

ROCK MATRIX AND FRACTURE ANALYSIS OF FLOW IN
WESTERN TIGHT GAS SANDS

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In this quarterly we report progress in the following areas of advanced core analysis of low permeability gas sands.

- a) Surface Area Measurements
- b) Helium Porosimetry
- c) Comparison of Permeabilities for Preserved Cores and for Cores Subjected to Drying
- d) Pore Space and Fractures as Related to Diagenesis of Multiwell Sandstones
- e) Imbibition Behavior of Tight Sands

a) Surface Area Measurements

A relationship was noted previously¹ between surface area per unit pore volume and the amount of water held at surfaces by adsorption or capillarity in very small pores, as revealed by water vapor adsorption/desorption measurements. This has prompted a more detailed look at the significance of surface area as determined by BET measurements for tight gas core samples since these may provide a useful indication of how much pore volume is in the form of microporosity. Fig. 1 shows the relationship between outgassing temperature and measured surface area for several crushed samples from MWX1 42-25. These measurements are made by a single point BET technique. This is a rapid method of obtaining approximate values for surface area based on nitrogen desorption at one partial pressure.

These measurements were made with a partial N_2 pressure of 0.3 using one to two gram, crushed core samples. Samples are outgassed for a minimum of 15

minutes at the lowest temperature in a stream of the 30% nitrogen in helium mixture. Successive cycles of adsorption and desorption show little or no change in surface area, and baseline measurements of gas compositions are very stable, both indicating that the outgassing process is effectively completed.

Increase in outgassing temperature, however, does have a major effect on surface area measurements. Some samples show a steady increase in surface area with temperature; others increase more dramatically as the temperature exceeds 150°C. At temperatures in the range of about 150 to 300°C, the duration of heating becomes an important factor. If the sample is then left at ambient temperature under the nitrogen/helium atmosphere, the surface area decreases with time. A systematic investigation of these effects is now underway.

Use of the single point method presupposes that the nitrogen partial pressure of nitrogen used for the measurement is in the linear portion of the BET curve and that the intercept of that curve is not far from zero. These assumptions have been validated for a typical tight gas sample by a ten point adsorption/desorption BET analysis performed by Quantachrome. Fig. 2 shows the BET plot obtained from adsorption for a portion of rock taken from MWX1 42-25 which was passed through a jaw crusher and then a roll crusher. Linearity does extend to the 0.3 partial pressure, and closure of the adsorption/desorption isotherms is at a higher partial pressure, around 0.4, as shown in Fig. 3. Assuming the intercept to be zero and calculating the slope based on the 0.3 partial pressure measurement would result in a value for surface area of 2.7 instead of the multipoint value of 2.9 M²/g.

The observed single point surface areas measured in our laboratories for portions of the crushed MWX 42-25 sample were still lower (2.3-2.5 M²/g) than that calculated from the Quantachrome results, and vary somewhat from one sample to another. Results are also shown in Fig. 1 for Sample 1 which had been crushed using a mortar and pestle. The discrepancy between surface areas measured for Samples 2a and 2b and the Quantachrome results may be due to outgassing conditions. Quantachrome outgassed at 110°C in a vacuum, whereas the single point samples were outgassed under the N₂-He mixture. Decreasing pressure may have the same effect as increasing temperature, perhaps driving off either water or organic compounds which are retained at 110°C and ambient pressure. This observation has important implications for core handling before laboratory measurements and will be investigated further. Overall, the Quantachrome results confirm that the single point method gives a reasonable approximation of surface area for tight gas samples, while pointing out the necessity to consider the effects of sample treatment on this and other measurements.

b) Helium Porosimetry

All the porosities reported in previous summaries of work on this project have been obtained from gravimetric measurements. The core is weighed dry and reweighed fully saturated with a fluid of known density to obtain a value for pore volume which can be compared with the bulk volume calculated from measurements of external dimensions. This procedure is very reproducible, provided the dry core state is well-defined.

In the case of the tight gas sand samples, however, there is considerable uncertainty as to choice of satisfactory conditions for the dry core state. Oven drying to some reproducible weight may cause irreversible changes in core flow properties. Presently recommended core treatment of samples with high water adsorption due to clay involves equilibration of samples at some controlled relative humidity at which water saturation in these samples is not negligible.^{2,3} Porosities calculated from these initial weights may vary by several porosity percent from those obtained with more thoroughly dried samples, and since total porosity is often less than 10%, these differences may be as much as 20 or 25% for estimates of porosity for a given sample.

It is useful, therefore, to have an alternative method for establishing porosity of the tight gas samples, and helium porosimetry provides a simple, quick, yet accurate porosity measurement. Pressure measurements can be reproduced to within $\Delta 0.05$ psi which for a typical measurement on a 1" by 1" tight gas core sample represents a change in volume of less than .015 ml or roughly 1.5% pore volume. Investigation of differences between the helium results obtained with a Temco helium porosimeter and those obtained previously by gravimetric methods is being continued.

c) Comparison of Permeabilities for Preserved Cores and for Cores
Subjected to Drying

MWX3 42-4, a preserved core sample (not allowed to dry after recovery from the formation), has been selected to investigate the effect of drying procedure on permeability. One core plug was cut, and water saturation was increased to 100% using deaerated distilled water.

After measuring absolute water permeability, the core was allowed to dry by evaporation until a desired water saturation was established. Since the sample pore volume was unknown at this stage of the experiment, we assumed a porosity of 7% for approximate calculation of water saturations. Permeabilities to nitrogen gas were measured at approximately 60%, 45%, 30% and 15% water saturations. Measurements were for first unloading, with Klinkenberg permeabilities being determined at 5000 and 500 psi confining pressures.

