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(54) Titre : PROCÉDE DE SEPARATION DU BITUME D'UNE MOUSSE DE SABLE BITUMINEUX AU MOYEN DE SOLVANT

(54) Title: SOLVENT PROCESS FOR BITUMEN SEPARATION FROM OIL SANDS FROTH

(57) **Abrégé/Abstract:**

A paraffinic solvent is mixed with bitumen froth containing water and solids. Sufficient solvent is added to induce inversion when the mixture is subjected to gravity or centrifugal forces. The emulsion reports to the water phase and a dry bitumen product virtually free of inorganic solids, is obtained.

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1                   **"SOLVENT PROCESS FOR BITUMEN SEPARATION**  
2                                   **FROM OIL SANDS FROTH"**

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4                                   **ABSTRACT OF THE DISCLOSURE**

5  
6                   A paraffinic solvent is mixed with bitumen froth containing water and solids.  
7                   Sufficient solvent is added to induce inversion when the mixture is subjected to  
8                   gravity or centrifugal forces. The emulsion reports to the water phase and a dry  
9                   bitumen product virtually free of inorganic solids, is obtained.

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**FIELD OF THE INVENTION**

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This invention relates to a paraffinic solvent addition method for separating water and solids from bitumen froth.

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**BACKGROUND OF THE INVENTION**

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The present invention has been developed in connection with a plant for extracting bitumen from the Athabasca oil sand deposit. At this operation, the oil sands are surface-mined and the contained bitumen is separated from the sand and recovered using what is known as the Clark hot water extraction process ("CHWE"). (The terms "oil" or "bitumen" are used interchangeably herein to identify the hydrocarbon content of oil sand.)

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The CHWE process is well known to those in the industry and is described in the patent literature. The "front end" of the process, leading up to the production of cleaned, solvent-diluted bitumen froth, will now be generally described.

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The as-mined oil sand is firstly mixed with hot water and caustic in a rotating tumbler to produce a slurry. The slurry is screened, to remove oversize rocks and the like. The screened slurry is diluted with additional hot water and the product is then temporarily retained in a thickener-like vessel, referred to as a primary separation vessel ("PSV"). In the PSV, bitumen globules contact and coat air bubbles which have been entrained in the slurry in the tumbler. The buoyant bitumen-coated bubbles rise through the slurry and form a bitumen froth. The sand in the slurry settles and is discharged from the base of the PSV, together with some water and a small amount of bitumen. This stream is referred to as "PSV underflow". "Middlings", comprising water containing non-buoyant bitumen and fines, collect in the mid-section

1 of the PSV.

2 The froth overflows the lip of the vessel and is recovered in a launder. This  
3 froth stream is referred to as "primary" froth. It typically comprises 65 wt. % bitumen,  
4 28 wt. % water and 7 wt. % particulate solids.

5 The PSV underflow is introduced into a deep cone vessel, referred to as the  
6 tailings oil recovery vessel ("TORV"). Here the PSV underflow is contacted and  
7 mixed with a stream of aerated middlings from the PSV. Again, bitumen and air  
8 bubbles contact and unite to form buoyant globules that rise and form a froth. This  
9 "secondary" froth overflows the lip of the TORV and is recovered. The secondary  
10 froth typically comprises 45 wt. % bitumen, 45 wt. % water and 10 wt. % solids.

11 The middlings from the TORV are withdrawn and processed in a series of sub-  
12 aerated, impeller-agitated flotation cells. Secondary froth, typically comprising 40 wt.  
13 % bitumen, 50 wt. % water and 10 wt. % solids, is produced from these cells.

14 The primary and secondary froth streams are combined to yield a product froth  
15 stream, typically comprising 60 wt. % bitumen, 32 wt. % water and 8 wt. % solids.  
16 This stream will typically have a temperature of 80°C.

17 The water and solids in the froth are contaminants which need to be reduced  
18 in concentration before the froth can be treated in a downstream refinery-type  
19 upgrading facility. This cleaning operation is carried out using what is referred to as a  
20 "dilution centrifuging circuit".



1 More particularly, the combined froth product is first deaerated and then  
2 diluted with sufficient solvent, specifically naphtha, to provide a solvent to froth ("S/F")  
3 ratio of about 0.45 (w/w). This is done to increase the density differential between the  
4 bitumen on the one hand and the water and solids on the other. The diluted froth is  
5 then processed in a scroll-type centrifuge, to remove coarse solids. The bitumen  
6 product from the scroll machine is subsequently processed in a disc-type centrifuge,  
7 to remove water and fine clay solids.

8 The "cleaned" bitumen product from the dilution centrifuging circuit typically  
9 contains 3 to 5 wt. % water and about 0.6 wt. % solids.

10 The underflows from the TORV, the flotation cells and the dilution centrifuging  
11 circuit are discharged as tailings into a pond system. Water is recycled from this  
12 pond for use as plant process water.

13 There are two significant problems associated with producing a cleaned diluted  
14 froth still containing such quantities of water and solids. Firstly, one is precluded from  
15 shipping the product through a commercial pipeline that is conveying discrete  
16 shipments of a variety of hydrocarbon products. Such pipelines require that any  
17 product shipped must contain less than 0.5 wt. % B S and W (bottom settlings and  
18 water). Because of this requirement, one must upgrade the cleaned diluted froth  
19 produced by the dilution centrifuging circuit in a refinery-type upgrading circuit located  
20 close to the mining site, before shipping it. Providing and operating an upgrading  
21 circuit at the mine site is very expensive. Secondly, there is a build-up in the  
22 concentration of chlorides in plant process water that occurs over time. This build-up  
23 arises from recycling water from the tailings pond to the tumbler and re-using the  
24 tailings water as part of the water used as process water. In addition, the incoming oil

1 sands contain salt which adds to the chloride content in the process water. Keeping  
2 in mind that the cleaned diluted bitumen product from the dilution centrifuging circuit  
3 contains a significant fraction of plant water, chlorides are brought by this fraction into  
4 the upgrading circuit. These chlorides are harmful in the upgrading circuit, as they  
5 cause corrosion and catalyst fouling.

6 The industry has long understood that it would be very desirable to produce a  
7 dry diluted bitumen froth product containing less than about 0.5 wt. % water plus  
8 solids. Stated alternatively, it would be desirable to separate substantially all of the  
9 water and solids from the froth.

