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PIPELINE TRANSPORTATION OF  
HEAVY OIL-IN-WATER EMULSIONS

BY



BARBARA WYSLOUZIL

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH  
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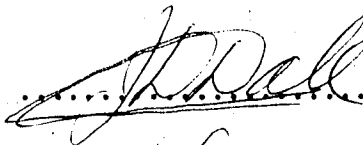
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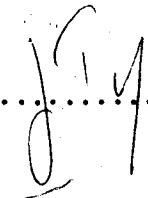
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## ABSTRACT

The pipeline transportation of heavy oils as oil-in-water emulsions has been proposed as an alternative to blending the crude with natural gas condensate or other diluent. A laboratory scale closed loop, with a 2 cm (3/4 inch) diameter test section, was constructed to investigate the behaviour of an emulsified Cold Lake crude oil under pipeline flow. Pressure drop was measured as a function of flow rate for freshly produced emulsions to establish generalized correlations of friction factor versus Reynolds number. Stability was observed over long term pipeline flow. The time at which the emulsion breakdown occurred was found to be a function of oil concentration and flow rate. This was clearly indicated by a simultaneous change in the system variables of pressure drop, temperature and power required to turn the pump at constant speed.

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# LIST OF SYMBOLS, NOMENCLATURE OR ABBREVIATIONS

cm	centimeter
cs	centistoke
cp	centipoise
D	pipe inner diameter
e	pipe roughness
f	friction factor ( $D\Delta P/2Lv^2\rho$ )
k	Boltzmann's constant
kg	kilogram
km	kilometer
kPa	kilopascal
l	liter
L	length of pipe section
m	meter
mA	milliampere
mPa·s	millipascal-second
P	pressure
Q	flow rate
Re	Reynolds number ( $Dv\rho/\mu$ )
T	temperature
v	bulk velocity
$\gamma$	kinematic viscosity ( $m^2/s$ )
$\eta$	viscosity
$\mu$	absolute viscosity (mPa s)
$\rho$	density



## CHAPTER 1 : INTRODUCTION

### 1.1 Heavy Oil Occurrence and Transportation

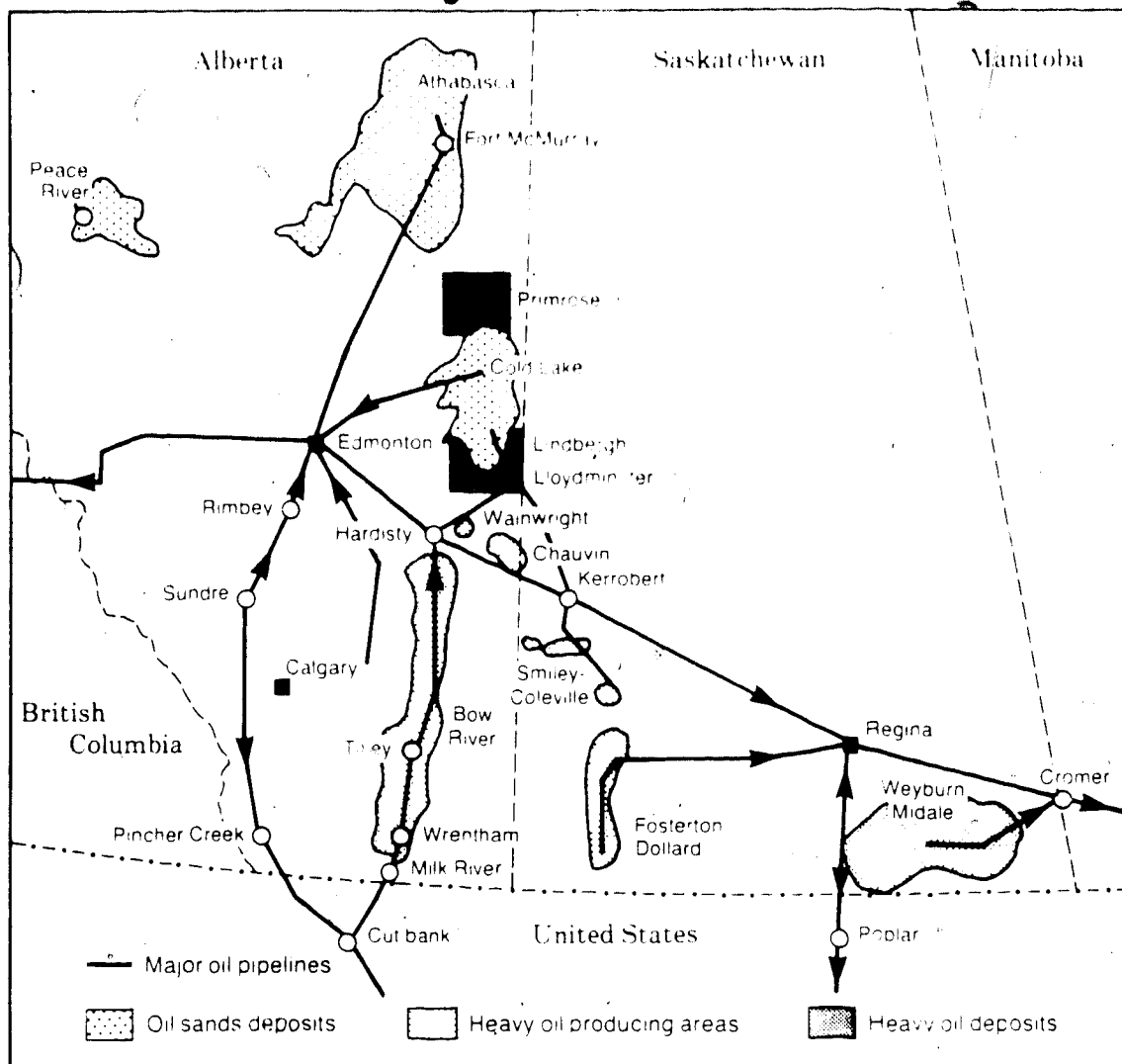
As reserves of conventional crude oils continue to decrease, Canada is becoming increasingly dependent on heavy oil and tar sands as a source of hydrocarbon liquids. In general, heavy crude oils and bitumen have an API gravity of  $10^{\circ}$  to  $20^{\circ}$  at  $15^{\circ}\text{C}$  and have viscosities ranging from 300 to several thousand centistokes at  $37.8^{\circ}\text{C}$  ( $100^{\circ}\text{F}$ ) [1]\*.

The major deposits of heavy oil in Western Canada are shown in Figure 1 [2]. These constitute a significant portion of the total oil reserves in this region. Table 1 [3] gives an estimate of the heavy crude oil and bitumen reserves for the top 10 countries in the world and an estimate for the world totals. Canada clearly has a large portion of the total estimated reserves. Thus transportation of heavy crude to processing plants is an important subject for Canadian producers.

The usual economic range of crude oil viscosity for pipeline transportation is 10-1000 cs ( $10^{-5}$ - $10^{-3}$  m<sup>2</sup>/s) at pipeline temperature. Figure 2 shows the variation of oil viscosity with temperature for a number of crude oils, both heavy and light [4]. With ground temperatures in Alberta in the range from  $-5^{\circ}$  to  $15^{\circ}\text{C}$  ( $23^{\circ}$  to  $68^{\circ}\text{F}$ ), heavy crude oils are not transportable in conventional pipelines.

---

\* Numbers in square brackets refer to references.



**Figure 1:** The major heavy oil deposits, producing areas and oil sands deposits of Western Canada (from [2]).

Table 1: Estimated remaining total reserves of heavy and extra heavy crude oil and bitumen. Summary of top 10 countries and world totals (million barrels, from [3])

Country	Heavy		Extra-Heavy		Bitumen		Total
	Reserves	Undiscovered	Reserves	Reserves	Undiscovered		
Venezuela	10,512	165,500	57,067	2		233,082	
Canada	240	3,679		48,777	85,000	137,696	
U.S.S.R.	1,958	20,510		112,369		134,737	
Nigeria	985	31,000	1,022	4,000		37,007	
Mexico	524	16,000	18			16,543	
U.S.A.	7,926	2,214	14	2,722	880	13,757	
Iran	5,380	1,300				6,680	
Madagascar	600			5,460		6,060	
Iraq	1,768	3,900				5,668	
Saudi Arabia	996	2,900				3,896	
World Totals	42,329	248,892	58,848	173,643	85,880	609,629	

Note: 1 barrel = 0.159 m<sup>3</sup>

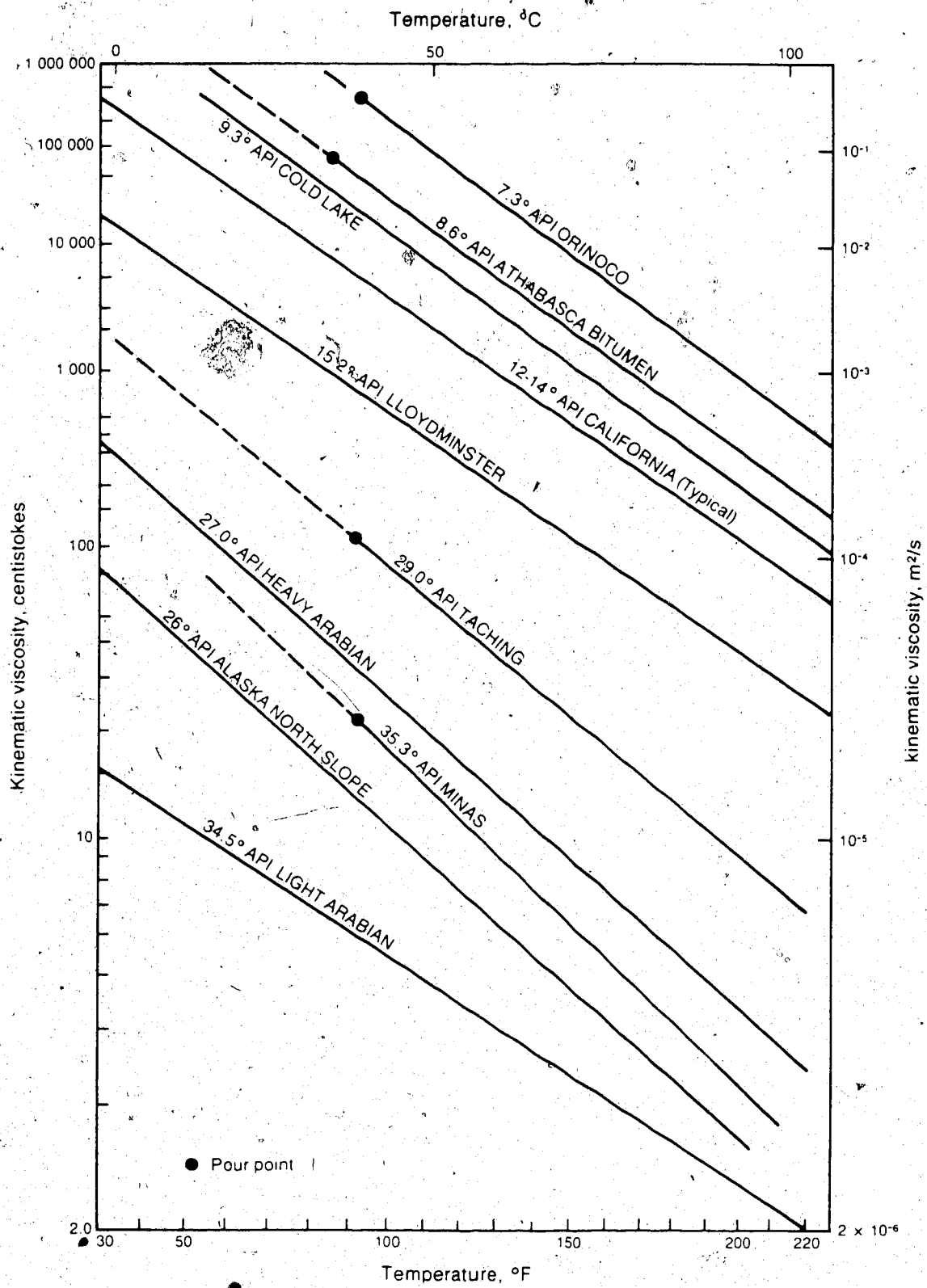


Figure 2: Crude oil and bitumen viscosity data (from [4]).

Current methods for transporting heavy crude oils and bitumen include:

- 1) trucking the oil
- 2) diluting the heavy crude with a light crude or natural gas condensate
- 3) heating the oil
- and 4) upgrading the heavy crude or bitumen on site to form a partially refined or synthetic crude.

Trucking is useful in moving small amounts of material. Thus trucks are primarily used to bring material from remote pilot plants or lease tanks to central locations [5].

The reduction in viscosity that is obtained by blending condensate with heavy crude oils is shown in Figure 3 [4]. This technique is currently practiced to move Lloydminster crude through the Interprovincial Pipeline system. Alberta Energy Company operates a dual pipeline system to transport blended heavy oil from Cold Lake to Edmonton, Alberta and to return the recovered diluent for reuse [6]. Recent forecasts for increases in total bitumen and heavy oil production, indicate that there is a pending diluent shortage as early as 1990 if blending remains the major solution to transportation [2].

Hot oil pipelines have been in operation world wide since the late 1950's to transport both heavy crude oil and heavy fuel oil [4]. One of the most significant is the Alyeska pipeline which transports 70°C (160°F) crude oil 1300 km (800 miles) in a 122 cm (48 inch) pipeline from Prudhoe Bay to Anchorage, Alaska.

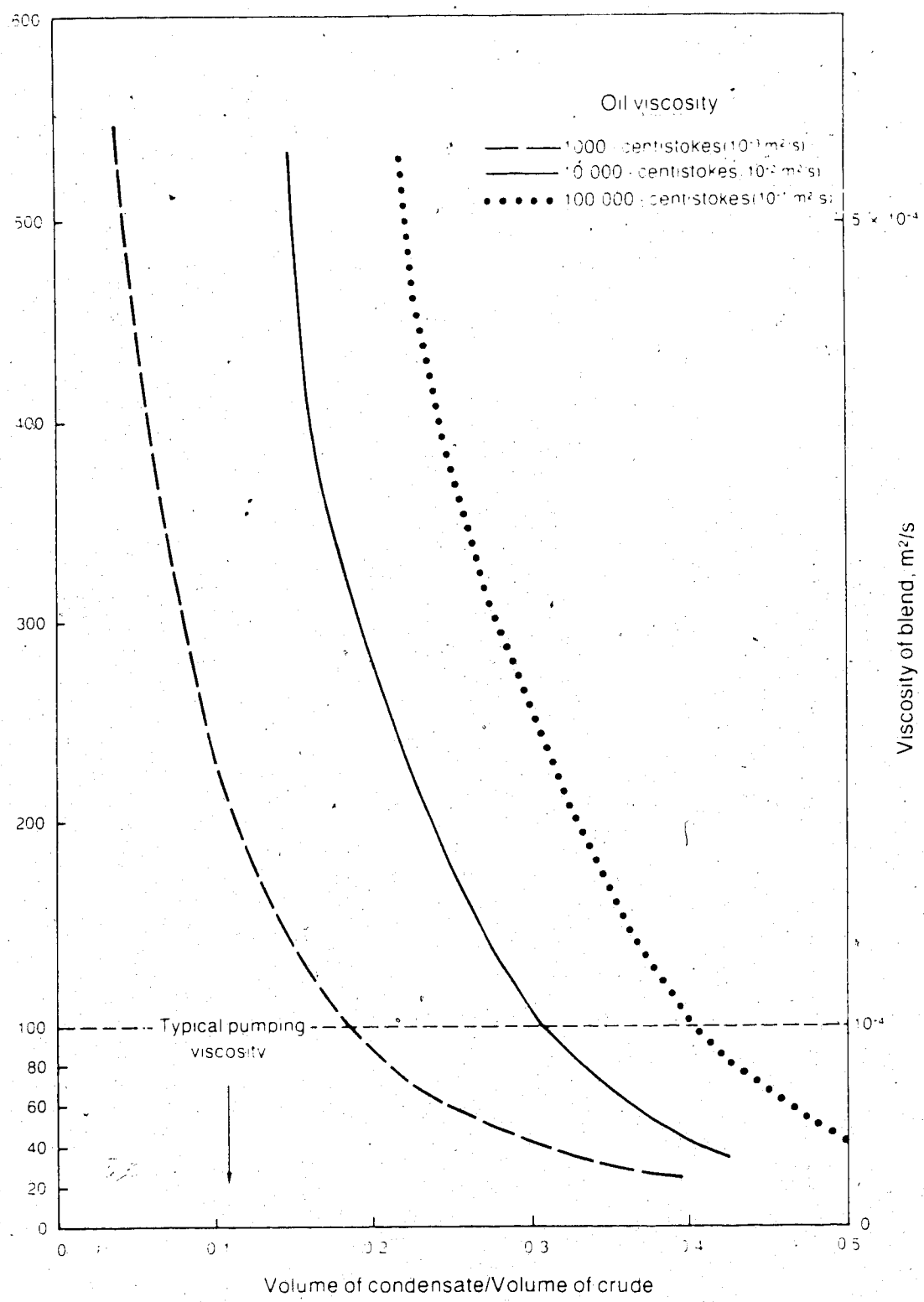


Figure 3: Blended heavy oil viscosity data (from [4])

Syncrude Canada Limited and Suncor Limited both operate large scale oil sands plants where synthetic crude oil is produced by upgrading of mined Athabasca bitumen. This upgraded crude oil is then easily transported in conventional pipelines.

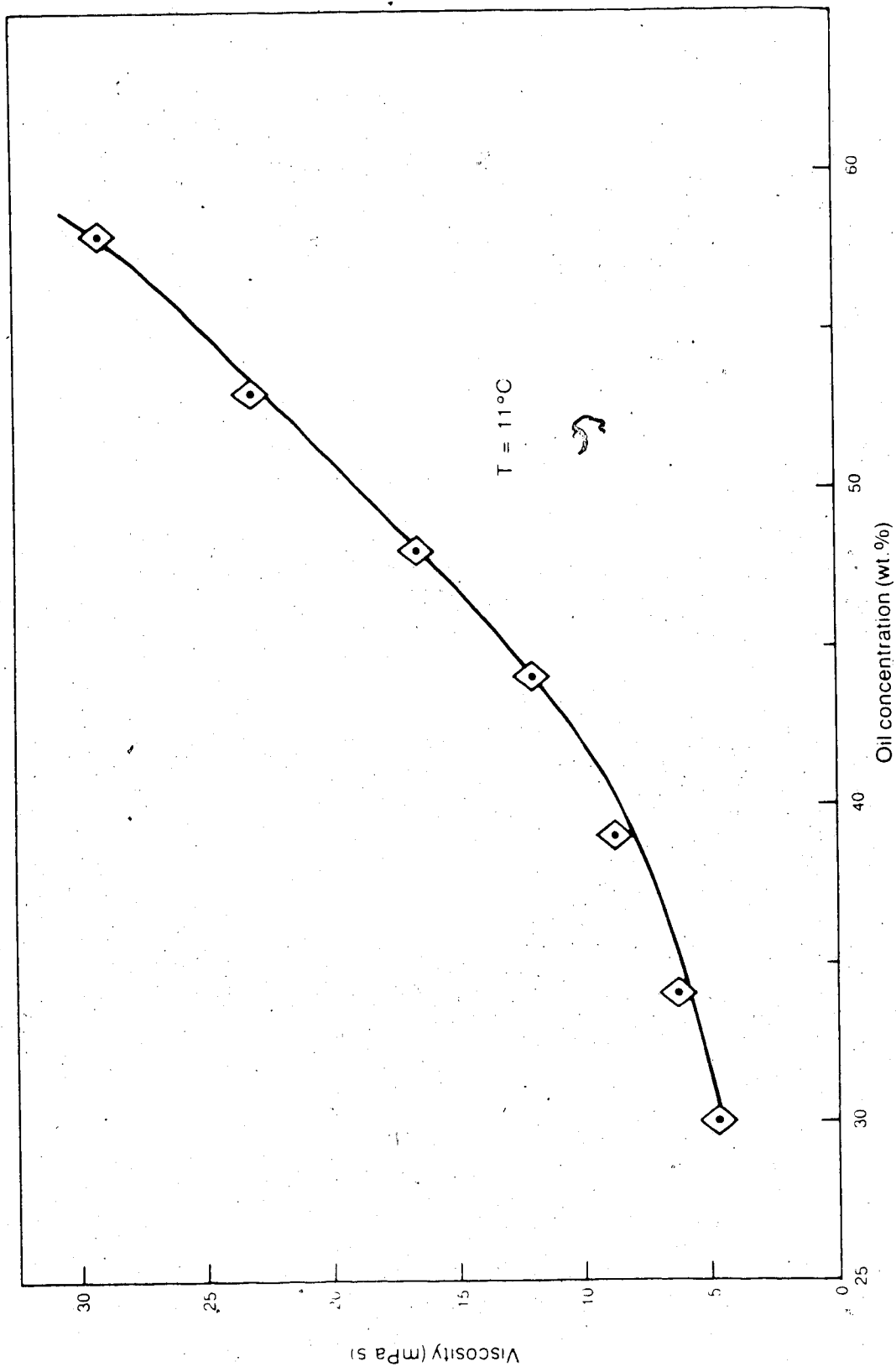
### 1.2 Emulsion Pipelining

Another method to overcome the high viscosity barrier is to emulsify the crude oil in water to produce a low viscosity emulsion which can be transported in a conventional pipeline.

The viscosity of an emulsion more closely reflects the viscosity of the continuous phase. Consequently, the viscosity of a heavy crude oil-in-water emulsion is much less than the viscosity of the heavy crude itself. For example, a 50% emulsion of a Cold Lake crude has a viscosity of only 20 mPa·s at 15°C, as opposed to the crude oil viscosity of over 400,000 mPa·s at 15°C. Figure 4 shows the variation of the emulsion viscosity with oil concentration for emulsions made with this crude at 11°C.

The emulsion pipelining concept consists of three steps.

- 1) The heavy crude oil or bitumen is emulsified in water in the presence of a surface active agent.
- 2) The emulsion is transported in a conventional pipeline system.
- 3) The emulsion is broken at the receiving terminal and the oil is recovered for further processing.



**Figure 4:** The variation of emulsion viscosity with oil concentration for a Cold Lake heavy crude oil.



Two examples of operating emulsion pipelines are mentioned in the literature. Emulsions of a waxy crude oil in water have been successfully used in a pipeline system in Indonesia since 1962 [7]. Marsden and Ranghavan reported that Getty Oil Ltd. has operated a 13 mile heavy oil emulsion pipeline in the Kern Area of California for about thirty years [8], transporting a 13.5°API crude as a 50% oil emulsion. Further investigation revealed that emulsions are not formed before the oil and water are introduced into the pipeline, nor are they considered desirable [9]. Thus this is not an emulsion pipeline in the context presented here. Neither pipeline directly addresses the problems that would be encountered in the Canadian context, those of long distance transportation of heavy crude emulsions in a cold climate.

### 1.3 Emulsion Pipelining Literature Review

There are a large number of patents available that describe the advantages of operating with oil-in-water emulsions rather than crude oil or water-in-oil emulsions. Most patents describe the great reduction in viscosity possible when viscous crude oils are emulsified in water. The types of surfactant are detailed, as well as the concentration of the surfactant and the oil/water ratios to be used. Pipelining of the resulting emulsion is then recommended as a method of transporting viscous crudes. Very few patents describe actual pipeline tests.

Appendix A lists the patents dealing with emulsion pipelining. A summary of each patent is presented.

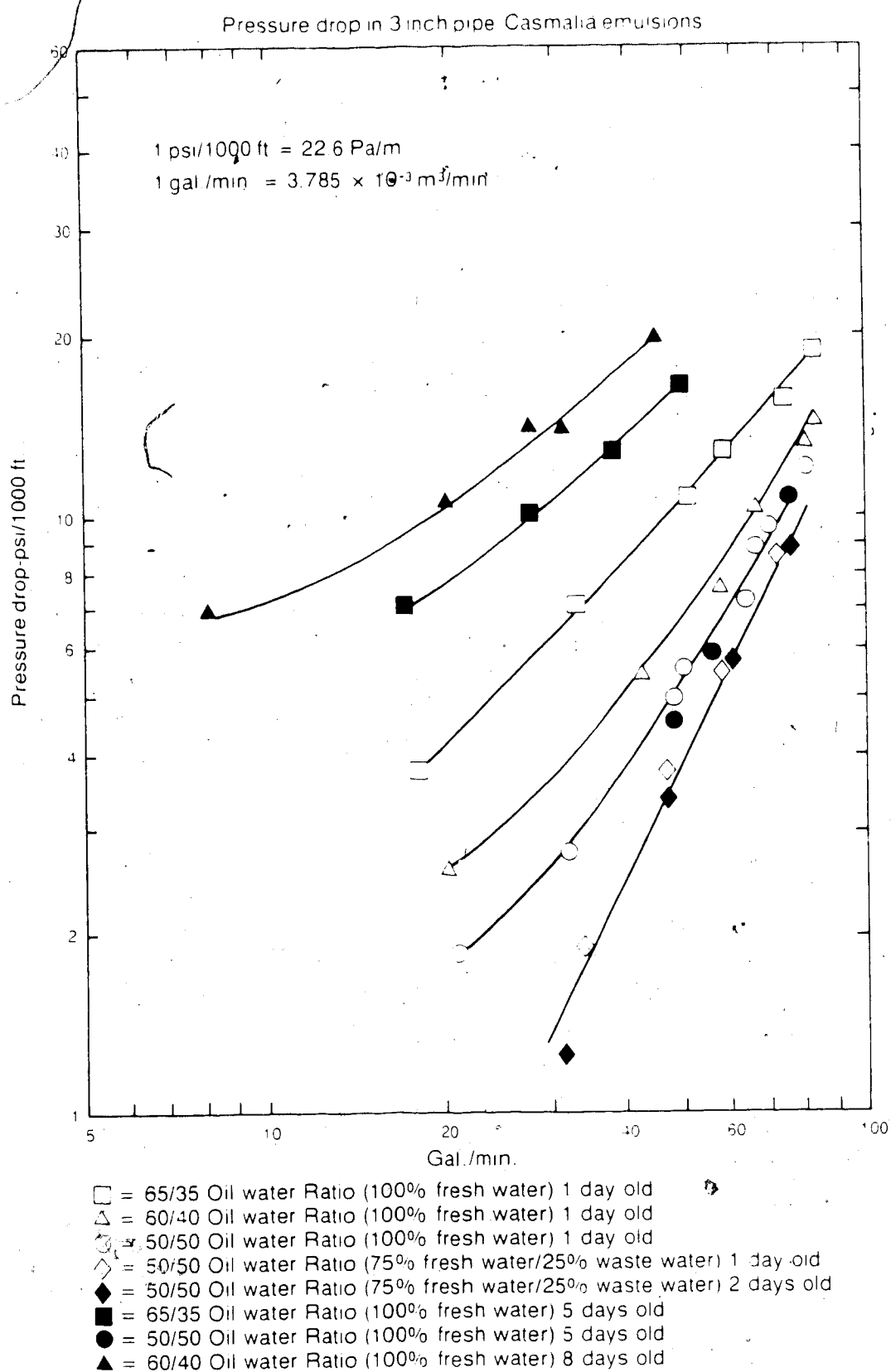
The patent with the most direct bearing on the present work is U.S. 3487844 [10], assigned to Simon, McAuliffe, Poynter and Jennings of the Chevron Research Company, Jan. 6, 1970. Here, crude oil is emulsified in the presence of an emulsifying agent with water containing a base such as sodium hydroxide (NaOH). No emulsifying agent is required if the crude is asphaltic and contains saponifiable constituents capable of forming effective emulsifying agents with the base solution. If the crude oil is non-asphaltic, the recommended procedures are to 1) add an emulsifying agent, such as a commercial emulsifier, 2) mix in some asphaltic crude or 3) add an extract obtained from a low gravity asphaltic crude.

The recommended oil to water ratios range from 50/50 to 70/30 on a volume basis. The recommended sodium hydroxide concentration is between 0.05 to 1.4% by weight based on total water. Table 2 illustrates the ranges of sodium hydroxide that were used to produce 70% by volume oil-in-water emulsions [10].

Several large scale tests were conducted by the authors of the above patent to demonstrate the feasibility of the process. Six batches of Casmalia crude oil-in-water emulsion were made and circulated through 600 m (1970 feet, equivalent corrected length) of a 7.6 cm (three inch) pipe. Figure 5 shows the variation of pressure drop with flow rate and the effects of oil concentration, water quality and age of the

Table 2: Effective ranges of sodium hydroxide required in the aqueous phase to form 70% by volume oil-in-water emulsions (from [10]).

Crude Oil	NaOH Concentration, weight percent in Water
Midway - Sunset A	0.4 - 1.4
Midway - Sunset B	0.05 - 0.5
Midway - Sunset C	0.1 - 1.0
West Coalinga A	0.05 - 0.3
West Coalinga B	0.1 - 0.5
Boscan	0.05 - 0.3
Casmalia	0.1 - 0.6
Cat Canyon	0.1 - 1.0



**Figure 5:** The variation of pressure drop with flow rate for Casmalia crude oil-in-water emulsions (from [10]).

emulsions.

A second series of tests was conducted in a 4 km (2.5 mile) long 10 cm (four inch) pipeline. During these tests the emulsion was allowed to stand for several days to simulate pipeline down conditions. No startup difficulties were encountered.

This patent clearly demonstrates the feasibility of emulsion pipelining by making, transporting and breaking Casmalia crude oil emulsions. However, generalized correlations, in the form of friction factor versus Reynolds number are not presented and long term stability tests were not attempted.

The viscosity of oil-in-water emulsions is less sensitive to temperature than the viscosity of the crude oil itself. In U.S. Patent 3670752, June 20, 1972 [11], Marsden and Rose proposed transporting Prudhoe Bay crude oil-in-brine emulsions at low temperatures to avoid environmental damage. By cooling the emulsions to  $-10^{\circ}$  to  $-1^{\circ}\text{C}$  ( $15^{\circ}$  to  $30^{\circ}\text{F}$ ) pipelines in the arctic could be buried without thawing the permafrost. In addition, gas produced with the crude oil could be redissolved in the cold emulsion and transported in the same pipeline.

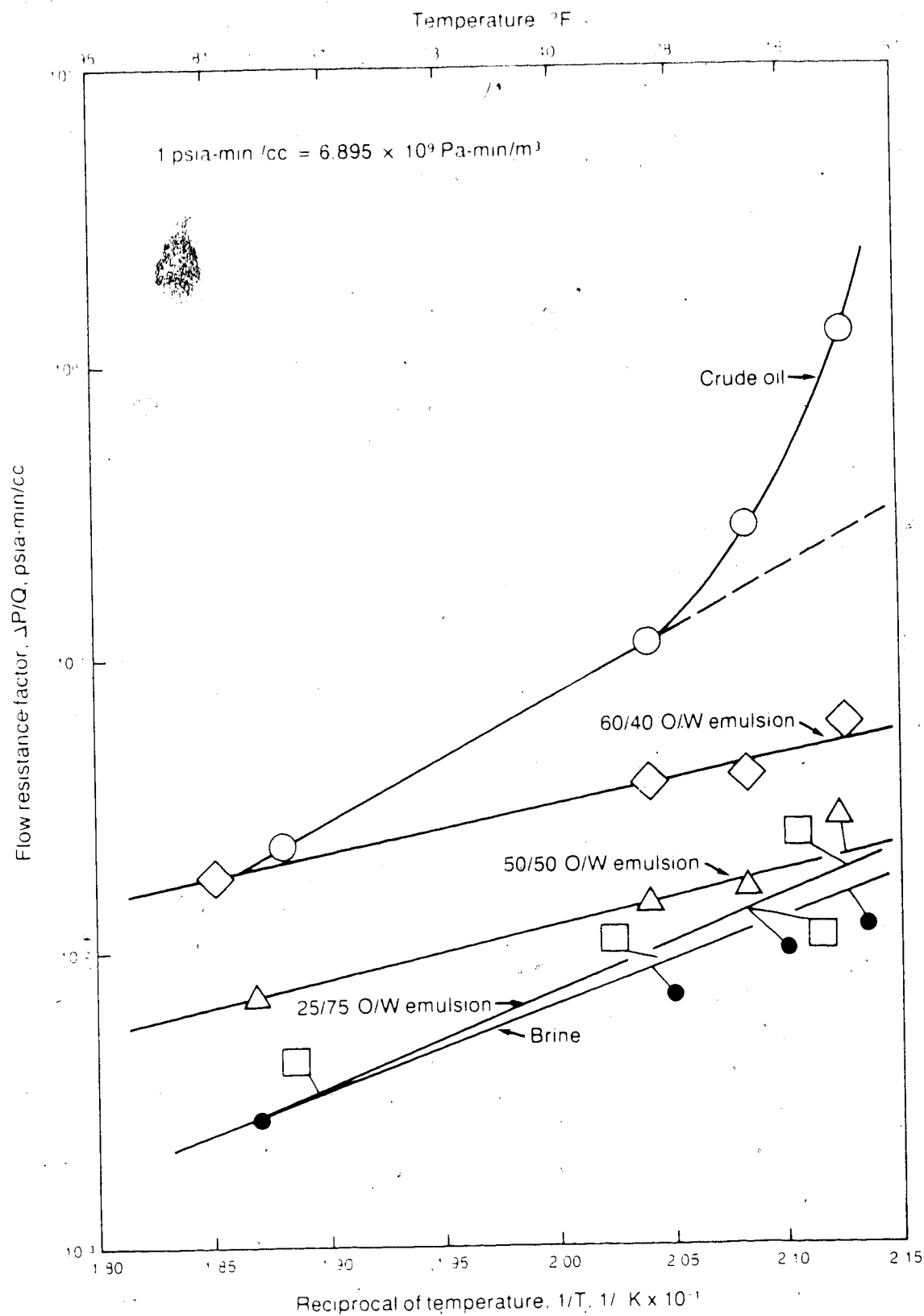
Marsden and Rose prepared emulsions with Prudhoe Bay crude, 20% by weight NaCl solutions and from 0 to 2% Triton X-114 emulsifier [12]. The pressure drop of these emulsions was measured across a 3.4 m (134 inch) long, 0.6 cm (1/4 inch) inner diameter coiled glass tubular flow resistor at a given temperature and flow rate. The viscosity of the

emulsions was measured both on-line and for samples taken from the system. All of the flow experiments were conducted in the laminar flow regime. The results were correlated as the variation of flow resistance with the inverse of temperature at different emulsion concentrations. This is shown in Figure 6. The results were not generalized further and the question of stability was not considered.

Zakin, Pinaire and Borgmeyer considered the effects of oil viscosity, temperature and oil concentration on the turbulent flow characteristics of oil-in-water emulsions [13]. As well, the effects of these variables on the specific energy loss were evaluated.

Stable emulsions were made from various refined mineral oils, tap water and 0.5% Triton X-114 emulsifier using a Monrohouse colloid mill. The range of oil concentrations investigated was from 50 to 75% by weight.

Oil viscosity was varied from  $1.53$  to  $61.5 \times 10^{-6} \text{ m}^2/\text{s}$  at  $35^\circ\text{C}$ . This was found to have little effect on the pumping energy requirements for emulsions in this range of concentration. Temperature was varied from  $30^\circ$  to  $45^\circ\text{C}$  and resulted in a decrease of less than 5% in pressure drop. This decrease was attributed to the decrease in the viscosity of the continuous water phase. Oil concentration was found to have little effect on the variation of pressure gradient with velocity but had a marked effect on the energy loss per mass of oil transported in the fully turbulent region. These last two findings are illustrated in Figure 7 and Figure 8.



**Figure 6:** The variation of flow resistance factor with reciprocal temperature for various oil-in-water emulsions (from [12])

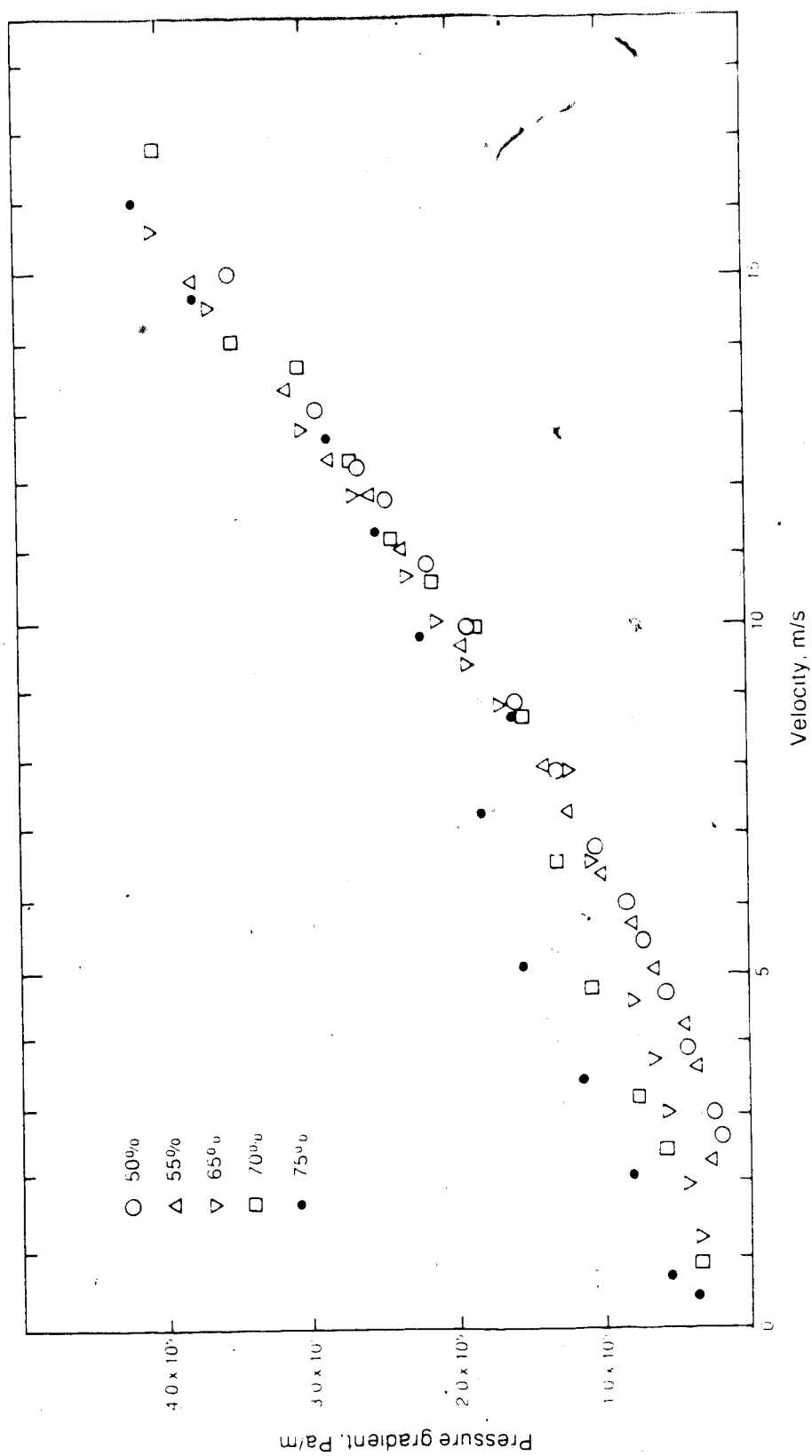
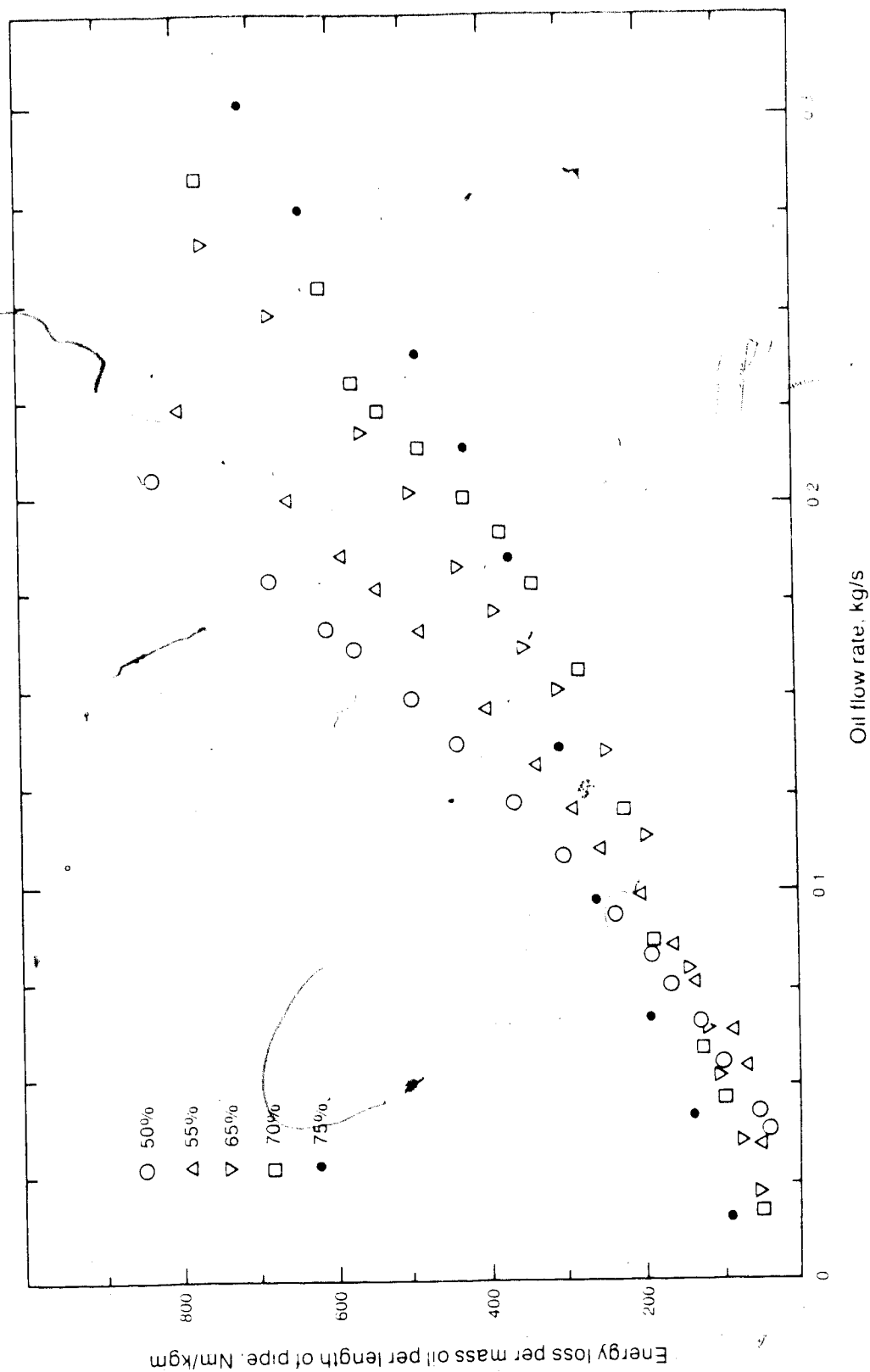


Figure 7: The variation of pressure gradient with velocity for 50 to 75% oil-in-water emulsions (from [13])





**Figure 8:** The variation of energy loss per mass of oil per length of pipe with oil flow rate for 50 to 75% oil-in-water emulsions (from [13]).

The variation of pressure drop with flow rate was compared with that predicted by the Dodge-Metzner equation for purely viscous non-Newtonian fluids. For the emulsions studied, the measured pressure drops were consistently 8 to 26% lower than those predicted by the Dodge-Metzner correlation. This was attributed to the visco-elastic character of the concentrated emulsions.

The range of generalized Reynolds number investigated was from 300 to 10000. No investigation regarding the stability of these emulsions was conducted.

In U.S. Patent 4343323, August 10, 1982 [14], Kessick and St. Denis described a dual pipeline system for heavy crude oil-in-water emulsions. Emulsions were made with crude oils from the Cold Lake and Lloydminster areas of Alberta using a sodium hydroxide solution that had been deaerated and had a pH of at least 11. Measurements showed that the emulsions had viscosities low enough to permit pumping and pipeline transportation even at 4°C. By breaking the emulsions with slaked lime both the oil and the sodium hydroxide solution were recovered. The sodium hydroxide solution could then be recycled to the front of the pipeline system. No flow tests were conducted with emulsions.

In U.S. Patent 4276094 [15], Gutnick and Rosenberg described an emulsifier, "alpha emulsan", produced by the bacterial action of *Acinetobacter Caloaceticus*. This bioemulsifier was found to be a good emulsion stabilizer. However, a surfactant was still required to form the initial emulsion.

Petroferm U.S.A. used forty barrels of a Boscan Venezuela crude oil to form a 70% oil-in-water emulsion using a surfactant and the emulsan bioemulsifier [16]. This was circulated in the system shown in Figure 9 at the conditions listed in Table 3. After 4 days of continuous pumping, the flow was stopped for three days. The pressure drops and flow rates measured after restarting the system were similar to those observed before the shut down. No data were presented. The total distance travelled during the test was 600 km (380 miles).

Briant, Fruhman, Quemada and Makria studied the stability and rheology of emulsions made with Boscan crude oil from Venezuela [17]. The aqueous solutions contained 0.25 to 1% Tall Oil and 0.0625 to 0.25% NaOH. Emulsions were made by heating the aqueous phase to 80°C and adding the crude oil which was at 120°C, while agitating with an Ultraturex emulsifier.

The most stable emulsions were made with distilled water, a Tall Oil/NaOH ratio of 4, with >1% Tall Oil. The emulsions were found to be shear thinning. For a given shear rate, the apparent viscosity increased rapidly with oil concentration. Surfactant concentration, salinity and age of the emulsions also affected the apparent viscosity.

Further work by Fruhman and Briant [18] included two Canadian heavy crude oils. Similar results were obtained in the rheological behaviour. Droplet size and the chemical nature of the disperse and continuous phases were found to influence the viscosity at high shear

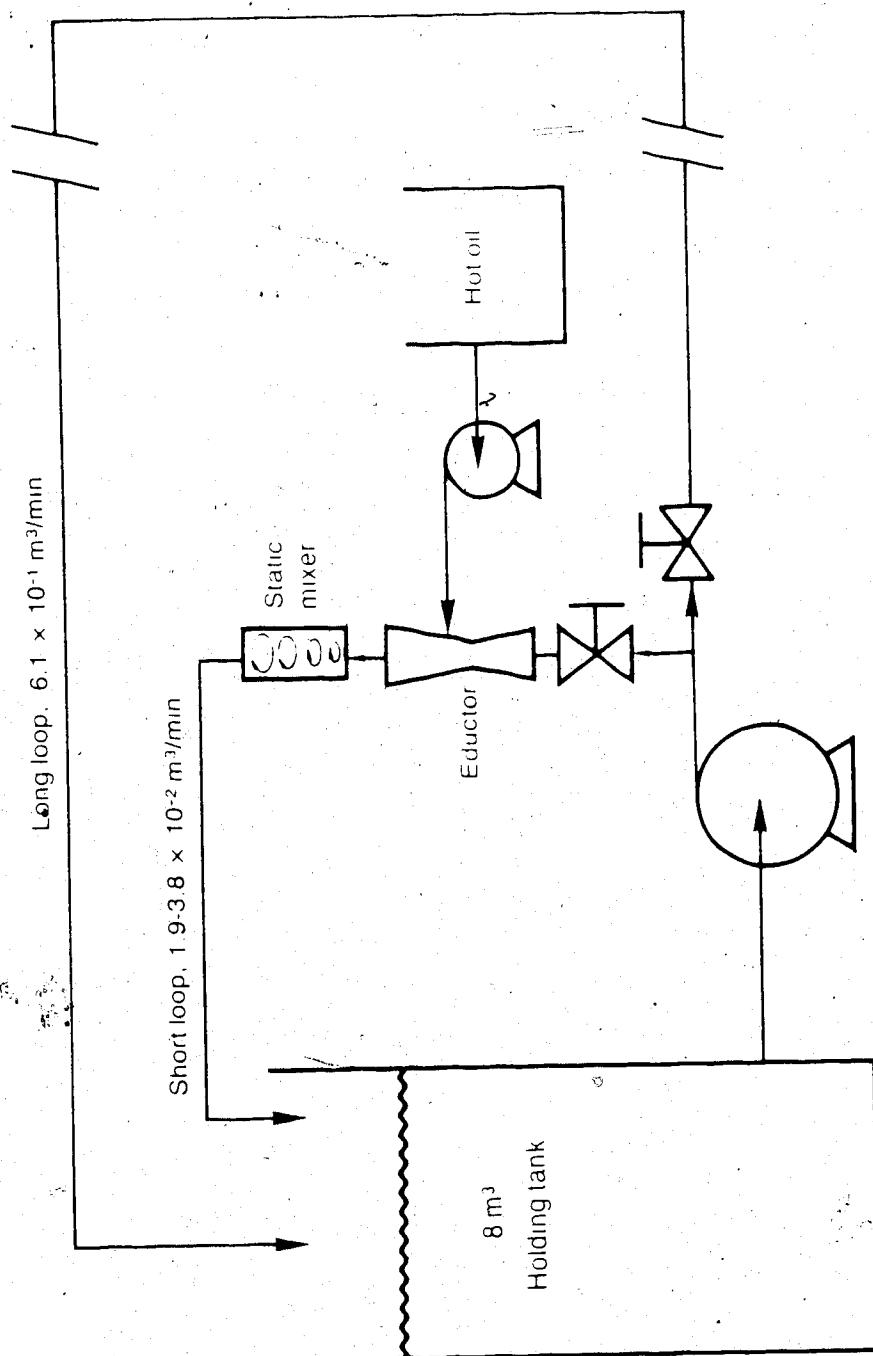


Figure 9: Pipeline test loop used by Petroterm U.S.A. for 70% Boscan crude oil-in-water emulsion flow tests (from [16])

Table 3: Summary of emulsified crude pilot test data  
for Petroferm U.S.A. test loop (from [16])

Oil/Water Ratio	70/30
Surfactant/Oil Ratio	1/500
Total running time	96 h
Shutdown time prior to restart	64 h
Average flow rate during run	0.61 m <sup>3</sup> /min
Average flow velocity	2.0 m/s
Pipe inner diameter	0.079 m
Distance pumped	approximately 610 km
Number of pump transits	Approximately 530
Apparent viscosity of emulsion	70 mPas
Viscosity of Boscan crude oil at 60°F	190,000 mPas
Comparable flow rate in 20" line	34,000 m <sup>3</sup> /day
Comparable flow rate in 30" line	76,000 m <sup>3</sup> /day

rate.

