Application of Fluid Inclusion Studies to Oil and Gas Exploration

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National University of Ireland, Galway.
The Application of Fluid Inclusion Studies to Oil & Gas Exploration.

Overview of Fluid Inclusion Studies and Applications

Sampling, Sample Preparation, Fluid Inclusion Petrography

Analytical Methods

Microthermometry, Geothermobarometry

Review of Case Histories from the OIL & Gas Exploration Sector

Parson’s Pond project—Dr James Conliffe, GSNL

Tour of Facilities, MAF-IIC Laboratories, Memorial University.
What are Fluid Inclusions?

Fluid inclusions are tiny bubble-like inclusions in crystals. They are composed of liquid ± solid ± gas phases in varying proportions. They represent fluid trapped during or after crystal growth.

They have the potential to elucidate the PTVX properties of fluids during many geological processes e.g. mineralisation, petroleum migration, magmatic and metamorphic processes.

And now the Biosphere and Martian exploration!
Fluid Inclusions

- Early work by H.C. Sorby (1858)
FLUID INCLUSION STUDY METHODS.

PETROGRAPHY

MICROTHERMOMETRY

LASER RAMAN

UV MICROSCOPY

FLUORESCENCE LIFETIME MICROSCOPY

ELECTRON MICROSCOPY.
What Are Fluid Inclusions?
Microscopic volumes of fluid (liquid ± gas ± solid) trapped during and/or after mineral growth in a rock.

Where Are They Found?
Most geological settings. Includes Sedimentary Basins such as in Rockall & Porcupine Regions.

Why Study Fluid Inclusions?
1. Timing of fluid movements
2. Recording fluid movement events.
3. Chemistry of fluids.
4. Temperature of fluid entrapment.
5. Pressure of fluid entrapment.
6. Exploration for Oil & Gas and Mineral Deposits.
1 micron (μm) = 0.001mm

20 microns = 0.02mm

○ Ballpoint of biro.
What are Fluid Inclusions

Aqueous Fluid Inclusions
What are Fluid Inclusions

H₂O + CO₂
Fluid Inclusions
What are Fluid Inclusions

Daughter and/or captive minerals
What are Fluid Inclusions

Hydrocarbon Bearing Fluid Inclusions

Transmitted Light Only

UV Light
Occurrence and distribution

MOST INCLUSIONS < 100 MICRONS AND COMMONLY RANGE BETWEEN 2 and 20 MICRONS.
THE TOP TEN*:

*RATINGS ARE BASED ON
ROBUSTNESS, TRANSLUCENCY
AND OCCURRENCE

1-Quartz
2-Fluorite
3-Halite
4-Calcite
5-Apatite
6-Dolomite
7-Sphalerite
8-Barite
9-Topaz
10-Cassiterite
Hydrothermal Systems

- Meteoric Water Systems
- Magmatic Water Systems
- Seawater System
- Metamorphic Water System
- Basinal Water/Hydrocarbon System
FLUIDS IN IRISH GRANITES

Main Fluid Inclusion Types In Granites & Veins

A

L2
L1

Aqueous Carbonic + NaCl (Magmatic)

Main Occurrence: Granite Quartz + Granite Related Mineral Veins

B

Aqueous + NaCl (Meteoric)

Main Occurrence: Granite Quartz + Granite Related Mineral Veins

C

Aqueous + NaCl + CaCl₂ (Mixed Fluids - Lower Carboniferous to Triassic ?)

Main Occurrence: Calcite/Quartz Veins + Base Metal
Also Granite Quartz + Granite Related Mineral Veins
Molybdenite Re-Os age determinations

Aqueous-carbonic fluids trapped in Mo-Cu quartz veins

Zircon U-Pb age determinations
MACE HEAD MOLYBDENITE BEARING QUARTZ VEIN
CLASSIC DOUBLE BUBBLE AQUEOUS CARBONIC FLUID INCLUSION

CARBON DIOXIDE (L)

SALINE AQUEOUS PHASE (L)

CARBON DIOXIDE (V)

TRAPPED IN GALWAY GRANITE QUARTZ ~ 407 Ma.
1. Scottish Granite Mo-system.

