

Chapter 4: Coring, Preservation and Handling

4.1 Introduction

Large financial resources are invested in RCAL and SCAL core analysis programmes, and a wide range of accurate experimental determinations can be carried out. However, cores are expensive to obtain and represent a very dilute sampling of the reservoir rock. It is clear that the samples used in such studies should be as representative as possible of the reservoir rock at depth if the final data is to be credible, and an efficient use of the financial resources devoted to them. Samples of the reservoir rock and the fluids they contain can be, and are commonly, altered by the process of obtaining them (coring, recovery, wellsite handling, shipment, storage, and preparation for experimentation). This chapter gives an overview of the alteration processes that may be at work, together with some of the techniques available to reduce alteration, and preserve the rock and fluid properties. The choice of core preparation techniques is increasingly being made by using pre-screening information on the preserved core. This approach is highly recommended.

4.2 The Coring Process

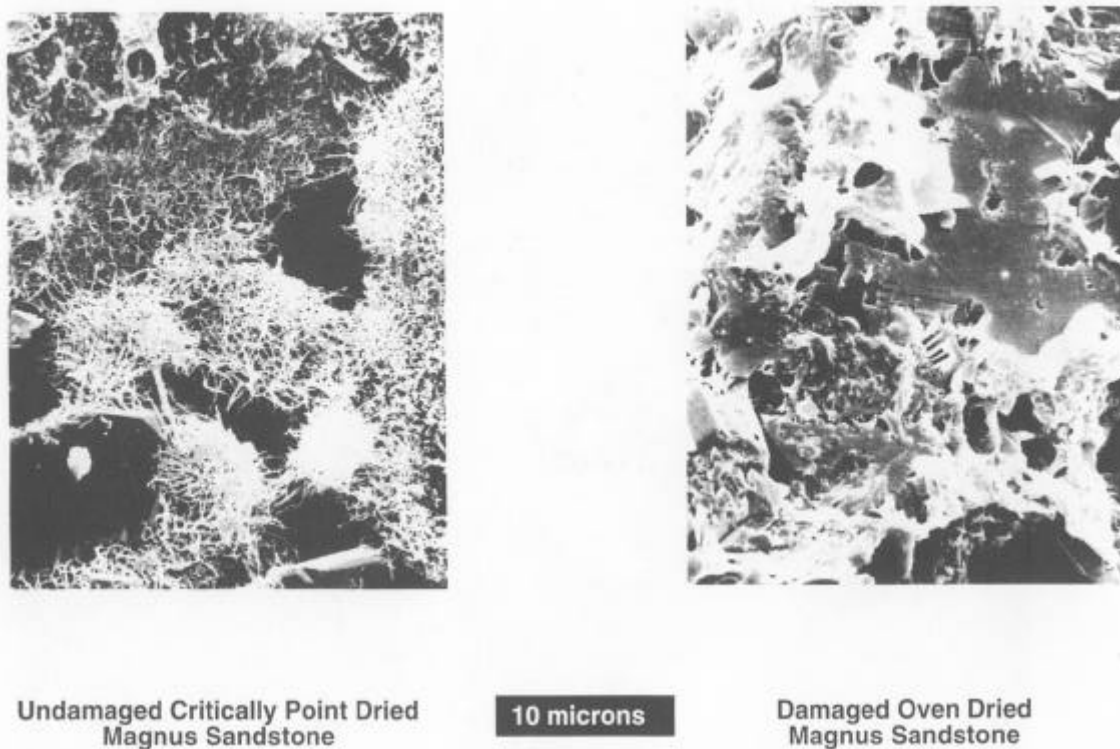
Reservoir rock undergoes changes during the coring process and on storage before reaching the laboratory. The changes which occur are shown in Figure 4.1. Some of the changes are reversible whilst others are irreversible but preventable. In most cases it is possible to leave all or part of the core in a usable state. It is essential to use preserved core for certain SCAL tests and for meaningful assessment of routine data.

