

NMR CHEMICAL SHIFT SELECTIVE IMAGING OF INDIVIDUAL FLUIDS IN SANDSTONE AND DOLOMITE CORES

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ABSTRACT

A number of approaches to chemical shift selective NMR imaging are possible, and several have been demonstrated. We have been evaluating the use of a pre-saturation method to obtain chemical shift selective, fluid specific, images of refined oil and aqueous brine in dolomite and sandstone cores. In the latter case, by injecting deuterated isopropanol, it was further possible to obtain fluid selective images for each of the three fluids present in the core. To our knowledge, this is the first demonstration by either NMR or X-ray CT imaging of fluid selective images for three individual fluids in a rock. 3D reconstructions of the fluid selective slice images are presented for the sandstone core.

INTRODUCTION

The ability to visualize the location of specific fluids such as oil and water in a core sample is extremely important for the study of multi-phase flow and the various factors that influence it. While X-ray CT imaging has been quite useful for determining the distributions of such fluids, these images are complicated by the response of the rock matrix and typically require the addition of high levels of contrast agent. ^1H NMR images of core fluids, on the other hand, are only indirectly affected by the rock matrix and in many cases may not require the use of contrast agents at all.

The indirect effects of the rock matrix on the NMR properties of the fluids can be severe, however. One principal effect is on the width of the ^1H NMR signal for the water or oil phase. This signal broadening effect arises from the difference in the bulk magnetic susceptibilities of the fluid and the rock matrix and is proportional to the static magnetic field strength. Depending on the nature of the rock, the broadening can be sufficient to prevent the spectral resolution of the NMR signals for oil and water and can severely limit image resolution as well. The other principal effect of the rock matrix is on the relaxation times of the ^1H NMR signals for the fluids, i.e., the time during which the signal can be manipulated or detected. The most seriously affected relaxation time is the spin-spin relaxation time (T_2) which can be so short that an image may not be obtainable with normal experimental parameters or techniques. Edelstein, et al. (1988), have demonstrated this effect for the shaley regions of sandstones.

In terms of being able to separately image the individual fluids in a rock core, two categories of samples can be described: (1) those for which the NMR line broadening is moderate and spectral resolution between water and oil is still observable, and (2) those for which the line broadening is severe and the water and oil signals are no longer resolvable. For the former category, the spectral separation (i.e., the chemical shift) of the water and oil can be used to obtain fluid selective images through what is usually called chemical shift imaging. A number of approaches to chemical shift imaging are possible. Edelstein, et al. (1988) have illustrated the use of selective excitation to obtain fluid selective images for dolomite samples and have also illustrated the use of differences in relaxation times to obtain fluid selective images in Bakers dolomite. The latter method has also been demonstrated by Hall and Rajanayagam (1987) for Berea sandstone, which because of extensive line broadening is a case where chemical shift selective imaging is not possible. Other approaches when chemical shift selective imaging is not possible include fluid doping such as Baldwin and Yamanashi (1988) used for Berea sandstone or multi-nuclear NMR imaging where different nuclides are used to image the different fluids.

Another approach to chemical shift imaging has been described by Keller, et al. (1987) for obtaining water or fat selective images in human subjects. This method employs a pre-saturation pulse to null the water or fat (oil) NMR signal before the imaging pulse sequence is executed. The advantage of this method is that it involves only a minor modification of the standard multi-slice "spin warp" imaging pulse sequence. Multi-slice, multi-echo imaging is still possible, and the imaging parameters are basically the same as the standard spin warp parameters. We have been using this pulse sequence to obtain fluid specific images for Bakers dolomite and for Bentheim sandstone cores saturated with brine and refined oil. These rocks induce only moderate broadening of the fluid NMR signals; the oil and water signals can still be spectroscopically resolved and therefore, chemical shift imaging is possible. We present here examples of oil selective and water selective images for these two samples. In addition we have constructed 3D representations of the fluid distributions for the sandstone sample. Finally, we present fluid selective images illustrating one approach to chemical shift imaging when three different fluids are present in a core. By using fully deuterated isopropanol, it was possible to obtain fluid selective images for alcohol, oil and water in Bentheim sandstone.