10 Many potential solutions have been explored. These have included  
11 electrostatic desalting, water-washing, chemicals addition, third stage centrifuging and  
12 high temperature froth treatment. However, no effective and practical technique has  
13 yet emerged which would produce dry bitumen with little accompanying bitumen loss  
14 with the water.

15 There are various reasons why no successful technique has yet been devised  
16 for cleaning bitumen froth to reduce the water plus solids content below 0.5 wt. %.  
17 The major reason is that the water remaining in naphtha-diluted bitumen froth is finely  
18 disseminated in the bitumen as globules having a diameter of the order of 3 microns  
19 or less. The mixture is an emulsion that tenaciously resists breakdown.

1 In this background, only the CHWE process has been mentioned. There are  
2 other water extraction processes - such as the known OSLO process, the Bitumen  
3 process, and the Kryer process - which also produce bitumen froth which can be  
4 cleaned by this invention.

5 With this background in mind, it is the objective of the present invention to  
6 provide a new method for cleaning bitumen froth, produced by a water extraction  
7 process, which method is effective to better reduce the water plus solids content,  
8 preferably to about 0.5 wt. % or less.

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### 10 **SUMMARY OF THE INVENTION**

11 The present invention is directed toward the breaking of the water emulsion in  
12 bitumen froth. The invention is based on the discovery that a paraffinic solvent, if  
13 added to the bitumen froth in sufficient amount, causes an inversion of the emulsion.  
14 That is, the emulsion, a complex mixture of water, bitumen, solvent and solids, which  
15 is initially in the hydrocarbon phase, is transferred into the aqueous phase. As a  
16 result of the inversion, contained water effectively separates from the diluted froth  
17 under the influence of gravity or centrifugal forces. The product is essentially dry  
18 diluted bitumen, preferably having a solids and water content less than 0.5 wt. %.  
19 (This product is hereafter referred to as dry bitumen.) This product is better suited for  
20 upgrading as it is reduced in chloride content, relative to the product of the prior art. It  
21 also meets pipeline requirements as to water plus solids content.

22 It is believed that the water globules agglomerate in the presence of the critical  
23 concentration of the paraffinic solvent and acquire the capacity to segregate from the  
24 hydrocarbon.



1 In a preferred embodiment, the invention involves a method for cleaning  
2 bitumen froth containing water and particulate solids contaminants, said froth having  
3 been produced by a water extraction process practised on oil sands, comprising:  
4 adding paraffinic solvent to the froth in sufficient amount to produce a solvent to froth  
5 ratio ("S/F") of at least 0.6 (w/w); mixing sufficiently to disperse the solvent in the  
6 bitumen; and subjecting the mixture to gravity or centrifugal separation for sufficient  
7 time to reduce its water plus solids content to less than about 0.5 wt %. Most  
8 preferably the solvent used is a mixture of low molecular weight alkanes with chain  
9 lengths from about C<sub>5</sub>-C<sub>16</sub>, such as natural gas condensate, added in sufficient  
10 amount to produce a solvent to froth ratio of about 1.0 (w/w).

11 The invention is characterized by the following advantages:

- 12 • substantially all of the water and solids can be removed from the froth  
13 by diluting it with sufficient paraffinic solvent;
- 14 • bitumen losses with the separated water are only slightly lower than for  
15 the conventional process;
- 16 • unless the amount of solvent added is high enough to cause  
17 asphaltenes to precipitate, the asphaltene content in bitumen lost with  
18 the water is no higher than that normally associated with bitumen - thus  
19 the lost bitumen can be recovered from the water using conventional  
20 techniques; and
- 21 • the new method has been shown to be effective at relatively low  
22 temperatures (40 - 50°C), which raises the possibility that the extraction  
23 process can be run at lower temperatures.

1           The method of this invention involves the mixing of the solvent with the  
2 bituminous froth in a vessel for a sufficient time to ensure the complete dispersion of  
3 the solvent into the froth. Normally, this can be carried out in a stirred tank with a  
4 nominal retention time of 5 minutes. The separation itself can be carried out in the  
5 same vessel by stopping the agitation and permitting the water droplets to separate  
6 under the influence of gravity. In a continuous process, the separation can be  
7 conducted in a separate settling vessel which is connected by piping to the mixing  
8 vessel.

9           Broadly stated, the invention is directed to a method for cleaning bitumen froth  
10 containing water and particulate solids contaminants, said froth having been  
11 produced by a water extraction process practised on oil sand, comprising:

12           adding a sufficient amount of paraffinic solvent to the froth to induce inversion;  
13           mixing the froth and the solvent for a sufficient time to disperse the solvent in  
14 the froth;

15           subjecting the mixture to gravity or centrifugal separation for a sufficient period  
16 to separate substantially all of the water and solids from the bitumen to produce dry  
17 diluted bitumen; and

18           pumping the dry diluted bitumen through a pipeline to an upgrading circuit.

19           In another aspect the invention is directed to a method for delivering oil sand-  
20 derived bitumen through a pipeline, comprising:

21           a) subjecting the oil sand to a water extraction process to obtain a bitumen  
22 froth, the bitumen froth containing water and particulate solids contaminants;

23           b) cleaning the bitumen froth to remove water and particulate solids  
24 contaminants, the cleaning process comprising:

1 adding a sufficient amount of paraffinic solvent to the froth to induce inversion;  
2 mixing the froth and solvent for a sufficient time to disperse the solvent in the  
3 froth;  
4 subjecting the mixture to gravity or centrifugal separation for a sufficient period  
5 to separate substantially all of the water and solids from the bitumen to produce dry  
6 diluted bitumen; and  
7 c) pumping the dry diluted bitumen through a pipeline to an upgrading circuit.  
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### 9 **DESCRIPTION OF THE DRAWINGS**

10 Figure 1 is a plot showing the residual water content remaining in the oil phase  
11 over time in a gravity settling test where the bitumen froth has been diluted with  
12 various solvents at conditions which are conventional: 80°C, S/F ratio 0.45 w/w. The  
13 Plant 7 naphtha represents the conventional solvent used in the commercial plant  
14 owned by the present assignees;

15 Figure 2 is a plot similar to Figure 1, showing the residual water content  
16 remaining in the oil phase over time in a gravity settling test for runs conducted at the  
17 same conditions as those of Figure 1, except that the S/F ratio was increased to 0.91  
18 - of significance is the elimination of water from the oil phase at this S/F ratio when  
19 heptane is the solvent used;

