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**CHEMSEAL 3808-A2 PENETRATION INTO
SMALL LEAK PATH**

By M. R. Carruth, Jr. and R. F. DeHaye

Materials and Processes Laboratory
Science and Engineering Directorate

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NOMENCLATURE

| <u>Symbol</u> | <u>Definition</u> |
|---------------|-------------------------------------------------------------------------|
| b_n | coefficients for polynomial curve fit |
| d | diameter |
| g | acceleration due to gravity |
| h | height fluid has moved into tube due to capillary action |
| H | maximum height a fluid moves into capillary tube due to surface tension |
| l | total leak path length |
| L | length viscous fluid had moved into capillary tube or leak path |
| P | pressure |
| Q | dV/dt or the volume rate of change with time |
| r | radius |
| t | time in seconds |
| u | viscosity |
| v | dL/dt or velocity |
| V | volume |
| ρ | density |
| τ | surface tension |

TECHNICAL MEMORANDUM

CHEMSEAL 3808-A2 PENETRATION INTO SMALL LEAK PATH

INTRODUCTION

A leak of nitrogen tetroxide (N_2O_4) oxidizer from the attitude control system of the Space Shuttle Discovery was discovered prior to launch of STS-26. One method of fixing the leak was proposed by MSFC. This fix involved the passing of a "shuttle-cock" past the leaking Dynaflo fitting and sealing the vent tube containing the fitting with Chemseal 3808-A2. One question to be addressed was whether the Chemseal will flow into the leak path and provide a better seal than if the sealant just covers the leak path. To address this question, the physical size of the leak was predicted using data on the leak rate, properties of N_2O_4 , and Poiseuille's law for viscous flow in a capillary tube [1]. This same formula, in a different form, is used to determine if the Chemseal will flow into the expected leak size. Experiments were conducted using fluids of known viscosity and small capillary tubes in order to verify the applicability of the formula to the N_2O_4 leak.

Following verification of the formula, it was applied to the Shuttle leak problem. Data were obtained in the laboratory on the viscosity of the Chemseal 3808-A2 as a function of time following mix of parts A and B, which initiates the cure process. The viscosity increase with time occurs because effectively a new material is evolving. This time dependence was included in the time integral of the formula for viscous flow into a capillary tube. Predictions for penetration into the expected leak path were made.

ANALYTICAL FORMULATION

Leak Size

The leak rate of N_2O_4 was 0.4 lb per day. The Dynaflo fitting, which was discovered to be leaking, is in the vent tube of the oxidizer tank for the Space Shuttle Discovery attitude control system. The fitting has a metal-to-metal seal and the width across this area is 0.33 cm. If the assumption is made that the leak is due to a scratch across the sealing surface, or an equivalent path, the size of the scratch may be calculated using Poiseuille's equation given in Reference 1,

$$Q = \frac{\pi r^4 P}{8 \mu l} ,$$

the necessary properties of the oxidizer, and the known leak rate.

If the above equation is used to solve for the scratch radius it becomes,

$$r = \left(\frac{8 u l Q}{P \pi} \right)^{1/4}$$

Using cgs units and data on N_2O_4 and the leak rate, u is 0.0042 poise, L is 0.33 cm, P is 1.65×10^7 dynes/cm², and Q is 1.5×10^{-3} cm³/s. With these values, the radius of the leak, r , is 0.004 cm. This is a diameter of 3 mils for the leak path.

The equation for the velocity of viscous flow through a pipe is taken from Reference 1 and is based on Poiseuille's equation. It is given by

$$v = \frac{P d^2}{32 u L}$$

If the velocity term is changed to differential form of distance traveled with time and integrated, the viscous fluid flow progress into the pipe with time is given,

$$L = \sqrt{\frac{d^2 P t}{16 u}} \quad (1)$$

The pressure exerted by a column of fluid is given by $P = \rho g h$. A fluid will move into a capillary until the force of gravity on the column is equal to the force caused by surface tension to pull the fluid into the capillary. When the fluid is at the end of the capillary, but has not yet moved into it, there is an effective pressure on the fluid due to the surface tension. As the fluid moves into the column, the effective pressure "pushing" the fluid upward changes. Therefore, the net pressure is $P(h) = (\rho g H - \rho g h)$. Going back to the differential form of Poiseuille's equation and allowing the pressure to be variable with h , $P(h)$, and integrating, the time required to arrive at a height $h(t)$ in the column is given by,

$$t = \frac{32 u}{\rho g d^2} \{-h(t) - H \ln[H-h(t)] + H \ln H\} \quad (2)$$

EXPERIMENTS

Flow Into Capillary Tube With Constant Pressure

An experiment was conducted by applying a constant pressure to a viscous fluid and forcing it into a capillary tube. If the results of the data obtained and equation (1) are consistent, then some confidence can be placed on the formula's application to the flow of sealant into the N_2O_4 leak path.

The flow of viscous fluids into a capillary tube was measured using a video camera to record the rise of the fluid with time. The camera allowed a 1/30 of a second resolution from frame to frame. To examine the flow for a constant applied

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pressure, a hollow metal fixture was fabricated and filled with the test fluid. The glass capillary was attached by a fitting with an O-ring seal. A pressure line was attached to the metal fixture so that a pressure of known magnitude was applied to the fluid. The rise of fluid into the glass capillary was recorded by the video camera. A scale was also in view of the camera so that the rate of rise could be accurately measured.

Figures 1 through 5 compare data obtained with the prediction obtained using equation (1). The time zero in the data could not be obtained accurately. Therefore, the data are shifted so that the first data point lies directly on the predicted curve. The other data points fall along or near the curve indicating a favorable comparison. The time from actual zero to the time of the first data point is also reasonable even though that datum was not directly obtained.

Flow Into Tube Due to Capillary Action

Another experiment was performed to gain additional confidence in the ability of the equations specified above to describe the flow of a viscous fluid into the leak path. In this case the pressure is allowed to vary and is, as described above, leading up to equation (2). This equation, therefore, will be compared with the data obtained in the described manner.