2. Lake District Granite Mo-system.

3. Connemara Granite Mo-system.

4. Newfoundland Granite Mo-system.


Euhedral molybdenite crystal in Ackley City Granite, Newfoundland. Pen length = 14 cm
Oil and Gas Genesis

- Burial to depths of 2 to 4 km heats the black shale.
  - Heating breaks the organics down into waxy kerogen.
  - Kerogen-rich source rocks are called oil shales.
- Continued heating breaks down kerogen.
- Oil and gas form in specific T ranges.
  - Oil and gas – 90° to 160°C.
  - Gas only – 160° to 250°C.
  - Graphite – >250°C.
Example of cross-plot used for describing the burial of sediments through time. The subsidence curves describe the burial history of the sediments. The figure indicates when they are within the oil/gas window. If organic rich sediments are uplifted, they also cool, and stop generating hydrocarbons until they are reburied.
TWO-PHASE (L+V) HYDROCARBON BEARING FLUID INCLUSIONS CELTIC SEA BASIN, OFFSHORE IRELAND.

TRAPPED IN VEIN CALCITE, NEGATIVE CRYSTAL SHAPES = PRIMARY, TRAPPED DURING GROWTH OF CALCITE, TERTIARY(?).
Geofluid Research Group
Fluid Inclusion microthermometry
Sample Collection

- Recent USA halite samples (up to 150ka)
  - Searles Lake, Death Valley, California
- Messinian Italian halite and gypsum (~5-7 Ma)
  - Halite from the Crotone basin
  - Gypsum from the Rossano basin
- Miocene-Cretaceous-Triassic Spanish gypsum
  - Valencia province
  - Terruel province
  - Almeria province
- Permian Irish gypsum
  - Kingscourt Co.Cavan
Fluid Inclusion Petrography

(a) Italian halite

(b) Italian halite

(c) Irish gypsum

(d) Spanish gypsum
Figure 1. Composite diagram showing the location of the Mugford Group in Northern Labrador and the stratigraphic setting of the Sunday Run Formation.
Figure 1. Sunday Run Formation sample showing quartz pebbles. This is a specially prepared fluid inclusion wafer. Pebbles are <5mm across and contain an abundance of fluid inclusions (FI) containing fluids that are at least 2 billion years old. These FI are the focus of this study.
Three-phase inclusions in Tourmaline-(Trichites)

Two liquids and a gas phase occupy the cavity

Strongly indicates a natural gemstone
Application of Fluid Inclusion Studies to Oil and Gas Exploration

LECTURE 2:

SAMPLING AND PETROGRAPHY
1st column ~5 cm in longest dimension. 2nd column the fluid inclusion wafers are 4 x 2.2 cm and 3rd column photomicrographs taken under x40 magnification.
Orthoscopic Illumination

Light path through the microscope for fluid inclusion petrography. Substage condensing lens must be in place just below the thin section/wafer.
1 micron (μm) = 0.001 mm

20 microns = 0.02 mm

20 microns

<10 microns

1 mm

X100

• Ballpoint of biro.
Inclusion morphologies
## Compositional Classification

<table>
<thead>
<tr>
<th>Inclusion Type</th>
<th>Essential Phases</th>
<th>Typical Examples</th>
<th>Inclusion Type</th>
<th>Essential Phases</th>
<th>Typical Examples</th>
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<tr>
<td>Monophase liquid</td>
<td>( L = 100% )</td>
<td><img src="image" alt="Example" /></td>
<td>Multiphase solid</td>
<td>( S &lt; 50% )</td>
<td><img src="image" alt="Example" /></td>
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<tr>
<td>Liquid-rich two-phase</td>
<td>( L &gt; 50% )</td>
<td><img src="image" alt="Example" /></td>
<td>Multisolid</td>
<td>( S &gt; 50% )</td>
<td><img src="image" alt="Example" /></td>
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<tr>
<td>Vapour-rich two-phase</td>
<td>( V = 50 \text{ to } 80% )</td>
<td><img src="image" alt="Example" /></td>
<td>Immiscible liquid</td>
<td>( L_1, L_2 )</td>
<td><img src="image" alt="Example" /></td>
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<tr>
<td>Monophase vapour</td>
<td>( V \geq 100% )</td>
<td><img src="image" alt="Example" /></td>
<td>Glass</td>
<td>( GL &gt; 50% )</td>
<td>not shown</td>
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</table>
Genetic Classification

**Primary origin**
1. Inclusions are parallel to growth zones or crystal faces.
2. Inclusions occur in a three-dimensional random distribution.
3. Inclusions are isolated, occurring at distances > 5 x inclusion diameter away from adjacent inclusions.
4. Large size relative to host crystal.