20 Figure 2a is a plot showing the residual water content remaining in the  
21 hydrocarbon phase after treatment of bitumen froth with paraffinic solvents of different  
22 molecular weights, ranging from butane to heptane at different solvent/froth ratios;



1 Figure 2b is a plot comparing the impact of three types of impurities (olefins,  
2 naphthenes and aromatics) on the ability of a pure paraffin (Heptane) to produce dry  
3 bitumen;

4 Figure 3 is a plot showing the residual water content remaining in the oil phase  
5 after 30 minutes of settling time for runs using heptane as the solvent at different S/F  
6 ratios. Conditions: centrifuging at 2000 rpm for 10 mins., 80°C - the results indicate  
7 that inversion occurred at a S/F ratio of about 0.75 - 0.80;

8 Figure 4 is a plot showing the residual water content remaining in the oil phase  
9 over time in a gravity settling test using: (a) natural gas condensate ("NGC") as the  
10 solvent for runs at different S/F ratios, and (b) the results of a single run using Plant 7  
11 naphtha as the solvent at a high S/F ratio - of significance is the inversion for NGC at  
12 an S/F ratio of about 1.00 to 1.20.

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#### **DESCRIPTION OF THE PREFERRED EMBODIMENT**

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A comparative testing program was undertaken under laboratory conditions.

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Different solvents were added to bitumen froth as diluents. The solvents varied in

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aromatic and paraffin contents. Various solvent/froth ratios were tried for each

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diluent. Various temperatures were tried. After adding the solvent, the diluted froth

19

was centrifuged or gravity settled and the residual water, chloride and solids contents

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in the bitumen fraction were determined. The resulting data were then assessed.

21

In the course of the testing, certain discoveries were made, as described

22

below. The inventive process is based on these discoveries.



1 More particularly, the test program involved the following materials and  
2 procedures:

3 A single froth was used for all of the test runs. This froth assayed as follows:

4	oil (or bitumen)	-	66.22 wt. %
5	water	-	24.59 wt. %
6	solids	-	9.65 wt. %

7 The solvents used in the test are set forth in Table 1.

Table 1

## Solvents Used For Water Removal Studies From Froth

	Class.**	Solvent	Source	Aromatics Content (%)	b.p. (°C)	Density (g/ml)
1						
2						
3						
4	A	Pt. 7 Naphtha	SCL Pt. 7	~15%	82 ~ 171	0.770
5	A	Aromatic naphtha	Esso	~98%	143 ~ 186	0.872
6	A	Toluene	Fisher Sci.	100%	111	0.866
7	A	Xylene	Fisher Sci.	100%	139	0.868
8	P	Hexane	Fisher Sci.	0%	69	0.664
9	P	Heptane	Fisher Sci.	0%	98	0.684
10	P	i-Octane	Fisher Sci.	0%	100	0.688
11	P	Hexadecane	Fisher Sci.	0%	287	0.773
12	P	Bayol 35(Light paraff.oil)*	Fisher Sci.	very low	light	0.780
13		Cyclohexane	Fisher Sci.	0%	81	0.779
14		Cyclohexane	Fisher Sci.	0%	83	0.810

15 \* indicates a trade mark

16 \*\* A - designates an "aromatic" or non-paraffinic solvent

17 P - designates a "paraffinic" solvent

1 The solvent used in applicants' commercial operation is referred to as Plant 7  
2 naphtha. This solvent is applied in the plant with a solvent/froth ratio of about 0.45. It  
3 will be noted that Plant 7 naphtha has an aromatics content of approximately 15%.

4 Water contents in solvent-diluted bitumen and settled water samples were  
5 determined by Karl-Fischer titration.

6 The procedure for the gravity settling runs was as follows, unless otherwise  
7 described. Bitumen froth and diluent samples were separately placed into a water  
8 bath operated at the temperature desired for the run. Once at temperature, samples  
9 of froth and diluent were weighed out, to yield the desired solvent/froth ratio for the  
10 run, and combined in a 32 ounce mixing jar. The diluent and froth in the jar were  
11 mixed at 500 rpm for 10 minutes using a blade mixer.

12 Upon completion of mixing, the mixture was allowed to stand in the jar in the  
13 bath to effect gravity settling.

14 Samples were taken at 0, 5, 15, 30, 60, 90 and 120 minute intervals. The  
15 location of the sampling point was about the mid-point of the hydrocarbon fraction.  
16 The collected samples were analyzed for water content.

17 Two samples of diluted bitumen product were collected from each run after  
18 120 minutes of settling. One was analyzed for chloride content; the other was  
19 analyzed for solids content.

20 The procedure for the centrifuging runs was as follows, unless otherwise  
21 described. The bitumen froth and diluent samples were pre-heated to the run  
22 temperature in a water bath. Once at temperature, samples of froth and diluent were  
23 weighed out, to yield an 80 ml sample having the desired solvent/froth ratio, and  
24 transferred into a 125 ml glass jar.

25 The glass jar was placed in a shaker and shaken rigorously for 5 minutes, to  
26 mix the components.

1           The mixture was then introduced into a 100 ml centrifuge tube and spun at  
2 2000 rpm for 10 minutes.

3           After centrifuging, two diluted bitumen product samples were taken. One  
4 sample was analyzed for water content. The other was analyzed for chloride content.

5           In the Examples below, solids content of the product bitumen was analyzed by  
6 the Dean Stark method. This involves placing the samples in a porous thimble in a  
7 distillation apparatus with toluene. The sample is washed by refluxing hot toluene to  
8 dissolve all of the "bitumen" which accumulates in the still pot. Water condenses  
9 overhead and is trapped in a "measuring boot" in the condenser. "Solids" remain  
10 behind in the porous thimble, and are weighed after the distillation is complete.

11           Samples of product bitumen from the Paraffin Froth Treatment process which  
12 have been subjected to the Dean Stark analysis generally contained in the range of  
13 0.00% - 0.15% (w/w) solids. Conventional coker feed bitumen contains  
14 approximately 1% (w/w) solids.



1 Example I

2 In this test, a group of solvents were tested at a S/F ratio of 0.45 (w/w), to  
3 assess their capability to remove froth water with gravity settling. The test was run at  
4 80°C. The solvents are described in Table I and identified in Figure 1.