As discussed earlier, the video camera is also used in this experiment to measure the flow of a fluid of known viscosity into the glass capillary tube. The resolution of the video camera allows accurate measurement of the position of the fluid column as a function of time. These data are compared to equation (2). The experimental setup is slightly different than before. The glass capillary tube is held stationary with the video camera focused on it so that the bottom open end is in the bottom of the camera view. A beaker of the test fluid sits on a lab jack which is slowly raised. As the fluid comes in contact with the end of the capillary tube it rises into the tube and this is recorded by the video camera for later analysis.

Figures 6 through 13 show the comparison of experimental data points with the predicted curve generated using equation (2). Numerous capillary tube sizes and fluid viscosities are shown. In all cases, the comparison is good and in some it is excellent. The times for the fluid to rise into the capillary due to surface tension ranges from seconds to many minutes. The only experimental data used in the prediction was the maximum height, H, since the surface tension of the fluid was not known and this is the standard way in which it is determined. Several fluids of known surface tension were tested this way to confirm that experiment procedures and techniques gave accurate data. The surface tension of a liquid that wets the capillary tube is given by

$$\tau = \frac{H \rho g r}{2}$$

APPLICATION TO SEAL LEAK

The previous experimental work has given some confidence to the ability of the Poiseuille's equation to predict the flow of viscous fluids into capillary tubes. It will therefore be used to predict the flow of Chemseal 3808-A2 into the leak whose size was predicted earlier.

After the Chemseal 3808-A2 parts A and B are mixed, the setting up and curing process begins, which means that the viscosity will be increasing with time. Data were obtained in the laboratory for the viscosity and are shown in Figure 14 [2]. This time varying viscosity must be accounted for in the equation used for prediction of flow into the leak. If the differential form of the equation which leads to equation (1) is taken but viscosity allowed to vary with time, the length that the fluid will travel into a capillary from one time t_1 to another time t_2 (where the mixing of Chemseal parts A and B occur at time $t = 0$), is given by,

$$L = \sqrt{\frac{d^2 P}{16} \int_{t_1}^{t_2} \frac{dt}{b_0 + b_1 t + b_2 t^2 + b_3 t^3}} \quad (3)$$

The laboratory data which were collected on the viscosity as a function of time following mix were fit to a cubic polynomial for use in integrating equation (3) and $u(t) = b_0 + b_1 t + b_2 t^2 + b_3 t^3$. By choosing the proper limits of integration for equation (3), the flow into the leak due to application of a constant pressure to the Chemseal after a given time following its mixing can be determined. Figures 15 and 16 give predictions for the length that the Chemseal will penetrate as a function of time after mix. Two leak sizes are shown on these two figures. The different curves on each figure indicate the time interval between Chemseal mix and its application to the leak.

CONCLUSIONS

The available data on the N_2O_4 leak indicates that the leak could be due to a scratch across the sealing surface of the Dynaflo fitting of approximately a 3-mil equivalent diameter. The Poiseuille equation, as given in Reference 1, was compared with various experimental data and found to correctly predict the flow of a viscous fluid into a small capillary tube. The equation was applied to the case of Chemseal 3808-A2 flow into the predicted leak size. The time dependence of the viscosity of the Chemseal after mix was taken into account. For a capillary path length the order of the leak size predicted by the known N_2O_4 leak rate, the Chemseal should penetrate to form a good seal even an hour after mix. However, better penetration is obtained if the Chemseal is applied as soon as possible after mix. The calculations do indicate that the penetration length reaches a limiting value both due to the length penetrated as well as the increasing viscosity.

The Chemseal has a small surface tension and does wet the stainless steel tube material. This surface tension will produce an added effective pressure that is not accounted for in the calculations given in this paper. However, calculations indicate that the effect of surface tension will be small compared to the pressure to be applied to the Chemseal.

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1. Urquart, L. C. (editor): Civil Engineering Handbook. McGraw-Hill Book Company, Inc., 1959.
2. Gibson, H. and Dolan, F.: Personal communication, MSFC.

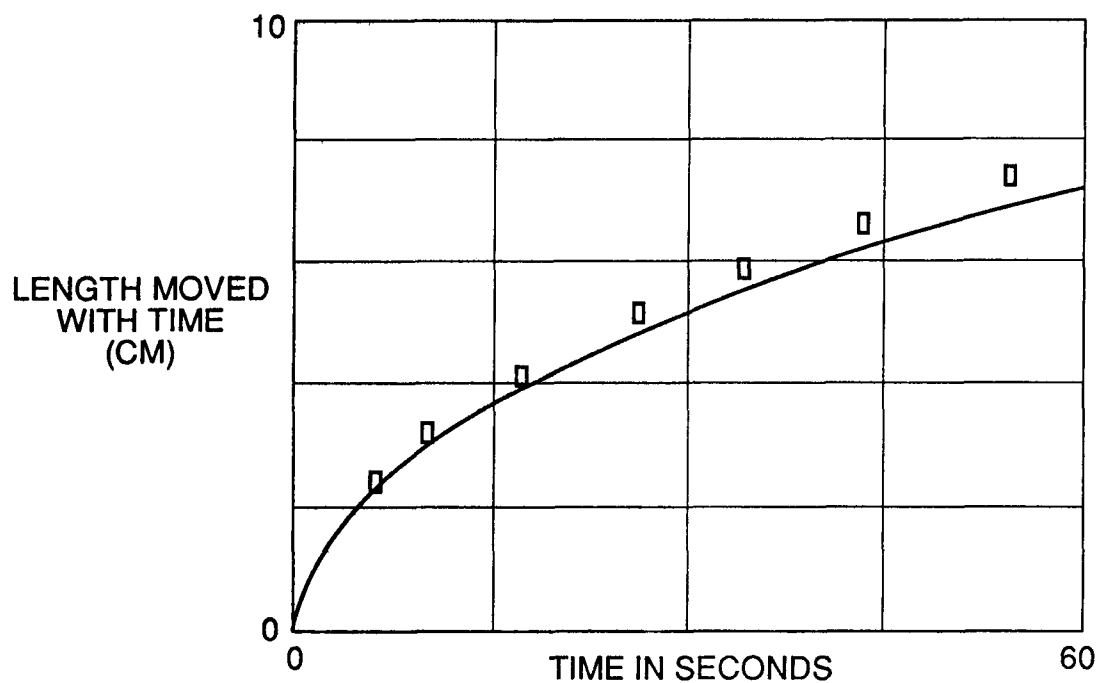


Figure 1. Prediction curve and data points for viscous fluid flow into a capillary tube. Tube diameter: 19 mils, pressure: 27 psi, viscosity: 296 poise.