As expected, relative permeability decreased with increase in confining pressure and water saturation (Figs. 4 and 5). Increase in sensitivity of relative permeability to confining pressure is illustrated by the plot of K_{rg} vs. P with saturation as parameter (Fig. 5) and in plots of K_{rg} vs. S_w (Fig. 6).

Following these measurements, the core plug was dried in an oven at 110°C until constant weight was obtained. This weight was used as a basis for calculation of water saturations. Gas permeability was measured for first unloading with K_{∞} values determined at 5000 and 500 psi overburden pressures. The plug was then resaturated with deaerated distilled water, and redeterminations of relative permeabilities to gas were attempted.

After making absolute gas permeability measurements on the dry core, the plug was resaturated with distilled water. After absolute water permeability measurements had been made, the plug was found to have fractured across its length close to one end. There was enough core left to continue making relative permeability measurements. A water saturation of 61.33% was established by evaporation, and relative permeability to gas was measured. Results

for the preserved and oven dried core are compared in Fig. 7. Drying appeared to have caused an increase in matrix permeability and range of confining pressure over which permeability was measurable at this comparatively high water saturation. However, the plug was again found to be fractured, this time at its center. Such fracturing of rock matrix is unusual in our experience. Therefore, we regard results obtained to date for 42-4 as strictly preliminary. Two more plugs have been cut from 42-4 for further measurements on the effects of drying preserved cores.

d) Pore Space and Fractures as Related to Diagenesis of Multiwell Sandstones

A generalized diagenetic history of Multiwell sandstones was derived using the following methods: 1) petrographic examination of over 500 thin sections, 2) preparation of polystyrene pore casts from selected sandstones, and 3) examination and photography of rock chips and pore casts with the scanning electron microscope. Particular attention was given to the nature and distribution of porosity (both primary and secondary) and fractures. Diagenetic characterization of Multiwell sandstones is still in progress; Fig. 8 is a summary of preliminary results.

Cements

Calcite cement was the first to form. It developed early in the burial history of the host sandstones, as shown by the loosely packed nature of calcite-cemented detrital grains. Diagenetic calcite commonly occurs as large, poikilotopic crystals; such cements are typical of early formed concretions and phreatic carbonates in many clastic units. Calcite cements are best developed

in non-fluvial rocks, presumably due to availability of seawater to the coastal and marine sediments. Detrital feldspar grains are commonly replaced by calcite, especially in marine sandstones.

Syntaxial quartz overgrowths post-date calcite cements in the Multiwell sandstones. Diagenetic quartz occurs in intergranular areas that were not previously filled by calcite. Quartz-cemented sandstones typically show more evidence of compaction than areas cemented by calcite, indicating that diagenetic quartz formed relatively late in the burial history.

Clays were the last major cements to form in the Multiwell sandstones. These clays consist primarily of kaolinite, chlorite, and illite and occlude, to varying degrees, both primary and secondary pores.

Porosity

Both primary and secondary pores occur in Multiwell sandstones, although secondary porosity dominates. Dissolution porosity in detrital feldspars is mostly restricted to fluvial sandstones, where calcitization of unstable feldspar grains during calcite cementation was incomplete. Secondary porosity after calcite is present in both fluvial and marine sandstones. Significant amounts of primary porosity were noted in only a few fluvial sandstones.

Feldspar dissolution occurred after quartz cementation. Solution porosity in feldspars is best developed in fluvial sandstones where calcitization of feldspars was less complete than in marine deposits. Bituminous residues are locally present within dissolved feldspars of some fluvial sandstones, but not

within secondary porosity after calcite. These relationships constrain the relative timing of hydrocarbon migration, and indicate that dissolution of feldspar and calcite did not occur simultaneously.

Fractures

Preliminary assessment of the nature and distribution of fractures has been accomplished through examination of over 500 thin sections from Multiwell sandstones. Fractures are very rare in most thin sections, probably because of a sampling bias toward homogeneous plugs that are representative of "matrix" characteristics. We are presently searching cores for fractures. Thin sections will be prepared from fractured sandstones and then examined for evidence of diagenesis and remaining porosity.

At least two, and possibly four, episodes of natural fracturing are represented in the thin sections. Calcite-filled fractures are the most abundant type, and appear to have formed contemporaneously with precipitation of intergranular calcite cements. No remaining thin-section porosity was noted in these fractures, although vuggy porosity associated with large calcite-filled fractures has been noted in some cores.⁴

A second generation of fractures formed after completion of calcite cementation, but prior to development of quartz overgrowths. These fractures are rare (only two examples were noted), and are filled by diagenetic quartz. Residual porosity was noted in one of the quartz-filled fractures.

A possible third generation of fractures is recorded in a few fluvial sandstones. These fractures contain bituminous residue, but the possibility remains that this residue was mobilized into artificial fractures during epoxy injection of the samples. Fractures containing no diagenetic products are common and represent a possible fourth episode of fracturing. However, it seems likely that most or all of these fractures were produced artificially, during coring or thin-section preparation.

e) Imbibition Behavior of Tight Sands

Imbibition rates into low permeability gas sands provide a further approach to investigating flow properties, effective pore size for flow, and gas entrapment in tight gas sands. Imbibition of fracturing fluid into the fracture zone and subsequent clean up (reversal of imbibition) are of special interest in attempts to improve the productivity of tight gas sands.

