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## HIGH-TEMPERATURE SETTLING OF BITUMEN FROM AOSTRA'S UNDERGROUND TEST FACILITY

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1990

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by

R.J. Mikula, I.S. Parsons, V.A. Munoz, W.W. Lam, C. Payette and K.C. McAuley

### ABSTRACT

Several bitumen samples from AOSTRA's Underground Test Facility were obtained (heat exchanger outlet) in order to characterize the emulsion droplet size distribution and to ultimately establish whether or not high temperature settling could successfully be used to separate the bitumen and water phases. Characterization of the dispersed phase was not straightforward since the samples varied. The samples would sometimes be separated into a large bitumen mass and significant free water and sometimes be quite fluid with dispersed bitumen. It was our opinion that sampling contamination, perhaps with residual soaps, lead to some samples remaining as a bitumen in water emulsion without separating. Normally, one would expect that the bitumen would separate from the free water.

Preliminary characterization of the solids in the feed was also done since it is known that mineral/solids composition can influence emulsion formation and the stability of rag layers in bitumen/water separation schemes. High temperature settling evaluations proved the feasibility of this type of settling as a method of bitumen separation producing a product of less than 5% water. Good results were achieved with temperatures from 190 to 220°C and 250 to 1000 ppm demulsifier. Separation without demulsifiers was not successful. Product samples were examined microscopically to determine the dispersed water size distributions.

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1990

## INTRODUCTION

The feasibility of using high temperature settling to separate the bitumen and water phases in the UTF produced fluid was investigated using CANMET's pressure vessel. The relatively low solids content of the UTF fluids meant that it might be a good candidate for inverse separation with the bitumen phase heavier than the water phase. The objectives of the experiments were to demonstrate the feasibility of the process and to define the dispersed phase droplet size distribution in order to support design of a scaled up version.

Prediction of the settling and separation behaviour is not straightforward since the produced fluid is a bitumen in water emulsion whereas the separation performance is determined by the separation of water from the bitumen phase. Therefore, the important parameters are the bitumen viscosity and the dispersed water phase size distribution.

As the bitumen separates from the water phase (since the produced fluid is initially a bitumen in water emulsion), there is a point at which the emulsion inverts, or becomes water in bitumen. The ultimate performance of the settling step is then determined by how efficiently the water separates from the bitumen phase. Based on other data, it was felt that a water content of less than 5% in the bitumen product could be flashed from the system by simply reducing the pressure. Producing a 5% water in bitumen product would then demonstrate the feasibility of the high temperature settling process.

## EXPERIMENTAL

A Carl Zeiss research microscope photometer with an incident light system (reflected light) was utilized for the optical microscopic analyses of the core samples. The white light source was a halogen lamp with an average brightness of 1,750 cd/cm<sup>2</sup>.

The optical examination of the samples was carried out using three forms of illumination. Plane polarized incident light, which was obtained with a polarizer set at 45° from the vertical, placed between the light source and

the vertical illuminator. The second type was partially crossed polarized light. In this case a polarizer was set in the incident beam, to produce plane polarized incident light, and a second polarizer (analyzer) was introduced in the reflected beam at  $90^\circ$  with respect to the polarizer. By rotating the analyzer a few degrees, partially crossed polarization was obtained.

The third form of illumination was the examination of the fluorescent characteristics of the samples using a high pressure mercury lamp with an average brightness of  $170,000 \text{ cd/cm}^2$ . The selection of the wavelength of the incident beam was accomplished with a combination of filters which provided a range from 450 to 490 nanometers (blue light).

The electron microscopes used for this project are a Hitachi S-2500 equipped with only an energy dispersive spectrometer and a Hitachi X-650 equipped with both wavelength and energy dispersive spectrometers. The energy dispersive spectrometer (Tracor 30-mm<sup>3</sup> Si(Li) detector) was used to produce all of the x-ray spectra. The incident beam current was approximately 0.2 nA at 25 KeV unless otherwise specified. The x-ray fluorescence which is excited by the incident electron beam is characteristic for each of the elements present in the sample.

An EMscope 2000 cryo-system is used to prepare the frozen hydrated emulsion samples. The procedure involves freezing the sample in a liquid nitrogen slush and keeping it frozen and under vacuum in the cryo-stage. The sample is then fractured to reveal the interior of the frozen emulsion before transferring it to the cold stage in the SEM (Hitachi X-650). The cold stage in the SEM is a Hexland DN302; its temperature is maintained at  $\sim 90^\circ\text{K}$  by an Oxford ITC4 nitrogen heat exchanger and temperature-controller unit. The energy-dispersive x-ray detector used in the cryo-stage work is a 30-mm<sup>3</sup> Si(Li) Tracor Northern detector coupled to the SEM and to the automated image analyser. Low electron energy (15-20 keV) and short acquisition times are used while accumulating x-ray spectra in the spot mode, in order to minimize beam damage and local evaporation of the sample.

In order to evaluate froth settling at elevated temperatures, it is necessary to contain significant pressures in order to prevent the boiling of the water phase. Figure 1 illustrates the design of the pressure vessel showing the sampling bulkhead and the heating tape. Figure 2 is a schematic of the sampling point and thermocouple locations. The vessel was heated

over a period of time and then cooled before the samples were extracted. This procedure was then modified to provide for sample extraction while the vessel was near the reaction temperature. Samples were extracted into a sealed, cooled container in order to prevent "flashing" of the hot product once the pressure is released. The water, bitumen and solids content were determined by a standard centrifuge method, and aliquots were saved for subsequent Dean Stark analysis.

## RESULTS AND DISCUSSION

Figure 3 shows the density curves for both bitumen (8° API) and water as a function of temperature. At temperatures above about 120°C, the bitumen phase becomes denser than water, allowing for separation of the bitumen and water components with the bitumen at the bottom. Stokes law predicts that separation velocity depends upon the density difference, and inversely upon the radius of the dispersed phase and the viscosity of the continuous phase. Figure 4 shows the viscosity dependence of the bitumen as a function of time. Clearly, with higher temperatures, both the viscosity and the density differences are working to promote separation. With higher temperatures, however, greater costs are incurred: the cost of heat as well as the cost of a vessel which can withstand the associated higher steam pressures. Since the AOSTRA UTF fluid is produced at approximately 220°C, this was the upper limit of temperatures used. The 'chicken wire and bubblegum' design of our pressure vessel did not allow for close control of the temperature and short excursions above this temperature may have occurred for short times. These are not likely to have made any significant difference in the water cuts observed. Figure 5 shows a typical temperature profile from the four thermocouples illustrated in Figure 2. Some of the structure and cross-over of the temperatures are believed to be due to the different heat capacities of the bitumen and water phases. Initially the bitumen is less dense than the water and will float. As the temperature rises above about 150°C, this floating bitumen (which has already probably coalesced to some extent), will begin to sink, forcing relatively cooler water to the middle and upper region. This has the effect of causing a temperature drop (briefly) in the middle thermocouple. This phenomena would not occur in a more evenly heated vessel. However, the sampling does not occur until the temperatures are

within 5 degrees (after approximately 90 minutes), so it is not expected to be significant in determining the water content in the bitumen product.

The residence time is obviously very important in determining the ultimate water content in the dispersed phase and unfortunately, it is not possible to accurately estimate this because of the significant heating time required. The ninety minutes is clearly an upper limit since for the majority of that time, the bitumen was in fact the upper phase.

Characterization of the nature of two of the supplied samples was done in order to determine some baseline information on the feed samples. This is included here for completeness although it is probably not relevant to the separation of the bitumen and water via high temperature settling. The sample variability has already been mentioned. In addition, since the water content in the bitumen is determined by the settling of water through a continuous bitumen phase, it is not relevant to determine the bitumen size distribution in the water phase. Furthermore, the only sample that was bitumen in water was probably contaminated during sampling since it would be expected that during shipment the dispersed bitumen phase of the produced fluid would coalesce into a continuous bitumen phase and some free water. In fact this was the case for the subsequent samples and it was this bitumen continuous component of the sample which was used for the high temperature settling evaluations. Nevertheless, the following microscopic observations are interesting in that they point out the potential complexities in attempting to model separation in these types of systems.

Figure 6 shows the bitumen in water component of the UTF cooling coil sample (Feb 16, 1990). This illustrates a wide size range of bitumen from about 30 microns to one micron. In addition, it shows a multi-level emulsion which is difficult to model. The corresponding sample from the heat exchanger was quantified as to the size distribution of the dispersed phase. This data is shown in Figure 7.

Of significantly more importance is the data on the size distribution of the remaining water in the bitumen product from the high temperature settling experiments. Figure 8 shows the size distribution from a very long residence time (three hours) run without demulsifier. It was possible to characterize this emulsion (water in bitumen) with the optical microscope because of the relatively large droplet size. Unfortunately, sampling problems prevented a more detailed analysis than to say that the feed

bitumen was 60% water and that the product bitumen was 15% water as determined via a spin test.

Initial problems with the heating and temperature consistency of the vessel meant that this bitumen was above a slightly cooler (and therefore denser) water layer. Once these problems were solved, however, the bitumen products from the vessel were all sampled from below the water phase. These results are discussed in more detail in the Appendix A. Dean Stark analysis were done on the feed and products from five runs which included two with 1000 ppm demulsifier at about 220°C, (Champion X-888, based on total feed), one with no demulsifier, at 220°C and two at about 190°C with 500 ppm and 250 ppm demulsifier. All of these tests produced bitumen products of less than 5% water with a water residue of less than 5% bitumen. The feed samples were the separated bitumen from the heat exchanger outlet at the AOSTRA UTF site. The normal sample would be a bitumen in water emulsion produced at 220°C; these tests therefore represent a worse case in terms of bitumen separation since the water trapped in the coalesced bitumen phase represents considerably more water and more tightly bound water than would be found in a system in which the water phase was always above or lighter than the bitumen phase. It would be reasonable to expect that significantly more efficient separations could be achieved with fresh emulsion, without the cooling step which is necessary when transporting samples. The cost of demulsifier X-888 is \$2.50/litre (for 1000 litre lots). This represents a cost of about 40 cents per barrel of fluid treated, at a rate of 1000 ppm. At a rate of 250 ppm, this becomes 10 cents per barrel of fluid or approximately 20 cents per barrel of bitumen produced. As noted, the treatment rates can be expected to be even lower in operation since these tests represent a worse case.