Figure 4.1 Changes to Core Properties during Coring

RESERVOIR	AS RECEIVED	CHANGE
Reservoir Pressure	Ambient Pressure	Stress Relief Fluid and Rock Expansion
Swi	Soi	So Reduced
Reservoir Temperature	Ambient	Wax Precipitation
Formation Brine	Mud Filtrate	Salinity
Anaerobic	Aerobic	Oxidation

Drilling of the core is invariably carried out at very high bottom hole pressure differentials, thus the core is effectively water-flooded with mud filtrate, and the original contents partly displaced. The outer surface of the core will be invaded by *mud particles*; the depth of invasion being dependent upon permeability. This zone should be avoided when sampling. The rest of the core will have had its original hydrocarbon content, and formation water displaced by *mud filtrate*; the extent depending upon the core permeability and original fluid saturations. These changes are not always harmful as the core can usually be restored in the laboratory. More important changes can occur if the rock contains minerals sensitive to water salinity. For example, contact with low salinity water can mobilise poorly adhered clay particles, giving a small possibility that core can arrive in the laboratory with mobilised fines, which are not significantly mobile in the reservoir. In a similar fashion the wetting characteristics of the rock may be altered by surfactant mud additives. These changes are usually unavoidable but if formations are known to be particularly sensitive, it may be possible to modify mud composition and reduce overpressure to minimise damage. For complete preservation of wettability on cores above the transition zone, coring with lease crude is necessary. Water saturation may then also be retained intact, allowing better estimation of initial reservoir oil saturation. For transition and water zone a bland mud formulation will do the least harm to original rock properties.

Figure 4.2 The Sensitivity of Clay to Conventional Drying

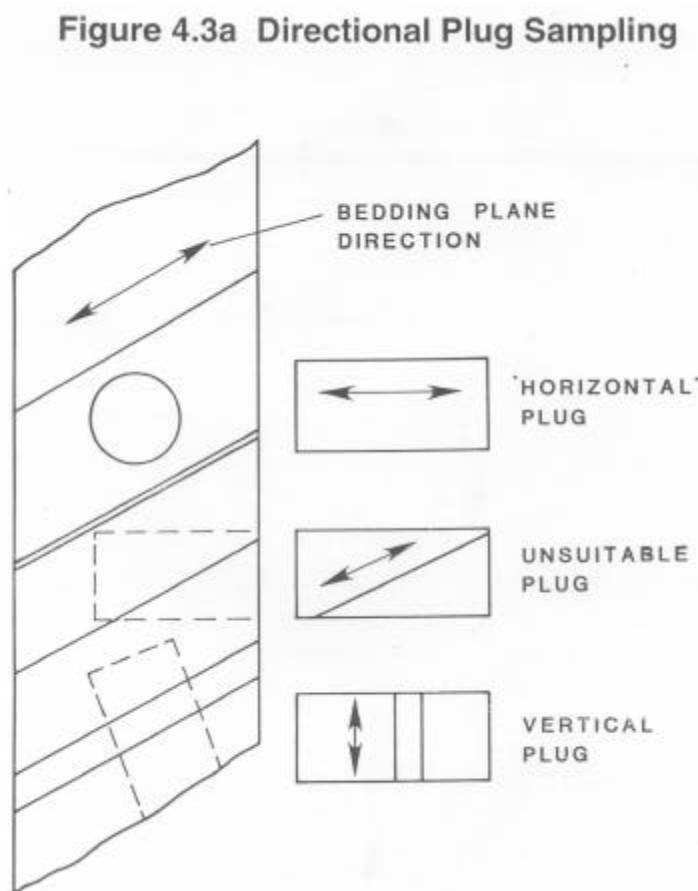


Drying can be the worst that can happen to core after removal from the barrel. If interface sensitive clays, e.g., fibrous illite are present they can be *irreparably* damaged by drying (Figure 4.2) and any permeability measurements made on such core will be valueless. Thus it

is necessary to preserve some core in the state that it leaves the barrel either by immersion in simulated formation brine or by wrapping in foil and wax. The latter technique is the minimum required for samples intended for wettability measurements, but for straightforward assessment of water zone permeabilities immersion in brine is adequate. The necessity for preserved core will be more fully covered under relevant sections below.

4.3 Plug Sampling and Cleaning (Unpreserved Core)

Standard techniques are applied unless the core is very heterogeneous or likely to be damaged by routine cleaning methods.