5 As previously stated, the S/F ratio of 0.45 is that used in the commercial plant  
6 dilution centrifuging circuit. Plant 7 naphtha is the solvent used in the circuit. The test  
7 Temperature (80°C) is the same as that used in the plant circuit.

8 The results are tabulated in Table 2 and presented in Figure 1.

9 As shown, the solvents with high aromaticity gave equivalent or better water  
10 removal when compared to the paraffinic solvent-heptane, at this S/F ratio.

11 In all of the runs, the residual water content in the diluted bitumen product after  
12 120 minutes of settling was still in excess of 3%.

13 In summary, at the conventional S/F ratio, the aromatic solvents were as good  
14 at inducing water separation as the paraffinic solvent; none of the solvents reduced  
15 the water content below 3%.

TABLE 2  
Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C  
Using Different Solvents at S/F Ratio = 0.45

1	2	3	4	5	6	7	8	9	10	11	12	13	14
Settling time	Heptane	Pt.7 naphtha	Tol/Hep = 1/1 Toluene	Xylene	Water Content in Oil Phase (%)								
mins													
0	15.67	14.81	14.67	14.36	13.50								
5	5.93	5.84	5.24	4.95	4.69								
15	5.35	5.25	5.14	4.05	4.51								
30	5.16	4.93	4.82	4.05	4.23								
60	4.48	4.36	4.28	4.08	4.00								
90	4.33	4.22	4.15	4.07	3.82								
120	4.25	4.11	4.10	3.95	3.69								

1 Example II

2           This example reports on a group of runs involving gravity settling and which  
3 were carried out at 80°C using various solvents at a relatively high S/F ratio of 0.91  
4 (w/w).

5           The results are shown in Table 3 and Figure 2.

TABLE 3  
Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C  
Using Different Solvents at S/F Ratio = 0.91

1	2	3	4	5	6	7	8	9	10	11	12	13	14
Settling time	Pt.7 naphtha	Toluene	Aromatic Naphtha	Heptane	Water Content in Oil Phase (%)								
mins													
0	10.89	9.13	9.41	9.32									
5	3.74	3.47	2.41	4.28									
15	3.44	3.21	2.26	3.78									
30	3.02	3.05	2.14	<0.10									
60	2.76	2.74	2.09	<0.10									
90	2.47	2.47	1.91	<0.10									
120	2.27	2.25	1.80	<0.10									



1 It will be noted that, at an S/F ratio of 0.91 (w/w), the residual water content in  
2 the oil phase was reduced from about 4% (Example I) to about 2 - 2.5% for the  
3 aromatic solvents tested.

4 However, the heptane run at the same S/F ratio gave a dramatically different  
5 result. After about 15 minutes of settling time, an apparent inversion of the emulsified  
6 water was initiated and virtually all of the emulsion settled into the water phase after  
7 30 minutes of settling.

8 Heptane is a paraffinic solvent. These runs disclose the discovery that a  
9 paraffinic solvent at a sufficient S/F ratio will remove substantially all of the water from  
10 diluted bitumen froth when gravity settled.

11

### 12 Example III

13 In this test, runs involving gravity settling were carried out at 80°C using  
14 various solvents at increasing S/F ratios.

15 The results are presented in Table 4.

16 It will be noted that for heptane, the residual water content could be reduced to  
17 a low value (0.1%) in decreasing settling time as the S/F ratio was increased above  
18 about 0.80.

19 The data shows that an inversion can be obtained using heptane when the S/F  
20 ratio is at least about 0.80. This inversion is initiated in less time as the ratio is further  
21 increased.

22 The Table 4 data further shows that the aromatic solvents (toluene, aromatic  
23 naphtha, Plant 7 naphtha) were not capable of producing dry bitumen product at high  
24 S/F ratios of 0.91 and 1.35.

TABLE 4

Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C  
Using Different Solvents at Different Solvent To Froth Ratios

	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>
1						
2						
3						
4	<u>Solvent</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>	<u>Heptane</u>
5	Solvent/Froth Ratio (w/w)	0.70	0.75	0.80	0.91	1.35
6		Water Content in Oil Phase (%)				
7	Settling time (min)	0	11.45	11.36	9.32	8.81
8		5	4.44	4.24	4.28	2.28
9		15	1.48	2.96	3.78	<0.1
10		30	1.04	0.31	<0.1	<0.1
11		60	0.56	0.11	<0.1	<0.1
12		90	0.26	0.13	<0.1	<0.1
13		120	0.13	<0.1	<0.1	<0.1
14	<u>Solvent</u>	<u>Toluene</u>	<u>Toluene</u>	<u>Aromatic</u>	<u>Plant 7</u>	<u>Plant 7</u>
15				<u>Naphtha</u>	<u>Naphtha</u>	<u>Naphtha</u>
16	Solvent/Froth (w/w)	0.91	1.35	0.91	0.91	1.35

1 TABLE 4 (Continued)

2	<u>Solvent</u>	<u>Toluene</u>	<u>Toluene</u>	<u>Aromatic</u>	<u>Plant 7</u>	<u>Plant 7</u>
3				<u>Naphtha</u>	<u>Naphtha</u>	<u>Naphtha</u>
4						
5	Settling time (min)	0	9.13	9.41	10.89	8.03
6		5	3.47	2.41	3.74	2.71
7		15	3.21	2.26	3.44	2.40
8		30	3.05	2.14	3.02	2.08
9		60	2.74	2.09	2.76	1.71
10		90	2.47	1.91	2.47	1.47
11		120	2.25	1.8	2.27	1.22

Water Content in Oil Phase (%)

1 Example IV

2 This example reports on runs involving centrifugation separation and use of  
3 hexane as the solvent. The results are presented in Table 5. The runs were  
4 conducted at temperatures ranging from 30°C to 60°C with increasing S/F ratios. The  
5 other runs were conducted at varying temperatures with a constant S/F ratio.

6 The results indicate that inversion occurs for hexane at 60°C at a S/F ratio of  
7 about 0.6. It further suggests that the S/F ratio required for inversion diminishes with  
8 a lighter solvent.

9 The results further indicate that the invention is operative at temperatures  
10 which are low (e.g. 40°C) relative to conventional temperatures (80°C) for dilution  
11 centrifuging.