Recent work by Rhodes and Pal [19] with oil-in-water emulsions showed that at dispersed phase concentrations of less than 55%, these emulsions behaved like Newtonian fluids in both the laminar and turbulent flow regimes.

#### 1.4 Emulsion Stability

The major concern in emulsion pipelining is the stability of the emulsions during transportation in a pipeline. Emulsions must be stable over a wide range of temperatures and shear rates so that they will not separate extensively or invert in the pipeline. Emulsions must however be easily broken at the receiving terminal to recover the oil. This section will discuss the basic concepts of emulsion stability and in particular the stability of emulsions under shear.

Emulsions are thermodynamically unstable systems because of their large surface free energy per unit volume. The stability of emulsions over extended periods of time is the result of three mechanisms : charge stabilization, stearic stabilization and stabilization by adsorbed species. Forces that act on the disperse phase of an emulsion may enhance stability or they may aid in separating the emulsion into its two component phases.

External force fields, such as gravity and applied electrostatic forces, act on the dispersed phase particles of an emulsion over an essentially

infinite distance. On an interparticle level, electrical double layer forces, dispersion and hydrodynamic forces act on the droplets. As well, diffusional forces are present due to bulk concentration gradients. Details concerning the molecular structure of the droplets and interfaces are required when considering coalescence and phase inversion.

The instability of an emulsion is determined by observing changes in the structure of the emulsion. Any overall changes in the structure of an emulsion are described by Tadros and Vincent [20] as combinations of five basic changes. These basic changes are

- 1) Establishment of a bulk droplet concentration gradient with no change in droplet size. This process is referred to as creaming or sedimentation.
- 2) The buildup of aggregates of droplets in the emulsion with no change in the basic droplet size. This process is called flocculation.
- 3) The formation of larger droplets by the coalescence of droplets that have come together by sedimentation or flocculation. This results in a change in the droplet size distribution and is referred to as coalescence.
- 4) The change in droplet size due to mutual miscibility of the disperse and continuous phases. For a polydisperse system the process known as Ostwald ripening results in larger droplets forming at the expense of smaller droplets.
- 5) The reversal of the continuous and disperse phases to form the inverted emulsion.

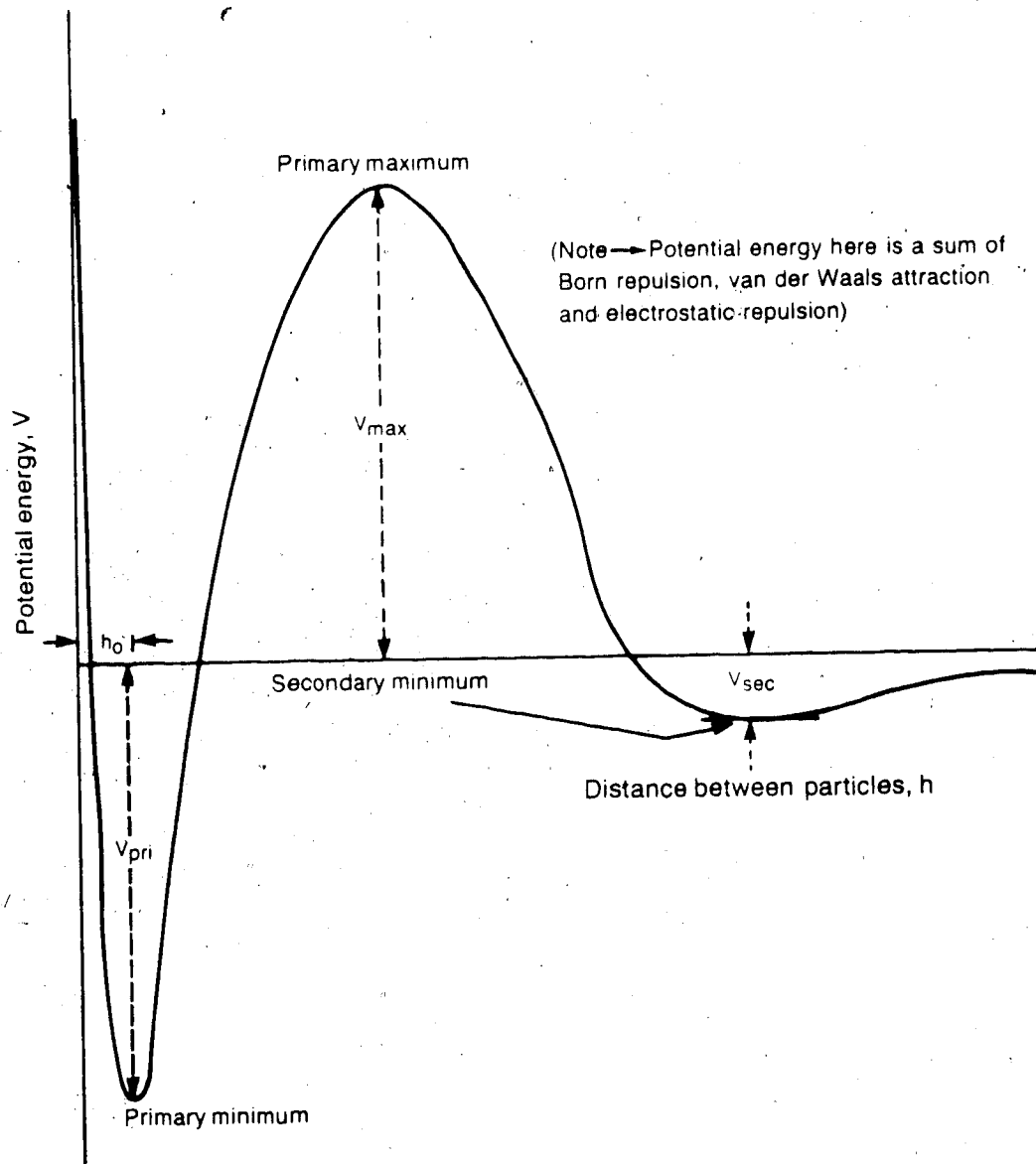
The stability of emulsions has been studied by considering the forces that operate between two dispersed phase particles. The theory that describes the competition between electrostatic repulsion and van der Waals attraction between two dispersed phase particles is the DLVO theory. This theory was developed independently by Derjaguin and Landau [21] and Verwey and Overbeek [22]. Figure 10 [23] is the potential energy curve for two isolated particles when the following forces are considered

- 1) short range Born repulsion
- 2) long range attractive van der Waals forces and
- 3) long range electrostatic repulsive forces due to double layer overlap.

Depending on the relative magnitudes of these forces the curve may exhibit up to two minima and one maximum. At the minima, van der Waals attractive forces predominate over repulsive forces and the droplets form stable binary pairs. The minimum at the lowest droplet separation distance is called the primary minimum while the minimum that occurs at larger droplet separation is called the secondary minimum. The energy of repulsion at the primary maximum acts as an activation energy controlling the rate of pair formation, or coagulation, in the primary minimum.

If the potential energy maximum is large compared to the thermal energy of the particles,  $kT$  (where  $k$  is Boltzmann's constant and  $T$  is the absolute temperature), the system should be stable against coagulation





**Figure 10:** The variation of potential energy between two particles with distance (from [23]).

due to the random Brownian motion of the particles, also called perikinetic coagulation. A height of  $15kT$  is considered insurmountable [24]. If the potential energy barrier does not exist, rapid coagulation occurs.

Flocculation may occur in the secondary minimum if it is deep enough to prevent separation of the particles by Brownian motion. A depth of about  $-3kT$  is usually sufficient [25]. Particles flocculated in the secondary minimum are relatively mobile and may frequently be separated by slight stirring. Particles coagulated in the primary minimum are essentially in contact.

Although the potential energy maximum for droplets in an emulsion may indicate the emulsion should be stable, externally applied forces can still be used to cause flocculation in the primary minimum. If the particles are brought into interaction distance by stirring, the process is called orthokinetic flocculation.

Both van de Ven and Mason [26] and Zeichner and Schowalter [27] have analysed the stability of colloid dispersions in flow fields by performing trajectory calculations on two particles. They calculated the conditions under which doublets formed under simple shear and extensional flow and whether these doublets were in the primary or secondary minima.

Practical application of the DLVO theory to bitumen-water systems has been demonstrated by Takamura and Chow [24]. Bitumen emulsion stability

for dilute emulsions (4% bitumen by volume) was found to agree well with that predicted by DLVO theory for different concentrations of aqueous salt solutions. As well, shearing experiments were found to be in agreement with predictions based on trajectory analysis.

No information was found on the stability of concentrated emulsions under shear.

## CHAPTER 2 : EQUIPMENT AND EXPERIMENTAL PROCEDURES

### 2.1 Outline of Experimental Program

The experimental program consisted of three stages. These were:

- 1) design, building and calibration of the laboratory pipeline loop
- 2) screening of emulsions made with different oils and emulsification techniques
- 3) investigating the pressure drop versus flow rate behaviour of fresh emulsions and stability of emulsions during long term pipeline flow.

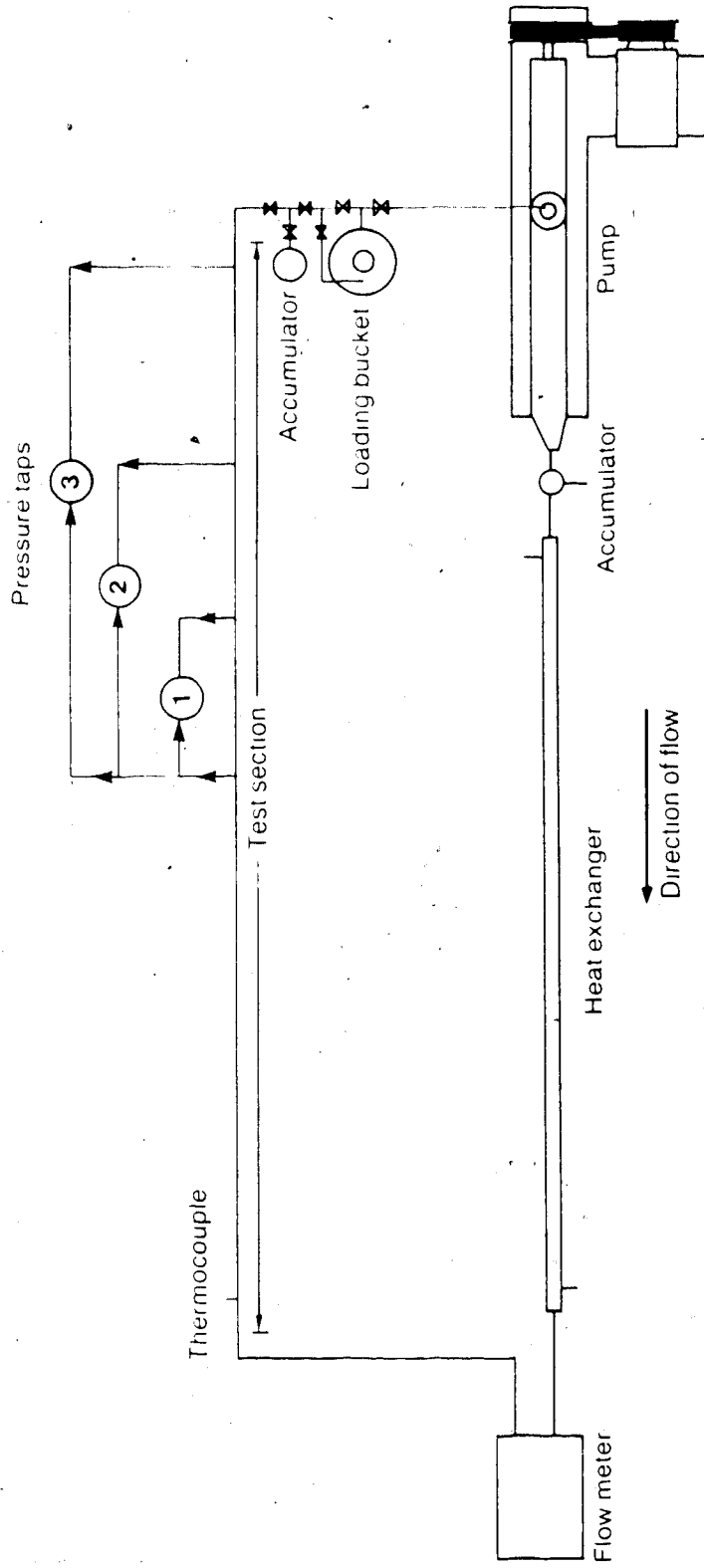
### 2.2 Pipeline Test Loop : Equipment

#### 2.2.1 Overview of Equipment

A plan view of the experimental pipeline is shown in Figure 11. The test loop was designed to establish steady flow of the emulsion at a constant temperature and permit measurement of the emulsion flow rate and the differential pressure due to flow in the test section.

Several considerations contributed to the final sizing of the pipeline and the major pieces of equipment. These included

- 1) the amount of laboratory space available
- 2) an emulsion product viscosity of 10 to 20 mPa s
- 3) desired Reynolds number ranges of 5000 to 10000 and later 500 to 2500



**Figure 11:** Plan view of pipeline test loop

4) no more than 50 liters of emulsion to be required per test.

The major components in the system are 1) a loading bucket, 2) a 3L8 Moyno pump, 3) a 3.6 m long double pipe heat exchanger with a surface area of  $3.8 \text{ m}^2$ , 4) a Micromotion mass flow meter, 5) a 5 meter test section, 6) three Rosemount model 1151 differential pressure transducers, 7) two accumulators and 8) an outlet hose.

The test fluid, emulsion or an oil, is introduced into the system via the loading bucket. It then proceeds through the pump to the heat exchanger. Following the heat exchanger is the flow meter where the mass flow rate (kg/min) of the emulsion is measured. The emulsion then enters the test section. Temperature and differential pressure are measured here. After the test section the emulsion is returned to the pump. The following sections describe the major components in the system.

### 2.2.2 Pump and Drives

The pump is a three stage Moyno pump, model 3L8, manufactured by Robbins-Meyers. This is a single screw type positive displacement pump consisting of a single threaded helical rotor rolling eccentrically in a double threaded helix. The rotor and stator form a series of sealed cavities 180 degrees apart, that progress along the pump as the single helix rotates. The rotor and stator are constructed of hardened steel and Buta N rubber respectively. The pump has a maximum discharge pressure of 1500 kPa (225 PSI) and a capacity of 2.5 l/s (40 U.S.

gallons per minute) at 500 RPM. With an appropriate choice of drive motor and pulleys, test flows can be varied from 0.066 to 3.33 l/s.

Moyno pumps are designed to handle slurries and pastes under low shear conditions. Shear rates experienced by the fluid in the pump are a function of speed and design constants. For a size 8 rotor element, the pump manufacturer gives the shear rate at 100 RPM as  $77 \text{ s}^{-1}$ . This shear rate is much lower than the shear rate experienced by the fluid in the pipe. For example, at 30 RPM the shear rate in the pump is about  $23 \text{ s}^{-1}$  while the shear rate at the wall of the 2 cm (3/4 inch) pipe ( $8v/D$ ) is  $240 \text{ s}^{-1}$ . The shear rate in the pump may be important, as excessively high shear rates could distort the overall effects of pumping the emulsions for long periods of time.

Two motors were required to cover the various flow rate ranges in this study. For the high flow rate studies a 15 horsepower A.C. motor with a fixed speed of 1160 RPM was used. Pump speed was varied by using a Hi-Lo variable speed pulley drive manufactured by Sepco-Metcom, on the motor. A 50 cm (24 inch) sheave on the pump shaft reduces the driven speed of the pump to the desired range. For the low flow rate studies a 2 horsepower D.C. motor with a variable speed controller was used. The speed of the motor can be varied from 0 to 1750 RPM, but should be kept above about 900 RPM for long term running. A 10:1 reduction gearbox together with a V-belt and two pulleys reduce the output speed to the desired range. For a given motor and pulley setup the flow rate during a given test can usually be varied on the order of 3:1. The electrical power required by the D.C. motor was monitored during an experiment.

### 2.2.3 Accumulators

Although the discharge from the Moyno pump is relatively smooth, some problems were initially experienced with vibration in the system, especially when the system was run as a closed loop. Two accumulators were added to the system along with several pieces of flexible gasoline pump hose. With these modifications the vibration was much reduced. The installation of the flexible hoses also helps in isolating the pump from the test section and makes the alignment of the various parts of the circuit much easier.

The first accumulator is a 1 l Valvcon accumulator. This is simply a pressure vessel containing a rubber bladder to separate the process fluid from a pressurizing gas. As long as the gas pressure is between 20 and 80% of the pump discharge pressure, the accumulator dampens the variations in the flow rate and pressure and reduces vibration throughout the system.

The second accumulator was added to the system during the very low flow rate experiments. This accumulator is a glass column that can be pressurized slightly with nitrogen and does not contain a separating membrane. The pressure in this accumulator can be adjusted more finely and is required to even out the flow variations at the very low flow rates. This accumulator is near the pump suction and has two additional advantages. The first is that it allows the whole system to be run slightly above atmospheric pressure and thus avoids the possibility of



air entrainment into the system. The second is that higher flow rates are possible because the additional pressure prevents cavitation in the pump. By filling the column with the emulsion, samples drawn from the system during a test are replaced by material from the column without opening the system to atmosphere.

#### 2.2.4 Heat Exchanger

A 3.6 m double-pipe counter current heat exchanger is in between the pump and the flow meter. Heat generated by flow in the system is removed via the heat exchanger. Initially, cooling water was drawn from the city cold water tap, run through the exchanger and discarded. An ethylene glycol/ water recirculating system was introduced when city water temperatures rose above 6°C. The recirculating system provides adequate cooling for the low flow rate tests. Refrigeration was provided by a Blue M PCC-34-C portable cooling unit, with the final temperature controlled by a Haake D1 heater/ circulator. Cooling water temperatures were maintained within 1° to 2°C once steady state was achieved.

The pipeline loop was insulated to reduce temperature fluctuations in the system. Most of the tests were run at about 10°C to try and more closely simulate actual pipeline conditions. This was not possible for two of the very high flow rate tests where city water was too warm and the recirculating system could not handle the heat load. These tests were run at 25°C.

### 2.2.5 Flow Meter

Two Micromotion flow meters are used to cover the range of flow rates of interest. A model C-100, calibrated for 0-400 kg/min, is used for the high flow rate studies. A model B-50, calibrated for 0-45 kg/min, is used for the low flow rate studies.

The flow sensor in this type of a meter is a U-shaped tube which is vibrated at its natural frequency. The movement of the fluid, in combination with the vibration, causes the tube to twist. Magnetic position detectors determine the amount of twist and produce a linear signal proportional to the mass flow rate of the fluid. The meter is not affected by the fluid viscosity, entrained gases or material buildup on the wall. As well the meter appears as essentially another piece of pipe to the flowing emulsion.

### 2.2.6 Test Section

The test section is a 5 m single piece of 3/4 inch Schedule 40 ASTM grade A105 seamless black pipe. This is the type of pipe commonly used in pipeline applications. The first 2.5 m provide an unobstructed region for flow to develop fully before any measurements are taken. This corresponds to a distance of approximately 125 pipe diameters.

The minimum entrance length in laminar flow was calculated by Langhaar [28] to be  $0.058Re$  pipe diameters for circular pipes. For a Reynolds number of 2000 this corresponds to about 115 pipe diameters. In

turbulent flow, Olson and Sparrow [29] report the entrance length in circular tubes is of the order of 20 pipe diameters and is only moderately affected by the magnitude of the Reynolds number. Thus an entrance length of 125 pipe diameters should be sufficient to establish fully developed flow in the test section before pressure measurements are made.

Four collars were welded to the pipe at the pressure tap points to hold the necessary fittings. The pressure taps themselves are 0.8 mm (1/32 inch) holes drilled into the pipe. Two half collars were welded on the pipe at either end of the test section to hold thermocouple fittings. Thermocouples were placed into the fluid itself at these two points. Surface thermocouples were attached along the length of the test section to check for temperature variation along the pipe.

#### 2.2.7 Pressure Transducers

Rosemount differential pressure transducers are used to measure three pressure drops from a common high side to points 0.76, 1.52 and 2.44 m downstream. Different transducers are used to cover the wide range of pressure drops that were measured. Transducer calibrations ranged from 0-2 kPa to 0-150 kPa.

The sensing element of the differential pressure transducer is a stretched spring element separated from the process fluid by isolating diaphragms and oil fluid fill. The spring is deflected by an imposed differential pressure and this deflection is detected by capacitor

plates on both sides. The maximum spring deflection is 0.10 mm (0.004 inch).

#### 2.2.8 Data Acquisition

The five measurements taken during a test were temperature, flow rate and three differential pressures. A Fluke data logger, Model 2240B, scanned the instruments at predetermined intervals. The measurements were displayed and the results printed on a paper tape. A Tektronix 4923 tape deck simultaneously recorded the data on a magnetic tape.

The data logger is capable of reading thermocouples directly. The remaining instruments generate 4-20 mA signals. Precision resistors were used to convert the mA signals to voltages for logging by the Fluke. The data logger was calibrated occasionally using a Rosemount Model 262 field calibrator, to establish the voltages corresponding to 4 and 20 mA.

The magnetic data tapes are compatible with a Tektronix computer which is used for data analysis and plotting.

#### 2.2.9 Loading System and Valving

Figure 12 illustrates the valves and other equipment required to introduce and remove material from the pipeline loop. All valves are 1 inch Jenkins compact body ball valves.

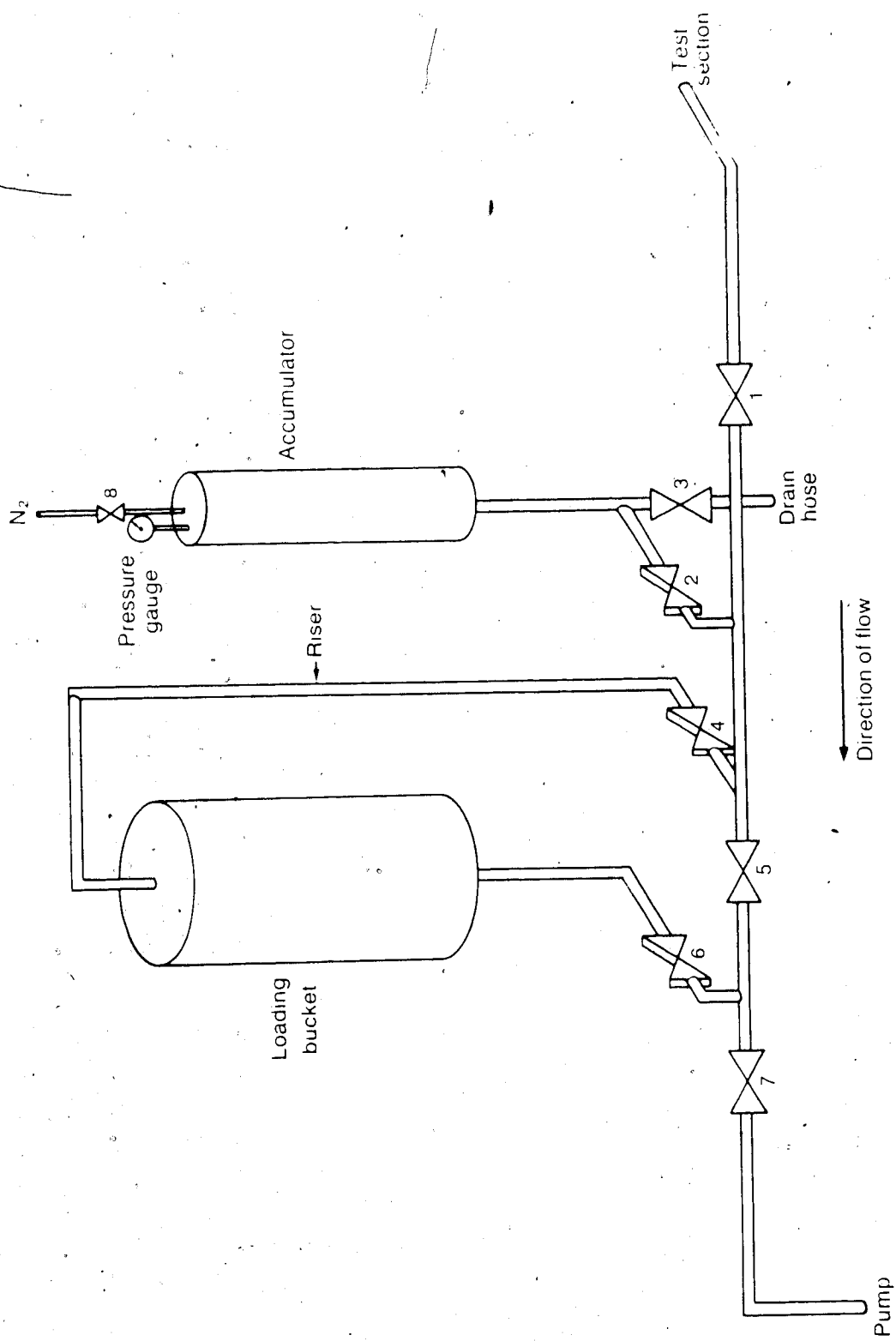


Figure 12: Valve configuration of the emulsions flow circuit.

To introduce a fluid (either water or oil) into the empty system, valves 1,4,6 and 7 are opened and valves 2 and 5 are closed. The fluid is poured into the loading bucket and the pump started. When the level of the fluid in the loading bucket drops, more fluid is added until recirculating fluid enters the bucket via the riser. The fluid is then allowed to recirculate until all the air in the circuit has been removed.

The test emulsions were not introduced into the system this way. Rather, the system was first filled with the aqueous solution used to make the emulsion and the solution was then displaced by the test emulsion. In this manner air in the system could be removed before the emulsion was introduced. The sequence of events to displace the aqueous solution in the pipeline with the emulsion is as follows.

The circuit and loading bucket both contain the aqueous solution to be displaced. Valves 1,2,3,6 and 7 are opened with valves 4 and 5 closed. Flow is started and the aqueous solution in the bucket enters the system forcing the solution from the system through the drain hose attached to valve 3. When the level of solution in the loading bucket drops to the top of the bulkhead fitting at the bottom of the loading bucket, flow is stopped. Valves 2 and 6 are closed. Any solution remaining in the loading bucket is removed manually.

The emulsion is now introduced into the loading bucket. Valves 2 and 6 are reopened and the flow is started. The loading bucket volume is approximately equal to the volume of the rest of the system. When the

level of the emulsion in the loading bucket begins to get low, more emulsion is added. This is done either while the emulsion is still flowing, or by interrupting the flow in the manner described above.

By the time about one and a half loading buckets of emulsion have been added to the system, all of the aqueous solution has left the system. At this point valves 2 and 3 are closed and valves 4 and 5 are opened. This stops the flow of material out of the system and starts circulation of the material through the system and through the bucket.

Usually two loading buckets full of the emulsion were used to displace the first fluid and fill the system. A third bucket of the emulsion was used to provide enough fluid to the system for sampling during a run. Initially, to insure proper mixing of the emulsion in the system, valve 5 was closed forcing all of the material to circulate through the loading bucket. Later, valve 5 was opened and valves 4 and 6 were closed to reduce the amount of emulsion that needs to be cooled to operating temperature.

After the emulsion has cooled but before a test starts, valves 2 and 6 are opened with valve 8 vented to atmosphere. Emulsion from the bucket enters the system and the emulsion level in the accumulator rises. This process is aided at low flow rates by throttling valve 5. When enough emulsion has been added to the accumulator, valve 6 is closed as is valve 8. Nitrogen is added to the accumulator through valve 8 to control the pressure of the system at this point and thus increase the pressure of the whole system slightly.

### 2.3 Pipeline Test Loop : Calibration

Before starting flow tests in the pipeline loop with the emulsions, the test section was characterised with respect to inner diameter, length and relative roughness of the test section. Both values were determined by conducting flow tests with Newtonian oils.

In the laminar flow regime, pressure gradient is only a function of the inner diameter and is not a function of the relative roughness. The friction factor,  $f = \Delta P / (2Lv^2\rho)$ , is related to the Reynolds number,  $Re = Dv\rho/\mu$ , by the relationship  $f = 16/Re$ . Flow tests can therefore be used to determine the value for inner diameter by:

- 1) using a Newtonian oil
  - 2) determining the density and viscosity behaviour of the test fluid as functions of temperature
  - 3) measuring the pressure drop in the test section as a function of flow rate and temperature
- and 4) plotting the test results as friction factor against  $1/Re$  for different values of inner diameter until the best fit of  $f = 16/Re$  is found.

The oil used for the laminar flow experiments was a mixture of two straight mineral oils produced by Esso Petroleum Canada, Faxam 22 and Faxam 68. The mixture was allowed to circulate for several days in the pipeline before the flow tests were conducted. The physical properties of the oil were determined immediately following the flow test on a



sample drawn from the test loop. The viscosity of the oil was determined as a function of temperature using a Haake Rotovisco viscometer. The density of the oil was determined as a function of temperature using a gravity bottle. At 25°C this oil had a viscosity of 57 mPa s and a density of 868 kg/m<sup>3</sup>.

Figure 13 shows the variation of friction factor with  $1/Re$  for the laminar flow data. A value of inner diameter of 2.12 cm (0.836 inches) results in  $f Re = 15.5$ , which is within 3% of the theoretical value. The book value for inner diameter of this type of pipe is 2.09 cm (0.824 inches), which compares well with the determined value.

In the turbulent flow regime, friction factor is a function of both the inner diameter and the pipe relative roughness. With the inner diameter determined by laminar flow experiments, turbulent flow experiments can be used to determine the relative roughness of the test section by

- 1) using a Newtonian fluid
  - 2) determining the density and viscosity behaviour of the test fluid as functions of temperature
  - 3) measuring the pressure drop of the test fluid in the test section as a function of flow rate
- and 4) plotting the results as friction factor against Reynolds number on a Moody plot.

The Moody plot, illustrated in Figure 14, correlates friction factor as a function of Reynolds number and relative roughness. It is based on the data of many investigators as correlated by Moody [30]. The lines

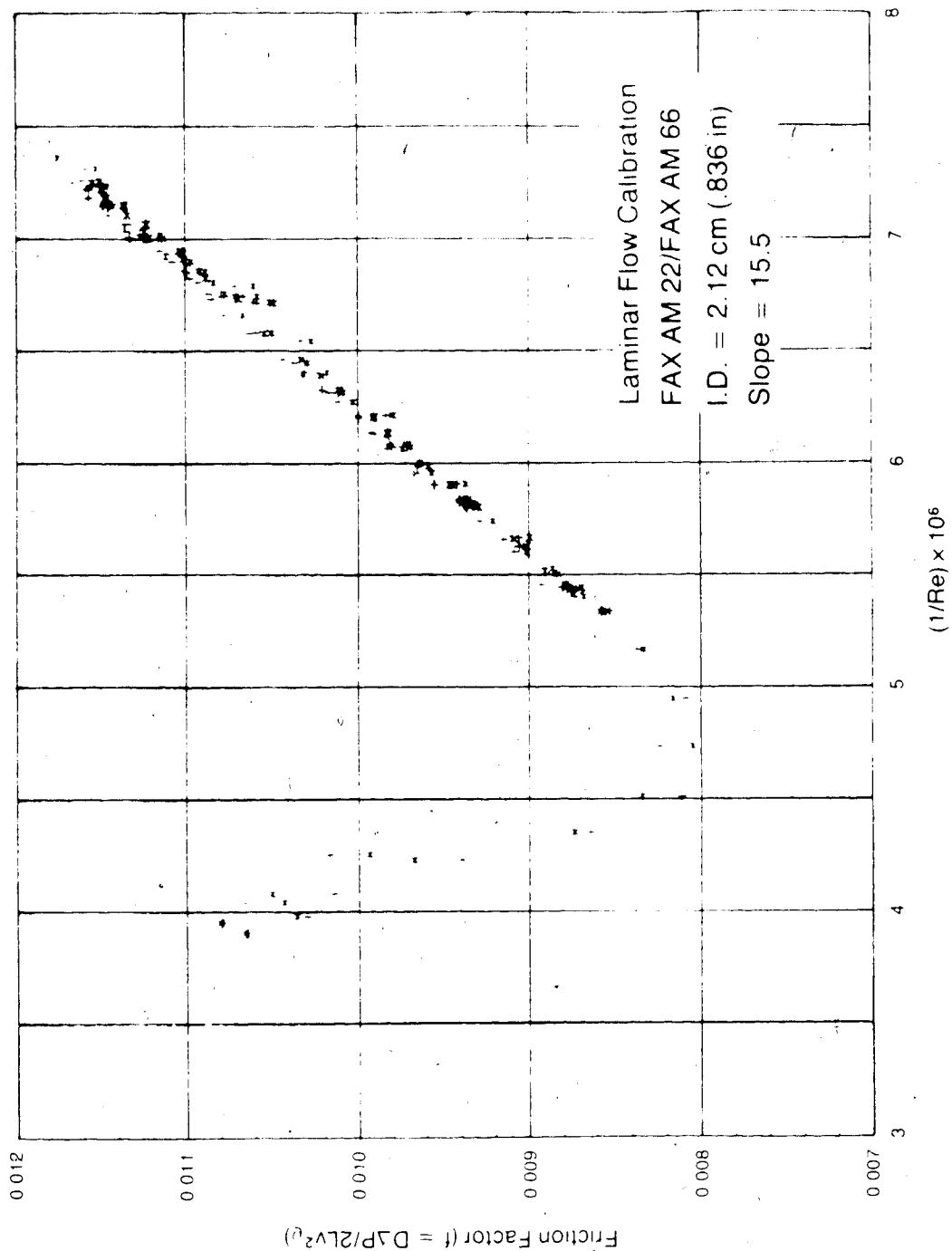
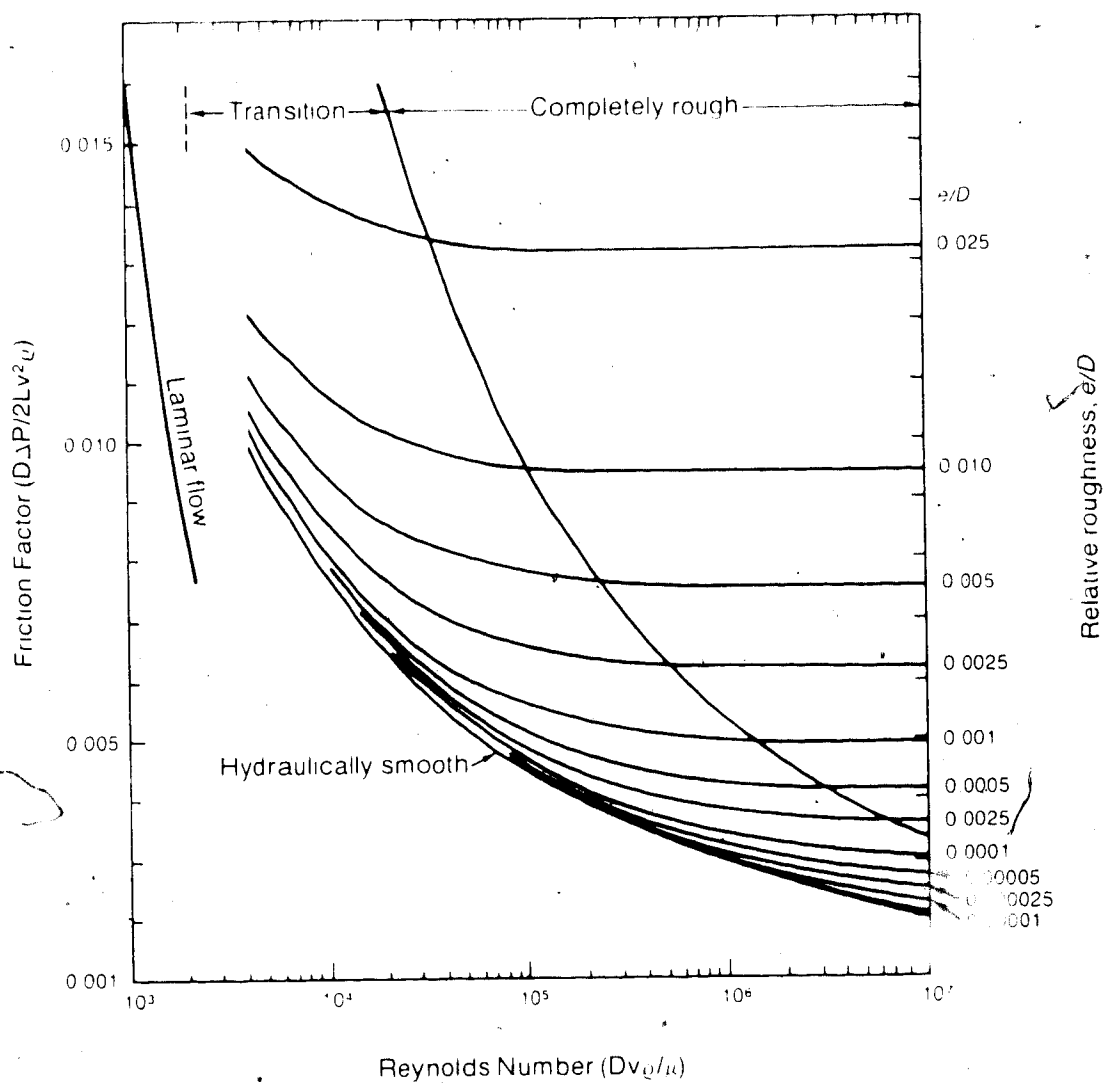


Figure 13: The variation of friction factor with 1/Reynolds number.



**Figure 14:** Friction factor as a function of Reynolds number and relative roughness for commercial pipe (from [31]).

on the diagram are well represented by the equation due to Churchill [32].

$$f/2 = ((8/Re)^{12} + 1/(A+B)^{1.5})^{1/12}$$

where

$$A = (2.457 \ln(1/((7/Re)^{0.9} + 0.27e/D)))^{16}$$

$$B = (37530/Re)^{16}$$

After plotting the data as friction factor against Reynolds number, lines for various values of relative roughness may be generated using Churchill's equation until the best fit of the data is found. This value is taken to be the relative roughness,  $e/D$ , of the pipe in the test section.

The oil used for turbulent flow calibration was Voltesso 35, a mineral oil containing a 0.05% of an antioxidant, produced by Esso Petroleum Canada. The oil was circulated for several days before the calibration tests were done. The physical properties of this oil were determined on samples taken from the material circulating in the pipeline upon completion of a flow test. The same methods were used to determine the physical properties of this oil as in the laminar flow experiments. The viscosity of the oil was 14 mPa s at 25°C and the density was 858 kg/m<sup>3</sup> at 25°C.

The data are plotted in Figure 15 as friction factor versus Reynolds number using the value for the inner diameter found in the laminar flow tests. The relative roughness is established by drawing in the lines

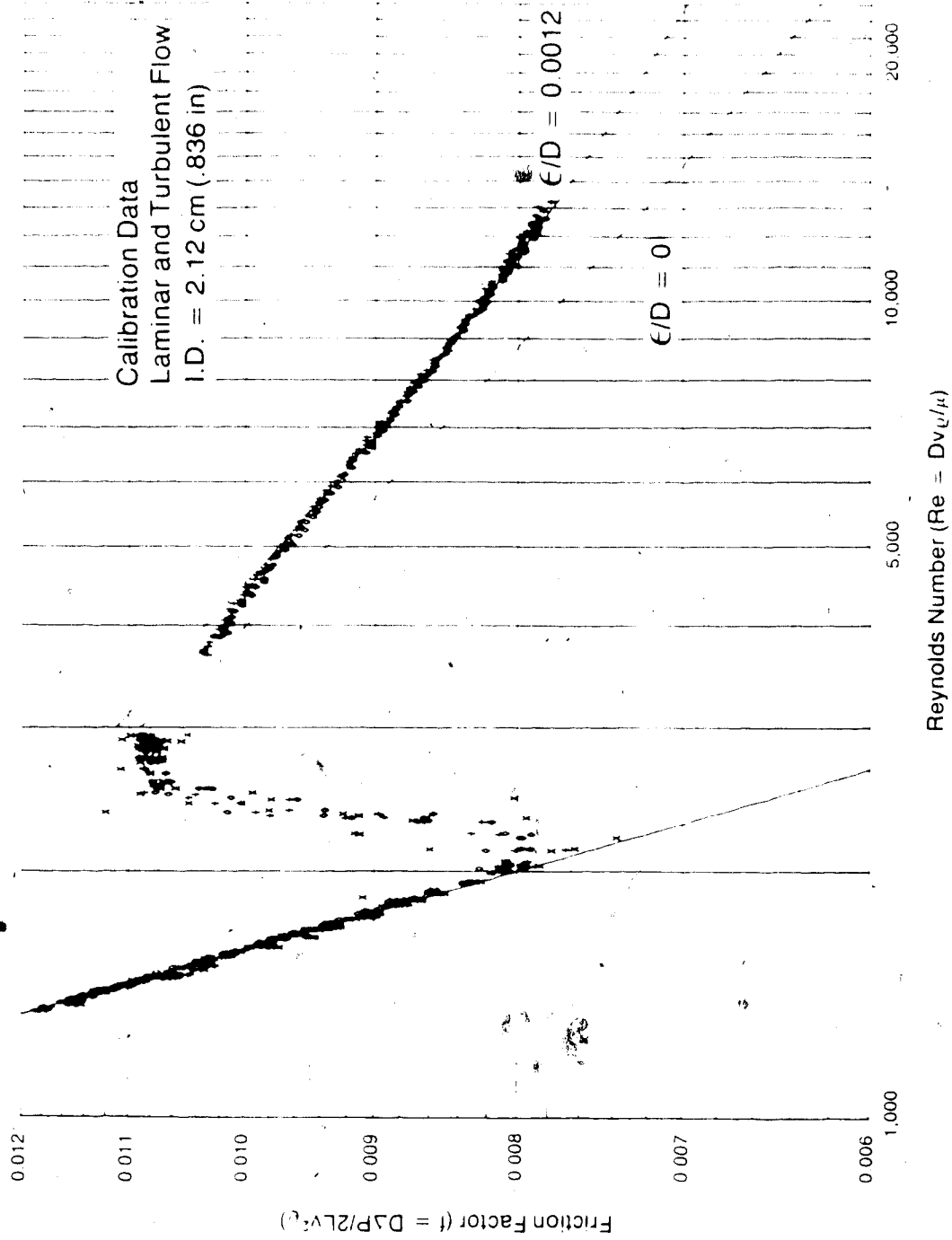


Figure 15: Summary of friction factor - Reynolds number data collected with Newtonian oils.

found on a Moody plot and determining which value of relative roughness best fits the data. Figure 15 summarizes the laminar and turbulent flow data. The best fit of the turbulent flow data corresponds to  $e/D=0.0012$ . This value of relative roughness lies between those reported for 3/4 inch drawn tubing (0.00008) and 3/4 inch commercial steel pipe (0.0024) [31] and is therefore considered reasonable for the seamless pipe used in these experiments.

The ultimate objective of this study is to compare the behaviour of an emulsion to that of a Newtonian oil. Therefore the calibration line for the turbulent flow data is necessary to allow comparison between the emulsion data and the Newtonian oil data in regions where the Reynolds numbers do not overlap.

## 2.4 Physical Property Determinations

The physical properties required for the emulsions are

- 1) density
- 2) viscosity
- and 3) oil/water ratios.

### 2.4.1 Density

The emulsion density versus temperature behaviour was determined using a Paar Digital Density Meter model DM-55. This device consists of a U-shaped sampling tube which is rigidly supported at its open ends. The tube is vibrated at its natural frequency and the period of oscillation

T is measured. The density  $\rho$  is determined by

$$\rho = (T^2 - B) / A$$

where A and B are instrument constants determined by calibration with two materials of known density, usually water and air.

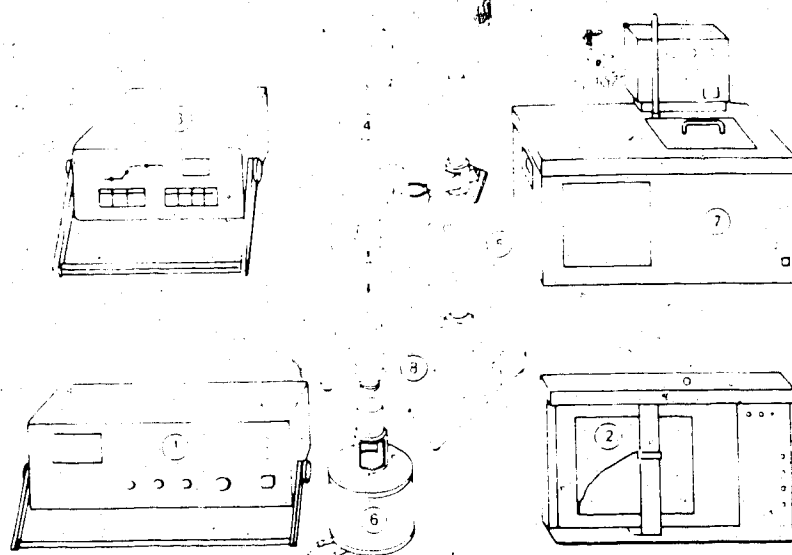
The procedure used to do the measurements is outlined in the ASTM test procedure D 4052-81.

#### 2.4.2 Viscosity

The rheological properties of the emulsions were measured using a Haake Rotovisco RV-12 equipped with an M-500 measuring head and NV sensor system. This is a Couette type viscometer with a rotating bob and stationary cup.

Values of absolute viscosity were determined with this instrument and flow curves of shear stress versus shear rate were generated. The temperature of the sample was controlled by an external water jacket. Viscosity measurements were made at 25°C during the emulsion screening program and at pipeline temperature during the pipeline test program. Figure 16 illustrates the components of the viscometer system.

Before a sample was taken, the water jacket was allowed to equilibrate to the desired temperature. The sensor system was removed from the viscometer assembly and sample was introduced into the cup. A clean disposable plastic eyedropper was used to transfer some of the emulsion from the sample jar to the cup. The tip of the eyedropper was cut off



- 1- Basic instrument ROTOVISCO RV 12
- 2- Recorder xy/t
- 3- Speed programmer PG 142
- 4- M500 measuring-drive-unit
- 5- Stand
- 6- Temperature vessel
- 7- Thermal liquid constant temperature circulator
- 8- Sensor system

Figure 16: Components of the viscosity measurement system.



for those samples where larger bitumen droplets clog the regular opening. The bob was then placed in the cup and the viscometer was reassembled.

Readings are taken as soon as possible after the sample has been introduced and the temperature has stabilized. Initially it was only possible to measure shear stress at four shear rates. A straight line was then drawn through these points and the viscosity was determined from the slope of the line. Later, a PG142 programmer was added to the system which allowed flow curves to be generated. These were straight lines as well.

#### 2.4.3 Oil / Water Ratios

The oil used in the emulsion pipelining experiments contained about 8% water. Thus the actual oil concentration of each emulsion was determined by analysis. Samples taken during a long term pumping test were also analysed for oil/water content to observe any changes from the initial composition.

All emulsion samples were analysed by the Oil Sands Analytical Laboratory of the Alberta Research Council. The procedure was a modified Dean Stark analysis, a copy of which is given in Appendix B.

## 2.5 Emulsion Screening

The objectives of the emulsion screening program were to find an emulsion suitable for the pipeline experiments and establish a method for making larger batches of emulsion. The emulsion formulation followed that described by Kessick and St. Denis, U.S. Patent 4343323 [14], and was therefore not studied in great detail.

The criteria used for selecting an oil were:

- 1) good emulsion formation in a 0.1% by weight NaOH solution
- 2) stability of the emulsion on standing
- 3) stability of the emulsions under shear in the viscometer
- 4) reproducibility of the emulsions as indicated by viscosity measurements
- and 5) availability of the oil.

The variables studied in the screening program were:

- 1) oil type
- 2) oil concentration
- 3) aqueous phase formulation
- and 4) emulsification equipment.

Two oils from the heavy oil fields of eastern Alberta were the possible candidates. The first was a Cold Lake crude recovered by steam stimulation and field treated. This oil was available through the Alberta Research Council Oil Sands Sample Bank, reference number 82-01. Table 4 lists the physical properties of this crude oil as reported by

Table 4: Reported physical properties of Cold Lake crude oil, sample number 82-01

## Oil Sands Physical Properties Data Base

**Sample Identification**

SAMPLE I.D. NO: 82-01  
 COUNTRY: Canada  
 STRATIGRAPHIC UNIT: Clearwater  
 LOCATION: LSD SEC. 4 TWP. 65 RGE. 3 W 4M  
 METHOD OF PRODUCTION: Steam-Stimulation  
 SOURCE OF SAMPLE: Esso Resources  
 OTHER SAMPLE DESCRIPTION: 82-01; sample had 8.62% water which was extracted

CLASSIFICATION: Bitumen  
 FIELD (POOL): Cold Lake

**Hydrocarbon Characteristics**

GRAVITY 9.999  
 RELATIVE DENSITY 15/15 deg.C 0.9994  
 VISCOSITY (Cps.) 15 deg.C 410,233  
 25 deg.C 86,943  
 60 deg.C 2,751  
 100 deg.C 220  
 POUR POINT deg.C 225  
 MOLECULAR WEIGHT 552  
 CARBON RESIDUE R wt.% 12.0  
 CARBON RESIDUE C deg.C 300  
 ASH wt.% 0.05  
 CARBON wt.% 83.91  
 HYDROGEN wt.% 10.46  
 NITROGEN wt.% 0.60  
 SULPHUR wt.% 4.50  
 OXYGEN wt.% 1.26  
 VANADIUM ppm wt 155  
 NICKEL ppm wt 67  
 SATURATES wt.% 17.34  
 AROMATICS wt.% 12.49  
 POLAR I wt.% 21.45  
 POLAR II wt.% 13.18  
 POLAR III wt.% 17.94  
 ASPHALTENES wt.% 16.61  
 ACID NUMBER 0.88  
 HEAT OF COMBUSTION J/g 41,041

**Simulated Distillation**

CUT TEMP. Vol.% Sum  
 0 deg.C VOL.%  
 IBP 0.5  
 150  
 175  
 195  
 225 1.75 2.25  
 250 1.56 3.81  
 275 1.85 5.66  
 300 3.48 9.14  
 325 3.61 12.75  
 343 2.60 15.35  
 375 4.74 20.09  
 400 4.05 24.14  
 425 4.00 28.14  
 450 4.01 32.15  
 475 4.00 36.15  
 500 4.01 40.16  
 524 3.72 43.88  
 RESIDUE 56.12 100.00

the Oil Sands Sample Bank. The second was a dewatered bitumen supplied by Esso Resources Canada Ltd. This material was recovered from produced oilfield emulsions by removing excess water by distillation.