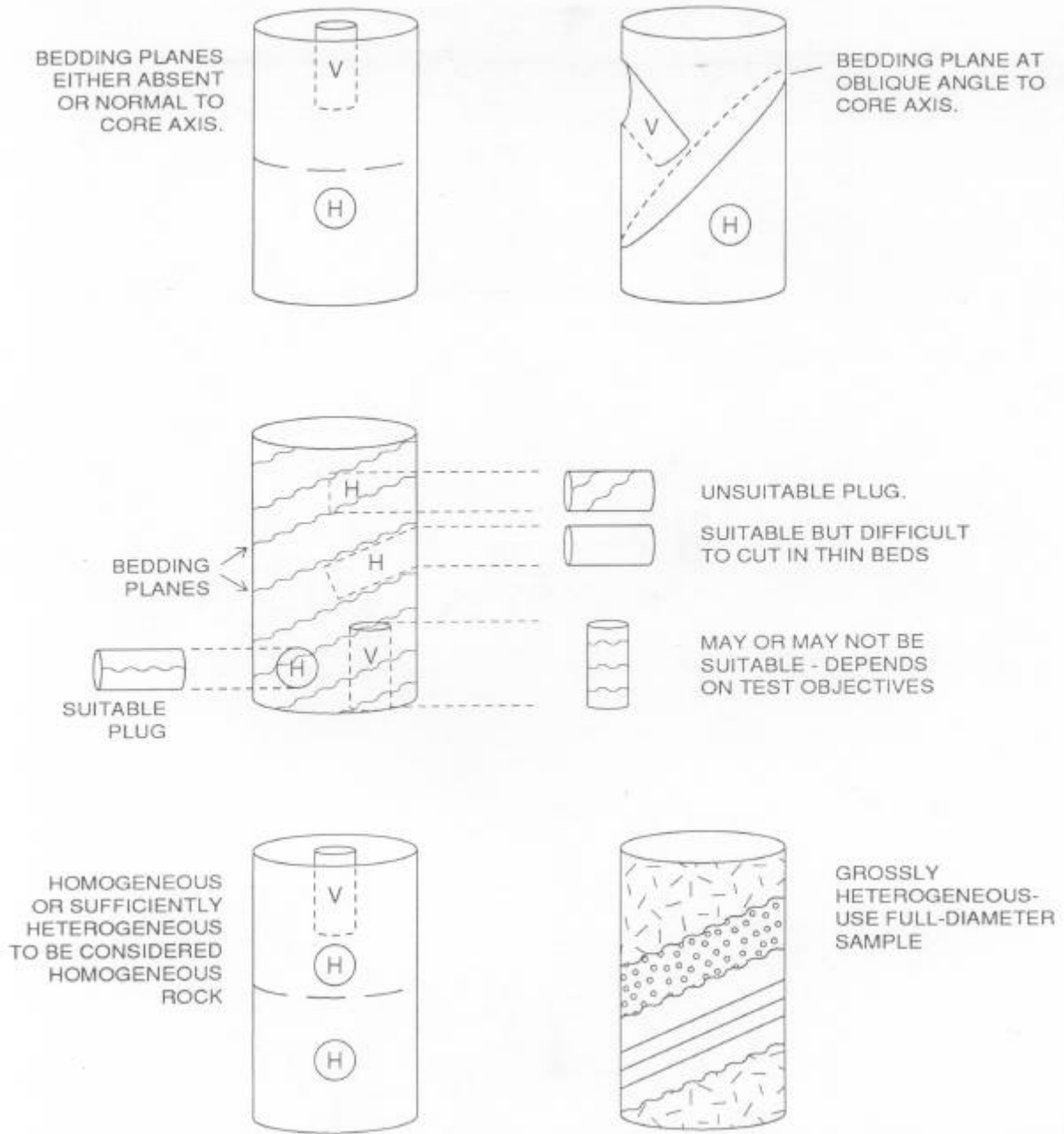


One or one and a half inch diameter sample plugs are drilled and trimmed to between two and three inches long with simulated formation brine as lubricant. If the composition of formation brine is unknown, a five percent sodium chloride brine is used. Plugs are taken at regular intervals (often every 25 cm), parallel to bedding planes for horizontal permeability (see Figure 4.3a). Further plugs normal to the bedding plane are taken if required for vertical permeability. The sampling interval can either, be increased, if the core is from a formation known to be homogeneous; or varied if the core contains thin shaly bands making it difficult to produce intact plugs. Thin shaly bands are avoided unless frequent and representative. Figure 4.3b analyses the suitability of core plugs for homogeneous, thickly bedded and thinly bedded whole core.

