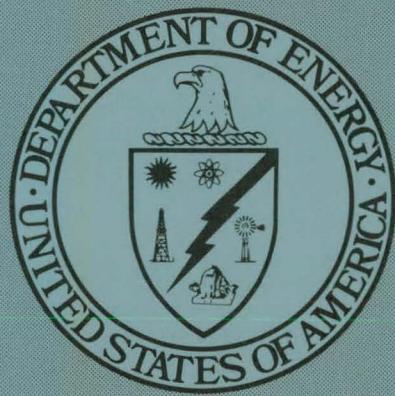


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**SIMULATED IN SITU RETORTING OF OIL SHALE  
IN A CONTROLLED-STATE RETORT**

**III. Dynamic Oil Film Thickness on Partially  
Retorted and Unretorted Shale**

By  
John J. Duvall

February 1982

Laramie Energy Technology Center  
Laramie, Wyoming

TECHNICAL INFORMATION CENTER  
UNITED STATES DEPARTMENT OF ENERGY

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ABSTRACT

The amount of oil washed from the partially retorted and unretorted shales from 14 interrupted runs of the controlled-state retort has been used to estimate dynamic oil film thickness on those shales. The data obtained indicated that factors that affect oil viscosity determine oil film thickness. For example, in the heated region of the retort, temperature was the controlling factor for oil film thickness. In the unheated region controlling factors included proximity to the heated region, gas composition and flow rate, retorting advance rate, and breadth of retorting zone. Factors that affected oil composition and thereby increased viscosity, such as increased gas velocity, oxygen in the retorting gas, slower retorting advance rate, and thinner retorting zone, increased oil film thickness. In the unheated region of the retort the oil film was thickest nearest the heated region gradually tapering to a more or less constant value approximately 1 meter from the heated region. Oil shale particle size did not affect oil film thickness.

INTRODUCTION

Retorting of oil shale, a vast natural resource, has received much attention in recent years. In several schemes for retorting oil shale, product oil flows down over partially retorted and unretorted shale. Mathematical models have been developed describing retorting (1,2,3) and should lead to a better understanding of the processes involved and how to maximize energy production. A factor of some importance to these mathematical models is the thickness of the product oil film that exists on the partially retorted and unretorted oil shale below the retorting zone during retorting (see Figure 1). This oil film consists of two parts, a static part that would stay on the shale if it was allowed to drain and a dynamic part that is flowing down over the shale. This paper uses data from interrupted runs (4) of the controlled-state retort

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<sup>1</sup> Research Chemist

(CSR), to estimate the dynamic oil film thickness. Factors that affect the viscosity of the product oil such as gas flow rate, temperature and retorting gas composition have been shown to affect oil film thickness. A thicker oil film can have the consequence of decreasing oil yield and affecting the composition of the product oil through coking.

## EXPERIMENTAL PROCEDURE

The CSR and its method of operation have been described in detail previously (4,5). Briefly, it is a vertically mounted, 4m long, 7.62 cm ID steel tube that is surrounded by a contiguous series of electric heaters that are nominally 15.2 cm long (see Figure 2). Temperatures are measured in a 2.54 cm steel tube that is concentric to the outside tube. Experiments are conducted by filling the annulus between the tubes with crushed, sieved oil shale and then successively turning on the heaters starting with the top heater. Input gas flows down through the retort during the experiment.

The oil shale used in the experiments reported here was obtained from the Department of Energy facility near Rifle, Colorado. Four separate batches of shale were crushed and screened into the desired size ranges to yield the material used. A 18 kg sample of each shale batch was screened into 0.32 cm cuts to determine the size distribution within a batch. Data pertaining to the batches of raw shale are shown in Table 1. Several sets of retorting conditions were used and they are shown in Table 2.

For an interrupted experiment, the retorting zone was allowed to pass part way down the shale bed and then the retort was shut down, laid on its side and water was passed through the center tube and over the outside tube to quickly cool the retort and stop the retorting and the movement of the product oil down the retort. The retort was then cut into sections that corresponded to the 15.2 cm heaters and the oil coated shale samples obtained were maintained separately. The oils coating the surface of the shale and the retort tubes of each section were washed from the shale with cyclohexane and the dried, washed shale was weighed. Solvent was evaporated from aliquots of each of the oil solutions and the residual oil was weighed to determine the total amount of oil in each section.

The oils collected at the bottom of the CSR were analyzed through Hempel distillation and the specific gravities of the oils and each of the distillation fractions were determined. These data along with the weights of oil washed from the shale and retort tubes were used to estimate the volume of oil washed from each section of retort. The oil volume was used with the combined surface area of the retort tubes and shale to calculate the oil film thickness in each oil wet section of the retort. The method used to calculate shale surface area and the data used for the calculation of surface areas, oil film thicknesses, etc. for the 14 experiments are shown in the appendix of this report.

## RESULTS AND DISCUSSION

The data on oil film thickness on partially retorted and unretorted, unheated oil shale are shown in Tables A3 through A17 in the appendix and in the next several figures. There are two groupings of data, one from the heated region of the retort and the unheated region of the retort. Within each of the two general groupings are smaller groupings depending on retorting parameters.

### Heated Region of the Retort

A statistical analysis of the data of Tables A3 through A17 for those zones of the CSR located in heated region of the CSR is shown in Table 3. An analysis of the slopes and y intercepts shows two general groupings plus values from two experiments that do not fit into either grouping. The first group includes CSR 14, 17, 28, 31, 32, 58, and 60, the experiments run in  $N_2$  atmosphere. The second grouping includes CSR 23, 25, 26, 27, and 37, the three experiments run in a  $N_2/O_2$  atmosphere plus an experiment run at a high flow rate relative to other retorting parameters (CSR 23) and an experiment using a very slow heating rate. The two experiments that do not fit into either grouping are CSR 19, an experiment done with a wide retorting region and CSR 33, an experiment done with smaller shale pieces than usual.

Figure 3 shows the data for the first grouping, most of the  $N_2$  atmosphere experiments, and Figure 4 shows the data for the second grouping of experiments. Least squares analysis gave the heavy line on each figure while statistical analysis (6) gave the area between the lighter lines, i.e., variance in y. The statistical analysis shows the variance in y, oil film thickness, to be  $\pm 16\%$  in Figure 3 and  $\pm 13\%$  in Figure 4. The data show a temperature dependence for oil film thickness in the heated region of the retort. The difference between the two major groupings is most likely a consequence of retorting conditions that yielded oils with different viscosities. Indeed, previous work (7) has shown a smaller amount of low boiling materials in the oils washed from the shale of the  $N_2/O_2$  experiments than in the  $N_2$  experiments; this would cause a higher viscosity which would give thicker oil films. Also, CSR 23 was conducted with a relatively high gas flow rate which would cause lower boiling materials to be swept away, again giving higher viscosity and thicker oil films. CSR 37 was conducted at a very slow heating rate, retorting advance rate, etc. (8); that meant that the oil films on the shale were exposed to gas flow for much longer periods of time, again causing lower boiling materials to be swept away resulting in higher viscosities and thicker oil films.

### Unheated Region of the Retort

The data for the unheated regions of the retort show a broader dependence of oil film thickness on retorting conditions than do the data for the heated regions of the retort. Examples are given in the following paragraphs and figures. The figures show oil film thickness for those sections of the retort whose final or maximum temperature was 100°C or less plotted versus zone of the retort (left to right on the figures reads top to bottom of the retort) with zone 3 on each figure corresponding to the highest unheated zone of the retort. The data for the lowest zone of the retort was left off the figures because of end effects.

Data are shown in figure 5 for two experiments for which all retorting parameters were the same except nitrogen flow rate (7.3 scmh/m<sup>2</sup> for CSR 17 and 68.9 scmh/m<sup>2</sup> for CSR 23). The oil film for CSR 23 was considerably thicker than that for CSR 17, probably because the higher nitrogen flow rate swept away the lower boiling materials leaving a more viscous oil on the surface of the shale as suggested previously (4). Also, the oil film thickness was greatest in the region of the retort immediately below the heated region gradually thinning to a more or less constant value further down the retort (best shown in the data for CSR 17 which had the longest unheated region of any of the experiments).

The data in Figure 6 are from experiments conducted with different retorting advance rates (30.5 cm/hr for CSR 19 and 7.6 cm/hr for CSR 58 and CSR 60. The latter two were duplicate runs). These data show that broadening of the retorting region from approximately 15 cm to 30 cm (4) decreased the oil film thickness by a factor of around two (as can be estimated from Figure 6). Again it can be seen that the oil film thickness was greatest in the region of the retort immediately below the heated region.

Figure 7 shows data for experiments in which the retorting advance rates (1.5 cm/hr. for CSR 17 and 7.6 cm/hr for CSR 58 and 60) and heating rates (0.22°C/min. for CSR 17 and 1.1°C/min. for CSR 58 and 60) were different. The oil film near the bottom of the retort was somewhat thinner in CSR 17 than the average of CSR 58 and 60. However, two retorting parameters were varied between the experiments and either could have been the major factor. Similar data are presented in Figure 8 for experiments conducted using the same retorting conditions as in Figure 5 (CSR 25 the same as CSR 17 and CSR 26 the same as CSR 58 and 60) except a N<sub>2</sub>/O<sub>2</sub> atmosphere was used instead of N<sub>2</sub>. Again, the data taken at the slower retorting advance rate show a thicker oil film but whether heating rate or retorting advance rate had the major effect is inconclusive except for agreement with the conclusions drawn from Figure 6.

Figures 9 and 10 compare oil film thickness data for sets of experiments conducted using the same retorting conditions, within sets, except the retorting atmosphere was either N<sub>2</sub> or N<sub>2</sub>/O<sub>2</sub>. The data indicate that, in general, experiments conducted in a N<sub>2</sub>/O<sub>2</sub> atmosphere had a thicker oil film than experiments conducted in a N<sub>2</sub> atmosphere.

Data on the effect of shale particle size on oil film thickness are shown in Figures 11-13. The data were taken from three pairs of experiments, where the shale sizes were 1) -0.95, +0.12 cm; 2) -1.3, +0.32 cm; or 3) -1.9, +0.95 cm. Each of the six experiments was conducted using the same retorting conditions (see Table 2). The lines in Figures 12 and 13 are averages determined through polynomial regression analysis; however, the data for the two experiments depicted in Figure 11 were too different to be analyzed in that manner.