The two concentrations investigated were 40 and 55% oil by weight. This gave a wide range of emulsion viscosity and stability behaviour.

The choice of aqueous solutions was between:

- 1) distilled water containing 0.1% by weight NaOH
- and 2) distilled water containing 0.1% by weight NaOH and 0.02% by weight sodium hydrosulphide ( $\text{Na}_2\text{S}_2\text{O}_4$ ), as an oxygen scavenger.

The two types of equipment available for emulsification were:

- 1) a paint stirrer
- and 2) a Polytron PT35 homogenizer.

The test program consisted of making about 200 g of each emulsion and investigating the viscosities of the fresh and aged emulsions over the period of several days. The change in viscosity of a sample on shearing at a constant shear rate in the viscometer was observed.

A summary of all the tests is given in Appendix C. The trends that can be observed are

- 1) The viscosity of the emulsions increases with the age of the sample.
- 2) The viscosity of a given sample increases with shearing time in the viscometer.

- 3) The dewatered bitumen emulsions are too unstable to be used in the pipeline experiments.
- 4) The oxygen scavenger does not appear to improve the stability of the emulsions.
- and 5) The best emulsions are made with the Cold Lake crude using the Polytron homogenizer.

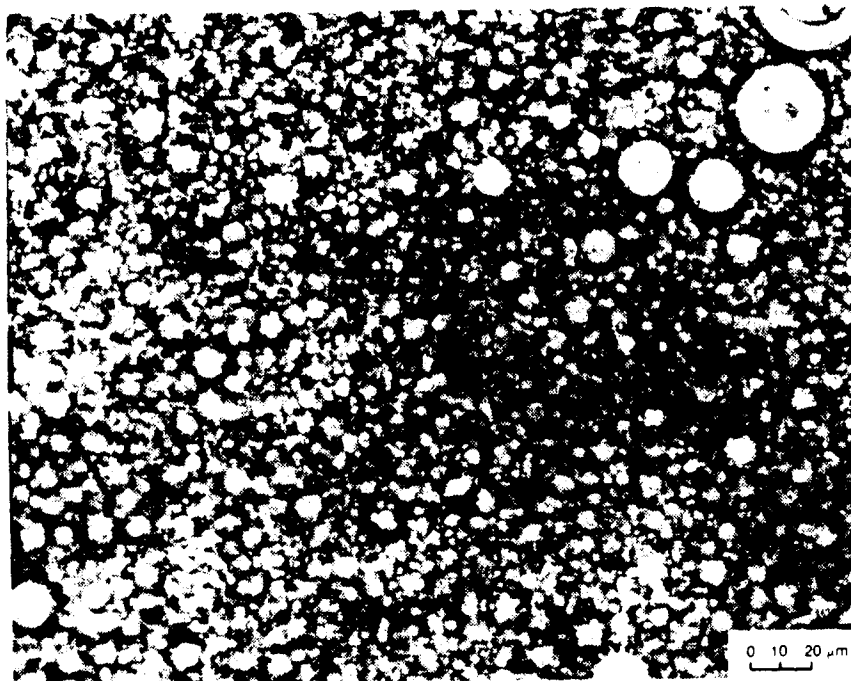
Figure 17 is a typical photograph of a Cold Lake crude emulsion enlarged 400X. The emulsion was made with the Polytron homogenizer and a 0.1% by weight NaOH solution. From the given scale, most of the droplets are in the range from 4 to 6 microns.

Based on the results from the emulsion screening program, the oil chosen for the emulsion pipelining tests was the Cold Lake crude.

## 2.6 Emulsion Preparation

Emulsions for use in the emulsion pipelining experiments were prepared in the following manner.

Distilled water was heated to 90°C to remove any dissolved gases. An aqueous solution was made with the hot distilled water, 0.1% by weight sodium hydroxide (NaOH) and 0.02% by weight sodium hydrosulphide ( $\text{Na}_2\text{S}_2\text{O}_4$ ). Approximately 6 kg of room temperature Cold Lake crude oil was poured into a tared, 20 l stainless steel bucket and weighed. The correct amount of aqueous solution at 80°C was then added to the oil to produce the desired nominal oil concentration in the emulsion. A



**Figure 17:** Photograph of Cold Lake crude oil-in-water emulsion through microscope enlarged 400x.

spatula was used to contact the oil and water phases, which formed an emulsion with gentle stirring.

The emulsion was then homogenized using a Polytron PT45 homogenizer with a PTA45/6 generator for 10 minutes at power level 5 and for 1 minute at power level 5.5. The homogenizer relies on mechanical shear to disperse the oil in the water with no ultrasonic component. No attempts were made to investigate the effects of power level or homogenization time on the physical or flow properties of the emulsion.

Three 20 l pails of emulsion were made for each test. The emulsion was introduced into the test loop as soon as possible after it was made.

## CHAPTER 3 : EXPERIMENTAL RESULTS

### 3.1 Objectives of the Emulsion Pipelining Experiments

The experimental program had two objectives. The first was to develop generalized correlations of friction factor versus Reynolds number that could be applied to pipeline design. The second objective was to investigate the effects of oil concentration and shear rate on the distance an emulsion can be pumped before breaking down.

### 3.2 Overview of the Pipelining Experiments

Two types of tests were performed with a given batch of emulsion. The first test was a short test usually taking about half an hour. Here the fresh emulsion was introduced into the pipeline and allowed to circulate until its temperature reached the desired test temperature. This was usually about 10° to 14°C. Starting at the lowest possible value, the flow rate was increased slowly in steps to the maximum value and then decreased in the same manner to the low value. At each flow rate the emulsion temperature and the pressure drops in the test section were recorded. This pattern was then repeated. At the end of the test a sample of the emulsion was taken to determine the density and viscosity versus temperature behaviour. The data were then used for the correlation of friction factor with Reynolds number.

The second type of test involved pumping the emulsion in the closed loop pipeline at constant flow rate and temperature. Measurements of the



pressure drops, flow rate and temperature were made every half hour for the duration of the test. Samples were taken during the test to monitor changes in the emulsion viscosity and oil concentration. The test was continued until a breakdown in the emulsion was observed.

The experimental program involved two levels of nominal oil concentration. For each level of oil concentration tests were made at five different flow rates.

The oil concentration levels were nominally 45% and 55% by weight, but due to the initial water present in the oil used, the actual values varied somewhat. The overall flow rate range covered in all tests was from 4 to 200 kg/min. For any given test the flow rate could only be varied by a factor of three. Different pulley and drive motor arrangements were required to achieve the desired flow rates.

Table 5 summarizes the conditions present for the tests that were successfully completed. The following information is given for each test.

- A) The test name which indicates the oil type, CLC = Cold Lake crude, the nominal oil concentration and the emulsion batch.
- B) The range of flow rates (kg/min) covered in the determination of the variation of friction factor with Reynolds number.
- C) The range of Reynolds numbers corresponding to the above flow rates
- D) The flow rate (kg/min) chosen for the long term pumping test.
- E) The corresponding value of shear rate,  $8*v/D$ , ( $s^{-1}$ ).

Table 5: Summary of the friction factor, Reynolds number and long term flow tests successfully completed

		Flow Rate Level				
		1	2	3	4	5
Nominal 45% Emulsions	Test name	CLC-45-10	CLC-45-08	CLC-45-09	CLC-45-05	CLC-45-11
	Flow rate range for friction factor - Reynolds No. tests (kg/min)	5.3 - 12.6	7.4 - 27	13 - 41	88 - 160	100 - 190
	Reynolds No. range for friction factor - Reynolds No. tests	500-1200	740-2700	1500-4700	8200-15200	10800-22000
	Flow rate for stability test (kg/min)	7.4	14	40	125	190
	Shear rate during stability test (s <sup>-1</sup> )	130	250	720	2300	3400
	Initial oil concentration (wt%)	42	43	39	45	42
Nominal 55% Emulsions	Test name	CLC-55-13	CLC-55-10	CLC-55-11	CLC-55-09	CLC-55-12
	Flow rate range for friction factor - Reynolds No. tests (kg/min)	4.2 - 9.6	8.0 - 24	13 - 41	92 - 192	125 - 200
	Reynolds No. range for friction factor - Reynolds No. tests	190-450	500-1200	650-2100	4000-8300	11000-17700
	Flow rate for stability test (kg/min)	7.4	14	28	125	190
	Shear rate during stability test (s <sup>-1</sup> )	130	250	500	2300	3400
	Initial oil concentration (wt%)	49	50	49	57	48

F) The concentration of oil in the emulsion (wt% oil). This is the value determined by analysis of a sample taken immediately after the short test.

Appendix D contains a complete description of each test and the data that were generated.

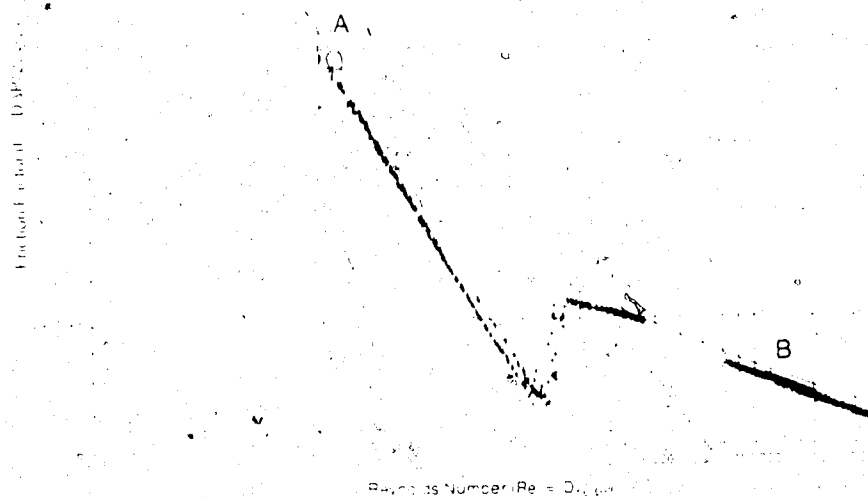
Tests done but not recorded in Table 5 are as follows. Test CLC-45-03 and test CLC-45-04 were the first attempts at working with emulsions in the pipeline. They served to establish the repeatability between flow tests from day to day and between emulsion batches. The best way to run the long term tests was determined and the method for cleaning the system between tests was established.

Long term tests CLC-45-06 and CLC-45-07 were not completed successfully. Test CLC-45-06 was stopped when the pump could no longer be turned at constant speed due to insufficient motor horsepower. Test CLC-45-07 was not completed due to a gearbox failure. However both tests are useful in establishing repeatability between identical long term tests when they are compared to the successfully completed test CLC-45-08.

### 3.3 Variation of Pressure Drop with Flow Rate

The data collected on the variation of pressure drop with flow rate are reduced to the dimensionless variables friction factor,  $f = \Delta P / 2L v^2 \rho$ , and Reynolds number,  $Re = D v \rho / \mu$ . The variation of the friction factor,  $f$ , with the Reynolds number,  $Re$ , is illustrated in Figure 18 for the nominally 45% oil emulsions and in Figure 19 for the nominally 55% oil

## Summary of 45% Emulsions

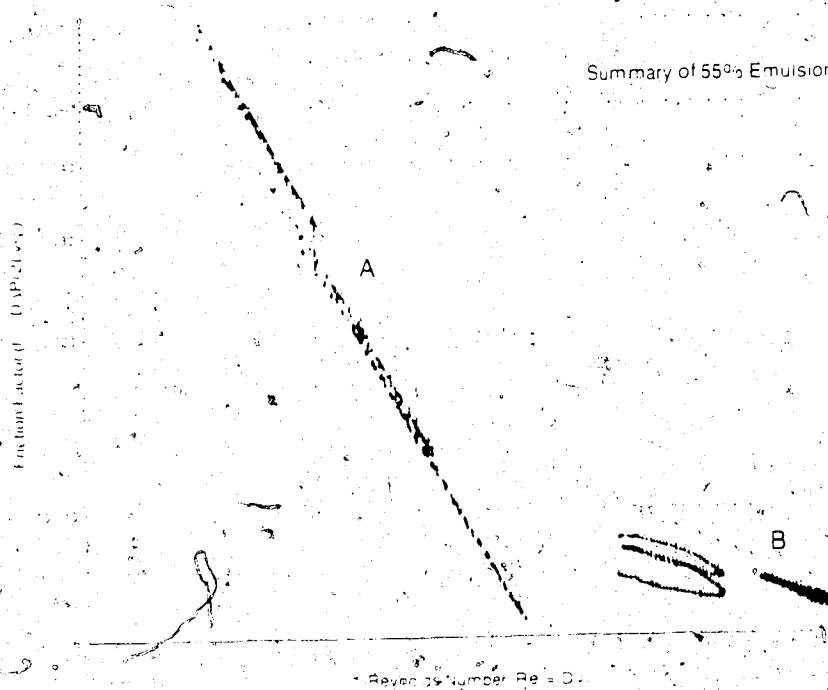


**Figure 18:** The variation of friction factor with Reynolds number for the nominally 45% emulsions.

A:  $f = 16/Re$

B: Calibration line for Newtonian Oil

## Summary of 55% Emulsions



**Figure 19:** The variation of friction factor with Reynolds number for the nominally 55% emulsions.

emulsions. The value of friction factor plotted is the average of the three values calculated for the different lengths of the pipe.

The range of flow rates covered over the course of all of the tests is 4 to 200 kg/min. This corresponds to a Reynolds number range of 500 to 22000 for the nominally 45% oil emulsions and a Reynolds number range of 190 to 17700 for the nominally 55% oil emulsions. As well, these flow rates correspond to a range in bulk velocity of 0.3 to 9 m/s. The measured pressure drops range from 0.3 to 140 kPa.

It was only possible to increase the flow rate by a factor of about three over the lowest flow rate during any given test. The 50 fold variation in flow rate overall was achieved by a careful choice of drive motor, gear reduction and pulleys to turn the pump at appropriate speeds for each of the tests. The friction factor - Reynolds number data in Figures 18 and 19 are the test results from five different emulsion batches at each nominal concentration level.

Figures 18 and 19 show that the emulsion data lie below the calibration line for the Newtonian oil. The 45% emulsion data lie within 10% of the calibration data. The 55% emulsion data lie somewhat lower.

The transition from the laminar to turbulent flow regimes for the 45% emulsion occurs between Reynolds numbers of 2200 and 2500, as was the case for the Newtonian oil. Flow rates for the 55% emulsions were not suitable to observe the transition in the flow regime.

The agreement between the different batches of 45% emulsion over the different ranges in Reynolds number is very good. The agreement between different 55% emulsion tests is also reasonable.

The hysteresis in the friction factor - Reynolds number behaviour for the 55% emulsion CLC-55-09 did not occur during any of the other tests. The friction factor decreased with increasing Reynolds number, returning to a higher level than initially, when the Reynolds number was decreased. This pattern was repeated when the flow rates were varied again, but not to as great an extent. This batch of emulsion, CLC-55-09, had the highest initial oil concentration at 57% by weight oil.

#### 3.4 Long Term Emulsion Pumping Tests

During a long term pumping test an emulsion was circulated under conditions of constant flow rate and temperature until significant departure in the operating conditions of the system from the steady state or pseudo steady state values were observed. These changes include large departures in differential pressures or temperature. No limit was placed on the length of time required for such changes to occur. When such changes did occur the emulsion was considered to have broken down.

The variables studied in the long term pumping tests were oil concentration and flow rate. The data recorded during a long term

pumping test were:

- 1) flow rate
- 2) temperature
- 3) differential pressure
- 4) oil concentration
- 5) emulsion viscosity

and 6) current drawn by the motor (where possible).

Flow rate, temperature and differential pressure were recorded every 30 minutes. The oil concentration was analysed from samples taken from the system about once or twice a day. These samples were also used to check the viscosity of the emulsion as an indication of changes in the structure of the emulsion. The current drawn by the D.C. motor was recorded continuously on a strip chart recorder. The differential pressure data was generalized by calculating the friction factor,  $f = D\Delta P / 2Lv^2\rho$ , to compensate for all changes in flow rate and temperature.

For each level of nominal oil concentration, long term pumping tests were conducted at five different flow rates. For the nominally 45% emulsions, the tests were conducted at 7.4, 14, 40, 125 and 190 kg/min. For the nominally 55% emulsions, the tests were conducted at 7.4, 14, 28, 125 and 190 kg/min. Tests were conducted in both the laminar and turbulent flow regimes. All tests were conducted between 10° and 14°C except for those at the highest shear rate level, where the refrigerated cooling system did not have adequate capacity and the city water was only available at about 15°C. These two tests were carried out at about

25°C.

The data collected during the long term pumping tests are presented as the variation of a given quantity with distance. The distance an emulsion remains stable is considered to be a more meaningful parameter for designing a pipeline than time. Time and velocity are the measured quantities, thus the distance travelled during a test is calculated as the product of the time average bulk velocity and time.

Figure 20(a)-(d) form a typical record of a long term turbulent flow test, test CLC-45-09. The four graphs present the variation with distance of a) friction factor, b) temperature, c) oil concentration and d) current drawn by the D.C. motor. In Figure 20(a), the friction factor remains steady for some time, then there is a sharp decrease followed by a second more stable period. At constant velocity changes in the friction factor are due to changes in the pressure drop alone. This change in the pressures and the subsequent more stable period are accompanied by an increase in the overall temperature in the system, Figure 20(b), and the current required by the D.C. motor to turn the pump at constant speed, Figure 20(d). At this time it also becomes impossible to get meaningful viscosity data, on samples taken from the system, as shear stress readings at constant shear rate in the viscometer were very erratic. The oil concentration decreases steadily throughout the course of a run, Figure 20(c). Table 6 gives the emulsion viscosity as a function of distance travelled during the test. Viscosity decreases during the test, becoming more erratic towards the end of the test. These trends were also observed in tests CLC-45-05 and



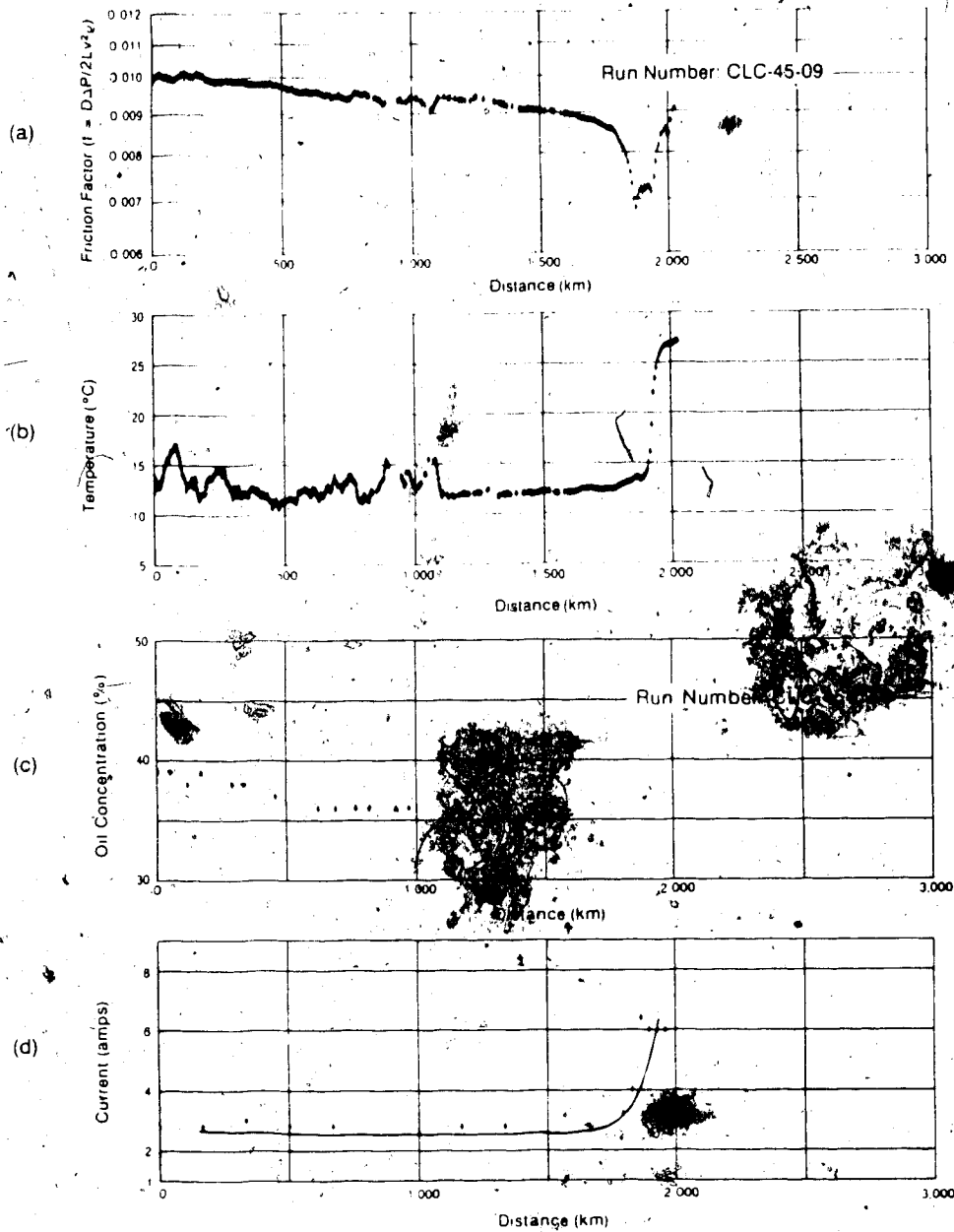


Figure 20: Record of test CLC-45-09

Table 6: The variation of emulsion viscosity with distance travelled for test CLC-45-09

Sample No.	Distance km	Oil Concentration (wt.%)	Emulsion Viscosity (cp @ °C)
2	0	39	9.1 @ 12.1
3	47	39	8.8 @ 14.1
4	117	38	8.6 @ 12.5
5	170	39	8.8 @ 10.8
6	290	38	8.2 @ 11.8
7	336	38	8.2 @ 11.6
8	453	38	8.2 @ 10.3
9	523	37	8.2 @ 10.3
10	616	36	7.2 @ 12.1
11	689	36	NA
12	763	36	7.3 @ 12.8
13	813	36	NA
14	922	36	12.1 @ 13.5
15	969	36	10.5 @ 13.7
16	1086	37	9.1 @ 13.7
17	1242	35	NA
18	1399	33	NA
19	1442	34	NA
20	1558	33	7.0 @ 11.5
21	1718	33	NA
22	1878	20	NA

NA = not available

CLC-55-09.

Figures 21(a)-(d) form a typical record of a long term laminar flow test, test CLC-55-10. In Figure 21(a), the friction factor decreases steadily throughout the course of the test. This is followed by a sharp increase in the friction factors and an accompanying increase in the system temperature, Figure 21(b), and current requirements of the D.C. motor, Figure 21(d). Again, viscosity measurements were no longer possible after the sharp change in system variables. Oil concentration decreases throughout the course of the test, Figure 21(c). Table 7 gives the viscosity of the emulsion as a function of distance travelled. Viscosity drops steadily throughout the test. These trends were also observed in tests CLC-45-10 and CLC-55-13.

Tests CLC-45-11 and CLC-55-12 were both done at very high flow rates. Figures 22(a)-(c) form the record of test CLC-45-11. Table 8 gives the viscosity of the emulsion as a function of distance travelled. The system is extremely steady for more than 10,000 km, which corresponds to more than 14 days of continuous running. The test was terminated at this point. There are essentially no changes in the values of friction factor, Figure 22(a), temperature, Figure 22(b) or oil concentration, Figure 22(c), observed. Viscosity measurements were possible throughout the course of the test and the value for viscosity remained essentially constant. It was not possible to monitor the current requirements of the A.C. motor used in this test.

Tests CLC-55-11 and CLC-45-08 did not follow the general trends

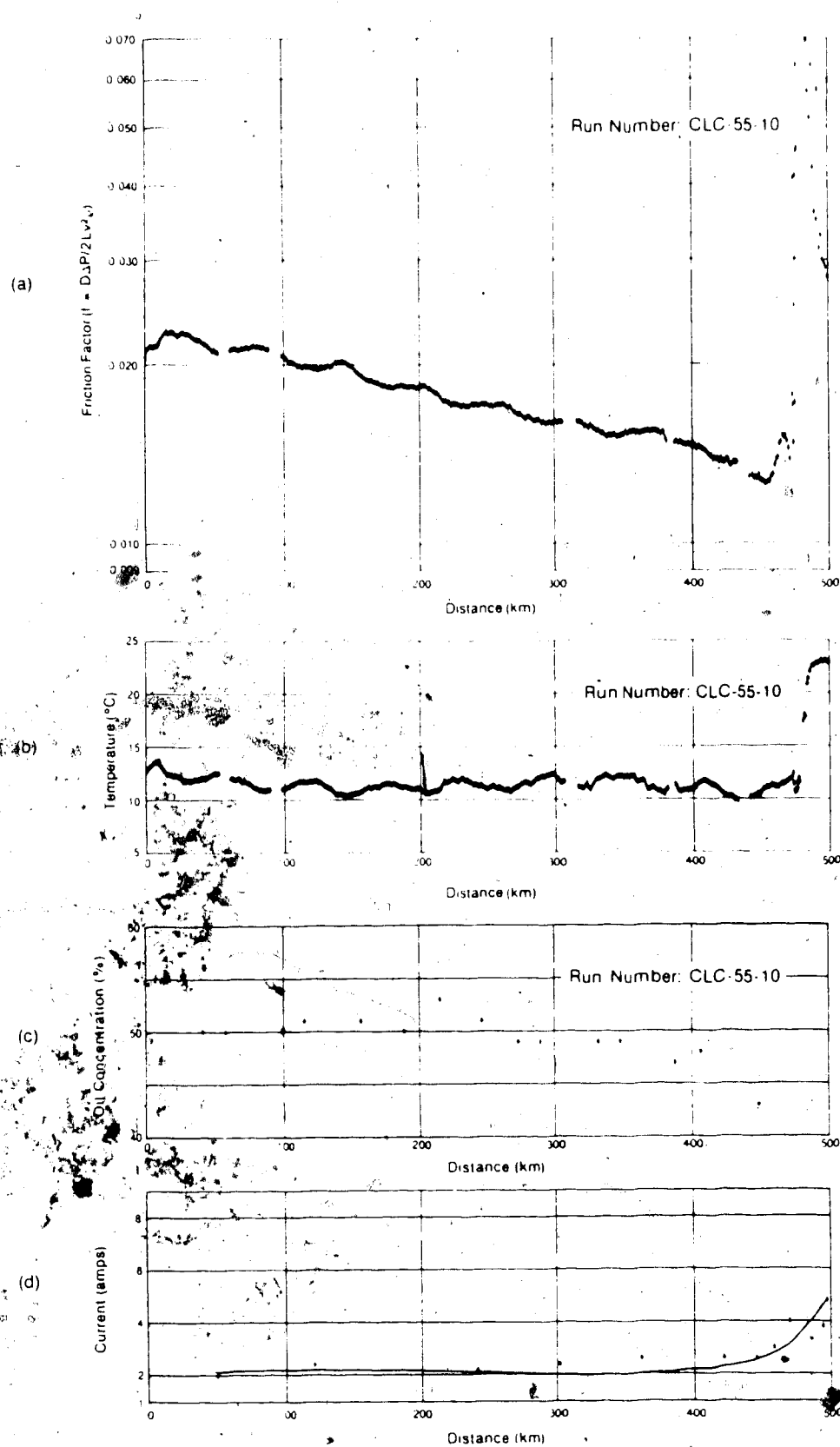


Figure 21: Record of test CLC-55-10

Table 7: The variation of emulsion viscosity with distance travelled for test CLC-55-10

Sample No.	Distance km	Oil Concentration (Wt.%)	Emulsion Viscosity (cp @ °C)
2	0	50	20.0 @ 12.7
3	41	50	22.2 @ 11.7
4	58	50	21.5 @ 11.9
5	99	50	20.4 @ 11.8
6	116	51	19.7 @ 11.9
7	157	51	19.5 @ 11.0
8	189	50	19.0 @ 11.3
9	215	53	18.5 @ 9.7
10	246	51	16.9 @ 10.8
11	273	49	16.9 @ 10.3
12	289	49	14.8 @ 11.5
13	332	49	
14	347	49	
15	388	47	14.2 @ 11.4
16	406	48	
17	449	43	
18	463		

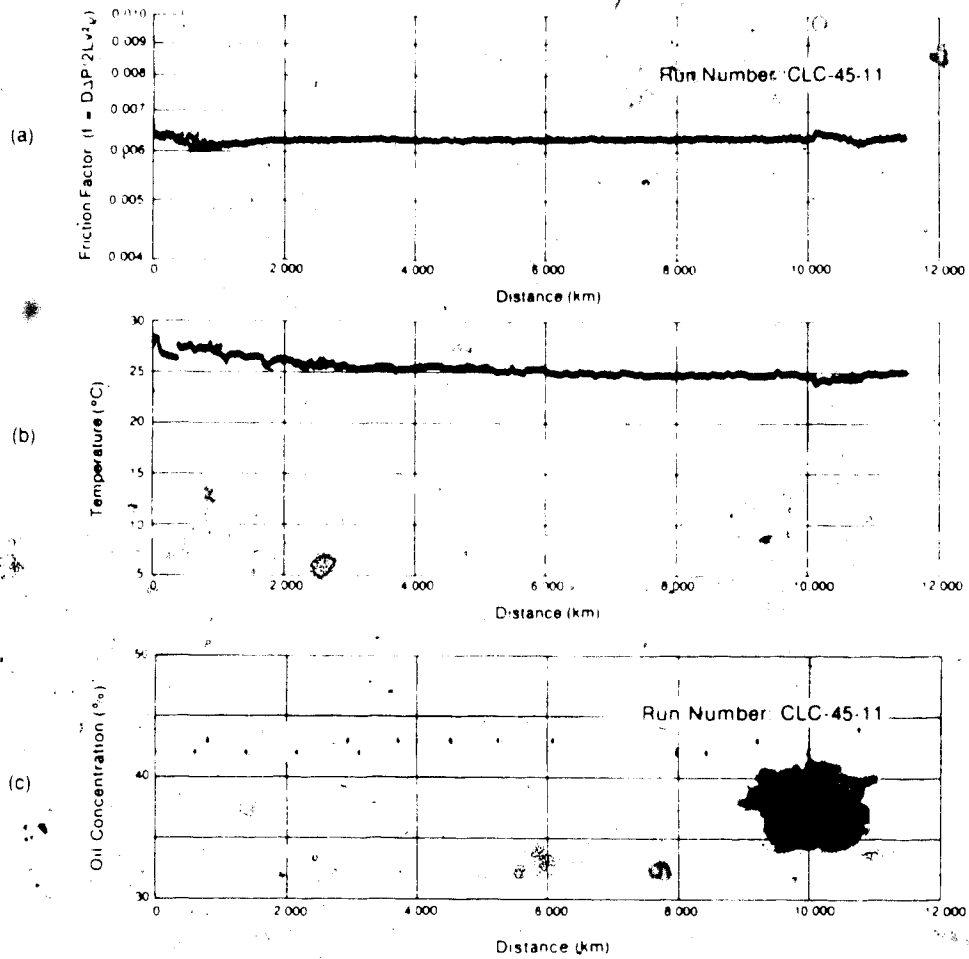


Figure 22: Record of test CLC-45-11

Table 8: The variation of emulsion viscosity with distance travelled for test CLC-45-11

Sample No.	Distance km	Oil Concentration (wt.%)	Emulsion Viscosity (cp @ °C)
2	0	42	8.3 @ 24.0
3	600	42	6.7 @ 26.8
4	794	43	7.0 @ 27.0
5	1380	42	7.0 @ 26.4
6	2160	42	7.0 @ 25.4
7	2930	43	6.9 @ 25.3
8	3110	42	7.1 @ 25.1
9	3710	43	7.0 @ 25.3
10	4504	43	7.7 @ 25.1
11	5250	43	
12	6090	43	7.5 @ 24.7
13	6930	43	7.0 @ 24.9
14	7970	42	6.7 @ 24.9
15	8420	42	7.2 @ 25.0
16	9200	43	7.2 @ 25.1
17	9980	42	7.2 @ 25.0
18	10760	44	7.6 @ 25.0

established above. Figures 23(a)-(d) form the record of test CLC-55-11. Calculation of the initial Reynolds number (1500) indicates this test should be in the laminar flow regime. As expected, the friction factor decreases for some time but then becomes extremely erratic, jumping between two levels, Figure 23(a). The temperature remains fairly steady, Figure 23(b), as does the current requirement of the motor, Figure 23(d), during this time. This was taken as an indication that the test was running in the transition zone between laminar and turbulent flow. The test was considered complete when the emulsion temperature shot up despite constant cooling water temperature and flowrate. As well the current drawn by the D.C. motor increased at this time, Figure 23(c). Table 9 gives the emulsion viscosity as a function of the distance travelled. Viscosity dropped steadily throughout the test.

Figures 24(a)-(d) form the record of test CLC-45-08. Again the initial Reynolds number (1500) indicates the test should be in laminar flow. The friction factor however, increases after the beginning of the run to remain steady for the rest of the test, Figure 24(a). The test ends in the manner typical of a turbulent flow test. This was taken as an indication that the test had gone from the laminar to turbulent flow regimes and remained in the turbulent flow regime until the test was completed. Table 10 gives the viscosity of the emulsion as a function of the distance travelled. Viscosity dropped steadily during the test.

Figure 25 compares a section of the pipe after a run with a clean piece of pipe. Deposition on the pipe walls of either oil or water-in-oil



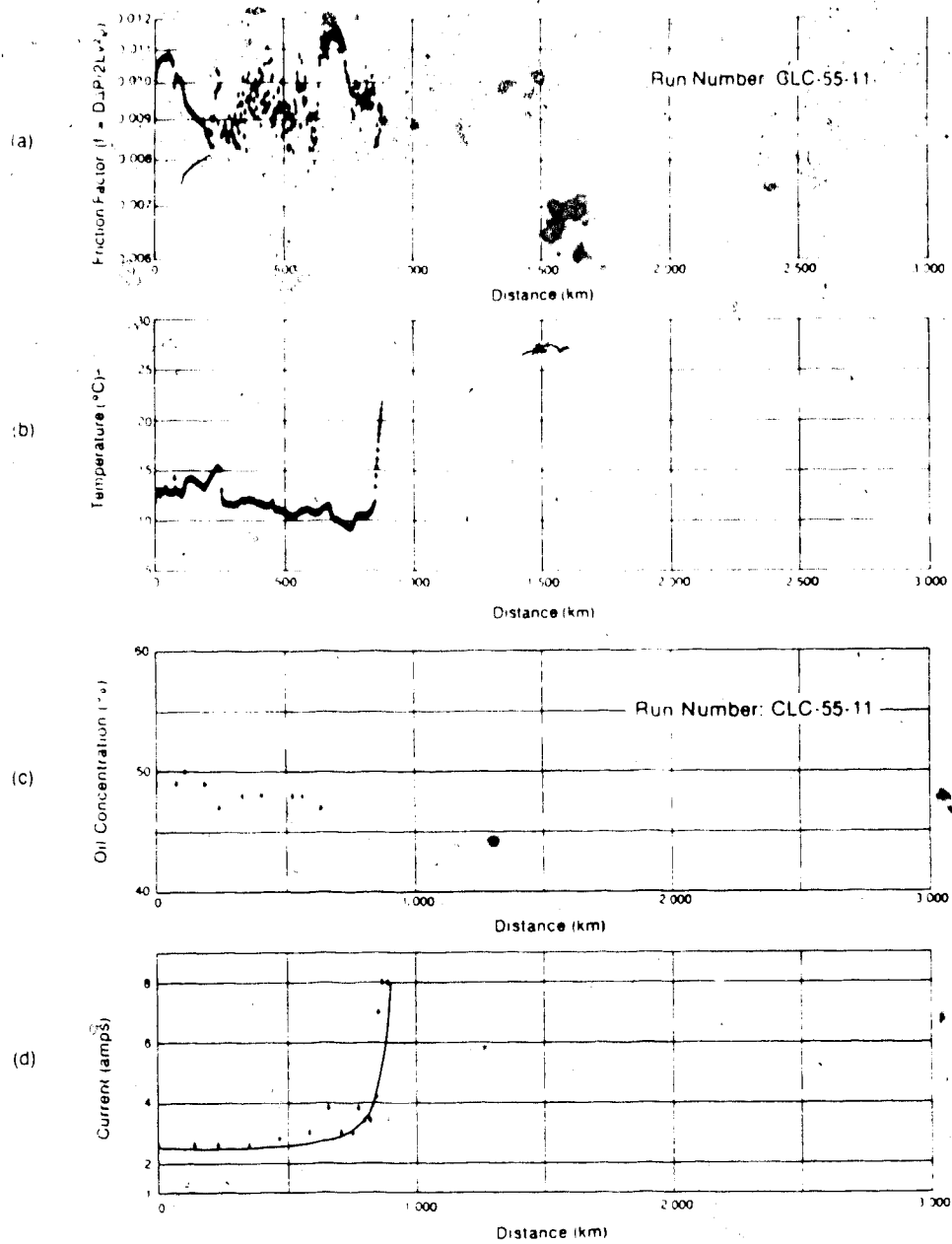


Figure 23: Record of test CLC-55-11

Table 9: The variation of emulsion viscosity with distance travelled for test CLC-55-11

Sample No.	Distance km	Oil Concentration (wt.%)	Emulsion Viscosity (cp @ °C)
2	0	49	20.9 @ 12.0
3	80	49	21.6 @ 12.9
4	112	50	21.6 @ 12.2
5	187	49	18.5 @ 12.7
6	243	47	17.6 @ 14.6
7	332	48	17.8 @ 14.6
8	407	48	17.4 @ 10.5
9		48	
10	522	49	
11	562	48	
12	636	47	
13	749	12	
14		18	

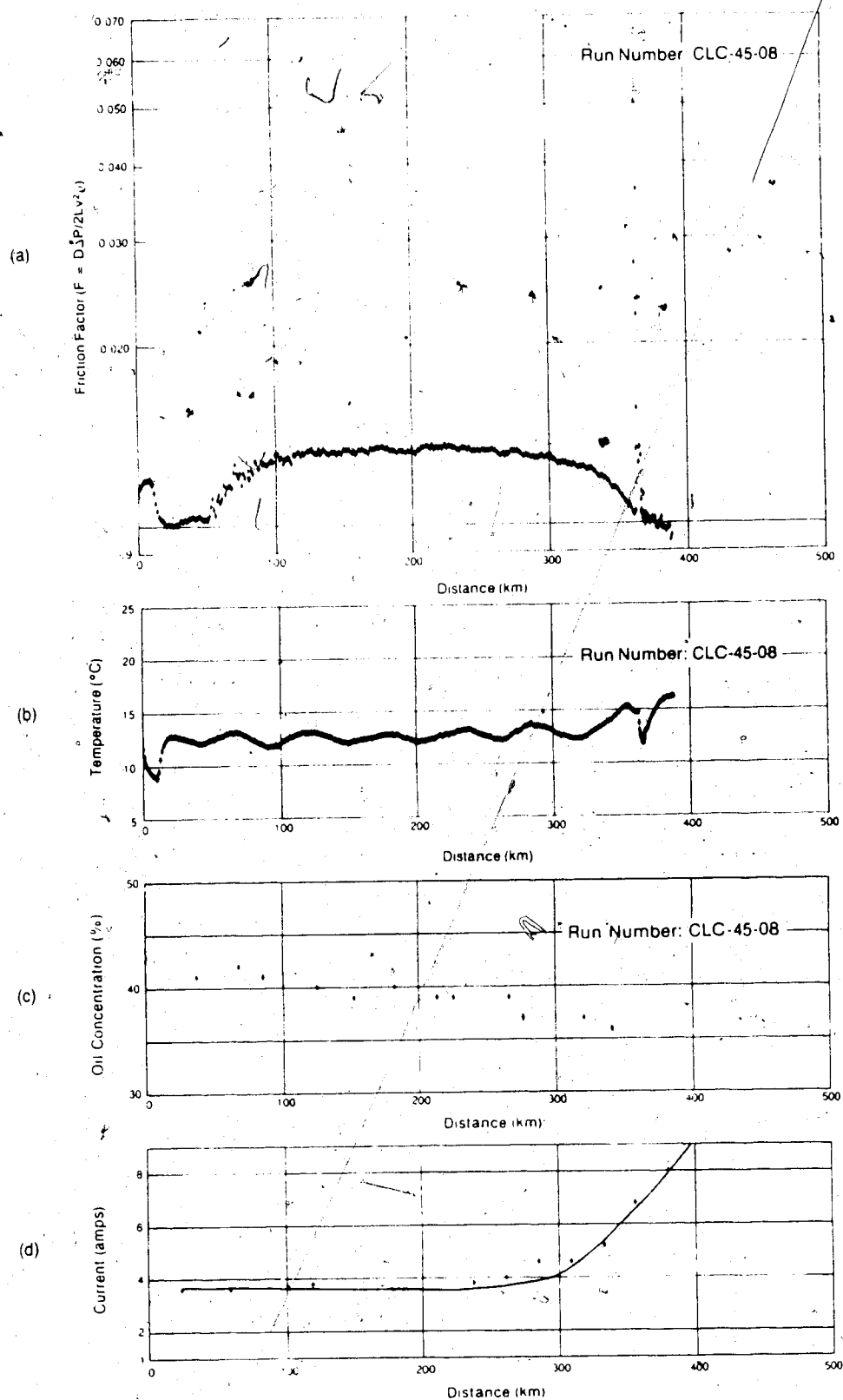


Figure 24: Record of test CLC-45-08

Table 10: The variation of emulsion viscosity with distance travelled for test CLC-45-08

Sample No.	Distance km	Oil Concentration (wt.%)	Emulsion Viscosity (cp @ °C)
2	0	43	10.4 @ 12.1
3	37	41	10.1 @ 12.0
4	68	42	10.1 @ 12.7
5	96	41	9.9 @ 11.5
6	125	40	9.2 @ 12.7
7	152	39	9.0 @ 11.6
8	182	40	9.7 @ 12.5
9	213	39	9.0 @ 11.7
10	225	39	9.4 @ 11.8
11	266	39	8.5 @ 11.8
12	283	37	7.5 @ 12.6
13	321	37	7.4 @ 12.6
14	341	36	7.5 @ 14.3
15			

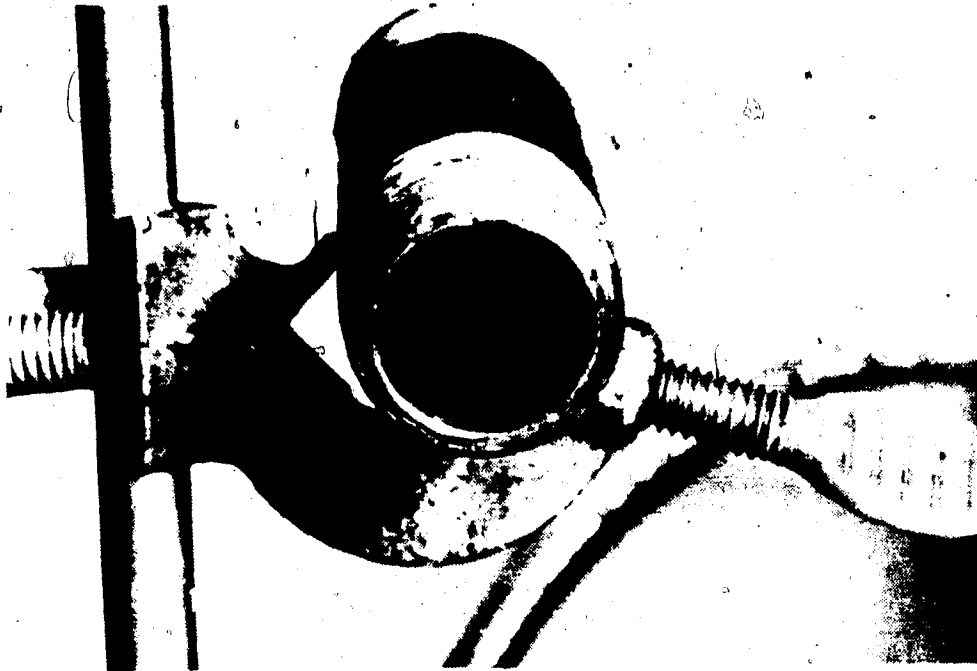


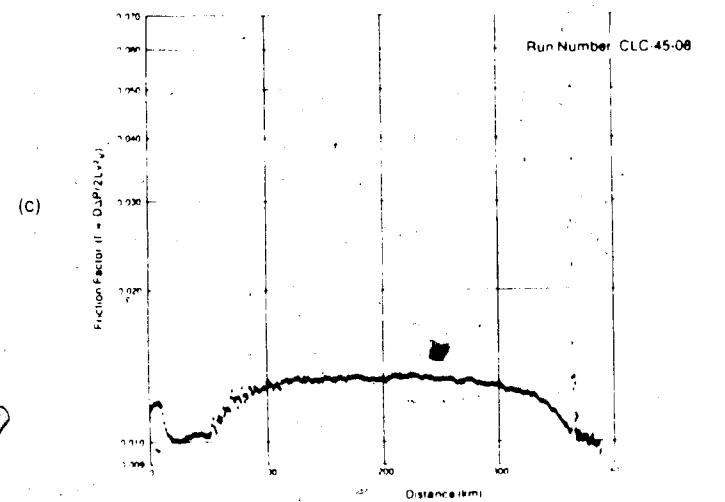
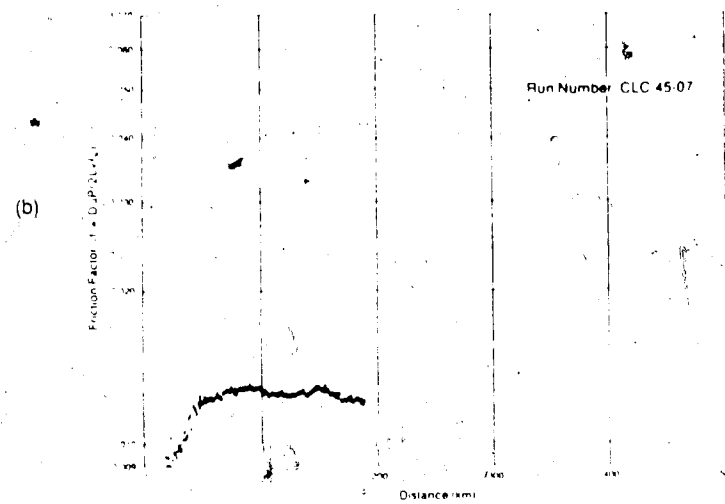
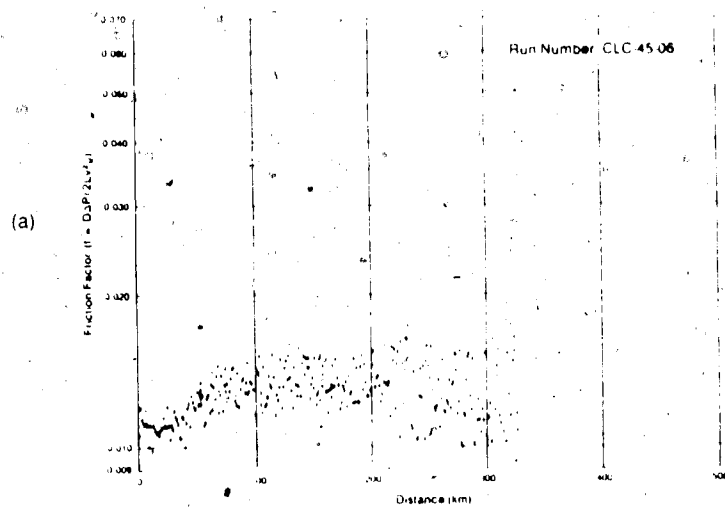
Figure 25: Removable section of pipe a) before emulsion flow test and b) after long term emulsion flow test

emulsion is obvious. This observation is consistent with the observed decrease in oil concentration throughout the course of each test. Oil concentration typically fell about 7% through the course of a test.

Repeatability of the long term tests is shown in Figures 26(a)-(c) by comparing the results of three long term tests under the same conditions. Test CLC-45-06 was not completed because the motor used to turn the pump began to overheat after about 320 km and could no longer turn the pump at constant speed. Overheating of the motor reflects an increase in the energy required to maintain a constant flow rate in the system and indicates the end of a test is near. The large scatter in the pressure data during this test was considered unacceptable and changes to the system were made before the next test was begun. Test CLC-45-07 was terminated when a reduction gearbox failed. Test CLC-45-08 was the successfully completed test. All three tests show the same friction factor-distance behaviour. Tests CLC-45-06 and CLC-45-08 compare well in the distance travelled to emulsion breakdown. Thus repeatability of the long term tests appears to be acceptable.

Based on the test results, a critical distance is defined for each emulsion as the distance an emulsion travels under reasonably steady conditions before exhibiting the changes characteristic of an emulsion breakdown. Table 11 summarizes the results of all the tests successfully completed giving the distance travelled in kilometers to emulsion breakdown and the shear rate present.

