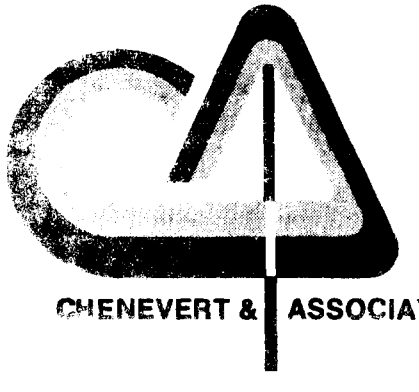
DEVONIAN SHALE - FRACTURING FLUIDS STUDIES

FINAL REPORT

SEPTEMBER 9, 1977

PRESENTED BY,

MARTIN E. CHENEVERT



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Summary

Ten shale samples were tested for their reactivity to water and other fracturing fluids. Three of the ten were found to be highly reactive with water. Kerosene and Methanol (with KCl water) minimize swelling.

X-ray diffraction and water adsorption isotherm data show these shales to contain only the slightly swelling clays Illite and Chlorite.

Experimental Data and Procedures

Refer to Progress Report No.1, June 7, 1977, Progress Report No. 2, July, 1977, Progress Report No. 3, August 1, 1977.

Swelling Tests

Table I and Figures 1 - 9 present all results obtained. As shown, only the 5300', 2740' and 3007' shales experienced a high degree of swelling when exposed to fresh water. The 2763' and 3027' shales had moderate swelling and the 4917', 4933', 5369' 3458', and 3896' shales had a low degree of swelling when exposed to fresh water. These shales experienced swelling similar to other shales previously reported[1].

Test Fluids Results

Shale samples were exposed to the six different test fluids shown in Table 2. All shales could not be exposed to all fluids because of limited shale samples available. Table 3 lists test results for the three highly swelling shales tested in order of increased swelling from left to right.

Water Base Fluids - a mixture of 30% Methanol and 70% KCl water (2% by weight KCl) reduced swelling more that the other water base fluids tested. For the three highly swelling shales their reduction varied from 30% to 76%. The other fluids tested had different degrees of inhibition. In order of preference fluid C was second and fluid D and E were next with some uncertainty as to which one was preferred.

The usefulness of KCl has been documented in the literature,[2] however, Methanol is a new approach which warrents field evaluation.

Oil Base Fluids - as expected[3] very little swelling (only .005%) was produced by kerosene (Fluid F). Figures 6 and 7 show these data for the 3458' and 3027' shales.

X-Ray Diffraction

Table 4 presents x-ray diffraction data for eight shales. As shown the shales contain a predominance of Illite and Chlorite clays and no Montmorillonite. These data plus the swelling data show that Illitic - Chloritic shales do swell provided they contain large quantities of these clays.

Adsorption - Isotherms

Table 5 presents the Adsorption Isotherm data. In general these eight shales had very flat (low adsorption) isotherms which is consistent with the x-ray diffraction data presented in Table 4.

Recommended Future Work

Some uncertainty exists as to what is the optimum mixture of Methanol and KCl water. This could be further defined by performing swelling tests at various Methanol/KCl water ratios.

It was surprising how much swelling variation was experienced within one well. As Table I shows the 5300' shale became swollen by .65% after 10 hours and the 5369' shale became swollen by only .05%. A more detailed swelling responce within zones of interest would be most informative.

Conclusions

1. Some of the shales exposed in the two wells studied have rather high swelling tendencies.

2. Swelling can be minimized by using the kerosene oil base fluid or the Methanol water base fluid.

3. The shales contain only Illite and Chlorite type clays.

References

1. Chenevert, M. E., "Shale Alteration by Water Adsorption", Journal of Petroleum Technology, September, 1970.

2. O'Brien, Dennis and M. E. Chenevert, "Stabilizing Sensitive Shales With Inhibited Potassium-Based Drilling Fluids", Journal of Petroleum Technology, September, 1973.

3. Chenevert, M. E., "Shale Control With Balanced Activity Oil - Continuous Muds", Journal of Petroleum Technology, October, 1970.

Work Reported by,

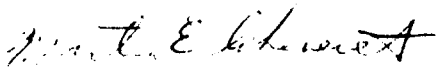

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TABLE I

WATER-SHALE, SWELLING RESULTS

Columbia Gas Well No. 20338, Wise County, West Virginia

Depth Feet	% Swelling After 10 Hours
4917	.05
4933	.15
5300	.65
5369	.05

Columbia Gas Well No. 20402, Lincoln County, West Virginia

Depth Feet	% Swelling After 10 Hours
2740	.70
2763	.30
3007	.70
3027	.40
3458	.08
3896	.08

TABLE 2

TEST FLUIDS

A. Fresh Water

B. 30% Methanol + 70% (Water + 2% KCl)

C. 2% KCl

D. Fresh Water + 7 Gal. HC-2/1000 Gal. + 1/2 Gal. Cla - Sta/1000 gal

E. Fresh Water + 2 Gal F-75/1000 Gal + 5 Gal L-42/ 1000 Gal

F. Kerosene

TABLE 3

Test Fluids - Shale, Swelling Results

Columbia Gas Well No. 20338, Wise County, West Virginia

Depth	Best Fluid (Lowest Swelling)			Worst Fluid (Highest Swelling)	
	↓				↓
5300	B	C	E	A	D

Columbia Gas Well No. 20402, Lincoln County, West Virginia

Depth	Best Fluid (Lowest Swelling)			Worst Fluid (Highest Swelling)	
	↓				↓
2740	B	C	D	A	E
3027	B	C	D	A	E

TABLE 4

X-Ray Diffraction Analysis

Columbia Gass Well No. 20338, Wise County, West Virginia

4917'		4933'	
α- Quartz	37.7%±	α- Quartz	49.2%±
Feldspar	1.3%±	Feldspar	2.7%±
Pyrite	2.6%±	Pyrite	.9%±
Illite	39.4%±	Illite	36.2%±
Chlorite	19.0%±	Chlorite	11.0%±

5300'		5369'	
α- Quartz	23.8%±	α- Quartz	21.3%±
Feldspar	1.0%±	Feldspar	1.0%±
Pyrite	3.2%±	Pyrite	3.5%±
Illite	49.5%±	Dolomite	7.5%±
Chlorite	22.5%±	Illite	36.2%±
		Chlorite	30.5%±

Columbia Gas Well No. 20402, Lincoln County, West Virginia

2740'		3027'	
α- Quartz	33.1%±	α- Quartz	23.8%±
Feldspar	1.3%±	Feldspar	1.0%±
Illite	45.3%±	Pyrite	2.3%±
Chlorite	20.3%±	Illite	46.7%±
		Chlorite	26.2%±

3455'		3896'	
α- Quartz	23.1%±	α- Quartz	23.1%±
Feldspar	1.0%±	Feldspar	1.0%±
Pyrite	1.9%±	Pyrite	2.7%±
Illite	57.2%±	Illite	65.5%±
Chlorite	16.8%±	Chlorite	7.7%±

TABLE 5

Adsorption Isotherm Analysis

Columbia Gas Well No. 20338, Wise County, West Virginia

Environment, Relative Vapor Pressure p/p$_o$	Water Adsorption, % by Weight			
	4917'	4933'	5300'	5369'
0.100	0.25	0.14	0.35	0.22
0.295	0.27	0.17	0.43	0.30
0.505	0.64	0.46	0.97	0.56
0.775	0.95	0.78	1.39	0.87
0.800	1.15	0.87	1.75	1.02
0.920	1.53	1.21	2.31	1.53
0.960*	3.29	2.80	4.84	2.89

Columbia Gas Well No. 20402, Lincoln County, West Virginia

Environment, Relative Vapor Pressure p/p$_o$	Water Absorption, % by Weight			
	2740'	3027'	3455'	3896'
0.100	0.35	0.30	0.32	0.26
0.295	0.35	0.35	0.33	0.29
0.505	0.70	0.66	0.66	0.68
0.775	1.08	0.93	0.90	1.05
0.800	1.16	1.13	1.04	1.22
0.920	1.25	1.53	1.67	2.17
0.960*	3.33	2.95	3.27	4.95

* Condensate noted in these vials.