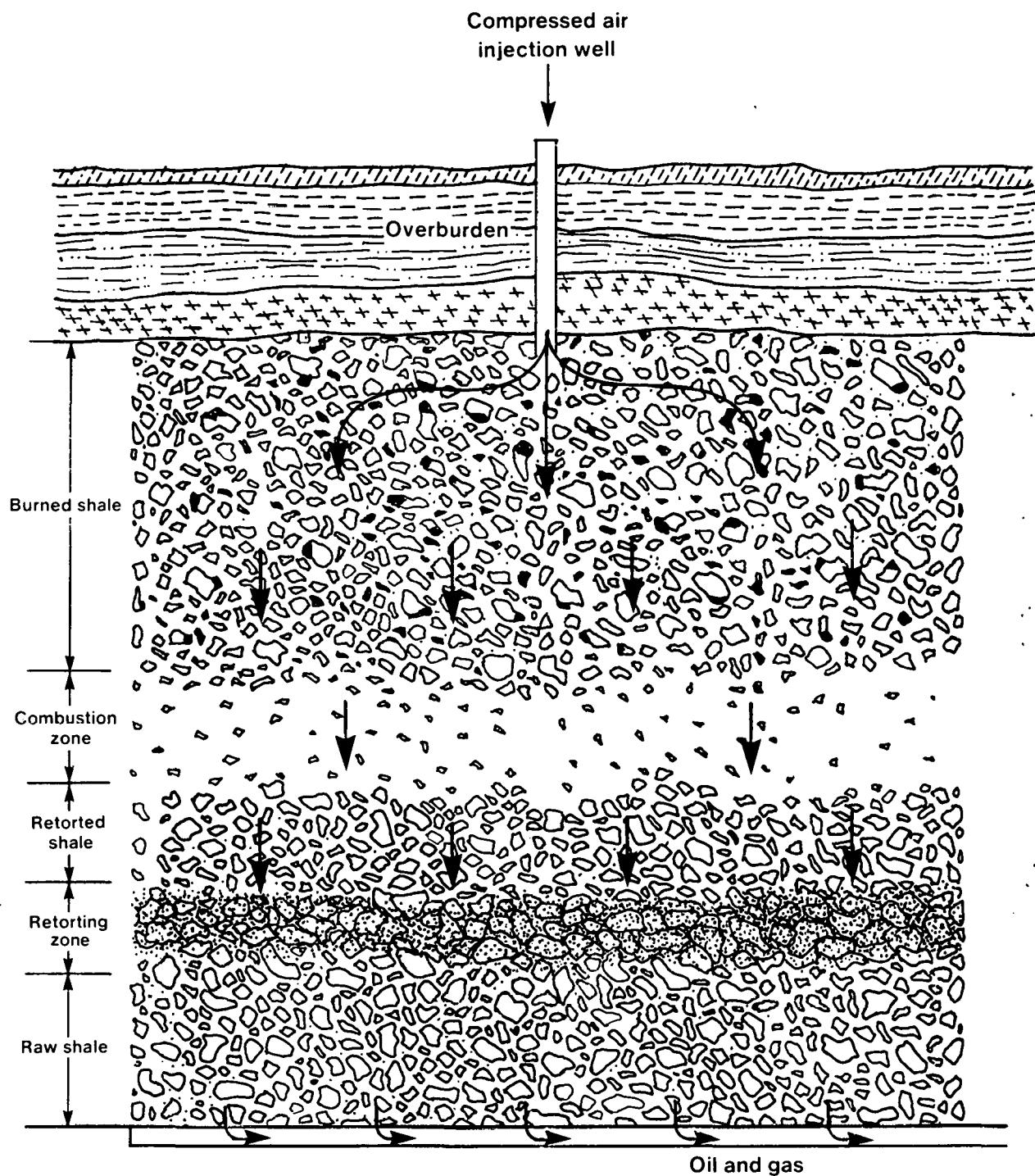
In fact, the data for five of these experiments showed that within experimental error, shale particle size had no effect on oil film thickness for the range of particles studied in this work. Further analysis of the data for CSR33 showed that blockage had occurred in the retort near the retorting zone. For example, the periodic gas analyses performed during the experiment showed very low nitrogen values, at or near 0 percent, during the last third of the experiment. This would indicate blockage in the retort causing the nitrogen to leak out of the retort probably through the top gasket. This blockage apparently stopped oil flow below the retorting zone allowing most of the oil to drain from the unretorted shales which gave low values for oil film thickness. Further evidence for the blockage was the fact that overall oil yield (combined receiver oil and oils washed from shale) from CSR 33 was 17 percent lower than that from CSR 32. The oil produced that was prevented from flowing down the retort coked in the retorting zone. Evidence for this is shown in Table 4 where organic carbon analyses of the retorted shales for CSR 32 and CSR 33 are compared. Organic carbon remaining on the retorted shale of CSR 33 was higher than that for CSR 32 indicating that significant coking took place. The coking should have produced hydrocarbon gases in more abundance than usual but the same blockage that caused the coking prevented effective analysis of the gases. The blockage apparent in CSR 33 was an example of how a thicker oil film in the retorting zone could reduce oil yield.

Because oil film thickness appeared to be related to viscosity, an attempt was made to quantify that relationship. Table 5 lists the viscosities at two temperatures for the receiver oils of most of the experiments along with the average oil film thickness in the unheated region of the retort. It should be pointed out that the boiling point composition of the oils washed from the shale usually differed somewhat from that of the receiver oils (4) and, therefore, the viscosities of the oils washed from the shales probably varied somewhat from those of the receiver oils. However, because it was impossible to get an accurate determination of viscosity from the oils washed from the shale, that of the receiver oils was assumed to be a good approximation. The data from Table 5 were plotted in Figures 14 and 15. The lines drawn are the result of least squares analysis (heavy line) and the determination of the variance along the y axis. The data show some scatter but do give approximations for the relationship of viscosity of oil to oil film thickness for two different temperatures.

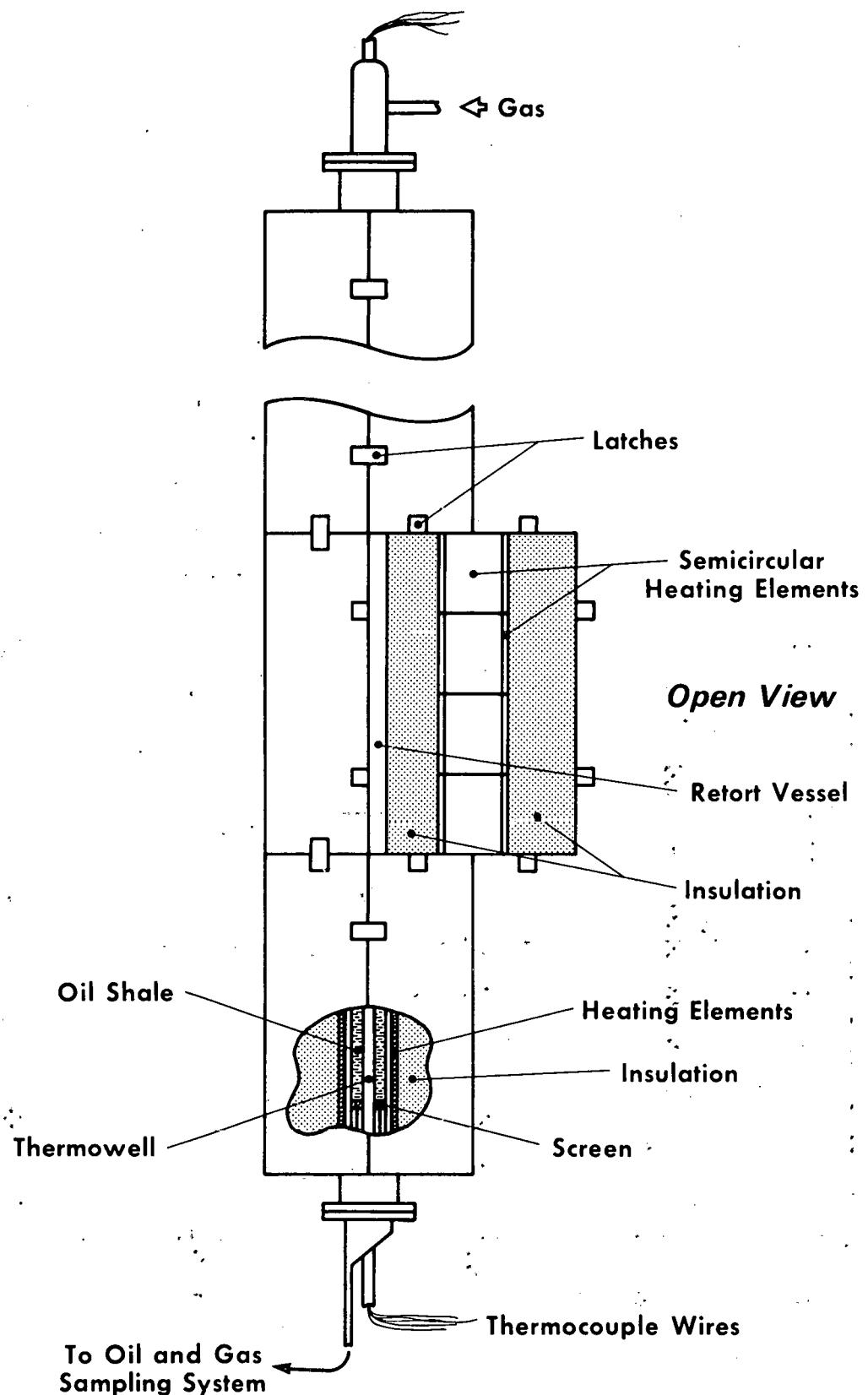
## SUMMARY AND CONCLUSIONS

Fourteen interrupted experiments have been conducted using the CSR and the oil film thicknesses have been calculated for the partially retorted and unretorted shales in the retort. These data show that, in the heated regions of the retort, temperature and retorting conditions were the factors that controlled oil film thickness. In the unheated region of the retort several factors became important including the following:

- 1) distance from the heated region - the oil film was thickest just below the heated region gradually thinning to a more or less uniform thickness in 7-8 15 cm sections;
- 2) retorting gas velocity - a faster gas flow rate resulted in a thicker oil film;
- 3) slower retorting advance rate and smaller retorting zone had the thicker oil film;
- 4) retorting atmosphere - a comparison of oil film thickness between a  $N_2$  atmosphere and a  $N_2/O_2$  atmosphere showed that the latter had the thicker oil film; and
- 5) coking - thicker oil films in the retorting zone can cause more oil coking which lowers the yield of oil.



**Figure 1. Schematic diagram of an in situ oil shale retorting process**



**Figure 2. Controlled-state retort**

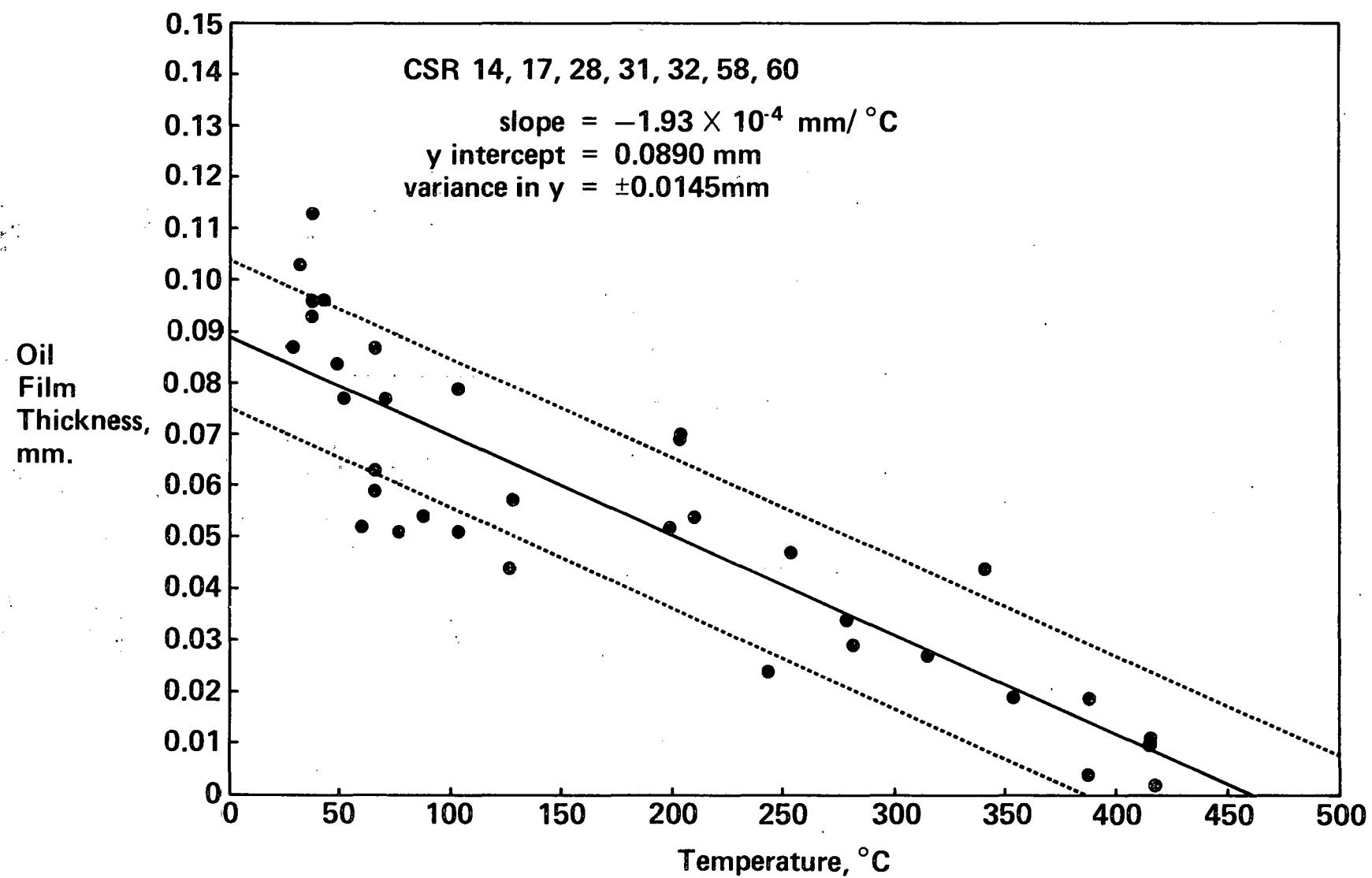


Figure 3. Effect of Temperature on Oil Film Thickness, Group I.