Products cannot be transported in a conventional pipeline without being



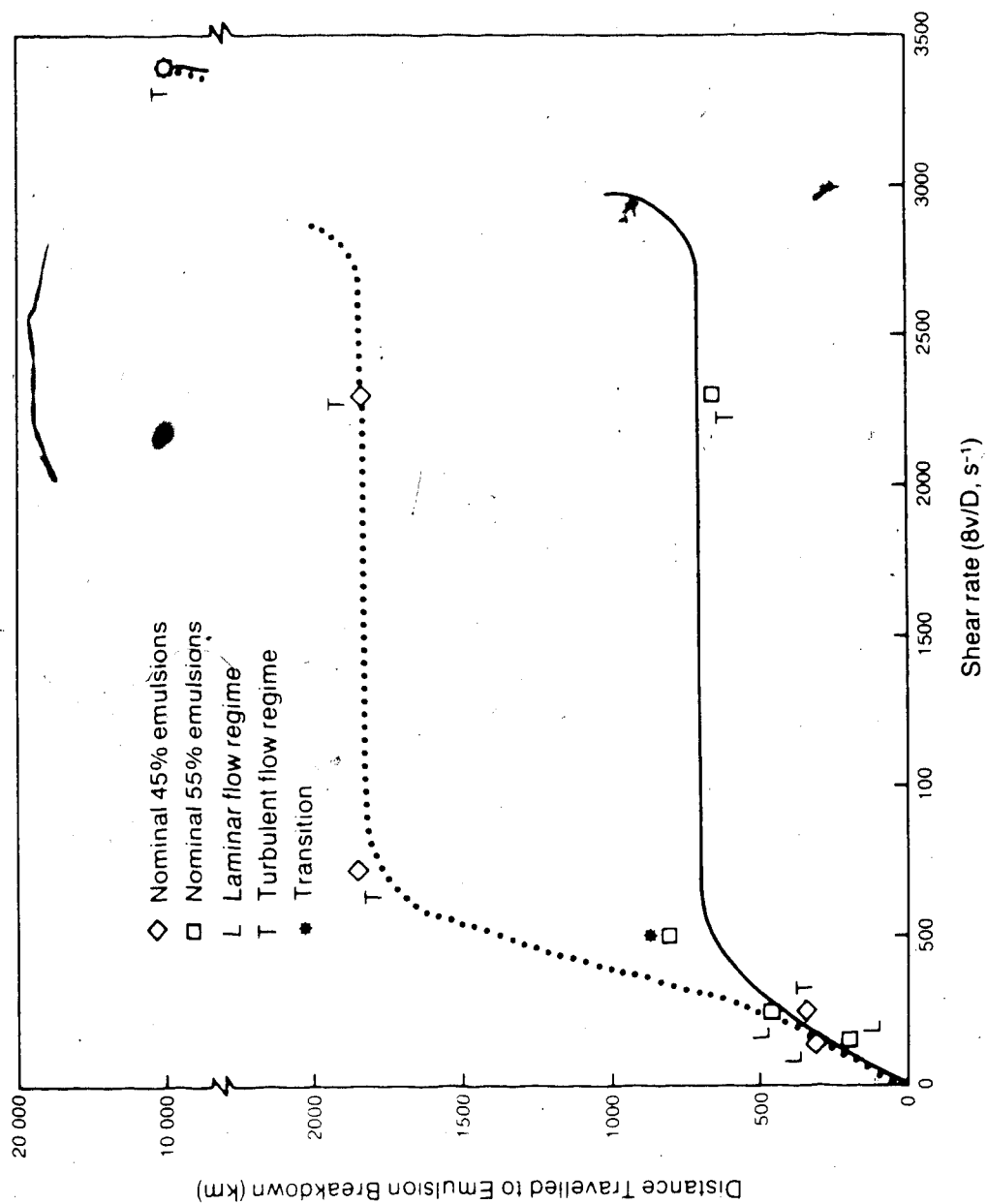
**Figure 26:** Comparison of the variation of friction factor with distance travelled for long term test CLC-45-06, CLC-45-07 and CLC-45-08

Table 11: Summary of distance travelled to emulsion breakdown for the long term pumping tests successfully completed

	Test Name	Shear Rate ( $s^{-1}$ )	Distance Travelled to Emulsion Breakdown (km)
Nominal 45% Emulsions	CLC-45-10	130	310
	CLC-45-08	250	340
	CLC-45-09	720	1850
	CLC-45-05	2300	1840
	CLC-45-11	3400	> 10000
Nominal 55% Emulsions	CLC-55-13	130	200
	CLC-55-10	250	460
	CLC-55-11	500	800
	CLC-55-09	2300	650
	CLC-55-12	3400	> 10000



subjected to shearing forces. The value of shear rate at the wall of the pipe is given as  $3*v/D$  and this was the shear rate used to correlate the results. Thus the distance travelled at a shear rate of zero would be zero km. Figure 27 summarizes the results, from Table 11, of all the tests successfully completed as the variation of distance travelled to emulsion breakdown with the shear rate ( $3*v/D$ ).



**Figure 27:** The variation of distance travelled before emulsion breakdown with shear rate ( $8v/D$ ).

## CHAPTER 4 : DISCUSSION OF RESULTS

### 4.1 Variation of Pressure Drop with Flow Rate

Over the wide range of Reynolds numbers covered, Figures 18 and 19 showed that the agreement between the emulsion  $f$  Re variation and the  $f$  Re variation obtained using a single phase Newtonian fluid, was good. Friction factors are consistently 5 to 12% lower than those predicted based on the measured physical properties of the emulsions. The deviation from the Newtonian calibration curve indicates that the emulsion in the pipeline does not behave as a single phase fluid. This trend was also noticed by Zakin [13] when measured pressure drops for pseudo-plastic emulsions in the turbulent regime were consistently lower than those predicted by the Dodge-Metzner equation. Zakin considered the deviation to be the result of the viscoelastic nature of the emulsions. No measurements were made with the emulsions to determine if they were visco-elastic in nature.

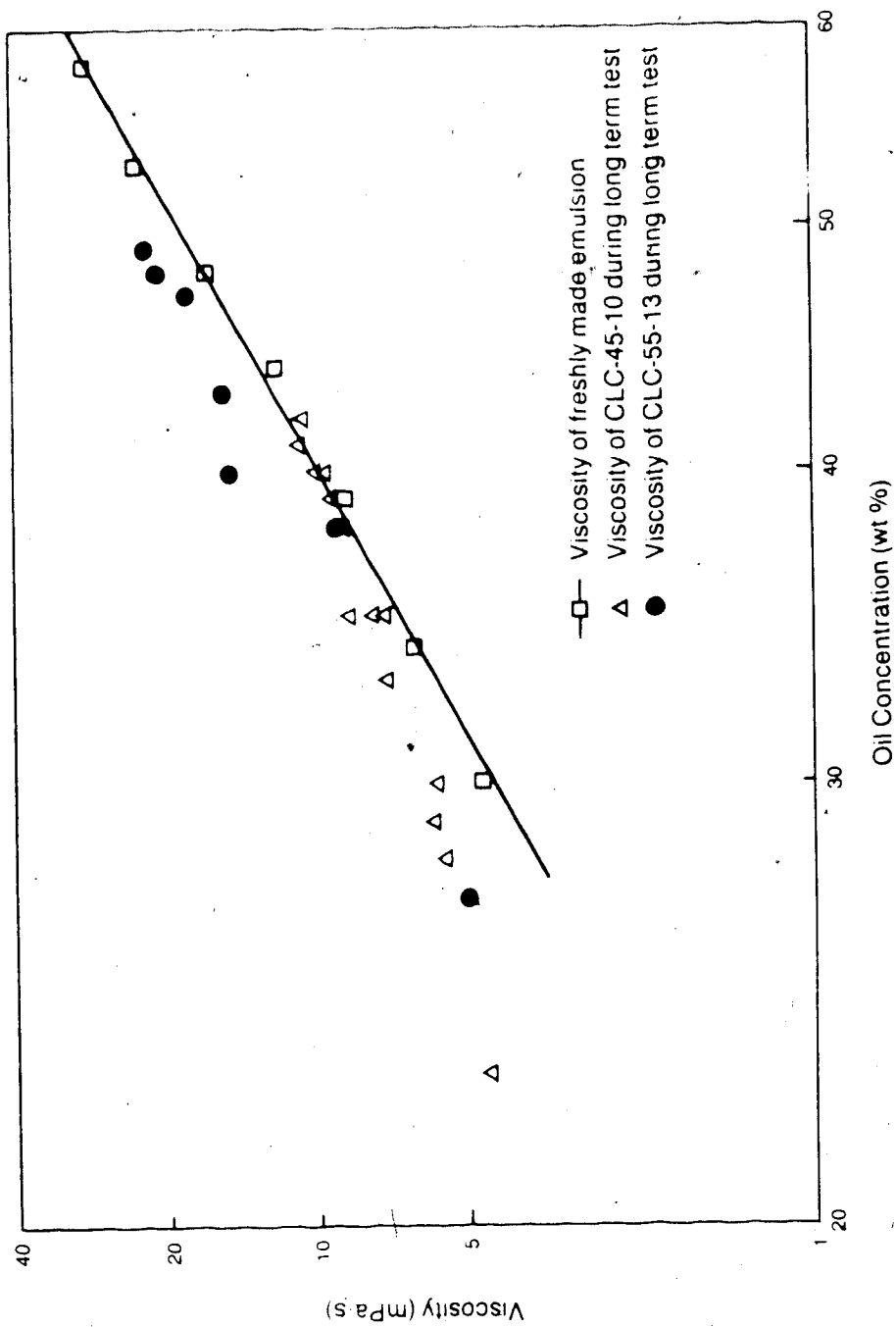
The deviation from the calibration curve is higher for the nominally 55% emulsions than for the nominally 45% emulsions. In the range of emulsion concentrations tested in this study, the measured physical properties, including the viscometry data, should give good estimates of pressure drop in a pipeline. However, should much higher oil concentrations be used, measured friction factors in a pipeline are required.

#### 4.2 Long Term Pumping Tests

The long term pumping tests were undertaken to investigate the stability of the emulsion during flow in a pipeline. The overall trend observed in the long term continuous pumping tests was that the system remained steady for some time, followed by a rapid breakdown in the emulsion. The oil concentration decreased steadily during the course of a test, as did the viscosity measured for the emulsion. Samples taken at the end of test separated rapidly into oil and water layers, indicating complete breakdown of the emulsion. This is a result of the coalescence of the emulsion oil droplets during flow in the pipeline.

During a typical long term test, as illustrated in Figure 20 by test CLC-45-09, emulsion breakdown is readily observed by the simultaneous change in several of the system variables. Friction factors changed sharply, at the same time the temperature of the system rose and the current drawn by the D.C. motor to turn the pump at constant speed increased. It was no longer possible to obtain meaningful viscosity measurements with the viscometer on samples taken from the pipeline. This was because the shear stress readings at constant shear rate became erratic due to the presence of large bitument droplets which bridge between the rotor and the cup of the viscometer.

Figure 28 illustrates the variation in viscosity with concentration for freshly made Cold Lake crude emulsions together with the emulsion viscosity measurements made during long term tests CLC-45-10 and CLC-55-13. These data indicate that the continual decline in viscosity



**Figure 28:** The variation of viscosity with concentration for freshly made Cold Lake crude and during the course of tests CLC-45-10 and CLC-55-13

during a long term pumping test is related to the decline in oil concentration in a manner similar to the fresh emulsion data. Sherman [32], showed that the viscosity of dilute emulsions decreases with an increase in particle size as well as with a decrease in concentration. Visual observation of the emulsion droplets in samples taken during a test did indicate that the oil droplet size was increasing although no systematic particle size analysis was carried out. From Figure 28, it appears then that prior to breakdown the decrease in emulsion viscosity was primary due to the decrease in oil content rather than the change in the oil droplet size.

The decrease in friction factor during the initial part of a long term test is a reflection of the decrease in the observed pressure drop at a constant flow rate. The density of an emulsion can be assumed to remain constant during the course of a test as the relative density of the oil used is given as .9994 at 15°C (Table 4). Thus the decrease in pressure drop is due to the observed decrease in emulsion viscosity, a result of the loss of oil from the emulsion. The more rapid decrease in friction factor observed in the laminar flow tests, as opposed to the turbulent flow tests, is reasonable since friction factor is more sensitive to Reynolds number in the laminar flow regime than in turbulent flow. The deposition of oil on the walls of the pipe decreases the inner diameter of the pipe and at constant mass flow rate, a decrease in inner diameter should be reflected in an increase in pressure drop. As this was not the case, the reduction in emulsion viscosity was the dominant cause for the observed reduction in pressure drop.

The rapid increase in temperature at the end of a test, Figure 20(b), is seen as a result of two processes. There was a reduction in the heat transferred to the cooling water due to coating of the pipe walls with oil. The oil coating has a thermal conductivity less than 1/100th of that of the pipe. As well the energy added to the system increased significantly when the emulsion breaks down, Figure 20(d).

Emulsions that exhibit stability on standing may frequently be broken by subjecting them to mechanical forces such as shearing forces that occur during stirring or in pipeline flow. Both van de Ven and Mason [26] and Zeichner and Schowalter [27] studied the effect of simple shear on the coalescence of two droplets. For a given value of surface potential, the rate of shear either enhanced or hindered the formation of doublets, which is the first step in the coalescence of droplets. At very high shear rates the formation of doublets was found to be hindered. This is in qualitative agreement with the findings of this study as illustrated in Figure 26. At the highest shear rates investigated emulsion breakdown was not observed which indicates that coalescence of the droplets was much hindered. The method of making emulsions was not varied in this study, thus the surface properties of the emulsion droplets should be essentially constant from test to test for a given nominal oil concentration. Shear rate and concentration are therefore the variables which will determine the stability of a particular emulsion under pipeline flow conditions.

In Figure 26, the distance travelled by an emulsion to breakdown at low values of shear rate does not appear to be a strong function of either

oil concentration or shear rate. All emulsions travelled between 200 and 450 km before breaking down. At intermediate values of shear rate, the distance travelled by an emulsion appears to be a strong function of oil concentration but not of shear rate. The nominally 45% emulsions travelled about 1800 km versus 700 km for the nominally 55% emulsions. At very high shear rates, neither the nominally 45% emulsion nor the nominally 55% emulsion had broken down after 14 days of continuous pumping (>10,000 km) and at this point both tests were terminated. Thus an actual value for the critical distance was not established for either nominal oil concentration.

Chemical analysis of the components of the emulsion for ions commonly associated with emulsion breaking are presented in Table 12. Samples of the heavy oil and aqueous solution are analysed 1) before emulsification, 2) after emulsification but before a long term pumping test and 3) after a long term test. The concentrations of ions commonly associated with emulsion breaking are not high enough to be the reason for emulsion breakdown. Consequently, it is believed that the emulsion breakdown is not chemical in nature, rather it is shear induced.

The major drawback of a fixed diameter pipeline loop is the inability to independently vary the values of Reynolds number,  $Dv\rho/\mu$  and shear rate,  $8v/D$ . Under actual pipeline conditions, the large pipe diameters give rise to high values of Reynolds number at relatively low values of shear rate for the same flow velocities and kinematic viscosities as used in this study. Thus it will not be practical to achieve the high values of shear rate of this study in a large diameter pipeline application.



Table 12: Chemical analysis for ions commonly associated with emulsion breaking

Bitumen samples are reported as  $\mu\text{g/g}$  bitumen.  
Water samples are reported as  $\mu\text{g/ml}$  water.

	Si	Na	Ca	K	Mg	Li	Sr	Al	Fe	Mn
<b>Component phases of emulsion</b>										
Initial dried bitumen	<3.6	1.4	2.3	N/A	<4.9	N/A	<0.2	<7.3	3.1	<0.2
Initial aqueous solution	0.5	705	<0.1	0.9	<0.2	<0.1	<0.1	<0.3	<0.1	<0.1
<b>CLC-45-08 Initial composite sample</b>										
Dried bitumen	14.7	153	<2.0	N/A	<8.2	N/A	<0.4	<12.3	0.8	<0.2
Aqueous solution	0.9	300	<0.1	0.7	<0.2	<0.1	<0.1	<0.3	<0.1	<0.1
<b>CLC-45-08 Sample taken after long term test</b>										
Dried bitumen	<5.2	240	1.9	N/A	<7.0	N/A	<0.3	<10.5	3.2	<0.2
Aqueous solution	2.4	485	0.3	1.2	<0.2	<0.1	<0.1	<0.3	<0.1	<0.1
	Zn	B	Ba	Cd	Cu	Pb	V	Ti	Ni	
<b>Component phases of emulsion</b>										
Dried bitumen	<0.7	1.0	<0.2	<0.2	<0.2	<4.9	188	1.5	69	
Initial aqueous solution	<0.1	<0.1	<0.4	<0.1	<0.1	<0.2	<0.1	<0.1	<0.1	
<b>CLC-45-08 Composite sample</b>										
Dried bitumen	<1.2	<1.6	<0.2	<0.4	1.0	<8.2	163	<0.4	54	
Aqueous solution	<0.1	0.2	<0.4	<0.1	<0.1	<0.2	<0.1	<0.1	<0.1	
<b>CLC-45-08 Sample taken after long term test</b>										
Dried bitumen	<1.0	<1.4	<0.2	<0.3	0.7	<6.8	159	<0.3	53	
Aqueous solution	<0.1	0.2	<0.4	<0.1	<0.1	<0.2	<0.1	<0.1	<0.1	

N/A = not available

The choice of a suitable pump must now be re-examined in view of the results in Figure 27. A low shear pump was chosen initially because it was felt that excessive shear in the pump would result in the redispersion of the emulsion while passing through the pump. Figure 27, however suggests that a low shear in the pump may enhance droplet coalescence. As the shear rate in the pump is lower than that in the pipeline, droplet coalescence may be occurring more readily in the recirculating test circuit than in an actual pipeline. Thus the stability results from all of the tests are conservative.

## CHAPTER 5 : CONCLUSIONS

The major conclusions that may be drawn from the work done with the 3/4 inch diameter pipeline loop are:

- 1) Simple viscosity measurements indicate the suitability of an emulsion for pipeline transportation but do not predict the stability of an emulsion under pipeline flow.
- 2) Fresh emulsions with concentrations less than 55% by weight oil give friction factor versus Reynolds number behaviour that agrees well with that predicted based on the measured physical properties of the emulsions.
- 3) The breakdown of an emulsion in the pipeline is clearly indicated by distinct changes in the system characteristics of pressure drop, temperature and power required to turn the pump at constant speed.
- 4) The stability of the emulsions under pipeline flow is a function of oil concentration and shear rate.
- 5) Emulsions are transported furthest when the shear rate is highest.

## CHAPTER 6 : RECOMMENDATIONS

Recommendations for further work include:

- 1) similar studies involving commercial emulsifiers. In particular, non-ionic emulsifiers should be investigated because of their ability to form emulsions with water containing high salt concentrations.
- 2) the effect of pipe diameter on emulsion stability.
- 3) particle size analysis on samples taken during a long term pumping test to enhance the understanding of the kinetics of droplet coalescence.
- 4) the mixing between sequential batches of emulsion or emulsion and oil in a pipeline. This is of significance in a common carrier pipeline system.
- 5) running identical tests with different pumps in the system.
- 6) more fundamental work studying the coalescence of concentrated emulsions in a simple shear device.

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## APPENDIX A : EMULSION PIPELINING PATENTS

The patent literature dealing with emulsion pipelining was reviewed and the major patents are summarized below. These summaries give the oil concentration and emulsification agents recommended in the patent. The summaries also note where actual pipeline experiments were conducted.

# EMULSION PIPELINING PATENTS

PATENT NUMBER AND ISSUE DATE	AUTHORS, ASSIGNEE	DESCRIPTION ( EMULSION FORMATION TECHNIQUES )
US 2981683 4 25 61	SIMPSON, W.C. SOMMER, H.J. SHELL	An oil-in-water suspension is made with 10 to 70% of a waxy crude oil having a pour point above 24 degrees C (75 degrees F) and the suspension is transported through a pipeline. No emulsifiers are required.
US 3006354 10 31 61	SOMMER, H.J. SIMPSON, W.C. SHELL	Oil-in-water emulsions containing 25 to 45% water are made with 35 to 100 ppm ammonia based on the weight of the oil. Tests with an emulsion made with a 16 degree API California crude on a 145 kilometer (90 mile) 25 and 30 centimeter (10 and 12 inch) pipeline were described briefly.
US 3425429 2 4 69	KANE, CHEVRON INC.	Crude oil is initially emulsified in 10 to 25% by volume of water containing 0.05 to 0.1% by volume of a nonylphenoxy polyethelenoxy ethanol surfactant. The solution is then reduced to 4 to 5% by volume of the oil. The formulas for the surfactants are (i) $R_1R_2ArO(CH_2CH_2)_nH$ and (ii) $R_1R_2ArO(CH_2CH_2O)_mH$ where $R_1, R_2$ are any alkyl, $Ar$ = phenyl and $n, m$ are 4 to 100.

US 3487844

1 6 70

SIMON, R.

MCAULIFFE, C. D.

POYNTER, W. G.

JENNINGS, H. Y.

CHEVRON RESEARCH

An emulsion is formed with 50 to 70% by volume water containing 0.05 to 1.4% by weight of a base, such as NaOH, transported through a pipeline to a second location and broken to separate the oil and water. Pipeline tests involving large batches of Casimalia crude oil are described and results presented. Methods for breaking the emulsions are also claimed.

US 3491835

1 27 70

GAGLE, D. W.

PHILLIPS

This is a general patent describing a process for transporting and desalting asphaltic crudes which are not pumpable at ambient temperatures. An oil-in-water emulsion is formed with a nonionic, cationic or anionic emulsifier, transported in a pipeline, broken and separated into oil and aqueous phases and part of the aqueous phase is returned for reuse. Specific surfactants claimed are polyoxyethylated alkylphenols or anionic surfactants of the form  $RO(C_2H_4O)_x(CH(CH_3)CH_2O)_y(C_2H_4O)_zH$  where R is aryl or alkaryl,  $x+y=0$  and  $z=20$  to 60 or  $x,y>0$  and  $x+z=50$  to 350,  $y=40$  to 60.

US 3519006

7 7 70

SIMON, R.

POYNTER, W. G.

An oil-in-water emulsion is made with 10 to 30% by volume of an aqueous solution containing 0.04 to 10% of a nonionic surfactant. Various surfactants are listed as being preferred.

US 3670752

6 20 72

MARSDEN, S. S.

ROSE, S. C.

An oil-in-brine solution is made with 30 to 60% by volume of a brine solution containing sufficient salts to prevent freezing at temperatures from -10 to -1 degrees C (15 to 30 degrees F), and 0.1 to 5% by

weight of a nonionic surfactant.

An integrated transportation system is described in which part of the fossil fuel is converted to methanol and used to transport the crude oil as an oil-in-methanol emulsion.

An emulsion is made with 10 to 40% by volume of an aqueous solution containing (a) 0.01 to 2% by weight of an anionic surfactant such as (i) alkyl sulphate salts with 6 to 20C or (ii) salts of unsaturated aliphatic carboxylic monobasic acids with 6 to 20C, (b) 0.05 to 5% by weight of a guanadine salt, (c) 0 to 1% of an alkalinity agent such as NaOH or KOH, (d) 0 to 1% of a nonionic surfactant of the formula  $RO(CH_2CH_2O)_nH$  where R is 6 to 20C alkyl or alkaryl,  $n=1$  to 20.

An emulsion is made with 15 to 70% by volume of an aqueous solution containing 0.001M to 0.5M of an alkali metal hydroxide and crude oil containing 1 to 10% by volume of a fatty acid or mixture of fatty acids.

An emulsion is formed with 8 to 60% by volume of an aqueous solution containing (a) 0.01 to 2.5% by weight of an oxyalkylated anionic surfactant and optionally (b) 0.01 to 1.0% by weight of an alkalinity agent such as NaOH, KOH or LiOH.

MANNESMANN  
ROHR

FLOURNOY, K. H.  
CARDENAS, R. L.  
CARLIN, J. T.  
TEXACO

CARLIN, J. T.  
POSEY, L. K. S.  
CARDENAS, R. L.  
TEXACO

KALFOGLOU, G.  
FLOURNOY, H.  
TEXACO

BE 824883  
5 15 75

US 3943954  
3 16 76

CA 1027349  
3 7 78

US 4099537  
7 11 78

US 4108193      FLOURNOY, K. H.      An emulsion is formed with 10 to 40% by volume of an aqueous solution  
8 22 78      CARDENAS, R. L.      containing (a) 0.01 to 2% by weight of an anionic surfactant such as  
CARLIN, J. I.      an alkyl sulphate salt with 6 to 20C or an unsaturated aliphatic  
TEXACO      carboxylic monobasic acid salt with 6 to 20C, (b) 0.01 to 1% by  
weight of an alkalinity agent such as NaOH, KOH or LiOH and (c) 0.01  
to 0.5% by weight of a guanidine salt or an oxyalkylated nitrogen  
containing aromatic compound.

US 4134415      FLOURNOY, K. H.      An emulsion is formed with 15 to 35% by volume of water containing  
1 16 79      ALSTON, R. B.      (a) 0.01 to 2% of an alkyl sulphonate or ethoxylated alkyl or  
BRADEN, W. B. JR.      alkaryl sulphonate, (b) 0.01 to 1.8% by weight of a rosin or naph-  
TEXACO      thenic acid soap and optionally (c) 0.02 to 1% by weight of a  
coupling agent such as glycol mono ether and/or an alkalinity agent  
such as NaOH or KOH.

US 4152290      FLOURNOY, K. H.      An emulsion is formed with 10 to 40% by volume of an aqueous solution  
5 1 79      CARDENAS, R. L.      containing (a) 0.01 to 2% of a alkyl sulphate salt or the salt of an  
CARLIN, J. I.      unsaturated aliphatic carboxylic monobasic acid, (b) 0.01 to 1% by  
TEXACO      weight of an alkalinity agent such as NaOH, KOH, or LiOH and option-  
ally (c) 0.01 to 0.5% by weight of a guanidine salt or oxyalkylated  
nitrogen containing aromatic compounds. Differs from US 4108193 in  
the formulae for the anionic surfactant.

US 4153573      KALFOGLOU, G.      An emulsion is formed with 8 to 60% by volume of an aqueous solution  
5 8 79      FLOURNOY, K. H.      containing (a) 0.01 to 2.5% by weight of an oxalkylated anionic  
TEXACO      surfactant and (b) 0.01 to 1% of an alkalinity agent such as NaOH,  
KOH, and LiOH. The aqueous      on is patented.

US 4153575      KALFOGLOU, G.      Same as US 4153573 but the emulsion itself is patented.

5 8 79      FLOURNOY, K. H.  
TEXACO

US 4162989      FLOURNOY, K. H.      Same as US 4152290 but the emulsion itself is patented.

7 31 79      CARDENAS, R. L.  
CARLIN, J. T.  
TEXACO

US 4192767      FLOURNOY, K. H.      An oil-in-water emulsion is formed by adding water containing 1) 0.01  
3 11 80      ALSTON, R. B.,      to 2% by weight ethoxylated quinoline sulphonate and 2) 0.01 to 1.8%  
BRADEN, W. B. JR.      by weight of rosin soap and/or naphthenic soap with 3) .02 to 1.0%  
TEXACO      of an optional coupling agent or .05 to 1.0% alkalinity agent.

US 4239052      MCCLAFLIN, G. G.      An oil-in-water emulsion is formed by adding 20 to 80% by volume  
12 16 80      CONOCO      water containing (a) 50 to 10000 ppm of an ethoxylated mono or  
dialkyl (6 to 15C) phenol containing 25 to 75 ethoxy groups and (b)  
50 to 10000 ppm of a water soluble alkaryl sulphonate. Formula for  
(b) is :  $\text{RARSO}_3\text{M}$  with molecular weight less than 410, where Ar is a



benzene, toluene, xylene or ethylbenzene residue, R is a 4 to 16C alkyl with a total of 6 to 16C in the R group(s), n and m are 1 or 2, M is Na, K or  $\text{NH}_4$ .

US 4246919  
1 27 81  
MCCLAFLIN, G.G.  
CONOCO

An oil-in-water emulsion is formed by adding 20 to 80% by volume of a water containing (a) 50 to 10000 ppm of an ethoxylated mono (8 to 10C alkyl) phenol containing 30 to 70 ethoxy groups and (b) 50 to 10000 ppm of an ethoxylated propylene glycol with an oxyethylene content of 10 to 50% by weight and a molecular weight of 1300 to 2900.

US 4246920  
1 27 81  
MCCLAFLIN, G.G.  
CONOCO

An oil-in-water emulsion is formed by adding 20 to 80% by volume of an aqueous solution containing 400 to 10000 ppm of an alkali metal or  $\text{NH}_4$  hydroxide and 10 to 500 ppm of a surfactant. The surfactant is either a water soluble 8 to 14C alkyl benzene sulphonate or a 4:1 by weight mixture of an ethoxylated 8 to 12C alkyl phenol containing 20 to 100 ethoxy groups and a polyethylene glycol with a molecular weight of 1000 to 3000.

US 4249554  
2 10 81  
MCCLAFLIN, G.G.  
CONOCO

An oil-in-water emulsion is formed by adding 20 to 80% by volume containing (a) 50 to 10000 ppm of an ethoxylated monoalkyl (8 to 12C) phenol containing 40 to 70 ethoxy groups and (b) 50 to 10000 ppm of an ethoxylated alcohol sulphate. Formula for (b) is :

$[\text{CH}_3(\text{CH}_2)_x\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OSO}_3]^\text{M}$  where  $x = 8$  to  $20$ ,  $n = 1$  to  $50$ , and  $\text{M}$  is  $\text{Na}$  or  $\text{NH}_4$ .

US 4265264

SIFFERMAN, T. R.

5 5 81

CONOCO

An oil-in-water emulsion is formed by adding 20 to 80% by volume water containing a combination of (a) 25 to 20000 ppm of a salt of an ethoxylated alcohol sulphate, (b) 50 to 20000 ppm of a second surfactant. Formula for (a) is:  $(\text{CH}_3(\text{CH}_2)_x\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OSO}_3)^\text{M}$  where  $x = 10$  to  $16$ ,  $n = 3$  to  $12$ ;  $\text{M}$  is  $\text{Na}$  or  $\text{NH}_4$ . Formula for (b) is one of

i)  $\text{HO}(\text{CH}_2\text{CH}_2\text{O})_a(\text{CH}(\text{CH}_3)\text{CH}_2\text{O})_b(\text{CH}_2\text{CH}_2\text{O})_c\text{H}$  where  $a$  and  $c$  are 2 to 10 and  $a+c$  is 4 to 20,  $b = 6$  to 30.

ii)  $\text{HO}(\text{CH}(\text{CH}_3)\text{CH}_2\text{O})_a(\text{CH}_2\text{CH}_2\text{O})_b(\text{CH}(\text{CH}_3\text{CH}_2\text{O})_c\text{H}$  where  $a$  and  $c$  are 3 to 15 and  $a+c$  is 6 to 30,  $b = 4$  to 20.

iii)  $\text{R}(\text{OC}(\text{CH}_3)\text{HCH}_2)_a(\text{CH}_2\text{CH}_2\text{O})_b\text{H}$  where  $\text{R}$  is a 10 to 18C linear alkyl,  $a = 6$  to 30,  $b = 4$  to 20.

US 4276094

GUTNICK,

6 30 81

ROSENBERG

BIOTECHNOLOGIE A.G.

This patent is primarily aimed at removal of hydrocarbon residues.

Aqueous solution containing .01 to 20 mg/ml of alpha emulsan and 5 to 40 mM of divalent cation is made with fresh water or brine. The

alpha-emulsan is a extracellular microbial protien associated lipopolysaccharide produced by Acinetobacter sp. ATCC 31012.

US 4285356

SIFFERMAN, T. R.

9 8 81

CONOCO

An oil-in-water emulsion is made by adding 20 to 80% by volume water of a mixture of two surfactants (a) 40 to 60% by weight of an alkyl polyether ethoxylated sulphate and (b) 60 to 40% by weight of an

alcohol ether sulphate. Formula for (a) is :

$R(OCHMeCH_2)_a(OCH_2CH_2)_bOSO_3M$  where R is 8-14C alkyl, a= 2 to 13, b= 1 to 3, M is Na, K or  $NH_4$ . Formula for (b) is :

$[CH_3(CH_2)_xCH_2(OCH_2CH_2)_nOSO_3]_m$  where x= 10 to 16, n= 3 to 12 and M is Na, K or  $NH_4$ .

where R is a 8 to 14C alkyl; a=2 to 13, b=1 to 3; M is Na, K or  $NH_4$ ; x=10 to 16, n=3 to 12.

MCCLAFLIN, G.G.

US 4287902

CONOCO

9 8 81

An oil-in-water emulsion is formed by adding 20 to 80% by volume of a mixture of methanol and water containing 500 to 10000 ppm of an alkaryl sulphonate with molecular weight of 415 to 470. The methanol to alkaryl sulphonate weight ratio is 0.1 to 10:1. The sulphonate formula is :  $RArSO_3M$  where R is 17 to 22C alkyl; Ar is phenyl, tolyl, xylyl or ethylphenyl; M is Na, K or  $NH_4$ .

CONTINENTAL OIL

CA 1108205

9 1 81

An oil-in-water emulsion is formed by adding 10 to 80% by volume water containing 50 to 20000 ppm of a polyoxyethylated alkyl sulphate. The formula is :  $CH_3(CH_2)_xCH_2(OCH_2CH_2)_nOSO_3M$  where M is Na or  $NH_4$ ; x= 8 to 20, n= 1 to 50.

CONOCO

CA 1113529

12 1 81

An oil-in-water emulsion is formed by adding 20 to 80% by volume water containing 100 to 2000 ppm of a water soluble alkaryl sulfonate with molecular weight below 410. Formula is :  $ArSO_3M$  where Ar is a phenyl, tolyl, xylyl or ethylphenyl group, mono- or di-substituted

by 4 to 16 alkyl, and the alkyl groups contain a total of 8 to 16C atoms, M is Na, K or NH<sub>4</sub>.

CA 1117568 2 2 82	CONOCO	An oil-in-water emulsion is formed by adding 20 to 80% by volume water containing 50 to 20000 ppm of an alkoxyated alcohol. The formula is : $RO(CH_2CH_2O)_nH$ where R is 10 to 20C alkyl, n= 5 to 40.
US 4333488 6 8 82	MCCLAFILIN, G.G. CONOCO	An oil-in-water emulsion is formed by adding 10 to 80% by volume water containing 1 to 3000 ppm of an adipate ester of a polyoxyethylene polyoxypropylene block polymer. The formula is : $H(CH_2CH_2O)_a(CH(CH_2CH_2O)_b(CH_2CH_2O)_c)H$ where a= 5 to 20, c= 5 to 20, a+c= 10 to 40, b= 6 to 30.

US 4343323 8 10 82	RESEARCH COUNCIL OF ALBERTA	Deaerated water containing a strong base (NaOH) with pH of at least 11 is used to make emulsions with heavy crudes containing surfactant forming groups. The NaOH solution is recovered in the breaking stage.
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US 4355651 10 26 82	MCCLAFILIN, G.G. CONOCO	An oil-in water emulsion is formed by adding 20 to 80% by volume water containing 100 to 5000 ppm of a polyoxyalkylated phosphate ester. The formula is : $(R(C_2H_4O)_x(C_3H_6O)_y)P(O)(OH)_n$ where R is a 2-30C alkyl; x= 2 to 7, y= 2 to 5 m= 1 to 3, n= 0 to 2, with m+n=3.
------------------------	----------------------------	--

CA 1143313

3 22 83

CONOCO

An oil-in-water emulsion is formed by adding 10 to 70% by volume water containing 250 to 50000 ppm of a polyethoxylated amine surfactant.

## APPENDIX B : PROCEDURE FOR DETERMINING THE OIL CONTENT OF EMULSIONS

Emulsion samples were submitted to the Oil Sands Analytical Laboratory of the Alberta Research Council for analysis of oil content. The method of analysis, OSRD Method 1.05, is given below.

### OSRD METHOD 1.05

#### OIL SAND DEAN STARK (WITH FILTER PAPER DETERMINATION FOR BITUMEN)

##### Scope

This method is a fast and inexpensive analysis for the determination of the bitumen, water and solids content of oil sand. This method can handle samples containing larger amounts of light hydrocarbons, but it is not suitable for samples rich in clay fractions. 40 - 140 g of oil sand are required for optimal analysis.

##### Summary

The sample is separated into bitumen, water and solids by refluxing with toluene in a Modified Dean Stark Extractor. Condensed solvent extracts the bitumen and water from the solids. The water azeotropically distills with the toluene and collects in the Dean Stark trap. The water and toluene are then separated on phase separating filter paper, and the water is determined gravimetrically. The solids in the thimble are oven dried and determined gravimetrically. The toluene-bitumen bottoms are transferred into a volumetric flask and made up to volume. An aliquot is taken and is evaporated on a glass fiber filter paper. The bitumen is determined gravimetrically.

##### Safety Considerations

Toluene : moderately toxic by skin absorption and inhalation. Possesses

irritant and anesthetic properties. Highly flammable.

### Apparatus

Figure B-1 shows the assembled apparatus. The condenser is an Allihn condenser 30 centimeters long, 4 centimeters in diameter with a 24/40 standard taper bottom joint protected by a Teflon sleeve.

Figure B-2 shows a detailed drawing of the flask. The flask is a 1000 milliliter round bottom flask with a 270 millimeter long neck and a 55/50 standard taper female joint.

Figure B-3 shows a detailed drawing of the still head. The still head is a combination of an adaptor and a water trap. It consists of a 55/50 standard taper male joint protected by a Teflon sleeve, a 24/40 standard taper female joint and a 24/40 Teflon stopper, a 24/40 standard taper female joint and a 30 millimeter glass side arm which terminates in a Teflon stopcock.

The neck of the flask and the top of the still head are insulated with 3 millimeter (1/8 inch) thick by 25 millimeter (1 inch) wide fiberglass tape.

Figure B-4 shows a detailed drawing of the thimble basket. The thimble basket is made from two aluminum wires and holds the extraction thimble.

Figure B-5 is a detailed drawing of the solvent distributor. The aluminum solvent distributor and locking ring hold the thimble basket in place.

Other equipment required for this procedure include :

- 1) 1000 milliliter, 335 watt heating mantle
- 2) 0 to 120 volt output, 10 ampere variable transformer
- 3) 600 milliliter beaker graduated in 50 milliliter increments
- 4) 75 millimeter diameter stemless powder funnel
- 5) funnel support for beaker

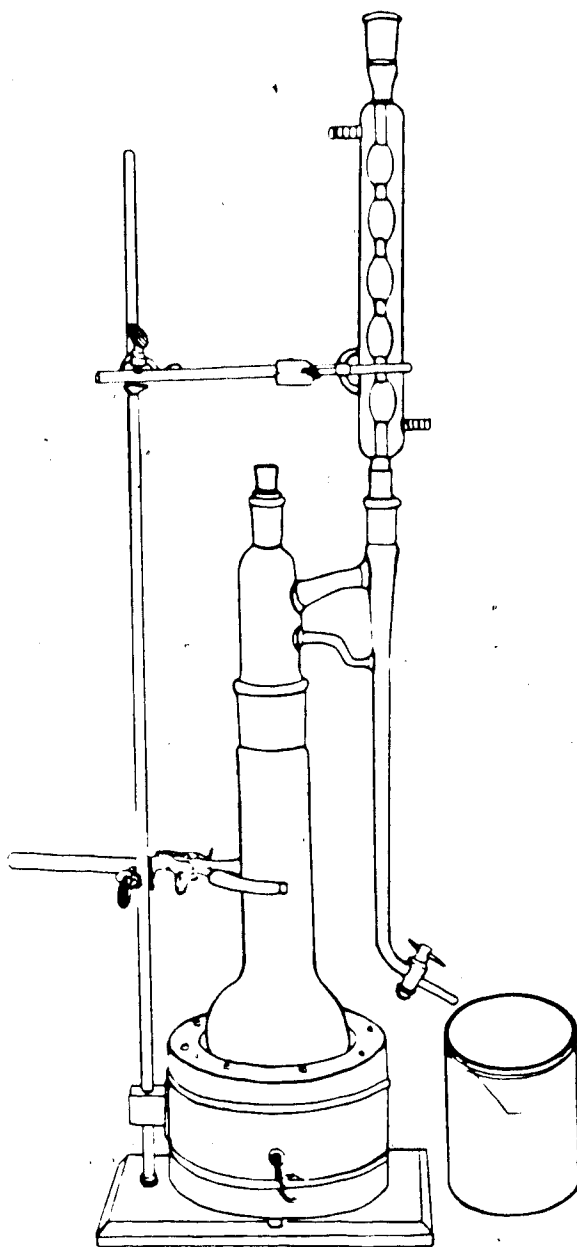


Figure B-1 : The modified Dean Stark extraction apparatus.



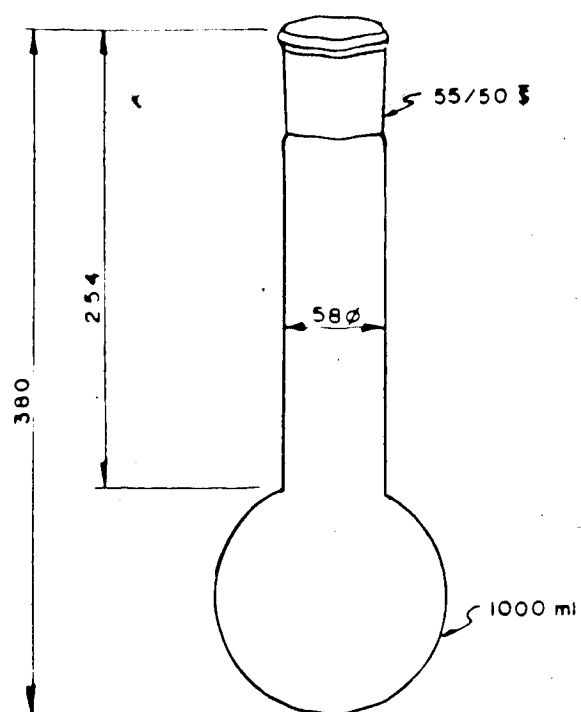


Figure B-2 : The extraction flask.



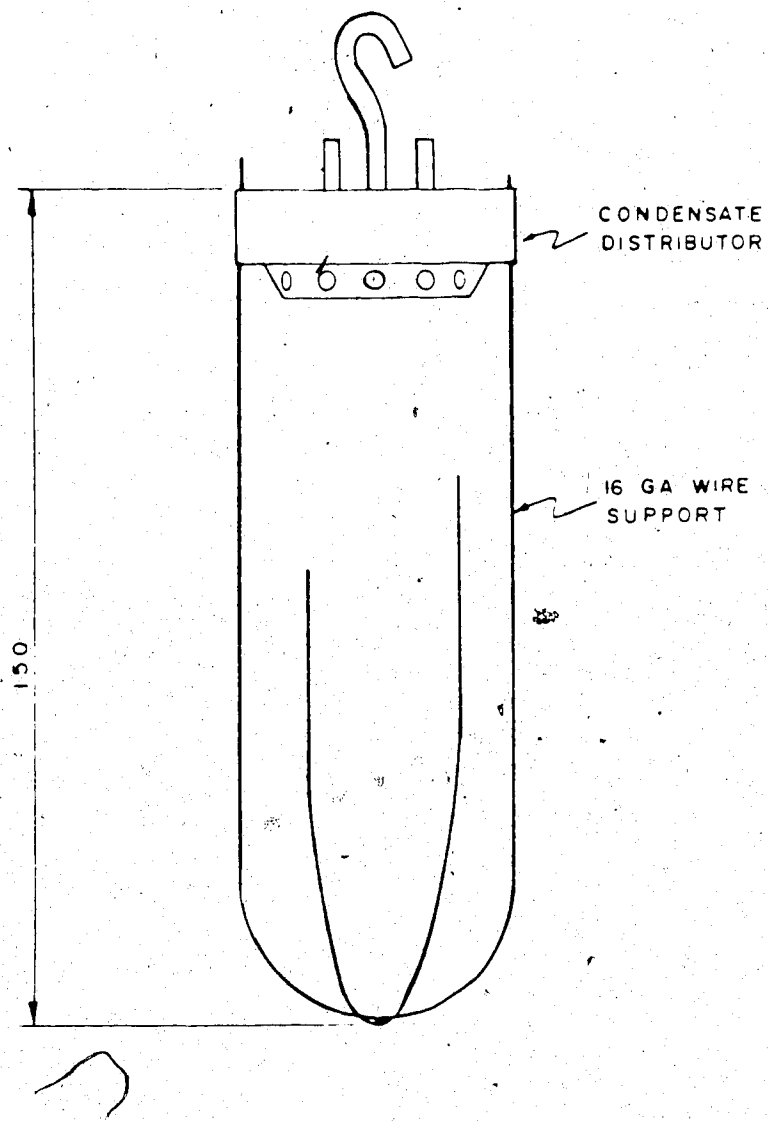


Figure B-4 : The basket support assembly.

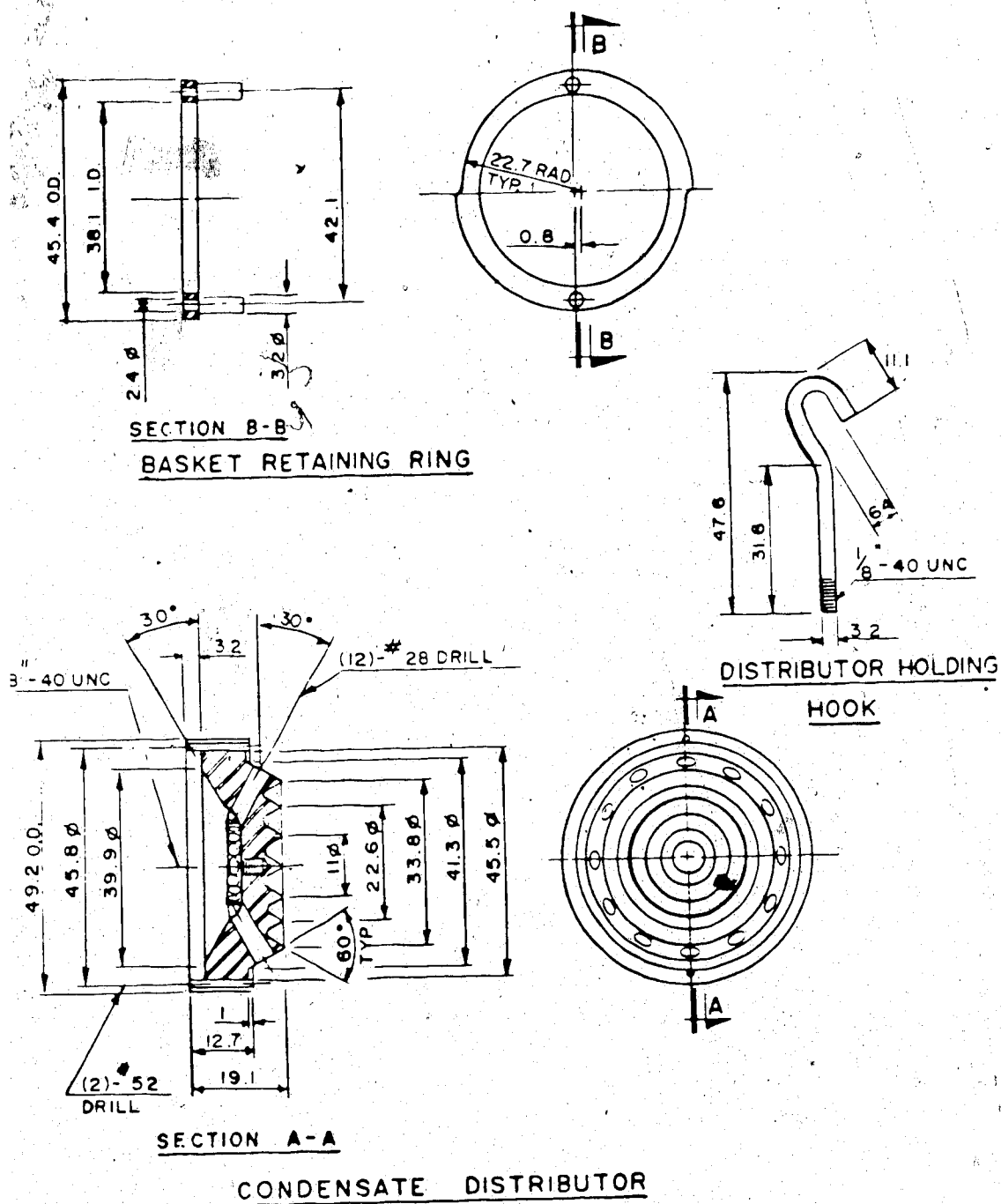


Figure B-5 : Basket retaining ring, condensate distributor and distributor holding hook.

- 6) water bottle with a 70 by 25 millimeter snap cap vial
- 7) 1200 gram capacity top loading balance readable to  $\pm 0.01$  gram
- 8) 500 millimeter volumetric flask
- 9) 5 millimeter pipet with bulb
- 10) 10 milliliter glass luer-lok tip syringe
- 11) 25 milliliter Erlenmeyer flask with ground glass top fitted with ground glass stopper
- 12) 100 millimeter diameter, 10 millimeter deep Petri dishes
- and 13) a drying rack consisting of a horizontal rod suspended between two retort stands approximately 1 meter apart. Ten 20 millimeter fold back clips are hung on the rod.

Materials and reagents required for this procedure are :

- 1) Whatman 43 by 123 millimeter cellulose extraction thimbles, single thickness, oven dried at  $105^{\circ}\text{C}$ , cooled and stored in a desiccator
- 2) Whatman IPS 15.0 centimeter diameter phase separating paper
- 3) 500 milliliters technical grade toluene
- 4) 15 centimeter diameter Reeve Angel glass fiber filter paper
- and 5) 0.45 micron by 25 millimeter diameter disposable Millipore Millex filter assemblies.

The extraction apparatus should be set up in a fume hood suitable for exhausting solvent vapours.