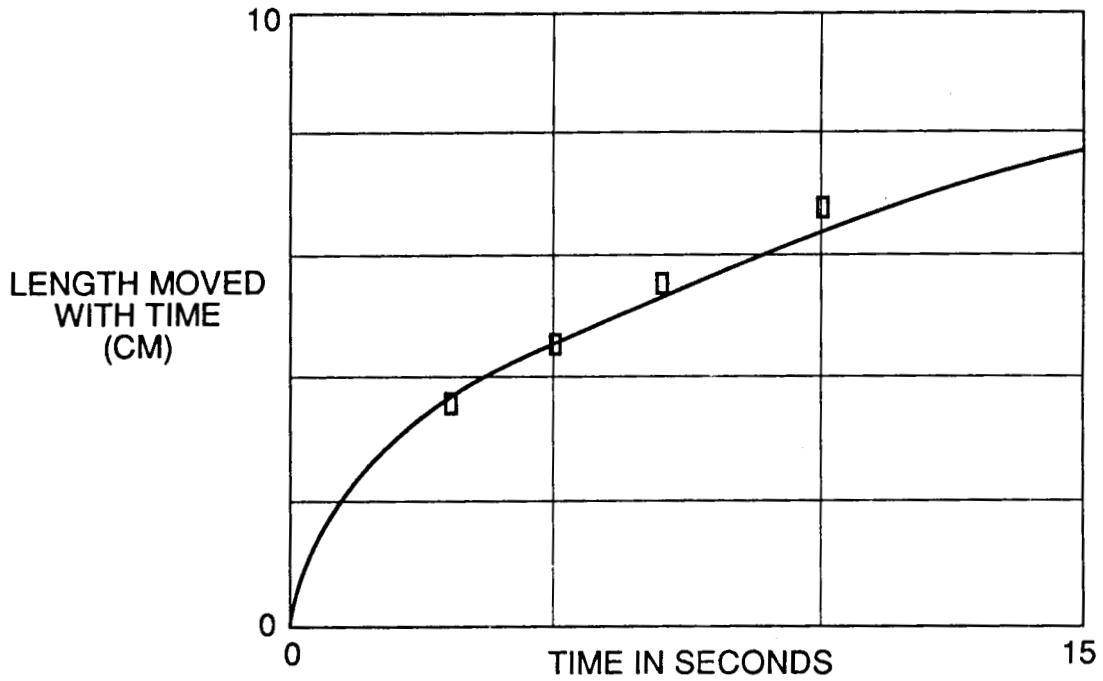


Figure 2. Prediction curve and data points for viscous fluid flow into a capillary tube. Tube diameter: 40 mils, pressure: 28 psi, viscosity: 296 poise.

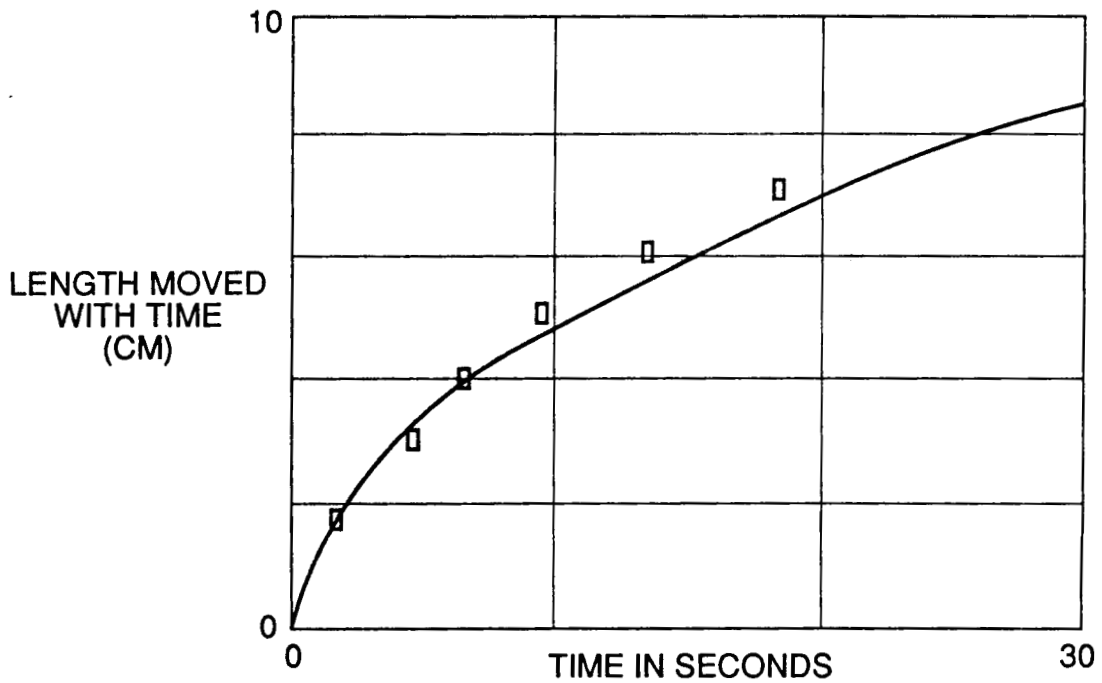


Figure 3. Prediction curve and data points for viscous fluid flow into a capillary tube. Tube diameter: 43 mils, pressure: 27 psi, viscosity: 590 poise.