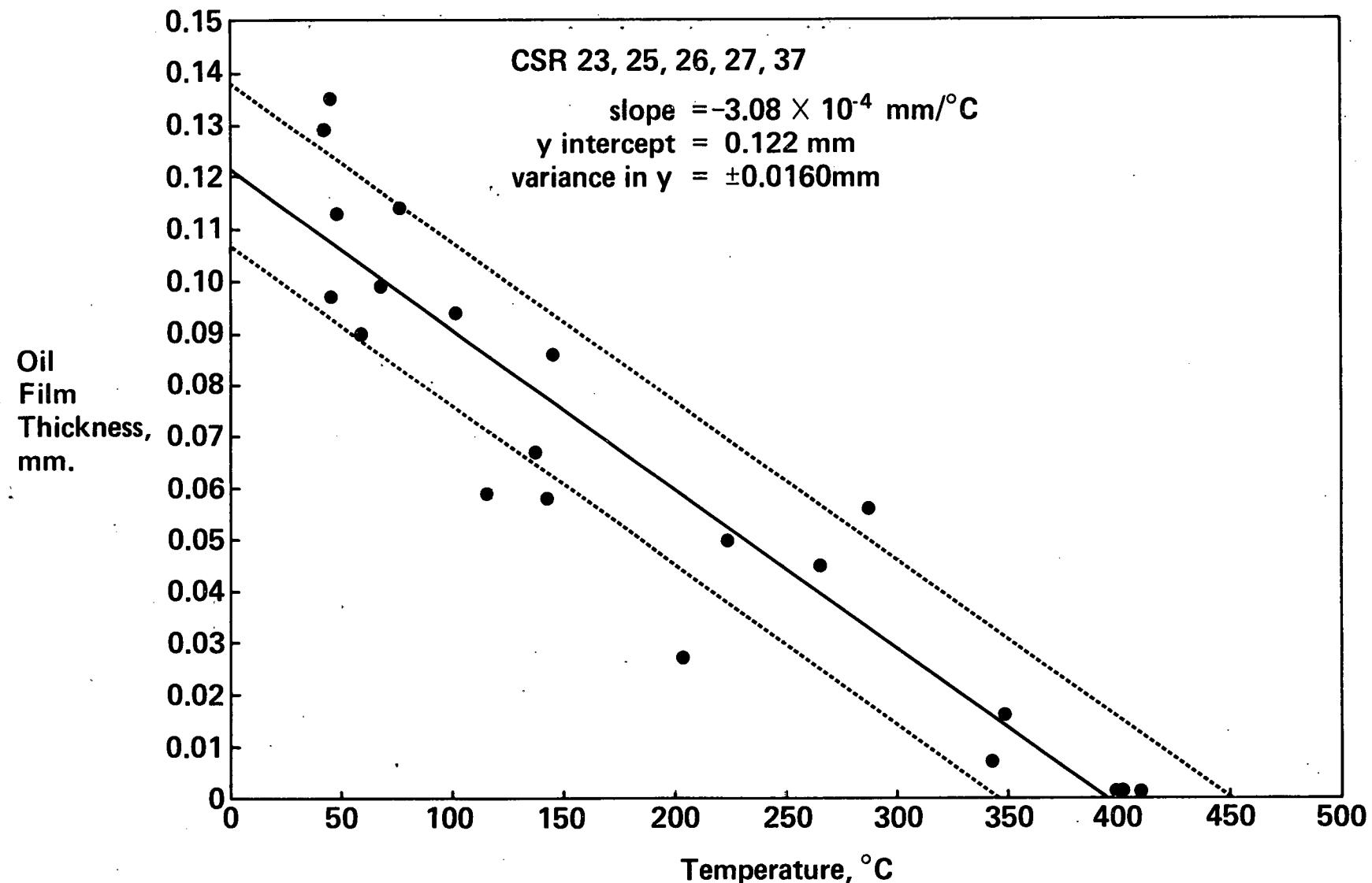


Figure 4. Effect of Temperature on Oil Film Thickness, Group II.

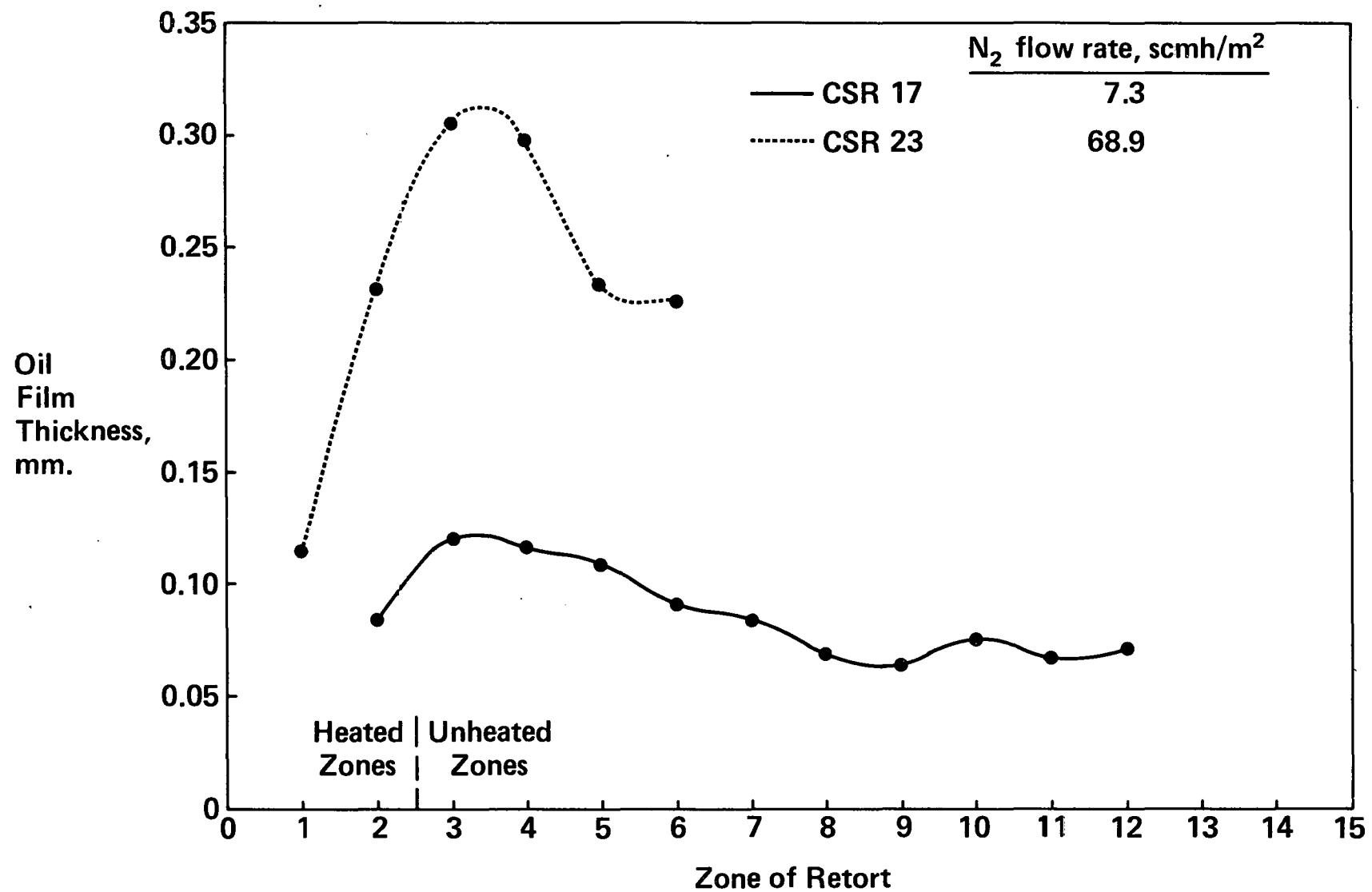


Figure 5. Effect of Gas Flow Rate on Oil Film Thickness.

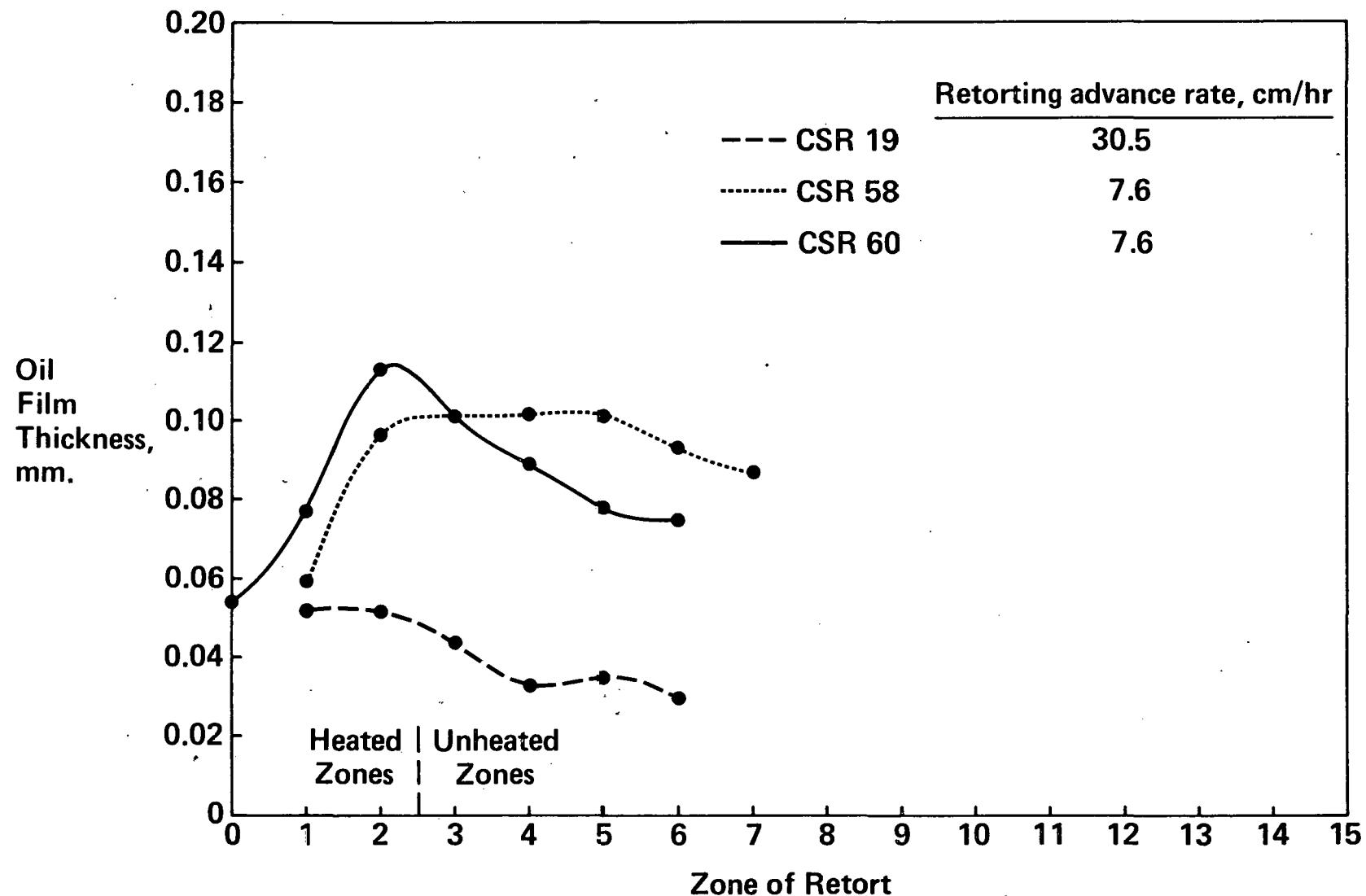


Figure 6. Effect of Retorting Zone Breadth on Oil Film Thickness.