#### Procedure

- 1) Turn on the fume hood and the condenser water. Place 400 milliliters of toluene in the round bottom flask.
- 2) Weigh the extraction thimble to the nearest 0.01 gram. Transfer 100 to 140 grams of oil sand into the thimble and reweigh to the nearest 0.01 gram. Take care that no oil sand sticks to the outside of the thimble.
- 3) Immediately place the thimble in the wire basket, attach the

distributor and suspend from the bottom of the still head. Fill the side arm with toluene. Assemble the apparatus as shown in Figure B-1, ensuring all the connections are vapour tight.

- 4) Turn on the variable transformer, set to 90, and reflux for two hours or until all the water has distilled over and the toluene dripping from the thimble is clear.
- 5) Weigh the water collected to the nearest 0.01 gram.
- 6) Open the stopcock and drain the water and toluene from the Dean Stark trap onto the phase separating paper. Close the stopcock. Transfer the water to the water bottle as soon as the toluene has filtered off. Open up the filter paper and collect all the small drops. Do not allow the water to sit on the filter paper for more than 30 seconds.
- 7) Open the stopcock and distill off 250 milliliters of the toluene into the graduated 600 milliliter beaker.
- 8) Turn off the transformer and allow the flask to cool for approximately 30 minutes.
- 9) Reweigh the water bottle to the nearest 0.01 gram.
- 10) Disassemble the apparatus. Remove the thimble, the basket and dry it overnight at 90°C in the oven. Cool the thimble and reweigh it to the nearest 0.01 gram.
- 11) Dry the glass fiber filters by placing an open box of filter in an oven at 110°C for 1 hour. Cool them in a desiccator. Number the filters and weigh to the nearest 0.00001 gram.
- 12) Quantitatively transfer the toluene-bitumen bottoms from the 1000 milliliter flask to the 500 milliliter volumetric flask and dilute to volume.

- 13) Rinse a 10 milliliter syringe with the sample extract. Withdraw approximately 7 milliliters of the extract and attach a 0.45 micron Millipore Millex Filter assembly to the 10 milliliter syringe. Filter the extract into a 25 milliliter Erlenmeyer flask. Stopper the flask if the solution aliquot is not to be taken immediately. Rinse the 5 milliliter transfer pipet with the extract solution. Support a weighed glass fiber filter on a Petri dish. Pipet a 5 milliliter aliquot of the solution onto the filter by continuously moving the pipet to disperse the extract evenly on the filter surface. Hang the filter from the drying rack to evaporate the solvent. The time required to dry the filter is critical and is selected using a solution of known bitumen concentration. 5 milliliter aliquots are applied to filters and these are weighed at varying times. The time which gives the correct bitumen weight is selected for sample analysis. The drying time is about 20 minutes and need only be determined for the particular laboratory conditions in use. Reweigh the filter paper to the nearest 0.00001 gram.

### Calculations

The bitumen, water and solids content of the the oil sand are given by :

$$\text{Weight bitumen} = (\text{weight bitumen on filter paper}) * 500\text{ml}/5\text{ml}$$

$$\% \text{ bitumen} = (\text{weight bitumen} / \text{weight oil sand}) * 100$$

$$\% \text{ water} = (\text{corrected weight water} / \text{weight oil sand}) * 100$$

where the corrected weight of water = (weight water \* A) + B, where A and B are the slope and intercept of the equation generated from a calibration procedure. Known weights of water added to the extractors are plotted against the weights of water recovered by extraction under conditions identical to those used in a regular extraction of oil sand. The water correction factor may change due

to factors relating to the efficiency of the condensers used in the apparatus.  $A=1.003\pm0.001$ ,  $B=0.090\pm0.009$

% solids = (weight solids / weight oil sand)\*100

% discrepancy = 100 - % bitumen - % water - % solids.

### Precision

Typical values for the precision of an oil sand determination are tabulated below. These data were derived from 6 replicate analyses performed by a technologist on a sample of Cold Lake oil sand.

		OSRD Method 1.05	OSRD Method 1.00
% bitumen	X	10.14	9.16
	S	0.10	0.13
	RSD	0.98%	1.41%
% water	X	7.25	7.37
	S	0.27	0.28
	RSD	3.74%	3.84%
% solids	X	82.80	82.80
	S	0.09	0.09
	RSD	0.10%	0.11%
% discrepancy	X	-0.24	0.67
	S	0.16	0.11



### Sources of Error

#### Bitumen

##### Too Low

- 1) The heat was too high or the water flow was too low in the condenser. The solvent front was condensing above the second bulb of the condenser. Light ends of the bitumen may be lost.
- 2) Incomplete extraction. The sand and thimble should be clean if the extraction is complete. A dark thimble and/or sand may indicate incomplete extraction.
- 3) The bottoms boiled dry. Part of the bitumen cracked and distilled off. The bitumen will be baked onto the flask.
- 4) The joints of the extraction apparatus were not vapour tight allowing vapour loss.
- 5) Poor volumetric technique.
- 6) The filter paper was dried too long before weighing.
- 7) Spillage.

##### Too High

- 1) The flask was not cleaned from the previous extraction.
- 2) The bitumen was insufficiently dried.
- 3) The extract in the 25 milliliter Erlenmeyer evaporated before the aliquot was taken.

## Water

### Too Low

- 1) The heat was too high in the condenser. The solvent front was condensing above the second bulb of the condenser and water vapour was lost through the condenser.
- 2) The thimble was not immediately set up in the extractor, or the condenser water was not on when the thimble was placed in the extractor. Water evaporated from the oil sand sample.
- 3) The joints of the extraction apparatus were not vapour tight allowing vapour loss.
- 4) The side arm and condenser were dirty. Water drops may remain stuck to the side of the glass.
- 5) The water was left too long on the filter paper. Water losses due to evaporation and absorption occurred.
- 6) Spillage.

### Too High

- 1) Water was present in the toluene. This often occurs when the toluene is taken from the bottom of a barrel or solvent can.
- 2) The thimble was improperly dried.

## Solids

### Too Low

- 1) Solids bumped over the top of the thimble. Particles of solids may be visible in the bottom of the volumetric if this occurred.
- 2) The thimble was improperly dried before use.

3) The oil sand contains large quantities of clay which have passed into the bitumen.

4) Spillage.

Too High

1) The thimble was improperly dried after extraction.

2) Incomplete extraction. The sand and thimble should be clean if the extracion is complete. A dark thimble and/or sand may indicate incomplete extraction.

## APPENDIX C : SUMMARY OF RESULTS FROM THE EMULSION SCREENING PROGRAM

The objectives of the emulsion screening program were to find an emulsion suitable for the pipeline experiments and to establish a method for making large batches of emulsion. Emulsion formulation followed that described by Kessick and St. Denis in U.S. patent 4343323 [14].

Stability was based on changes observed in the viscosity of fresh emulsions, and where possible aged emulsions, with time at constant shear rate in the viscometer. In Tables C-1 and C-2, this information is recorded as:

- 1)  $\eta_i$  = viscosity of emulsion as soon as possible after being placed in the viscometer.
- 2)  $\eta_t$  = viscosity of the emulsion after  $t$  minutes of being sheared in the viscometer at a measuring head speed of  $512 \text{ min}^{-1}$
- 3) other noticeable changes to the emulsion.

Table C-1 contains the results for the 40 and 55% by weight oil-in-water emulsions made with the Cold Lake crude. Tests were not conducted for every combination of oil concentration, emulsion formulation and emulsification equipment. Rather promising emulsions were investigated further and poor emulsions were abandoned. Table C-2 contains the results for the emulsions made with the dewatered bitumen.

Table C-1: Summary of screening test results for Cold Lake crude emulsions

Emulsion Formulation: Cold Lake Crude with 0.1% NaOH aqueous solution

wt % oil	Emulsification Method	
	Paint Stirrer	Homogenizer
40	Emulsion: CLC-40-01	No emulsion was made
	fresh: $\eta_1 = 6.0$ $\eta_3 = 5.8$ $\eta_{73} = 7.0$ 23 hrs: $\eta_1 = 6.3$ $\eta_7 = 6.6$ $\eta_{68} = 7.8$	
55	Emulsion: CLC-55-05	Emulsion: CLC-55-6 (fresh sample measurement not possible)
	fresh: $\eta_1 = 15.8$ $\eta_{59} = 28.4$ Viscosity increased quickly during the first 30 minutes and was still increasing after 60 minutes. After standing overnight, 3 tests were done. i) $\eta_1 = 35.7$ $\eta_{57} = 46.3$ ii) $\eta_1 = 36.8$ $\eta_{56} = 40.9$ iii) $\eta_1 = 38.2$ $\eta_{56} = 53.2$ The repeatability was poor after 30 minutes	27 hrs: $\eta_1 = 22.9$ $\eta_{59} = 29.6$ $\eta_{\text{minimum}} = 28.0$ @ 28 minutes 50 hrs: $\eta_1 = 32.0$ $\eta_{56} = 30.1$ Both samples exhibited the same viscosity behaviour with time after 30 minutes. Emulsion: CLC-55-08 (long term standing test) age (hrs): 5    7    24    48    72    96 $\eta_1$ 17.3   18.3   22.0   27.6   31.4   33.0 $\eta_{60}$ 26.2   27.9*   29.7   31.4   38.4 *t = 50 minutes

Emulsion Formulation: Cold Lake Crude with 0.1% NaOH and 0.02%  $\text{Na}_2\text{S}_2\text{O}_4$  aqueous solution

wt % oil	Emulsification Method	
	Paint Stirrer	Homogenizer
40	Emulsion: CLC-40-02	No emulsion was made
	fresh: $\eta_1 = 6.0$ $\eta_1 = 5.8$ $\eta_{71} = 7.5$ 21 hrs: $\eta_1 = 6.4$ $\eta_3 = 6.6$ $\eta_{70} = 8.1$	
55	Emulsion: CLC-55-03	CLC-55-07
	fresh: some difficulty with temperature control in viscometer. fresh: $\eta_1 = 16.8$ $\eta_{33} = 21.5$ Emulsion: CLC-55-04 fresh: $\eta_1 = 16.7$ $\eta_{32} = 23.2$ $\eta_{89} = 34.2$ The increase in viscosity with time was linear. Viscosity was still increasing after 90 minutes. Good agreement in viscosity behaviour between the two samples.	Emulsion was made but no viscosity work was done with this sample.

Table C-2: Summary of screening test results for dewatered bitumen emulsions

Emulsion Formulation: Dewatered Bitumen with 0.1% NaOH aqueous solution

wt % oil	Emulsification Method	
	Paint Stirrer	Homogenizer
40	Emulsion: DWB-40-01 fresh: $\eta_1 = 11.2$ $\eta_3 = 8.8$ $\eta_{60} = 11.1$ Sample stiffened overnight and no further measurements were taken.	Emulsion: DWB-40-02 Flow curves were generated using RV3 viscometer and appeared to be Newtonian. The viscosity versus time behaviour was not studied. Sample inverted after 2 days.
55	Emulsion: DWB-55-01 fresh: The fresh sample gave very steady viscosity readings with almost no increase for the first 60 minutes. fresh: $\eta_1 = 48.4$ $\eta_3 = 38.7$ $\eta_{60} = 40.0$ 1 hour: The sample had stiffened but was redispersed by stirring at 330 rpm for 12 minutes. Readings were made at measuring head speed of $\eta = 64$ rpm. $\eta_1 = 221$ $\eta_{20} = 149$ $\eta_{58} = 162$ ;	Emulsion: DWB-55-02 Flow curves were generated using RV3 viscometer and the fresh sample appeared to be Newtonian. The 1 hour old sample displayed severe hysteresis and had twice the viscosity of the fresh sample. The sample inverted after 6 hours (would not disperse in water).

Emulsion Formulation: Dewatered Bitumen with 0.1% NaOH and 0.02%  $\text{Na}_2\text{S}_2\text{O}_4$  aqueous solution

wt % oil	Emulsification Method	
	Paint Stirrer	Homogenizer
40	Emulsion: DWB-40-03 (sample spill during preparation) fresh: $\eta_1 = 9.6$ $\eta_2 = 9.0$ $\eta_{69} = 11.6$ 20 hours: $\eta_1 = 51.5$ $\eta_3 = 35.1$ $\eta_{56} = 26.5$ 23 hours: $\eta_1 = 50.8$ $\eta_3 = 36.6$ $\eta_{60} = 27.2$ 44 hours: $\eta_1 = 94.1$ $\eta_1 = 54.5$ $\eta_{61} = 33.4$ measuring head speed: $n = 256$ rpm Emulsion: DWB-40-04 (repeat sample) fresh: $\eta_1 = 9.4$ $\eta_2 = 8.5$ $\eta_{61} = 11.8$ Agreement between fresh samples is good.	No emulsion was made
55	Emulsion: DWB-55-03 fresh: $\eta_1 = 41.8$ $\eta_2 = 35.3$ $\eta_{61} = 52.3$ $\eta_{82} = 56.2$	No emulsion was made

#### APPENDIX D : EMULSION FLOW DATA

Emulsion pipeline flow data was collected on nine nominally 45% emulsions (CLC-45-03 through CLC-45-11) and five nominally 55% emulsions (CLC-55-09 through CLC-55-13). A brief description of the tests performed with each emulsion batch and the conditions present in the pipeline system during each test are given below. The data that were collected are presented graphically as:

- (1) the variation of friction factor with Reynolds number
  - (2) the variation of friction factor with distance travelled
  - (3) the variation of temperature with distance travelled
  - (4) the variation of emulsion oil concentration with distance travelled
- and (5) the variation of current drawn by the D.C. motor with distance travelled (where possible).

For each long term pumping test the viscosity and oil concentration of the emulsion are tabulated as functions of the distance travelled.

The order in which the tests were done is : CLC-45-03, CLC-45-04, CLC-45-05, CLC-55-09, CLC-45-06, CLC-45-07, CLC-45-08, CLC-55-10, CLC-45-09, CLC-55-11, CLC-45-10, CLC-45-11, CLC-55-12, CLC-55-13.

Emulsion : CLC-45-03

Description : This emulsion was used in a series of tests to establish the friction factor-Reynolds number behaviour of fresh emulsions. These tests established convenient operating, sampling and cleaning procedures for the emulsion flow experiments. The agreement between tests, despite changes in viscosity and temperature, established the validity of the Newtonian flow models used.

Table D-1: Conditions for tests with emulsion CLC-45-03.

	Test No. 1 to 3	Test No. 4
Flow Rate (kg/min)	86 to 160	64 to 88
Velocity (m/s)	4.1 to 7.5	3.0 to 4.2
Shear Rate ( $s^{-1}$ )	1500 to 2800	1100 to 1600
Reynolds Number	9,000 to 18,000	6,000 to 9,000
Temperature ( $^{\circ}C$ )	20 or 25	18 to 28

Emulsion : CLC-45-04

Description : This emulsion was used in a series of tests to establish the friction factor-Reynolds number behaviour of fresh emulsions and the agreement in flow behaviour between different batches of emulsion. Operating procedures for long term tests were established.

Table D-2: Conditions for tests with emulsion CLC-45-04.

	Test No. 1, 2, 6 & 7	Test No. 3 & 4	Test No. 9
Flow Rate (kg/min)	83 to 160	83 to 160	85 to 163
Velocity (m/s)	3.9 to 7.5	3.9 to 7.5	4.0 to 7.7
Shear Rate ( $s^{-1}$ )	1400 to 2800	1400 to 2800	1400 to 3000
Reynolds Number	9,000 to 18,000	6,000 to 14,000	9,000 to 18,000
Temperature ( $^{\circ}C$ )	about 20	9 to 12	11 to 20

Observations : Figure D-1 shows the results of friction factor-Reynolds number tests done with two different emulsion batches. The agreement between the tests is good.



Instrument Calibration :

Flow Meter 0-400 kg/min

Pressure Transducers 0-150 kPa

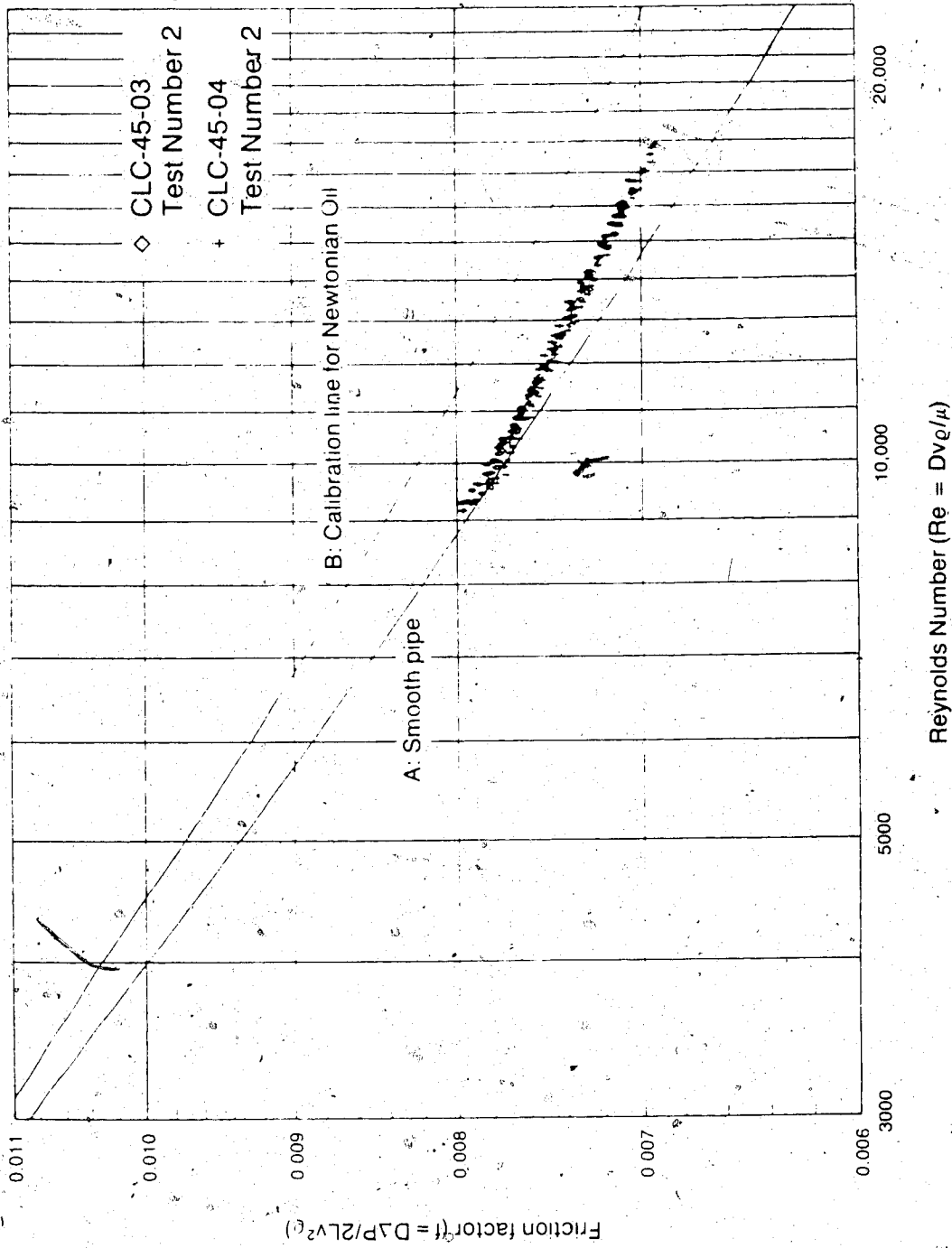


Figure D-1: The variation of friction factor with Reynolds number for tests with two different emulsion batches.

Emulsion : CLC-45-05

Description : Two tests were performed with this emulsion. Test number 1 investigated the friction factor-Reynolds number flow behaviour of the fresh emulsion over the same flow rate range as the tests with emulsions CLC-45-03 and CLC-45-04. The temperature during test number 1 was 10°C. Test number 2 was the first long term test at constant flow rate.

Table D-3: Conditions for tests using emulsion CLC-45-05.

	Test No. 1	Test No. 2
Flow Rate (kg/min)	88 to 160	125
Velocity (m/s)	4.2 to 7.5	6
Shear Rate ( $s^{-1}$ )	1600 to 2800	2300
Reynolds Number	8,200 to 15,200	11,500 (initially)
Temperature (°C)	10	10
Initial Emulsion		
Viscosity	11 mPas @ 10°C	
Oil Concentration	45 wt%	

Observations : Figure D-2 shows the results from the friction factor-Reynolds number test, test number 1. These data lie within 4% of the calibration line for the Newtonian oil and agree well with the data from previous tests with different emulsion batches, Figure D-1. Figures D-3 and D-4 show the results from test number 2, the long term flow test. After progressing smoothly for more than 1700 kilometers, friction factors decrease rapidly followed by an increase and second more stable period, Figure D-3. This second more stable period is accompanied by an increase in the emulsion temperature of 10°C, despite constant cooling water temperature and flow rate. At this time it was no longer possible to measure the viscosity of samples taken from the system as the shear stress readings at constant shear rate were erratic. Table D-4 shows the steady decrease in emulsion viscosity and oil concentration for samples taken during the test. The test was terminated when the system variables were no longer changing.

Time/Distance Travelled : 85 h/ 1840 km

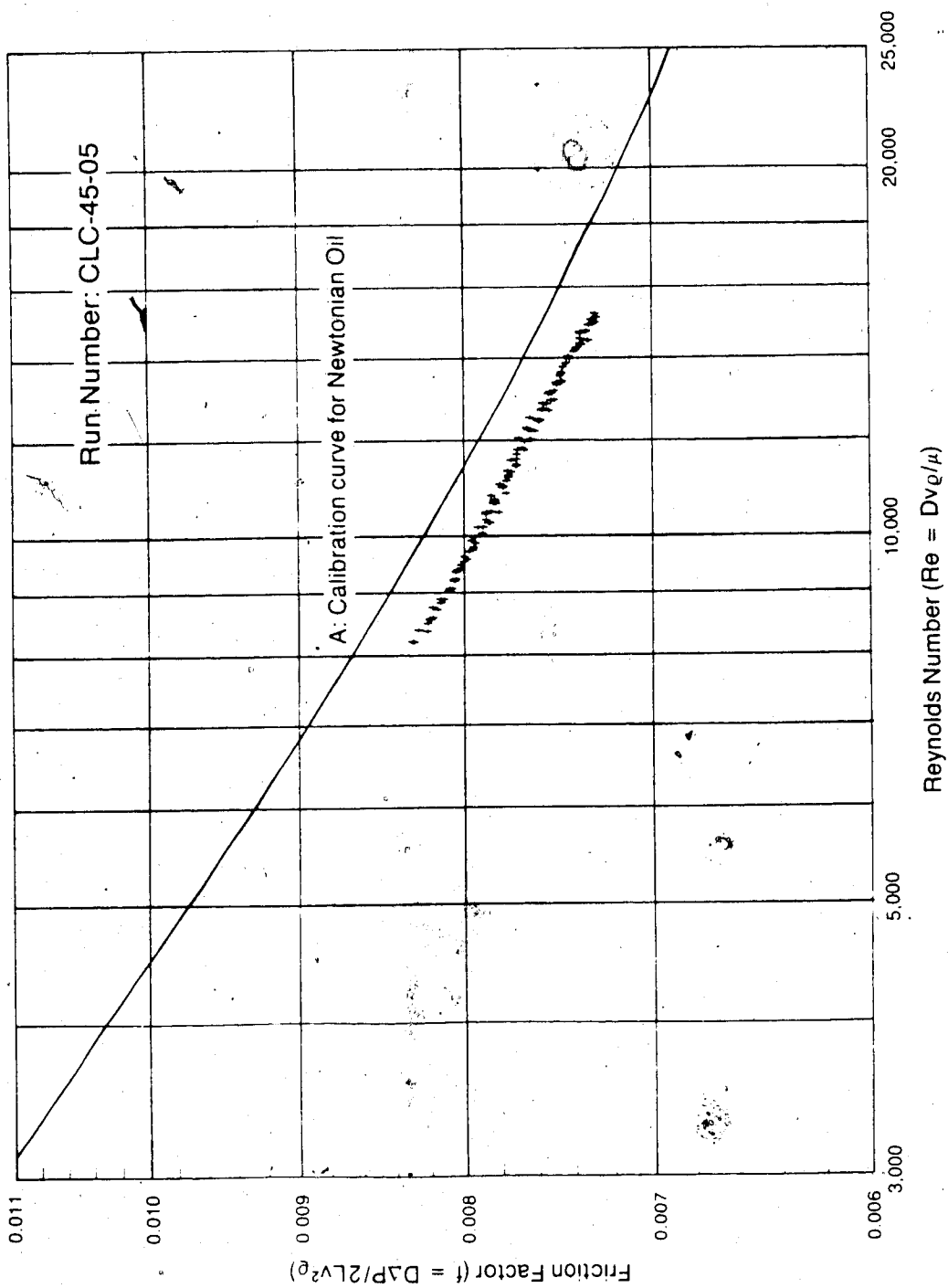
Instrument Calibration :

Flow Meter 0 to 400 kg/min  
 Pressure Transducers 0 to 150 kPa

Table D-4 : Oil concentration and emulsion viscosity as functions of real time and distance travelled for the long term test with emulsion CLC-45-05.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1	0	24:00	0.0	0	45	11.0 @ 10.0
2	1	08:00	8.0	173	45	10.5 @ 9.9
3	1	11:30	11.5	248	45	10.6 @ 9.8
4	1	15:30	15.5	335	44	10.1 @ 9.7
5	1	21:00	21.0	454	44	10.3 @ 9.8
6	2	09:00	33.0	713	44	10.5 @ 9.6
7	2	13:00	44.0	799	44	10.0 @ 9.2
8	2	21:30	55.5	983	45	9.8 @ 10.3
9	3	08:00	56.0	1210	41	8.0 @ 9.3
10	3	15:30	63.5	1372	40	7.8 @ 9.9
11	3	21:00	69.0	1490	41	7.9 @ 9.8
12	4	09:00	81.0	1750	40	N/A
13	4	16:00	88.0	1900	42	N/A
14	5	16:30	112.5	2430	37	N/A
15	5	24:00	120.0	2590	37	N/A
16	6	17:30	137.5	2970	37	N/A
17			175.0	3780	38	N/A

N/A = not available



**Figure D-2:** The variation of friction factor with Reynolds number for CLC-45-05.

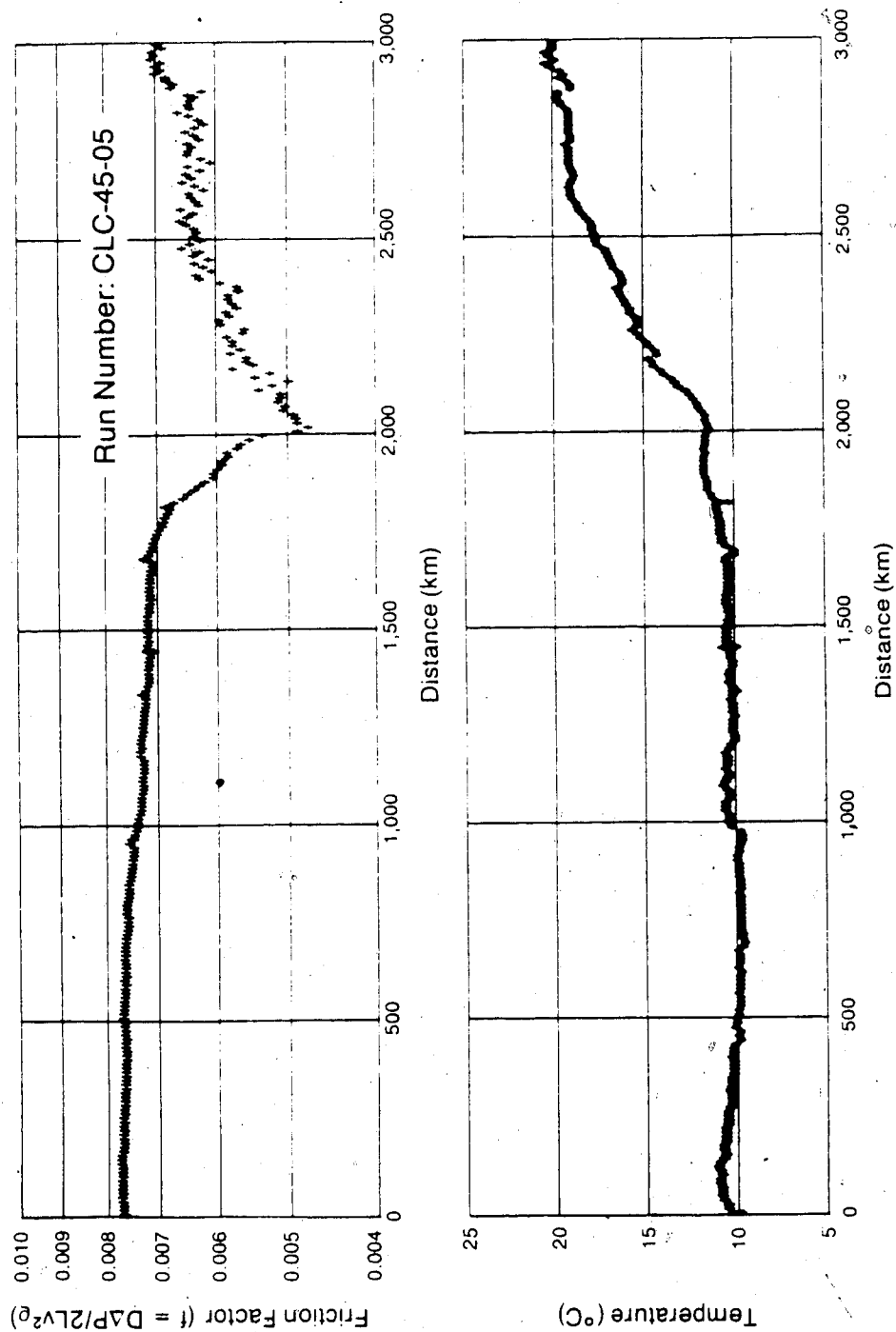


Figure D-3: The variation of friction factor and temperature with distance travelled for CLC-45-05.

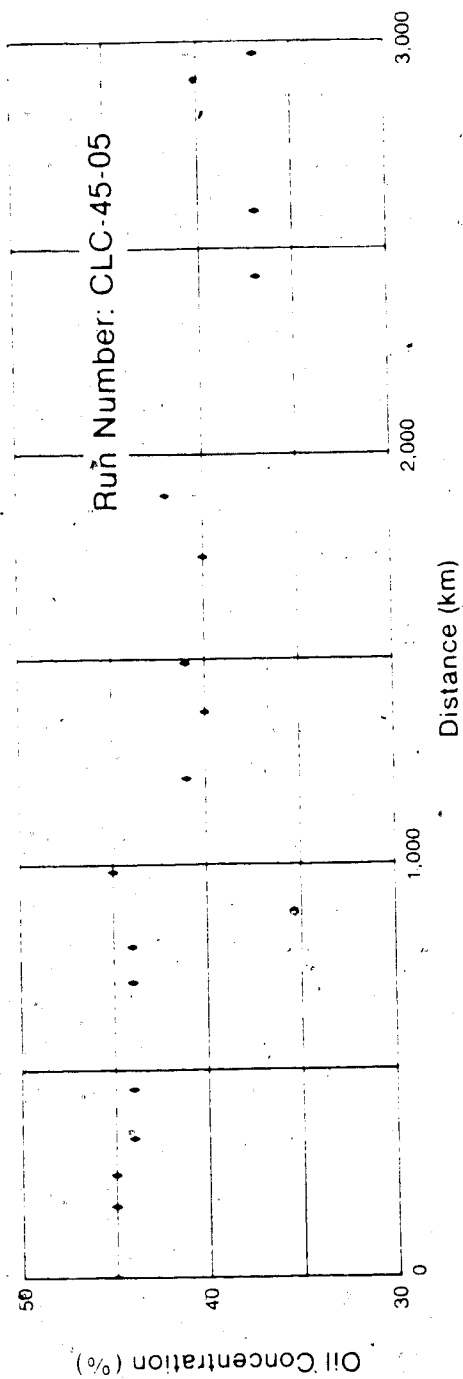


Figure D-4: The variation of oil concentration with distance travelled for CLC-45-05.

Emulsion : CLC-45-06

Description : The tests performed with this emulsion were the first laminar flow rate tests attempted. The desired Reynolds number was 1500. The effect of changing the flow regime on the distance the emulsion would remain stable was of interest. Decreasing the flow rate also decreases the shear rate.

Table D-5: Conditions for tests with emulsion CLC-45-06.

	Test No. 1	Test No. 2
Flow Rate (kg/min)	5.8 to 38	14
Velocity (m/s)	.27 to 1.8	.67
Shear Rate ( $s^{-1}$ )	100 to 680	250
Reynolds Number	570 to 3700	1500
Temperature ( $^{\circ}C$ )	14 to 15	12
Initial Emulsion		
Viscosity	11 mPas @ $10.0^{\circ}C$	
Oil Concentration	42 wt%	

Observations : Figure D-5 shows that the data from test number 1 are extremely scattered and not acceptable for comparison with the calibration line  $f=16/Re$ . Flow rate and discharge pressure from the pump were not steady enough to obtain good flow data during this test. Figures D-6, D-7 and D-8 show the results from the long term flow test. The friction factor data are very scattered for this test as well. The test was terminated after 320 kilometers (270 hours) because the 1/2 H.P. motor turning the pump began to overheat and the desired flow rate could not be maintained. Table D-6 shows the change in emulsion viscosity and oil concentration for samples taken during the test.

Instrument Calibration :

Flow Meter	0 to 45 kg/min
Pressure Transducers	0 to 10 kPa



Table D-6: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-45-06.

SAMPLE NO.	DAY	TIME	CUM.HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1	0	16:30	0.0	0	42	10.4 @ 14.2
2	1	09:00	16.5	39	43	10.0 @ 12.7
3	1	13:30	21.0	50	42	10.8 @ 12.2
4	1	16:00	23.5	56	42	10.0 @ 12.9
5	2	09:00	40.5	96	41	10.3 @ 11.6
6	2	13:00	45.0	107	41	11.5 @ 12.4
7	2	16:00	47.5	113	42	10.5 @ 12.4
8	3	09:00	64.5	153	39	9.5 @ 11.7
9	3	14:30	70.0	166	41	9.0 @ 11.9
10	4	15:30	95.5	227	38	8.6 @ 11.6
11	4	22:00	102.0	242	36	8.5 @ 11.3
12	5	09:30	113.5	270	34	N/A
13 SAMPLE TAKEN FROM UNCIRCULATED BUCKET					40	

N/A = not available

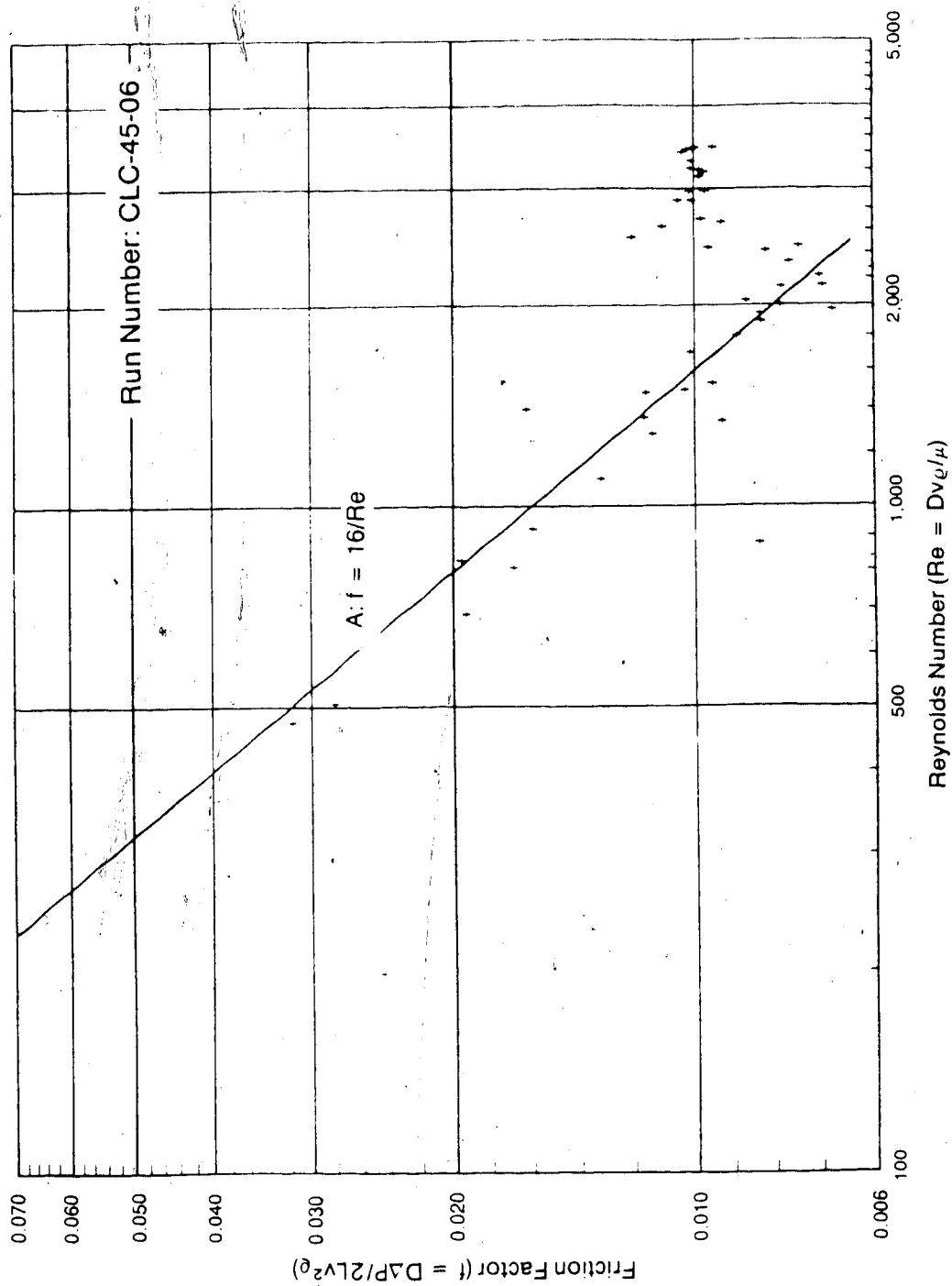


Figure D-5: The variation of friction factor with Reynolds number for CLC-45-06.

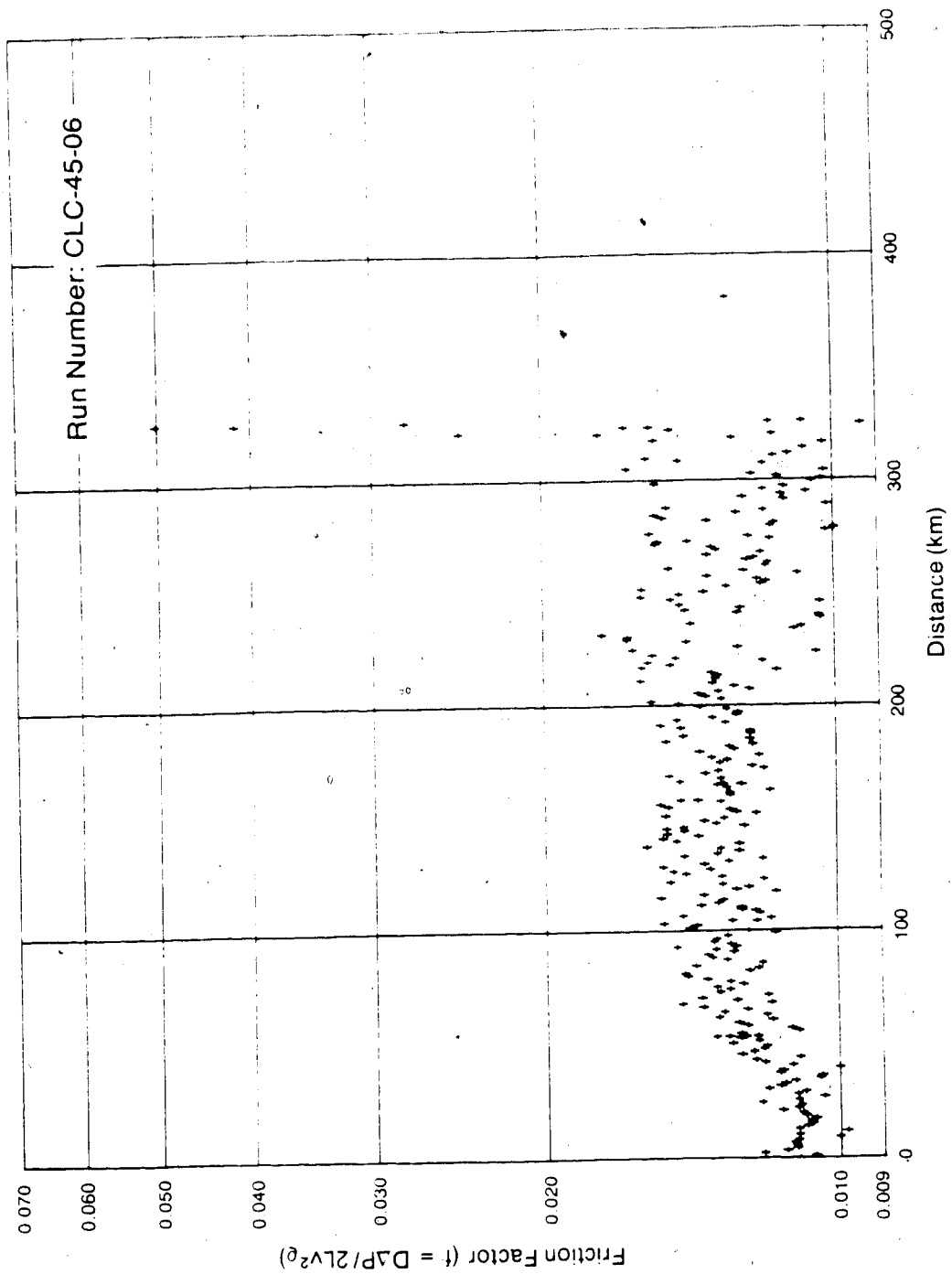


Figure D-6: The variation of friction factor with distance travelled for CLC-45-06.

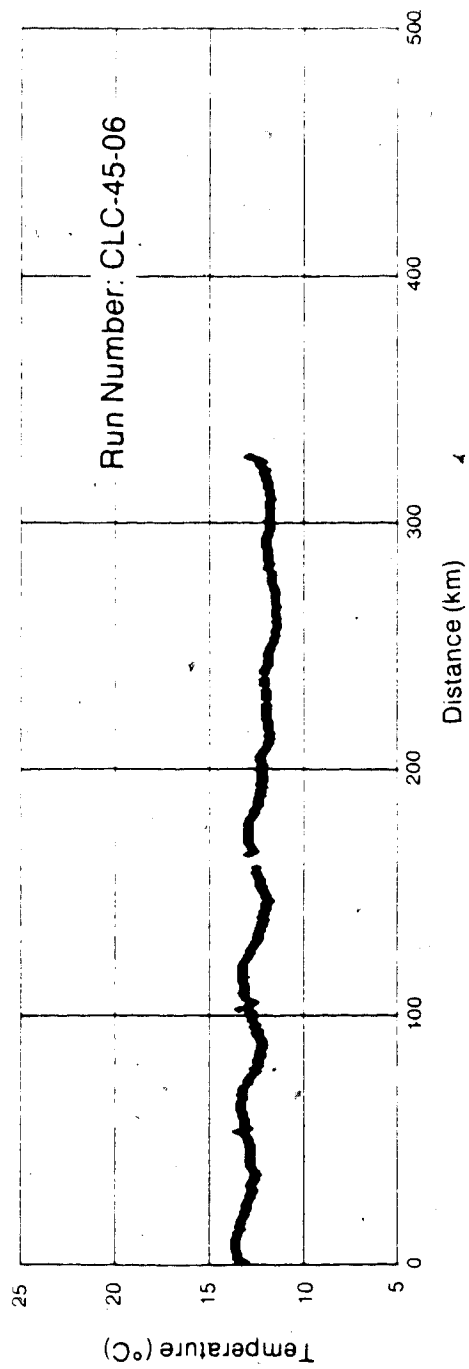


Figure D-7: The variation of temperature with distance travelled for CLC-45-06.

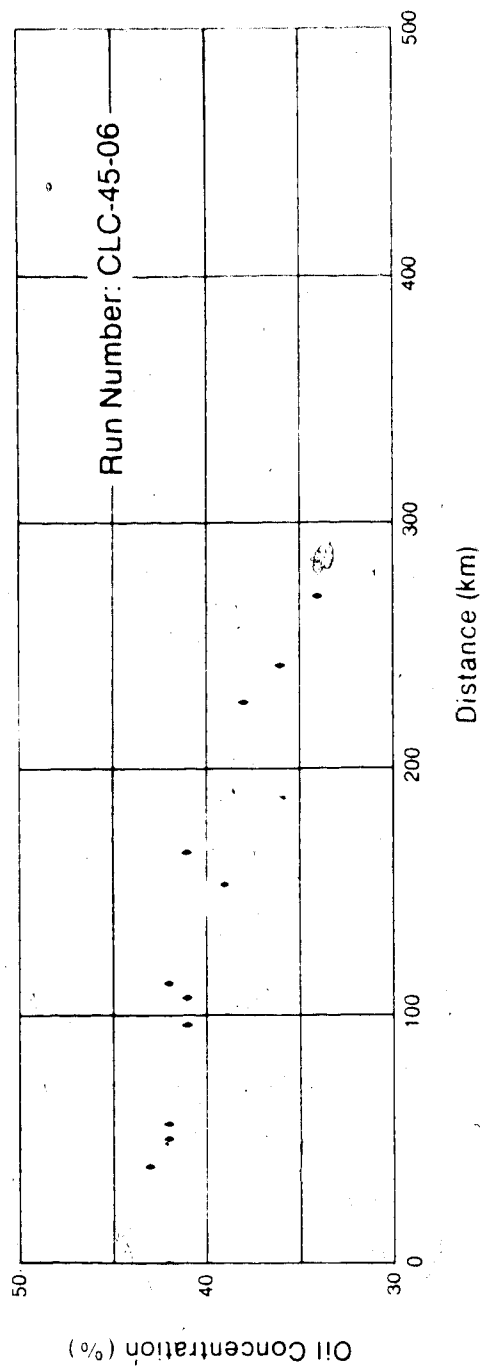


Figure D-8: The variation of oil concentration with distance travelled for CLC-45-06.

Emulsion : CLC-45-07

Description : These tests were repetitions of the tests performed with emulsion CLC-45-06. Steady flow rates and discharge pressures from the pump were achieved by adding a second accumulator to the pipeline test loop. The 1/2 horsepower motor was replaced with a 1 horsepower motor to provide the increased power required at the end of a long term test. Dampening on pressure transducers and the flow meter were increased by increasing the time constant on both to about 1.5 seconds.

Table D-7: Conditions for tests with emulsion CLC-45-07.

	Test No.1	Test No.2
Flow Rate (kg/min)	4.7 TO 36	14
Velocity (m/s)	.22 TO 1.7	.67
Shear Rate (s <sup>-1</sup> )	83 TO 640	250
Reynolds Number	560 TO 4300	1700
Temperature (°C)	14 to 15	13 to 15
Initial Emulsion		
Viscosity	8.5 mPas @ 14.1°C	
Bitumen Concentration	40 wt%	

Observations : Figure D-9 shows the friction factor-Reynolds number data lie within 12% of the calibration line  $f=16/Re$  and that the transition from the laminar to the turbulent flow regimes occurs at  $Re=2200$ . Figures D-10, D-11 and D-12 show the data from the long term flow test. The abrupt end to the test after 190 kilometers is the result of a reduction gear box failure. The emulsion temperature during the test, Figure D-11, is higher than the desired 10-12°C because the cooling water temperature had increased over that in previous tests. Figure D-11 shows that the friction factor behaviour during this test follows the same trend as during the long term test with emulsion CLC-45-06, Figure D-6.

Instrument Calibration :

Flow Meter	0 to 45 kg/min
Pressure Transducers	0 to 10 kPa

Table D-8: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-45-07.