Tests may also be carried out on full diameter core samples. This is necessary if plug sized samples do not contain a representative pore size spectrum. Fractures, vugs (very large pores) and stylolytes are typical structural features which necessitate measurement on full diameter (whole core) samples. The measurements made are the same as for plug samples, but a special core holder is necessary if horizontal permeabilities are required.

Plugs are cleaned by alternate extraction with hot toluene and methanol in Soxhlet extractors (Figure 4.4a and 4.4b) until no further discolouration of solvent occurs. This may take from a few, to several hundred hours depending upon permeability. Low permeability plugs are seldom completely free of residual brine and oil at this stage. Complete removal of residual fluids can only be achieved by prolonged Soxhlet extraction. Cores can also be cleaned by

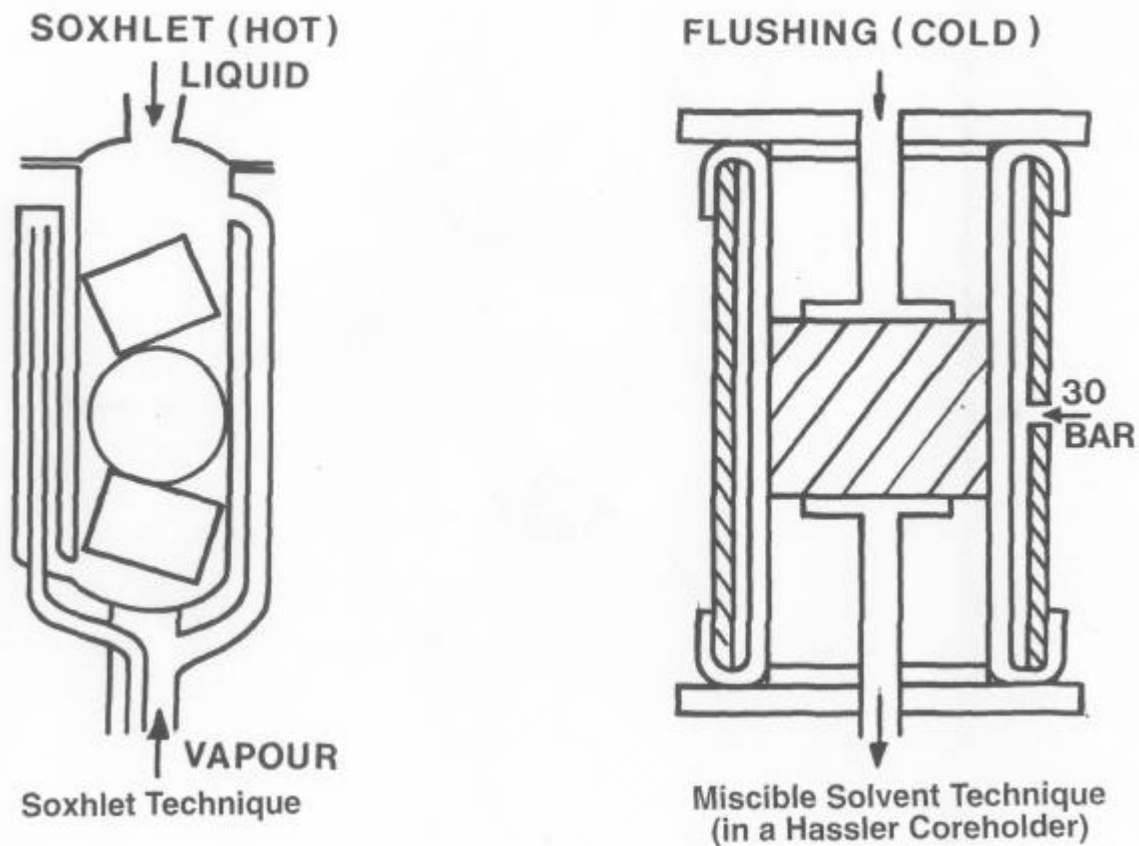
Figure 4.3b Suitability of Core Plugs



NOTE: WELL ORIENTATION MUST BE SPECIFIED WITH RESPECT TO PLUG ORIENTATION

flushing the core with alternate miscible solvents (e.g. toluene (for the oil phase) and methanol (for the water phase)) done hot or cold in a Hassler coreholder (Figure 4.4a; also see section 4.5). Both the aqueous (methanol) and oleic (toluene) cleaning phases exiting the rock can be bulked and submitted for analysis of the amount of water and individual hydrocarbons present.