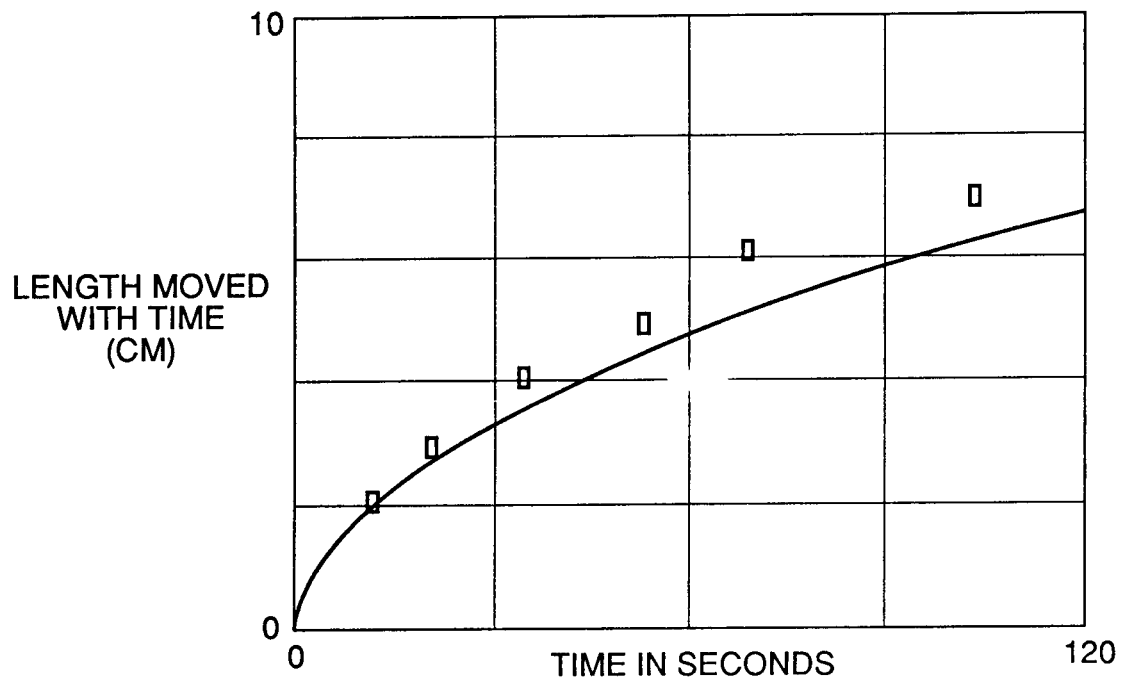


Figure 4. Prediction curve and data points for viscous fluid flow into a capillary tube. Tube diameter: 19 mils, pressure: 25 psi, viscosity: 590 poise.

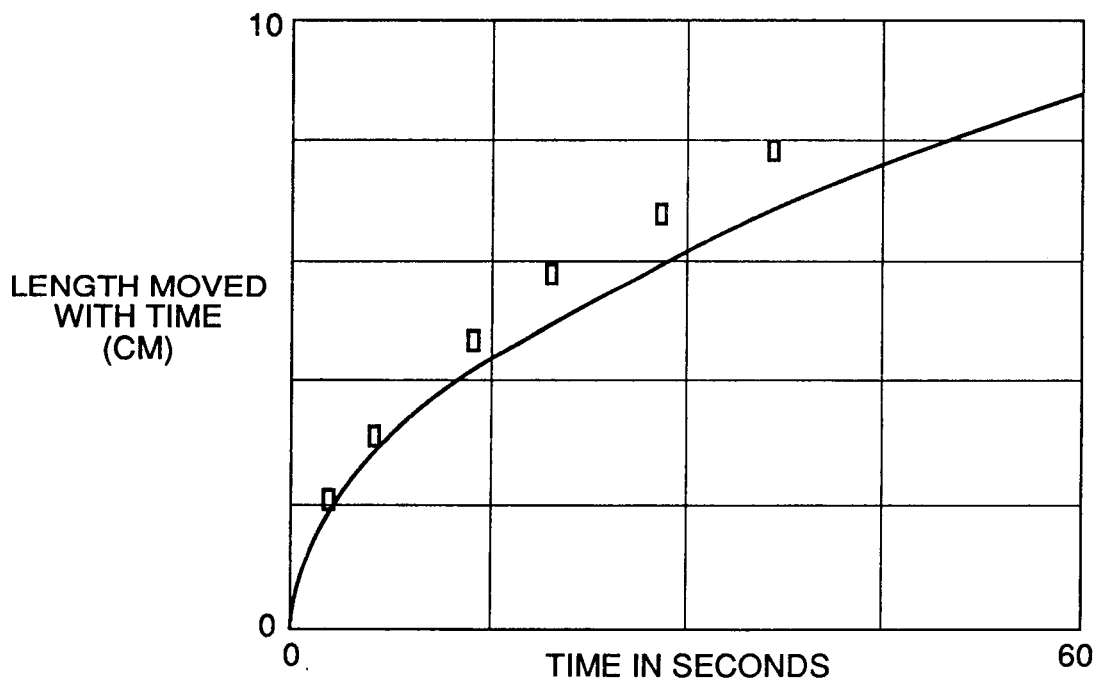


Figure 5. Prediction curve and data points for viscous fluid flow into a capillary tube. Tube diameter: 44 mils, pressure: 26 psi, viscosity: 1005 poise.

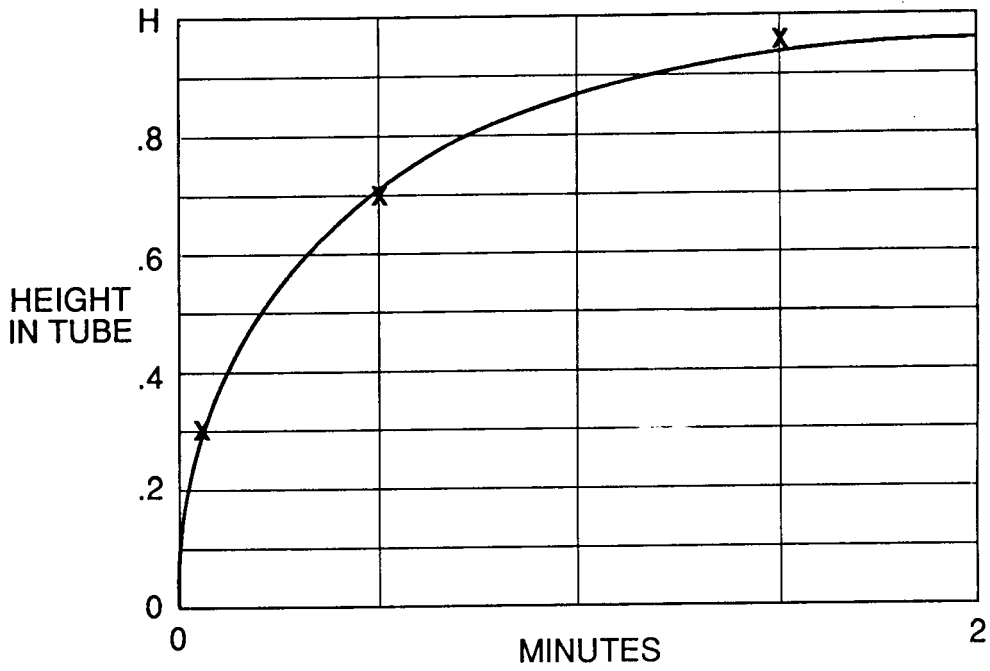


Figure 6. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 11 mils, viscosity: 50 centipoise.