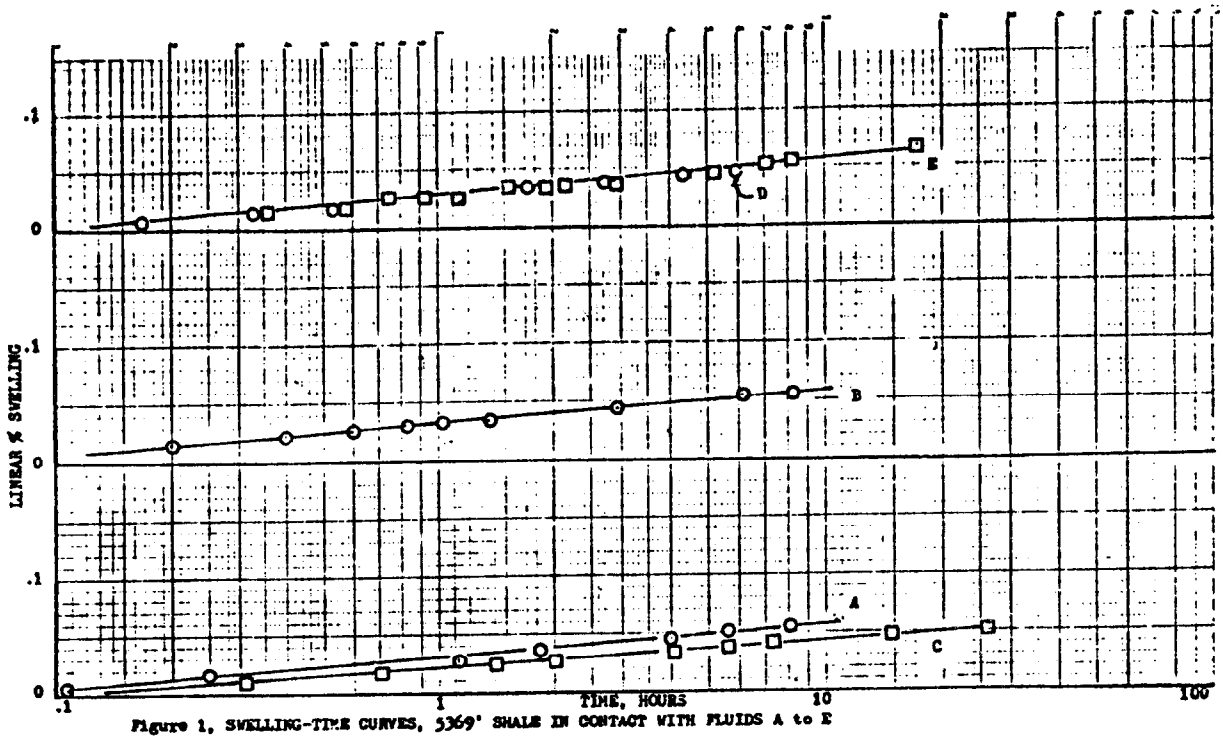

Figure 1, SWELLING-TIME CURVES, 5369' SHALE IN CONTACT WITH FLUIDS A to E

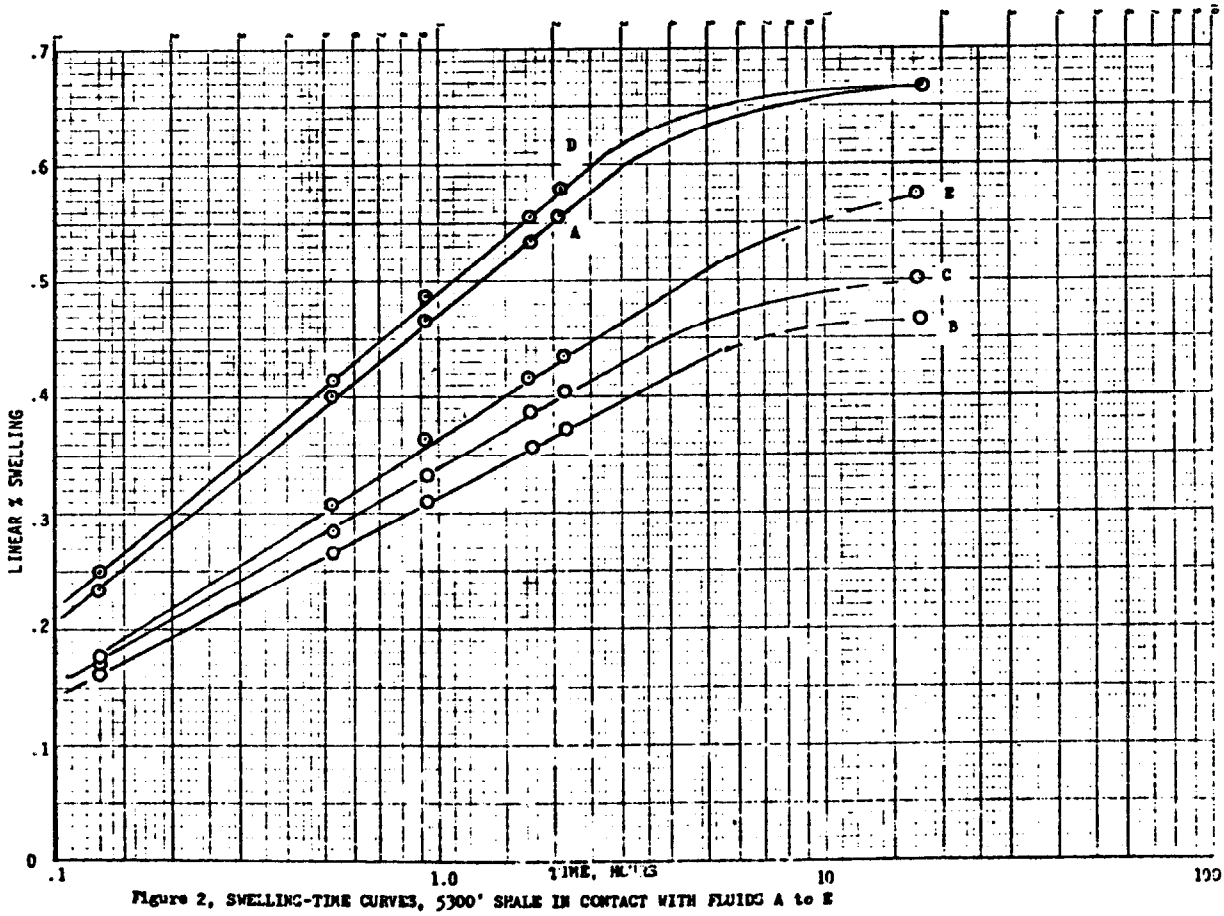

Figure 2, SWELLING-TIME CURVES, 5300' SHALE IN CONTACT WITH FLUIDS A to E

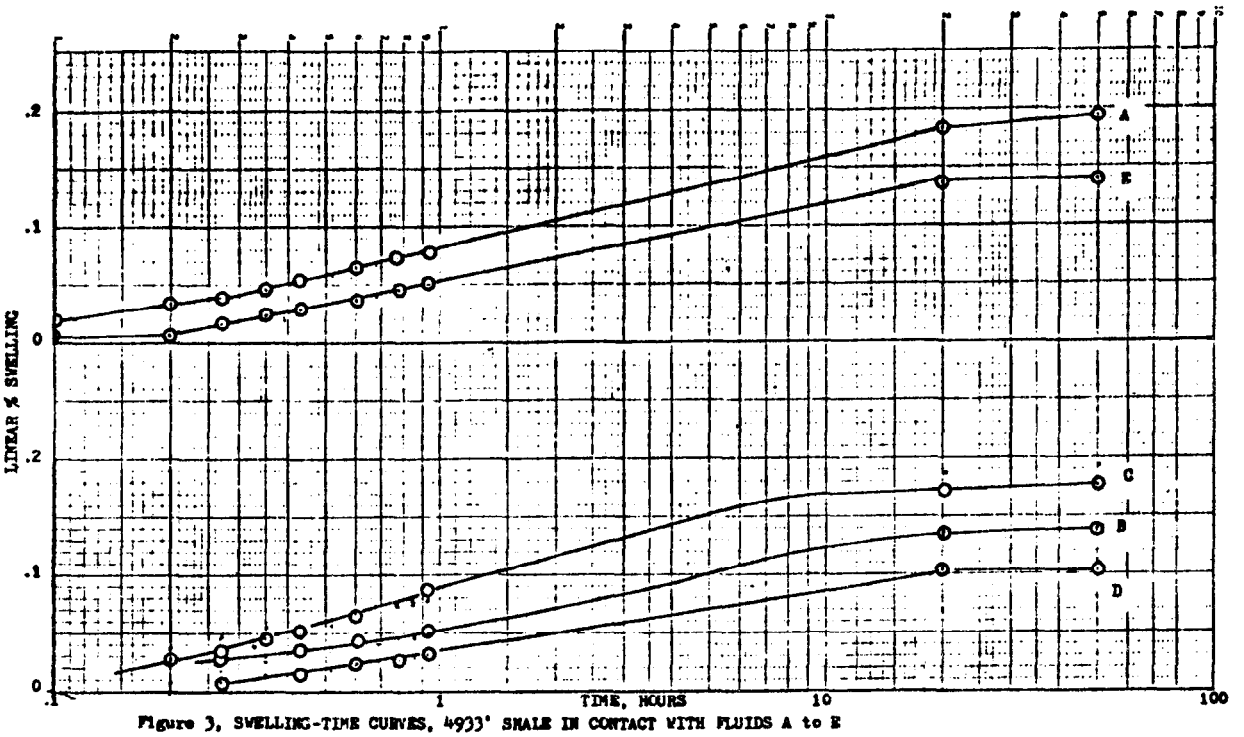

Figure 3, SWELLING-TIME CURVES, 4933' SHALE IN CONTACT WITH FLUIDS A to E