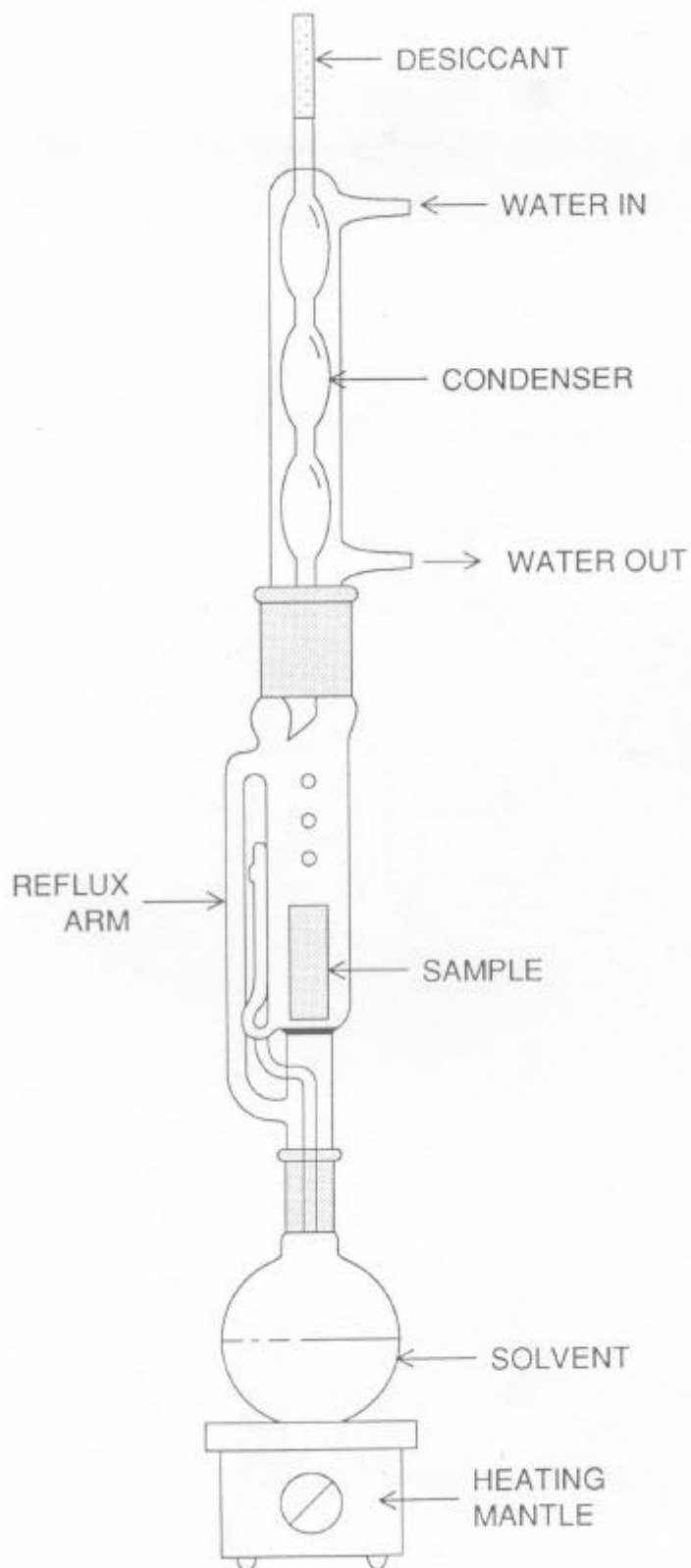
Figure 4.4a Core Plug Cleaning



Plugs are then dried to constant weight in a humidity controlled oven at 60°C, 40% relative humidity. Humidity controlled drying assists in restoring clays to nearer their reservoir state, and may assist in preventing any further damage. However, the Klinkenberg corrected equivalent liquid permeability from this type of drying process may still be larger than the actual brine permeability due to the destruction of the clay texture.