Determining the size distribution of the dispersed phase of the water in bitumen product emulsion was not possible with the optical microscope. Using freeze fracture and frozen hydrated observation of samples in the electron microscope, however, it was possible to obtain some information about the size distribution of the dispersed phase. Figure 9 shows some scanning electron microscope photos of the dispersed water phase in the 1000 ppm run from May 24. This run produced a bitumen product of about 1.6% water and 0.6% sand (sand being the solids, usually clays). Many fields of view were scanned before finding these droplets, all of which are less than



one micron in size and many of which are less than 0.5 microns. Figure 10 shows similar features in the bitumen product from the run with 250 ppm demulsifier and 190°C. This bitumen product also had a low water content and although it was nominally slightly higher than that in Figure 9, little or no water could be found.

The results presented here, although they clearly indicate the feasibility of high temperature settling as a technique for separating bitumen and water in the UTF fluid, are not definitive. Figure 11 shows some typical spin tests of feed samples. A noticeable interface emulsion or rag layer is seen. Although no evidence of this was noted in the high temperature settling experiments, a build up of such a layer during continuous operation cannot be discounted. In addition, residence times are not and can not be well understood with results from the present vessel. Resolution of these questions demands experiments with the as produced feed on a continuous basis. Stability of these interface layers and of water in bitumen emulsions has been correlated to the presence of iron compounds and characterization of the mineral composition could be important with continuous operation. Figure 12 shows an x-ray spectra of solids from the Dean Stark extraction of the feed sample. The spectra is typical of clays (aluminum and silica) and other oil sands minerals (iron, titanium and calcium). The chlorine and some of the silica is due to contamination from the thimble. Baseline mineral characterization could be important if upsets in separator operation occur due to inefficient separation or rag layer formation. With a complete characterization of mineralogy during normal operation, it could be easier to determine whether a change in efficiency is due to operational changes or to feed composition changes.

#### SUMMARY

It is possible with demulsifier additions of 250 ppm and at operating temperatures of 190°C to achieve a bitumen product of about 2% water and solids. This translates into a cost of about 20 cents per barrel of produced bitumen and means handling about one third of the fluid volumes downstream of the separator. These numbers represent the worse case since they involve the treatment of the coalesced (and aged) bitumen component of the UTF fluids. The worse case residence time estimated from these tests is

about one hour. With treatment of fresh froth, one could expect significant improvements in both residence time and the level of demulsifier addition required. In addition, with the option of flashing residual moisture it should be possible to get even more significant improvements.

Further work should be scaled up to an on line pressure vessel where questions about residence time and rag layer build up can be answered. Design of the vessel might benefit from viscosity information about the bitumen phase and potential dispersed water phase size determinations. The applicability of the observations discussed in this report are not known since the cooled, coalesced bitumen tested in these trials will bear little resemblance to the bitumen in water emulsion which would constitute the feed to an on line separation vessel. However, characterization of the products' dispersed water size distribution will be important in understanding operating parameters, as will characterization of mineralogy and organic nature of the feed during normal operation. This will be especially important in tracing and treating upset conditions.

Figure 1. Photograph of high temperature settling vessel.

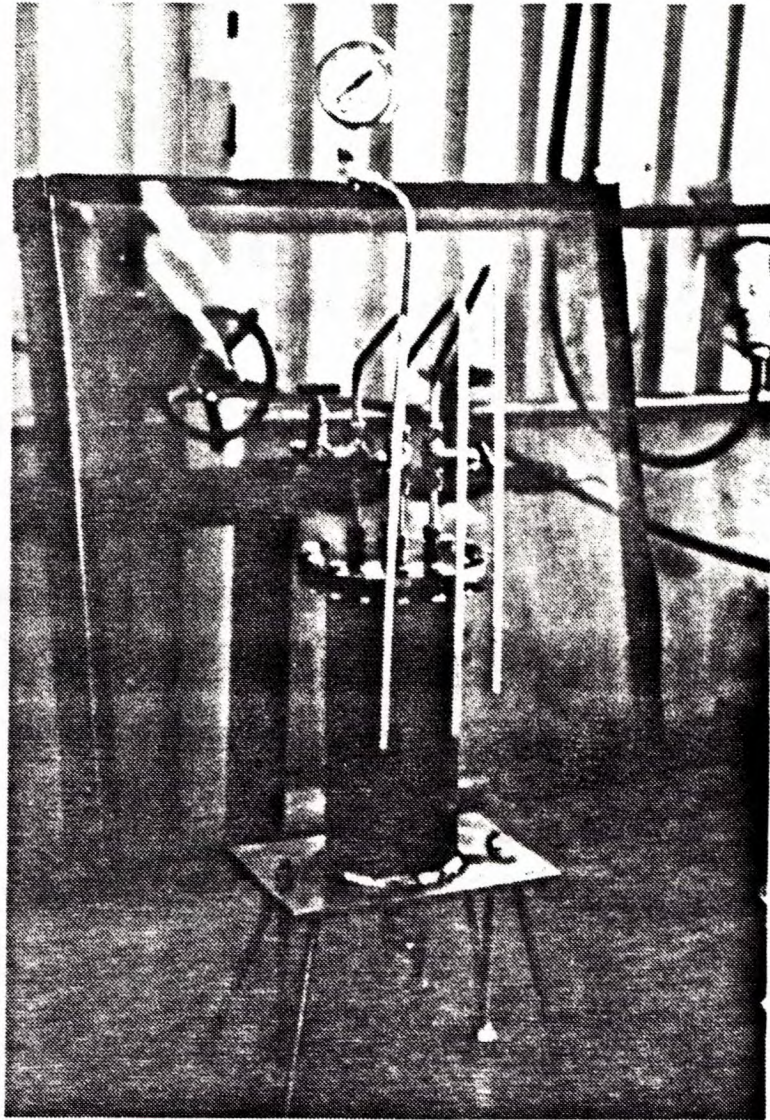


Figure 2. Schematic of vessel showing thermocouple and sampling locations.