EXPERIMENTAL

The Bentheim sandstone used for these experiments had a porosity of 23% and an air permeability of 1400 millidarcies. Thin section analysis as well as X-ray CT and single fluid NMR imaging indicated that the sample was fairly homogeneous. The clay content was determined by X-ray analysis to be less than 2%. The Bakers dolomite sample was less homogeneous; gradations in porosity were readily observed in single fluid NMR images as well as X-ray CT images. The core plugs were about 2" x 3" and were placed in plexiglass holders. The dry Bentheim core was flushed with CO₂ and then saturated with Soltrol-130. Saturation was monitored using an X-ray CT scanner. A 2% solution of CaCl₂ brine was injected until water breakthrough occurred. After NMR images were obtained, about 20 cc of isopropanol-d₈ were injected. This volume represents about 38% of the total pore volume. The Bakers dolomite core was flushed with CO₂ and saturated with Soltrol-130. The core was then injected with 0.25 M NaI brine until no further oil was displaced.

NMR images were obtained at 1.5 Tesla on the University of Arkansas GE Signa clinical imaging system using a 6" extremity rf coil. The gradients were about 1 G/cm. The cores were imaged in two series of interleaved slices for a total of 19 transverse

slices each with a thickness of 5 mm. The slice images were produced with 128 gradient increments and were acquired into 256 real data points with zero filling in the second dimension. Pulse sequence parameters include a TR of 0.8 seconds and a TE of 30 ms. A total of 6 scans were acquired. For the chemical shift selective images, a pre-saturation pulse sequence was used which employed a 16 ms pre-saturation sinc pulse to suppress either the water or the oil signal. The linewidths at this field for both the water and the oil signals were about 180-200 Hz for both the sandstone and dolomite cores. The spectroscopic in-plane resolution was about 0.5 mm; the digital in-plane resolution was about 0.6 mm. Total imaging time was about 20 minutes.

The images were stored on 9-track tape and transferred to a CEMAX 1500X graphics workstation (Withjack, 1988). Using the workstation, color scales could be substituted for grayscales, image intensity could be contoured, and contoured slices could be assembled into a 3D image. In order to orient the viewer as to where the selected fluids lie in the core, the 3D images are displayed in a translucent mode that superimposes a "shadow" of the overall core on the fluid image.

RESULTS AND DISCUSSION

Spectroscopic saturation is a condition largely peculiar to NMR spectroscopy and arises from two of its characteristics (Pople et al., 1959). First, the energy separation between the ground and excited spin states is sufficiently small that both energy levels are nearly equally populated at normal temperatures. Second, the excited spin state can have a relatively long lifetime (usually called the relaxation time). The intensity of the NMR signal (i.e., the probability that a spin transition is induced) is related to the spin population excess in the ground state. When sufficient spin transitions have been induced to equally populate the two energy levels, no NMR signal is observable for those spins. This condition of equally populated ground and excited energy states is called saturation.

The problem of suppressing a signal in an NMR spectrum has been a long-standing concern since the advent of Fourier transform NMR. This concern arises because of the dynamic range limitations imposed by the digitizer and is most severe for dilute aqueous solutions. A number of approaches to solvent signal suppression have been described over the years (Freeman, 1988) and are potential approaches for signal suppression in NMR imaging as well. Pre-saturation is one method that has proven useful in NMR spectroscopy. It consists of applying a frequency selective rf pulse to saturate the water signal, followed by the normal observation pulse sequence to excite and detect the solute signals. The saturated water signal can be suppressed by several orders of magnitude by this method. This same procedure is the basis of the chemical shift selective pulse sequence of Keller et al. (1987).

Single fluid ^1H NMR images were obtained for the Bakers dolomite core saturated with Soltrol-130. These images indicated a significant degree of heterogeneity for the core; regions of high porosity were observed as well as considerable gradations in porosity. A 0.25 M NaI brine was injected until no further displacement of oil was observed. At this point, the pre-saturation method was used to obtain fluid selective images. First, by irradiating the oil NMR signal with the pre-saturation pulse until the signal was spectroscopically "saturated" (i.e., no longer observable), it was possible to obtain slice images with intensity only due to the water NMR signal. Figure 1a shows a transverse slice near the outlet end of the core obtained by this method. The intensity is primarily determined by the amount of water present within each voxel region of the core. Regions of low intensity represent regions of high oil concentration. By irradiating the water NMR signal with the pre-saturation pulse it is possible to obtain similar images

with intensity only due to the oil NMR signal. Figure 1b shows the same transverse slice but now with the intensity representing oil concentration. Comparison of the two images show that the regions of low intensity in the water selective image correspond with the regions of high intensity in the oil selective image. A second pair of slices closer to the outlet are shown in Figure 2a and 2b. Again, a high degree of correspondence is observed between regions of high intensity in the water image (Figure 2a) and regions of low intensity in the oil image (Figure 2b).