If samples of plugs containing clays that are sensitive to drying are required for SEM analysis (e.g. Figure 4.2), then a sample of the core with the original fluid contents must be critical point dried. Ordinary drying destroys fine clay minerals because the interfacial forces associated with the retreating liquid-vapour interface are high enough to mash the clay structure. Critical point drying involves keeping a small sample of the core at pressure and temperature conditions of the critical point of the fluids. The fluids will then be evaporated from the sample without a liquid-vapour interface, which avoids destroying the fine clay structure. This is an expensive operation because it can take many days to perform on even the smallest sample chip. Consequently, it is almost never carried out for core plugs.

Figure 4.4b Soxhlet Cleaning Detail



SOXHLET EXTRACTOR

4.4 Core and Plug Preservation (SCAL Techniques)

Preserved core is almost always required for one or more of the following reasons:

- (i) Wettability determinations.
- (ii) Prevention of drying of interface sensitive clays.
- (iii) Maintenance of fluid saturations as received at surface.
- (iv) Other SCAL where drying is not desirable.
- (v) Unconsolidated or relatively uncompacted samples that exhibit strong porosity and permeability reductions with overburden stress.

Several methods of preservation are currently available and a choice can be made if the requirement for preserved core is specified. The methods are:

Under simulated formation brine or kerosene, for water and oil zone cores respectively. Cores are either kept under simulated formation brine in polymer containers with an airtight seal at ambient pressure (certain types of spaghetti jars are good for this); see Figure 4.5.

Wax coated, for all SCAL purposes and especially wettability and residual oil saturations. This technique, also called 'seal-peel', is widely used, and involves wrapping the core in layers of plastic and aluminium foil before being dipped in wax. Cores preserved in this way at the well site can be safely stored for moderately long periods and then be used for almost all SCAL purposes (Figure 4.5).

In deoxygenated formation brine or kerosene, for wettability measurements. Samples are kept in anaerobic jars which can be pressurised to 30 psi (Figure 4.5). The freshly cut core pieces are placed in the jars under deaerated simulated formation brine or kerosene, and the jars are then sealed. The remaining air is then purged with nitrogen, which is then raised to 30 psi pressure. The samples are then preserved under reservoir fluid and a blanket of inert gas. Providing that the pressure is maintained, the samples may be stored in this state for long periods.

Wrapped in cling film and frozen in solid CO₂ for fluid saturation measurements. This is used for unconsolidated

Figure 4.5 Core Plug Preservation Techniques



core. The samples are cooled using liquid nitrogen and are loaded into special containers. The containers can be transported packed in solid CO₂, and stored in special freezers. Plugs can be cut from the core using liquid nitrogen as the cutting fluid, and the plugs are then immediately loaded into special coreholders again, and stored frozen. The sample is thawed out and tested without being removed from the special coreholders in which they were initially loaded.

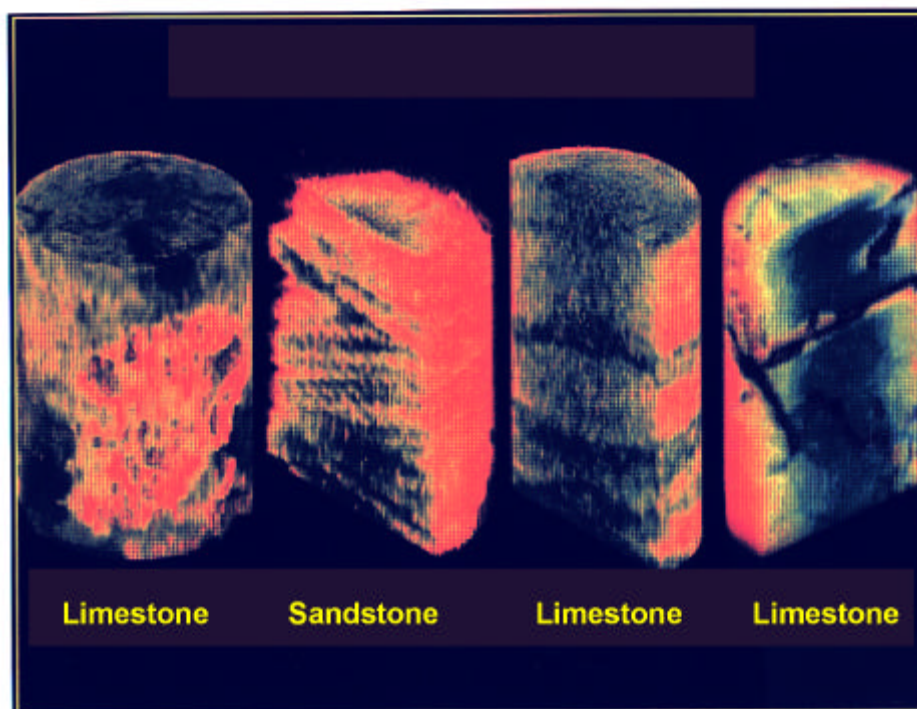
4.5 Cleaning and Treatment of Preserved Core