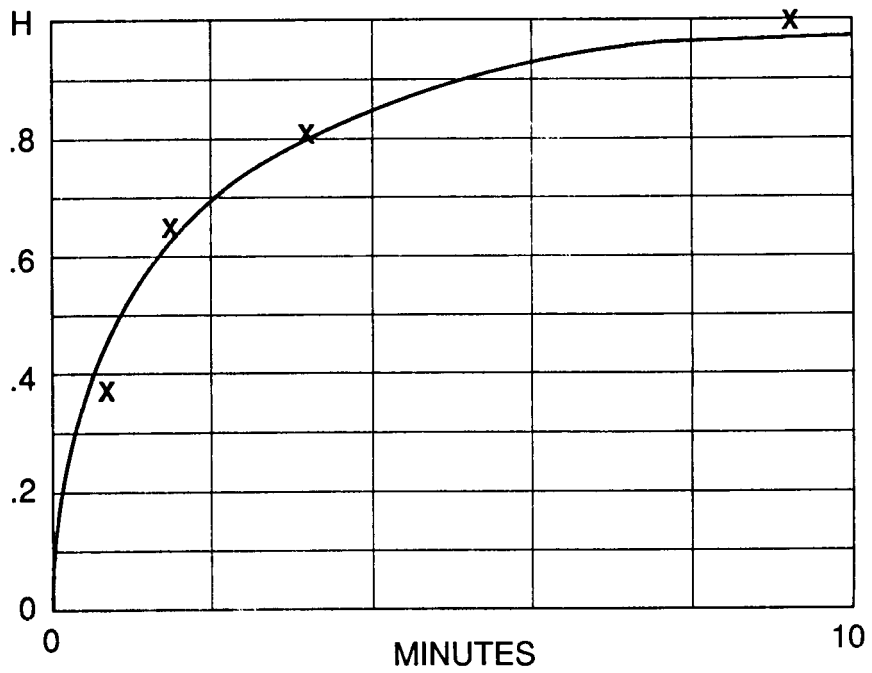


Figure 7. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 30 mils, viscosity: 50 centipoise.

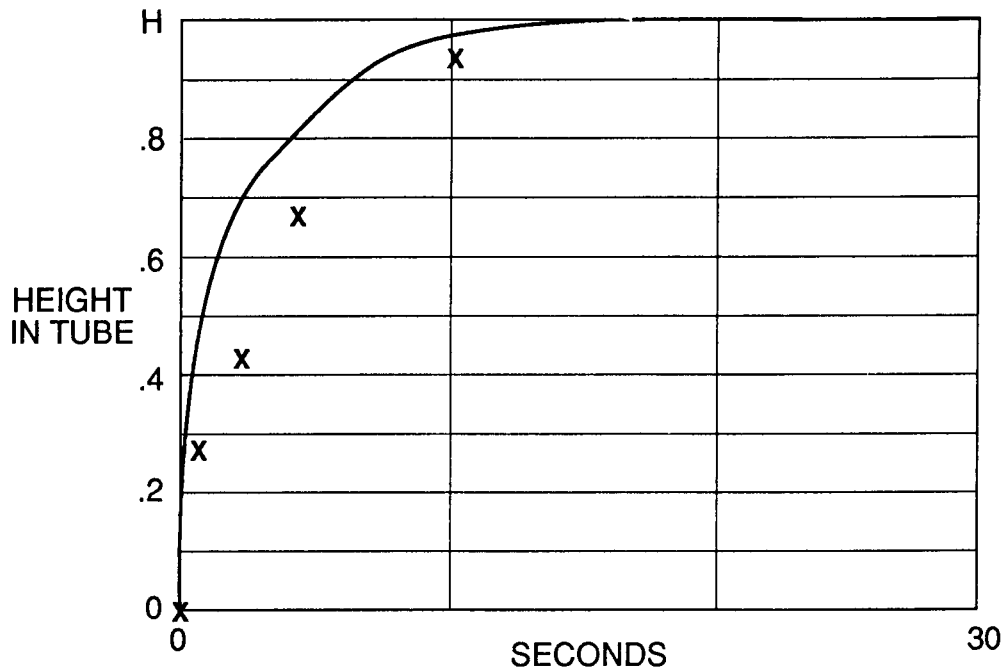


Figure 8. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 30 mils, viscosity: 98 centipoise.

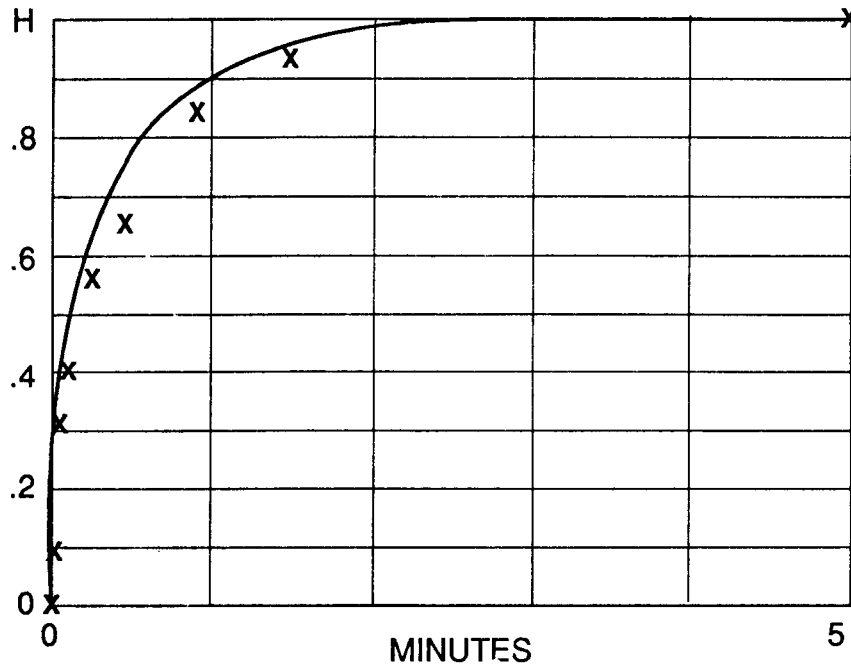


Figure 9. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 16 mils, viscosity: 98 centipoise.