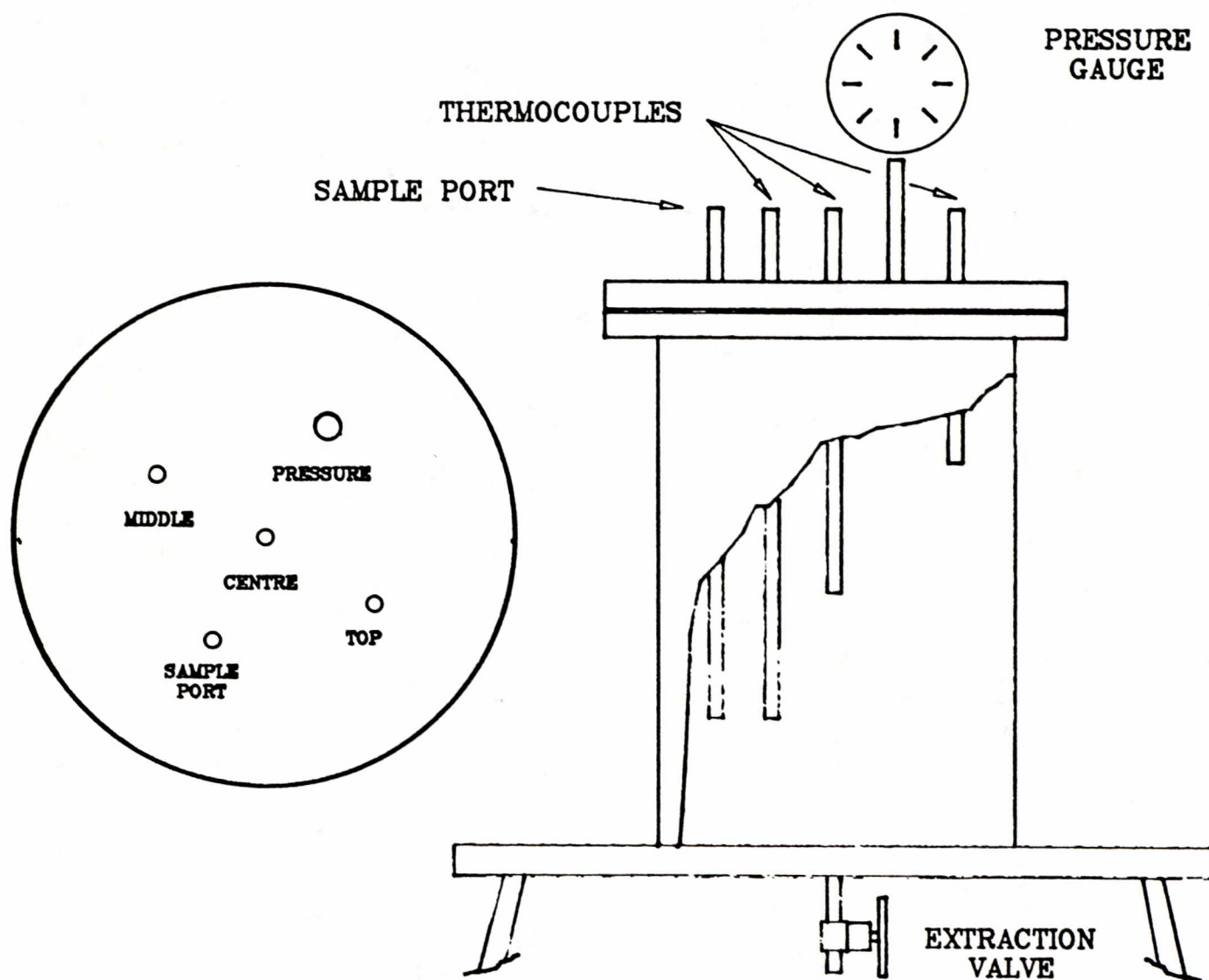
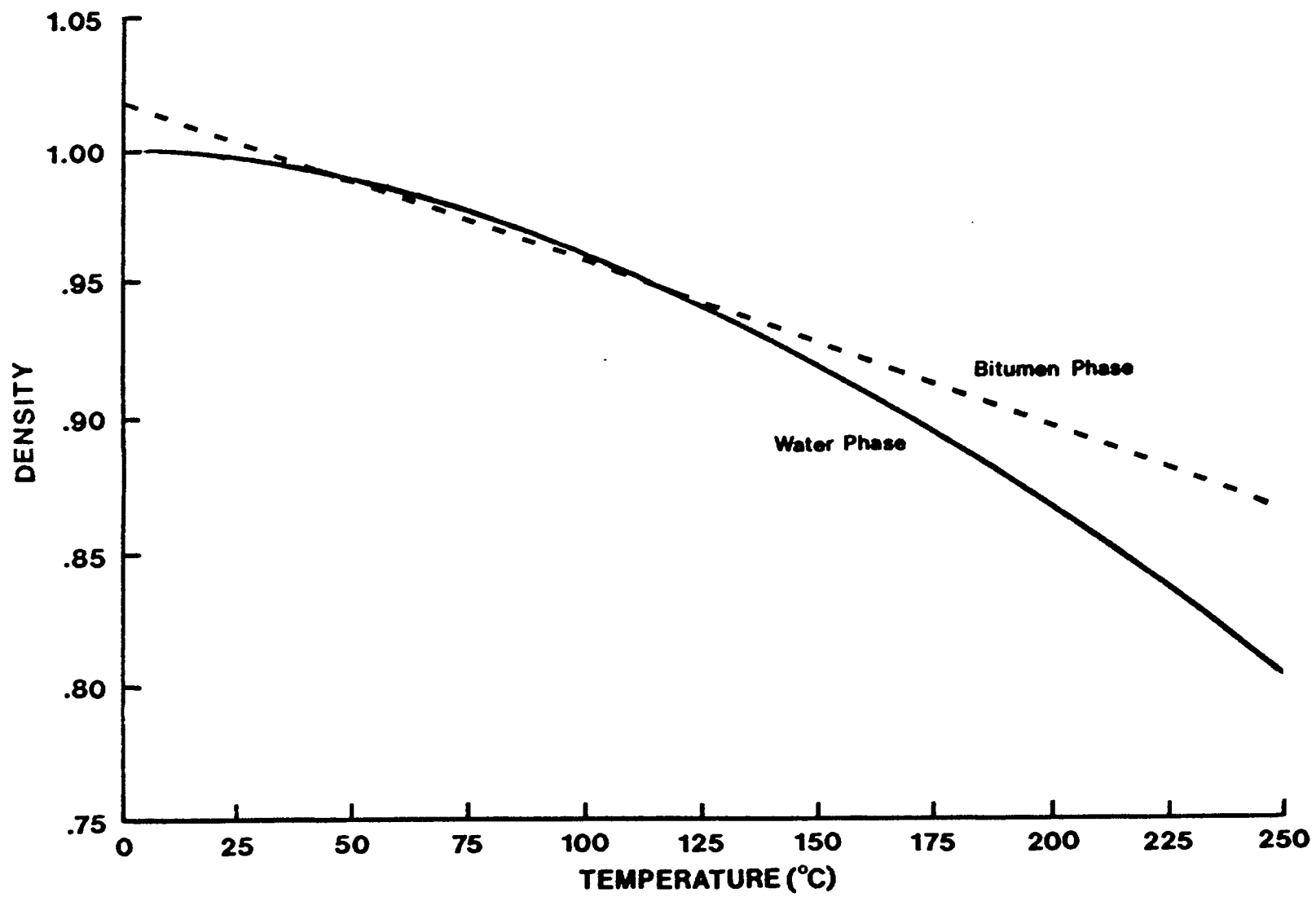


Figure 3. Density versus temperature curves for bitumen and water.





Bitumen Viscosity (cP)

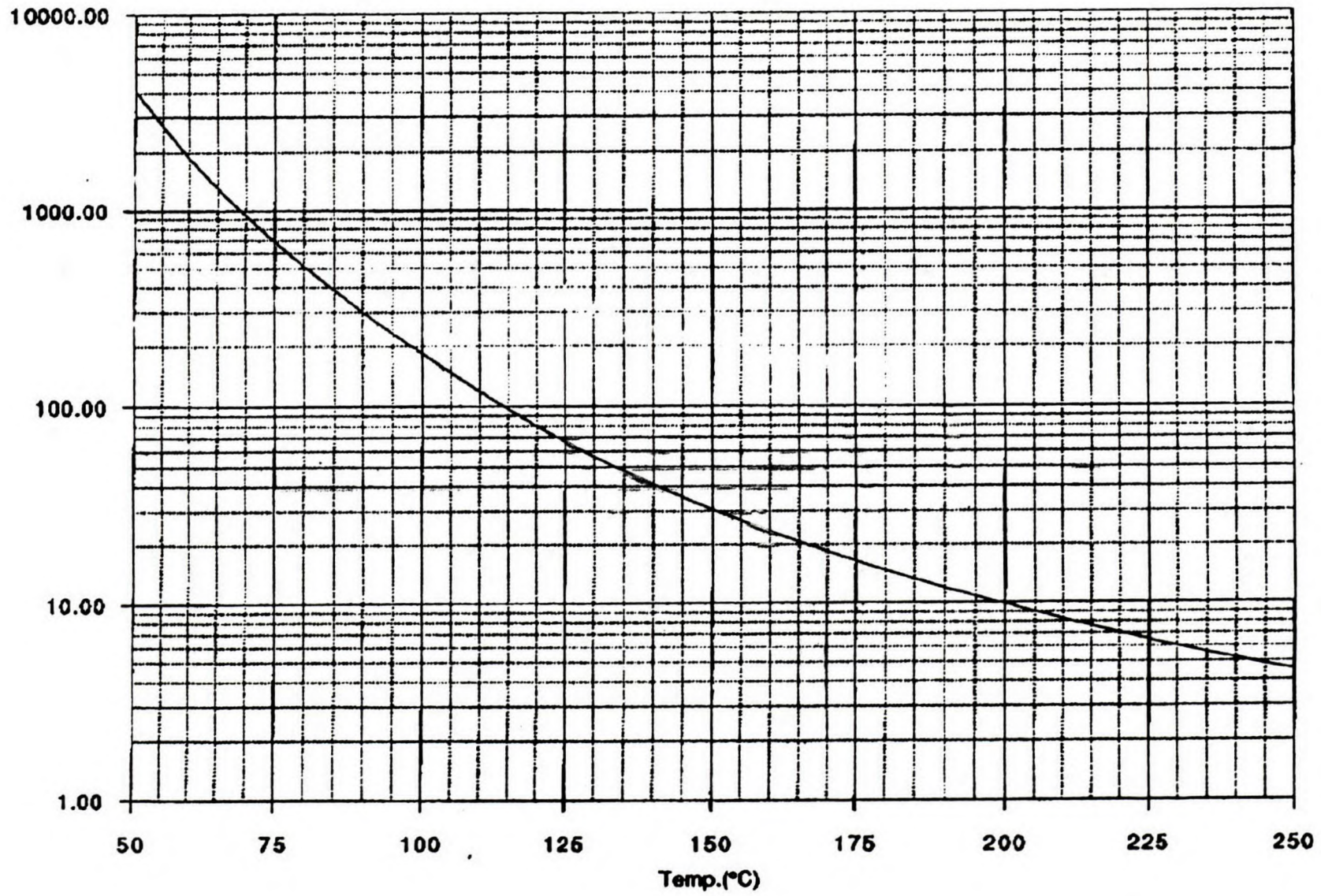


Figure 4. Viscosity versus temperature curve for bitumen.

Figure 5. Temperature profile of the four thermocouples shown in Figure 2 (typical).