Single fluid NMR images were obtained for a Bentheim sandstone core saturated with Soltrol-130. These images indicate a high degree of homogeneity for this core. The core was then injected with a 2% CaCl_2 brine solution. Fluid selective images were obtained as was described for the dolomite sample, using the pre-saturation pulse sequence. Figure 3a shows a water selective image for a transverse slice near the outlet end of the core. The image shows a higher concentration of water near the center of the slice. Figure 3b shows an oil selective image for the same transverse slice. The highest intensity lies near the periphery of the slice with a region of low intensity at the center.

The series of 19 slices for the water selective images were used to construct a 3D representation of the water distribution in the sandstone core as shown in Figure 4. A translucent phantom of the core is superimposed on the water selective image as a viewing aid. The fluid flow direction is from right to left. The water is primarily located at the faces of the core and in a channel down the center of the core. The 19 slices for the oil selective images were similarly used to construct a 3D representation of the oil distribution as shown in Figure 5. The oil is located in a sheath around the periphery of the core and is displaced from the faces of the core.

About 20 cc (ca. 0.38 total pore volume) of isopropanol- d_8 were injected into the core. Because the alcohol is fully deuterated, it will not contribute to the ^1H NMR images. Thus, by obtaining a total ^1H NMR image using the conventional spin warp method, the regions of low intensity should represent the regions primarily occupied by the alcohol. By contouring the region of low intensity, the 3D representation shown in Figure 6 is obtained, which shows the location of the injected isopropanol. Again, the fluid flow direction is from right to left. The volume represented by the alcohol comprises about 40% of the volume of the core. Subsequent fluid-selective images using the pre-saturation pulse sequence lead to the 3D reconstructions of the water selective images in Figure 7 and the oil selective images in Figure 8. The water and oil have been displaced from the inlet end of the core by the alcohol miscible injectant and the location of these fluids in the outlet end of the core is readily discernable.

CONCLUSIONS

There are several advantages associated with the pre-saturation approach to chemical shift NMR imaging. The method involves only a slight modification of the conventional spin warp imaging method; multi-slice, multi-echo imaging is still possible and the majority of the pulse sequence parameters are little changed from those of the spin warp method. One disadvantage is the need for a very homogeneous B_1 field (which is also required by the other approaches to chemical shift imaging). The qualitative images shown here do not indicate any serious variations in our B_1 field, but problems could arise with larger size cores. We are currently evaluating this method for the quantitative determination of fluid saturations where any variations in B_1 should become more evident.

We have successfully demonstrated one approach to the three fluid problem. Using isopropanol-d8 as the third fluid, it is possible to obtain fluid selective images for three fluids in a core. The isopropanol is imaged by obtaining a non-selective image of total ^1H intensity and assuming that regions of low intensity represent the alcohol. Oil selective and water selective images are then obtained using the pre-saturation pulse sequence.

ACKNOWLEDGEMENTS

We are very grateful for the help provided by Steve Graham, Eric Withjack and C. T. Yang of the ARCO X-ray CT lab.

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FIGURE CAPTIONS

Figure 1. (a) Water selective image of transverse slice of Bakers dolomite core; (b) Oil selective image of the same transverse slice.

Figure 2. (a) Water selective image of second transverse slice of Bakers dolomite core; (b) Oil selective image of the same transverse slice.

Figure 3. (a) Water selective image of transverse slice of Bentheim sandstone core; (b) Oil selective image of the same transverse slice.

Figure 4. 3D reconstruction of water selective slice images of Bentheim sandstone core before alcohol injection.

Figure 5. 3D reconstruction of oil selective slice images of Bentheim sandstone core before alcohol injection.

Figure 6. 3D reconstruction from non-selective slice images showing location of injected isopropanol.

Figure 7. 3D reconstruction of water selective slice images showing water distribution after alcohol injection.

Figure 8. 3D reconstruction of oil selective slice images showing oil distribution after alcohol injection.



Figure 1. (a) Water selective image of transverse slice of Bakers dolomite core;



Figure 1. (b) Oil selective image of the same transverse slice.



Figure 2. (a) Water selective image of second transverse slice of Bakers dolomite core;



Figure 2. (b) Oil selective image of the same transverse slice.