TABLE 5

Residual Water, Chloride and Solids in Hydrocarbon Phase

After Centrifuging Using Hexane as Solvent at Different Temperatures

4	Solvent	S/F	Mixing	Cent.	Water	Chloride
5		(w/w)	temp. (°C)	temp. (°C)	(%)	(ppm)
7	Hexane	0.50	60	60	2.95	24.0
8	Hexane	0.55	60	60	2.47	10.1
9	Hexane	0.60	60	60	<0.1	<1
10	Hexane	0.70	60	60	<0.1	<1
11	Hexane	0.80	60	60	<0.1	<1
12	Hexane	1.00	60	60	<0.1	2.2
13	Hexane	0.70	50	50	<0.1	<1
14	Hexane	0.70	40	40	<0.1	<1
15	Hexane	0.70	30	30	0.76	3.8
16	Hexane	0.70	60	30	<0.1	

1 Example IV-A

2 This example illustrates the effectiveness of pure paraffinic solvents of  
3 different molecular weights. The solvents tested were butane (C4), pentane (C5),  
4 hexane (C6), and heptane (C7). Froth treatment was carried out under conditions  
5 similar to those set out in Example IV. The solvent/froth ratio was varied for each  
6 solvent over the range 0.3 to 0.9. The results, shown graphically in Figure 2A,  
7 indicate that as the molecular weight of the solvent increased from C4 to C7, the  
8 solvent/froth ratio required to produce dry (zero water concentration) bitumen  
9 increased from slightly less than 0.5 for butane to about 0.8 for heptane. This  
10 example suggests that inversion occurs at an essentially constant molar ratio of  
11 solvent to bitumen. It will be appreciated from the foregoing that the molecular weight  
12 of the paraffin used must be taken into consideration in setting the solvent/froth ratio.  
13 At least for the paraffins tested, the trend appears to be that a higher molecular  
14 weight paraffin must be used in higher mass ratios than a lower molecular weight  
15 paraffin.

16 Example V

17 Table 6 illustrates the effect of temperature on water removal. Hexane was  
18 used as a diluent at a hexane/froth ratio of 0.7 w/w and the hydrocarbon samples  
19 were centrifuged at 2000 rpm for 10 minutes at temperatures different from the mixing  
20 temperature. The data illustrate that separation of the water from the hydrocarbon  
21 can be achieved at temperatures above about 30°C.

TABLE 6

Effect of Mixing Temperature and Centrifuging Temperature on

Separation of Water from Hexane Diluted Froth

Hexane/Froth Ratio = 0.7 w/w, Centrifuging 10 mins. at 2000 rpm

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Ratio: Mixing Temp °C/ Centrifuging Temp. °C (M°C/C°C)	M30/C30	M60/C30	M40/C40	M50/C50	M60/C60
Water Content in Hydrocarbon, wt. %	0.76	<0.10	<0.10	<0.10	<0.10

---

Example VI

Table 7 illustrates the solids content for the runs of Figure 2 resulting from the use of heptane solvent at 0.91 solvent/froth ratio, and residual solids contents for hydrocarbons where toluene and Plant 7 naphtha were used as diluents.

TABLE 7

Effect of Diluent Type on Solids Removal from Froth

Settling Temperature 80°C, S/F Ratio = 0.91

Diluent Type	Heptane	Toluene	Plant 7 Naphtha
Solids Residue in Hydrocarbon, wt. %	0.15	0.75	0.79

Example VII

This example reports on runs involving centrifugation separation and use of paraffinic, cycloparaffinic and olefinic solvents at varying temperatures and a S/F ratio of 1.00 w/w.

Table 8 illustrates the effect of cycloparaffinic (cyclohexane) and olefinic (cyclohexane) solvents on water removal at solvent/froth ratios of 1.0 w/w. It is clearly shown that non-paraffinic solvents do not achieve the water removal of paraffinic solvents.

TABLE 8

Residual Water and Chloride in Bituminous Froth Diluted with Various Hydrocarbon Solvents, After Centrifuging

	Solvent	Paraffinic Content	b.p.(°C)	Density (g/ml)	S/F (w/w)	Mixing Temp (°C)	Cent. Temp. (°C)	Water (%)	Chloride (ppm)
9	Hexane	100%	69	0.664	1.00	60	60	<0.1	2.2
10	Heptane	100%	98	0.648	1.00	80	80	<0.1	<1
11	i-Octane	100%	100	0.688	1.00	80	80	<0.1	<1
12	Hexadecane	100%	287	0.773	1.00	80	80	<0.1	<1
13	Bayol 35*	98+%		0.780	1.00	80	80	<0.1	<1
14	Cyclohexane	0%	81	0.779	1.00	80	80	2.04	16.5
15	cyclohexane	0%	83	0.810	1.00	80	80	2.36	19.0
16	* Trade Mark								
17	Bayol 35 is a blend of higher molecular weight paraffins (C <sub>12+</sub> )								



1 As shown:

- 2 • The paraffinic solvents (hexane, heptane, i-octane, hexadecane and  
3 Bayol 35) were all successful in producing dry (0.1%) diluted bitumen  
4 product. This group of paraffinic solvents included normal paraffins,  
5 isoparaffins (i-octane) and paraffin blends (Bayol 35);
- 6 • The cycloparaffinic and olefinic solvents were not successful in  
7 producing a dry diluted bitumen product;
- 8 • Residual chlorides in the hydrocarbon phase were less than 1 ppm  
9 when paraffinic solvents were used. Cycloparaffinic and olefinic  
10 solvents yielded higher chloride contents in the hydrocarbon, which  
11 were consistent with retention of salt in the residual water.

12 The term "paraffinic solvent" is used in the claims. This term is intended to  
13 cover solvents containing normal paraffins, isoparaffins and blends thereof in  
14 amounts greater than 50 wt. %. It is not intended to include olefins, naphthas or  
15 cycloparaffins.

1 Example VII-A

2           The experiments reported in this example examined the impact of compounds  
3 such as aromatics, olefins and naphthenes on the ability of paraffins to produce dry  
4 bitumen. These tests were carried out because paraffinic solvents which are  
5 inexpensive enough to use on a commercial scale are not pure paraffins. For  
6 example, natural gas condensate (NGC) contains about 83% paraffins, but also  
7 significant amounts of aromatics and naphthenes. Other tests reported on herein  
8 (see Table II) demonstrated that when NGC is used as the froth diluting solvent, the  
9 solvent/froth ratio required to produce dry bitumen is significantly higher than for pure  
10 paraffins, at approximately 1.0 (mass/mass). The inventors believed that it was  
11 important to identify compounds which are inhibitors to the action of paraffins in froth  
12 treatment. Such information could be useful for designing and producing an  
13 economically viable solvent from existing process materials.

