, compositional data of the present thermal fluids, with which the results of inclusion analysis are comparable, are occasionally available, although there is no reason to believe that these two fluids have the same origin. In addition to the results of gas analysis on quartz from the Broadlands BR-45 geothermal well (Table 2), results on vein-filling anhydrite from the Oku-aizu geothermal well are shown in Table 3, and gas compositions of the present fluids from these wells are shown in Table 4.

The CO₂ concentration of the present fluid
(0.84 in molality(m.)) from the 84N-2t well is somewhat lower than that of the inclusion fluid (1.2 m. on an average), and the present fluid from the BR-45 well is fairly lower (0.24 m.) than the inclusion fluid (1.1 m.). The samples from these two geothermal fields contain a large number of gaseous inclusions of various degrees of filling besides liquid inclusions. As the decrepitation method was used at 440-530°C in order to extract inclusion fluids, many gaseous inclusions must have been left intact but some of them were opened and might have effected on the results of analysis, although it was impossible to estimate the volume ratio of opened and un-opened inclusions. The difference of CO2 concentration between the present geothermal and inclusion fluids is probably due to either the difference of gas composition of fluid itself or contamination with gas-rich inclusions, or either of them. CH4 and N2 concentrations of the present fluids from both of the wells are much lower than those of inclusion fluids.

As the present study is a preliminary work to know a tendency of gas compositions of epithermal systems, it will be necessary to do more detailed studies in order to come to a conclusion concerning characteristics of fluids of individual systems.

5. CO2-CH4-H2-H2O System

When the liquid and gas phase coexist and the system is in equilibrium, CO2, CH4 and H2 concentrations in aqueous solutions are controlled by the following reactions.

\[
\begin{align*}
\text{CH}_4 + 2\text{H}_2\text{O} &= \text{CO}_2 + 4\text{H}_2 \quad (1) \\
2\text{CH}_4 + 2\text{H}_2\text{O} &= \text{CO}_2 + 6\text{H}_2 + \text{C (solid)} \quad (2)
\end{align*}
\]

Equilibrium constants of these reactions are expressed as follows.

\[
\begin{align*}
\text{Kp}_1 &= \frac{[\text{P(CO}_2)]\times[\text{P(H}_2^4)]}{[\text{P(CH}_4)]\times[\text{P(H}_2\text{O})^2]} \quad (3) \\
\text{Kp}_2 &= \frac{[\text{P(CO}_2)]\times[\text{P(H}_2^6)]}{[\text{P(CH}_4)]^2\times[\text{P(H}_2\text{O})^2]} \quad (4)
\end{align*}
\]

where Kp is the equilibrium constant and P(i) is the partial pressure of i species.

TRUESDELL (1984) expressed Kp values as a function of temperature as follows.

\[
\begin{align*}
\log\text{Kp}_1 &= 10.278 - 9082x(1/T) \\
\log\text{Kp}_2 &= -6.82 - 11801x(1/T) + 7.11x\log T
\end{align*}
\]

As GIGGENBACH (1980) expressed the partial pressure of water as a function of temperature at or near boiling condition of hydrothermal fluid and TRUESDELL (1984) gave Henry's Law constants as a function of temperature in a temperature range between 200°C and 350°C, H2 mole fraction [X(H2)] in solution is expressed as functions of CO2 and CH4 mole fractions [X(CO2) and X(CH4)] and temperature [TK], as follows.

For reaction (1),

\[
\log\text{X(H}_2) = \frac{[\log\text{X(CH}_4) - \log\text{X(CO}_2) - 13180x(1/T) + 0.01959xT - 5.9218]}{4}
\]

For reaction (2),

\[
\log\text{X(H}_2) = \frac{[2\times\log\text{X(CH}_4) - \log\text{X(CO}_2) + 0.02542xT - 7705x(1/T) - 7.11x\log T - 16.9179]}{6}
\]

As the mode of occurrence of fluid inclusions in samples from the Oku-aizu and Broadlands geothermal fields suggests the boiling of fluid at their trapping (TAKENOUCHI, 1988a; HEDENQUIST, personal communication), equation (7) or (8) is applicable to calculate H2 concentration of fluid.

Calculated H2 mole fractions of the inclusion fluids and present fluids of these two geothermal fields are shown in Table 5. Analyzed H2 concentrations of the present eothermal fluids are slightly lower but much closer to the calculated ones using equation (7) than those of equation (8), suggesting that the present fluids of the Oku-aizu and Broadlands are not controlled by carbonaceous matter in the surrounding rocks. Calculated H2 concentrations of inclusion fluids from the geothermal systems agree fairly well with those of the present fluids. This indicates that H2 concentrations of fluids may have not changed remarkably since the time of trapping of fluid inclusions when it is assumed that CO2 and CH4 concentrations of fluid inclusions did not change appreciably since the trapping.

Calculated H2 concentrations of fluid inclusion from the Kawarage silicified rock and Kasuga Nansatsu-type gold deposit are shown in Table 5 in comparison with those of the above-stated two geothermal fields, as it is inferred that the fluids of
these two epithermal systems were under the boiling condition (TAKENOUCHI, 1983, 1984).

6. Concluding Remarks

Gas compositions of fluid inclusions in quartz and anhydrite from epithermal gold deposits and geothermal areas were determined by the gas chromatography.