An apparatus has been constructed which permits imbibition rates into tight sands to be measured at chosen levels of overburden pressure. Results to date show, as expected, that cores with permeabilities which are pressure-sensitive also show a corresponding sensitivity of imbibition rate to confining pressure. The measured relationships between time and amount of liquid imbibed suggest that, if the displacement is close to piston-like, capillary imbibition pressures are determined by interface curvatures in the relatively large solution pores rather than the much narrower sheet pores. (On average, the sheet pores are only a few tenths of microns in width and are believed to control the permeability of tight sands.⁵) Further results are being obtained, and details will be presented in the next quarterly report.

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MWX1 42-25

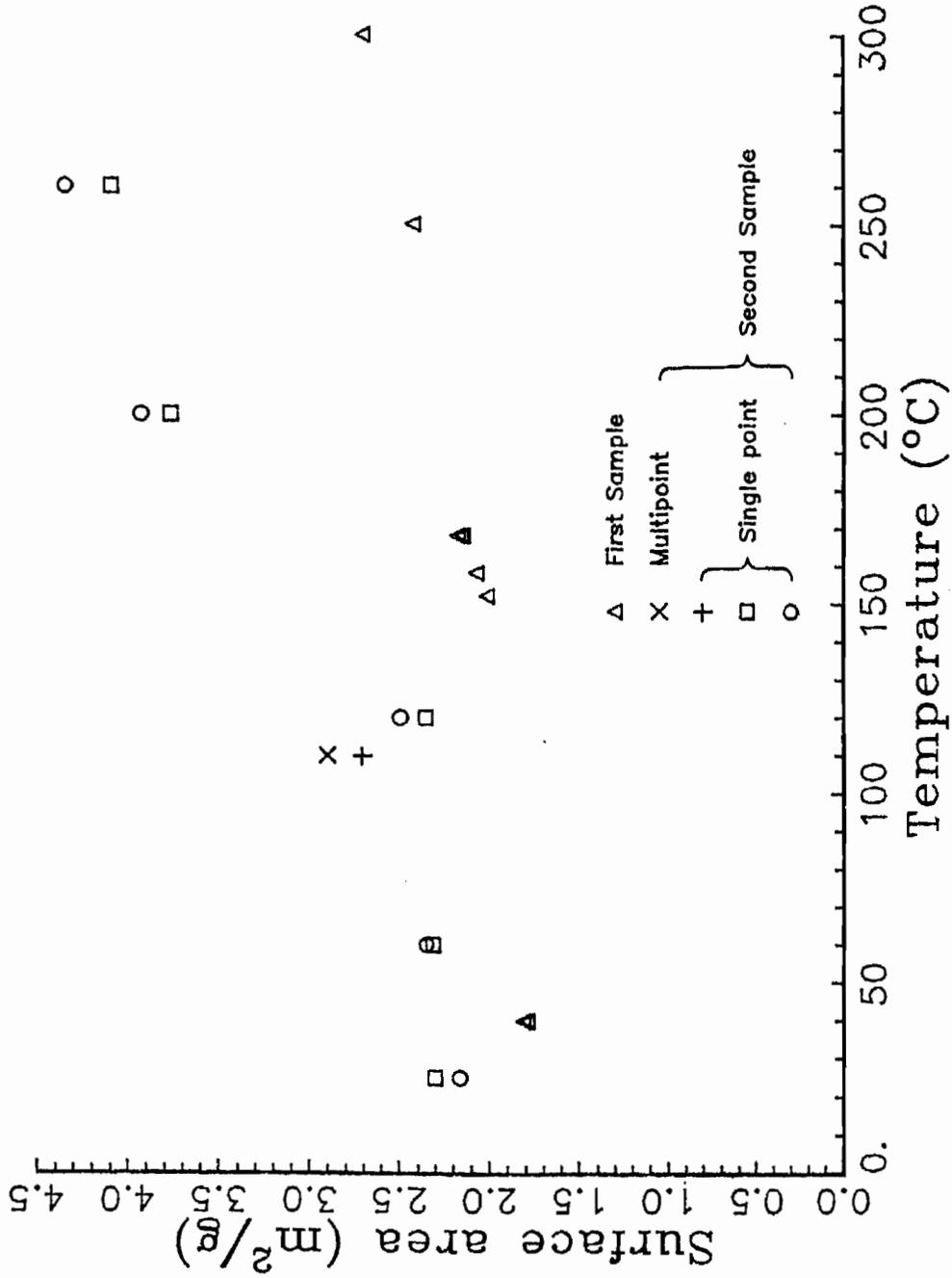


Figure 1. Surface area versus outgassing temperature for crushed sample of MWX1 42-25.