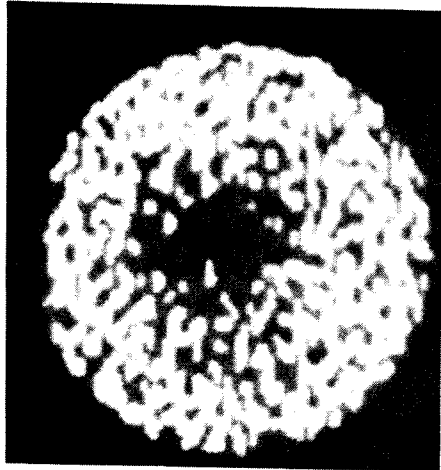


Figure 3. (a) Water selective image of transverse slice of Bentheim sandstone core;

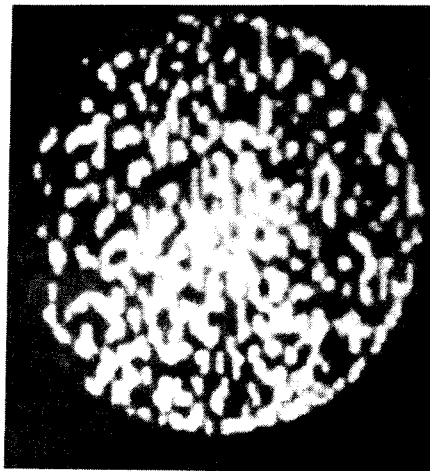


Figure 3. (b) Oil selective image of the same transverse slice.



Figure 4. 3D reconstruction of water selective slice images of Bentheim sandstone core before alcohol injection.