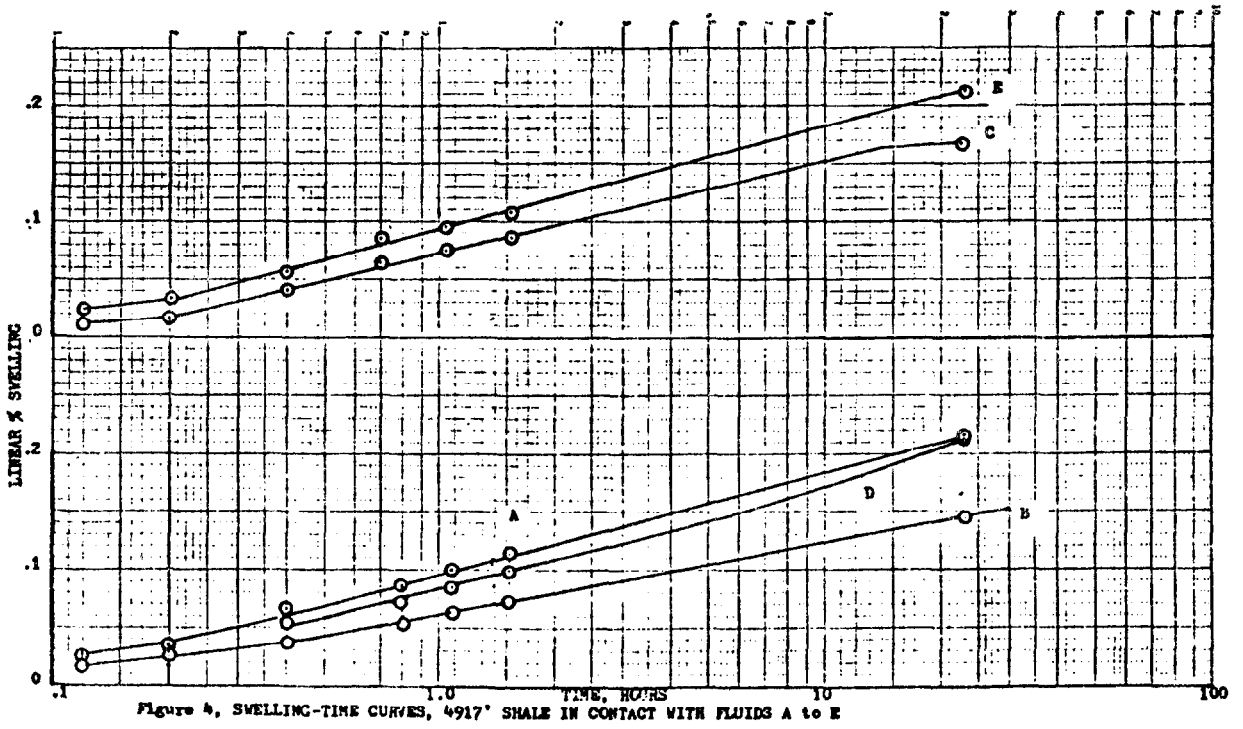

Figure 4, SWELLING-TIME CURVES, 4917' SHALE IN CONTACT WITH FLUIDS A to E

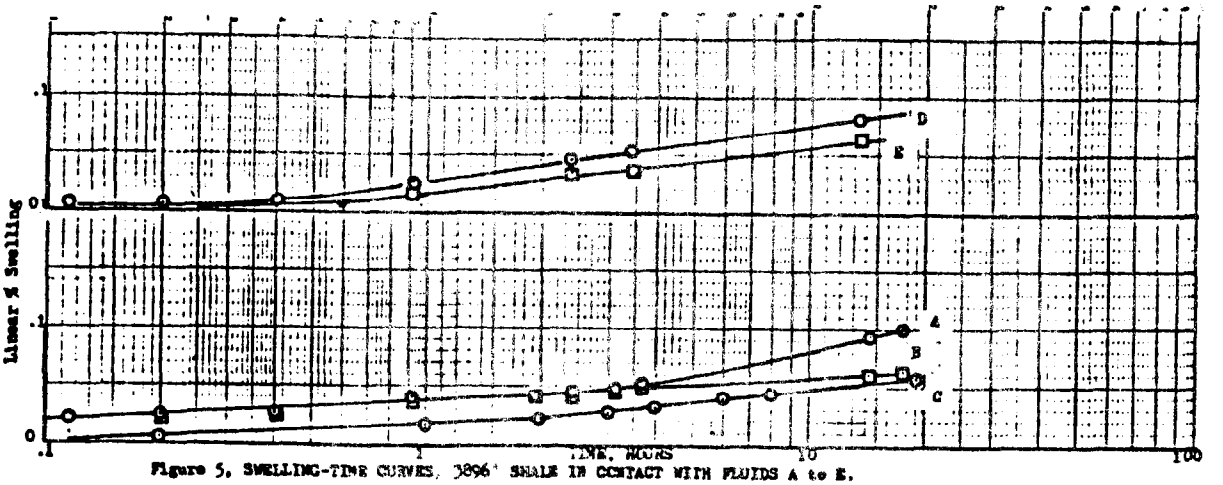

Figure 5, SWELLING-TIME CURVES, 3896' SHALE IN CONTACT WITH FLUIDS A to E.

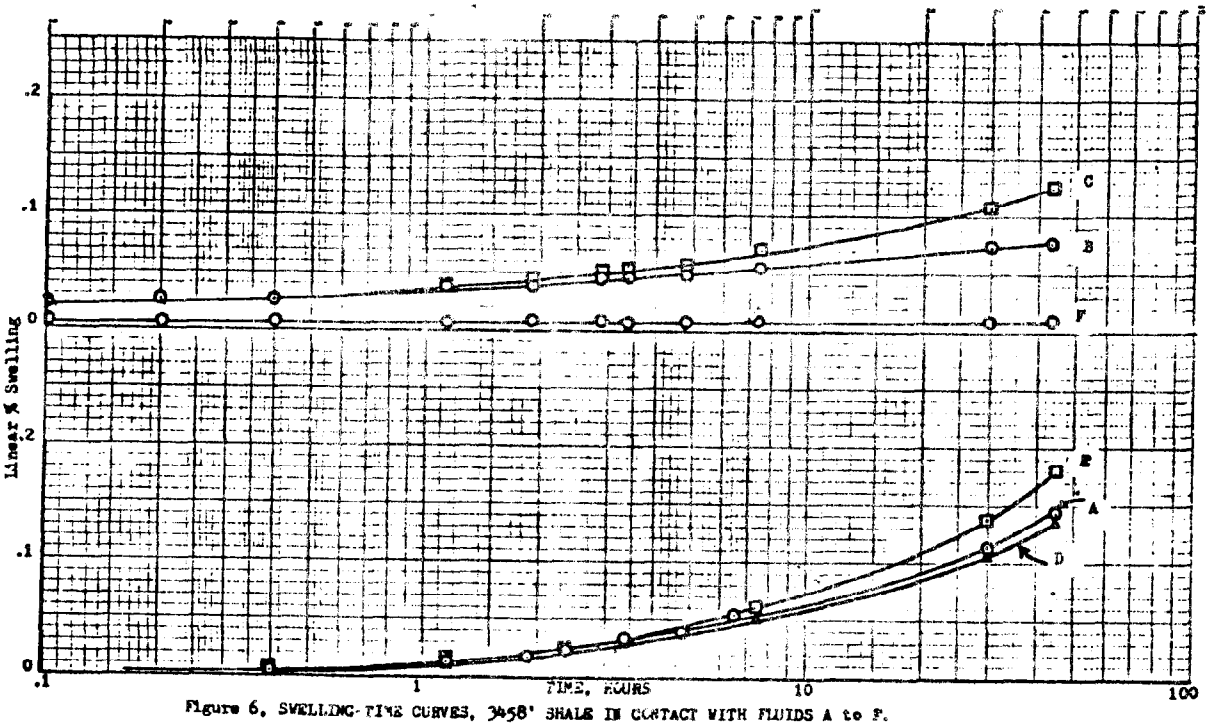

Figure 6, SWELLING-TIME CURVES, 3458' SHALE IN CONTACT WITH FLUIDS A to F.