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MWX1 42-25

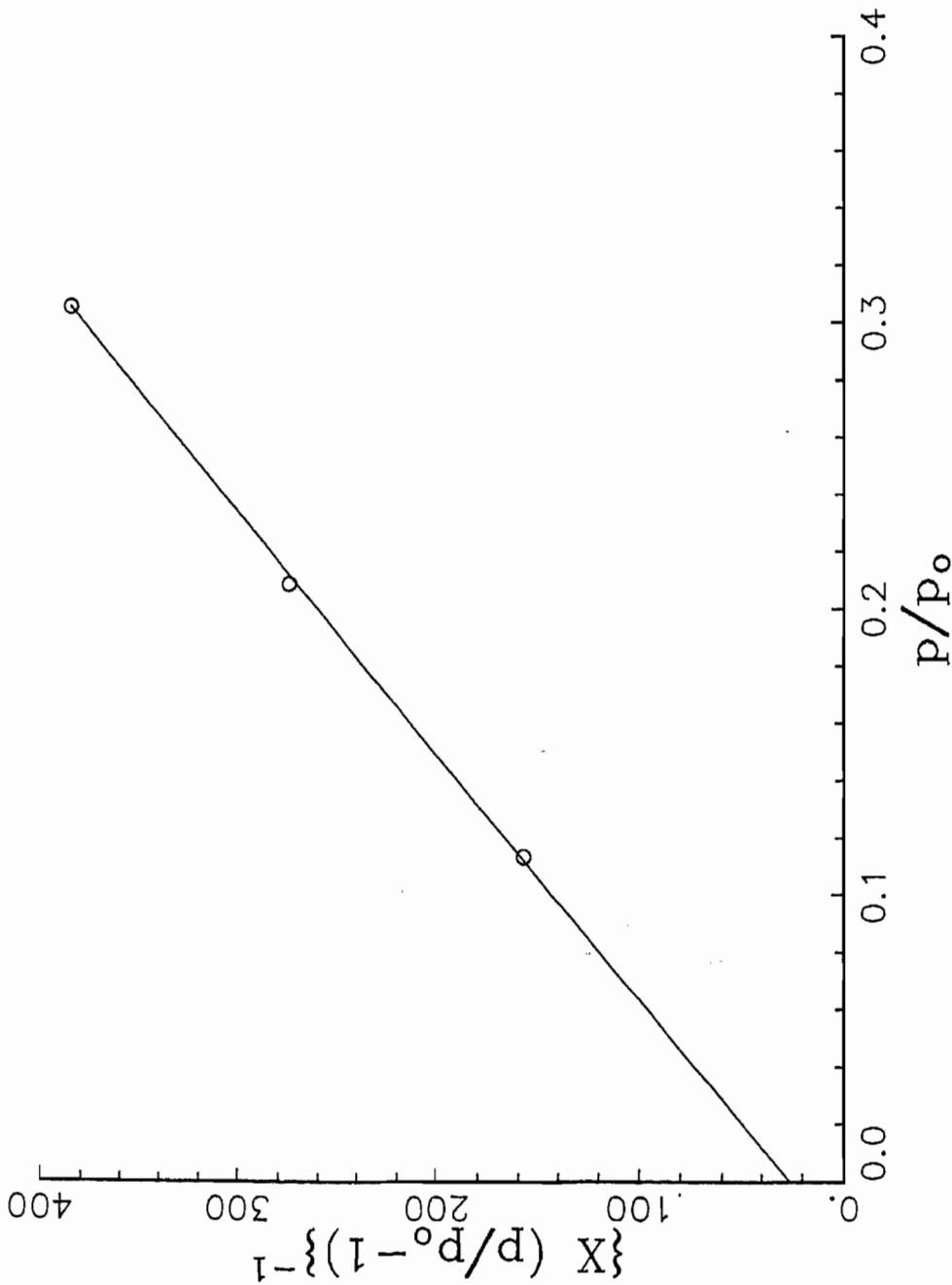


Figure 2. Relationship between nitrogen adsorption and partial pressure (BET plot).