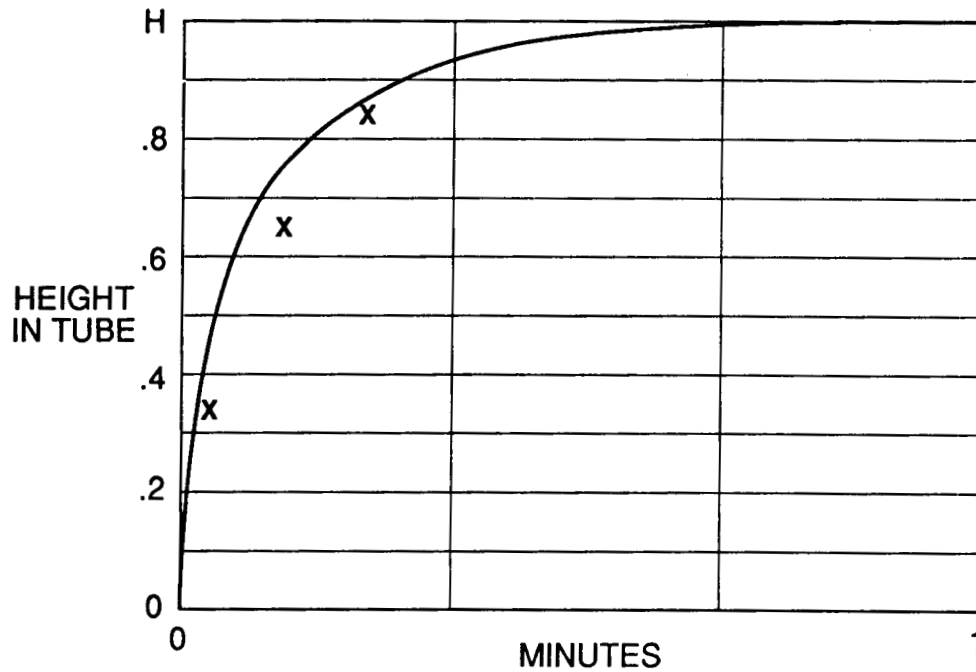


Figure 10. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 11 mils, viscosity: 9.7 centipoise.

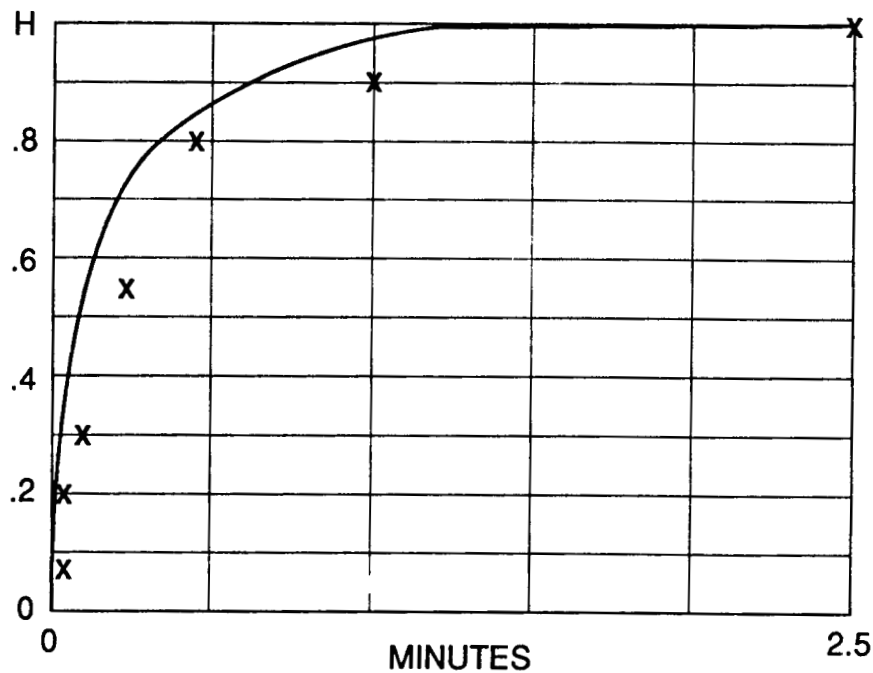


Figure 11. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 16 mils, viscosity: 50 centipoise.

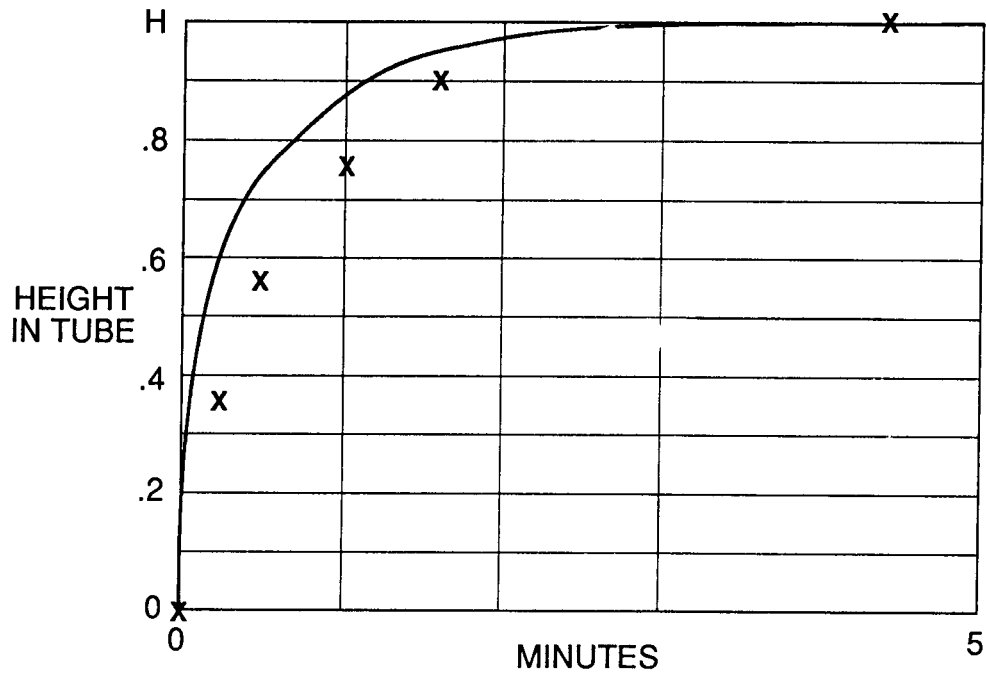


Figure 12. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 30 mils, viscosity: 10.2 poise.