NO.	TIME		DISTANCE		OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
	DAY	TIME	CUM. HRS	KM		
1 (COMPOSITE)			0	0.0	0	42 N/A
2	0	17:30	0.0	0	40	8.5 @ 14.1
3	1	09:30	17.0	41	40	8.5 @ 13.4
4	1	16:30	23.0	55	40	8.1 @ 14.4
5	2	10:00	40.5	98	39	7.6 @ 13.7
6	2	16:00	46.5	112	38	N/A
7	3	14:00	68.5	165	36	N/A

N/A = not available

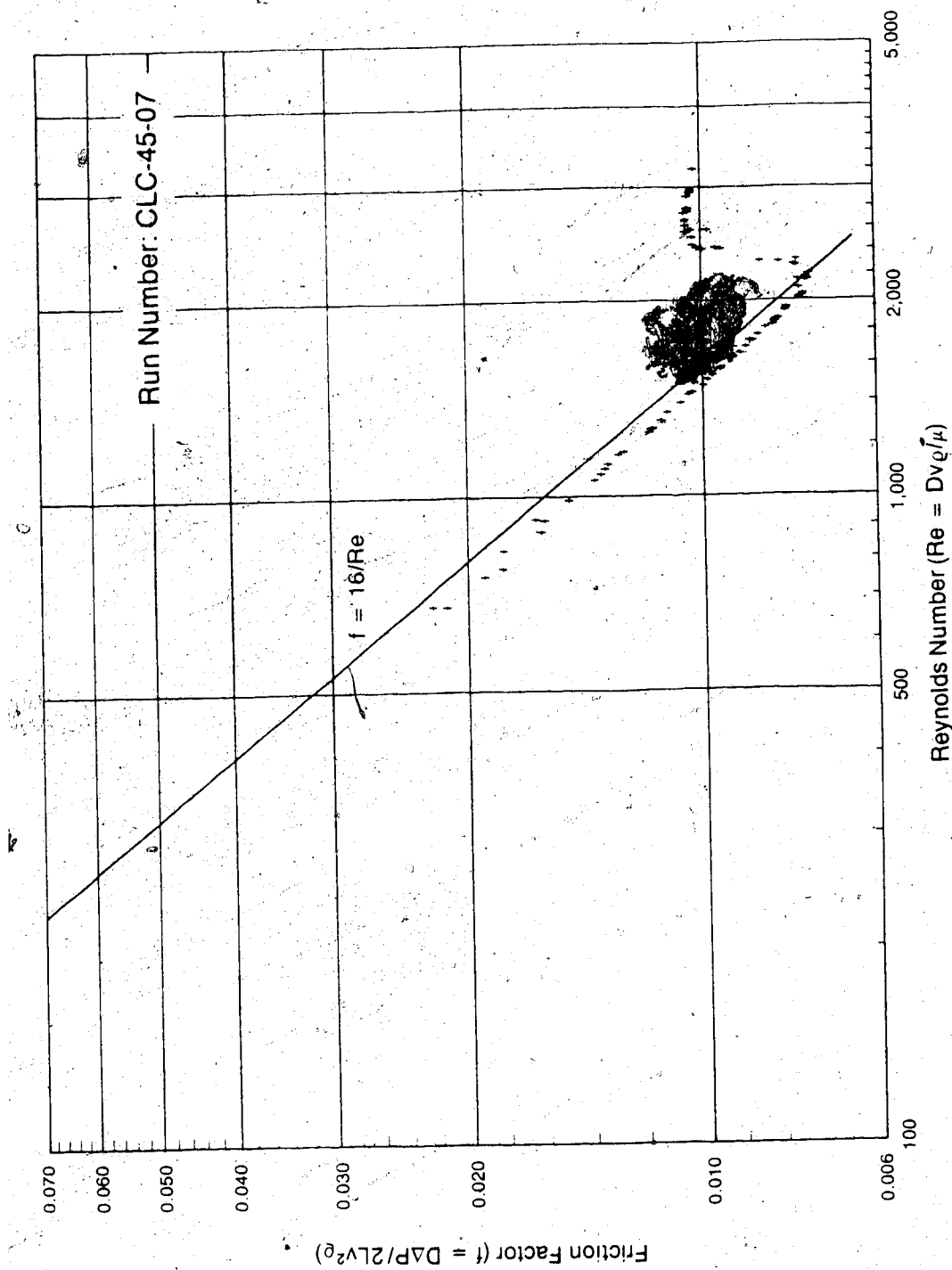


Figure D-9: The variation of friction factor with Reynolds number for CLC-45-07.



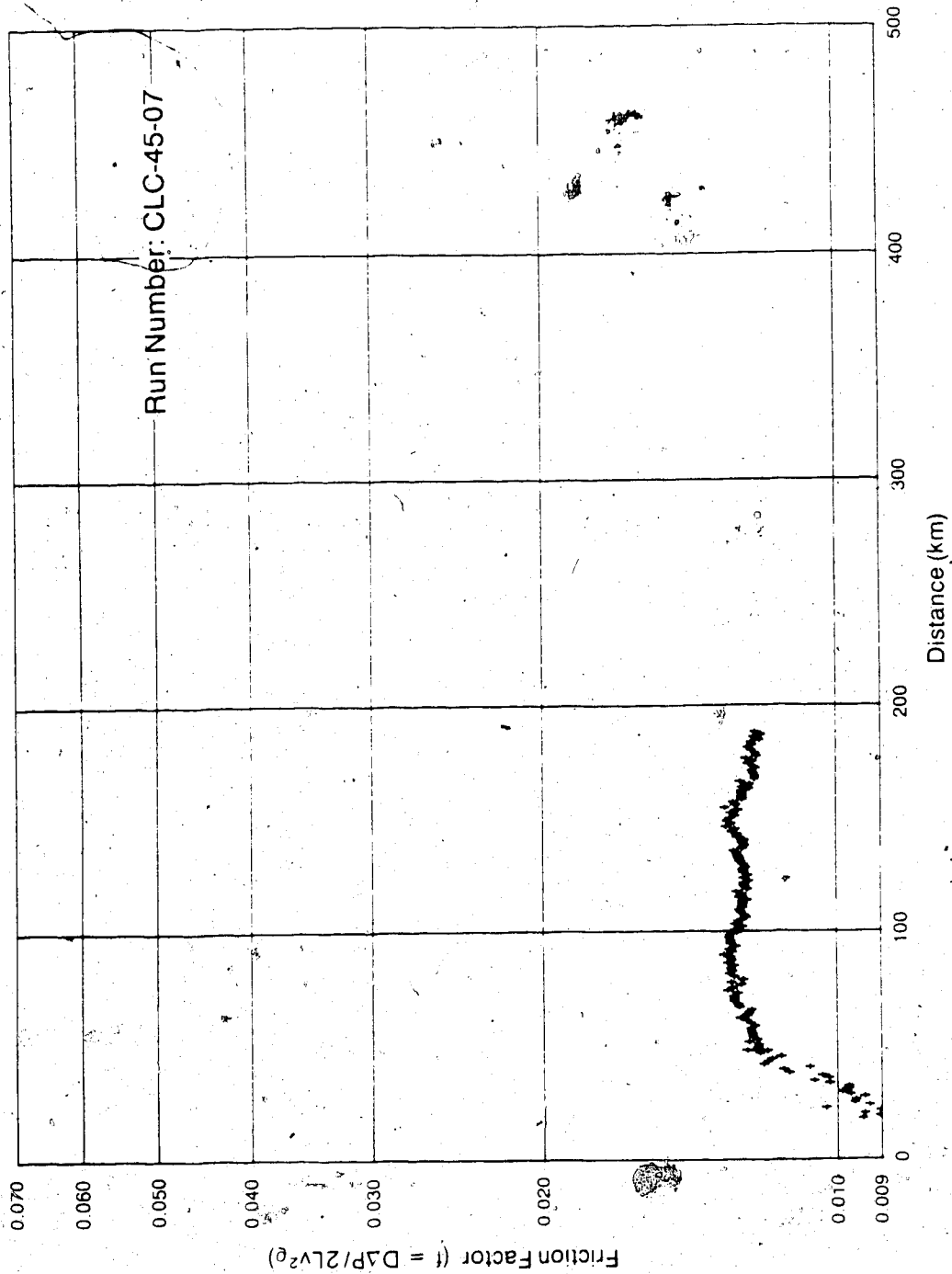


Figure D-10: The variation of friction factor with distance travelled for CLC-45-07.

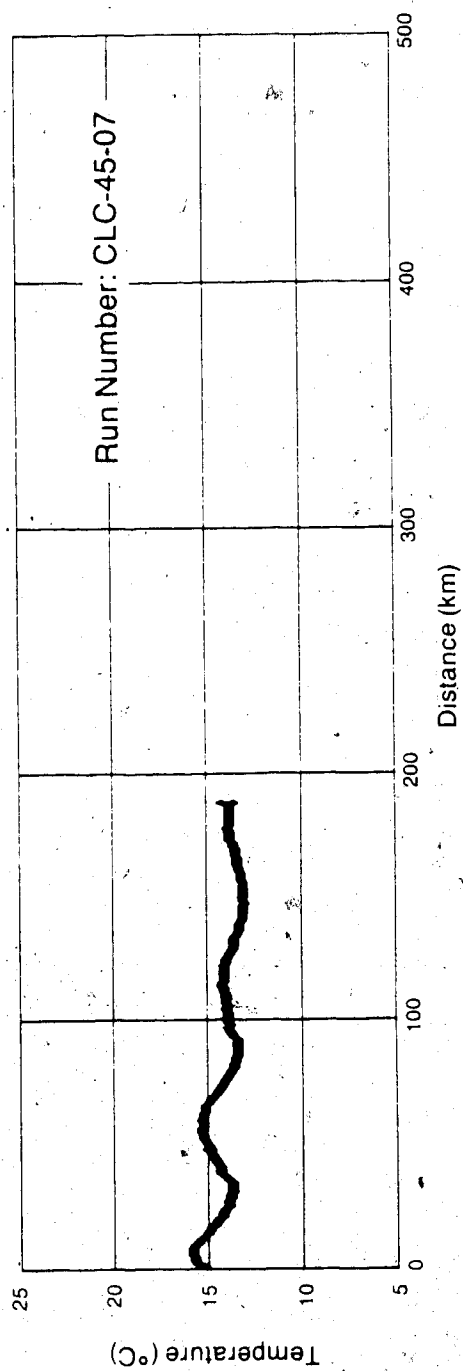


Figure D-11: The variation of temperature with distance travelled for CLC-45-07.

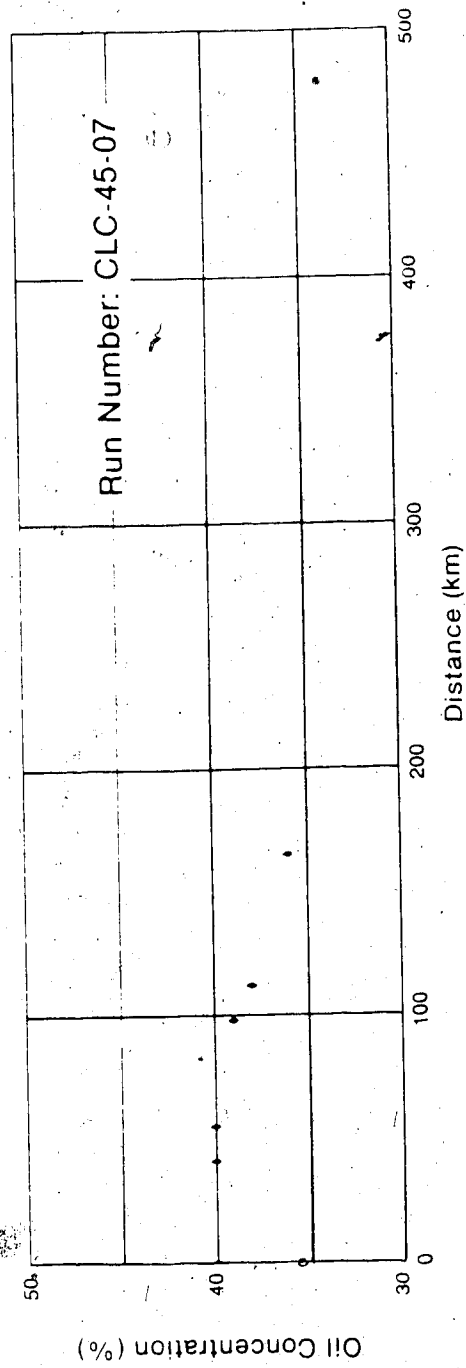


Figure D-12: The variation of oil concentration with distance travelled for CLC-45-07.

Emulsion : CLC-45-08

Description : These two tests were repetitions of the tests performed with emulsions CLC-45-06 and CLC-45-07. The broken gearbox was replaced with a larger unit and a refrigeration system was added to provide cooling water at a lower temperature.

Table D-9: Conditions for tests with emulsion CLC-45-08.

	Test No.1	Test No.2
Flow Rate (kg/min)	7.4 to 27	14
Velocity (m/s)	.35 to 1.3	.67
Shear Rate ( $s^{-1}$ )	130 to 450	250
Reynolds Number	740 to 2700	1500
Temperature ( $^{\circ}C$ )	12 to 14	12
Initial Emulsion		
Viscosity	10.4 mPas @ 12.1 $^{\circ}C$	
Oil Concentration	43 wt%	

Observations : Figure D-13 shows that the friction factor-Reynolds number data lie within 10% of the calibration line  $f=16/Re$  and that the transition from laminar to turbulent flow occurs at  $Re=2200$ . Figures D-14, D-15 and D-16 show the results from the long term flow test. The friction factor behaviour, Figure D-14, corresponds well with that observed in the two previous tests, Figure D-6 and Figure D-11. The high values for friction factor observed near the end of the test are the result of reduced flow rates that occurred when the drive belt to the pump slipped. The simultaneous increase in temperature of 5 $^{\circ}C$ , Figure D-15, and current drawn by the D.C. motor to turn the pump at constant speed, Figure D-16, as well as the decrease in measured pressure drop, indicated the end of the test. Table D-10 shows the decrease in emulsion viscosity and oil concentration for samples taken during the test.

Time/Distance Travelled : 140 h / 340 km

Instrument Calibration :

Flow Meter                      0 to 45 kg/min  
 Pressure Transducers        0 to 10 kPa

Table D-10: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-45-08.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1a	0		0.0	0	44	
2	0	17:30	0.0	0	43	10.4 @ 12.1
3	1	09:00	15.5	37	41	10.1 @ 12.0
4	1	22:00	28.5	68	42	10.1 @ 12.7
5	2	10:00	40.5	96	41	9.9 @ 11.5
6	2	22:00	52.5	125	40	9.2 @ 12.7
7	3	09:30	64.0	152	39	9.0 @ 11.6
8	3	22:00	76.5	182	40	9.7 @ 12.5
9	4	11:00	89.5	213	39	9.0 @ 11.7
10	4	16:00	94.5	225	39	9.4 @ 11.8
11	5	09:30	112.0	266	39	8.5 @ 11.8
12	5	16:30	119.0	283	37	7.5 @ 12.6
13	6	08:30	135.0	321	37	7.4 @ 12.6
14	6	17:00	143.5	341	36	7.5 @ 14.3
15						
COMPOSITE FROM BUCKET					41	

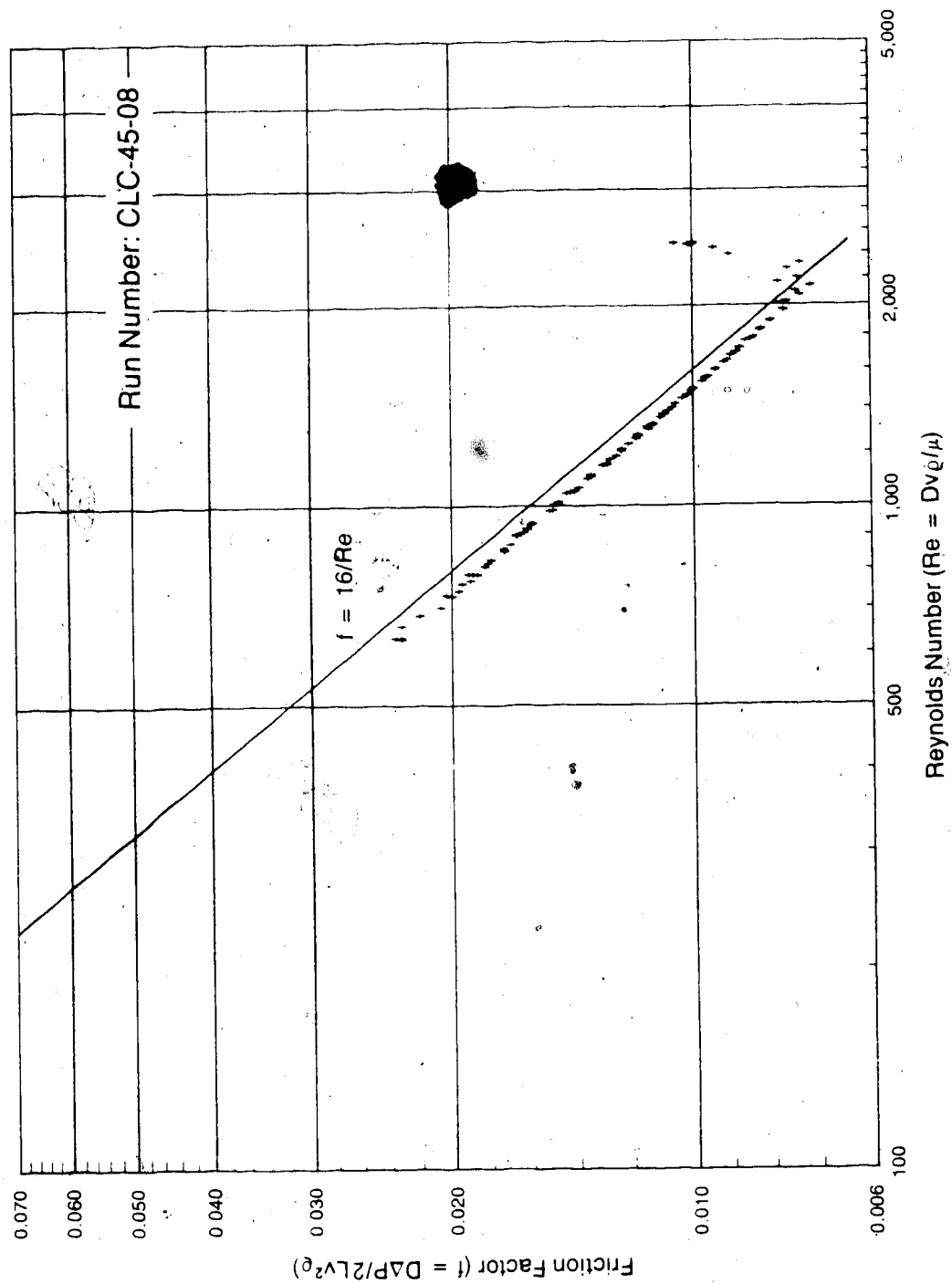


Figure D-13: The variation of friction factor with Reynolds number for CLC-45-08.

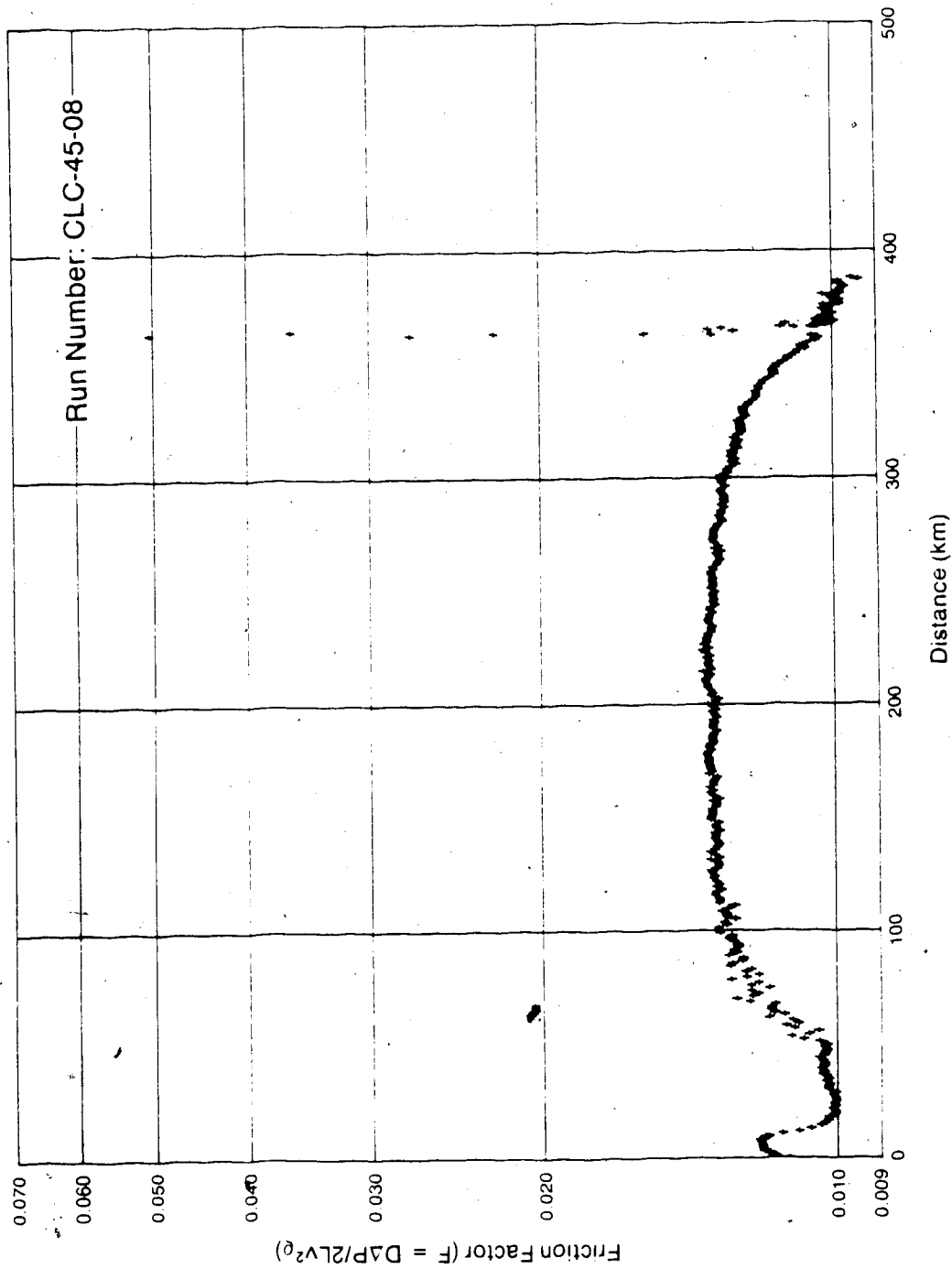


Figure D-14: The variation of friction factor with distance travelled for CLC-45-08.

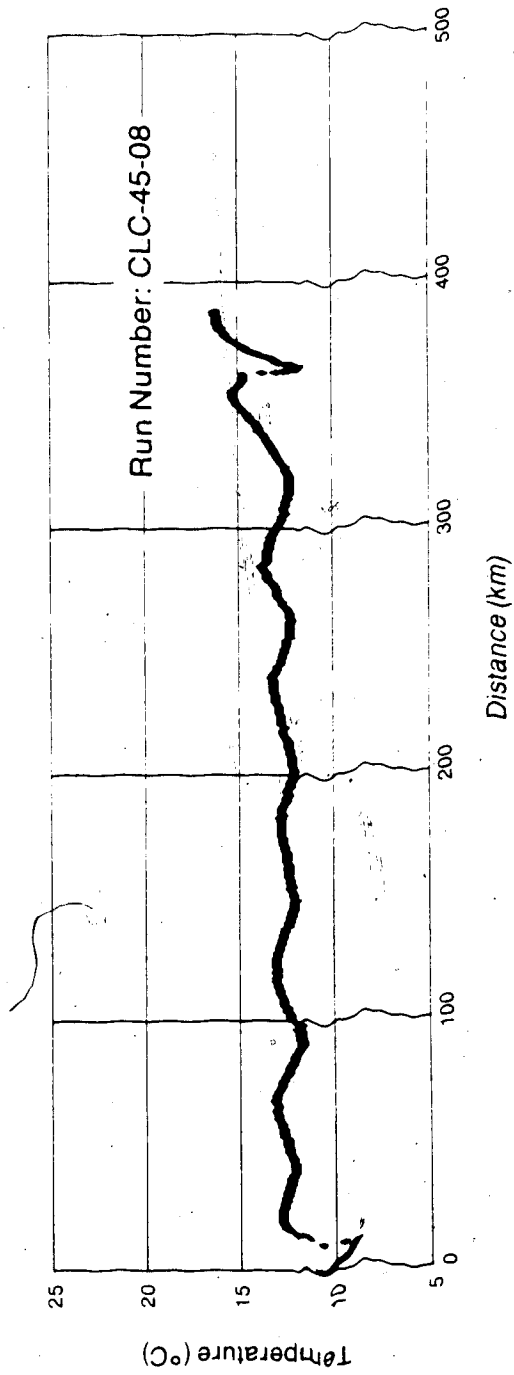


Figure D-15: The variation of temperature with distance travelled for CLC-45-08.



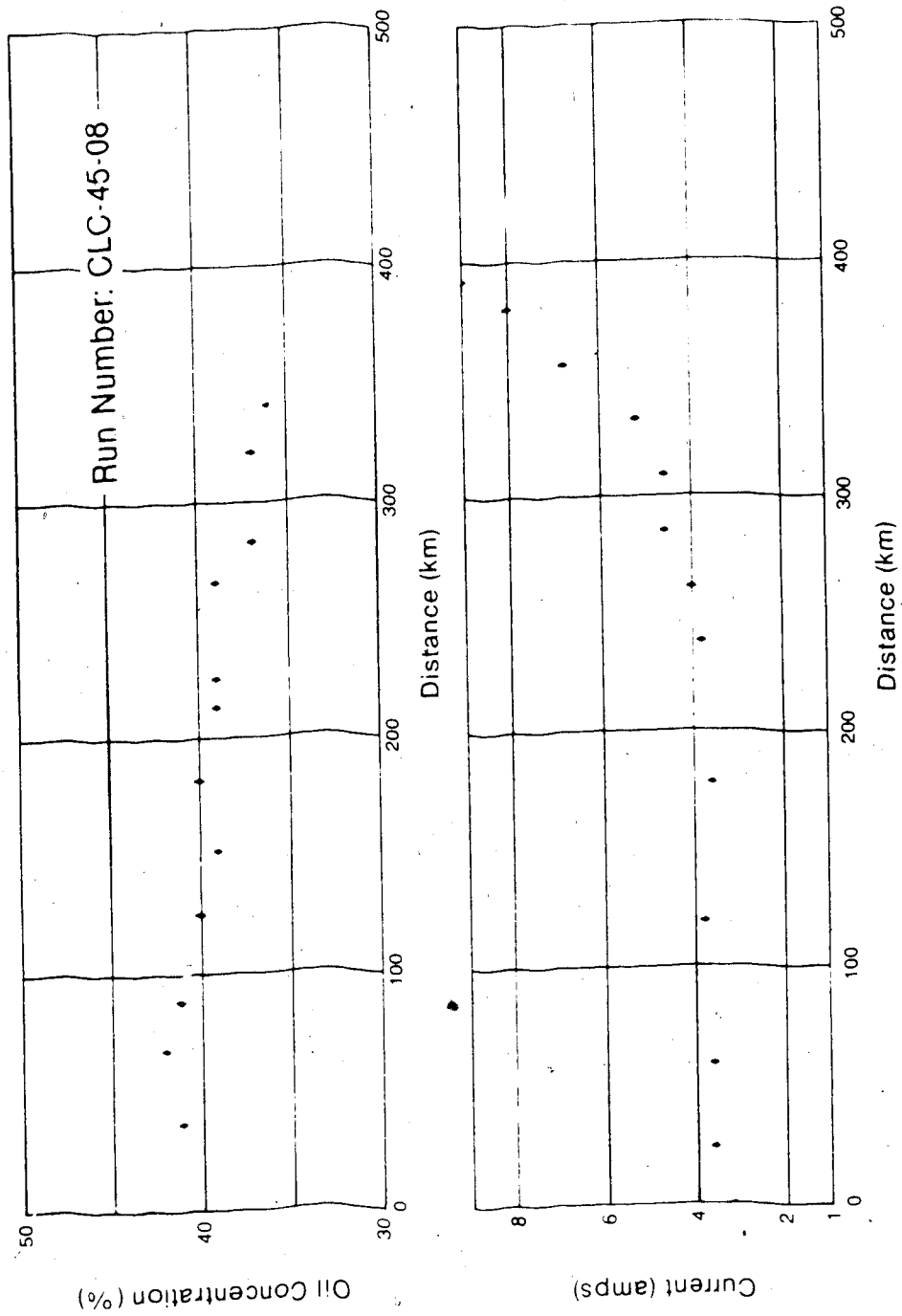


Figure D-16: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-45-08.

Emulsion : CLC-45-09

Description : These tests were performed at intermediate values of Reynolds number.

Table D-11: Conditions for tests with emulsion CLC-45-09.

	Test No.1	Test No.2
Flow Rate (kg/min)	13 to 41	40
Velocity (m/s)	.6 to 2.0	1.85
Shear Rate ( $s^{-1}$ )	230 to 740	720
Reynolds Number	1500 to 4700	4500
Temperature ( $^{\circ}C$ )	12 to 14	12
Initial Emulsion		
Viscosity	9.1 mPas @ 12.1 $^{\circ}C$	
Oil Concentration	39 wt%	

Observations : Figure D-17 shows that the friction factor-Reynolds number data lie within 2% of the calibration line  $f=16/Re$  in the laminar flow regime and within 5% of the calibration curve for the Newtonian oil in the turbulent flow regime. The transition from laminar to turbulent flow occurs at  $Re=2200$ . Figures D-18 and D-19 show the results from the long term flow test. Friction factor decreased only 10% in the first 1600 kilometers. There is a sharp drop in the friction factor at about 1800 kilometers followed by a recovery to nearly normal values. This recovery is accompanied by a 15 $^{\circ}C$  increase in temperature, Figure D-18, and a sharp increase in current drawn by the D.C. motor, Figure D-19. Temperature fluctuations in the system initially were due to a faulty controller in the refrigeration system. Table D-12 shows the decrease in emulsion viscosity and oil concentration for samples taken during the test.

Time/Distance Travelled : 270 h / 1850 km

Instrument Calibration :

Flow Meter	0 to 45 kg/min
Pressure Transducers	0 to 15 kPa

Table D-12: Oil concentration and emulsion viscosity as functions of real time and distance traveled during the long term test with emulsion CLC-45-09.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1 (COMPOSITE)				0	41	
2	0	14:30	0	0	39	9.1 @ 12.1
3	0	21:30	7.0	47	39	8.8 @ 14.1
4	1	08:00	17.5	117	38	8.6 @ 12.5
5	1	16:00	25.5	170	39	8.8 @ 10.8
6	2	10:00	43.5	290	38	8.2 @ 11.9
7	2	17:00	50.5	336	38	8.2 @ 11.6
8	3	10:30	68.0	453	37	8.2 @ 10.3
9	3	21:00	78.5	523	37	8.2 @ 10.3
10	4	11:00	92.5	616	36	7.2 @ 12.1
11	4	22:00	103.5	689	36	N/A
12	5	09:00	114.5	763	36	7.3 @ 12.8
13	5	16:30	122.0	813	36	N/A
14	6	09:00	138.5	922	36	12.1 @ 13.5
15	6	16:00	145.5	969	36	10.5 @ 13.7
16	7	09:30	163.0	1086	37	9.1 @ 13.7
17	8	09:00	186.5	1242	35	N/A
18	9	10:30	210.0	1399	33	N/A
19	9	17:00	216.5	1442	34	N/A
20	10	10:30	234.0	1558	33	7.0 @ 11.5
21	11	10:30	258.0	1718	33	N/A
22	12	10:30	282.0	1878	20	N/A
23	FROM BUCKET, NOT CIRCULATED				34	

N/A = not available

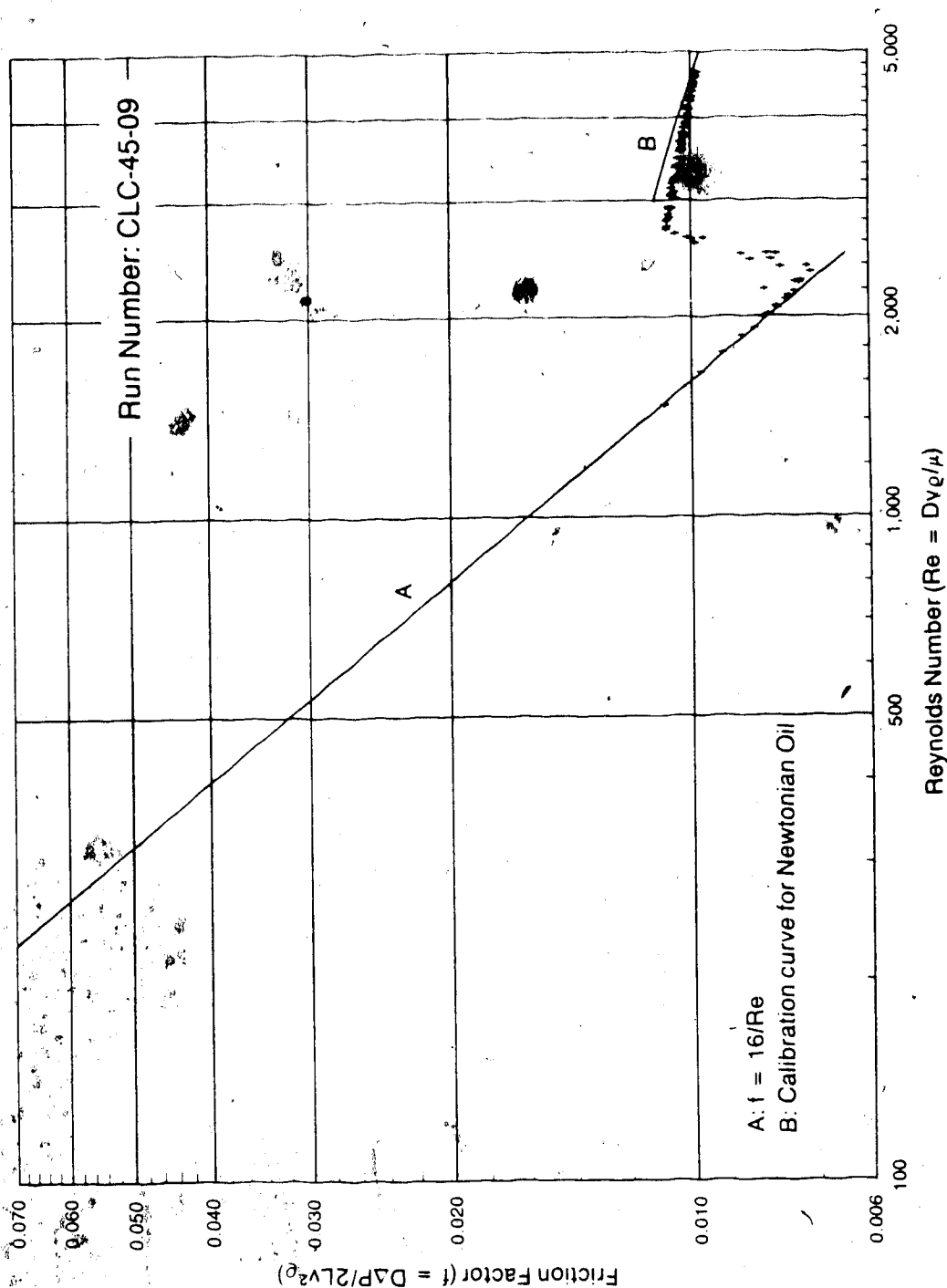


Figure D-17: The variation of friction factor with Reynolds number for CLC-45-09.

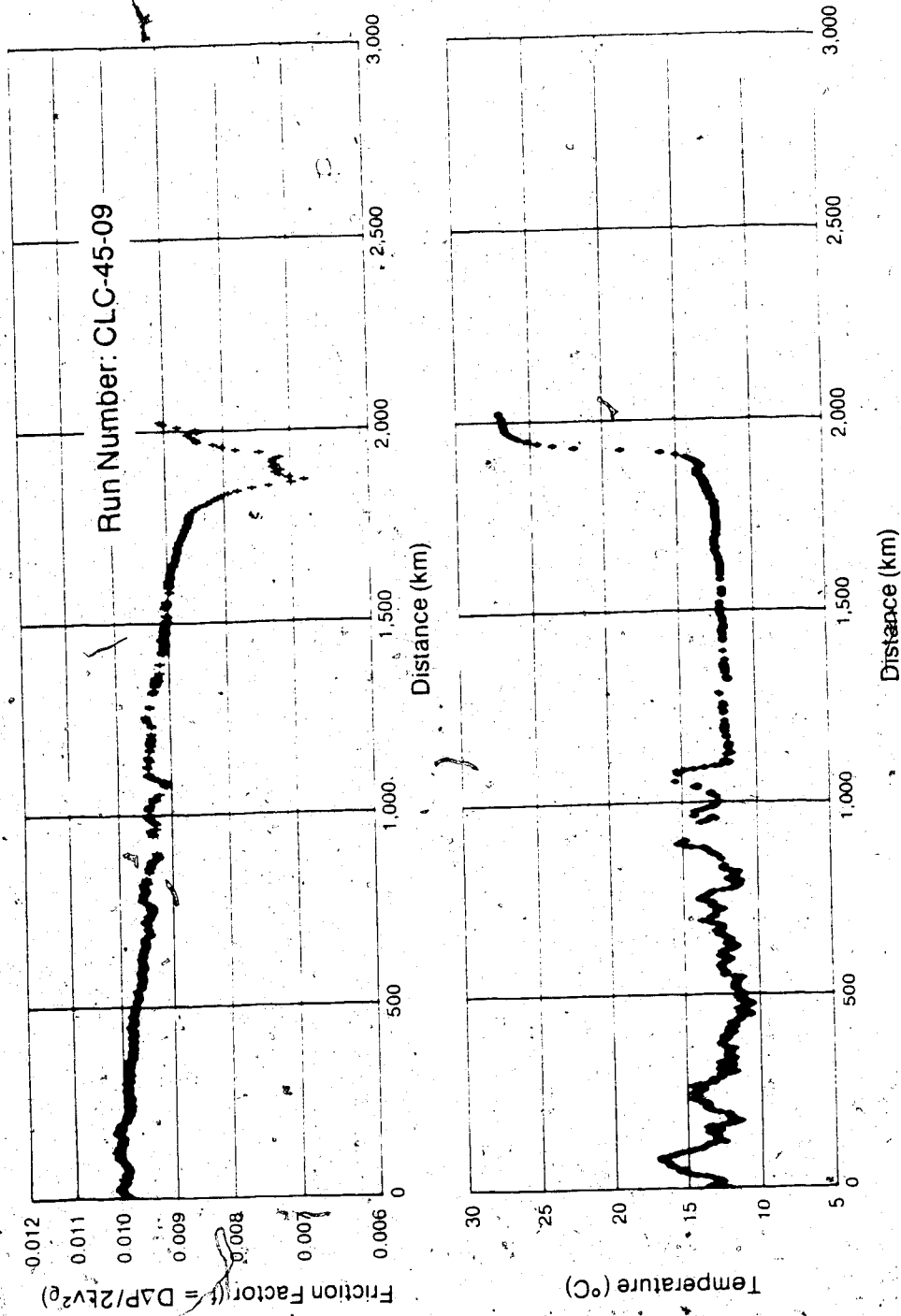


Figure D-18: The variation of friction factor and temperature with distance travelled for CLC-45-09.

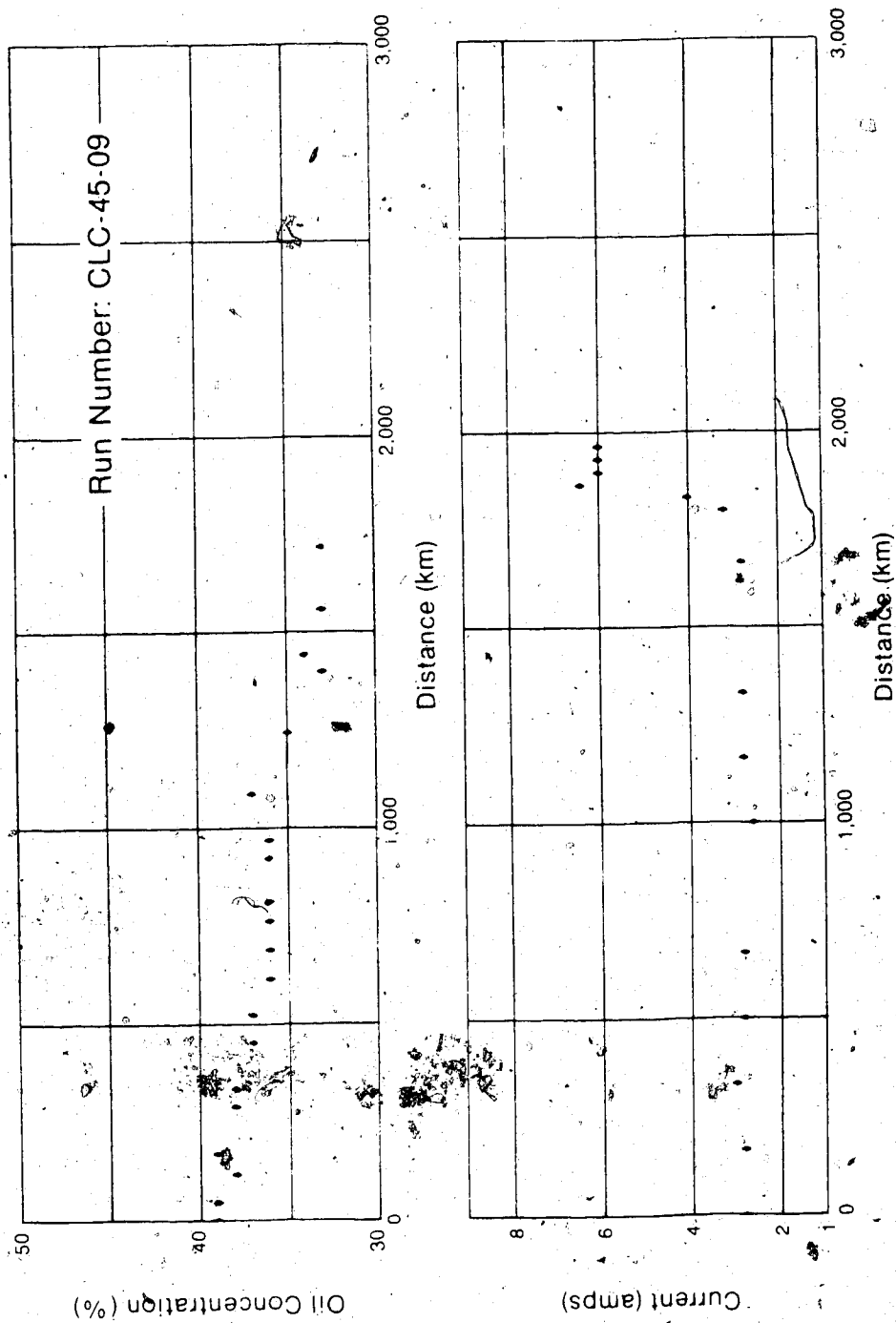


Figure D-19: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-45-09.

Emulsion : CLC-45-10

Description : These tests had the lowest flow rates for the 45% emulsions.

Table D-13: Conditions for tests with emulsion CLC-45-10.

	Test No.1	Test No.2
Flow Rate (kg/min)	5.3 to 12.6	7.4
Velocity (m/s)	.25 to .59	.35
Shear Rate ( $s^{-1}$ )	94 to 220	130
Reynolds Number	500 to 1200	650
Temperature ( $^{\circ}C$ )	11 to 13	12
Initial Emulsion		
Viscosity	10.8 mPas @ 12.6 $^{\circ}C$	
Oil Concentration	42 wt%	

Observations : Figure D-20 shows that the friction factor-Reynolds number data lie within 8% of the calibration line  $f=16/Re$ . Figures D-21, D-22 and D-23 show the results of the long term flow test. Friction factor decreases throughout the run becoming more scattered after a distance of 300 kilometers. After 350 kilometers, friction factors increase dramatically, Figure D-21, and temperature increases about 8 $^{\circ}C$ , Figure D-22. Current drawn by the D.C. motor increased from 1.5 to 3 amps, Figure D-23. The decrease in emulsion viscosity and oil concentration for samples taken from the system are shown in Table D-14. The pressure data from this test were taken from the Rosemount pressure transducers only as the data from the Viatran transducer were too scattered.

Time/Distance Travelled : 260 / 310 km

Instrument Calibration :

Flow Meter	0 to 45 kg/min
Pressure Transducer 1.	0 to 7 kPa (Rosemount)
2.	0 to 2 kPa (Rosemount)
3.	0 to 2 kPa (Viатran)

Table D-14: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-45-10.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1	COMPOSITE				43	
2	0	16:00	0.0	0	42	10.8 @ 12.6
3	1	09:00	17.0	21	42	10.8 @ 12.6
4	1	16:00	24.0	30	41	11.0 @ 11.0
5	2	09:00	41.0	52	40	10.2 @ 11.0
6	2	16:00	48.0	60	40	9.7 @ 11.2
7	3	09:00	65.0	82	39	9.4 @ 10.1
8	3	16:00	72.0	91	38	9.0 @ 10.6
9	5	11:00	115.0	145	35	7.4 @ 11.2
10	6	09:00	137.0	173	35	7.8 @ 10.5
11	6	16:00	144.0	181	35	8.7 @ 10.1
12	7	09:00	161.0	203	33	7.3 @ 11.5
13	7	16:00	168.0	212	30	5.8 @ 11.1
14	8	09:00	185.0	233	29	5.9 @ 10.2
15	8	16:00	192.0	242	28	5.6 @ 11.0
16	9	09:00	209.0	263	23	4.6 @ 10.6
17	9	16:00	216.0	272	21	3.7 @ 11.1
18	10	09:00	233.0	294	20	3.6 @ 10.4
19	10	16:00	240.0	302	21	3.7 @ 10.9
20	11	14:30	262.5	331	12	3.0 @ 10.8
21	12	19:00	291.0	367	12	3.6 @ 11.0



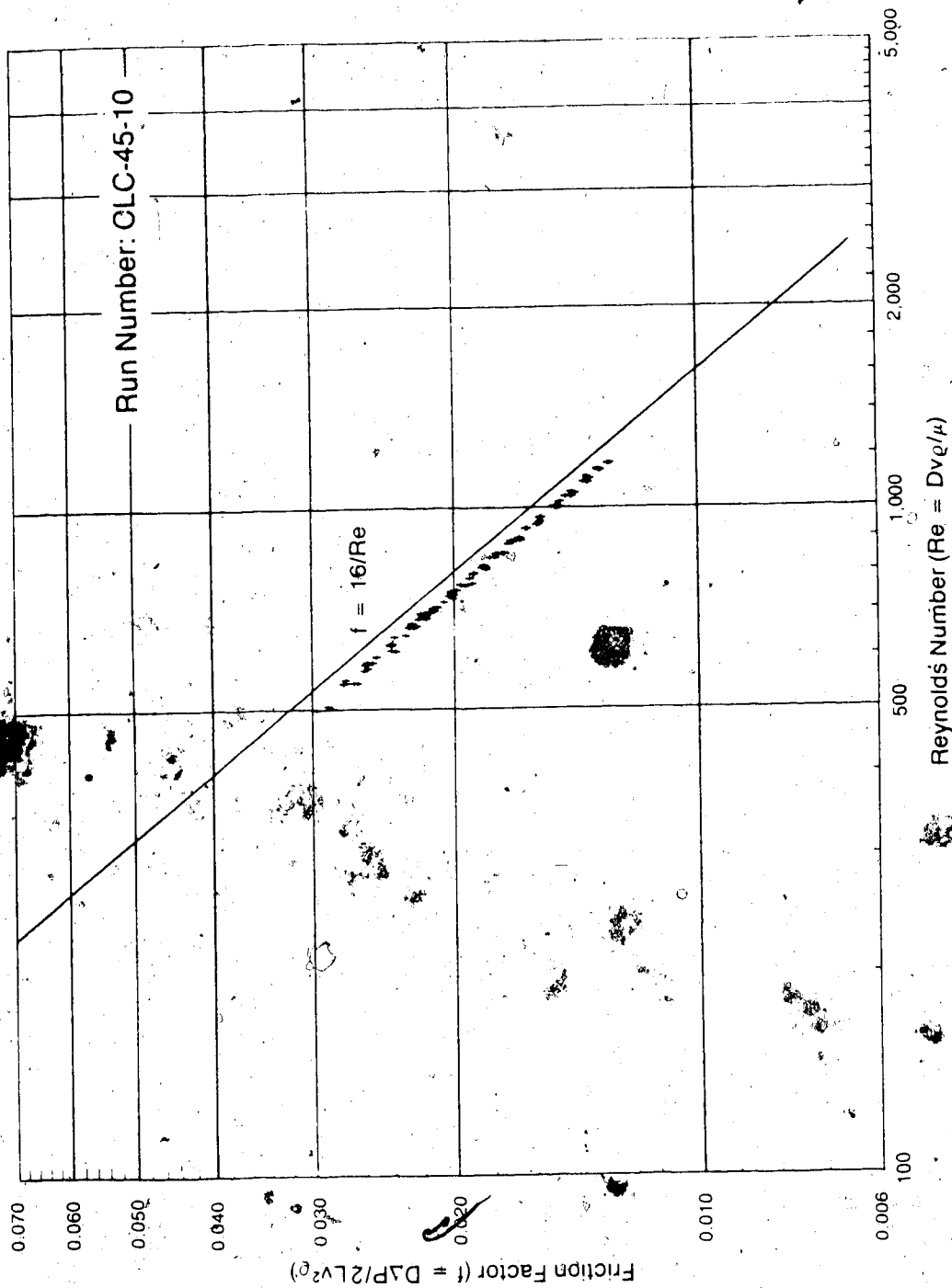


Figure D-20: The variation of friction factor with Reynolds number for CLC-45-10.