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MWX1 42-25

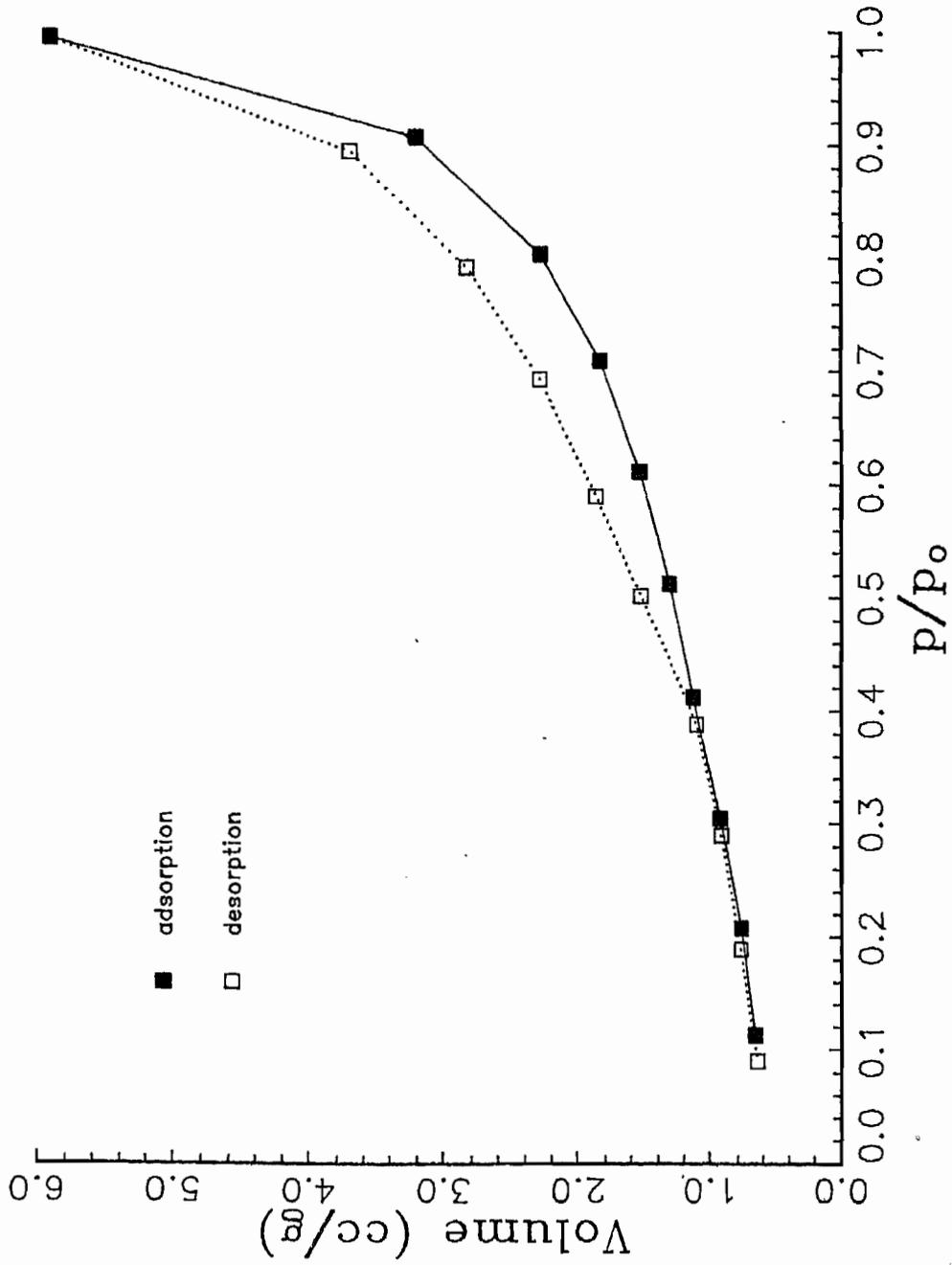


Figure 3. Adsorption-desorption hysteresis loop for nitrogen on MWX1 42-25.

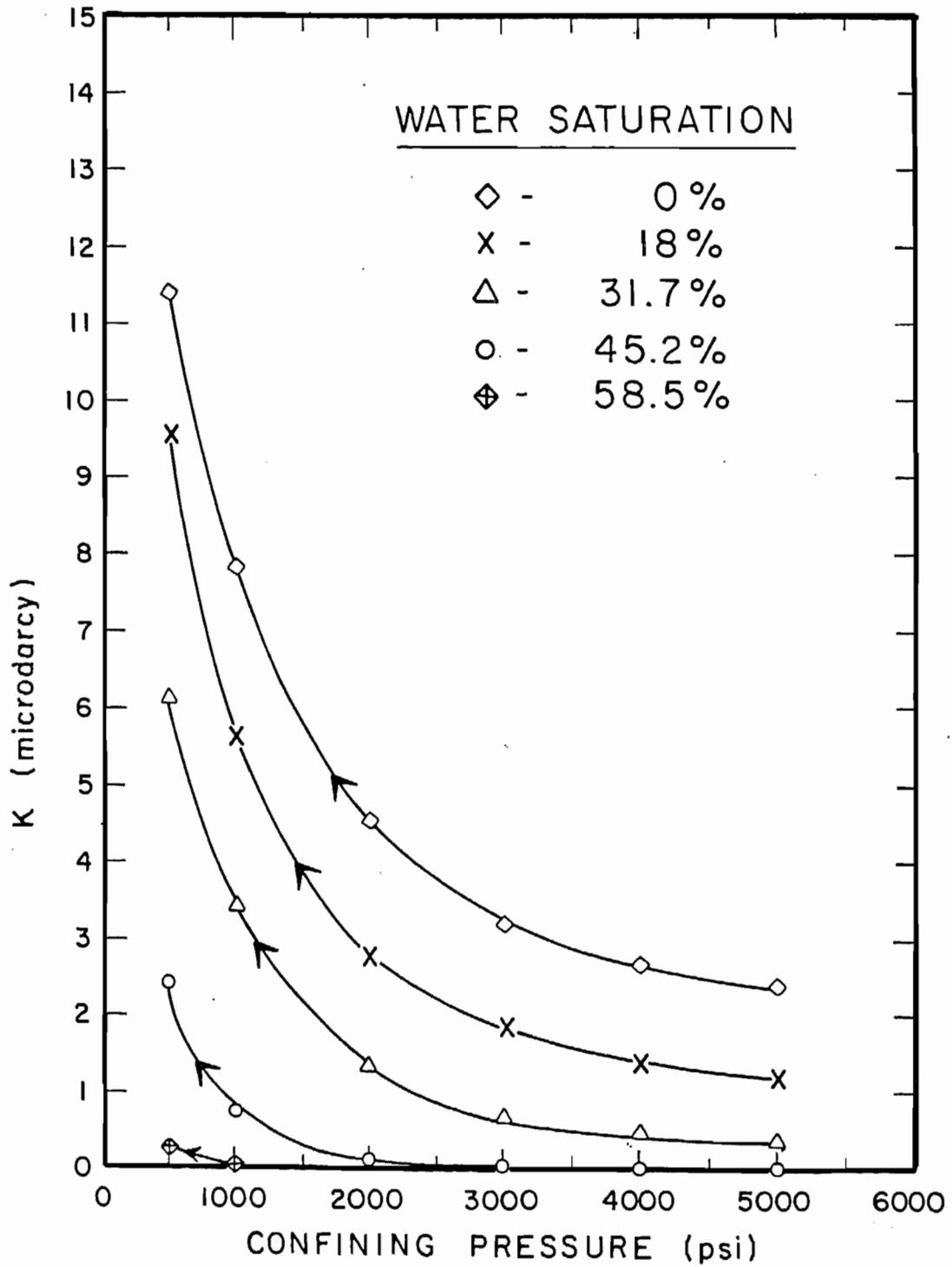


Figure 4. Gas permeability as a function of confining pressure with established water saturations from 0 to 58.5% for preserved MWX3 42-4.