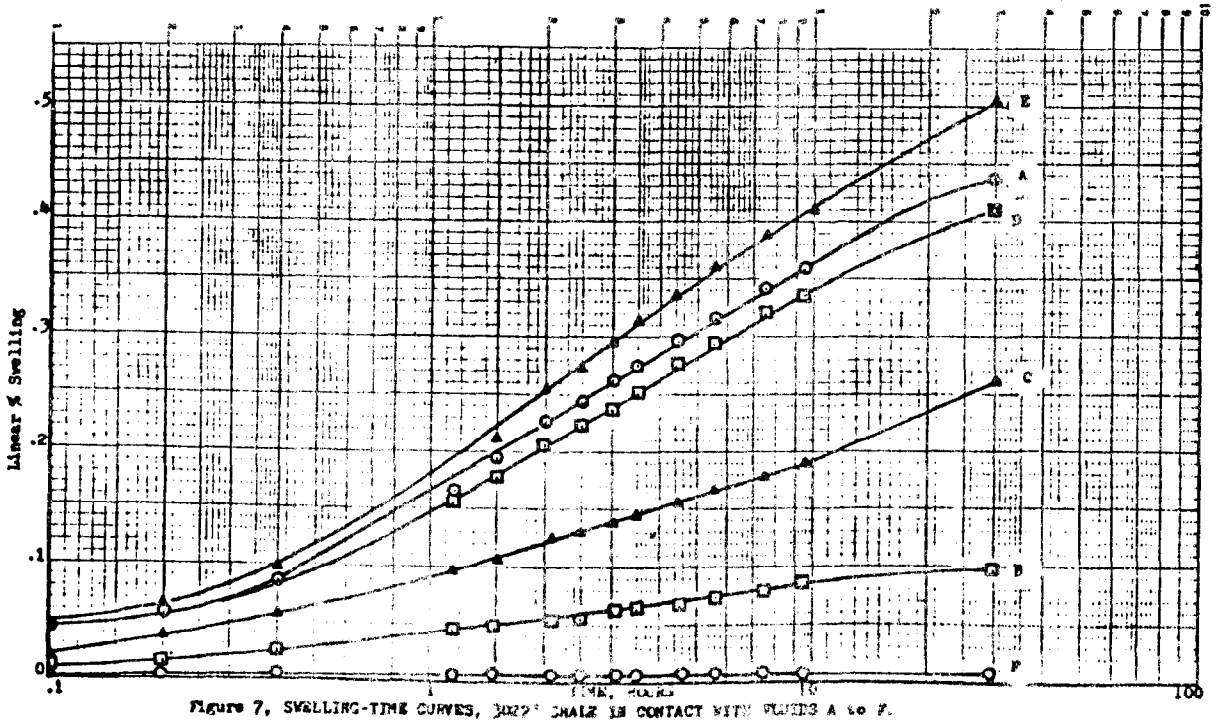

Figure 7, SWELLING-TIME CURVES, 3027' SHALE IN CONTACT WITH FLUIDS A to F.

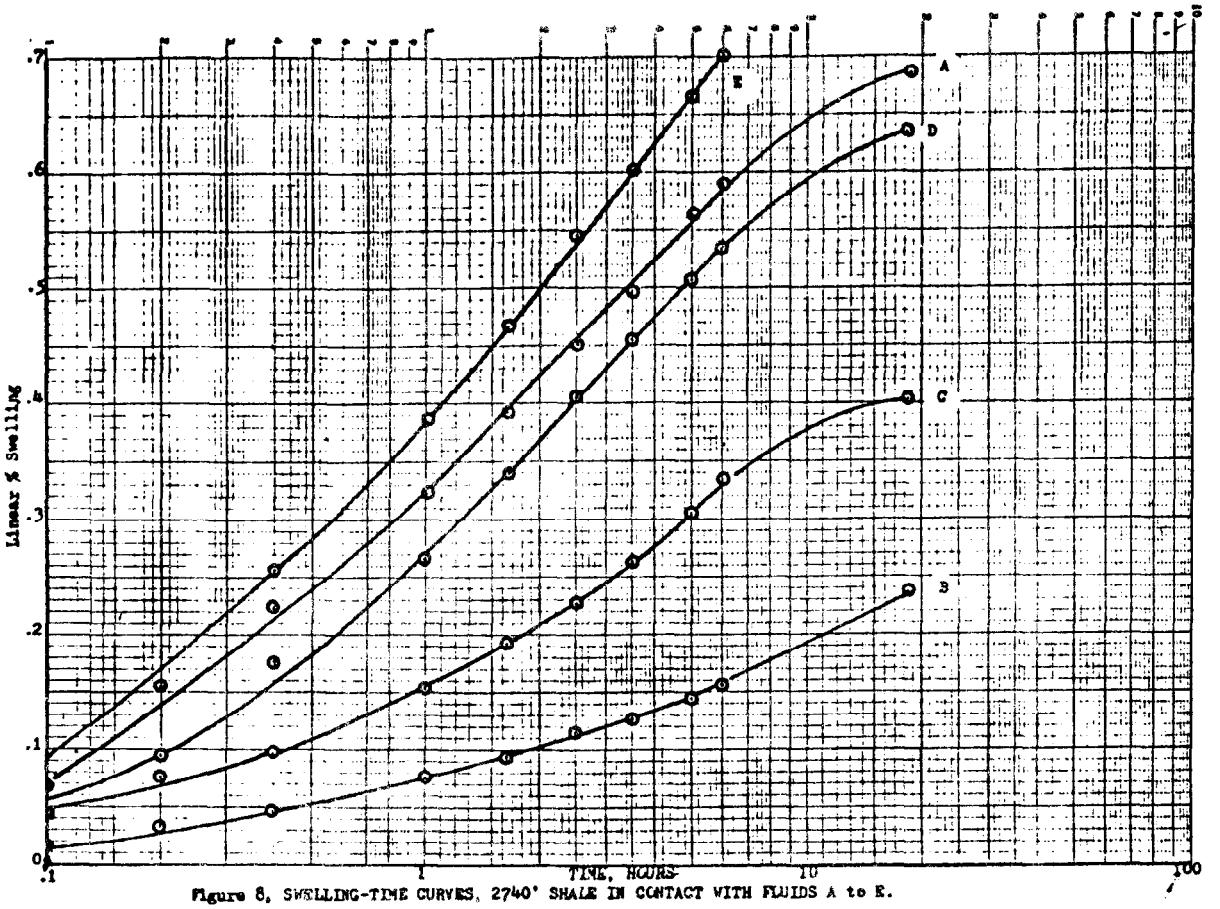

Figure 8, SWELLING-TIME CURVES, 2740' SHALE IN CONTACT WITH FLUIDS A to E.