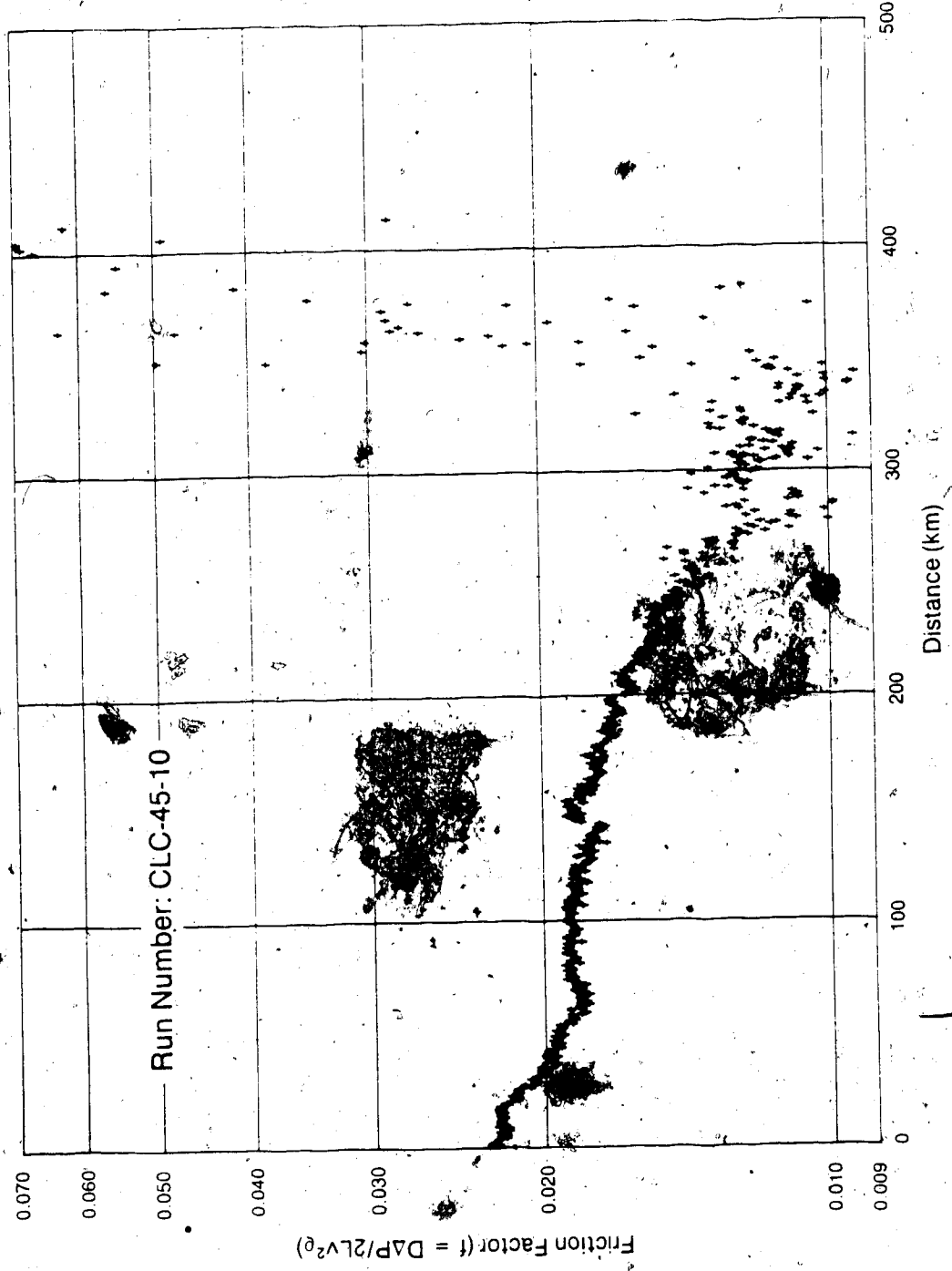


Figure D-21: The variation of friction factor with distance travelled for CLC-45-10.

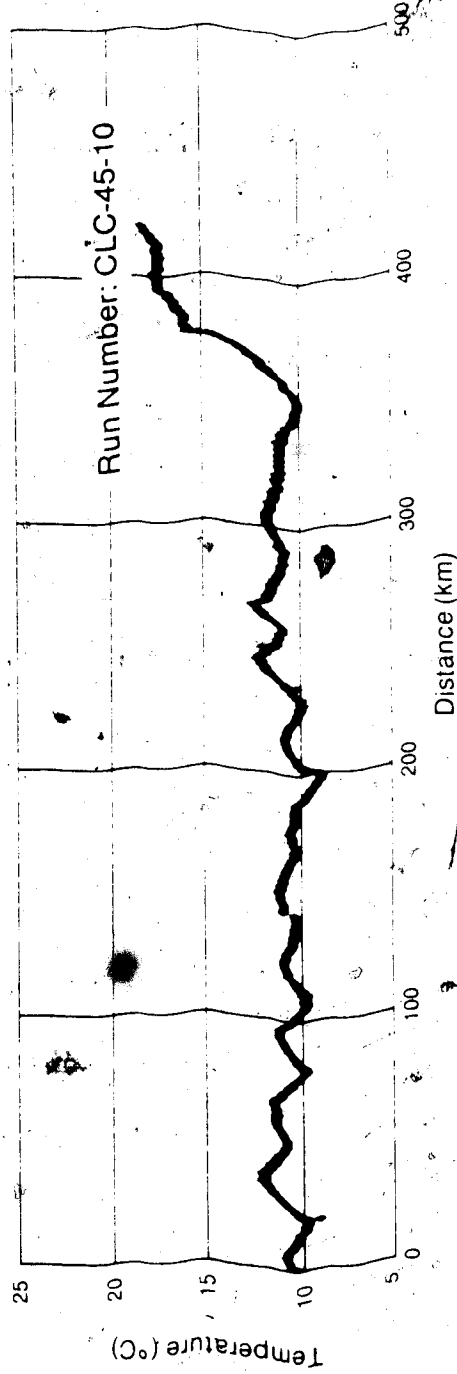


Figure D-22: The variation of temperature with distance travelled for CLC-45-10

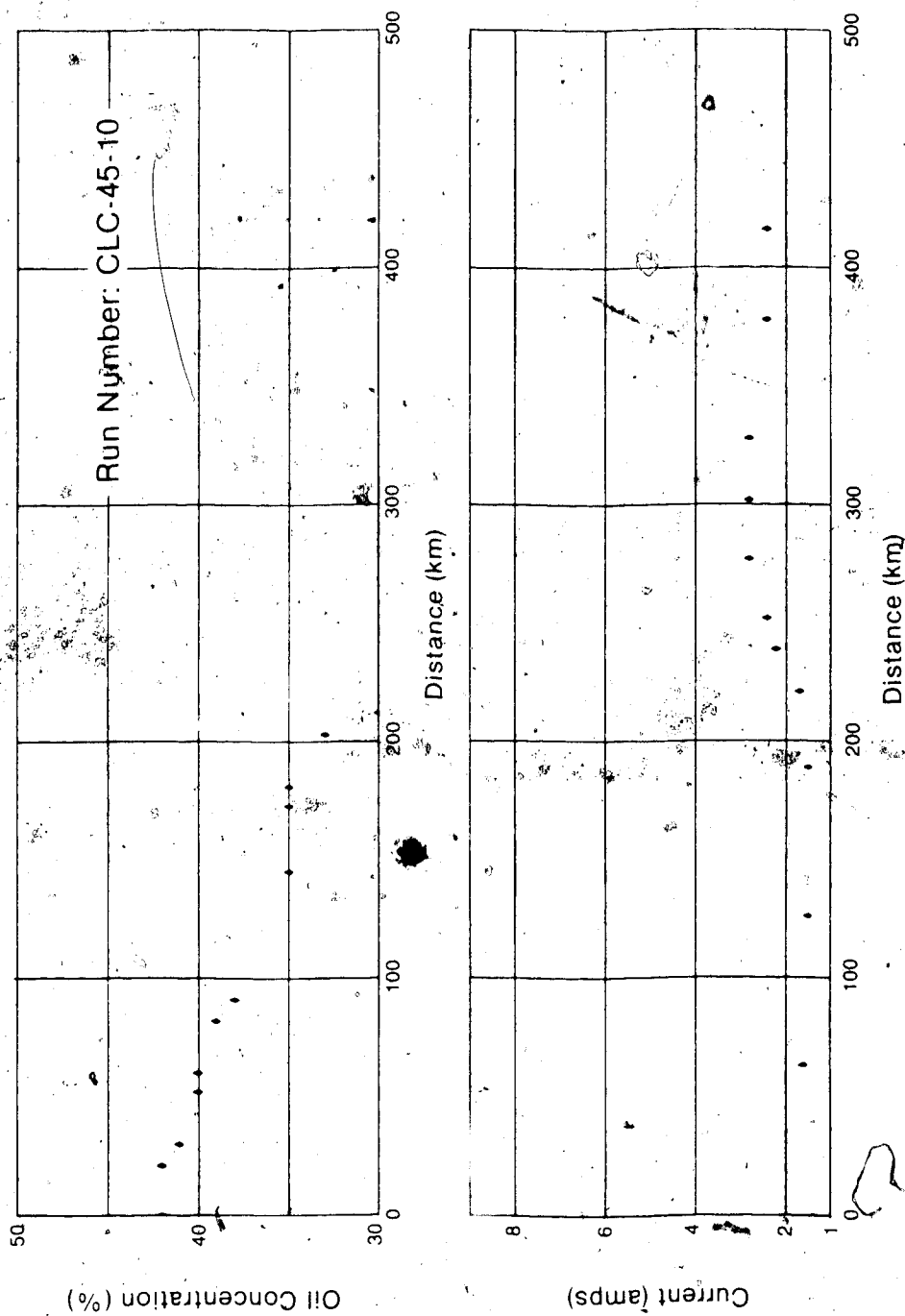


Figure D-23: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-45-10.

Emulsion : CLC-45-11

Description : These flow tests were performed at the highest flow rates for 45% emulsions. The temperatures in both tests were higher than previous runs due to a lack of adequate cooling water.

Table D-15: Conditions for tests with emulsion CLC-45-11.

	Test No.1	Test No.2
Flow Rate (kg/min)	100 to 190	190
Velocity (m/s)	4.8 to 9.1	9
Shear Rate ( $s^{-1}$ )	1800 to 3400	3400
Reynolds Number	10,800 to 22,00	*22,000
Temperature ( $^{\circ}C$ )	18 to 24	25
Initial Emulsion		
Viscosity	8.3 mPas @ 24 $^{\circ}C$	
Oil Concentration	42 wt%	

Observations : Figure D-24 shows the friction factor-Reynolds number behaviour of the fresh emulsion. The data lie within 6% of the calibration curve for the Newtonian oil. The data agree well with the data from the test with emulsion CLC-45-05, Figure D-2. Figures D-25 and D-26 show the results of the long term flow test. Friction factor, temperature and oil concentration do not change appreciably during the test. Table D-16 shows that emulsion viscosity remains constant during the test. After 14 days and more than 10,000 km of running the test was terminated.

Time/Distance Travelled : >14 days/10,000 km

Instrument Calibration :

Flow Meter	0 to 400 kg/min
Pressure Transducers	0 to 150 kPa

Table D-16: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-45-11.

SAMPLE NO.	DAY	TIME	CUM.HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1 COMPOSITE					44	
2	0	15:00	0.0	0	42	8.3 @ 24.0
3	1	09:30	18.5	600	42	6.7 @ 26.8
4	1	15:30	24.5	794	43	7.0 @ 27.0
5	2	09:30	42.5	1380	42	7.0 @ 26.4
6	3	09:30	66.5	2160	42	7.0 @ 25.4
7	4	09:30	90.5	2930	43	6.9 @ 25.3
8	4	15:00	96.0	3110	42	7.1 @ 25.1
9	5	09:30	114.5	3710	43	7.0 @ 25.3
10	6	10:00	139.0	4504	43	7.7 @ 25.1
11	7	09:00	162.0	5250	43	N/A
12	8	11:00	188.0	6090	43	7.5 @ 24.7
13	9	13:00	214.0	6930	43	7.0 @ 24.9
14	10	21:00	246.0	7970	42	6.7 @ 24.9
15	11	11:00	260.0	8420	42	7.2 @ 25.0
16	12	11:00	284.0	9200	43	7.2 @ 25.1
17	13	11:00	308.0	9980	42	7.2 @ 25.0
18	14	11:00	332.0	10760	44	7.6 @ 25.0

N/A = not available

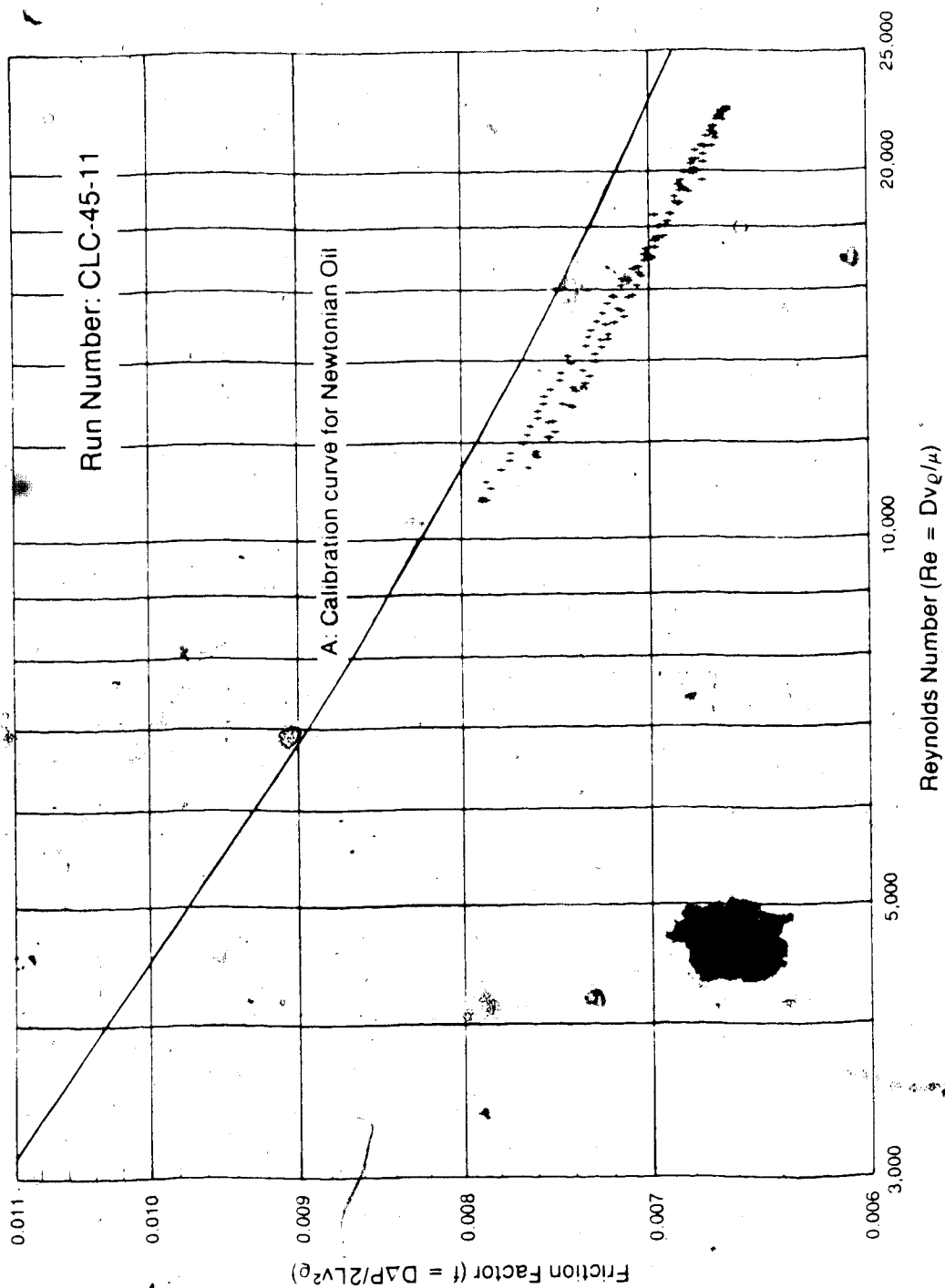


Figure D-24: The variation of friction factor with Reynolds number for CLC-45-11

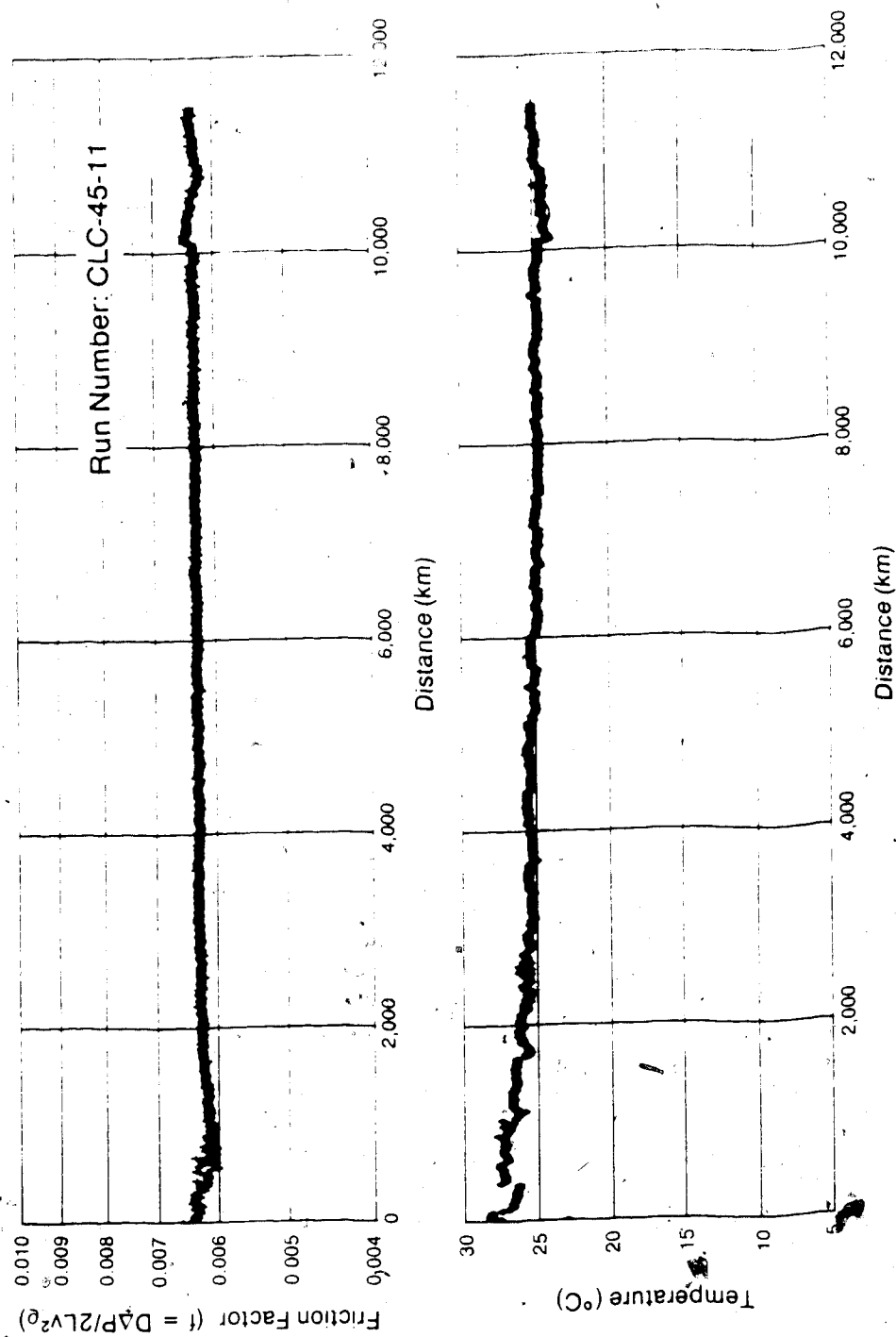


Figure D-25: The variation of friction factor and temperature with distance travelled for CLC-45-11.



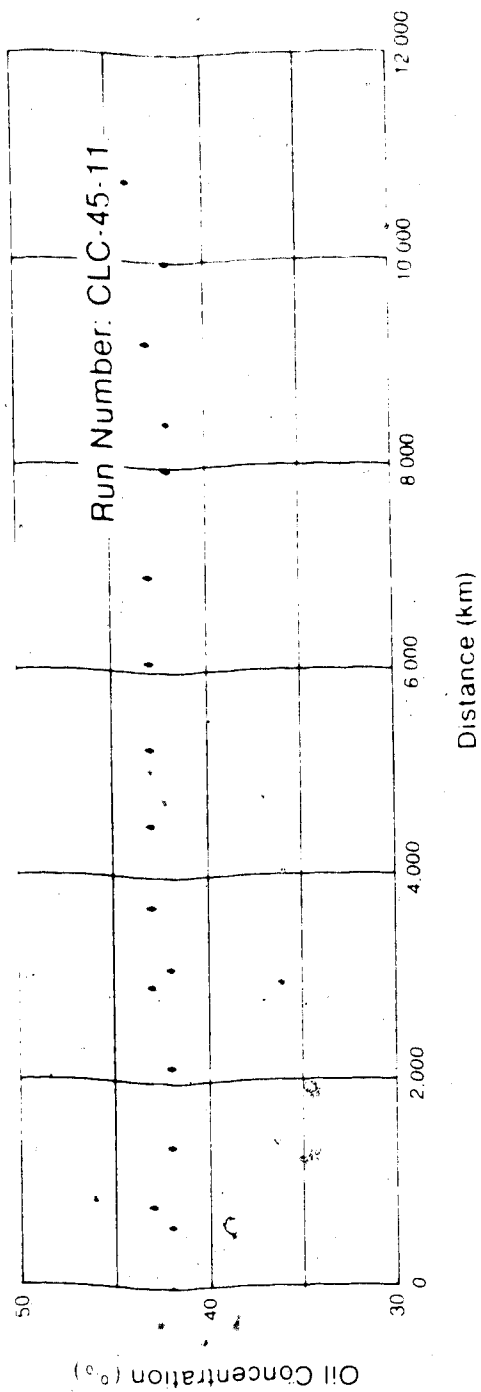


Figure D-26: The variation of oil concentration with distance travelled for CLC-45-11

Emulsion : CLC-55-09

Description : These tests were the first flow tests performed with a 55% emulsion. The same flowrates were covered as in CLC-45-05. This corresponds to a lower Reynolds number range due to the higher emulsion viscosity.

Table D-17: Conditions for tests with emulsion CLC-55-09.

	Test No. 1	Test No. 2
Flow Rate (kg/min)	92 to 92	125
Velocity (m/s)	4.3 to 9.0	6
Shear Rate ( $s^{-1}$ )	1500 to 3400	2300
Reynolds Number	4000 to 8300	5500
Temperature ( $^{\circ}C$ )	11 to 16	12
Initial Emulsion		
Viscosity	23 mPas @ $12^{\circ}C$	
Oil Concentration	57 wt%	

Observations : Figure D-27 shows the friction factor-Reynolds number data for this emulsion. The initial data are about 25% below the calibration line for the Newtonian oil. As the test progresses measured pressure drops move to within 15% of the predicted pressure drops based on the measured physical properties of the emulsion. This severe hysteresis was not observed elsewhere. Figure D-28 and D-29 show the results of the long term flow test. Initial friction factors are not as steady as in test CLC-45-05, Figure D-3, but appear to level off after 150 kilometers. The friction factor decreases abruptly after 650 kilometers and recovers to a second more stable value. Viscosity measurements were no longer possible on samples taken from the system after this point. The recovery in friction factors is accompanied by an increase in emulsion temperature of  $12^{\circ}C$ , Figure D-28. The test was stopped when the system no longer appeared to be changing. Table D-18 shows the changes in emulsion viscosity and oil concentration for samples taken during the test.

Time/Distance Travelled : 30 h / 670 km

Instrument Calibration :

Flow Meter 0 to 400 kg/min  
 Pressure Transducers 0 to 150 kPa

Table D-18: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-55-09.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1	0	14:30	0.0	0	57	23 @ 12.0
2	0	21:00	6.5	130	52	22 @ 11.6
3	1	08:30	18.0	390	52	22 @ 12.2
4	1	11:30	21.0	454	51	23 @ 11.9
5	1	16:30	26.0	562	50	14 @ 11.9
6	1	21:30	31.0	670	51	15 @ 12.0
7	2	15:30	49.0	1060	47	N/A
8	3		70.0	1510	47	N/A
9	4		85.	1836	47	N/A

N/A = not available

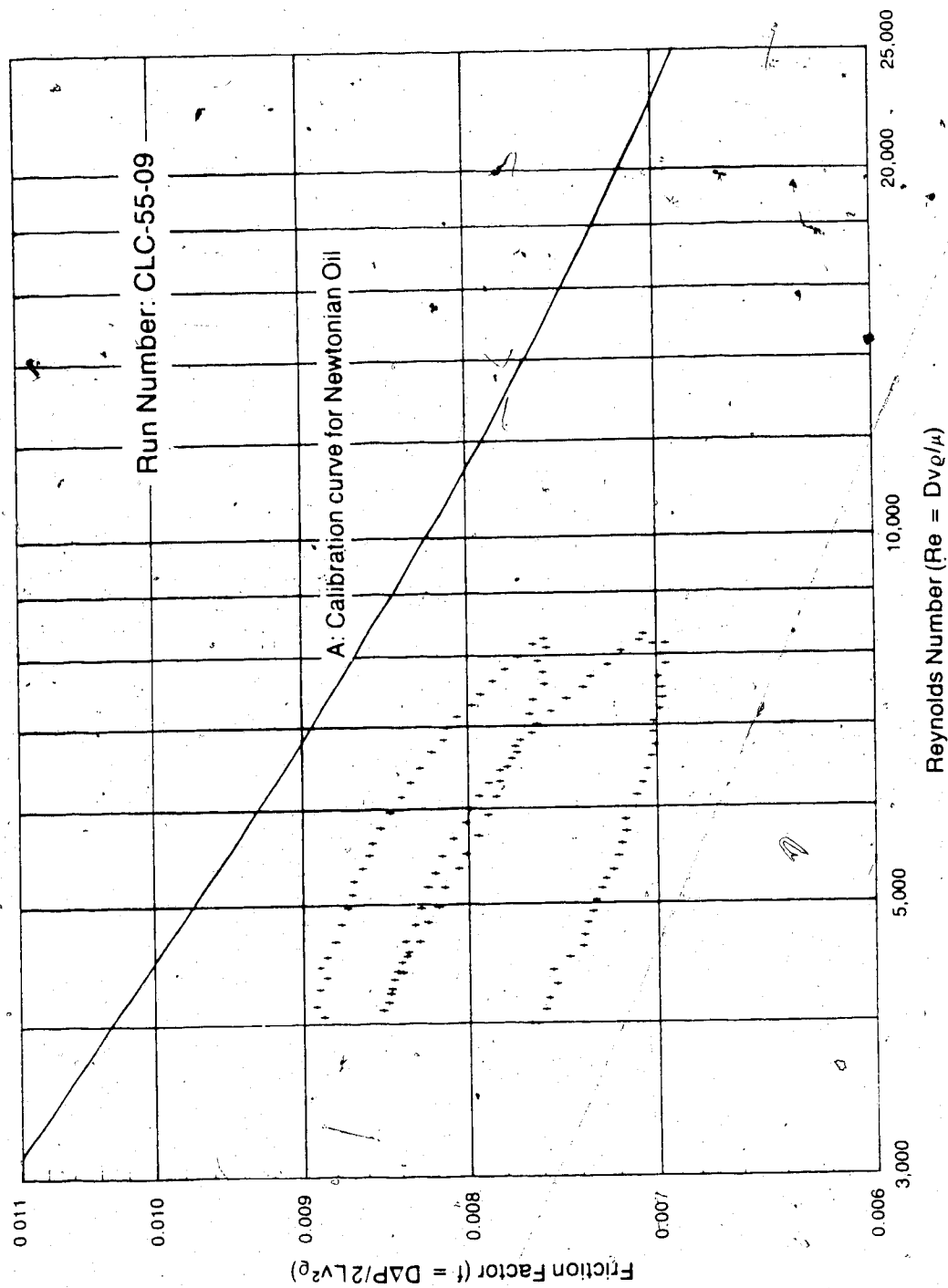
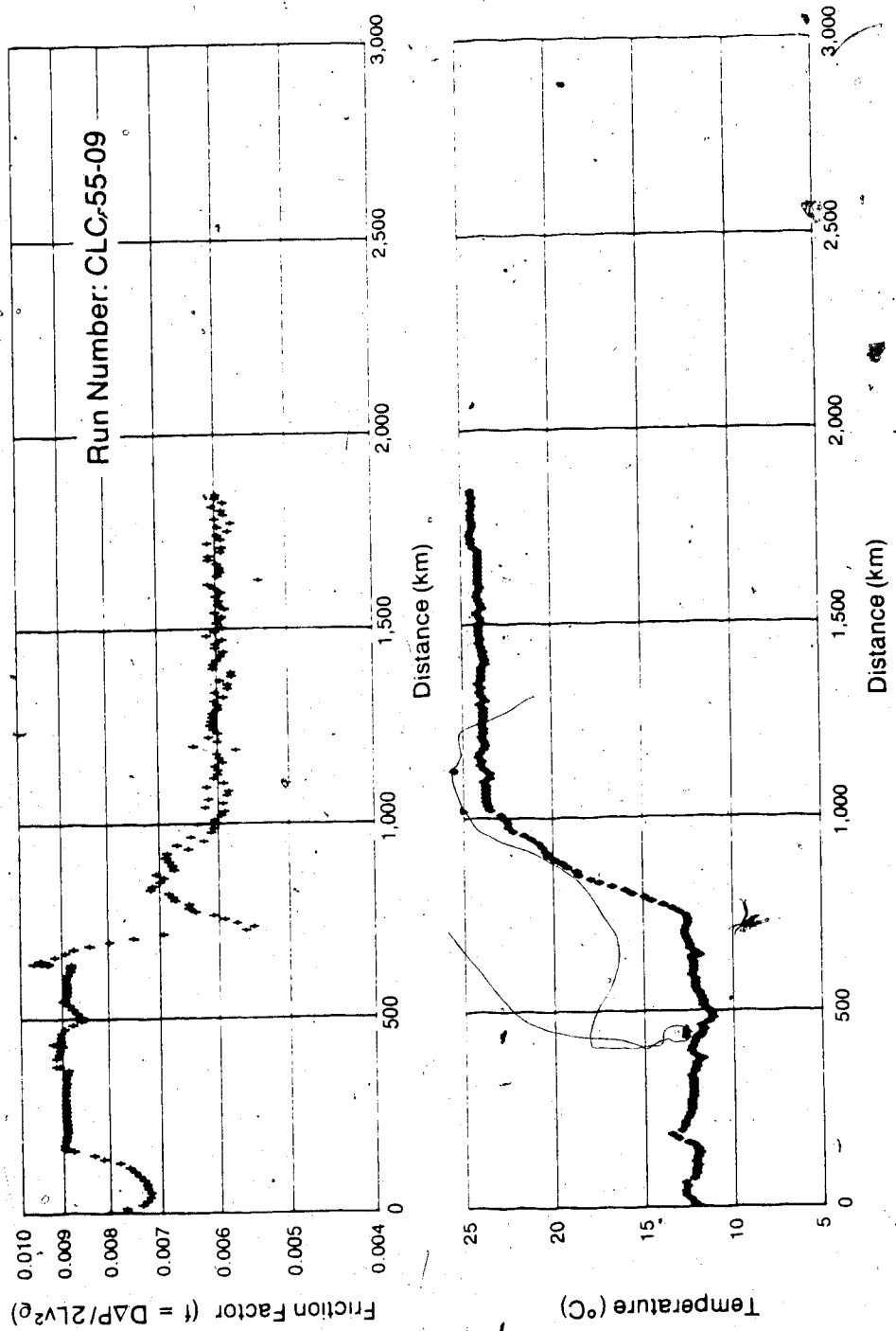


Figure D-27: The variation of friction factor with Reynolds number for CLC-55-09.



**Figure D-28:** The variation of friction factor and temperature with distance travelled for CLC-55-09.

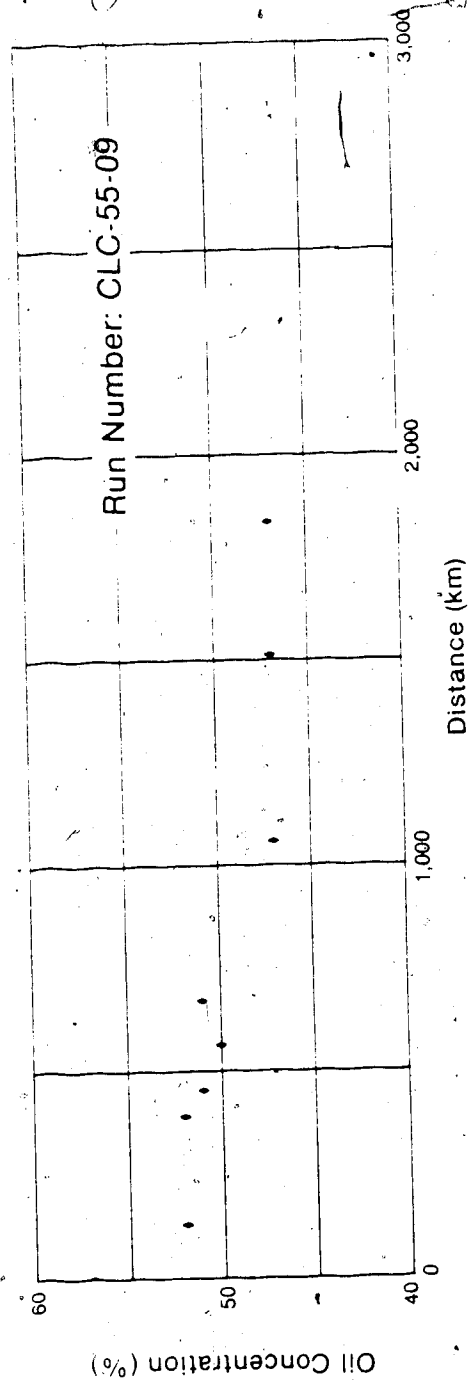


Figure D-29: The variation of oil concentration with distance travelled for CLC-55-09.

Emulsion : CLC-55-10

Description : These were the first laminar flow tests with a 55% emulsion. Flow rates were chosen to correspond with those used in tests with emulsion CLC-45-08. A 2 horsepower motor was used for this run and subsequent low flow rate runs to prevent problems due to insufficient horsepower at the end of a long term test.

Table D-19: Conditions for tests with emulsion CLC-55-10.

	Test No. 1	Test No. 2
Flow Rate (kg/min)	8.0 to 24	14
Velocity (m/s)	.38 to 1.1	.67
Shear Rate ( $s^{-1}$ )	140 to 420	250
Reynolds Number	500 to 1200	700
Temperature ( $^{\circ}C$ )	12 to 13	11 to 12
Initial Emulsion		
Viscosity	20.0 mPa @ 12.7 $^{\circ}C$	
Oil Concentration	50 wt%	

Observations : Figure D-30 shows the friction factor-Reynolds number data lie within 18% of the calibration line  $f=16/Re$ . No strong hysteresis is noticeable in the data. Figures D-31, D-32 and D-33 show the results from the long term test. The friction factor decreases continually during the test for 450 kilometers. After 460 kilometers, the friction factor suddenly increases rapidly. This is accompanied by an increase in temperature of 12 $^{\circ}C$ , Figure D-32, and an increase in the current drawn by the 2 horsepower motor from 1.9 to 3 amps, Figure D-33. Viscosity measurements on samples taken from the system were no longer possible after this had occurred. Table D-20 shows the changes in emulsion viscosity and oil concentration for samples taken during the run.

Time/Distance Travelled : 190 h / 460 km

Instrument Calibration :

Flow Meter	0 to 45 kg/min
Pressure Transducers	0 to 10 kPa

Table D-20: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-55-10.

SAMPLE NO.	DAY	TIME	CUM.HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
COMPOSITE					53	
2	0	16:00	0.0	0	50	20.0 @ 12.7
3	1	09:00	17.0	41	50	22.2 @ 11.7
4	1	16:00	24.0	58	50	21.5 @ 11.9
5	2	09:00	41.0	99	50	20.4 @ 11.8
6	2	16:00	48.0	116	51	19.7 @ 11.9
7	3	09:00	65.0	157	51	19.5 @ 11.0
8	3	22:30	78.5	189	50	19.0 @ 11.3
9	4	09:00	89.0	215	53	18.5 @ 9.7
10	4	22:00	102.0	246	51	16.9 @ 10.8
11	5	09:00	113.0	273	49	16.9 @ 10.3
12	5	16:00	120.0	289	49	14.8 @ 11.5
13	6	09:00	137.5	332	49	N/A
14	6	16:00	144.0	347	49	N/A
15	7	09:00	161.0	388	47	14.2 @ 11.4
16	7	16:30	168.5	406	48	N/A
17	8	10:00	186.0	449	43	N/A
18	8	16:00	192.0	463		
UNCIRCULATED MATERIAL FROM BUCKET					52	

N/A = not available



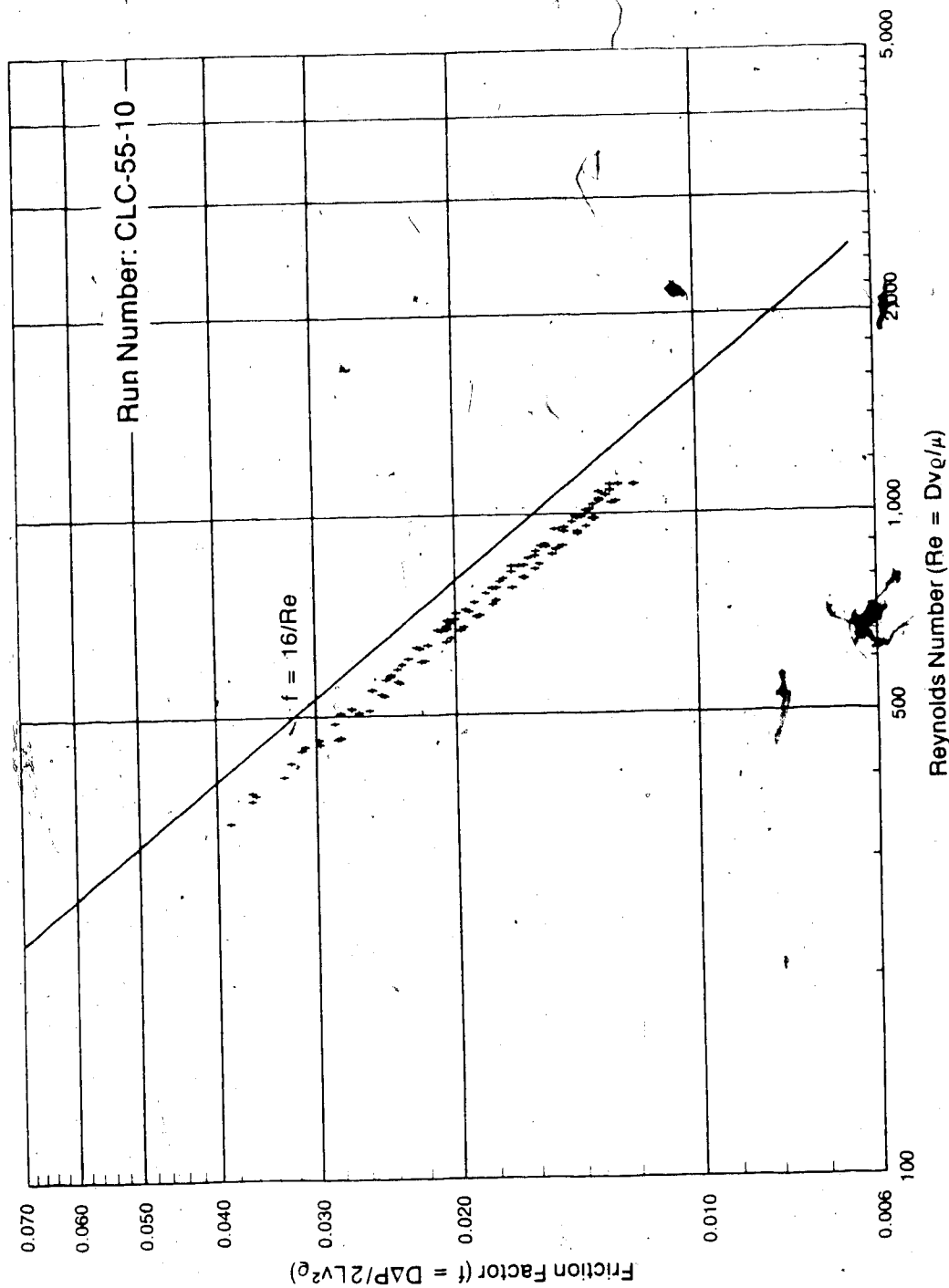


Figure D-30: The variation of friction factor with Reynolds number for CLC-55-10.

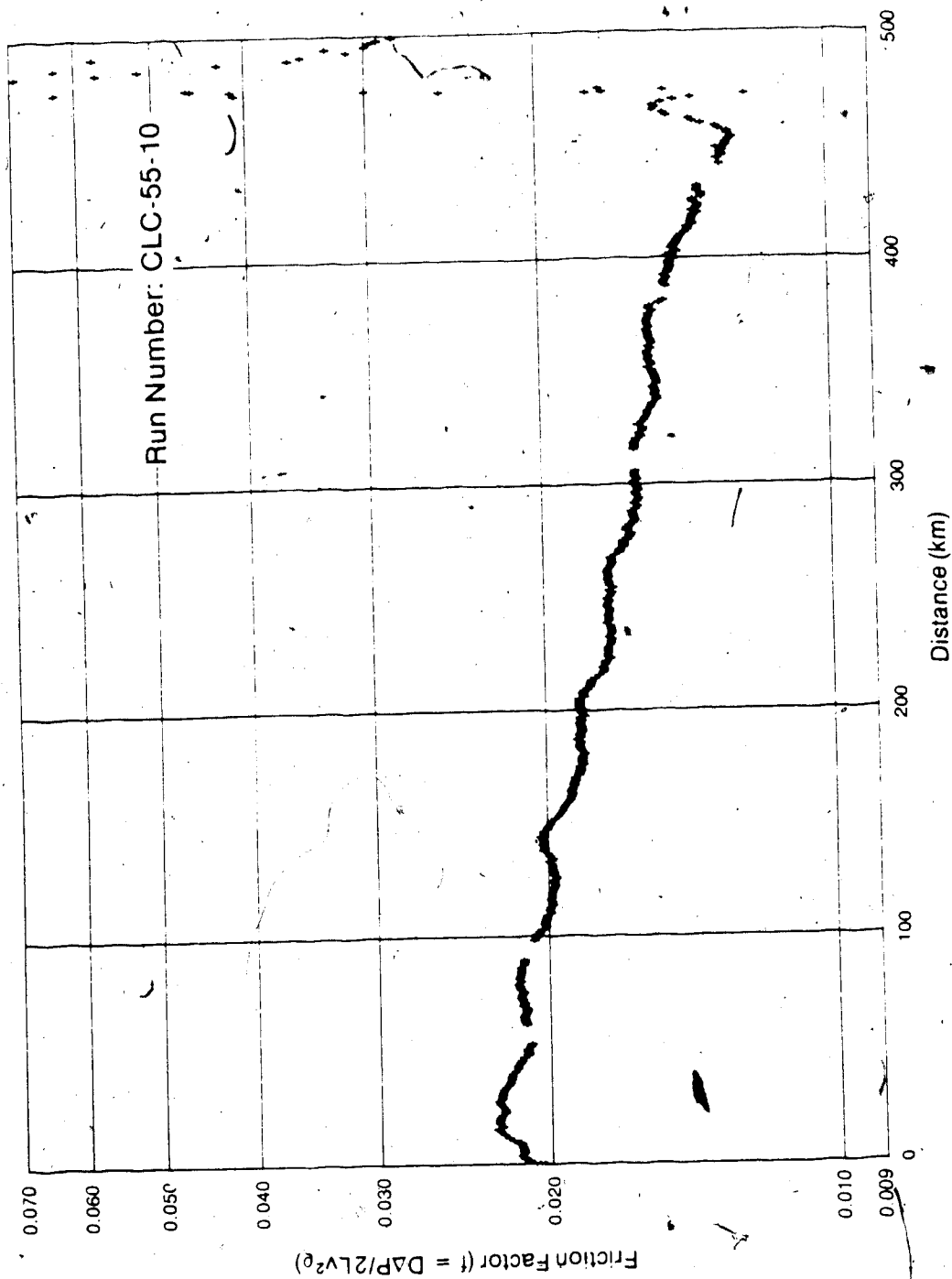


Figure D-31: The variation of friction factor with distance travelled for CLC-55-10

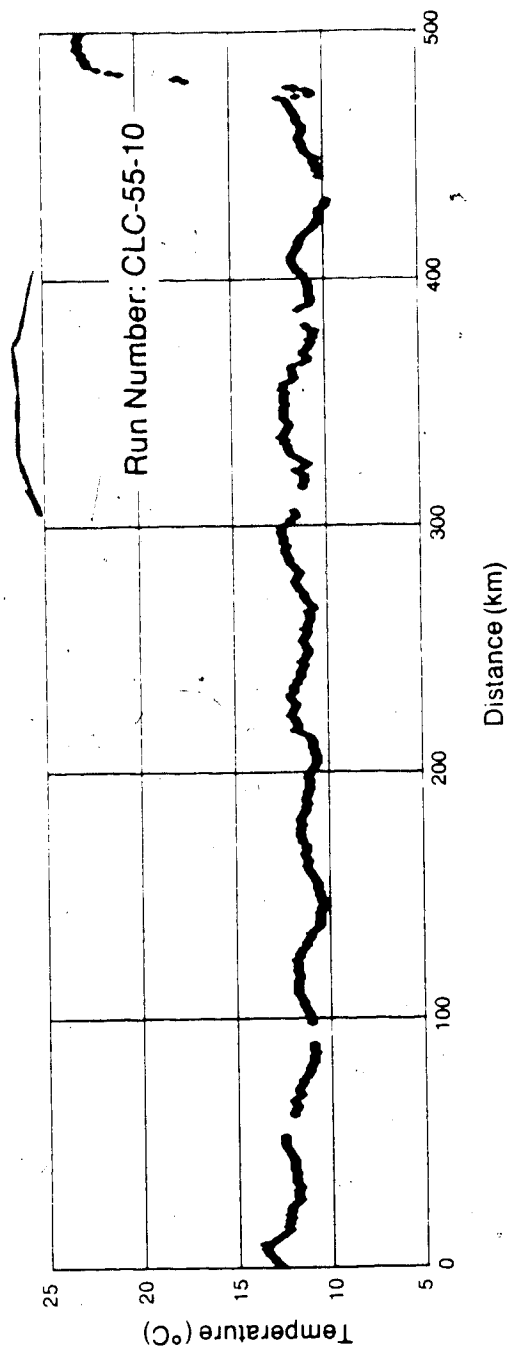


Figure D-32: The variation of temperature with distance travelled for CLC-55-10.

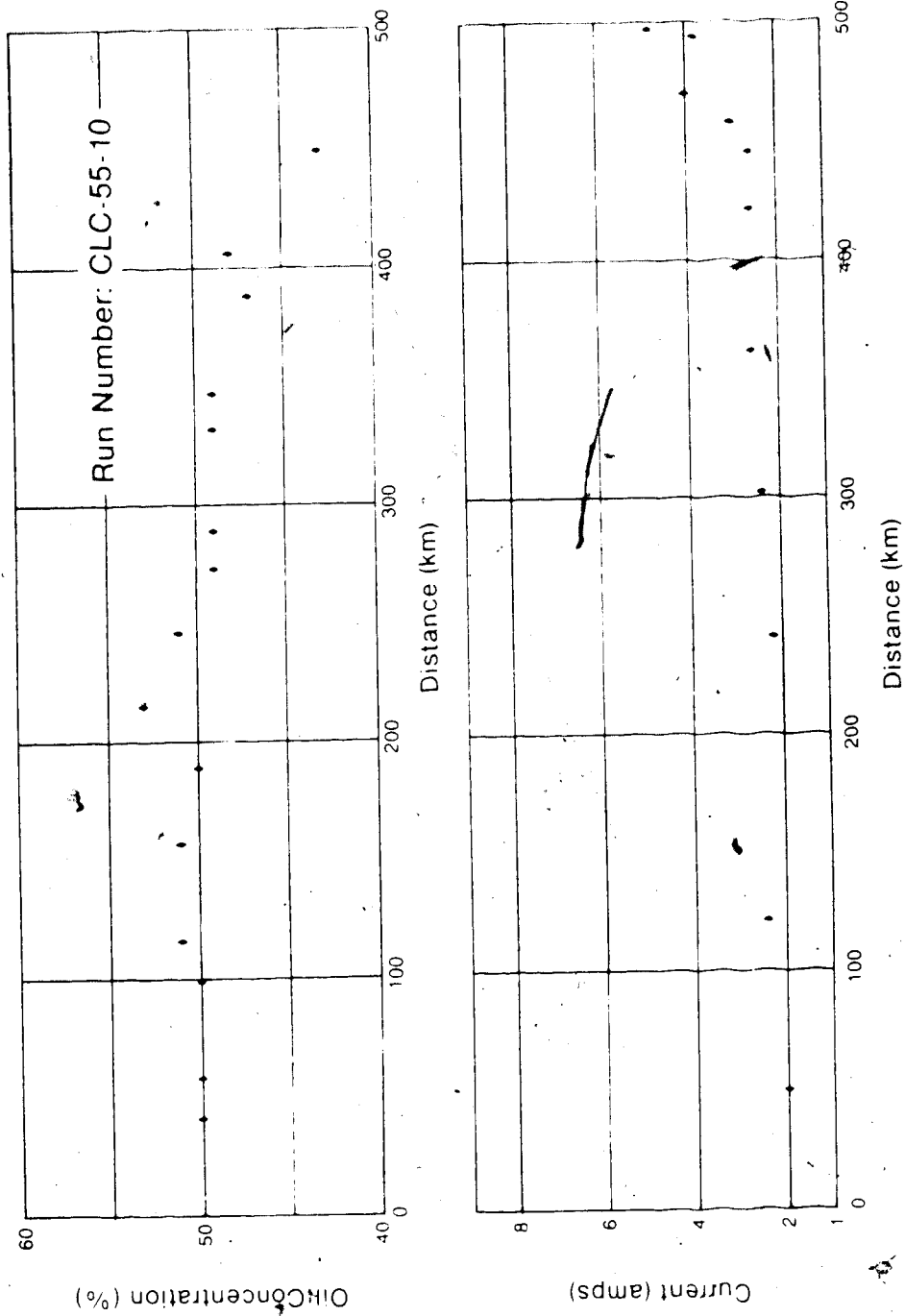


Figure D-33: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-55-10.

Emulsion : CLC-55-11

Description : These tests were done in the laminar regime at flow rates that were about double those used in the tests with emulsion CLC-55-10. Because the viscosity of the 55% emulsions is about twice that of the 45% emulsions the long term test had the same initial Reynolds number as the long term flow test with emulsion CLC-45-08.

Table D-21: Conditions for tests with emulsion CLC-55-11.