14           To minimize the effect of molecular weight, a family of compounds was  
15 selected that were close together in molecular weight, as follows:

- 16           • heptane (pure paraffin),
- 17           • methyl cyclohexane (naphthene), and
- 18           • toluene (aromatic).

1 The tests were carried out on a single froth sample using the procedure  
2 outlined above for centrifuging runs. The solvent mixtures tested contained heptane  
3 with varying concentrations of the three non-paraffinic test solvents, which were  
4 added on a volume/volume basis. For each solvent mixture, a series of tests was  
5 run, increasing the solvent/froth ratio until either a dry bitumen was produced, or the  
6 solvent/froth ratio became too high for more solvent to be added to the test vessels.

7 The results of the tests are summarized in Tables 8a, 8b, 8c and 8d and  
8 Figure 2b. Table 8a shows that for pure heptane, dry bitumen is produced at a  
9 solvent/froth ratio of 0.80. The effect of addition of an olefin (hexane) even at 30%  
10 (v/v), was relatively small. It increased the solvent/froth requirement to produce dry  
11 bitumen was from 0.8 to 1.0 (Table 8b). A more pronounced effect was observed with  
12 the addition of naphthene, methyl cyclohexane. Added at only 10% (v/v), it increased  
13 the effective solvent froth ratio from 0.8 to 1.0 (Table 8c). At 20% and 30%  
14 naphthene, the product contained measurable water at a solvent/froth ratio of 1.0.  
15 The addition of an aromatic, toluene, at 10% (v/v) increased the effective solvent/froth  
16 ratio to 1.2.

17 In Figure 2b, the effect of the three non-paraffinic compounds at 30% (v/v) are  
18 compared in terms of the water content remaining in the treated bitumen at varying  
19 solvent/froth ratios.

1 To summarize, this example suggests that aromatic and naphthene impurities  
 2 in a paraffinic solvent will significantly increase the amount of solvent required to  
 3 produce dry bitumen. Olefin impurities appear to be less important.

4 Table 8a

5 Summary of Results, Pure Paraffin (heptane)

6	7	8	9	10	11	12	13
Solvent	Upper	Solvent	Solvent	Density	Water		
to Froth	Phase	Density	Bitumen	Hydro-	Content of		
Ratio	Density		Ratio	carbon	HC Phase		
0.49	0.8453	0.6838	0.6980	0.8452	0.91		
0.61	0.8161	0.6838	1.0083	0.8155	0.37		
0.71	0.8035	0.6838	1.1768	0.8035	0.07		
0.80	0.8020	0.6838	1.2007	0.8020	0.01		



Table 8b

	Summary of Results, Olefin/Paraffin Mixtures																		
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	
	Hexane Concentration in Heptane	Solvent to Froth Ratio	Upper Phase Density	Solvent Density	Solvent to Bitumen Ratio	Density of Hydrocarbon	Water Content of HC Phase												
	10	0.50	0.8406	0.6832	0.7413	0.8399	1.31												
	10	0.70	0.8235	0.6832	0.9094	0.8233	0.25												
	10	0.80	0.8024	0.6832	1.1869	0.8024	0.04												
	10	0.98	0.7853	0.6832	1.4987	0.7853	0.00												
	20	0.50	0.92887	0.6826	0.2299	0.9283	1.62												
	20	0.70	0.8259	0.6826	0.8760	0.8259	0.21												
	20	0.81	0.8115	0.6826	1.0504	0.8114	0.05												
	20	1.01	0.8014	0.6826	1.196	0.8014	0.00												
	30	0.50	0.8475	0.6820	0.6743	0.8470	1.55												
	30	0.80	0.8056	0.6820	1.1257	0.8056	0.16												
	30	1.00	0.7870	0.6820	1.4455	0.7870	0.00												
	100	0.70	0.8427	0.6780	0.6887	0.8427	1.34												
	100	1.00	0.7826	0.6780	1.4693	0.7826	0.00												
	100	1.50	0.7659	0.6780	1.8771	0.7659	0.00												



Table 8c

## Summary of Results, Naphthene/Paraffin Mixtures

		Upper Phase Density	Solvent Density	Solvent to Bitumen Ratio	Density of Hydrocarbon	Water Content of HC Phase
1						
2						
3	Methyl					
4	Cyclo-hexane					
5	Concentration					
6	in Heptane					
7						
8	10	0.8472	0.6923	0.7454	0.8453	2.31
9	10	0.8341	0.6923	0.8699	0.8331	0.77
10	10	0.8209	0.6923	1.0209	0.8206	0.22
11	10	0.8084	0.6923	1.2028	0.8082	0.06
12	10	0.7944	0.6923	1.4585	0.7944	0.02
13	20	0.8643	0.7009	0.6712	0.8589	3.00
14	20	0.8123	0.7009	1.2479	0.8119	0.17
15	20	0.7975	0.7009	1.5395	0.7974	0.03
16	30	0.8594	0.7095	0.7399	0.8567	2.92
17	30	0.8333	0.7095	1.0342	0.8317	1.59
18	30	0.8234	0.7095	1.1818	0.8221	0.63
19	30	0.8137	0.7095	1.3458	0.8131	0.29
20	30	0.8013	0.7095	1.6115	0.8012	0.06

Table 8d  
Summary of Results, Aromatic/Paraffin Mixtures

	Toluene Concentration in Heptane	Solvent to Froth Ratio	Upper Phase Density	Solvent Density	Solvent Bitumen Ratio	Density of Hydrocarbon	Water Content of HC Phase
1							
2							
3							
4	10	0.50	0.8564	0.7021	0.7071	0.8556	1.67
5	10	0.80	0.8286	0.7021	1.0085	0.8284	0.61
6	10	0.90	0.8160	0.7021	1.1987	0.8157	0.15
7	10	1.20	0.7881	0.7021	1.8056	0.7881	0.00
8	15	0.50	0.8527	0.7112	0.7998	0.8519	1.86
9	15	0.82	0.8157	0.7112	1.3222	0.8156	0.60
10	15	0.99	0.8069	0.7112	1.5106	0.8067	0.16
11	15	1.21	0.7962	0.7112	1.7907	0.7960	0.03
12	30	0.51	0.8711	0.7387	0.7823	0.8706	1.76
13	30	0.80	0.8428	0.7387	1.1988	0.8421	0.82
14	30	1.00	0.8389	0.7387	1.2641	0.8387	0.38
15	30	1.21	0.8263	0.7387	1.5485	0.8263	0.23
16	30	1.51	0.8110	0.7387	2.0326	0.8109	0.07