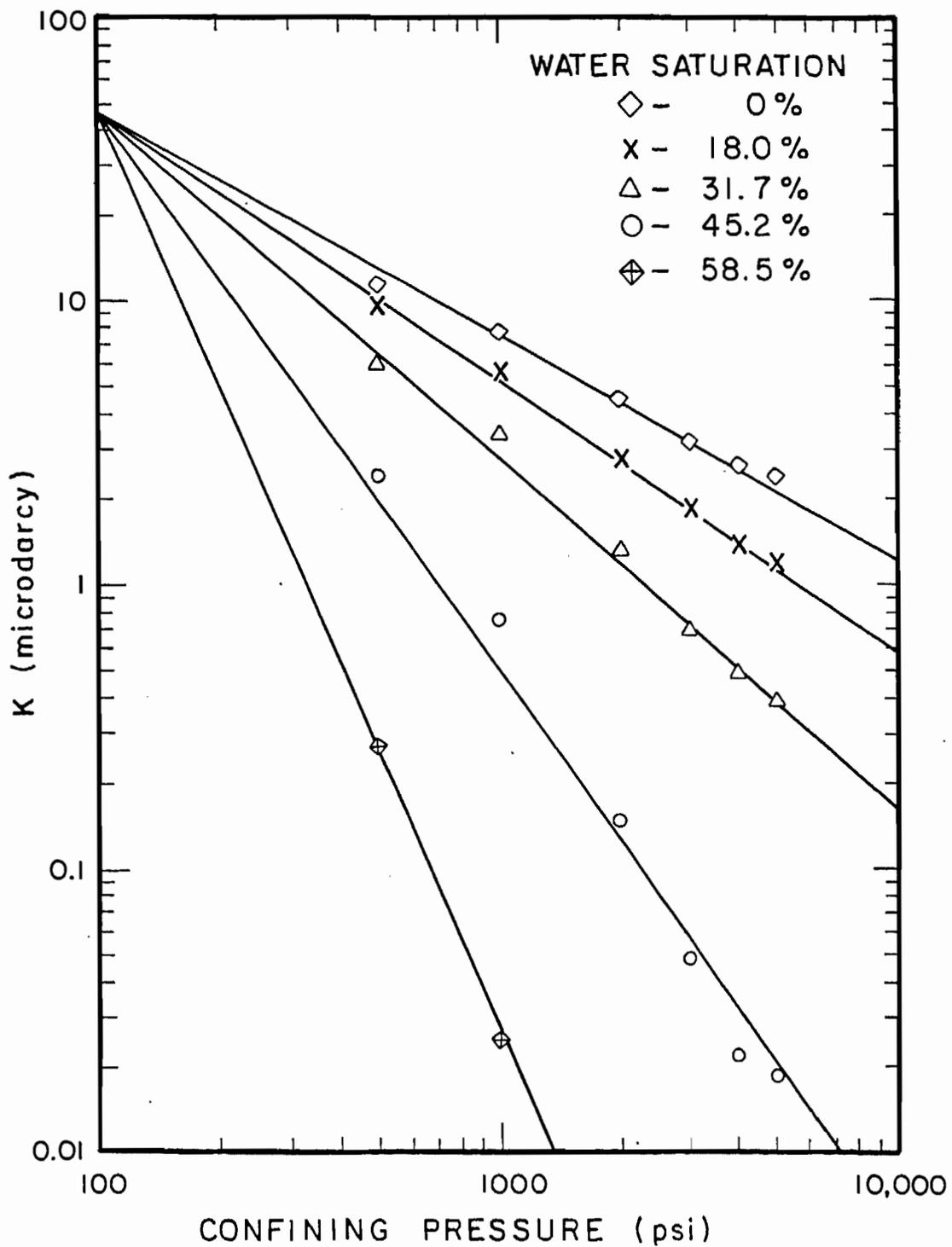


Figure 5. Log-log plot of gas permeability versus confining pressure at different levels of water saturation for preserved MWX3 42-4.