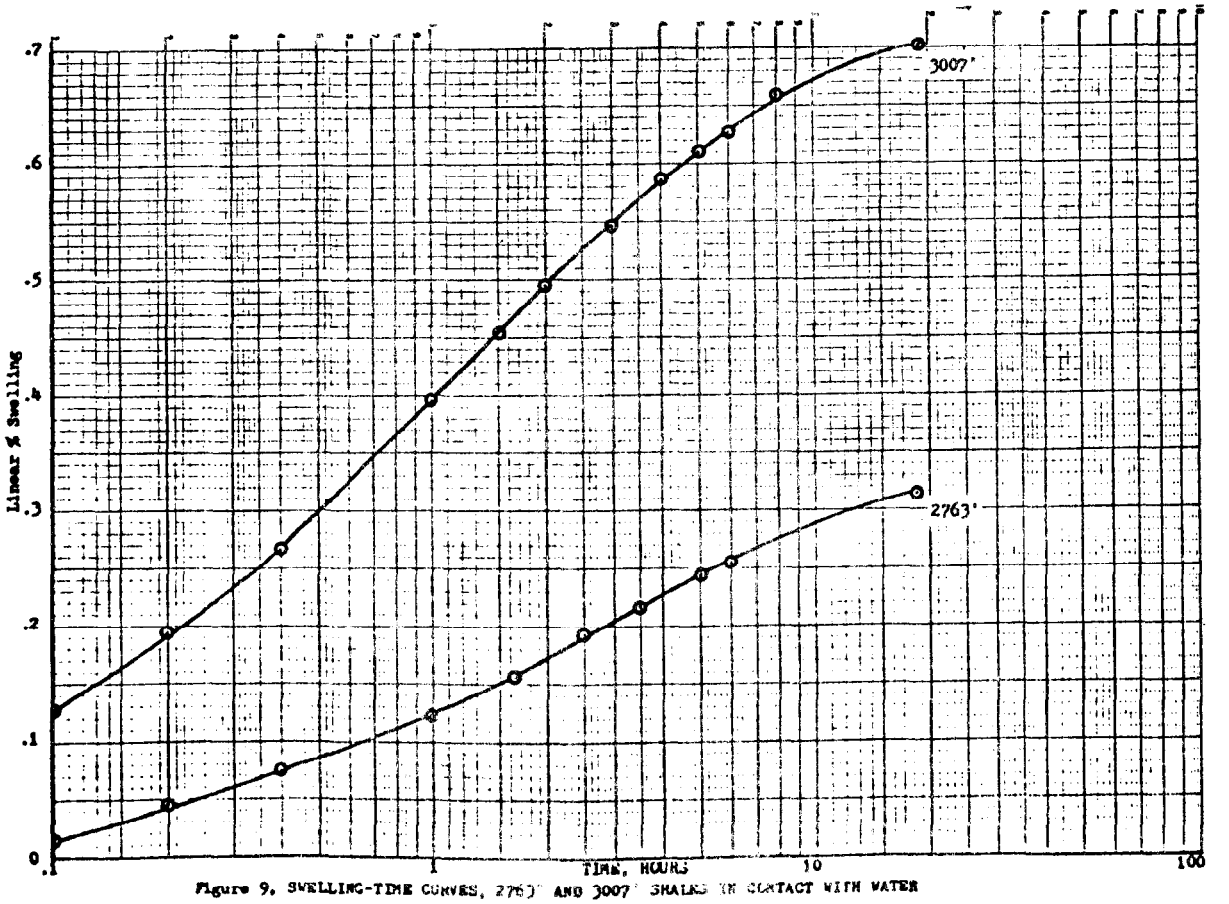

Figure 9, SWELLING-TIME CURVES, 2763' AND 3007' SHALES IN CONTACT WITH WATER

Devonian Shale - Fracturing Fluids Studies

Progress Report No. 1

June 7, 1977

Samples Received

On May 10, 1977 shale core samples 1 to 4 were received from well 22 and on May 27, 1977 samples 5-8 were received from well No. 20338. Table I lists the location and depths of the various samples. The first four were unpreserved and the second four were preserved in plastic bag wrappings and sealed in tin cans.

Sample Preparation

After receiving the shales, a $1\frac{1}{4}$" section of each sample was prepared for testing in the following manner.

a. Swelling tests

About 6 rectangular specimens, $\frac{1}{2}$" x $\frac{1}{2}$" x $1\frac{1}{4}$" were cut and sealed for future testing. They were cut using kerosene coolant so that hydration was minimized.

b. X-Ray diffraction and adsorption isotherms

The remainder of the $1\frac{1}{2}$" section was sealed and forwarded to "Spectrochemical Research Labs", Houston, Texas, for analysis.

Testing

Swelling tests were run on the 5369' interval using the various fluids listed in Table 2. This table lists the swelling results obtained after 8 hours of exposure to the test fluids.

Results

As shown in Table 2 the 5369' shale interval (as received) does not exhibit excessive swelling tendencies, and responds about the same when placed in the various aqueous solutions. The variation of 420 to 520 micro inches per inch is not felt to be significant. Future testing will help define experimental accuracy and substantiate such variations.

The level of 500 micro inches per inch is only 1/10 the amount of swelling experienced on other shales as shown in Figure 4 of the reference, "Shale Alteration by Water Adsorption", Journal of Petroleum Technology, September 1970.

It should be pointed out that even though the 5369' shale was received sealed in a can it might not be in its true in situ hydrational state. The shale may have adsorbed significant water while coreing. This aspect can be further defined once the adsorption isotherm is developed.

Exposure of the 5369' shale to kerosene shows kerosene to be essentially inert to the shale.

Conclusions

Compared to other argillaceous shales the 5369' shale, as received, does not have excessive swelling tendencies. All aqueous solutions produce similar swelling and may be damaging to the shale in normal

TABLE I

Shale Samples Received

Core Samples	Location	Zones	Depths	Intervals
1	Well 22	1	2763	-
2	"	2	3007	-
3	"	3	3458	-
4	"	4	3896	-
5	Well 20338	1	4917	4
6	"	1	4933	3
7	"	2	5300	2
8	"	2	5369	1

TABLE 2

Swelling Data - Shale from depth of 5369'

Fluid Type	Swelling after 8 hours micro inches per inch
1. Fresh Water	495
2. Fresh Water + 2% KCl	420
3. 30% Methanol +70% (Water + 2%KCl)	545
4. Fresh Water +2 gal F-75/1000 gal +5 gal L-42/1000 gal	520
5. Fresh Water + 7 gal HC-2/1000 gal + $\frac{1}{2}$ gal ClaSta/1000 gal	520
6. Kerosene	6

fracturing operations. Proppant imbedment tests could help evaluate such effects.

Future Work

Swelling tests are planned for the other seven samples. These data coupled with x-ray and adsorption data should define the water sensitivity tendencies of the shale and also evaluate the various frac fluids listed in Table 2.

Work reported by,

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Devonian Shale - Fracturing Fluid Studies

Progress Report No. 2

July 7, 1977

Testing

Swelling tests were run on the 5300', 4933' and the 4917' shales using the various fluids listed in Table 1.

Results

Figures 1 to 4 show the swelling response of the various shales immersed in the five test fluids. In order to compare results, values of swelling after eight hours were obtained from these graphs and listed in Table 1.

In general, fluid B (the 30% methanol mixed with 70% KCl water) showed the most consistent inhibition to the four shale samples tested. In all cases fluid A (fresh water) was most damaging. Results are varied, and cannot be adequately analyzed until all complementary shale information is obtained. This will be discussed in the final report.

Of the four shales tested the 5300' shale is most reactive to the fluids tested. When placed in fresh water it became swollen 3 to 6 times more so than the other shales.

Future Work

Swelling tests will be run on the other four shale samples submitted. These shales were not preserved when received therefore special efforts are being made to restore them to a constant state of dryness before they are tested.

X-ray diffraction and water adsorption data should be completed by August 15, 1977.

Work reported by,

Martin E. Chenevert, J.C.

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Table 1: Swelling data for Shale from Columbia Gas Well No. 20338, Wise County, West Virginia

Fluid Type	Linear % Swelling After 8 Hours			
	Depth of Shale			
	5369' Middle Brown	5300' Middle Gray	4933' Upper Gray	4917' Upper Gray
A. Fresh Water	.055	.65	.15	.175
B. 30% (Methanol) + 70% (Water + 2% KCl)	.055	.455	.12	.12
C. 2% KCl water	.041	.482	.16	.14
D. Fresh Water + 7 gal. HC-2/1000 gal. + ½ gal. Cla-Sta/1000 gal. (W-212)	.055	.66	.08	.16
E. Fresh Water + 2 gal. F-75/100 gal. + 5 gal. L-42/1000 gal.	.055	.54	.11	.17