**Criteria for Secondary origin**
1. Occurrence as planar groups, outlining healed fractures.
2. Thin, flat, or irregular shape.

**Criteria for Pseudosecondary origin**
Occurrence as for secondary inclusions, but with the fracture terminating inside the body of the crystal.
Genetic Classification

Idealized distribution of P, S and PS fluid inclusions in quartz (A) and fluorite (B). (from Shepherd et al., 1985)
PRIMARY FLUID INCLUSIONS IN VEIN QUARTZ.
TWO-PHASE (L+V) HYDROCARBON BEARING FLUID INCLUSIONS CELTIC SEA BASIN, OFFSHORE IRELAND.

TRAPPED IN VEIN CALCITE, NEGATIVE CRYSTAL SHAPES = PRIMARY, TRAPPED DURING GROWTH OF CALCITE, TERTIARY(?).
Fig. 8. Sketches of distribution of primary fluid inclusions in various minerals of the sedimentary realm (modified from Goldstein and Reynolds, 1994). (A) Calcite, (B) Dolomite, (C) Anhydrite, (D) Quartz overgrowth, (E) Felspar overgrowth, (F) Chevron halite, (G) Gypsum (modified from Lowenstern, written communication).
VEIN QUARTZ HOSTED FLUID INCLUSIONS

Thin-section of vein quartz from Glenmalure

Inclusions from earlier generation
VEIN QUARTZ HOSTED FI.

Inclusions from later generation
Several planar arrays of secondary fluid inclusions that formed in healed fractures. Cave-in-rock Fluorite, Illinois.
Fluid Inclusion Petrography

Schematic representation of fluid inclusions in a sandstone

• 3 generations of fluid represented
Fluid Inclusion Petrography

Schematic representation of fluid inclusions in a sandstone

Inclusions hosted in trails or clusters within detrital quartz grains

- Represent fluids activity prior to cementation
Fluid Inclusion Petrography

Schematic representation of fluid inclusions in a sandstone

Inclusions hosted within cements at grain overgrowth boundaries

- Represent fluids present during cementation
Fluid Inclusion Petrography

Schematic representation of fluid inclusions in a sandstone

Inclusions hosted in trails that crosscut grain boundaries

- Represent fluids present after cementation
5. Fluid Inclusion Petrography

Six main fluid inclusion types (Type 1, Type 2, Type 3, Type 4, Type 5 and Type 6) were recorded during the study. This fluid inclusion classification is based upon: (a) their phase relations (presence at room temperature of liquid + vapour phases or, liquid phase only) and (b) the textural relationship between inclusions and their host mineral and/or cement (Table 2). Fluid inclusions in all samples possess a range of morphologies: from ellipsoidal to irregular shapes, and rare negative crystal shapes.
~400 Ma aqueous carbonic fluid inclusion from the Galway Batholith

DOUBLE BUBBLE AQUEOUS-CARBONIC FLUID INCLUSION
Figure 6.38  Worked example to calculate the bulk density and wt% CO₂ content of an H₂O–CO₂ inclusion.

A. \( T_{\text{H₂O}} \) (L + V \rightarrow L) = 26°C = 0.7 g cm\(^{-3}\) (from Figure 6.17)

B. CO₂ phase occupies 30% by volume (visual estimate)
   H₂O phase occupies 70% by volume (visual estimate)

C. Assume (i) salt content of H₂O phase is low or negligible and therefore density can be taken as 1.0 g cm\(^{-3}\) at room temperature
   (ii) solubility of CO₂ in H₂O phase and H₂O in CO₂ phase are negligible at room temperature.