Fluids released from fluid inclusions in a vacuum system either by the crushing or decrepitation method, were separated into water and non-condensable gases. Gases consist mainly of CO₂, CH₄ and N₂. In case of crushing of quartz, a small amount of N₂ was generated by abrasion between stainless steel and quartz, and CH₄ was formed by a reaction between fractured fresh surfaces of quartz and CO₂. Decrepitation is preferable for extraction of inclusion fluids from quartz.

Gas concentrations of fluid inclusions in quartz from epithermal gold deposits and siliceous rocks are generally low less than 2 in molality and CO₂ predominates over CH₄. It is shown that H₂ concentrations of geothermal fluids calculated from the analyzed CO₂ and CH₄ concentrations on the assumption that the fluids were under the boiling condition and that the systems were not controlled with solid carbon, agree roughly with the analyzed H₂ concentration. As is shown in Table 5, calculated H₂ concentrations of geothermal fluids assuming that the fluids were not controlled with carbon, agree fairly well with the analyzed ones of the present geothermal fluids. This fact does not necessarily suggest the same age of hydrothermal fluids of the present geothermal systems and fluid inclusions, but possibly shows that the hydrothermal fluids of the Oku-aizu 84N-2t and Broadlands BR-45 drill hole were free from carbon in the surrounding rocks.

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References


BRYAN, F. R. and NEERMAN, J. C. (1962) : Gas chromatogra-
Appendix

Deposit (1)Location: (2)Abbreviation: (3)Remarks

Chitose (1)Hokkaido: (2)(D3): (3)Daikoku 3goh-hi-240mL

Seigoshi (1)Shizuoka: (2)(2V): (3)2goh-hi-52mL S2.
Th: 205-240°C.

Nebazawa (1)Gunma: (2)(M3V): (3)Manzai 3goh-hi-120mL
Th: 130-185°C; 205-255°C.

Kohryu (1)Hokkaido: (2)(1HV): (3)lgoh-uwabanhi 60mL.
Th: 215-275°C.
(2)(3Va): (3)3goh-hi-120mL No. 3R, Center of vein.
Th: 220-275°C.
(2)(3Vb): (3)ditto Wall side of vein.
Th: 205-260°C.
(SUGAKI et al., 1984).

Yatani (1)Yamagata: (2)(HT): (3)Higashi-tenguhi 1L N2
(SHIKAZONO, 1985). K-Ar age of adularia is 3.6±0.27 Ma (SHIKAZONO, 1985). Th: Not available.

Kasuga (1)Kagoshima: (2)(HK): (3)Honkohtai 20mL
(TAKENOUCHI, 1983; 1988b). Small quartz crystals in cavities of the Nansatsu-type (Hot spring-type) gold deposits, which have been formed in a silicified body with a thin alunite-kaolinite alteration zone in andesitic lavas and pyroclastic rocks. K-Ar age of natro-alunite is 5.5±0.4 Ma (IZAWA et al., 1984). Th: 165-240°C. Boiling of fluid at 230°C is presumed from the mode of occurrence of fluid inclusions.

Quartz phenocrysts of altered quartz porphyry in a silicified body with a thin alunite-kaolinite alteration zone, which has been formed in Miocene dacitic pyroclastic rocks. Th: 200-260°C. Boiling of fluid at 220°C is presumed from the mode of occurrence of fluid inclusions.

Oku-aizu (1)Fukushima: (2)(2t): (3)84N-2t drill hole, 1469.4 m.
Th: 230-250°C.
(2)(13t): (3)86N-13t, 1227.4 m.
Th: 155-175°C.
(2)(18t): (3)87N-18t, 1365.3 m.
Th: 175-190°C.
Samples were collected from anhydrite veins in drill cores. Boiling of fluid is presumed from the mode of occurrence of fluid inclusions (TAKENOUCHI, 1988a; 1991).

Broadlands (1)New Zealand: (2)(BR): (3)BR-45 drill hole, 490 m.
Fluid inclusion data. Th: 254-265°C (ave. 259°C); Tm: -0.5 -0.9°C (ave. -0.6°C); Boiling of fluid is presumed(J. W. HEDENQUIST, personal communication).

流体包有物のガスクロマトグラフ分析

武内 寿久雄

要旨：浅熱水成金鉱床および地熱系の石英、硬石膏中の流体包有物の主要ガス組成を真空抽出装置とガスクロマトグラフの組合わせによって分析した。真空中で流体包有物から放出されたガスを捕集し、ガスクロマトグラフに移すための特別なガス・サンプラーを考案した。包有物からの流体の解析には、ボール破砕法とデクレピテーション法とを試みたが、石英試料の場合にはボール破砕法では少量のN2とCH4が生成するので、デクレピテーション法の方が好ましいことが分かった。浅熱水成金鉱床と地熱系の流体包有物のガス濃度は一般に2モル濃度よりも少なく、主要ガス成分はCO2, CH4, N2で、CO2が最も多い。H2濃度は、流体が沸騰（気液二相）条件にあるH2O－CO2－CH4－H2系と仮定して、測定されたCO2, CH4濃度から計算によって求めめた。地熱系の場合、固相Cが共存しない系として求めたH2濃度は、地熱井で実測されたH2濃度に近いものであった。H2濃度は、浅熱水成金鉱床では10⁻⁶モル分率、地熱系では10⁻³モル分率であった。