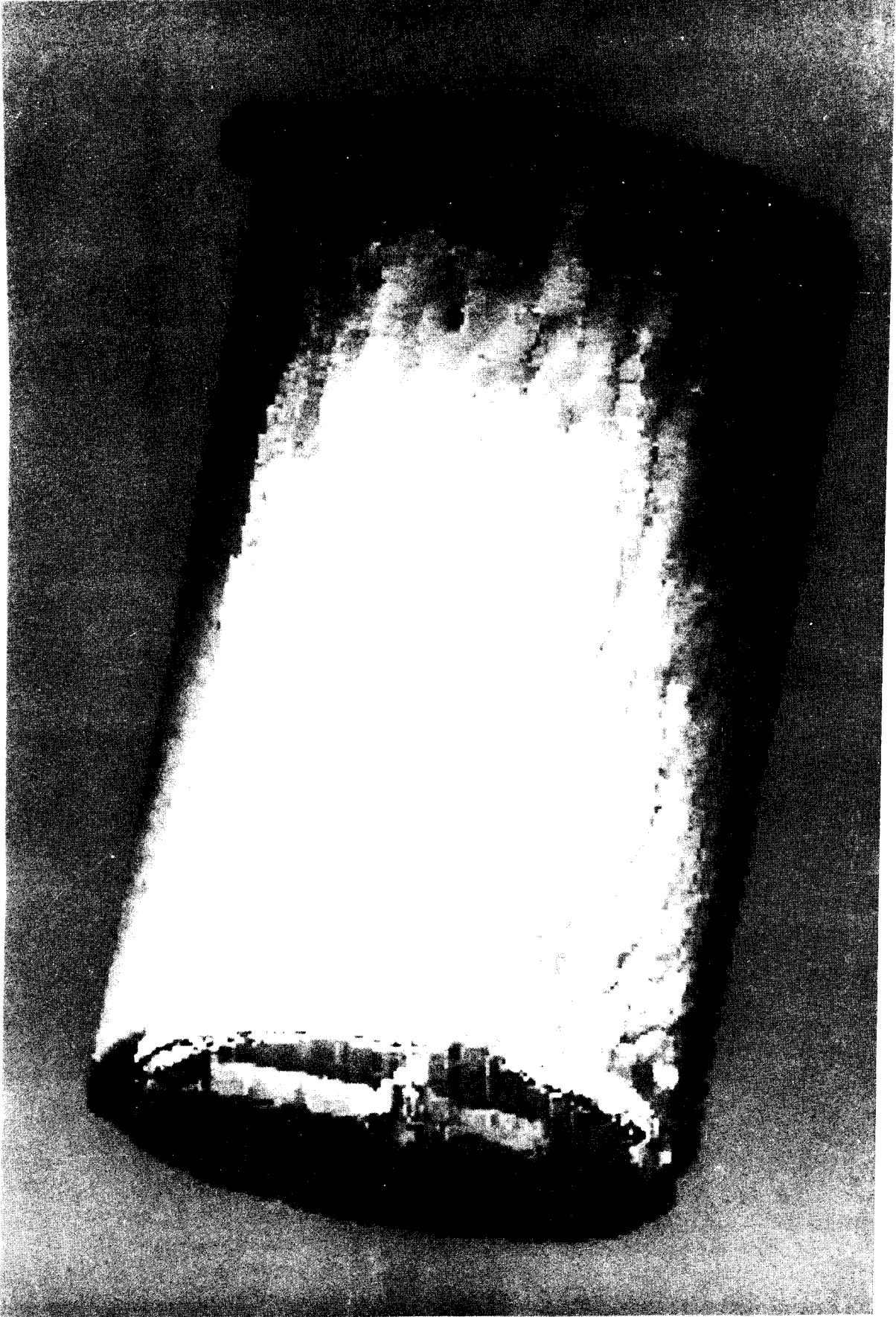


Figure 5. 3D reconstruction of oil selective slice images of Bentheim sandstone before alcohol injection.

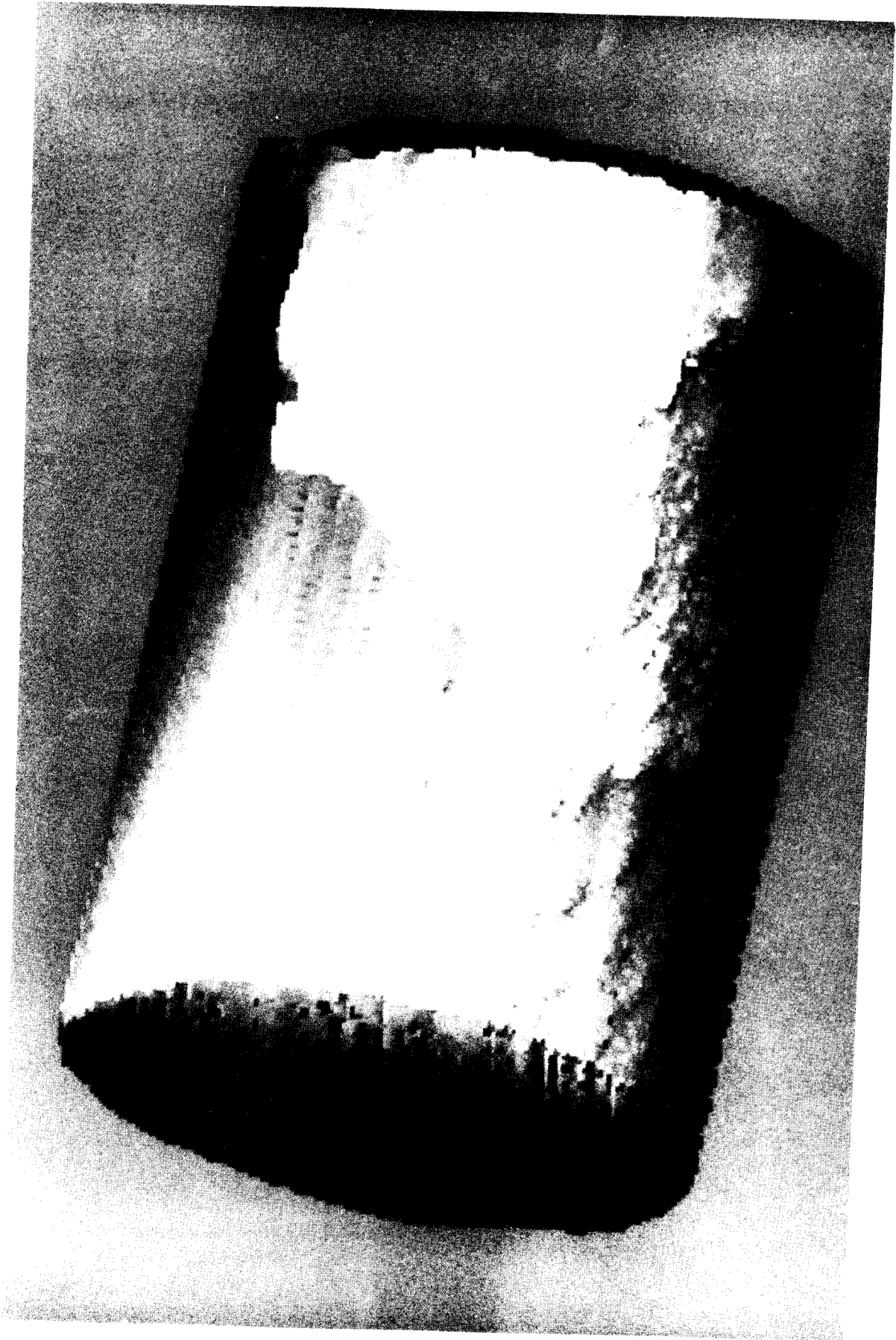


Figure 6. 3D reconstruction from non-selective slice images showing location of injected isopropanol.