D. Thus bulk density of H₂O – CO₂ inclusion is given by

\[
(0.3 \times 0.7) + (0.7 \times 1.0) = 0.91 \text{ g cm}^{-3}
\]

\[
\frac{(\text{vol} \times \rho)}{(\text{vol} \times \rho)}
\]

Wt% CO₂ is given by

\[
\frac{0.21}{0.91} = 23.1 \text{ wt% CO₂}
\]
Application of Fluid Inclusion Studies to Oil and Gas Exploration

LECTURE 3

FLUID INCLUSION ANALYTICAL METHODS
FLUID INCLUSION ANALYTICAL METHODS

POLARISED LIGHT MICROSCOPY

MICROTHERMOMETRY

UV-MICROSCOPY

CATHODOLUMINESCENSE

FLUORESCENCE LIFETIME MICROSCOPY

LASER RAMAN MICROSCOPY

SCANNING ELECTRON MICROSCOPY
Geofluids Research Laboratory, NUI, Galway

Microthermometric Unit
Fluid Inclusion Microthermometry

- Microthermometry is carried out using a Linkam THMSG 600 heating freezing stage
- Specific phase changes within an inclusion are measured
- Behaviour upon heating and freezing of inclusions is indicative of fluid composition and minimum trapping temperatures
UV Fluorescence

• Samples examined under ultra-violet (UV) light using a Nikon Diaphot microscope with an epifluorescence attachment

• Used to distinguish between HCFI and aqueous inclusions
UV Fluorescence

Transmitted Light Only

UV Light (365 nm)

Magnification x50
UV Fluorescence

Transmitted Light Only

UV Light (365 nm)

Magnification x100
CIE x, y chromaticity diagram

- R: Red
- B: Blue
- G: Green
- Y: Yellow
- O: Orange
- P: Purple
- Pk: Pink

Lower case: “ish” takes suffix
Fig. 5. Fluorescence colours of HCFI from the Porcupine Basin, plotted on CIE-1931 diagram.
UV Fluorescence

- When exposed to UV light, hydrocarbons emit light in the visible spectrum.
- The relationship between fluorescence colour of HCFI and chemical composition of oils is highly complex but can give a first approximation of the composition of the hydrocarbon.
CMAs are relatively inexpensive attachments to microscopes. A high voltage (10-30 keV) cold cathode gun discharges electrons in a low vacuum chamber (rough pump only). A plasma results that provides charge neutralization (no carbon coating necessary). A camera (film or digital) and/or monochromometer are attached to acquire images and/or wavelength scans of the light.
Cathodoluminescence

Evaluating minerals for heterogeneous growth (complex history, overgrowths, dissolution, crack infilling)

A: Casserite, SnO$_2$
B: Crinoidal limestone
C: Red = dolomite, orange = calcite; dark grey = baddeleyite (ZrO$_2$)
D: St Peter Sandstone; mature quartz with zoned authigenic quartz overgrowths
(from Marshall, 1988, CL of Geological Materials)
Figure 5: Upright ISS Alba system for Fluorescence Lifetime Imaging Microscopy.
Raman spectroscopy of FIs at NUI Galway
In situ microanalysis of FI phases (solid, vapour, liquid)
Raman peaks of gases trapped in FIs

![Raman spectrum](image)

- **CO2**
- **H2O**
- **N2**
- **CH4**
- **H2**
Hydrothermal vein from a Mo-Cu deposit, Newfoundland
Mixed Raman spectrum: quartz + opaque phase in Fl
SEM STUBS HOLDING UPRIGHT TO THE BEAM FRESHLY EXPOSED SURFACES OF VEIN MINERALS: QUARTZ, CALCITE, FLUORITE & GALENA FROM WEST OF IRELAND
OCTAHEDRON OF FLUORITE; FLUORITE HOSTED INCLUSION, CLARE, IRELAND.