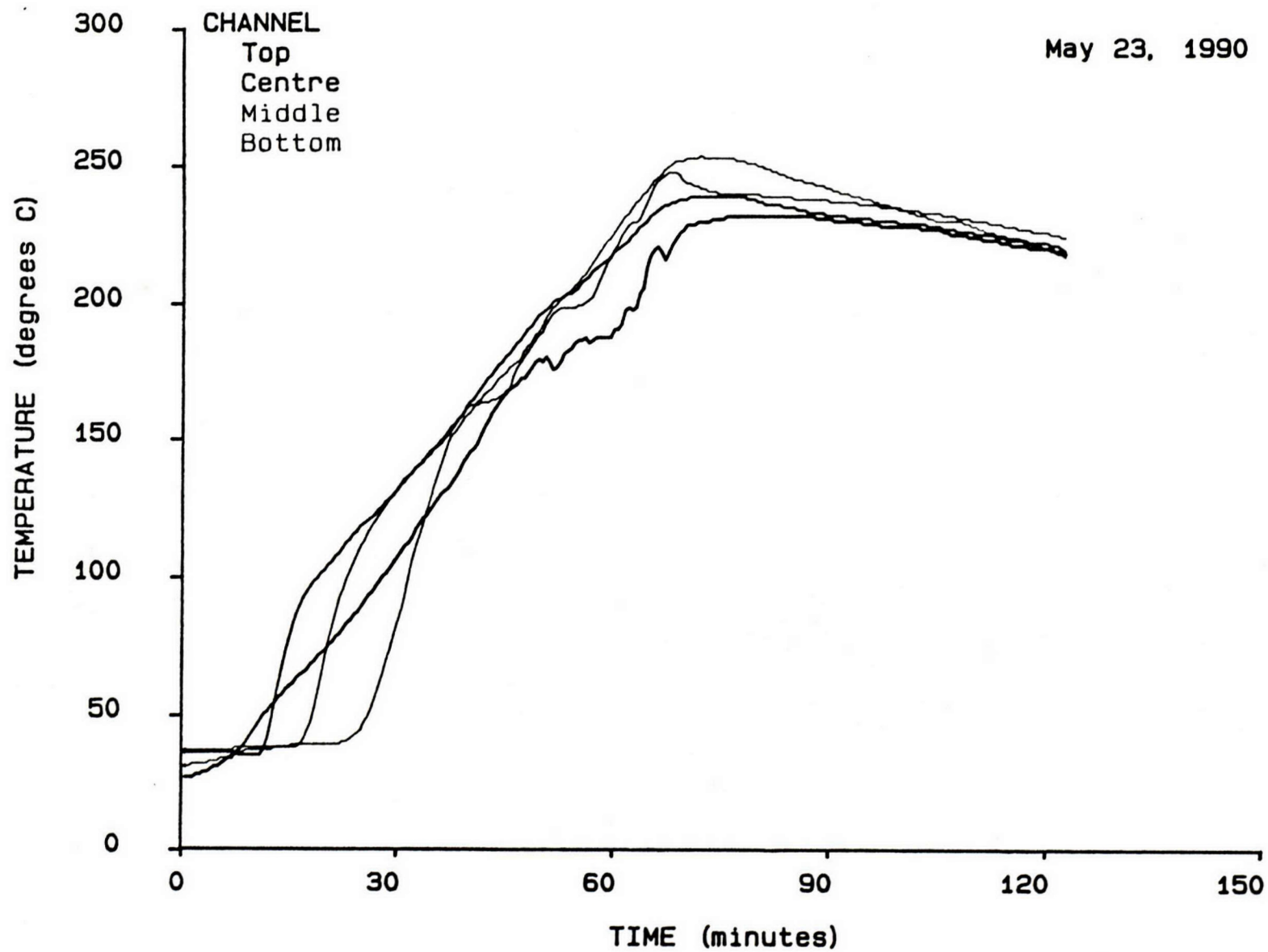




Figure 6. Dispersed oil droplet in UTF cooling coil sample showing multi-level emulsion. The arrow shows an oil in water in oil in water emulsion.

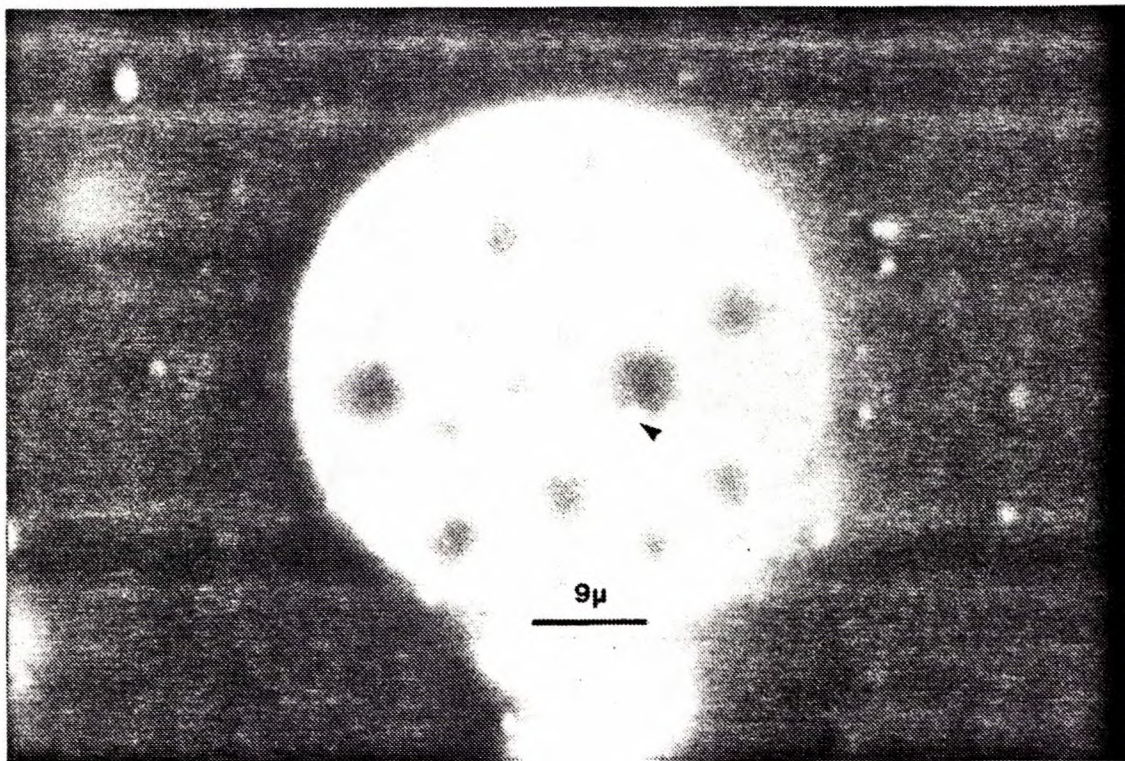
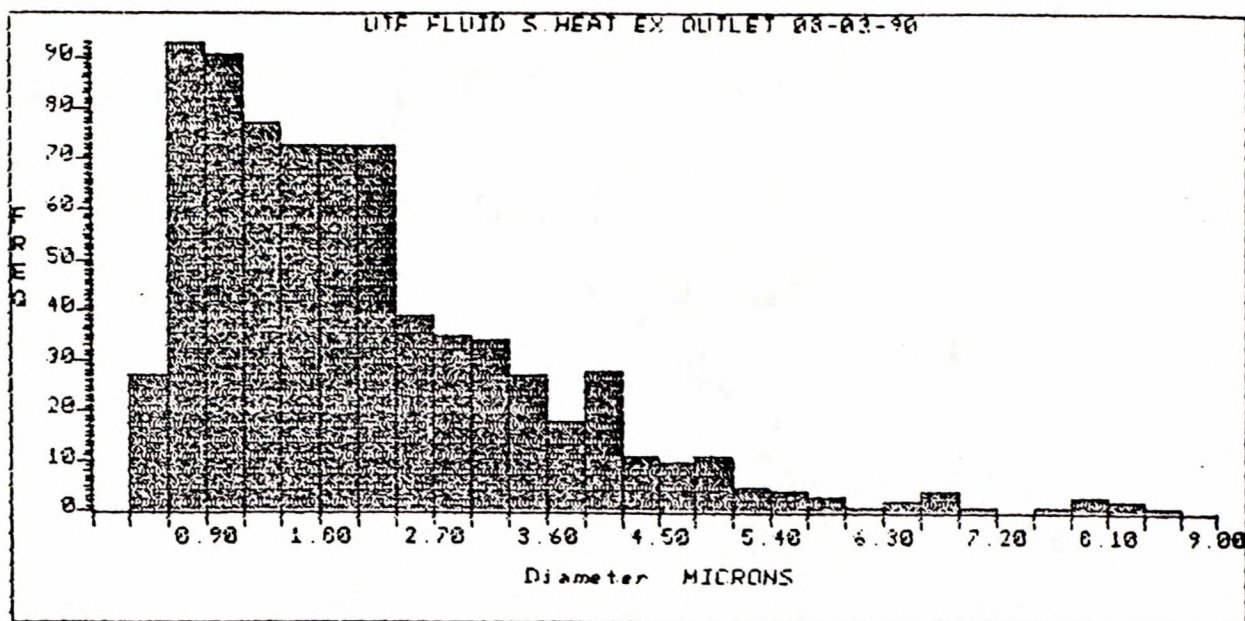




Figure 7. Histogram of the size distribution of the oil in water component of the feed emulsion (UTF heat exchanger output).

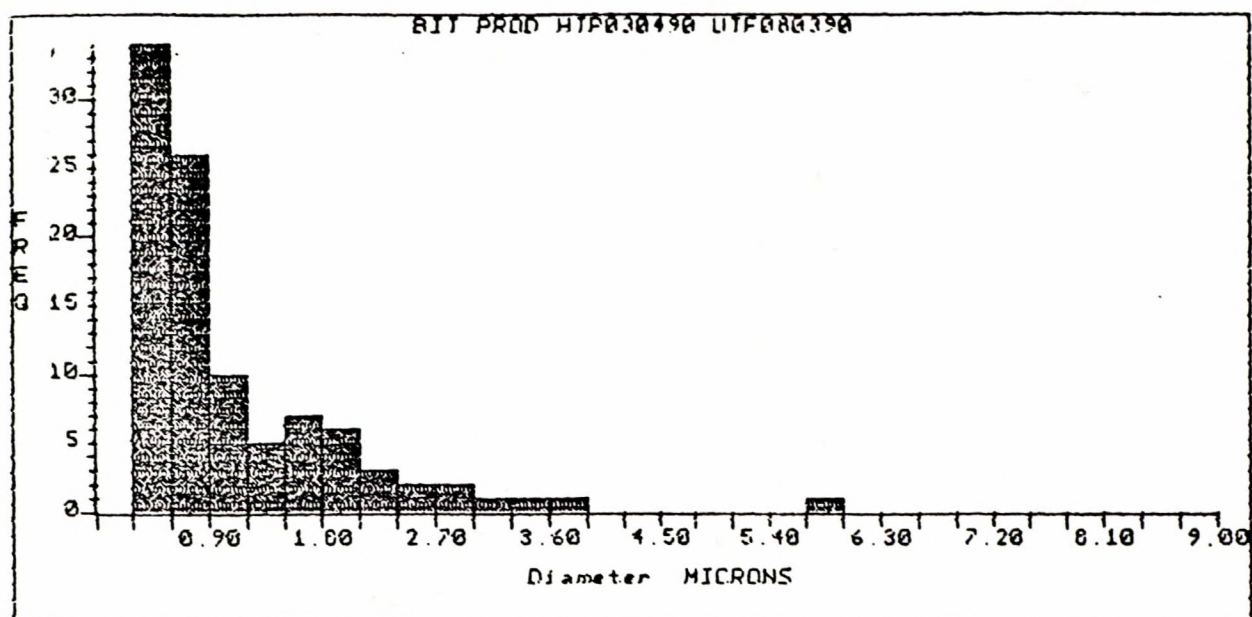


FILE NAME : FUTF000390.DAG  
 FILE LABEL : UTF FLUID HEAT EXT 08-03  
 DATE OF DATA COLLECTED: 2 Apr 90 12:58:51  
 DATE OF ANALYSIS: 2 Apr 90 13:43:13

#### SUMMARY STATISTICS

	Diameter
MEAN	2.159
STD DEVIATION	1.385
MAXIMUM	9.524
MINIMUM	0.476
VARIANCE	1.917
STD ERROR	0.002
GEOMETRIC MEAN	1.790
HARMONIC MEAN	1.492
MEDIAN	1.927
SKEWNESS	1.497
KURTOSIS	2.851
AUTO_CORRELATE	0.010
NUMBER OF DATA	747

Figure 8. Histogram of the size distribution of the water in the bitumen product from a bitumen settling experiment (15% water).