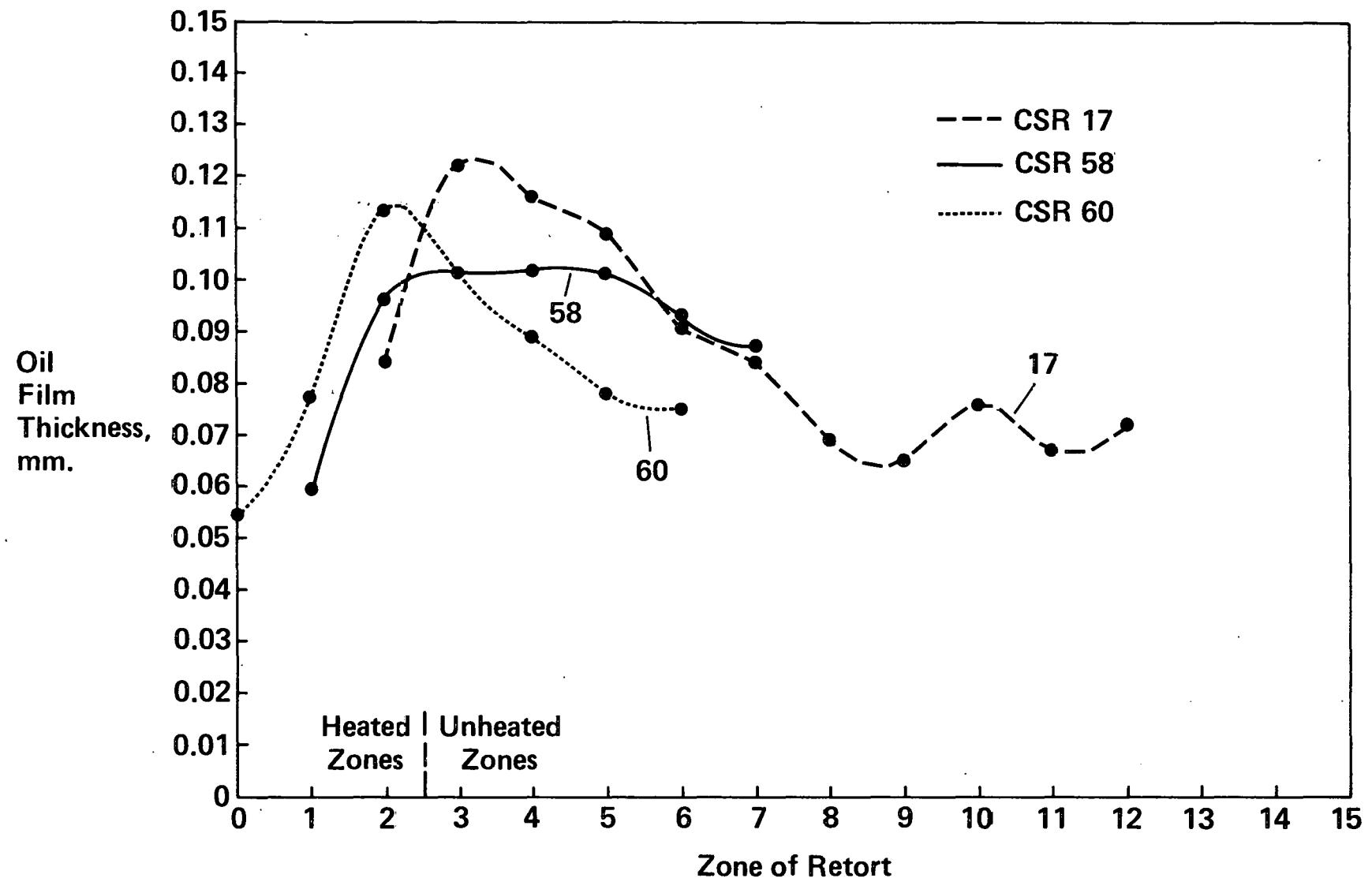


Figure 7. Effect of Heating Rate and Retorting Advance Rate on Oil Film Thickness,  $N_2$  Atmosphere.

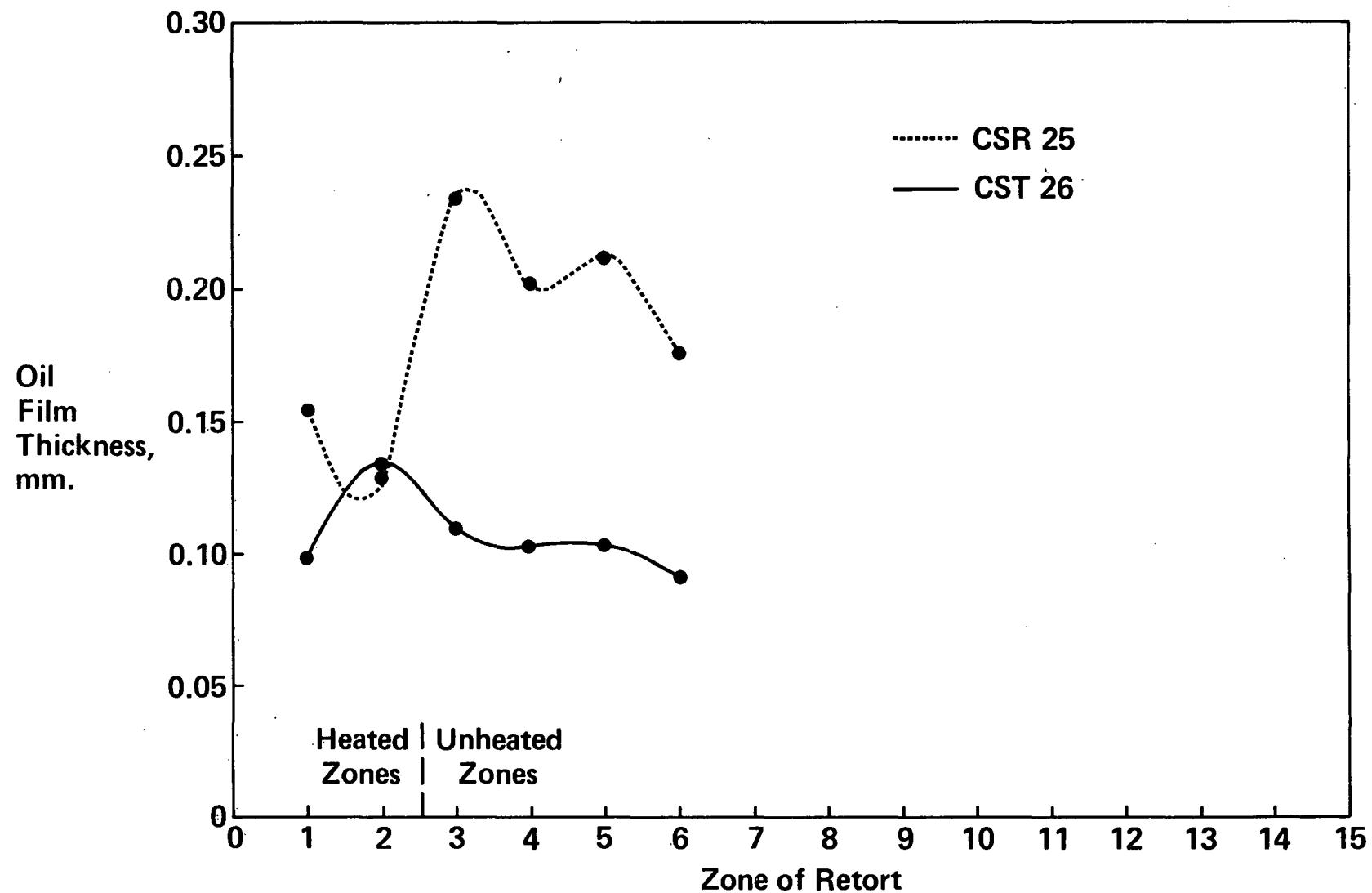


Figure 8. Effect of Heating Rate and Retorting Advance Rate on Oil Film Thickness,  $N_2/O_2$  Atmosphere.

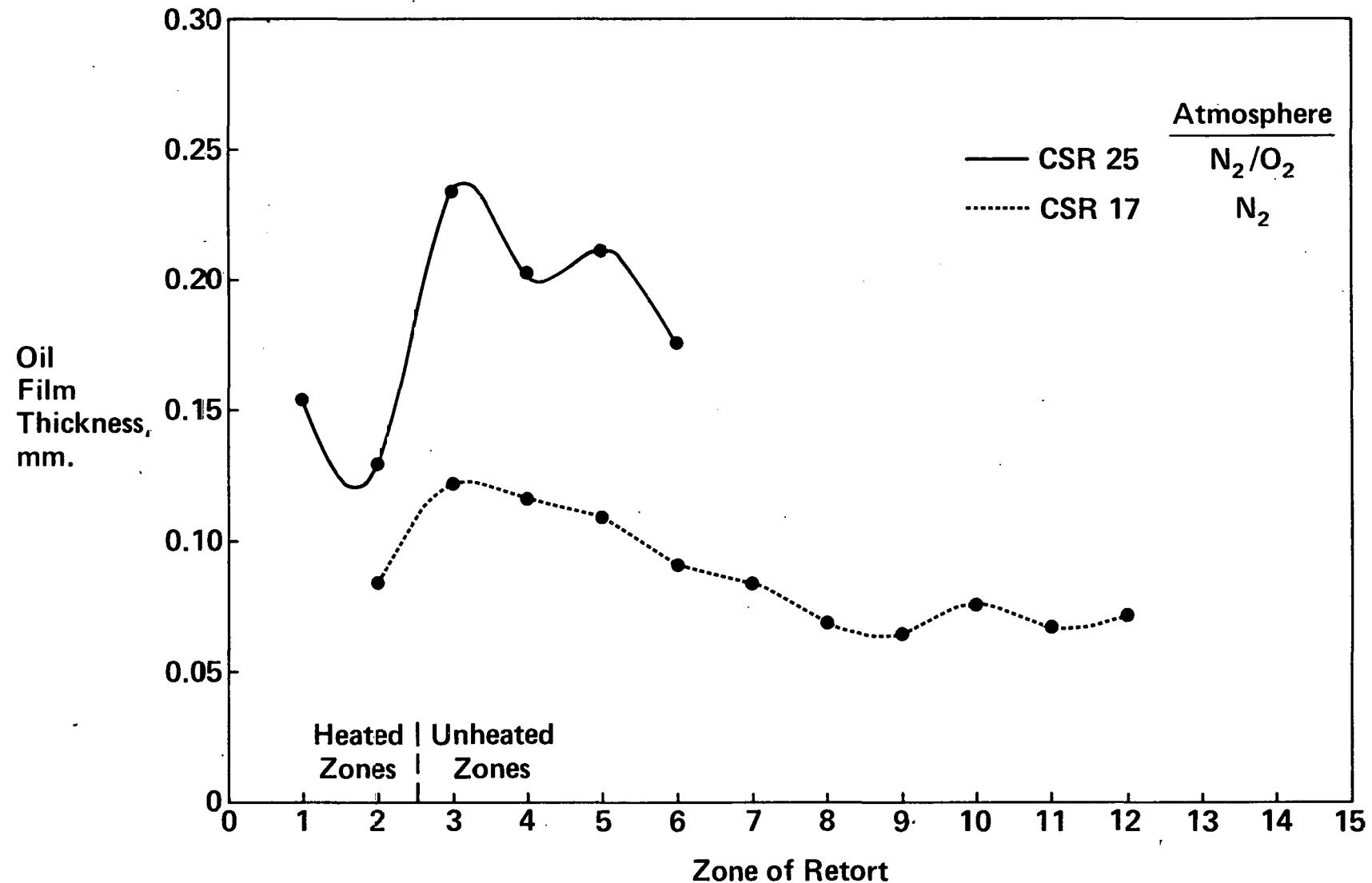


Figure 9. Effect of Retorting Atmosphere on Oil Film Thickness.