SPATIAL DENDRITES OF HALITE; FORMED ON SURFACE OF QUARTZ FROM FRESHLY OPENED HIGHLY SALINE INCLUSIONS, CONNEMARA
PYRITE CUBE IN OPENED INCLUSION IN QUARTZ FROM MACE HEAD, IRELAND

MICA IN GRANITE QUARTZ, MACE HEAD, IRELAND

SEVERAL EMPTY INCLUSIONS IN QUARTZ MACE HEAD, IRELAND.
Application of Fluid Inclusion Studies to Oil and Gas Exploration

LECTURE 4

FLUID INCLUSION MICROTHERMOMETRY AND FLUID P-T-X MODELLING
Fluid Inclusion Microthermometry

- Microthermometry is carried out using a Linkam THMSG 600 heating freezing stage
- Specific phase changes within an inclusion are measured
- Behaviour upon heating and freezing of inclusions is indicative of fluid composition and minimum trapping temperatures
DEPRESSION OF FREEZING POINT OF PURE WATER BY DIFFERENT ADDED SALT.
DENSITY-COMPOSITION-DEGREE OF FILL
## STEAM TABLES

### Table 17. Densities of 3 weight percent NaCl solutions, g/cm³

[The uncertainties in the densities are: 3-place figures ±0.005 and ±0.05 for 1-place figures.]

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*Extrapolated values
Isochore for aqueous fluid with ~7wt% NaCl

ISOCHORE

Density = 1.001 gm/cc

ISOCHORE = A line of constant density in P-T space
Microthermometric Unit

Geofluids Research Laboratory, NUI, Galway
Linkam THMS 600

Fluid inclusions are observed using high power objectives (×40, ×60 or ×100).

Technical Specifications

Temperature range: -180°C to +600°C
Temperature resolution: 0.1°C (-180 to +200°C)
1°C (+200 to +600°C)
Temperature control: fully automatic and programmable
Max sample size: 20 mm diameter;
1.5 mm thickness
Viewing area: 2.2 mm diameter
TWO-PHASE AQUEOUS INCLUSION FREEZING RUN

-25 C

-40 C

-80 C

-30 C

-15 C

-10 C
TWO-PHASE AQUEOUS INCLUSION ON A HEATING RUN

25 C  150 C  250 C

25 C  250 C  350 C
An animated introduction to fluid inclusions

by

A H Rankin
School of Earth Sciences and Geography
Kingston University, Surrey KT1 2EE

http://www.kingston.ac.uk/esg
The following slides illustrate how primary (P) and secondary (S) inclusions develop during crystal growth and healing of secondary fractures.

Some animation has been added
To see changes hit return

Aqueous solution

Crystal
Formation of Primary and Secondary inclusions

**primary** inclusions form during primary crystal growth often along growth zones.

**secondary** inclusions form after primary growth, usually along healed micro-fractures.
Inclusions formed from trapping of homogeneous aqueous fluid will develop vapour bubbles on cooling due to differential thermal contraction of fluid and host mineral.

The next slide shows groups of two phase (vapour-liquid) primary and secondary inclusions with distinctly different vapour-liquid ratios, indicative of different PT trapping conditions.
The following slides show the homogenisation of liquid and vapour in a two phase aqueous inclusion during laboratory heating. The temperature at which this occurs is the *Homogenisation temperature* (Th) The bubble returns on cooling.
Temperature of Homogenisation Th

350°C

25°C
0°C
-100°C
The next slides show the freezing and subsequent melting of a two phase aqueous inclusion. Note the first and last melting temperatures which tell us about composition.
First melting temperature - $T_{fm}$
Last ice melting temperature $T_m(\text{ice})$
The next slides show more complex aqueous inclusions with daughter minerals, liquid carbon dioxide and liquid hydrocarbons (oil)
The next slides illustrate the use of heating and freezing data for simple two phase aqueous inclusions.

Heating and freezing data (Th, Tfm, Tmice) are interpreted in terms of the simple H₂O and NaCl-H₂O systems.
Principle of fluid inclusion geothermometry

PVT diagram for pure water
Consider an inclusion trapped at a given temperature and pressure (T_t, P_t)
On cooling, the inclusion follows an isochoric PT path until it meets the L=V curve.
Beyond this point the inclusion cools along the L=V curve and a vapour bubble nucleates.
Continued cooling results in further shrinkage of liquid and growth of the vapour bubble.
On heating along the V/L curve, the liquid expands and the bubble shrinks.