1 Example VIII

2 It has long been recognized that asphaltenes will precipitate in pentane. It was  
3 reported by Reichert, C., Fuhr, B. J., and Klein, L. L., in "Measurement of asphaltene  
4 flocculation in bitumen solutions", J. Can. Pet. Tech. 25(5), 33, 1986, that the onset of  
5 asphaltene precipitation in pentane occurs when 1.92 ml/g of pentane is added to  
6 Athabasca bitumen. Considering the bitumen content (66.22%) in the tested froth  
7 sample, the asphaltene precipitation threshold is equivalent to 1.27 ml/g of pentane  
8 for the froth sample.

9 As previously established, the minimum solvent to froth ratios for hexane  
10 diluent and heptane diluent for water elimination are about 0.60 g/g and 0.80 g/g of  
11 solvent based on froth, respectively. By considering the densities of the diluents,  
12 these ratios are converted to 0.90 ml/g for hexane and 1.17 ml/g for heptane diluents.

13 Since asphaltene solubility in hexane and heptane is higher than in pentane, it  
14 appears that asphaltene precipitation should not be significant in hexane or heptane  
15 at S/F ratios close to the inversion point.

1 To further demonstrate that inversion of the emulsion and not asphaltene  
 2 precipitation was taking place, a test was conducted where heptane was added to  
 3 bitumen in different amounts and the quantities of asphaltene precipitating from the  
 4 solution was observed. The results are reported in Table 9 and clearly show that  
 5 asphaltenes begin to precipitate from solution at ratios in excess of approximately 1.0  
 6 w/w heptane to froth, which exceeds the inversion value of 0.8 w/w heptane to froth  
 7 as obtained from Figure 3.

8 TABLE 9

9 Asphaltene Precipitation Observations with Heptane Diluent

10

---

11 Heptane to bitumen ratio (w/w)	0.68	1.06	1.21	1.37	1.50	1.60	2.04	5.00
12 Equivalent heptane to froth ratio (w/w)	0.45	0.70	0.80	0.91	1.00	1.06	1.35	3.11
13 Asphaltene precipitation at room temp.	No	No	No	No	No	little	some	lots
14 Asphaltene precipitation at 80°C	No	No	No	No	No	little	some	lots

15 This point is significant for the following reason. There is a hydrocarbon loss with  
 16 the water fraction. If this loss is asphaltenes, then there is no practical way known to  
 17 applicants for recovering these lost hydrocarbons.



1 In conclusion, the foregoing examples support:

- 2 (1) That paraffinic solvents when used as diluents for froth treatment at  
3 appropriate S/F ratios will eliminate substantially all of the water and  
4 chloride from froth upon separation using centrifugation or gravity  
5 settling;
- 6 (2) Both normal and iso paraffinic solvents are efficient in generating dry  
7 diluted bitumen products;
- 8 (3) Sufficient paraffinic solvent to achieve inversion is needed to produce  
9 dry bitumen product - the critical S/F ratio will vary somewhat with the  
10 solvent used;
- 11 (4) The process works at low and high temperatures; and
- 12 (5) Asphaltene precipitation does not appear to be a problem.

13

14 Example IX

15 A typical commercial solvent, which is largely paraffinic and commonly consists  
16 of C<sub>4</sub> - C<sub>20</sub> hydrocarbons, is natural gas condensate ("NGL"). The composition of this  
17 solvent is compared with the Plant 7 naphtha in Table 10, in which the composition is  
18 described by various hydrocarbon classes.



TABLE 10

Typical Hydrocarbon Class Compositions of  
Natural Gas Condensate and Plant 7 Naphtha

Component	Paraffins	Naphthenes	Aromatics
Naphtha	43%	40%	17%
Natural Gas Condensate	83%	12%	5%

Table 11 and Figure 4 illustrate water removal at different solvent/froth ratios using natural gas condensate as a solvent. In this example, water and solids were eliminated from the hydrocarbon at solvent/froth ratios exceeding 1.0 w/w.

TABLE 11

Water Removal Results From Froth With  
Natural Gas Condensate As Diluent By Gravity Settling at 40°C

Solvent		NGC	NGC	NGC	Pt.7 Naphtha
Solvent/Froth Ratio (w/w)		0.80	1.00	1.20	1.35
Temperature (°C)		40	40	40	80
		Water Content in Oil Phase (%)			
Settling time (min)	0	8.83	8.16	7.58	8.03
	5	7.32	6.79	6.22	2.71
	15	6.01	2.8	<0.1	2.4
	30	1.75	<0.1	<0.1	2.08
	45	1.72	<0.1	<0.1	
	60	1.62	<0.1	<0.1	1.71
	90				1.47
	120				1.22

As shown, runs were carried out using S/F ratios of 0.80, 1.00, and 1.20. On the run having a S/F ratio of 1.00, the water removal increased dramatically (relative to S/F ratio = 0.80 run) and dry bitumen was produced. Stated otherwise, inversion was obtained using NGC at S/F ratio of 1.00 (w/w).

By comparison, a run using Plant 7 naphtha at 80°C and S/F ratio of 1.35 was unsuccessful in producing dry bitumen.

As stated, using NGC as the diluent at S/F ratios of 1.00 or greater resulted in substantially all of the water being removed from the oil. However a brownish rag layer was produced between the oil and water layers. See Figure 4 and Table 12.

TABLE 12

Rag Layers Produced During Gravity Settling with  
Natural Gas Condensate as Froth Diluent

Settling time (min)	<u>Rag layer/(rag layer +</u> NGC/Froth = 1.00(w/w)	<u>upper oil layer); Vol %</u> NGC/Froth = 1.20(w/w)
30	30%	25%
60	23%	17%
90	22%	15%
120	18%	13%
3 days	9%	8%
Composition of rag after 120 min settling	51.97% + 48.03% water plus solids	/

14

15 As settling was extended, the volume of the rag layer diminished. After settling  
16 for 120 minutes, the composition of the rag layer reached about 50% oil and 50%  
17 water plus solids.