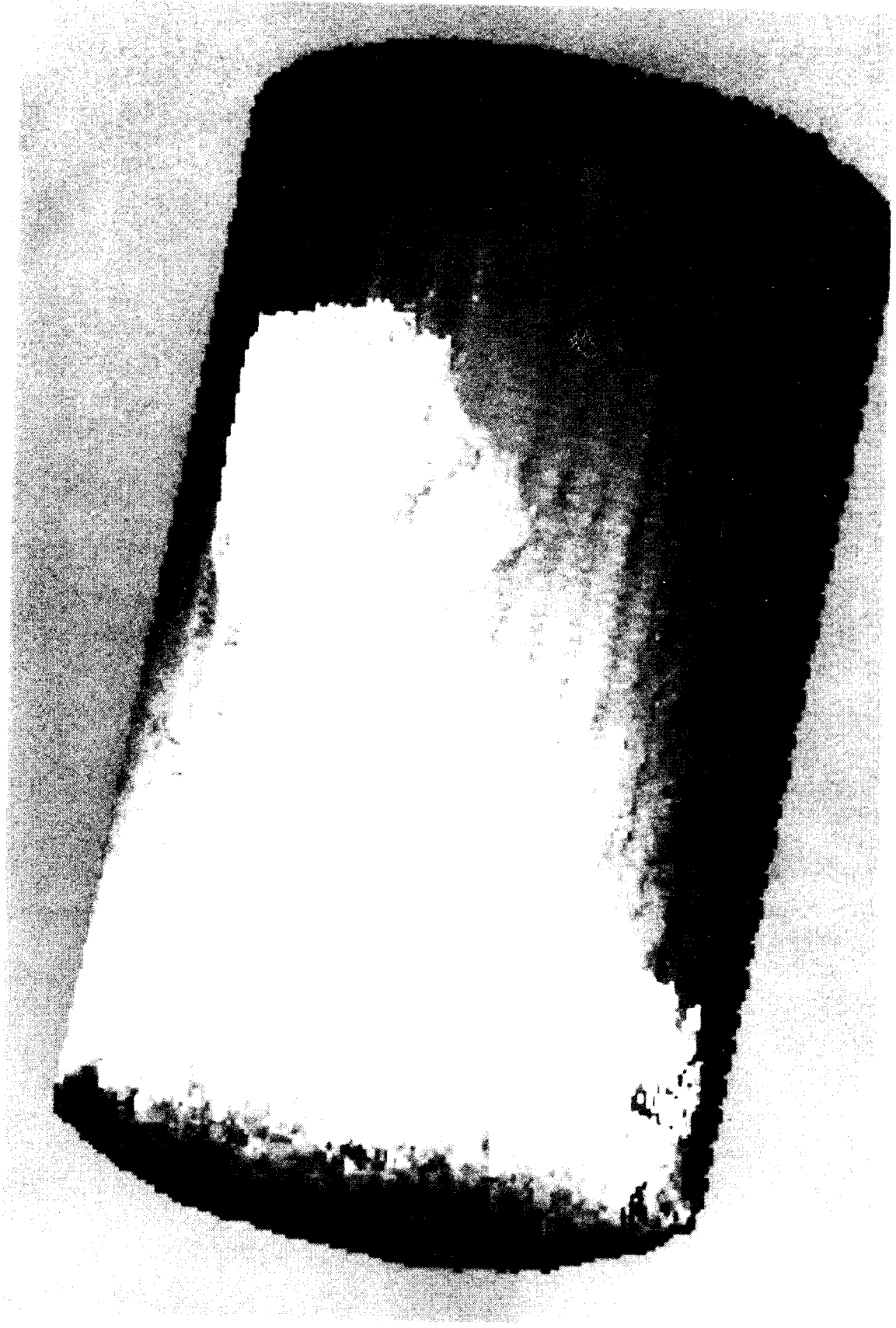


Figure 7. 3D reconstruction of water selective slice images showing water distribution after alcohol injection.



Figure 8. 3D reconstruction of oil selective slice images showing oil distribution after alcohol injection.