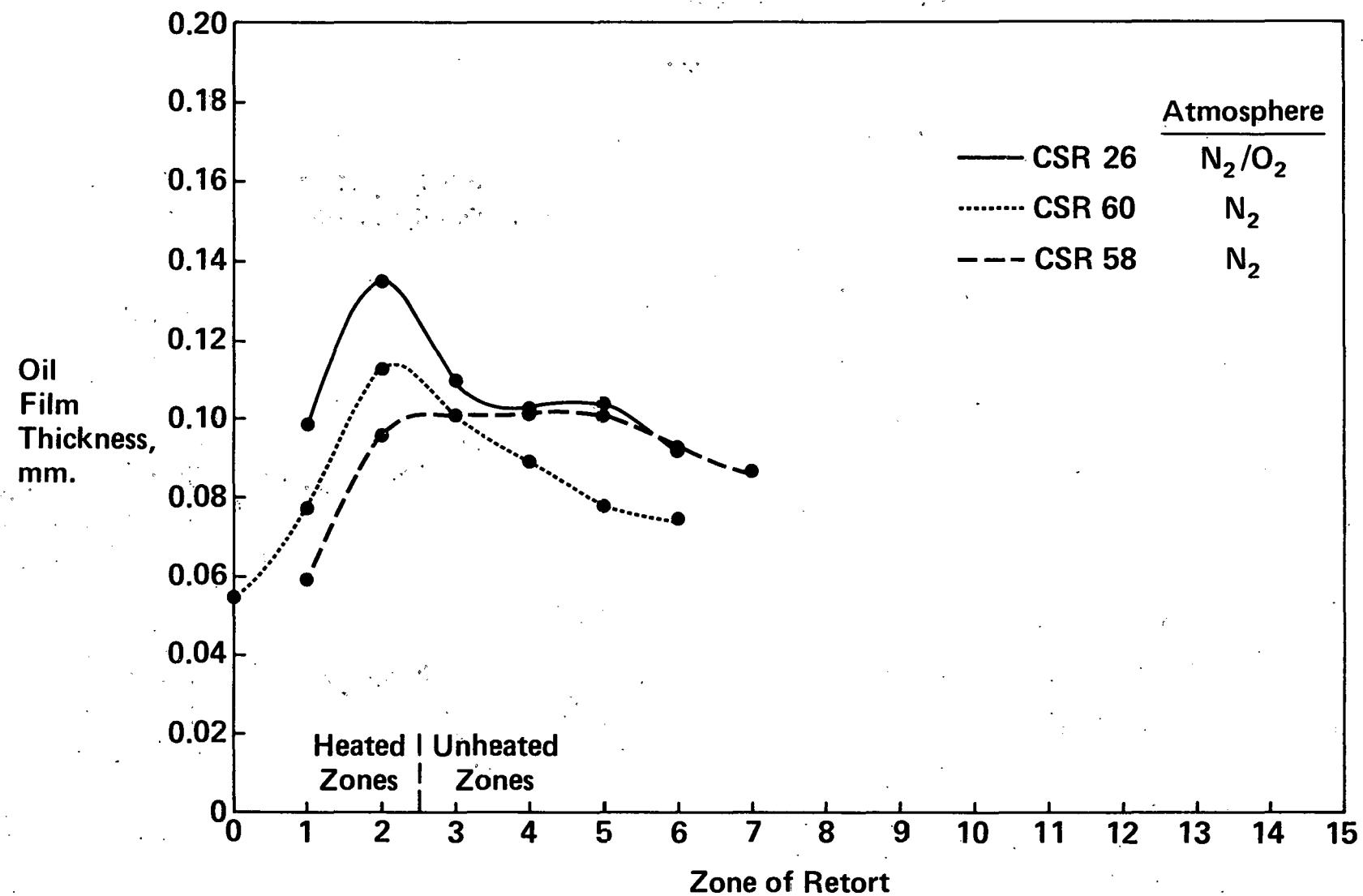


Figure 10. Effect of Retorting Atmosphere on Oil Film Thickness.

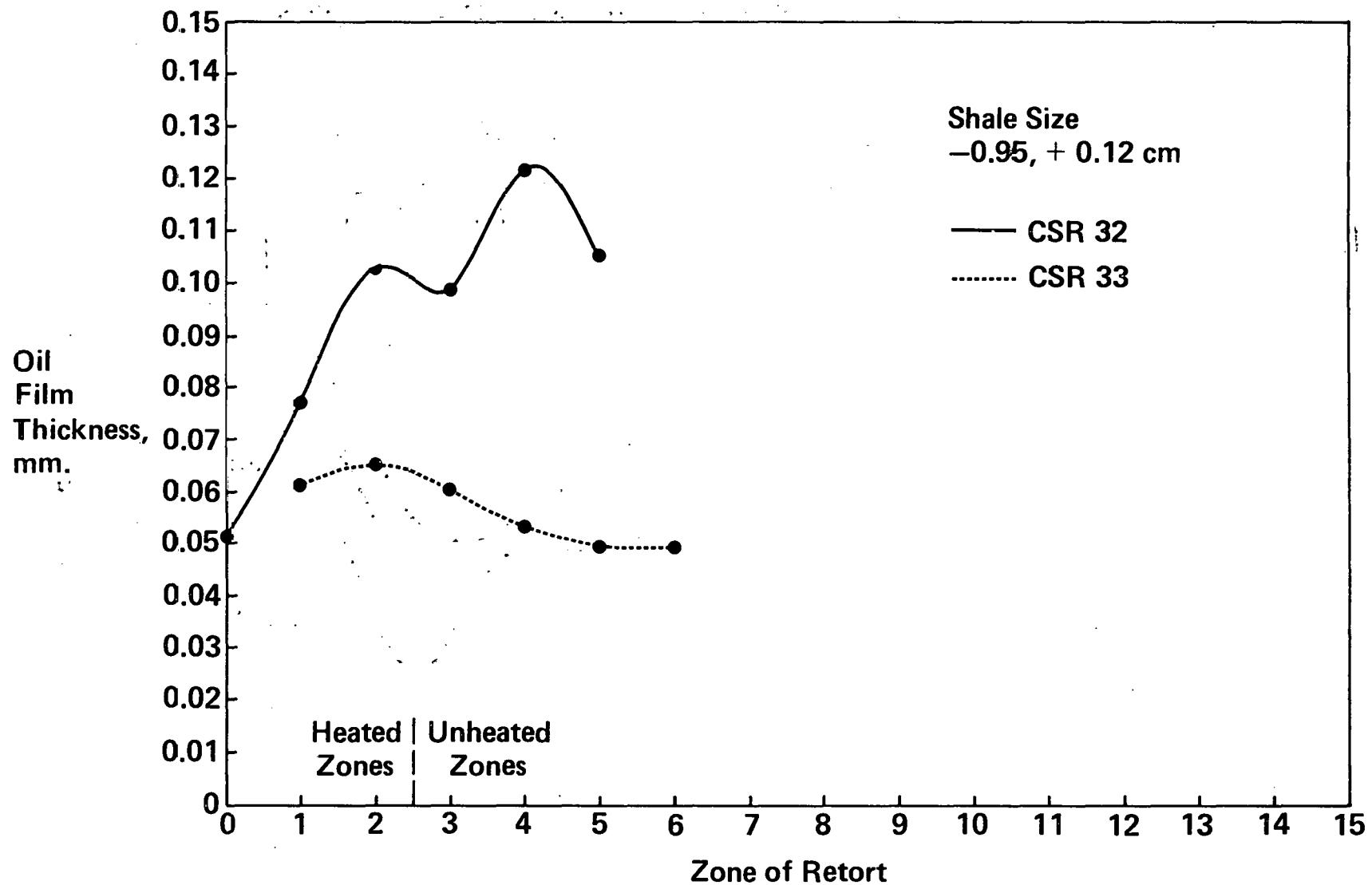


Figure 11. Effect of Oil Shale Particle Size on Oil Film Thickness.

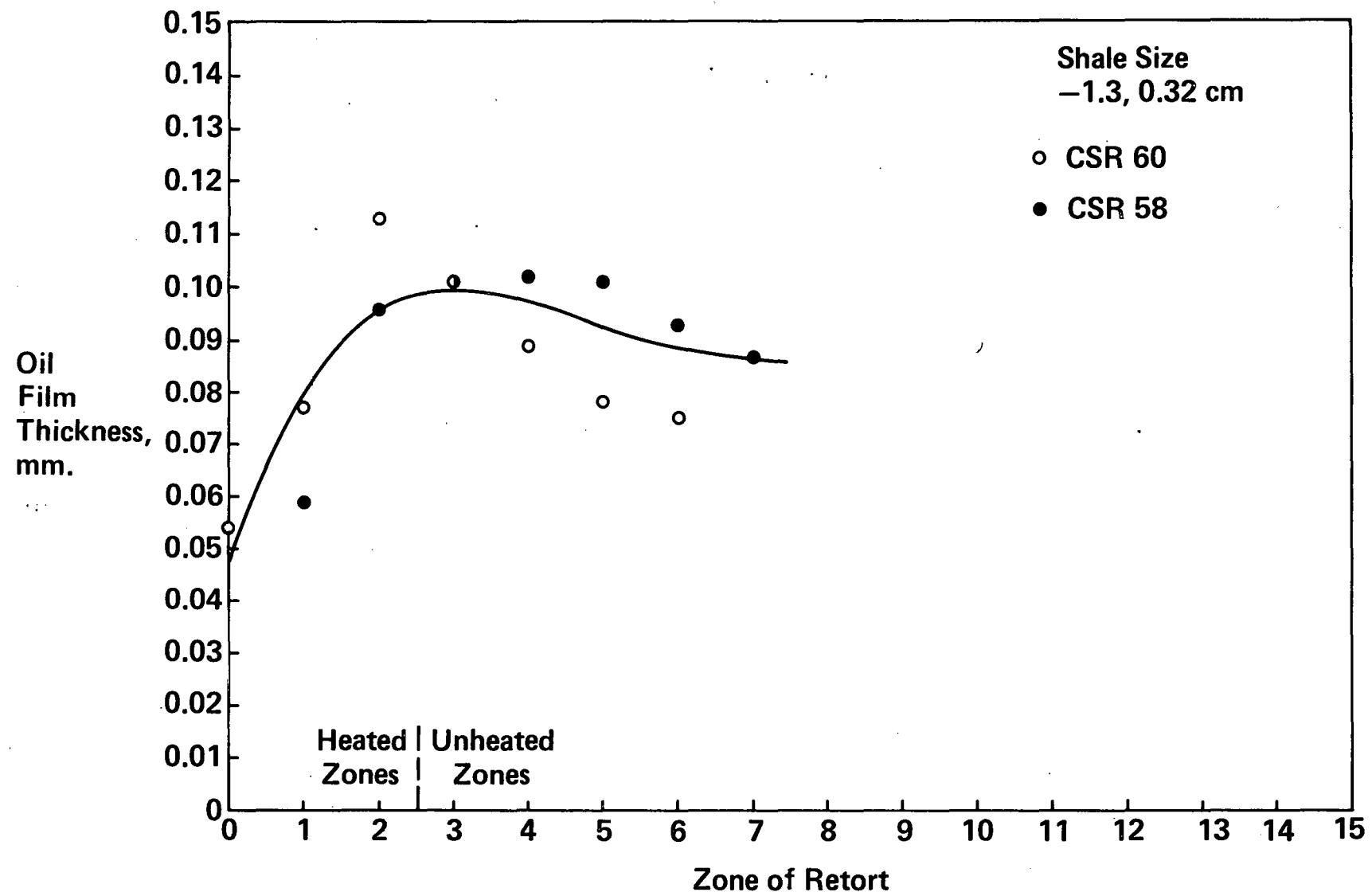


Figure 12. Effect of Oil Shale Particle Size on Oil Film Thickness.