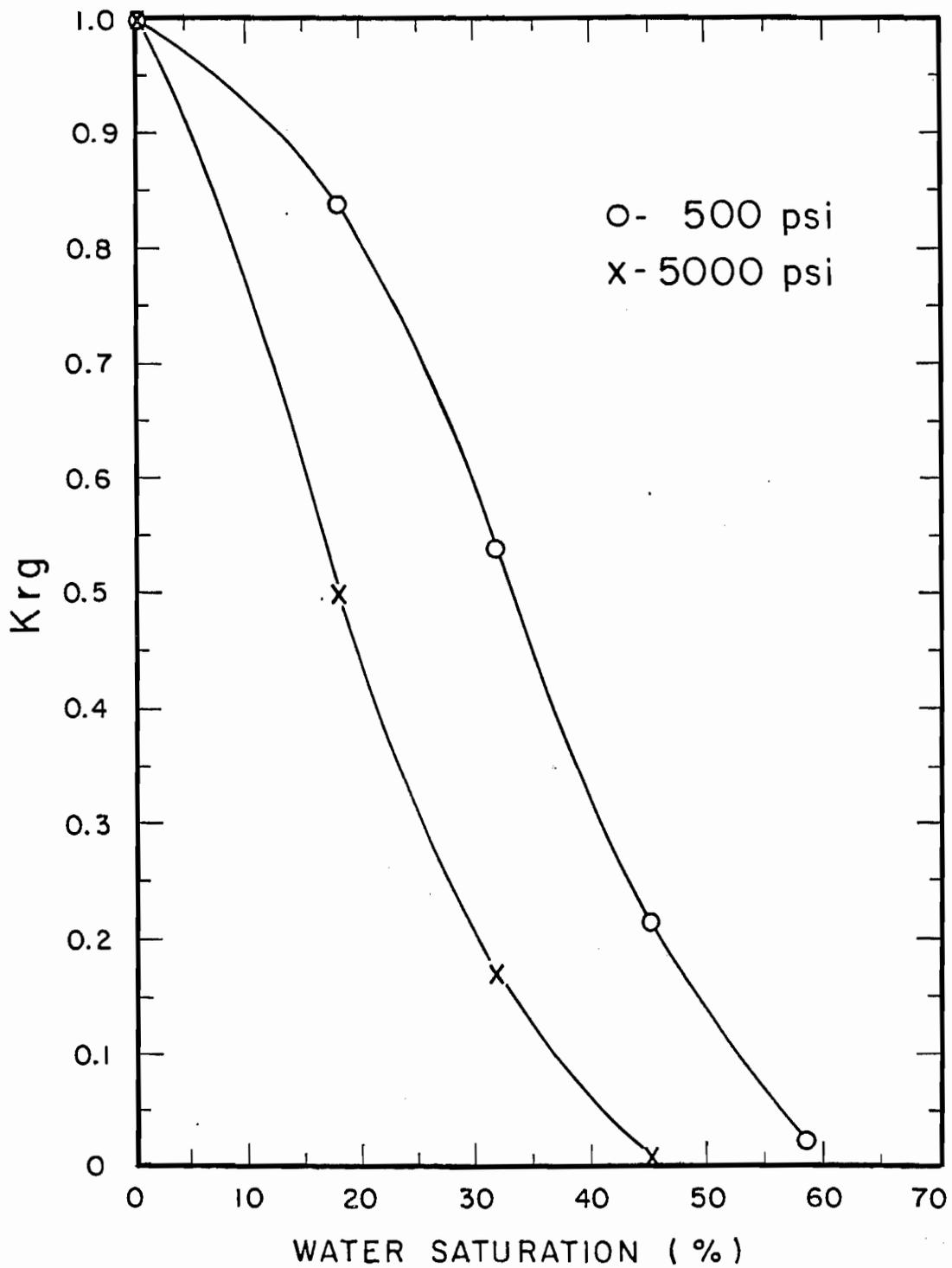


Figure 6. Relative permeability to nitrogen as a function of established water saturation at confining pressures of 500 and 5000 psi for preserved MWX3 42-4.