	Test No.1	Test No.2
Flow Rate (kg/min)	13 to 41	29
Velocity (m/s)	.6 to 1.9	1.3
Shear Rate ( $s^{-1}$ )	230 to 720	500
Reynolds Number	650 to 2100	1500
Temperature ( $^{\circ}C$ )		12
Initial Emulsion		
Viscosity	20.9 mPas @ 12.0 $^{\circ}C$	
Oil Concentration	49 wt%	

Observations : Figure D-34 shows the friction factor-Reynolds number data for the fresh emulsion lies within 20% of the calibration line  $f=16/Re$ . Figures D-35 and D-36 show the results of the long term flow test. In Figure D-35, the friction factor decreases initially in a manner similar to the laminar flow test with emulsion CLC-55-10. After about 200 kilometers, and for the next 400 kilometers, the friction factor appears scattered between two extremes. Towards the end of the test a more steady value of friction factor is achieved followed by a sharp decrease. This decrease in friction factor is accompanied by an increase in temperature of 12 $^{\circ}C$ , Figure D-35, and an increase in current drawn by the D.C. motor, Figure D-36. It was no longer possible to measure the viscosity of samples taken from the system. The changes in emulsion viscosity and oil concentration for samples taken during the test are given in Table D-22.

Time/Distance Travelled : 170 h / 800 km

Instrument Calibration :

Flow Meter                      0 to 45 kg/min  
 Pressure Transducers          0 to 15 kPa

Table D-22: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-55-11.

SAMPLE NO.	DAY	TIME	CUM.HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1 (COMPOSITE)					53	
2	0	16:00	0.0	0	49	20.9 @ 12.0
3	1	11:00	17.0	80	49	21.6 @ 12.9
4	1	16:00	24.0	112	50	21.6 @ 12.2
5	2	10:00	40.0	187	49	18.5 @ 12.7
6	2	22:00	52.0	243	47	17.6 @ 14.6
7	3	15:00	71.0	332	48	17.8 @ 14.6
8	4	09:00	87.0	407	48	17.4 @ 10.5
9					48	N/A
10	5	09:30	11.5	522	49	N/A
11	5	16:00	120.0	562	48	N/A
12	5	10:00	136.0	636	47	N/A
13	7	10:00	160.0	749	12	N/A
14	8				18	
15 (UNCIRCULATED BUCKET)				51		

N/A = not available

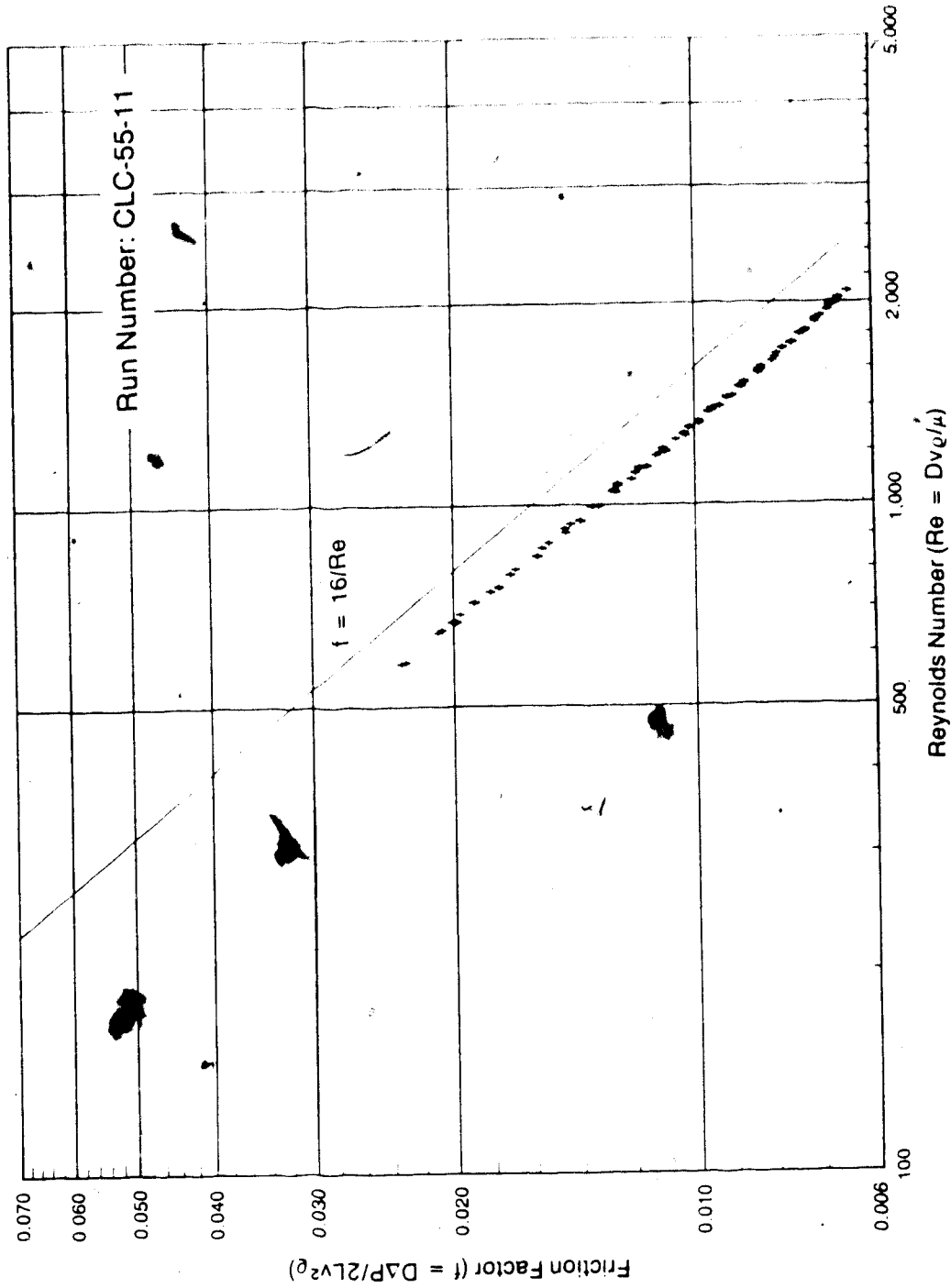


Figure D-34: The variation of friction factor with Reynolds number for CLC-55-11.

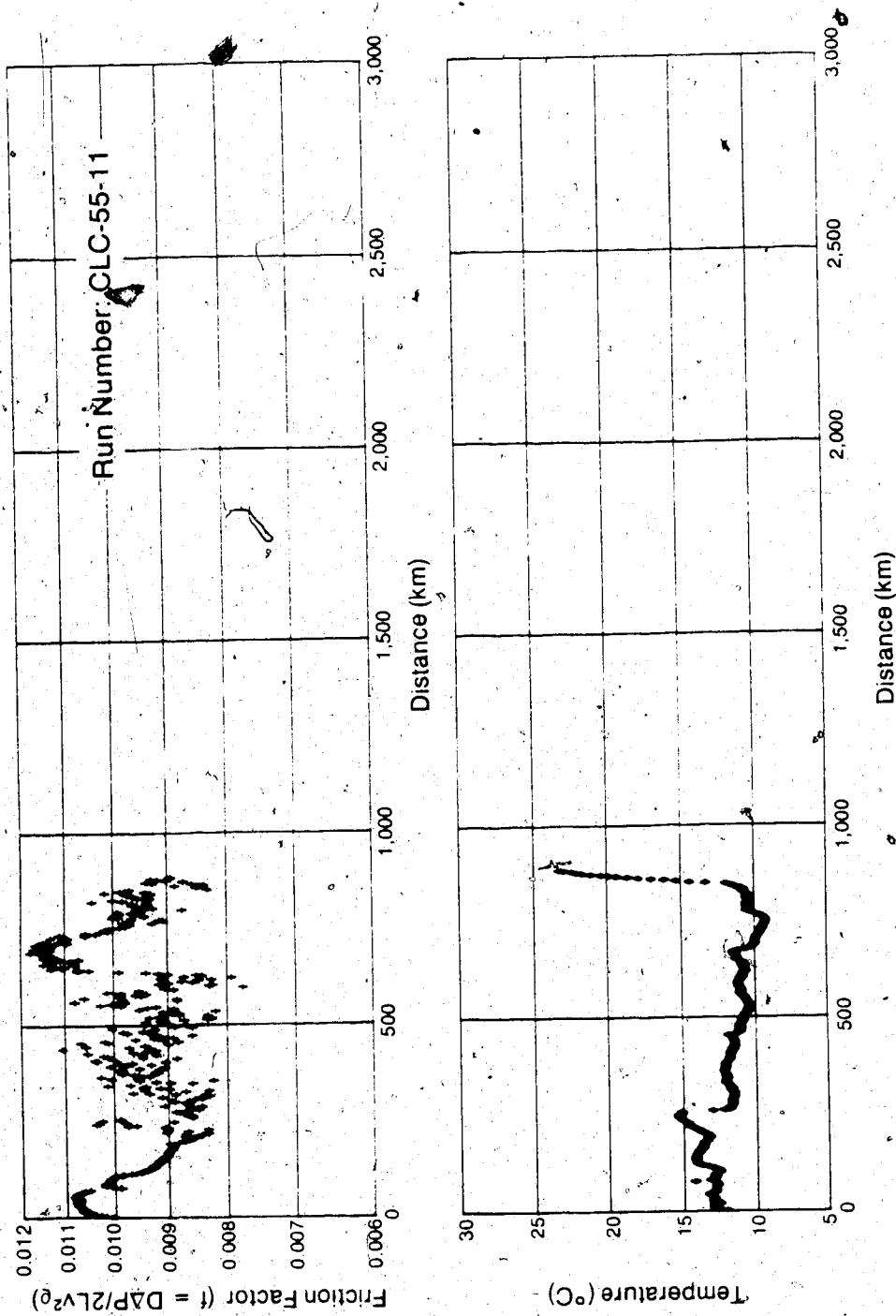


Figure D-35: The variation of friction factor and temperature with distance travelled for CLC-55-11.



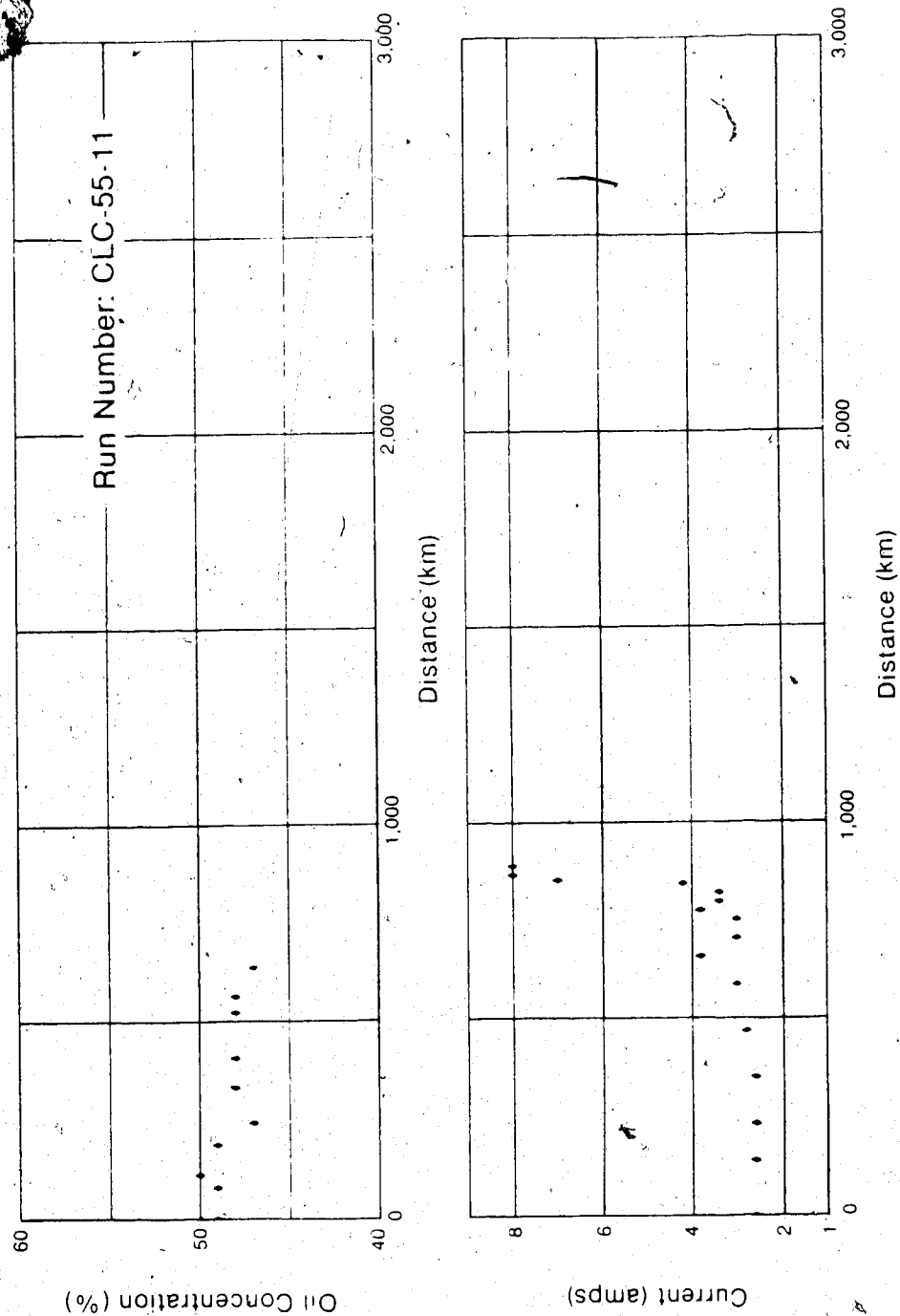


Figure D-36: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-55-11.

Emulsion : CLC-55-12

Description : These tests were run at the highest flow rates for 55% emulsions. The long term flow test was run at the same flow rate as the long term test with emulsion CLC-45-11. Temperature in both tests were substantially higher than other tests with 55% emulsions due to the lack of adequate cooling water.

Table D-23: Conditions for tests with emulsion CLC-55-12.

	Test No.1	Test No.2
Flow Rate (kg/min)	124 to 198	190
Velocity (m/s)	5.9 to 9.4	9
Shear Rate ( $s^{-1}$ )	2200 to 3500	3400
Reynolds Number	11,000 to 17,700	17,000
Temperature ( $^{\circ}C$ )	21 to 26	25
Initial Emulsion		
Viscosity	11.2 mPas @ 22.3 $^{\circ}C$	
Oil Concentration	48 wt%	

Observations : Figure D-37 shows the friction factor-Reynolds number data lie within 8% of the calibration line for the Newtonian oil. Although there appears to be about 4% scatter in the data, the results agree well with those from the test with emulsion CLC-45-11. Figures D-38 and D-39 show the results of the long term test. Friction factor, temperature and oil concentration do not change appreciably during the test and emulsion viscosity remains constant, Table D-24. After 16 days and more than 10,000 kilometers the test was terminated.

Time/Distance Travelled : >14 days / 10,000 km

Instrument Calibration :

Flowmeter

0 to 400 kg/min

Pressure Transducers

0 to 150 kPa

Table D-24: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-55-12.

SAMPLE NO.	DAY	TIME	CUM. HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
1 COMPOSITE					51	
2	0	17:00	0	0	48	11.2 @ 22.3
3	1	14:30	21.5	697	48	13.8 @ 25.3
4	2	13:00	44.0	1430	48	12.4 @ 25.3
5	3	13:30	68.5	2220	48	11.7 @ 25.3
6	4	14:30	93.5	3030	47	11.0 @ 25.4
7	5	14:00	117.0	3790	47	12.1 @ 24.5
8	6	09:00	136.0	4410	47	11.4 @ 24.5
9	7	11:00	162.0	5250	47	11.5 @ 24.5
10	8	11:00	186.0	6030	47	10.9 @ 24.5
11	9	11:00	210.0	6800	47	10.9 @ 24.3
12	11	16:00	263.0	8520	48	10.6 @ 25.0
13	12	10:00	281.0	9100	46	10.6 @ 25.0
14	14	09:00	304.0	9850	47	10.8 @ 24.9
15	14	11:00	330.0	10690	47	11.1 @ 25.1
16	16	14:30	381.5	12360	46	10.2 @ 25.0
PAIL NO. 1					51	
PAIL NO. 2					50	
PAIL NO. 3					50	

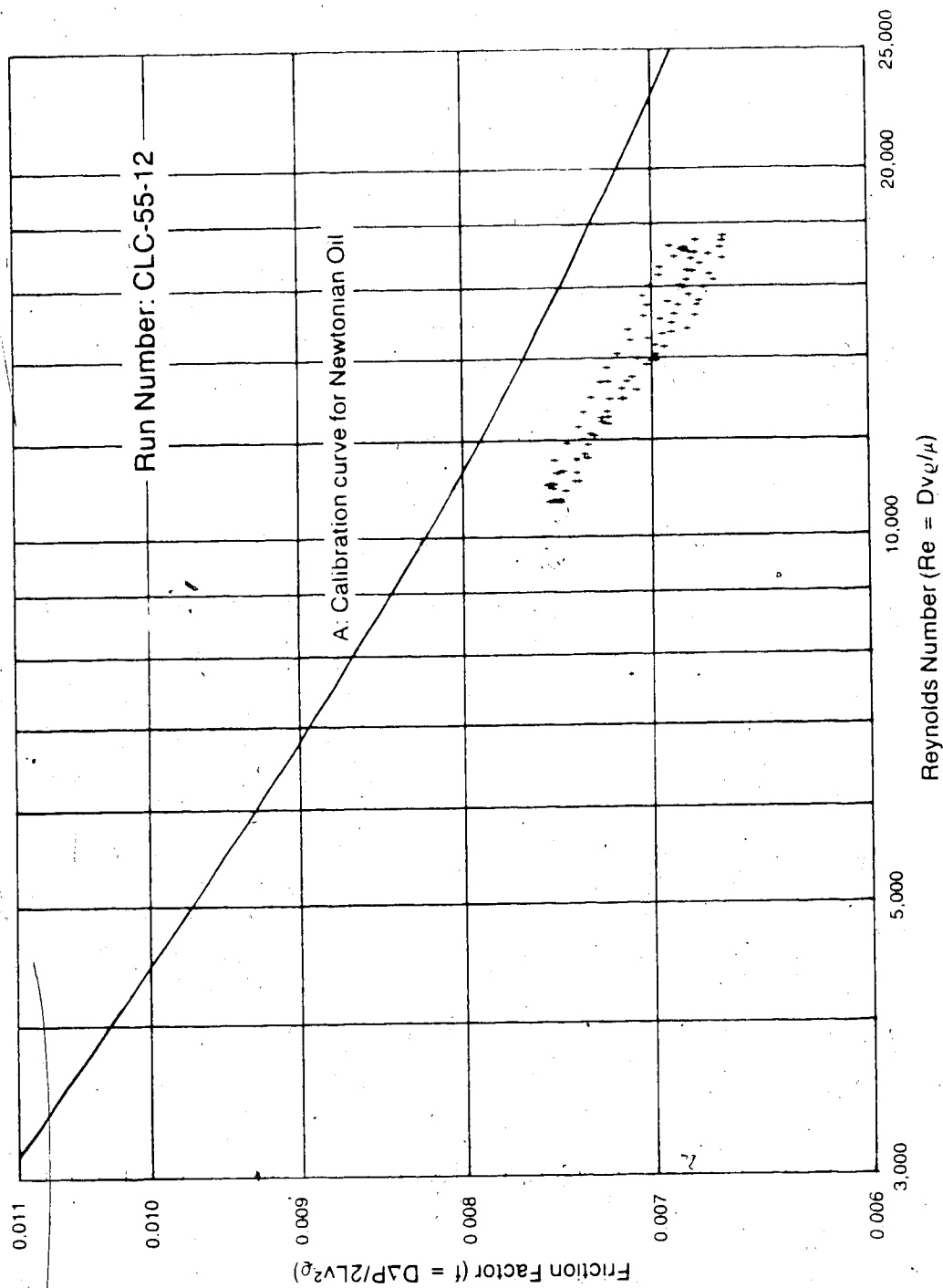


Figure D-37: The variation of friction factor with Reynolds number for CLC-55-12.

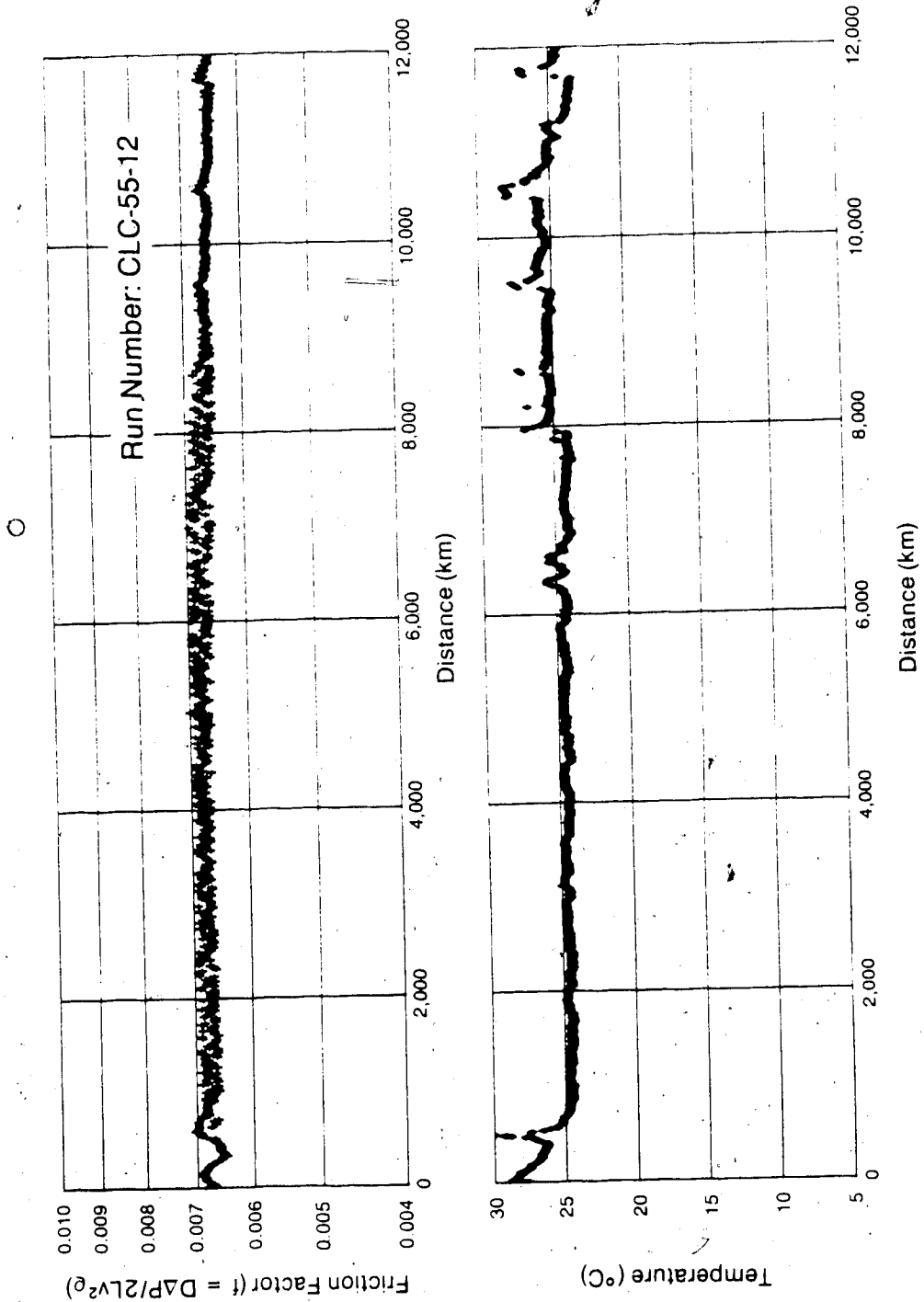


Figure D-38: The variation of friction factor and temperature with distance travelled for CLC-55-12.

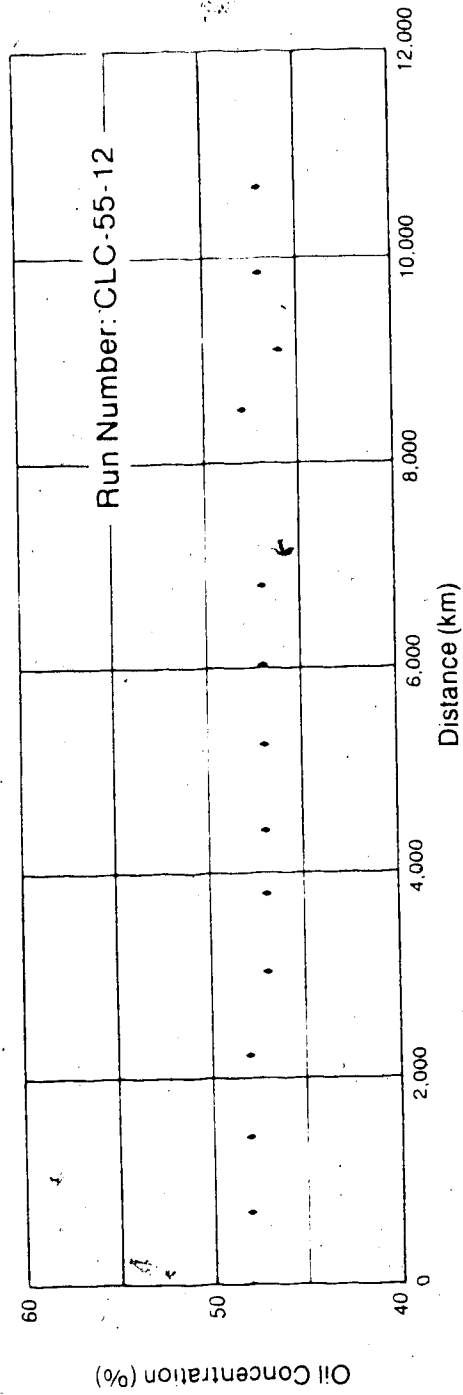


Figure D-39: The variation of oil concentration with distance travelled for CLC-55-12.

Emulsion : CLC-55-13

Description : These tests were performed at the lowest flow rate for 55% emulsions. The flow rates are the same as those used in the tests with emulsion CLC-45-10.

Table D-25: Conditions for tests with emulsion CLC-55-13.

	Test No.1	Test No. 2
Flow Rate (kg/min)	4.2 to 9.6	7.4
Velocity (m/s)	.20 to .45	.35
Shear Rate ( $s^{-1}$ )	75 to 170	130
Reynolds Number	190 to 450	325
Temperature ( $^{\circ}C$ )	9 to 11	10
Initial Emulsion		
Viscosity	22.2 mPas @ 9.8 $^{\circ}C$	
Oil Concentration	49 wt%	

Observations : Figure D-40 shows the friction factor-Reynolds number data lie within 2% of the calibration line  $f=16/Re$ . Figures D-41, D-42 and D-43 show the results from the long term test. In Figure D-41, friction factor decreases steadily for the first 200 kilometers, increasing abruptly after this point. The abrupt change in friction factor is accompanied by an increase in temperature of 10 $^{\circ}C$ , Figure D-42, and an increase in current drawn by the D.C. motor, Figure D-43. Table D-26 shows the decrease in emulsion viscosity and oil concentration for samples taken during the test. The pressure data from these tests were based on the data from the Rosemount pressure transducers only as the data from the Viatran transducer were too scattered.

Time/Distance Travelled : 170 h / 200 km

Instrument Calibration :

Flowmeter	0 to 45 kg/min
Pressure Transducer 1.	0 to 7 kPa (Rosemount)
2.	0 to 2 kPa (Rosemount)
3.	0 to 2 kPa (Viатran)

Table D-26: Oil concentration and emulsion viscosity as functions of real time and distance travelled during the long term test with emulsion CLC-55-13.

SAMPLE NO.	DAY	TIME	CUM.HRS	DISTANCE KM	OIL CONCENTRATION (wt%)	EMULSION VISCOSITY (mPas @ °C)
PAIL NO.1					53	
PAIL NO.2					52	
PAIL NO.3					50	
1 COMPOSITE					52	
2	0	17:00	0	0	49	22.2 @ 9.8
3	1	10:00	17.0	21	48	21.1 @ 10.4
4	2	11:00	42.0	53	17	18.2 @ 9.7
5	4	14:30	93.5	118	3	15.5 @ 9.1
6	5	12:00	115.5	145	40	15.0 @ 9.1
7	6	08:30	135.5	171	38	9 @ 9.0
8	7	09:30	160.5	202	27	5 @ 9.3



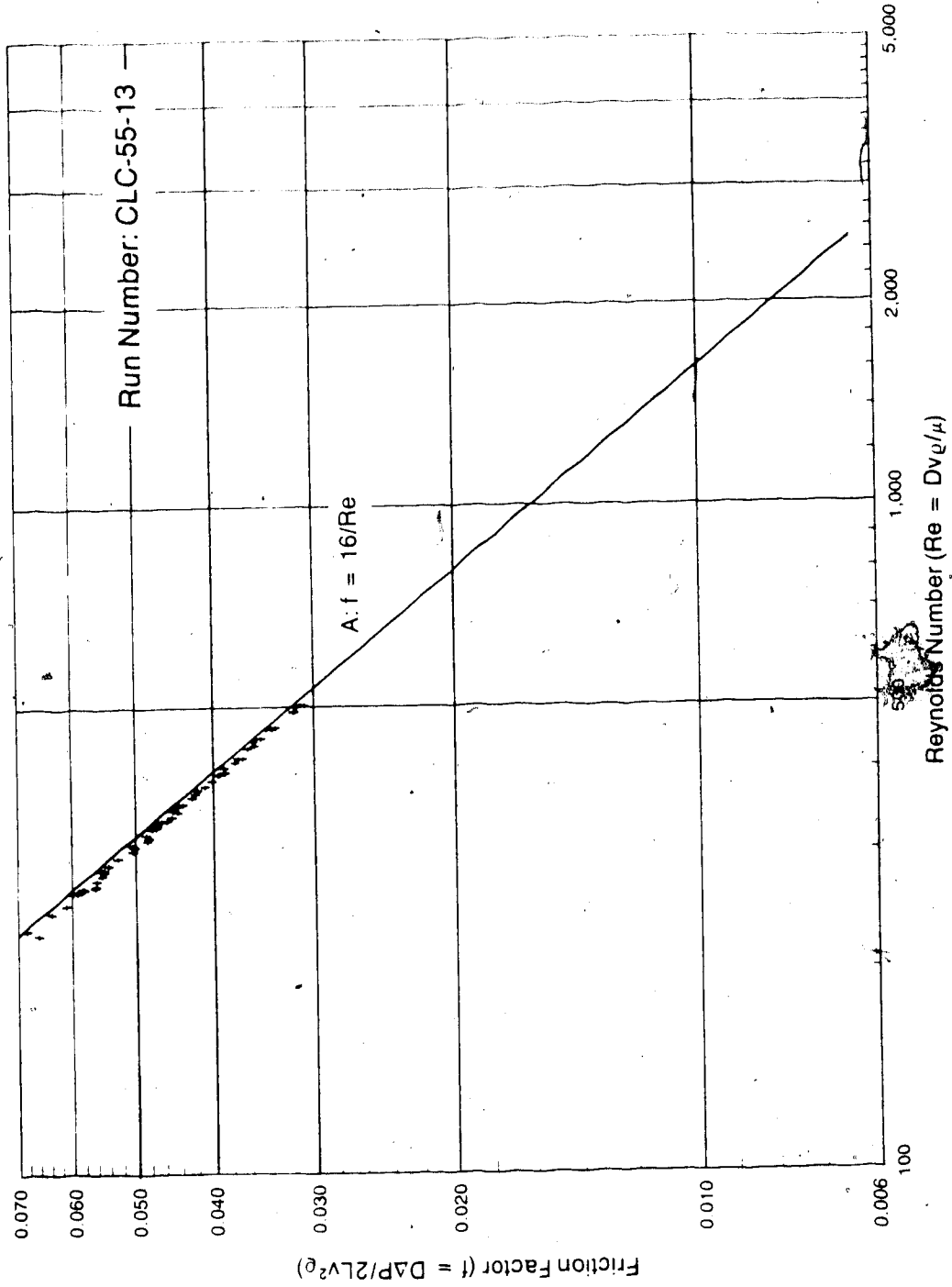


Figure D-40: The variation of friction factor with Reynolds number for CLC-55-13.

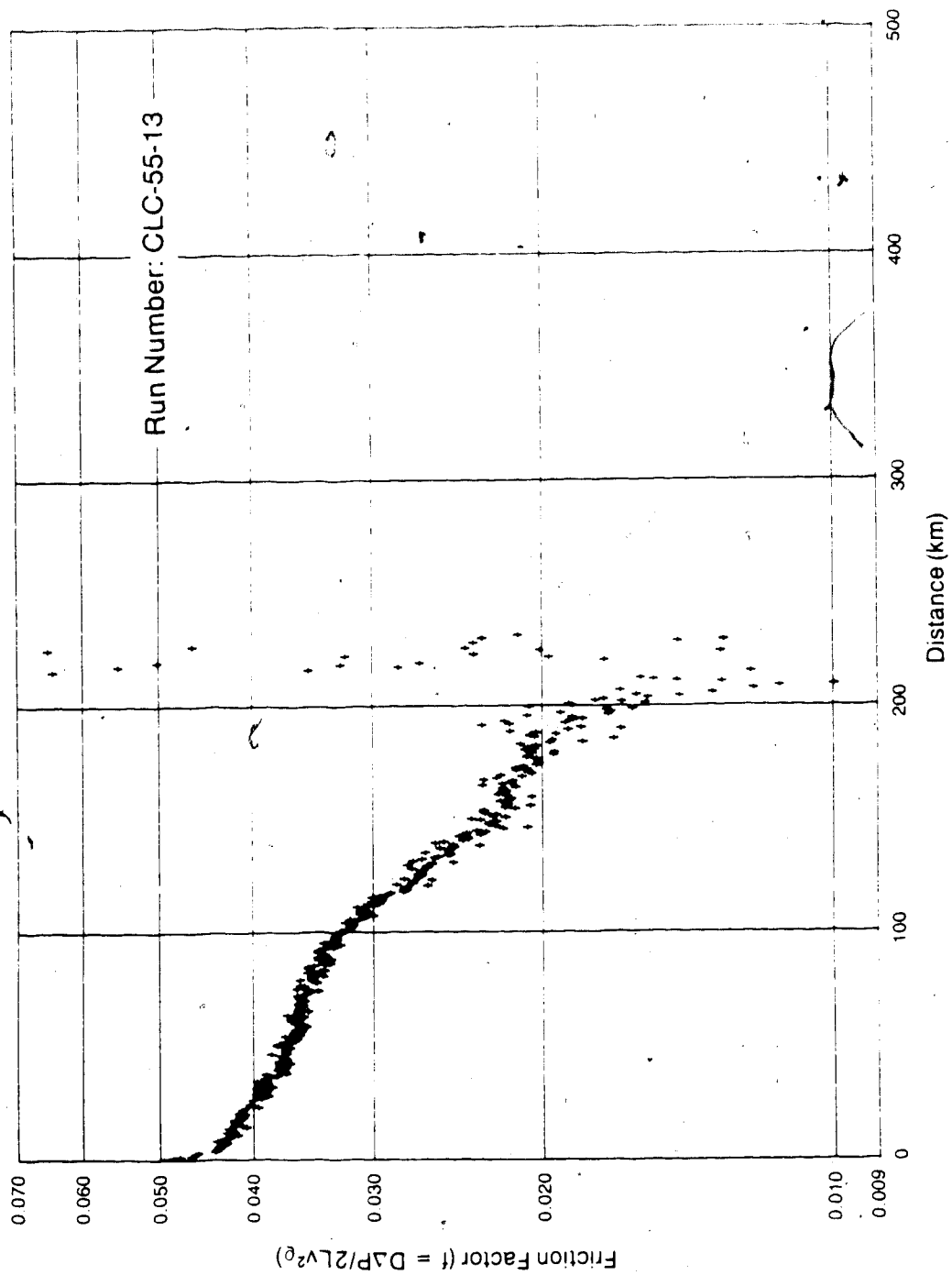


Figure D-41: The variation of friction factor with distance travelled for CLC-55-13.

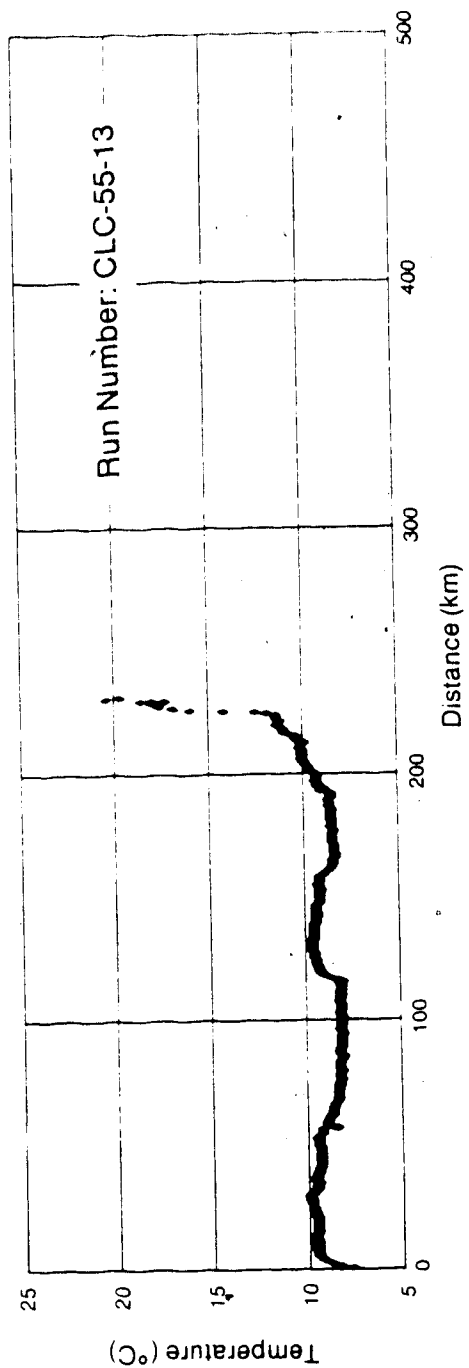


Figure D-42: The variation of temperature with distance travelled for CLC-55-13.

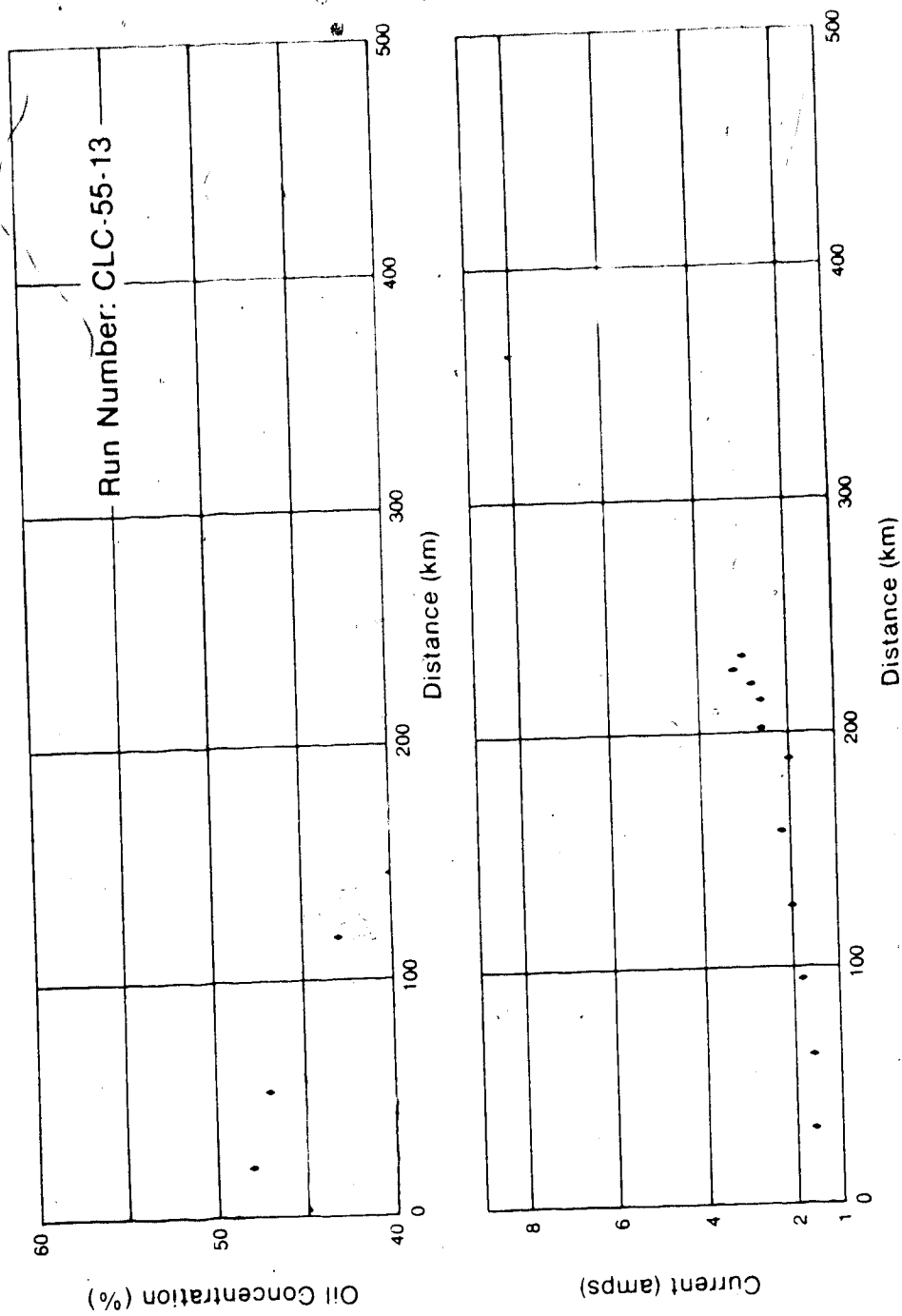


Figure D-43: The variation of oil concentration and current drawn by D.C. motor with distance travelled for CLC-55-13.

Figure D-44 summarizes the friction factor-Reynolds number data from tests with the emulsions CLC-45-05, CLC-45-08, CLC-45-09, CLC-45-10 and CLC-45-11. All of the data lie within 10% of the calibration lines established with the Newtonian oils. The data appear to be consistent for tests made with different emulsion batches.

Figure D-45 summarizes the friction factor-Reynolds number data from tests with the emulsions CLC-55-09, CLC-55-10, CLC-55-11, CLC-55-12 and CLC-55-13. All of the data lie within 25% of the calibration lines established with the Newtonian oils with the majority of data lying within 20% of the calibration lines. The data are less consistent between tests made with different emulsion batches but are still considered acceptable.

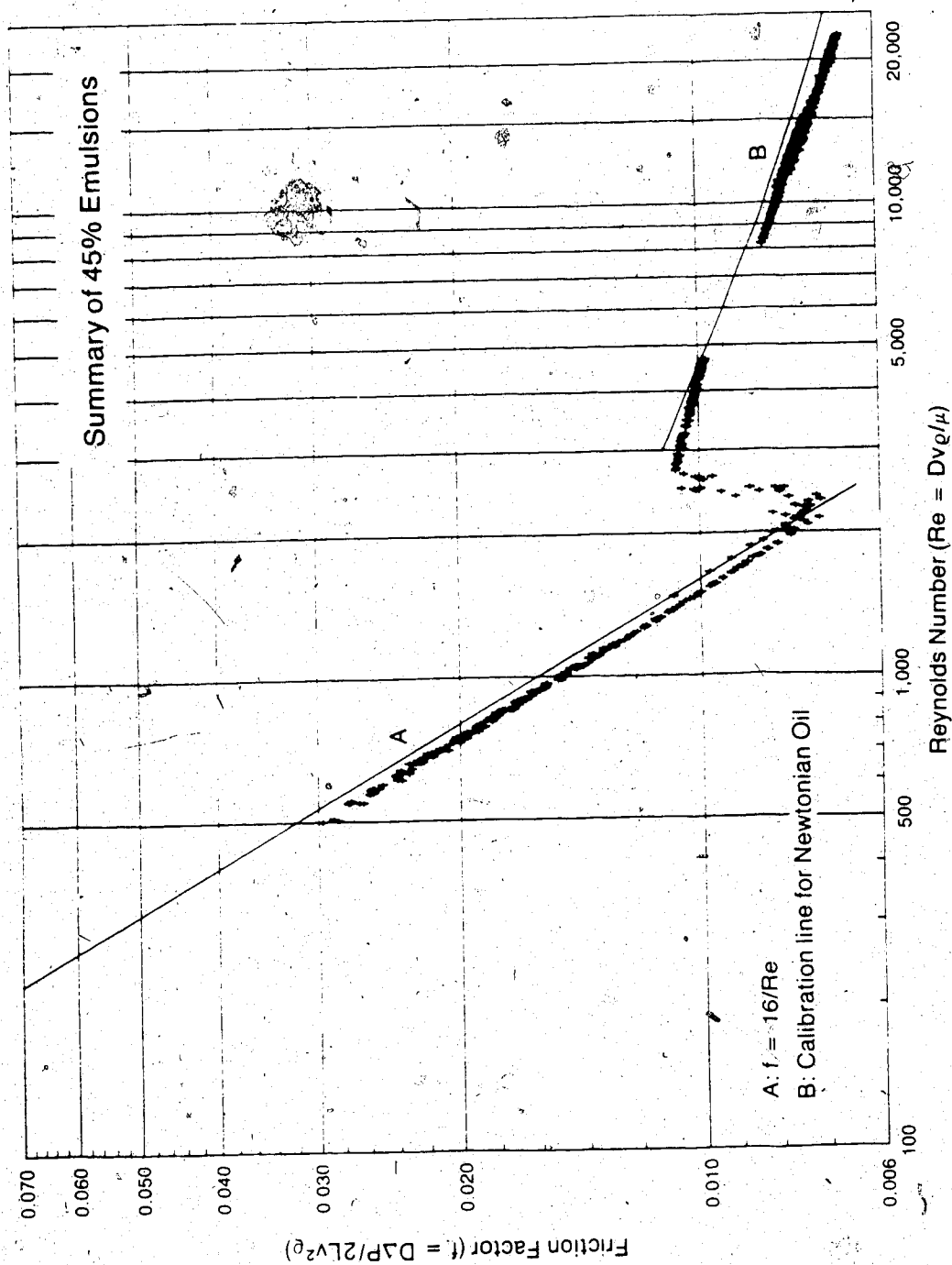


Figure D-44: Summary of the friction factor Reynolds number data for the nominally 45% emulsions

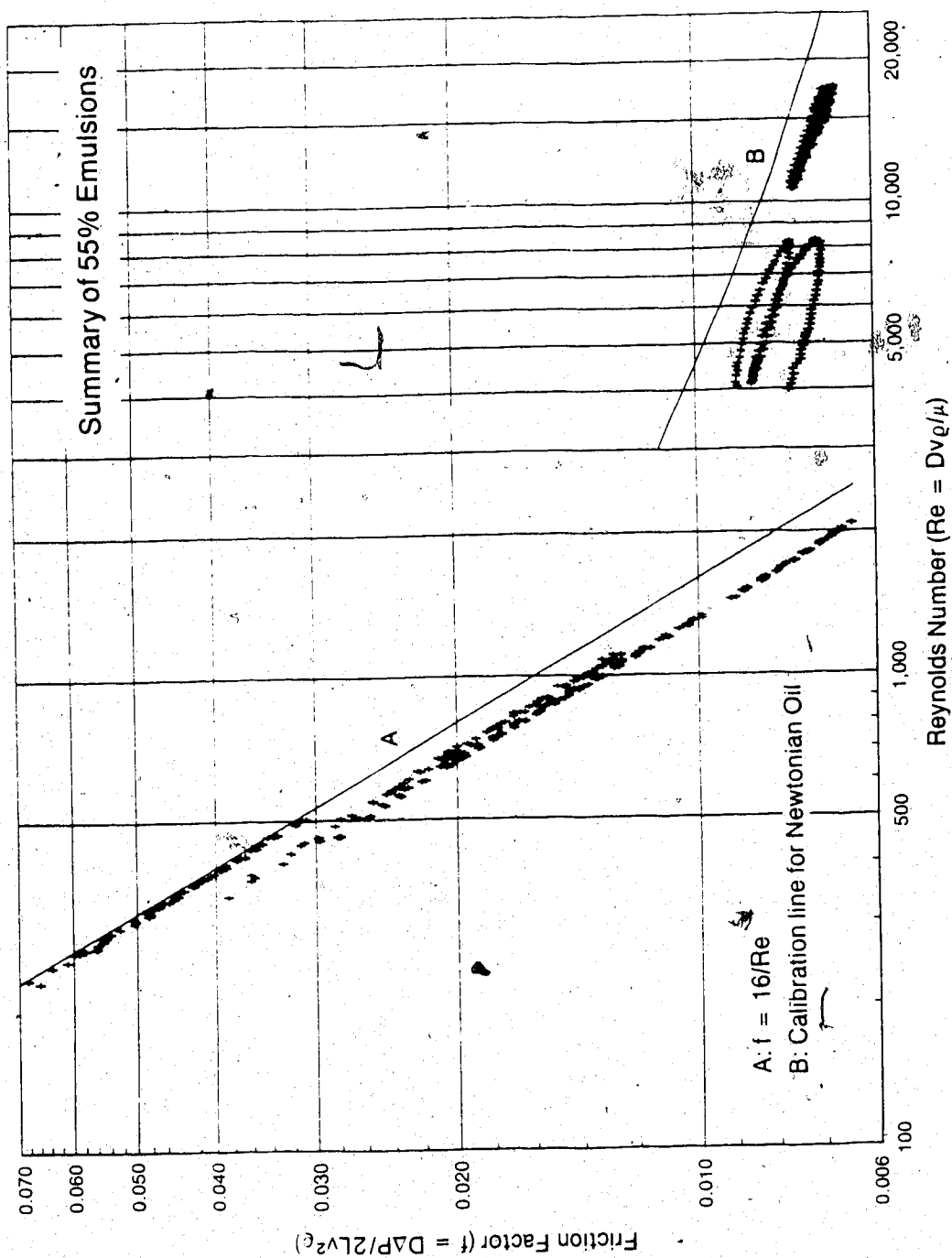


Figure D-45: Summary of the friction factor Reynolds number data for the nominally 55% emulsions