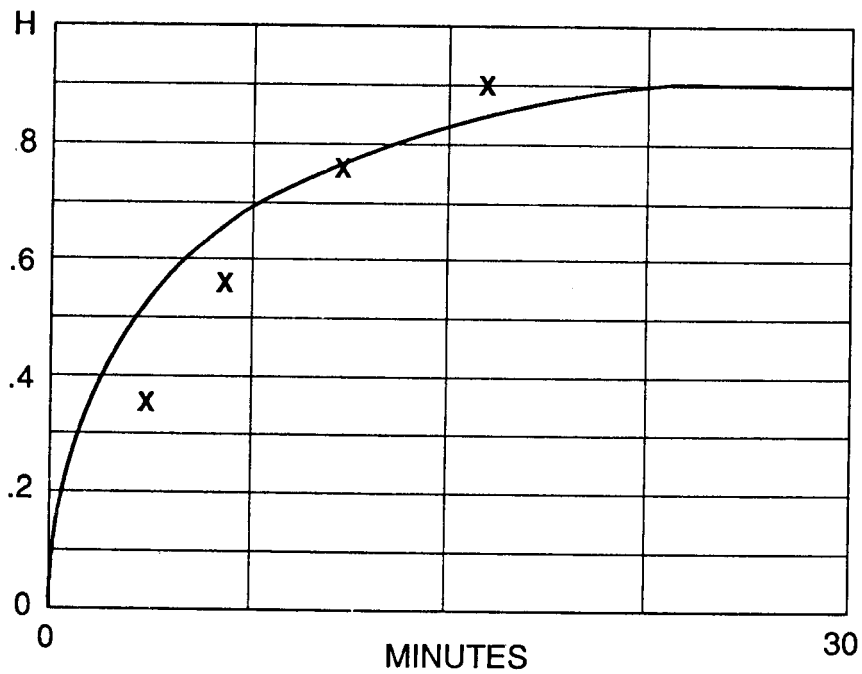


Figure 13. Prediction curve and data points for viscous fluid flow into a capillary tube by capillary action producing changing pressure due to column height increase. Tube diameter: 30 mils, viscosity: 294 poise.

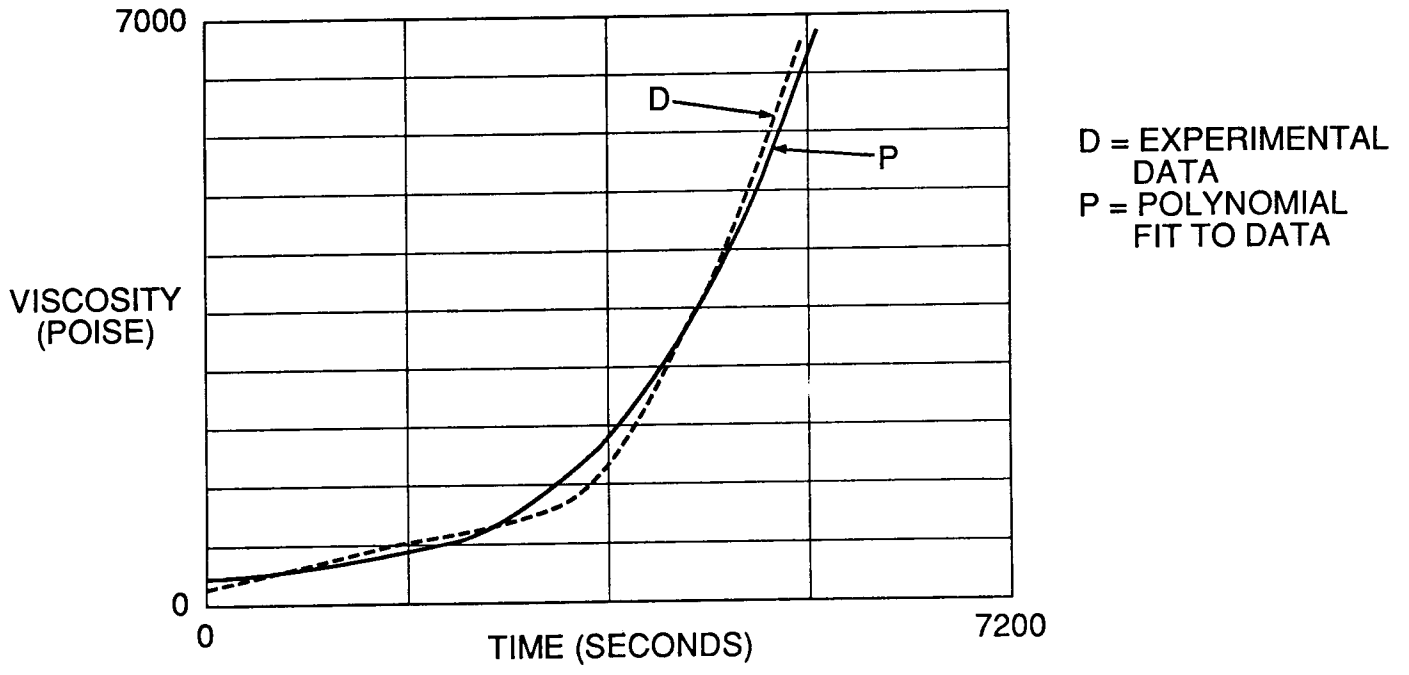


Figure 14. Viscosity versus cure time following mix of Chemseal 3808-A2 parts A and B. Curve D is fit exactly to data points and curve P is a cubic polynomial fit for use in analytical calculations.