FILE NAME : BHTPUTF080390.DAG  
 FILE LABEL : BIT HTP UTF 080390  
 DATE OF DATA COLLECTED: 5 Apr 90 14:16:33  
 DATE OF ANALYSIS: 5 Apr 90 14:50:15

#### SUMMARY STATISTICS

	Diameter
MEAN	1.128
STD DEVIATION	0.861
MAXIMUM	5.744
MINIMUM	0.517
VARIANCE	0.742
STD ERROR	0.009
GEOMETRIC MEAN	0.932
HARMONIC MEAN	0.817
MEDIAN	0.741
SKEWNESS	2.437
KURTOSIS	7.801
AUTO_CORRELATE	0.055
NUMBER OF DATA	99



Figure 9. Scanning electron micrograph of the dispersed water phase in a high temperature settling experiment (1000 ppm demulsifier, 220°C, 1.6% water in the product).





Figure 10. Scanning electron micrograph of the dispersed water phase in a high temperature settling experiment (250 ppm demulsifier, 190°C, 2.1% water in the product).





Figure 11. Photograph of typical feed sample spin tests showing interface emulsion layer.

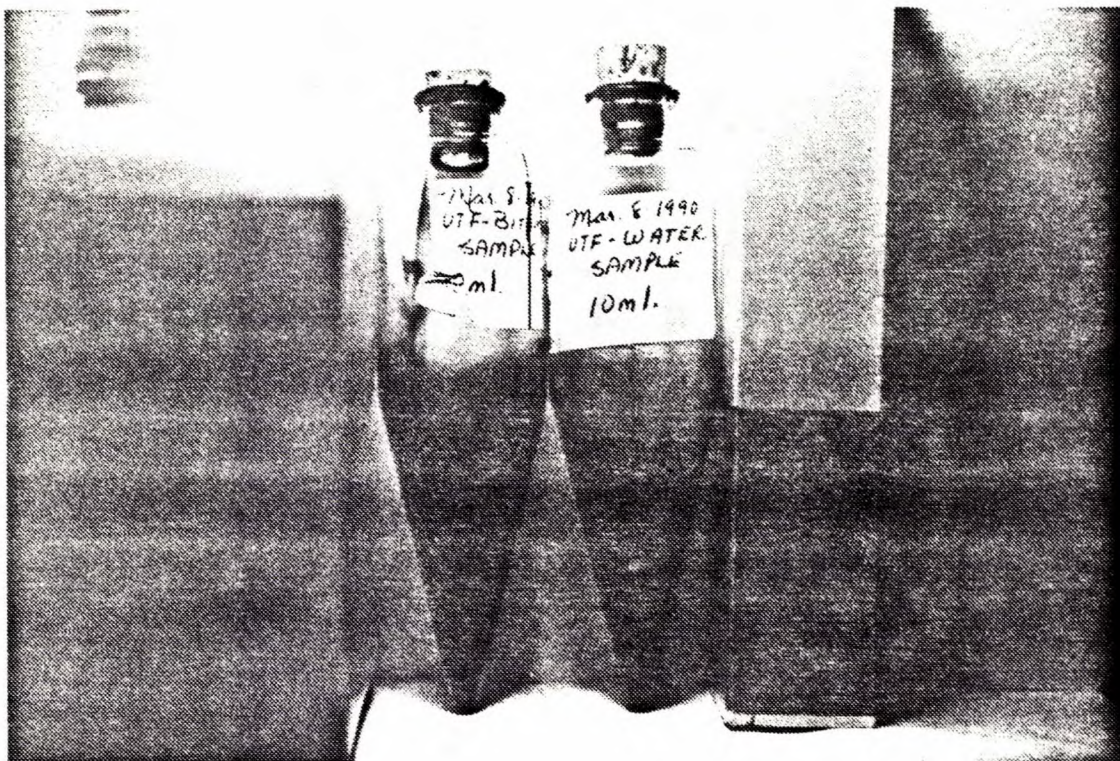
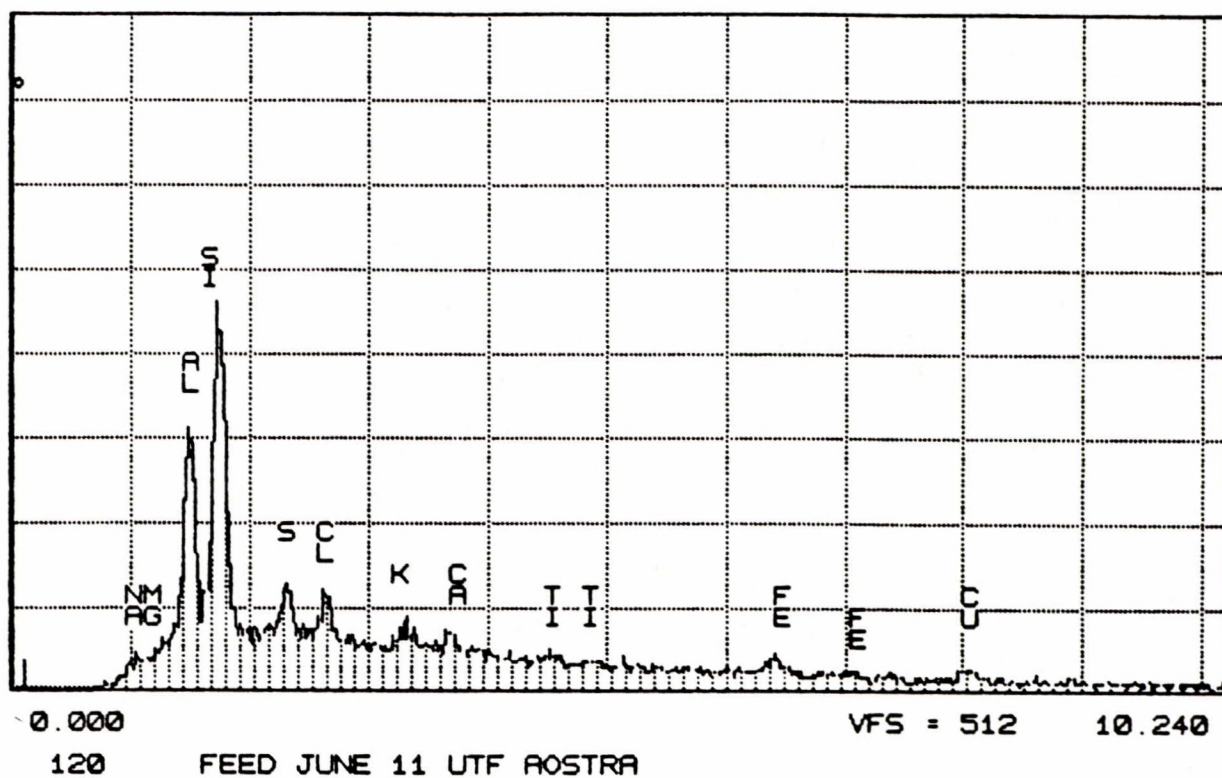


Figure 12. X-ray spectra of solids from Dean Stark extraction of the feed to the high temperature settling experiments.

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Cursor: 0.000keV = 0



## APPENDIX A

Feed: Sample received May 11, 1990. (Three 5 gallon gas cans)

## Dean Stark Test

Oil	Water	Sand
55.1%	44.7%	0.2%
53.7	46.0	0.2
53.3	46.5	0.2

## Bottle Tests.

Reagent	dose ppm	Spin Test Results	
		30 minutes	Water Clear
None		50%	0%
X-888	1000	46	26
MJ-21-6	500	52	12
	1000	55	18
MJ-21-8	500	47	15
	1000	54	8
MJ-21-7	500	45	2.5
	1000	50	2.5

Demulsifier X-888 was chosen based on the high percentage of clear water produced with separation. The water content is the water in the separated bitumen phase.

Run Date: May 23, 1990

Conditions: Heat to 210°C with 1000 ppm X-888 demulsifier. Heated barrel of reactor and bottom plate with heating tape. Four type K thermocouples measured the temperature rise (See Figure 2 for position). Sampling was from the bottom of the reactor through an ice bath and was started as soon as the reactor reached final temperature.