Treatment of preserved core for the tests mentioned above will be reviewed with the appropriate tests; but in general, sample plugs are drilled and trimmed using deoxygenated formation brine and stored under deoxygenated, depolarised kerosene or brine before testing.

There are several methods of cleaning core. The actual method used will depend upon the properties of the core. Usually the optimum method will be clear from *pre-screening information* on the core. Pre-screening measurements include:

- Core description
- Core lithology
- Assessment of consolidation
- SEM analysis of mineralogy and pore structure
- Petrographic analysis of mineralogy and pore structure
- XRD/XRF analysis for bulk and clay mineralogies
- CT scanning to assess core heterogeneities, Figure 4.6 (cross-bedding, and fractures)

Figure 4.6 CT Scan Pre-screening to Locate Cross-bedding and Fractures



This information is designed to identify possible problems with; (i) unconsolidated core, (ii) clay sensitivity, (iii) stress sensitivity, (iv) core mineralogical heterogeneity, (v) core structural heterogeneity (e.g. fractures, vugs, fossils, and cross-bedding).

The commoner specialist cleaning methods include:

- (i) Critical point drying
- (ii) Cold miscible solvent flushing
- (iii) Hot miscible solvent flushing
- (iv) Direct fluid replacement (oil for oil and brine for brine)

Core cleaning, where appropriate, is most often carried out using miscible solvent flushing techniques. The core if confined in a Hassler holder (Figure 4.4a) and cold solvent flowed through it. Cleaning is usually complete after flowing three 200 ml alternating portions each of methanol and toluene. Under certain circumstances only one portion of each solvent will be used, although it is commoner to use at least three portions of each. This is applied to cores known to contain mobile fines or where it is necessary to retain wettability modifying crude oil components in their existing state. In some circumstances the evolved solvents need to be quantitatively tested using chemical techniques for the water content, and the oil content and composition. In this case special dry methanol is used, and the toluene is replaced with a more efficient solvent such as CS₂ (very dangerous) or dichloromethane.

4.6 Unconsolidated Core

Unconsolidated core gives rise to particular problems in coring, storage, handling and plugging. Its extremely friable nature means that any rough handling damages the pore structure irreversibly, and samples can turn into a pile of mud in your hand. The most common method of handling, shipping, storage, and plugging this type of core is in a frozen state. The core is frozen with liquid nitrogen or dry ice as soon as it emerges from the coring barrel. It is then placed in a special core holder for the relevant experiment to be carried out. Thawing inside the coreholder, prior to the experiment is only carried out after the sample has been fully supported with the relevant applied confining pressures (see above).

4.7 Water Analysis

It is possible to obtain the initial water saturation and water composition from preserved whole core and core plugs by extracting the water. This is done by the Dean and Stark method. Figure 4.7 shows the Dean and Stark apparatus. The preserved sample is placed in a paper thimble in the large glass container and fluxed with hot solvent. The water evaporates, is carried by the solvent vapours into the long straight condenser in the top of the apparatus, cools, condenses and is trapped in the graduated part of the apparatus. The water saturation can be calculated by using the volume of the evolved water and a measurement of the porosity of the rock sample after the extraction process. The composition of the evolved fluids can also be analysed chemically, however, the water compositions more commonly used in SCAL applications derive from wireline formation testing.

Figure 4.7 Dean and Stark Water Extractor