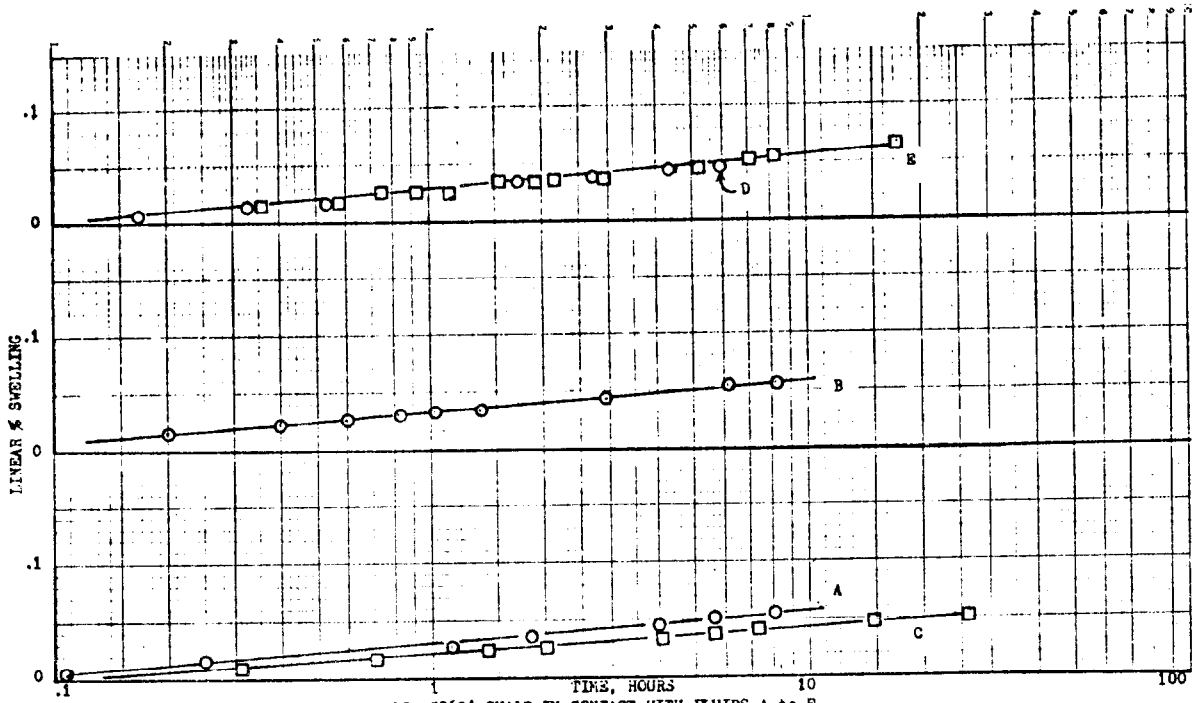

Figure 1, SWELLING-TIME CURVES, 5369' SHALE IN CONTACT WITH FLUIDS A to E

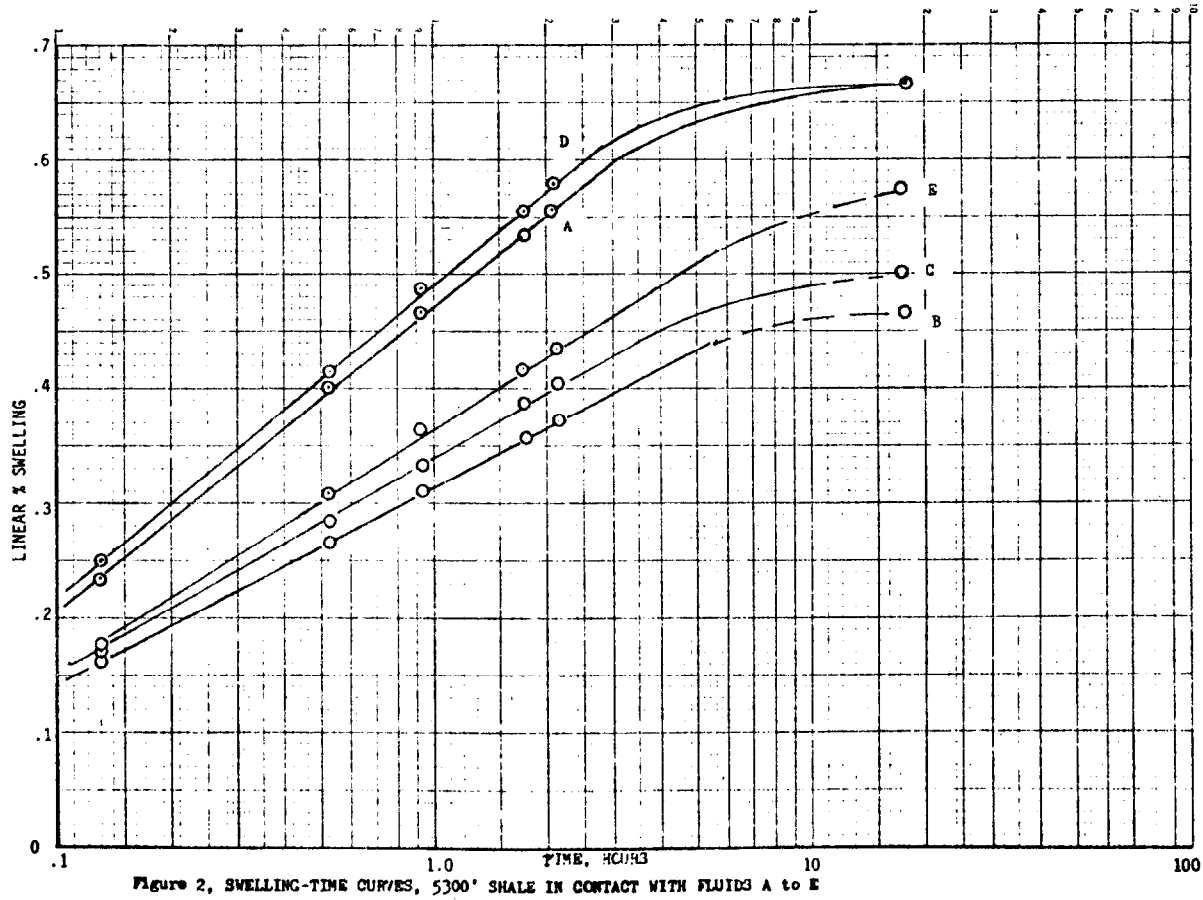

Figure 2, SWELLING-TIME CURVES, 5300' SHALE IN CONTACT WITH FLUIDS A to E

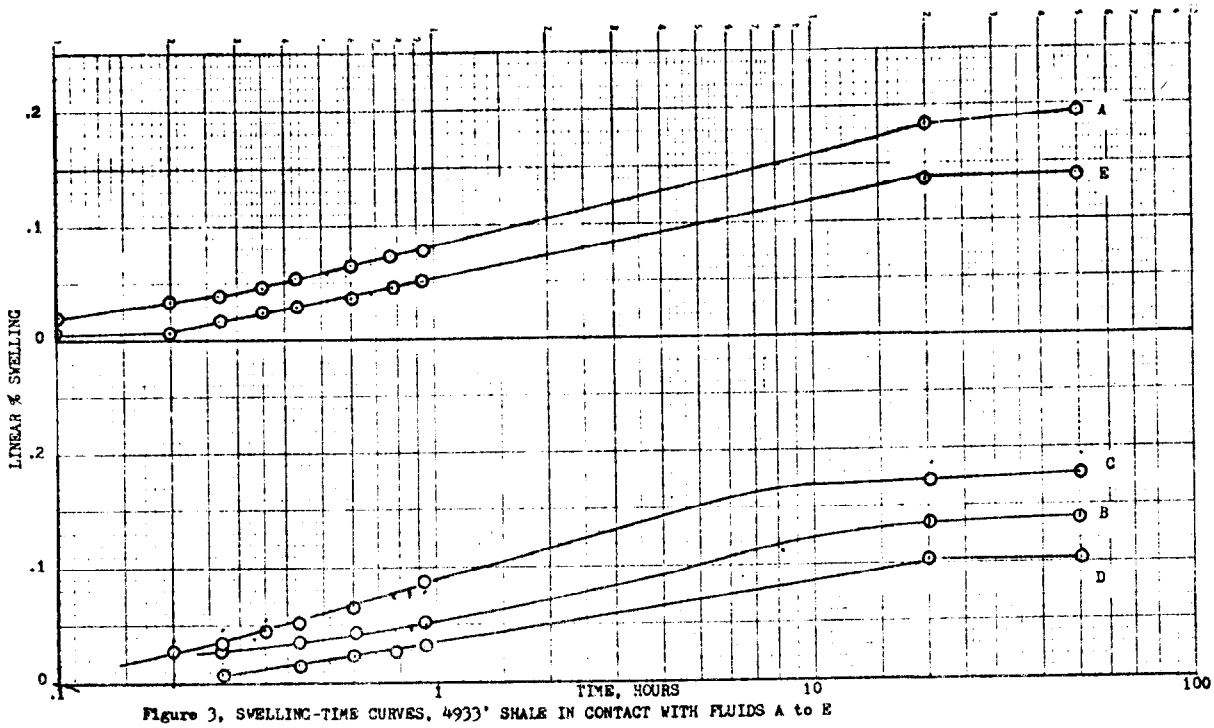

Figure 3, SWELLING-TIME CURVES, 4933' SHALE IN CONTACT WITH FLUIDS A to E

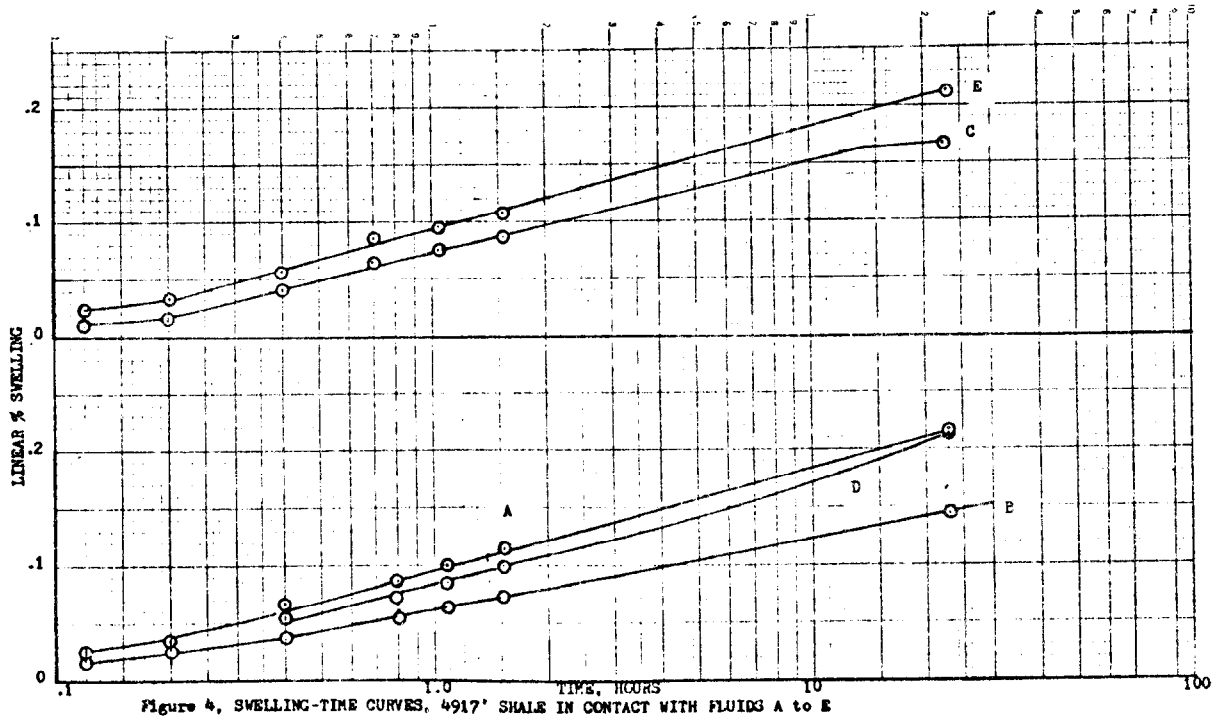

Figure 4, SWELLING-TIME CURVES, 4917' SHALE IN CONTACT WITH FLUIDS A to E

Devonian Shale - Fracturing Fluids Studies

Progress Report No. 3

August 1, 1977

Testing

Swelling tests were run on the 3896', 3458', 3027', 3007', 2763' and the 2740' shales in contact with the various fluids listed in Table 2.