Input: 1771 g

Recovered: 1736g	342.7g	(19.8%)
	278.2	(16.0)
	208.4	(12.0)
	35.0	( 2.0)
	872.0	(50.2)

Number 1 Dean Stark
Number 2 Dean Stark
Number 3 Dean Stark
Spin Test samples
Residue left in reactor

#### Spin Tests

1. 1.3% water with 0.0% clear
2. 2.0% water with 0.0% clear
3. 8.6% water with 0.0% clear

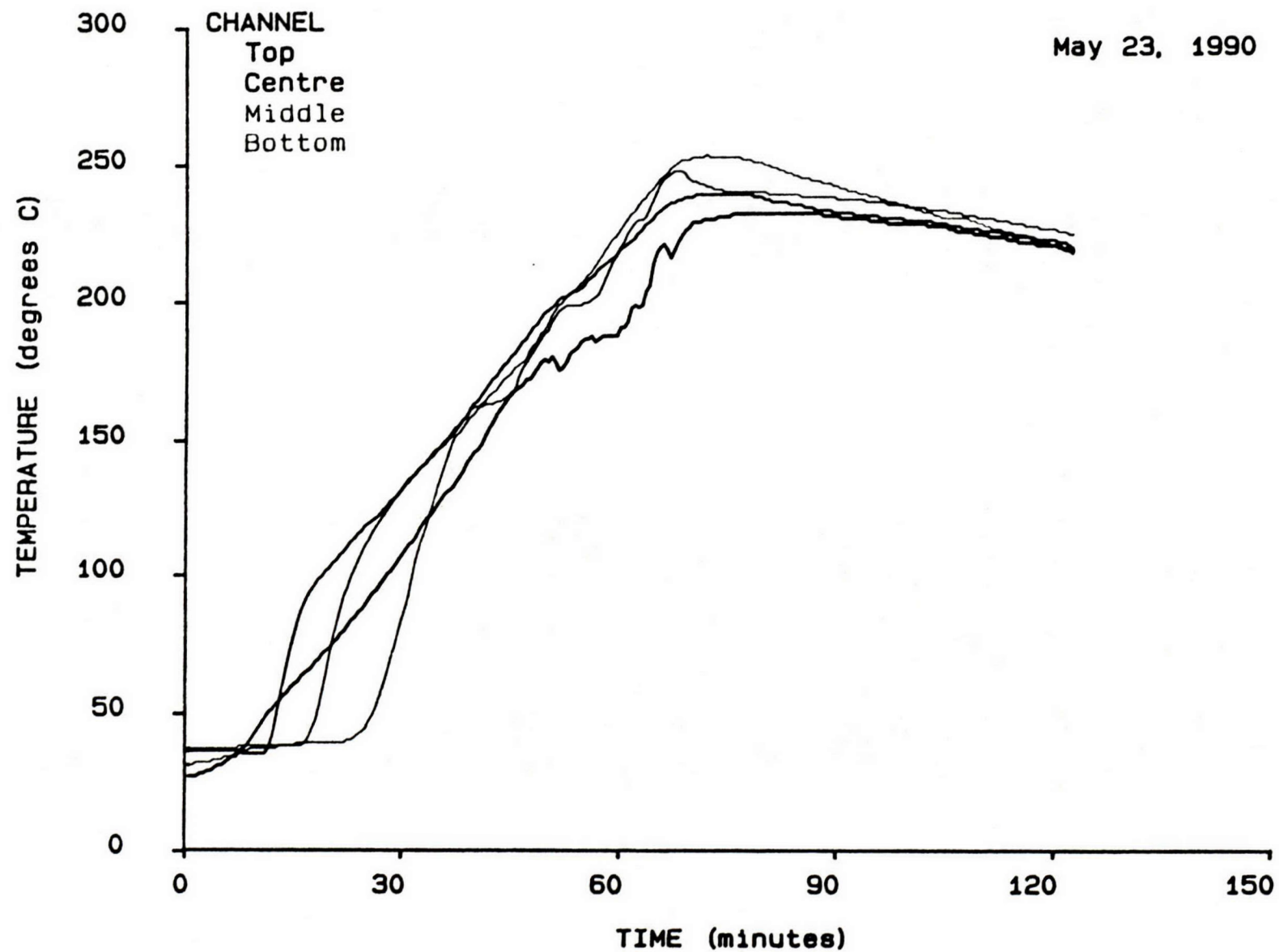
Sampled as temperature reached.  
Sampled after ten minutes  
Sampled after thirty minutes  
Water phase reached.

#### Dean Stark Analysis

	Oil	Water	Sand		Oil	Water	Sand
No.1	98.6%	1.1%	0.3%	Product	97.1	1.4	1.5
No.2	95.4	1.5	3.1	Residue	4.2	94.2	1.6
No.3	96.7	1.9	1.5	Recon Feed	49.5	48.9	1.6
Residue	4.2	94.2	1.6				

Comments: Maximum temperature reached 254°C with pressure at 460 PSI  
No water at start only oil.  
Sampled until only water was left.  
Residue density 1.000 (with hydrometer)





Run Date: May 24, 1990

Conditions: Heat to 210°C with 1000 ppm X-888 demulsifier. Heated barrel of reactor and bottom plate with heating tape. Four type K thermocouples measured the temperature rise (See Figure 2 for position). Sampling was from the bottom of the reactor through an ice bath and was started as soon as the reactor reached final temperature.

Input: 1772 g

Recovered: 1762g	318.8g	(18.1%)
	317.5	(18.0)
	183.7	(10.4)
	35.0	( 2.0)
	906.7	(51.5)

Number 1 Dean Stark  
Number 2 Dean Stark  
Number 3 Dean Stark  
Spin Test samples  
Residue left in reactor

#### Spin Tests

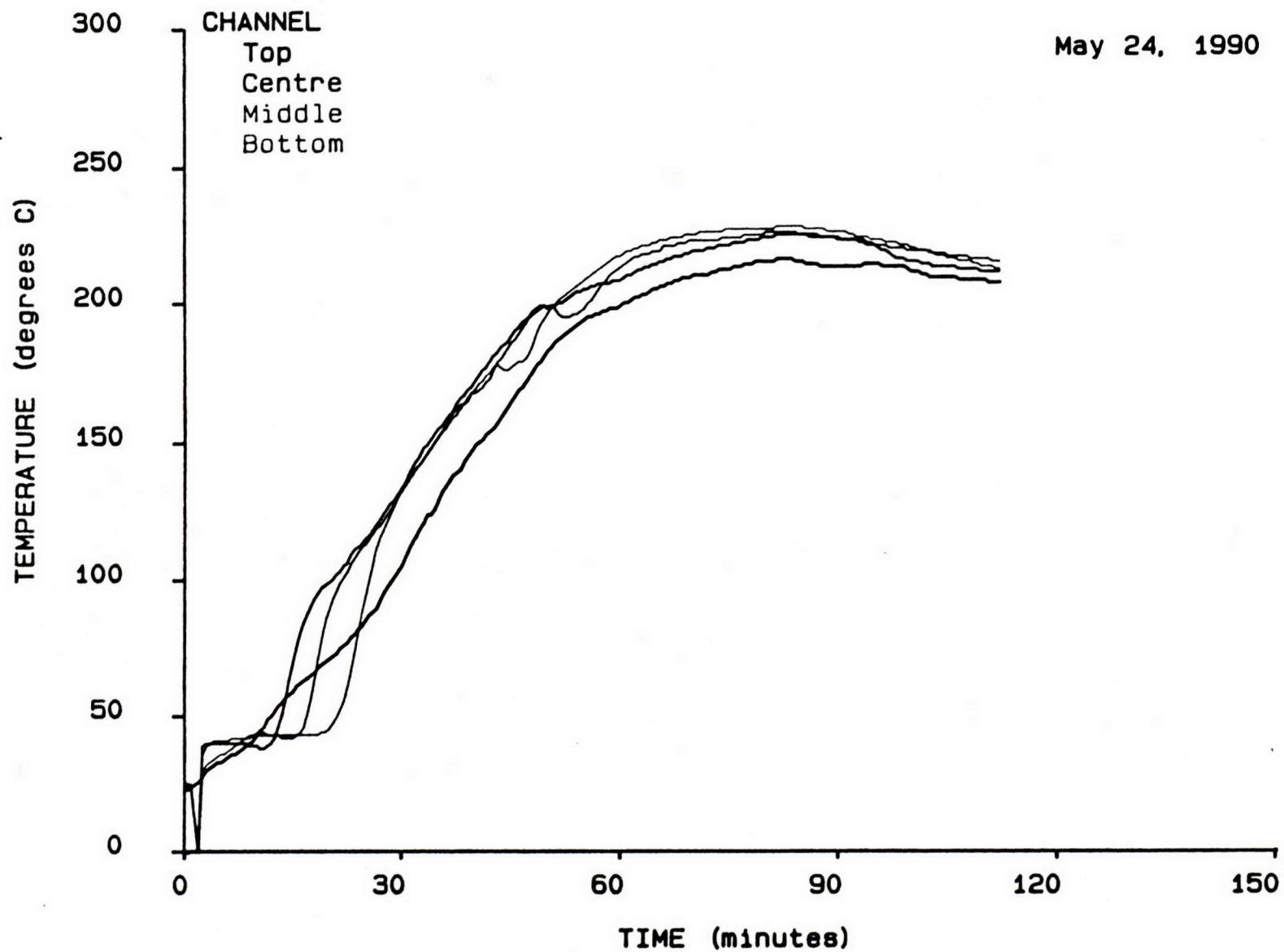
1. 1.2% water with 0.25% clear
2. 1.0% water with 0.5% clear
3. 1.0% water with 0.0% clear

Sampled as temperature reached.  
Sampled after thirty minutes  
Sampled after forty five minutes Water phase reached.

#### Dean Stark Analysis

	Oil	Water	Sand		Oil	Water	Sand
No.1	97.9%	1.6%	0.6%	Product	96.5	1.6	1.9
No.2	96.1	1.2	2.7	Residue	1.7	98.3	0.0
No.3	94.6	2.5	2.9	Recon Feed	46.7	51.4	0.9
Residue	1.7	98.3	0.0				

Comments: Maximum temperature reached 226°C with pressure at 310 PSI  
No water at start only oil.  
Sampled until only water was left.