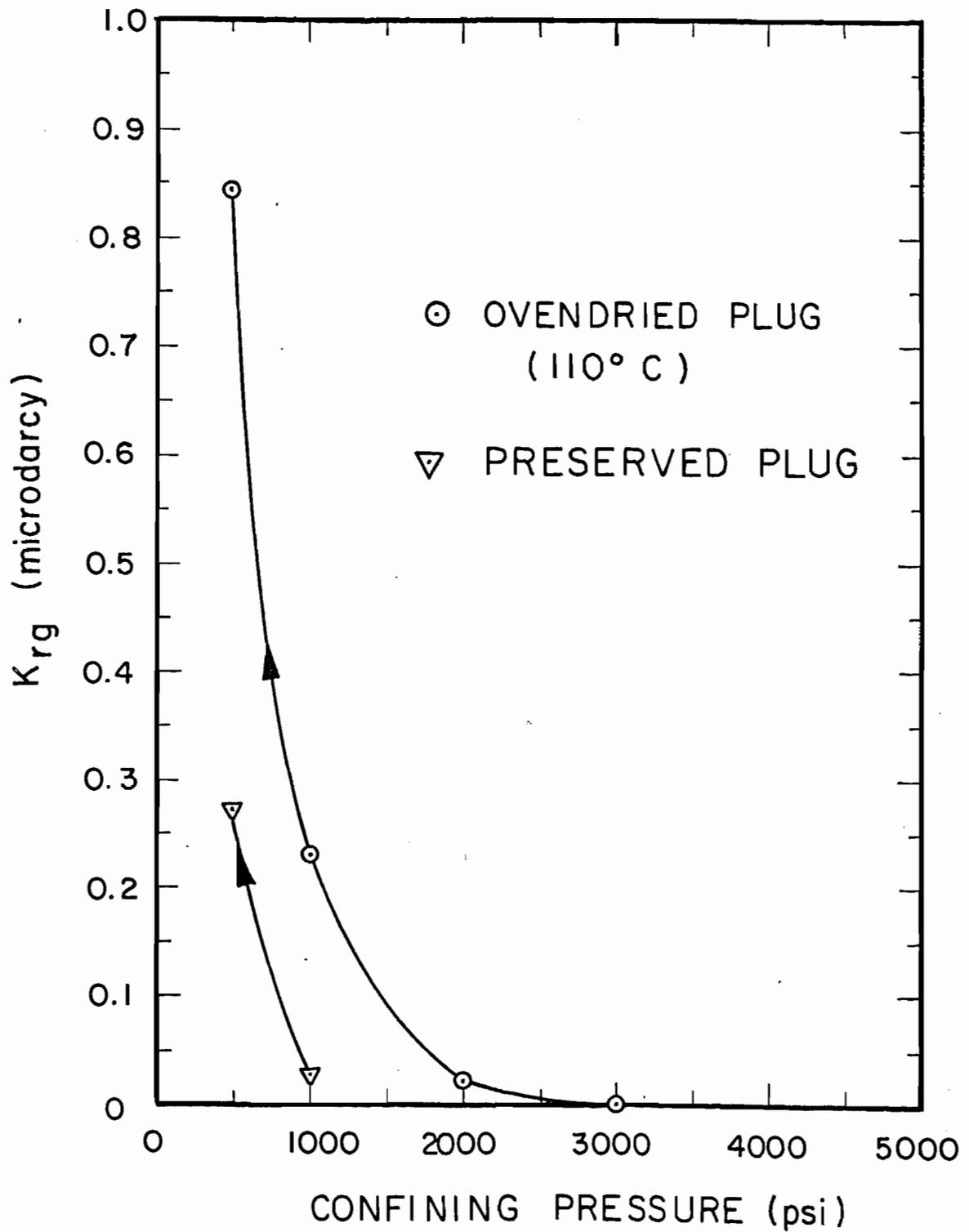


Figure 7. Preliminary results for the effect of drying on gas relative permeabilities.

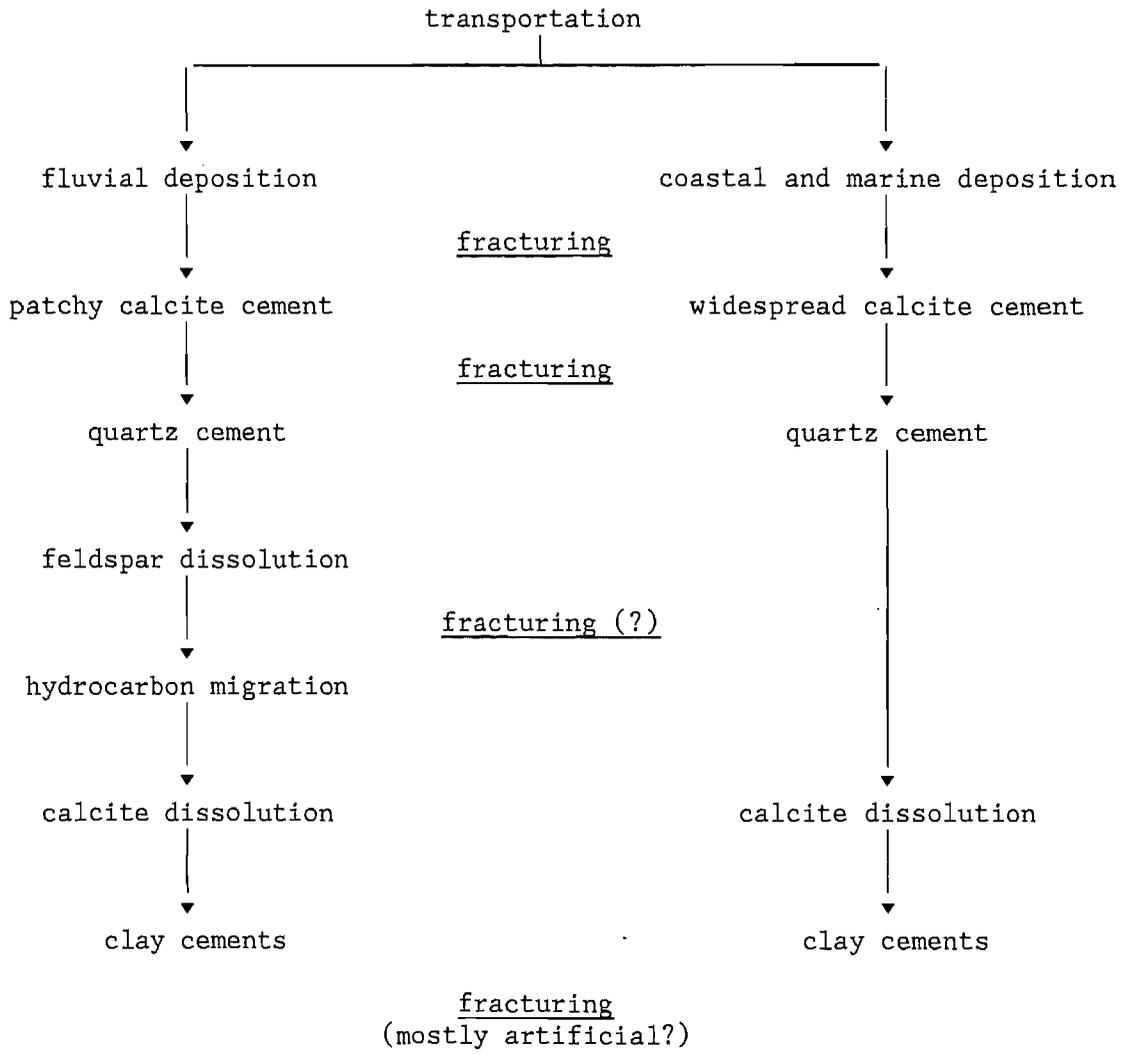


Figure 8. Generalized diagenetic history of Multiwell sandstones.