Shale Sample Preparation

All shales tested were un-preserved samples which had experienced some weathering. Because of this it was necessary to "normalize" the shales to a fixed adsorption potential before testing. This was done by placing the shale test specimens in a controlled humidity environment (R.H.=75%) for 30 days.

Results

Of the six shale zones tested the 2740', 2763', 3007' and the 3027' shales showed a high sensitivity to water. As in previous tests the methanol-KCl water fluid mixture showed the largest reduction in swelling response. Also shown in Figures 6 and 7 is the zero swelling response of kerosene.

Figure 9 presents data for the 2763' and 3007' shales in water. Because of the fragile nature of these shale samples more tests could not be run.

Future Work

X-ray diffraction and water adsorption isotherms are being developed.

Work reported by,

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Table 2: Swelling data for shale samples from Columbia Gas Well No. 20402, Lincoln County, West Virginia

Fluid Type	Linear % Swelling After 8 Hours Depth of Shale					
	3896'	3458'	3027'	3007'	2763'	2740'
A. Fresh Water	.072	.05	.34	.66	.275	.62
B. 30% (Methanol + 70% (Water + 2% KCl)	.055	.055	.08	--	--	.18
C. 2% KCl	.045	.07	.18	--	--	.36
D. Fresh Water + 7gal HC-2/1000 gal + ½ gal Cla-Sta/1000 gal	.07	.05	.32	--	--	.57
E. Fresh Water + 2 gal F-75/100 gal + 5 gal L-42/1000 gal	.055	.062	.39	--	--	.72
F. Kerosene	--	.005	.005	--	--	--

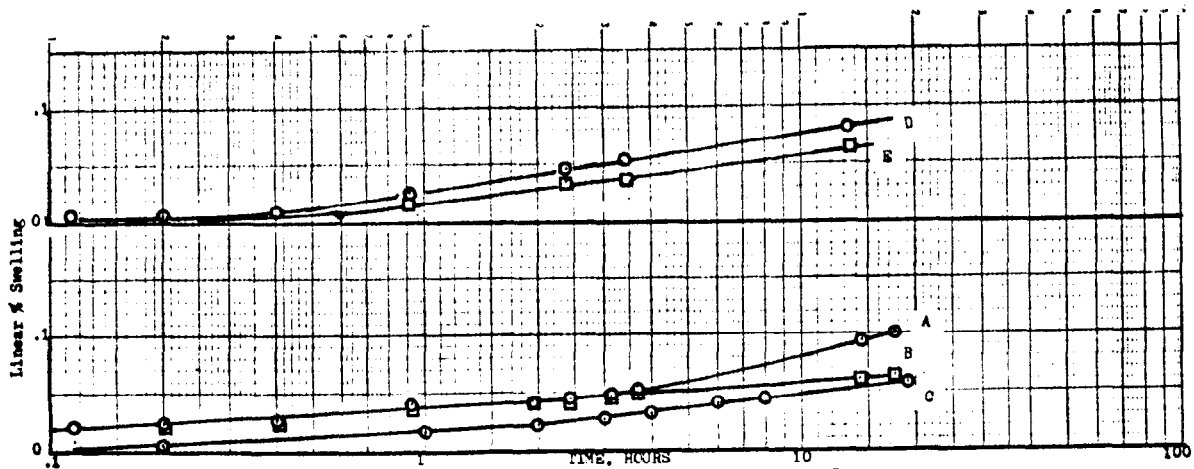

Figure 5, SWELLING-TIME CURVES, 3896' SHALE IN CONTACT WITH FLUIDS A to E.

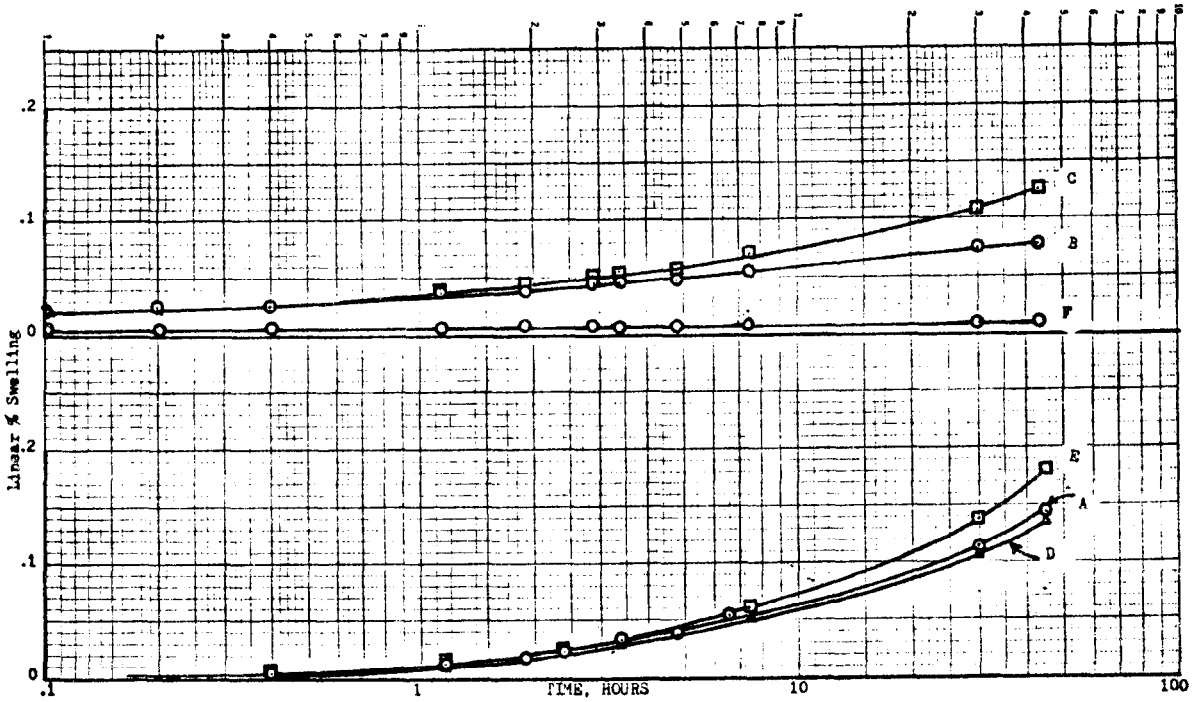

Figure 6, SWELLING-TIME CURVES, 3458' SHALE IN CONTACT WITH FLUIDS A to F.