Run Date: May 28, 1990

Conditions: Heat to 210°C without any X-888 demulsifier. Heated barrel of reactor and bottom plate with heating tape. Reactor was in vertical position. Four type K thermocouples measured the temperature rise (See Figure 2 for position). Sampling was from the bottom of the reactor through an ice bath and was started as soon as the reactor reached final temperature.

Recovered: 1613g

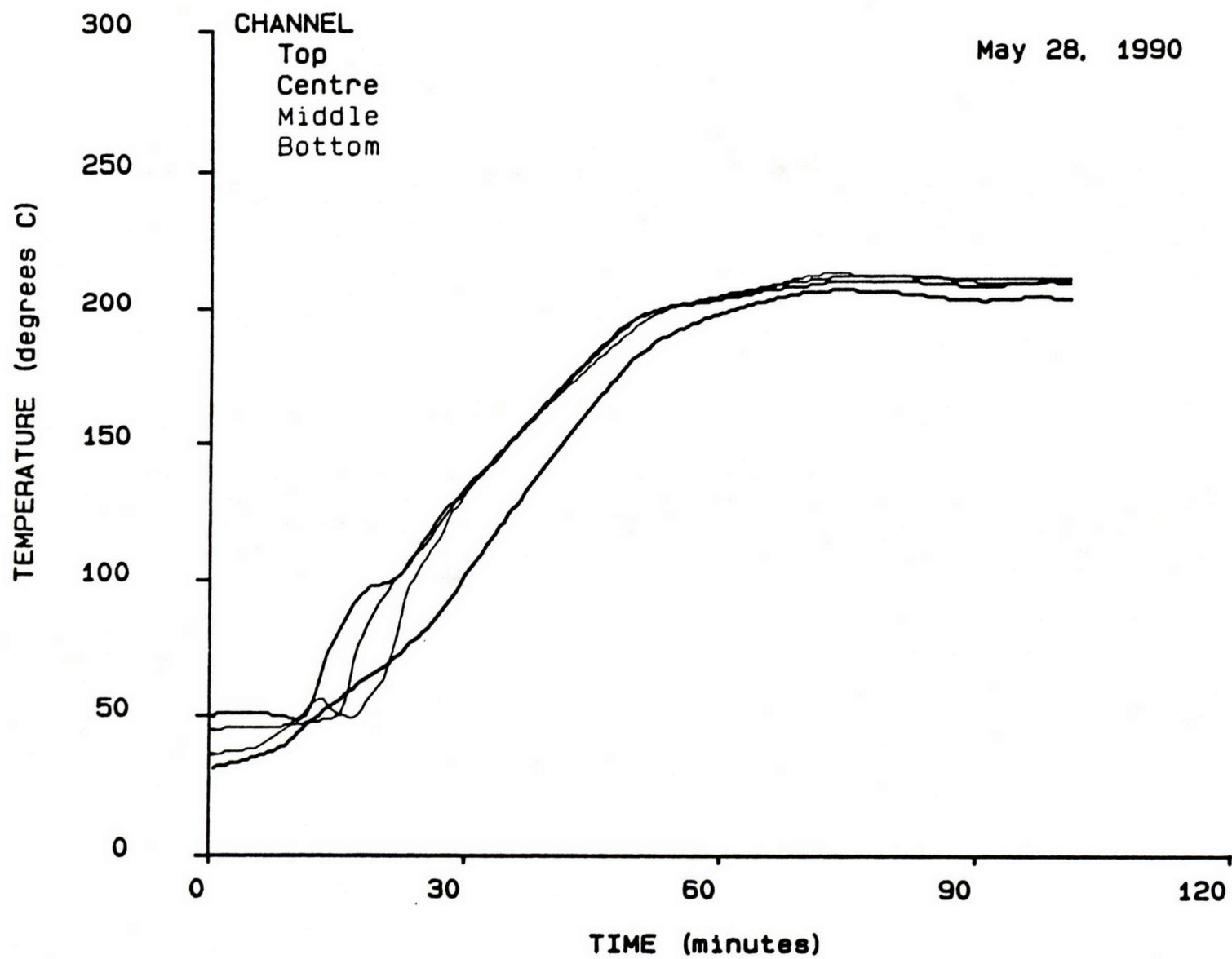
Dean Stark Test

	Oil	Water	Sand
Product	76.7%	23.1%	0.2%

Spin Tests

1.	35.0% water with 2.5% clear	Sampled after temperature reached setpoint.
2.	33.3% water with 2.5% clear	Sampled after twenty minutes

Comments: The oil was very wet and did not change. Stopped sampling after 161 g of product removed.  
One sample was kept for Dean Stark analysis from this run.  
Maximum temperature 212°C at 300 PSI



Run Date: June 15, 1990

Conditions: Heat to 190°C with 500 ppm X-888 demulsifier. Heated barrel of reactor and bottom plate with heating tape. Four type K thermocouples measured the temperature rise (See Figure 2 for position). Sampling was from the bottom of the reactor through an ice bath and was started as soon as the reactor reached final temperature.

Input: 1699 g

Recovered: 1690g    260.3g (15.4%)  
                          304.3 (18.0)  
                          220.7 (13.1)  
                          55.0 ( 3.3)  
                          849.3 (50.2)

Number 1 Dean Stark  
 Number 2 Dean Stark  
 Number 3 Dean Stark  
 Spin Test samples  
 Residue left in reactor

#### Spin Tests

1. 0.8% water with 0.8% clear
2. 0.5% water with 0.5% clear
3. 0.7% water with 0.7% clear

Sampled as temperature reached.  
 Sampled after second Dean Stark sample started  
 Sampled after third Dean Stark sample started. Water phase reached.

#### Dean Stark Analysis

	Oil	Water	Sand		Oil	Water	Sand
No.1	98.6%	1.2%	0.2%	Product	99.0	0.9	0.1
No.2	99.0	0.9	0.1	Residue	12.6	87.0	0.4
No.3	99.6	0.4	0.1	Recon Feed	54.2	45.6	0.2
Residue	12.6	87.0	0.4				

Comments: Maximum temperature reached 205°C with pressure at 200 PSI

No water at start only oil (temperature 193°C).

Sampled until only water was left.

Heating tape on barrel broke. Heated only from plate.

Run Date: June 19, 1990

Conditions: Heat to 190°C with 250 ppm X-888 demulsifier. Heated barrel of reactor and bottom plate with heating tape. Four type K thermocouples measured the temperature rise (See Figure 2 for position). Sampling was from the bottom of the reactor through an ice bath and was started as soon as the reactor reached final temperature.

Input: 1867 g

Recovered: 1833g    278.3g (15.2%)  
                          310.4 (16.9)  
                          300.8 (16.4)  
                          60.0 ( 3.3)  
                          883.8 (48.2)

Number 1 Dean Stark  
 Number 2 Dean Stark  
 Number 3 Dean Stark  
 Spin Test samples  
 Residue left in reactor

#### Spin Tests

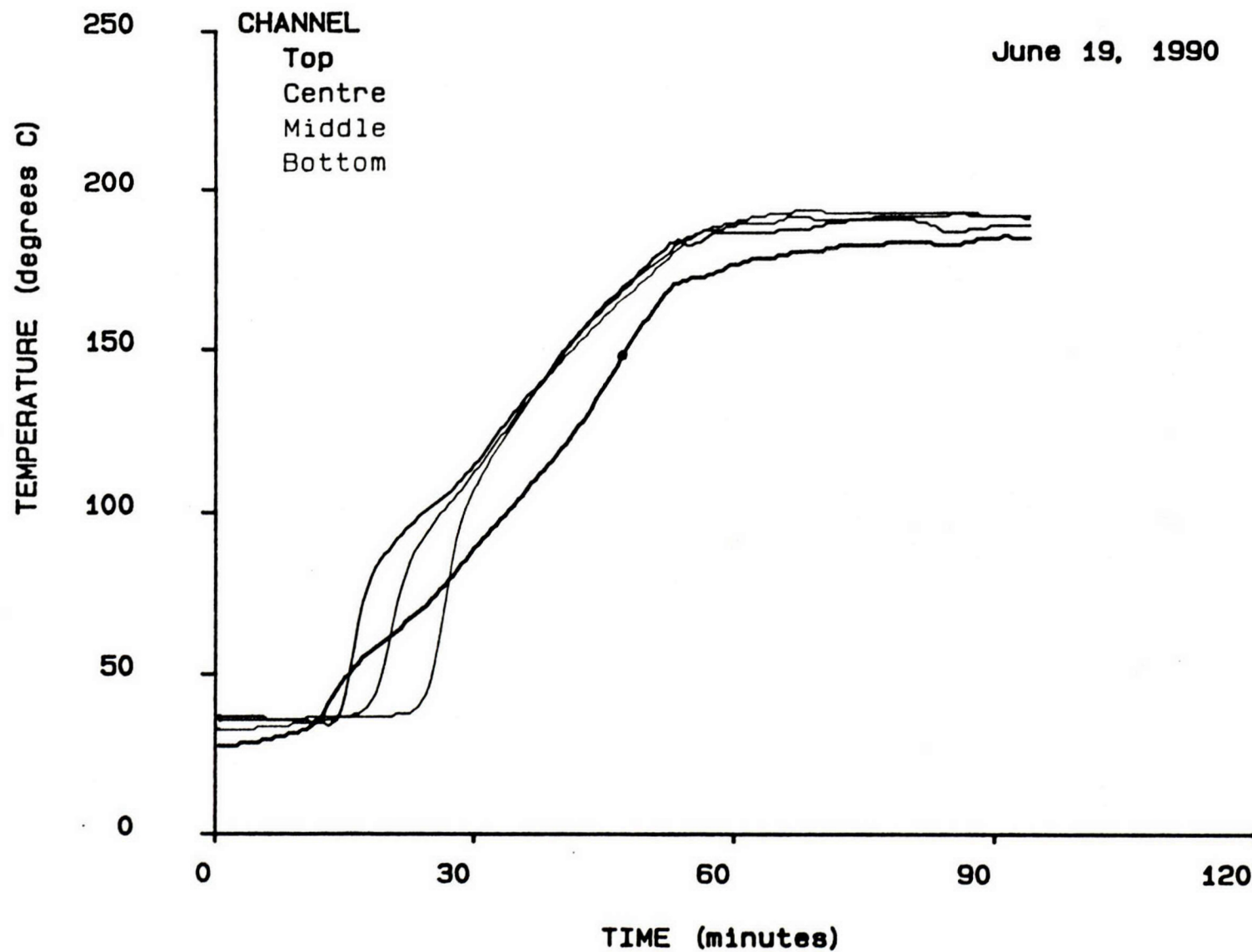
1. 5.5% water with 2.0% clear
2. 3.0% water with 2.0% clear
3. 3.0% water with 1.3% clear

Sampled as temperature reached.  
 Sampled after second Dean Stark sample started  
 Sampled after third Dean Stark sample started. Water phase reached.