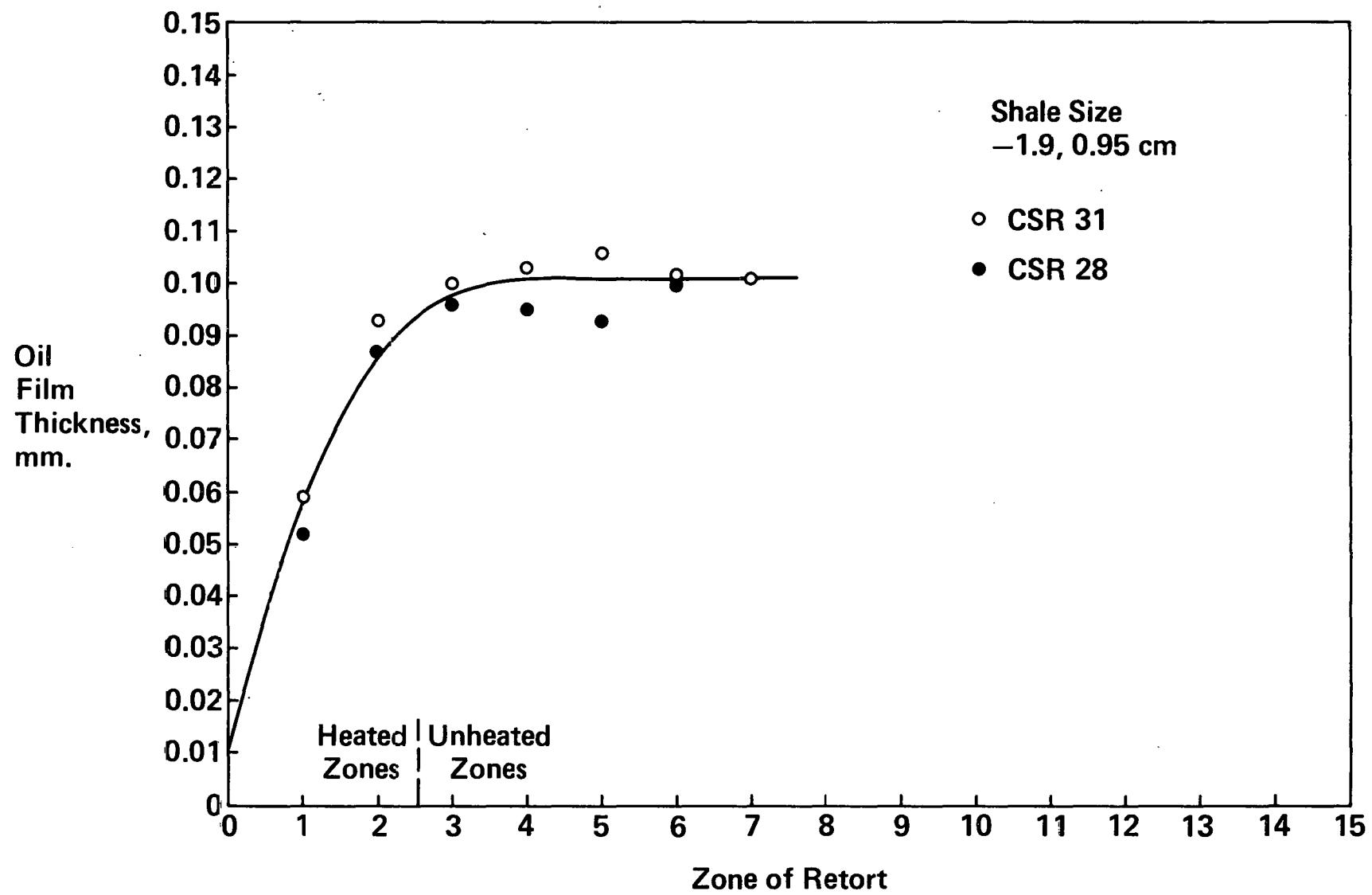


Figure 13. Effect of Oil Shale Particle Size on Oil Film Thickness, C.

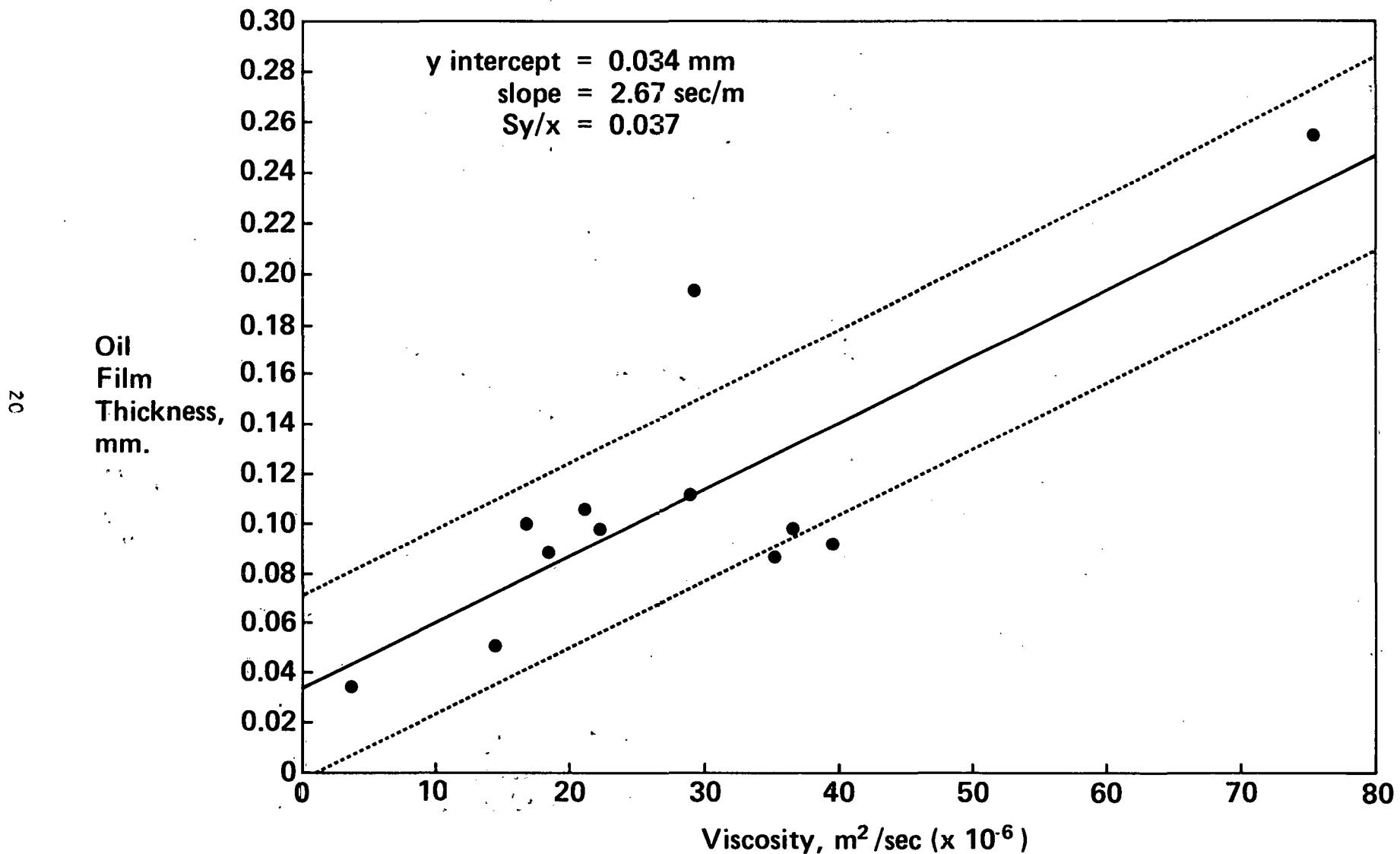


Figure 14. Effect of Viscosity ( $38^\circ\text{C}$ ) on Oil Film Thickness.

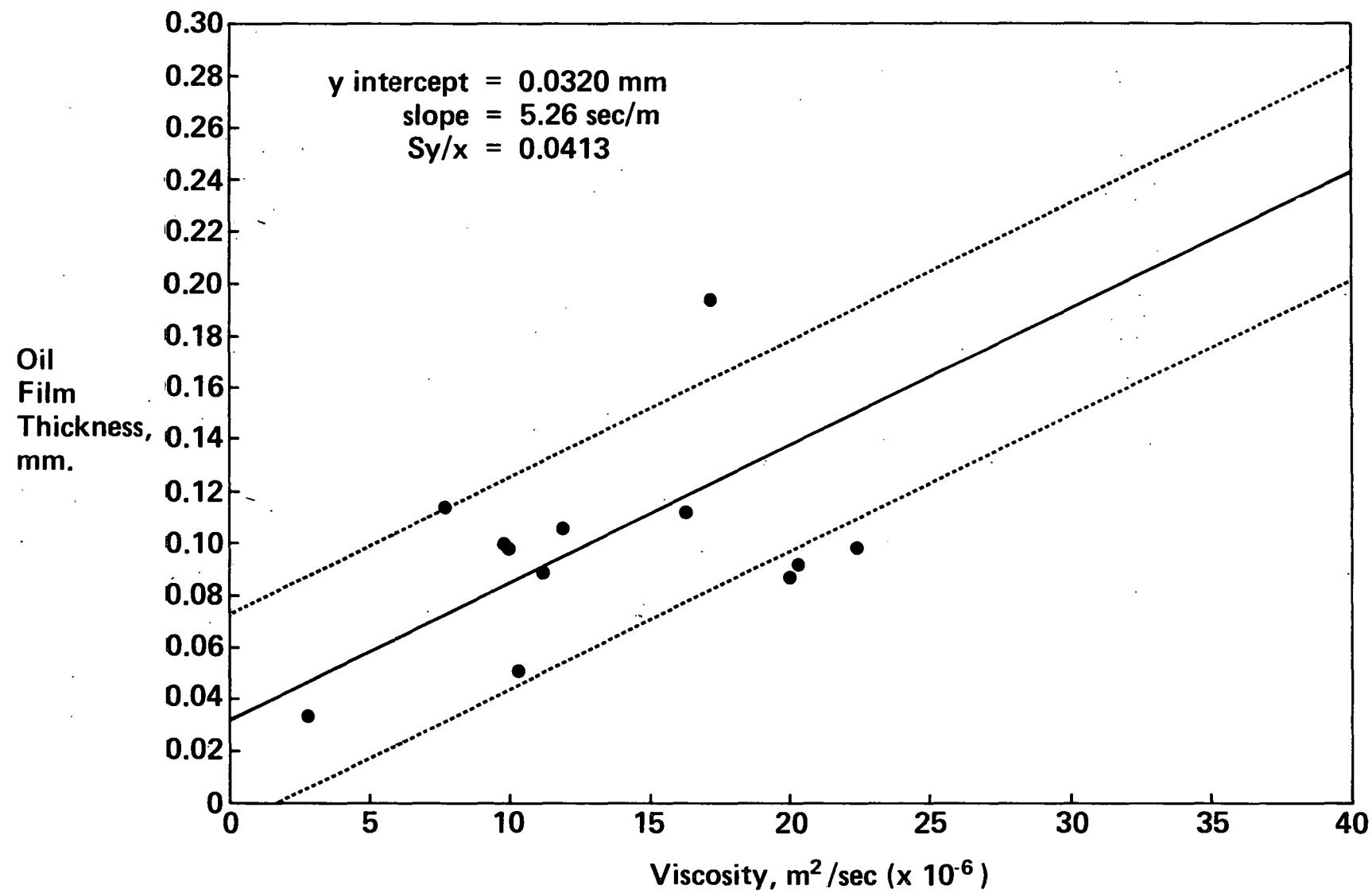


Figure 15. Effect of Viscosity (54°C) on Oil Film Thickness.