- Isochore (g/cc)
- Critical point

Pressure KBar

Liquid

Vapour

Pt

L-V curve

Tt

Temp °C

0 0.5 1.0

50 150 350
Until the bubble disappears at the homogenisation temperature (Th)
The point Th uniquely defines the isochore along which the inclusions originally cooled.
With continued heating the inclusion follows the original isochore.
If Pt is known, or estimated, the trapping temperature (Tt) can be determined.
The difference between Th and Tt is known as the *Pressure Correction*.
The bubble reappears on cooling and Th can be re-determined
Phase diagram for NaCl-H2O showing stability fields for halite, hydrohalite, liquid and vapour.
An inclusion with 10 wt.% solution cooled below 0°C does not form ice because of metastability.
Rapid cooling below the eutectic temperature (Te) is usually needed before the inclusion freezes.

- Ice + NaCl·2H₂O + V
- Ice + L + V
- L + V
- NaCl + L + V
- NaCl·2H₂O + L + V

Temperature °C

Weight % NaCl
On heating first melting (Tfm) occurs at -20.8 °C (Te), evident by “unlocking” of the vapour bubble.

- Ice + NaCl.2H₂O + V
- Ice + L + V
- L + V
- NaCl + L + V
- NaCl.2H₂O + L + V
- 0.1 °C
- -20.8 °C

Temperature °C

Weight % NaCl

Diagram showing the phase changes and melting points of ice and NaCl solutions.
Continued heating results in the melting of the last ice crystal (Tm\textsubscript{ice}) at -6°C.
Continued heating results in the melting of the last ice crystal (Tm_ice) at -6°C.

Temperature °C

-50 0 25

Weight % NaCl

0 10 20 30

Ice + NaCl_2H_2O + V

Ice + L + V

NaCl + L + V

NaCl_2H_2O + L + V

-20.8°C 0.1°C
Sedimentary/Diagenetic Environments

Range of Fluid Trapping Scenarios:

Fluid Inclusion Mineral Hosts
Fluid Salinities and Temperatures

Fig. 1. Schematic representation of environments in sedimentary systems with characteristic of fluids applicable to fluid inclusion work (a) and diagenetic processes important for each environment (b).
<table>
<thead>
<tr>
<th>Gradient Type</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geothermal gradient</td>
<td>20-50°/km burial</td>
</tr>
<tr>
<td>Hydrostatic gradient</td>
<td>100 bars/km burial</td>
</tr>
<tr>
<td>Lithostatic gradient</td>
<td>226 bars/km burial</td>
</tr>
</tbody>
</table>

Table 1. Commonly encountered conditions in diagenesis
<table>
<thead>
<tr>
<th>Dissolved Species</th>
<th>Eutectic temperature</th>
<th>Eutectic Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>-20.8</td>
<td>23.3% NaCl</td>
</tr>
<tr>
<td>KCl</td>
<td>-10.6</td>
<td>19.7% KCl</td>
</tr>
<tr>
<td>CaCl₂</td>
<td>-49.8</td>
<td>30.2% CaCl₂</td>
</tr>
<tr>
<td>MgCl₂</td>
<td>-33.6</td>
<td>21.0% MgCl₂</td>
</tr>
<tr>
<td>NaCl-KCl</td>
<td>-22.9</td>
<td>20.1% NaCl, 5.81% KCl</td>
</tr>
<tr>
<td>NaCl-CaCl₂</td>
<td>-52.0</td>
<td>1.8% NaCl, 5.81% CaCl₂</td>
</tr>
<tr>
<td>NaCl-MgCl₂</td>
<td>-35</td>
<td>1.56% NaCl, 22.74% MgCl₂</td>
</tr>
</tbody>
</table>

Table 5. Characteristic eutectic points for salts in inclusions
Isochore for aqueous fluid with ~7wt% NaCl

ISOCHORE

Density = 1.001 gm/cc

ISOCHORE = A line of constant density in P-T space
Phase envelopes for a series of generic oil compositions

![Graph showing phase envelopes and compositions with specific data points and labels such as C1, C2, C3, C4, C5, C6, C7x, and P and T of Trapping. The graph includes isochores and phase regions labeled as 1 Phase Petroleum Fluid, Volatile Oil, Liquid + Vapor, and Gas Condensate.]