#### Dean Stark Analysis

	Oil	Water	Sand		Oil	Water	Sand
No.1	97.1%	2.7%	0.2%	Product	97.8	2.1	0.1
No.2	98.8	1.1	0.1	Residue	3.2	96.4	0.4
No.3	97.3	2.7	0.1	Recon Feed	50.6	49.1	0.3
Residue	3.2	96.4	0.4				

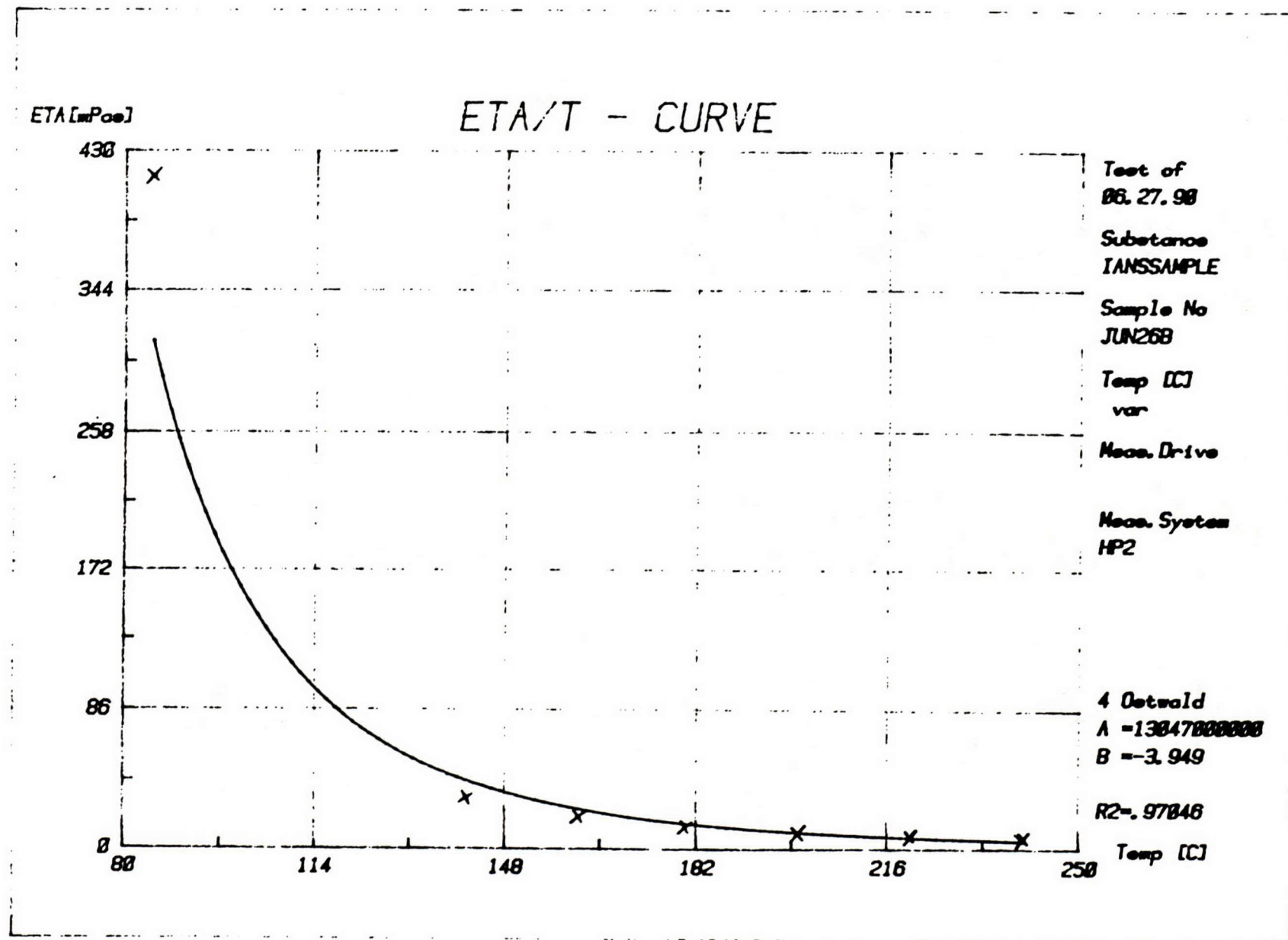
Comments: Maximum temperature reached 193°C with pressure at 205 PSI  
 No water at start only oil (temperature 190°C).  
 Sampled until only water was left.





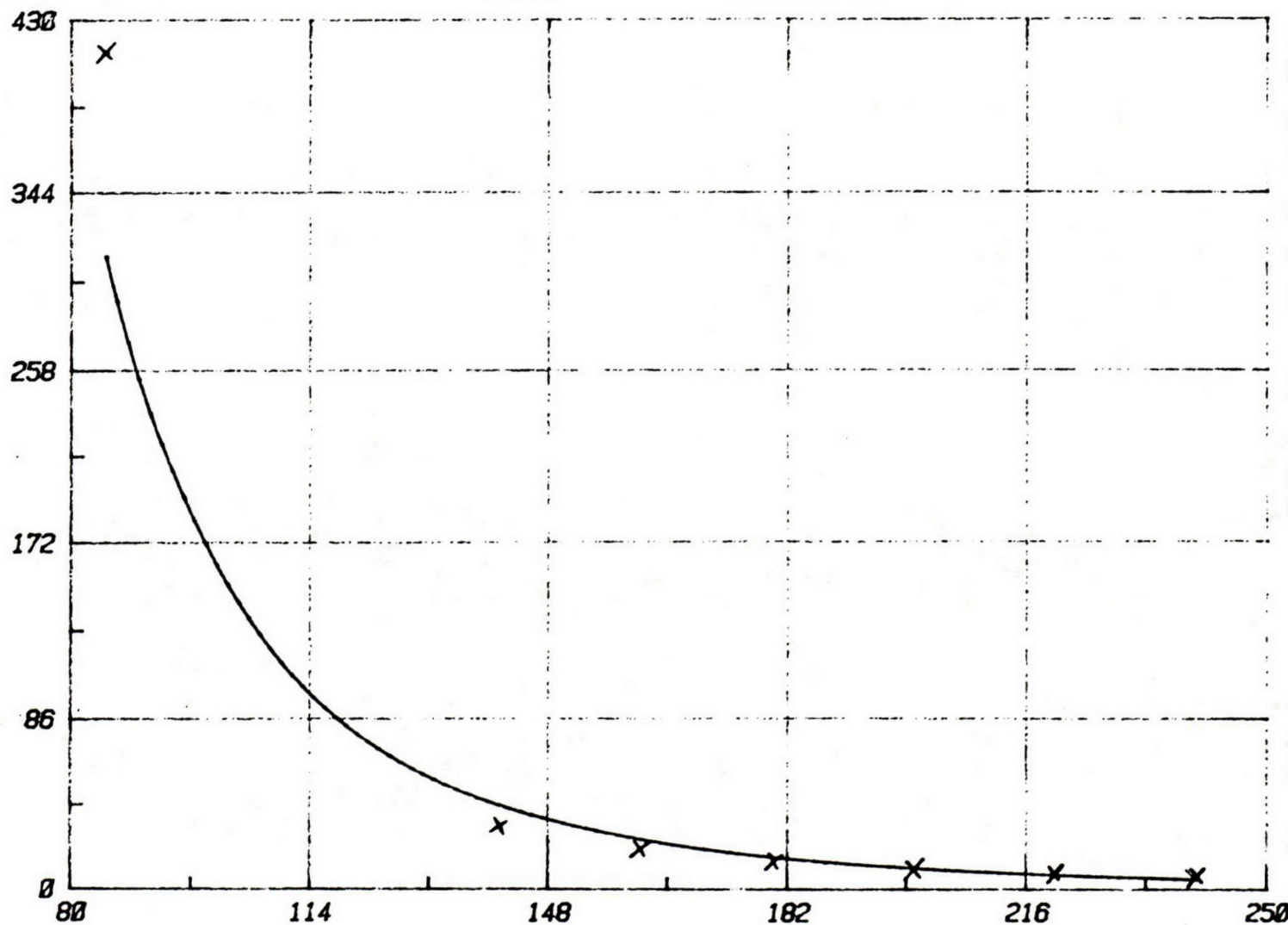
## APPENDIX B-VISCOSITY MEASUREMENTS

No.	ETA (mPas)	Temp (°C)
1	414.00	85.00
2	31.00	141.00
3	20.00	161.00
4	14.00	180.00
5	11.00	200.00
6	8.00	220.00
7	7.00	240.00



ETA[mPa.s]

# ETA/T - CURVE



Test of  
06.27.98

Substance  
IANSSAMPLE

Sample No  
JUN268

Temp [C]  
var

Meas. Drive

Meas. System  
HP2

4 Ostwald  
A = 13847000000  
B = -3.949

R2 = .97846

Temp [C]