TABLE 1  
RAW OIL SHALE DATA

Batch	Overall Size Range, cm (in)	Fischer Assay Yield, L/tonne (gal/ton)	Specific Gravity, (15.6°/15.6°C)
1	-1.3,+0.12 (-1/2,+3/64)	132.3 (31.7)	2.121
2	-1.3,+0.32 (-1/2,+1/8)	140.2 (33.6)	2.095
3	-1.9,+0.95 (-3/4,+3/8)	134.4 (32.2)	2.114
4	-0.95,+0.12 (-3/8,+3/64)	137.7 (33.0)	2.103

TABLE 2  
RETORTING CONDITIONS

CSR Run Number	Shale Batch	Heating Rate °C/min.	Retorting Advance Rate, cm/hr.	Input Gas Flow Rate scmh/m <sup>2</sup>	Input Gas	Number Sections Oil wet	Reference
14	1	0.56	2.5	14.8	N <sub>2</sub>	10	4
17	1	0.22	1.5	7.3	N <sub>2</sub>	15	4
19	1	1.1	30.5	7.3	N <sub>2</sub>	15	4
23	2	0.22	1.5	68.9	N <sub>2</sub>	10	4
25	2	0.22	1.5	7.3	N <sub>2</sub> /O <sub>2</sub>	10	6
26	2	1.1	7.6	7.3	N <sub>2</sub> /O <sub>2</sub>	9	6
27	2	2.2	15.2	68.9	N <sub>2</sub> /O <sub>2</sub>	10	6
28	3	1.1	7.6	7.3	N <sub>2</sub>	10	6
31	3	1.1	7.6	7.3	N <sub>2</sub>	10	7
32	4	1.1	7.6	7.3	N <sub>2</sub>	10	6
33	4	1.1	7.6	7.3	N <sub>2</sub>	10	7
37	2	0.02	0.2	0.7	N <sub>2</sub>	9	8
58	2	1.1	7.6	7.3	N <sub>2</sub>	10	4
60	2	1.1	7.6	7.3	N <sub>2</sub>	10	4

TABLE 3  
STATISTICAL ANALYSIS OF OIL FILM THICKNESS  
AS RELATED TO TEMPERATURE

CSR Number	Slope, mm/ $^{\circ}\text{C}$ $\times 10^{-4}$	y intercept, mm	Variance in y
14	2.20	0.106	0.0075
17	2.20	0.098	0.0039
19	0.96	0.063	0.0057
23	3.10	0.123	0.0183
25	3.35	0.129	0.0161
26	2.89	0.124	0.0249
27	2.93	0.121	0.0120
28	1.63	0.079	0.0123
31	1.73	0.089	0.0193
32	2.09	0.085	0.0171
33	1.05	0.066	0.0074
37	3.36	0.115	0.0175
58	1.30	0.084	0.0175
60	2.06	0.096	0.0202

TABLE 4  
ORGANIC CARBON CONTENT FOR SHALES

Section Number	CSR 32	CSR 33
1	4.87	6.96
2	3.35	4.58
3	3.43	3.94
4	3.35	4.17
5	4.00	4.81
6	4.09	4.93
7	3.81	5.23
8	4.36	5.49
9	3.61	7.05
10	5.24	7.13
11	4.51	8.39
12	3.46	7.37
13	4.01	7.45
14	4.77	5.82
15	7.31	9.37
16	16.89	17.10
17	15.77	13.95
18	15.09	14.00
19	15.12	13.90
20	16.13	14.44
21	14.81	14.33
22	14.43	15.45
23	15.24	14.31
24	15.51	13.16

TABLE 5  
VISCOSITY DATA

CSR Number	Viscosity, $m^2/sec$ ( $\times 10^{-6}$ )		OFT <sup>1</sup> mm
	38°C	54°C	
14	35.2	20.0	.087
17	ND	ND	.087
28	ND	ND	.102
31	21.0	11.9	.106
32	16.7	9.8	.100
33	14.4	10.3	.051
58	22.2	10.0	.098
60	18.4	11.2	.089
23	75.4	32.9	.255
25	29.2	17.2	.194
26	36.5	22.4	.098
27		7.7	.114
37	39.6	20.3	.092
19	3.7	2.8	.034
33	14.4	10.3	.051

<sup>1</sup> OFT = oil film thickness

## BIBLIOGRAPHY

1. Fausett, D. W. "A Mathematical Model of an Oil Shale Retort." Quarterly of the Colorado School of Mines, v. 70, 1975, pp. 272-313.
2. Braun, R. L. and R. C. Y. Chin, "Progress Report on Computer Model for In Situ Oil Shale Retorting." UCRL-52292, 1977, 28 pp.
3. Dockter, Leroy and H. Gordon Harris, "A Mathematical Model of Forward Combustion Retorting of Oil Shale." LETC/TPR-78/1, 1978, 20 pp.
4. Duvall, John J., "Simulated In Situ Retorting of Oil Shale in a Controlled State Retort: I. Nitrogen Atmosphere, Interrupted Runs." LETC/RI-79/7, 1979, 62 pp.
5. Duvall, J. J. and H. B. Jensen, "Simulated In Situ Retorting of Oil Shale in a Controlled-State Retort." Quarterly of the Colorado School of Mines, v. 70, 1975, pp. 187-205.
6. Duvall, J. J., unpublished data.
7. Bartke, T. C. and J. J. Duvall, "Effects of Particle Size on Retorting Oil Shale in a Controlled-State Retort." ANS Proceedings NTIS Number Conference770440, 1977, pp. 516-527.
8. Duvall, J. J. "Simulated In Situ Retorting of Oil Shale in a Controlled-State Retort: II. Heat Soaking Experiments, LETC/ RI-79/10, 1979, 24 pp.
9. Crow, Edwin L., Frances A. Davis, Margaret W. Maxfield, "Statistics Manual." Dover Publications, Inc., New York, NY, 1960, p. 156.
10. Duvall, John J., unpublished data.
11. Smith, John Ward, "Relationship Between Rock Density and Volume of Organic Matter in Oil Shales." LETC/RI-76/6, 1976, 11 pp.
12. Bird, R. Byron, Warren E. Stewart, and Edwin N. Lightfoot, "Transport Phenomena." John Wiley & Sons, Inc., New York, NY, 1960, p. 197.

## APPENDIX

### Calculation of Oil Shale Surface Area

The quantities needed to calculate oil film thickness that were known were the weight of oil washed from the surface, the weight of shale, the size distribution of shale, the volume of the section of the retort, and the density of the shale (9). The determination of oil film thickness required, in addition, a knowledge of the wetted surface area of the shale in a particular section of the CSR. An equation adapted from Bird, Stewart, and Lightfoot (12) allowed calculation of wetted surface area (a) per unit volume of bed as follows:

- 1)  $a = a_v (1 - E)$  where  $a_v$  is the "specific surface" (the total particle surface divided by the volume of the particles) and E is the void volume. The quantity  $a_v$  was used to define the mean particle diameter ( $D_p$ ) as follows:
- 2)  $D_p = 6/a_v$  where  $D_p$  equals the diameter for spherical particles. Combining equations 1) and 2) gives:
- 3)  $a = 6/D_p(1 - E)$ . The void volume (E) can be calculated from the weight of shale in a section, the volume of the section, and the density of the shale (9). The mean particle diameter ( $D_p$ ) of a shale particle of a batch of shale can be estimated from the particle size distribution of the shale (Table 1), the mean sieve opening (S) for a shale cut (e.g., the mean sieve opening for the shale cut -6.4, + 3.2mm is 4.8 mm), and the assumption that a shale particle can be treated as a rectangular box. The relationship of the lengths of the sides of a shale particle was estimated from actual measurements of 25 pieces of shale (see Table A1) picked at random from a batch of shale, to be  $1.6W \times W \times 0.6W$  where W is the width of the shale particle. Substitution of the mean sieve opening of the above example (4.8 mm) for W gives the volume of an average shale particle in that sieve cut:

$$\begin{aligned} \text{Volume} &= 1.6W \times W \times 0.6W \\ &= 0.96S^3 \\ &= 106 \text{ mm}^3. \end{aligned}$$

The mean diameter ( $D_p$ ) of a sphere of equivalent volume can be calculated as follows:

$$\text{Volume} = \frac{4\pi}{3} r^3$$

$$r = \left( \frac{106 \times 3}{4\pi} \right)^{1/3}$$

$$= 2.9 \text{ mm}$$

$$D_p = 2r = 5.8 \text{ mm}$$

These calculations gave the average particle diameters which are shown in Table A2 along with the particle size distribution and the average particle diameter for each batch of shale.

The average  $D_p$ 's of Table A2 were used in equation 3) to calculate the raw shale surface areas in each of the oil wet sections in interrupted CSR experiments as in the following example (for CSR 14, section 18).

$$a = \frac{6}{D_p} (1 - E)$$

$$a = \frac{6}{0.56} (1 - 0.45)$$

$$= 5.89 \text{ cm}^2/\text{cm}^3$$

The  $a$  above, when multiplied by the volume of that section of the retort ( $645 \text{ cm}^3$ ), gave  $3800 \text{ cm}^2$  as the calculated surface area of the shale in that section of the retort.

In addition, the surface area of the retort also must be taken into account. The surface area of the retort of CSR 14, section 18 was calculated to be  $507 \text{ cm}^2$ . Because the oil was flowing over the shale and retort surfaces and was not allowed to drain, the oil film thickness on the shale and retort was assumed to be the same and were added together to give the total surface area in each section of the retort. For CSR 14, section 18 this gave a total surface area of  $4307 \text{ cm}^2$ . This surface area divided into the cubic centimeters of oil ( $41.4 \text{ cm}^3$ ) washed from the surface of the retort and shale gives  $0.096 \text{ mm}$  as the oil film thickness on the shale and the retort pipe of section 18 of CSR 14.

These types of calculations led to the oil film thickness data shown in Tables A3 through A16.