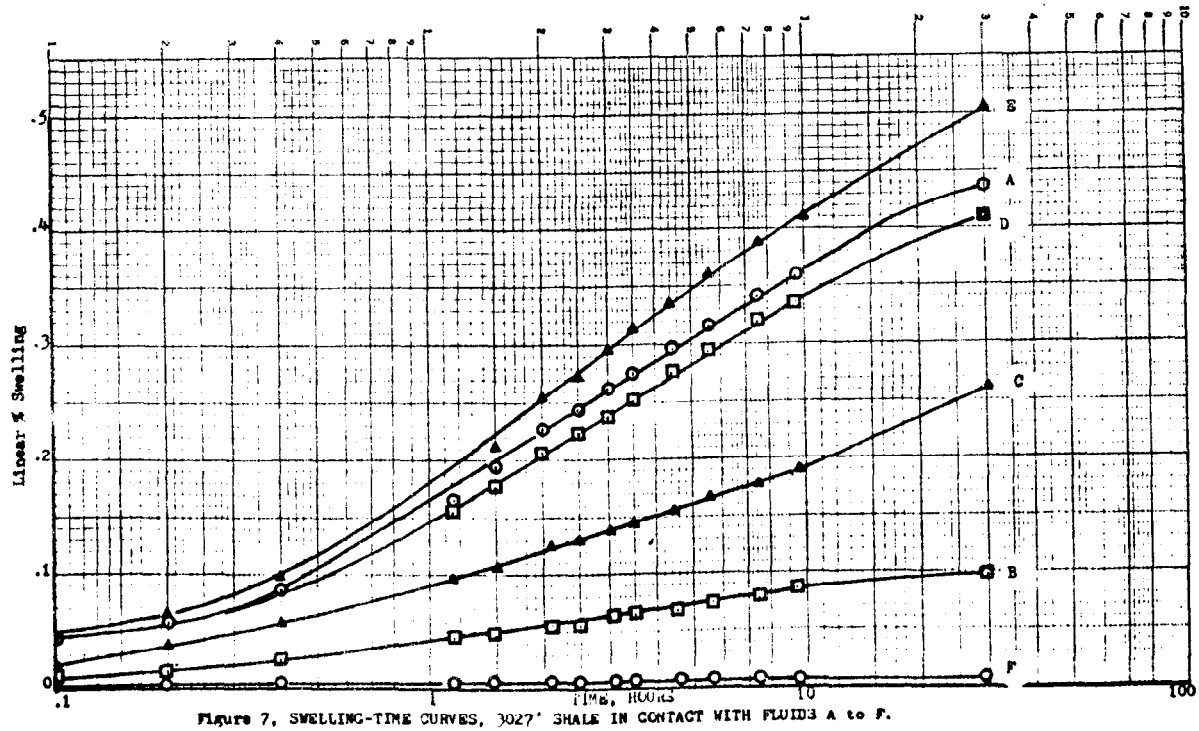

Figure 7, SWELLING-TIME CURVES, 3027' SHALE IN CONTACT WITH FLUIDS A to F.

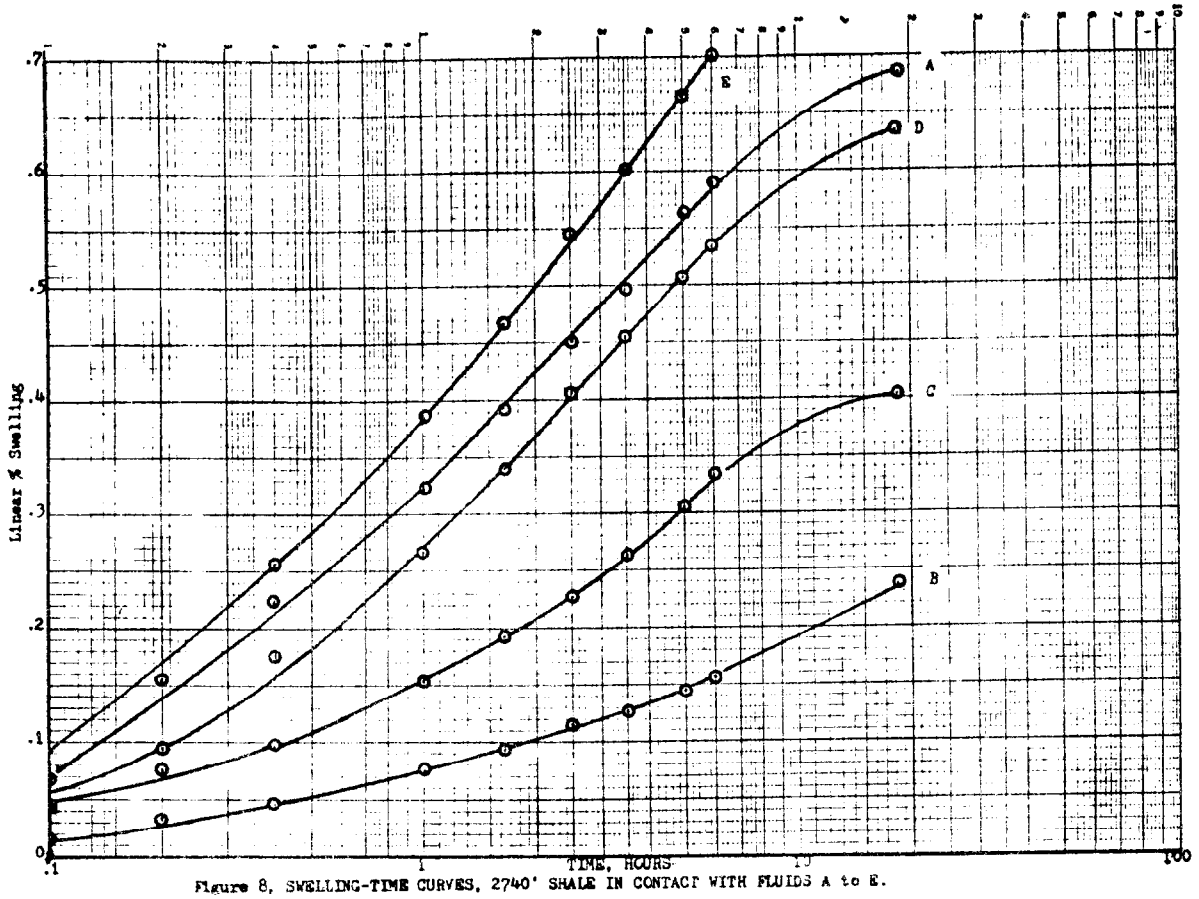

Figure 8, SWELLING-TIME CURVES, 2740' SHALE IN CONTACT WITH FLUIDS A to E.

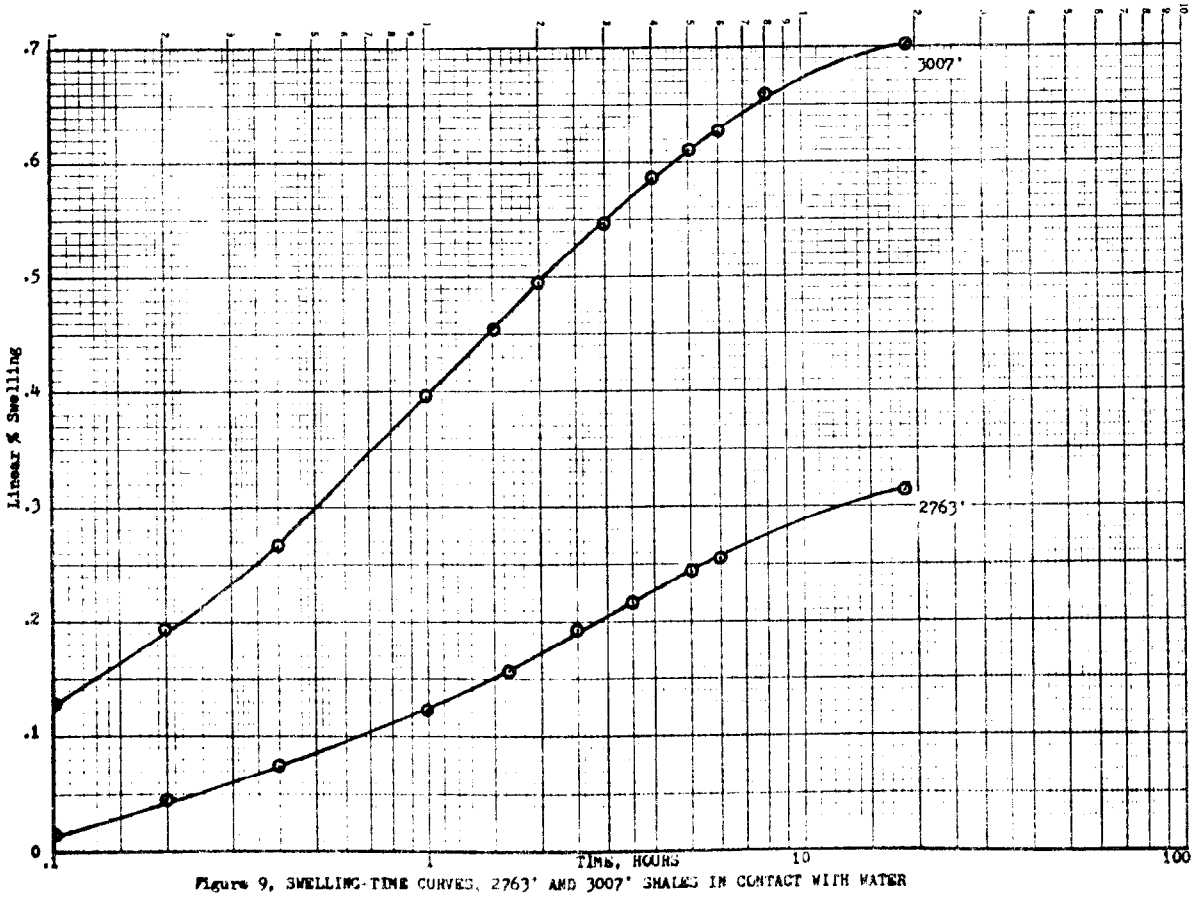

Figure 9, SWELLING-TIME CURVES, 2763' AND 3007' SHALES IN CONTACT WITH WATER