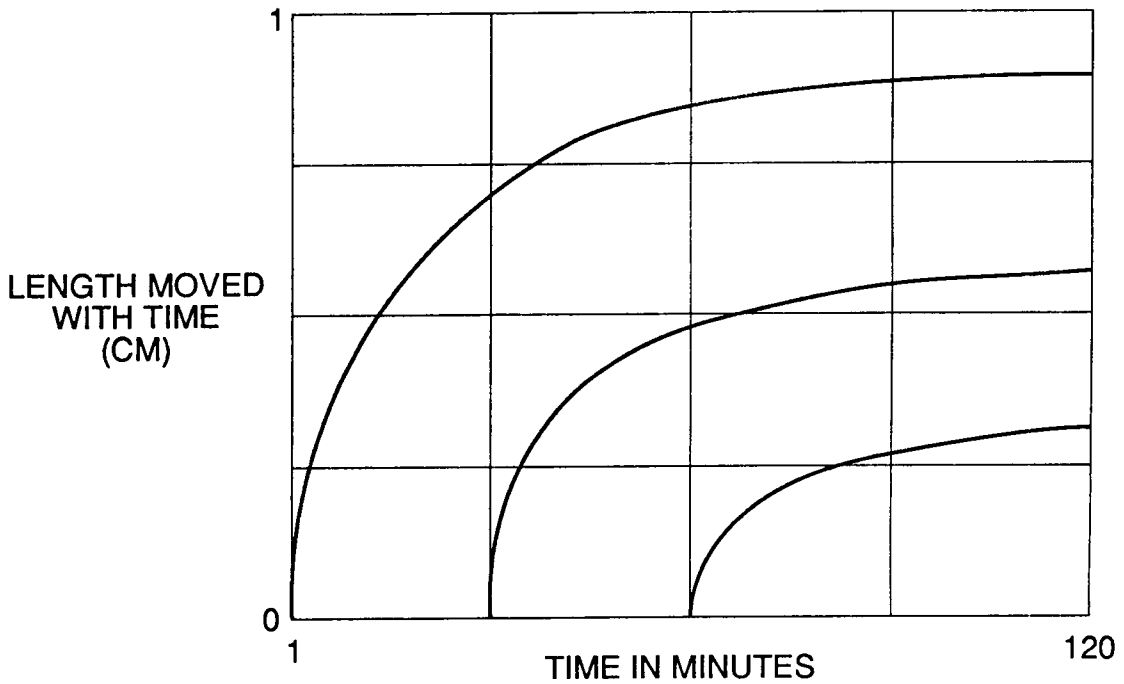


Figure 15. Chemseal flow into leak of 0.1-mil diameter scratch with 500 psi pressure applied constantly. The different curves represent time following Chemseal mix until application to the leak and pressure applied.

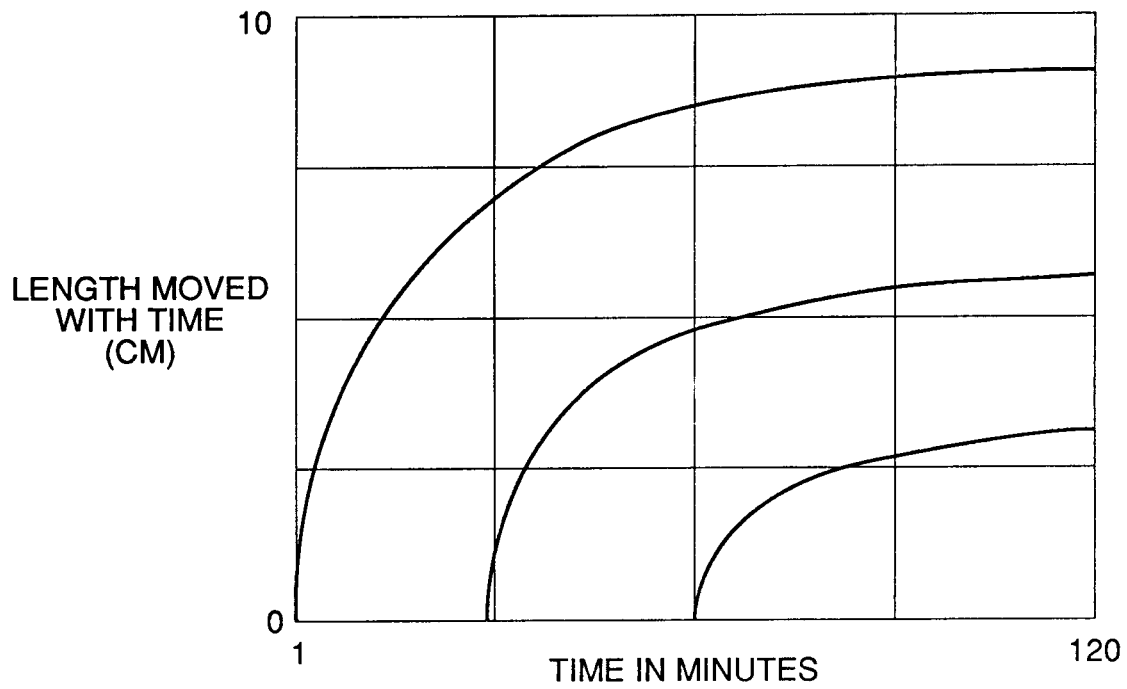


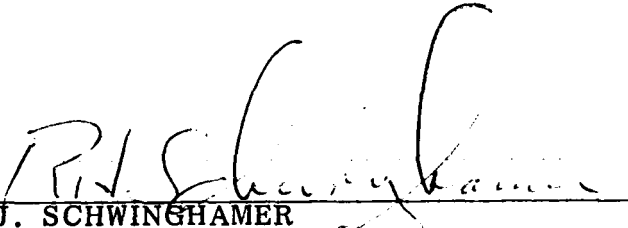
Figure 16. Chemseal flow into leak of 1.0-mil diameter scratch with 500 psi pressure applied constantly. The different curves represent time following Chemseal mix until application to the leak and pressure applied.

APPROVAL

CHEMSEAL 3808-A2 PENETRATION INTO SMALL LEAK PATH

By M. R. Carruth, Jr. and R. F. DeHaye

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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Director, Materials and Processes Laboratory