TABLE A1  
DIMENSIONS OF RAW SHALE PARTICLES PICKED AT RANDOM FROM  
A BATCH SIEVED TO -1.3, +0.3 cm (-1/2 in, +1/8 in)

Particle Number	L, cm	W, cm	H, cm
1	3.3	1.1	0.8
2	1.8	0.8	0.6
3	1.8	1.5	0.6
4	2.2	1.1	1.0
5	1.5	1.4	1.0
6	1.0	0.8	0.4
7	1.5	1.3	0.3
8	2.4	1.5	0.4
9	1.3	0.9	0.8
10	1.5	1.0	0.6
11	2.3	1.5	0.8
12	1.5	1.0	0.8
13	2.3	1.0	0.9
14	2.0	1.3	0.9
15	2.5	1.3	0.8
16	2.3	1.3	0.6
17	2.0	1.3	0.8
18	2.0	1.3	0.3
19	2.0	1.1	0.6
20	1.9	1.5	0.5
21	1.5	1.0	1.0
22	1.8	1.3	0.6
23	1.5	1.1	0.8
24	2.4	1.1	0.8
25	1.9	1.5	0.8
Average	1.9 ± 0.5	1.2 ± 0.2	0.7 ± 0.2

TABLE A2  
RAW OIL SHALE SIZE AND SIZE DISTRIBUTION DATA

Sieve Size Range (mm)	Mean Sieve Openings (mm)	D <sub>p</sub> (mm)	Fraction of Shale in Size Range Batch			
			1	2	3	4
-3.2,+1.2	2.2	2.7	0.02	-	-	0.20
-6.4,+3.2	4.8	5.8	0.30	0.26	-	0.37
-9.5,+6.4	7.9	9.7	0.39	0.26	-	0.43
-12.7,+9.5	11.1	13.6	0.29	0.48	0.44	-
-15.9,+12.7	14.3	17.4	-	-	0.37	-
-19.0,+15.9	17.5	21.4	-	-	0.19	-
D <sub>p</sub> for batch (mm)			5.6	7.2	15.2	3.4

TABLE A3  
DATA FOR CSR 14

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of pipe, cm <sup>2</sup>	Surface Area of shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of zone, °C
15	15.9	726	4.7	47	507	3657	0.011	416
16	17.1	764	30.8	48	547	3847	0.070	204
17	17.5	728	36.7	52	557	3665	0.087	66
18	15.9	746	41.4	45	507	3800	0.096	43
19	15.9	716	63.7	48	507	3617	0.155	ambient
20	17.1	763	44.4	48	547	3854	0.100	"
21	17.1	761	37.3	48	547	3832	0.087	"
22	15.9	732	36.8	46	507	3687	0.088	"
23	15.9	705	32.8	48	507	3551	0.083	"
24	21.9	818	41.7	57	699	4119	0.088	"

TABLE A4  
DATA FOR CSR 17

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
33	10	16.2	686	7.7	51	517	3456	0.019
	11	15.9	690	21.5	49	507	3475	0.054
	12	17.1	718	32.8	51	547	3616	0.079
	13	17.1	731	35.6	50	547	3680	0.084
	14	15.9	691	48.9	49	507	3491	0.122
	15	15.9	665	44.8	51	507	3349	0.116
	16	17.1	723	45.8	51	547	3652	0.109
	17	17.5	797	41.7	47	557	4026	0.091
	18	15.9	627	30.9	54	507	3157	0.084
	19	15.9	665	26.8	51	507	3359	0.069
	20	17.1	723	27.0	51	547	3757	0.065
	21	17.1	746	32.8	49	547	3273	0.067
	22	15.9	648	25.5	53	507	3273	0.067
	23	15.9	690	28.7	50	507	3486	0.072
	24	21.9	1030	51.9	45	699	5187	0.088

TABLE A5  
DATA FOR CSR 19

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
10	16.2	707	7.6	49	517	3650	0.019	382
11	15.9	708	12.1	48	507	3567	0.030	349
12	17.1	775	15.0	47	547	3902	0.034	310
13	17.1	799	18.3	46	547	4024	0.040	282
14	15.9	692	15.8	49	507	3485	0.040	260
15	15.9	739	18.3	46	507	3721	0.043	227
16	17.1	744	22.8	50	547	3745	0.053	174
17	17.5	788	27.6	48	557	3970	0.061	104
18	15.9	697	20.8	49	507	3509	0.052	77
19	15.9	685	19.8	52	507	3313	0.052	32
20	17.1	750	18.2	49	547	3776	0.044	ambient
21	17.1	722	13.4	51	547	3635	0.033	"
22	15.9	698	13.3	49	507	3485	0.035	"
23	15.9	692	11.7	49	507	3485	0.030	"
24	21.9	1182	16.3	37	699	5953	0.026	"

TABLE A6  
DATA FOR CSR 23

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
15	15.9	577	0.3	57	507	2297	0.001	399
16	17.1	739	15.8	49	547	2942	0.045	266
17	17.5	765	21.8	48	557	3192	0.058	143
18	15.9	687	37.0	49	507	2734	0.114	77
19	15.9	664	72.8	51	507	2644	0.231	49
20	17.1	757	113.0	48	547	3161	0.305	ambient
21	17.1	788	109.6	46	547	3137	0.298	"
22	15.9	729	79.5	46	507	2902	0.233	"
23	15.9	724	76.8	46	507	2881	0.227	"
24	21.9	1087	107.2	42	699	4327	0.213	"

TABLE A7  
DATA FOR CSR 25

CSR Section Number	Length of Section, cm	Shale, g.	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
15	15.9	593	0.2	56	507	2360	0.001	402
16	17.1	782	18.5	46	547	3112	0.050	224
17	17.5	754	23.8	49	557	3002	0.067	138
18	15.9	784	46.8	53	507	3120	0.154	56
19	15.9	784	46.7	42	507	3120	0.129	43
20	17.1	750	82.6	48	547	2986	0.234	ambient
21	17.1	737	70.6	49	547	2934	0.203	"
22	15.9	696	69.4	48	507	2769	0.212	"
23	15.9	707	58.5	48	507	2815	0.176	"
24	21.9	1155	76.9	38	699	4596	0.145	"

TABLE A8  
DATA FOR CSR 26

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
15	15.9	652	0.4	52	507	2594	0.001	410
16	17.1	757	20.0	48	547	3014	0.056	288
17	17.5	757	21.1	49	557	3014	0.059	116
18	15.9	711	33.0	47	507	2830	0.099	68
19	15.9	687	43.7	49	507	2734	0.135	46
20	17.1	712	37.3	51	547	2833	0.110	ambient
21	17.1	776	37.4	47	547	3089	0.103	"
22	15.9	685	33.3	49	507	2727	0.104	"
23	15.9	679	29.3	50	507	2702	0.092	"
24	21.9	910	34.4	51	699	3622	0.080	"

TABLE A9  
DATA FOR CSR 27

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
16	17.1	748	5.6	49	547	2977	0.016	349
17	17.5	779	31.3	47	557	3102	0.086	146
18	15.9	697	29.6	48	507	2775	0.090	60
19	15.9	695	36.8	48	507	2760	0.113	49
20	17.1	743	65.1	49	547	2956	0.186	32
21	17.1	779	53.4	47	547	3102	0.146	ambient
22	15.9	682	31.4	49	507	2715	0.097	"
23	15.9	716	32.6	47	507	2850	0.097	"
24	21.9	1158	61.6	38	699	4610	0.116	"

TABLE A10  
DATA FOR CSR 28

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
15	15.9	606	1.6	55	507	1128	0.010	416
16	17.1	724	6.5	51	547	1347	0.034	279
17	17.5	708	10.7	53	557	1317	0.057	129
18	15.9	683	9.2	50	507	1271	0.052	60
19	15.9	693	15.6	49	507	1290	0.087	29
20	17.1	654	16.9	55	547	1217	0.096	ambient
21	17.1	747	18.4	49	547	1388	0.095	"
22	15.9	698	16.8	49	507	1299	0.093	"
23	15.9	664	17.4	51	507	1235	0.100	"
24	21.9	1083	34.7	42	699	2016	0.128	"

TABLE A11  
DATA FOR CSR 31

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
5	15.9	693	4.9	49	507	1290	0.027	316
	17.1	639	12.0	57	547	1190	0.069	204
	17.5	766	11.7	49	557	1425	0.059	66
	15.9	678	16.5	50	507	1261	0.093	38
	15.9	682	17.8	50	507	1269	0.100	ambient
	17.1	713	19.3	51	547	1327	0.103	"
	17.1	741	20.4	50	547	1380	0.106	"
	15.9	666	17.8	51	507	1240	0.102	"
	15.9	666	17.8	51	507	1245	0.101	"
	21.9	1094	34.6	42	699	2036	0.126	"

TABLE A13  
DATA FOR CSR 32

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
15	15.9	650	1.4	52	507	5514	0.002	418
16	17.1	783	20.6	46	547	6642	0.029	282
17	17.5	814	32.6	45	557	6902	0.044	127
18	15.9	697	33.0	49	507	5912	0.051	77
19	15.9	735	52.0	46	507	6232	0.077	52
20	17.1	784	74.2	46	547	6648	0.103	32
21	17.1	770	69.7	47	547	6531	0.099	ambient
22	15.9	733	82.1	46	507	6218	0.122	"
23	15.9	736	71.7	46	507	6242	0.106	"
24	21.9	1108	75.3	41	699	9398	0.075	"

TABLE A12  
DATA FOR CSR 33

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
16	17.1	834	25.1	43	547	7072	0.033	360
17	17.5	828	27.4	44	557	7022	0.036	204
18	15.9	753	41.8	44	507	6385	0.061	66
19	15.9	741	44.2	45	507	6285	0.065	38
20	17.1	815	44.8	44	547	6912	0.060	ambient
21	17.1	813	39.5	44	547	6894	0.053	"
22	15.9	731	33.6	46	507	6200	0.049	"
23	15.9	752	34.0	44	507	6378	0.049	"
24	21.9	1206	46.0	35	699	10228	0.042	"

TABLE A14  
DATA FOR CSR 37

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
16	17.1	752	2.3	48	547	2993	0.007	343
17	17.5	800	10.0	46	557	3184	0.027	204
18	15.9	717	31.6	47	507	2583	0.094	102
19	15.9	730	33.3	46	507	2906	0.097	46
20	17.1	780	39.6	46	547	3105	0.108	ambient
21	17.1	759	32.3	48	547	3021	0.090	"
22	15.9	741	30.4	45	507	2949	0.088	"
23	15.9	707	26.4	48	507	2815	0.080	"
24	21.9	1164	50.9	37	699	4632	0.095	"

TABLE A15  
DATA FOR CSR 58

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Percent	Surface Area of Shale, cm <sup>2</sup>	Oil Shale Thickness, mm	Final Temperature of Zone, °C
15	15.9	722	13.4	46	507	2874	0.044	341
16	17.1	773	19.0	47	547	3077	0.052	199
17	17.5	789	21.7	47	557	3140	0.059	66
18	15.9	693	31.5	49	507	2757	0.096	38
19	15.9	665	31.9	51	507	2647	0.101	ambient
20	17.1	778	37.0	47	547	3096	0.102	"
21	17.1	772	36.7	47	547	3073	0.101	"
22	15.9	606	27.0	55	507	2410	0.093	"
23	15.9	338	16.1	75	507	1346	0.087	"
24	21.9	41	8.8	98	699	163	0.102	"

TABLE A16  
DATA FOR CSR 60

CSR Section Number	Length of Section, cm	Shale, g	Oil, cm <sup>3</sup>	Void Volume, Percent	Surface Area of Pipe, cm <sup>2</sup>	Surface Area of Shale, cm <sup>2</sup>	Oil Film Thickness, mm	Final Temperature of Zone, °C
45	15.9	666	5.9	51	507	2651	0.019	388
	17.1	768	16.8	47	547	3056	0.047	254
	17.5	738	19.0	50	557	2939	0.054	88
	15.9	737	26.3	45	507	2934	0.077	71
	15.9	710	37.6	47	507	2825	0.113	38
	17.1	767	36.4	47	547	3052	0.101	ambient
	17.1	793	33.0	46	547	3155	0.089	"
	15.9	695	25.4	48	507	2765	0.078	"
	15.9	769	26.6	43	507	3061	0.075	"
	21.9	922	45.6	50	699	3670	0.104	"

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