18 When the rag layer was separated from the other layers and centrifuged at  
19 2000 rpm for 10 minutes, the water and hydrocarbon separated, leaving oil containing  
20 less than 0.1% water.

1 Example X

2 This example reports on a run conducted in a scaled up pilot circuit using NGC  
3 as the diluent. The run was operated at 50°C and then the temperature was  
4 increased over time, reaching 127°C. The S/F ratio was maintained at about  
5 1.20(w/w).

6 The pilot unit used is outlined schematically in Figure 5.

7 The results are set forth in Table 13.

8 The pilot unit consisted of a feed system where froth and diluent were pumped  
9 through a heater and into a mixing vessel which had a nominal retention time of 2 - 5  
10 minutes. Pressures in the system were held at approximately 1000 Kpa. Product  
11 from the mixer was passed under pressure into the settling vessel which had a  
12 nominal 15 minutes residence time. The oil/water interface was monitored and  
13 controlled by a conductivity probe. The products, both hydrocarbon and slurry  
14 underflow, were discharged from the process through coolers and then the pressure  
15 released through positive displacement pumps.

16 The run continued for a period of 7-1/4 hours with approximately one-half of  
17 the operating time at 50°C and the other half at 117°C (ave).

18 The results show that dry diluted bitumen could be recovered when the  
19 process was operated at both temperatures. (See Table 13.)



TABLE 13

## Froth Treatment Pilot Test Results with Natural Gas Condensate as Froth Diluent

		Froth Flow Kg/min	Condensate Flow kg/min	Diluent Froth Flow kg/min	Settler Product kg/min	Settler Tails kg/min		
7	Run #1	0.823	0.881	1.704	1.10	0.60		
8	Run #2	0.823	0.966	1.788	1.39	0.40		
10								
11	Temperature Deg C		Mixing Speed	Pressure Kpa	Hydrocarbon Recovery (%)	Product Quality (%HC)	Chloride Removal (%) Wt. %	Hydrocarbon Solids Content
15	Run #1	49	500	1000	83.8	99.2	98	0.06
16	Run #2	117	500	1000	97.6	90.7	77	0.32

TABLE 14

## Centrifuging Results of Underflows From Pilot Runs

Underflow Sample	From 50°C pilot run; Natural gas condensate	From 120°C pilot run; Natural gas condensate	From 120°C pilot run; Plant 7 naphtha
Density of U/F before cent.	0.92g/ml	0.98g/ml	
Upper oil after centrifuging	33.8%	11.8%	9.0%
Rag after centrifuging	41.2%	3.4%	none
Water after centrifuging	14.7%	58.9%	71.3%
Bottom solids after cent.	10.3%	25.9%	19.7%
Water % in rag from cent.	73.8%	50.5%	/
Water % in recovered oil by cent.	<0.1%	<0.1%	0.35%

However, it was found that, at the low operating temperature (50°C), oil losses with the water and solids underflow were relatively high. At the high operating temperature (~120°C), the oil losses with the underflow were minimal. More particularly, samples of the underflow were centrifuged in a laboratory centrifuge at 2000 rpm for 10 minutes. The centrifuge contents separated into 4 layers, specifically: a clean oil layer; a viscous rag layer; a water layer; and a solids layer. The relative proportions are stated in Table 14. Most of the solids in the hydrocarbon were also removed.

1 In conclusion, the results teach that NGC can successfully be used as the  
2 diluent at low and high temperatures to yield dry diluted bitumen. However, the low  
3 temperature process produces relatively low quality underflow and the underflow has  
4 a relatively high rag content.

5

#### 6 Example XI

7 This example provides a detailed analysis of the product bitumen and the  
8 tailings which are produced in the froth treatment process described in Example X. In  
9 order to maximize the quality enhancement:

- 10 • a relatively high solvent/froth (natural gas condensate) ratio of 1.58 was  
11 used; and
- 12 • the run was conducted at 50°C.

13 The lower temperature was chosen on the basis of Example X which  
14 demonstrated that while the bitumen recovery was higher if the run was conducted at  
15 117°C, the product might be of higher quality if the temperature was 49°C. A  
16 summary of the operating conditions and process performance data for the run are  
17 shown in Table 15.

18 Three samples were taken from the unit:

- 19 • feed to the unit before dilution (froth);
- 20 • product from the unit (diluted bitumen); and
- 21 • tailings from the unit.

1           The composition and properties of the three fractions were determined using  
2 methodology which is well known in the field of bitumen upgrading. Analyses were  
3 carried out in accordance with the methods documented in the analytical methods  
4 directory of the LAN. The bitumen fraction of interest in the three samples was  
5 selected to be that fraction of hydrocarbon which boils above 343°C. The diluted  
6 bitumen product was sufficiently low in water and solids and was dilute enough that  
7 the fraction of the hydrocarbon with bp above 343°C could be directly recovered from  
8 the sample through distillation on a true boiling point distillation apparatus. The froth  
9 and tailings samples both had significant water and solids contents. Therefore,  
10 before distillation, they were first extracted with hot toluene in a Dean Starke  
11 apparatus to isolate the hydrocarbon portion of the sample. This was then subjected  
12 to the identical distillation process as the diluted bitumen sample.

13           The data from the analyses performed on the bitumen recovered from the  
14 three distillations is shown in Table 16. The results show that the process yielded a  
15 bitumen product which was significantly different from that obtained from the  
16 conventional froth treatment process. In particular, the product bitumen from this  
17 process in comparison to the standard product had:

- 18           • almost no extraneous matter;
- 19           • less aromatics;
- 20           • less micro-carbon residue;
- 21           • less nitrogen;
- 22           • less sulphur;
- 23           • less pentane insoluble material;
- 24           • a better carbon to hydrogen ratio; and
- 25           • a lower density.

26           "Extraneous matter" comprises material which can be measured as "solids" in



1 the Dean Stark analysis, plus any other toluene-insoluble material which can be  
2 recovered by filtration. It is basically a measurement of non-bitumen material.

3 There was also a drastic reduction in the viscosity as compared to the feed  
4 bitumen, from 1880 - 324 mPa.s @ 100°C.

5 In summary, this example demonstrates that it is possible to produce a highly  
6 purified dry bitumen product using the paraffin froth treatment process of the  
7 invention. The product bitumen has a water content of less than about 0.1%, an  
8 extraneous matter and solids content of less than about 0.1%, and a reduced  
9 viscosity of generally less than about 500 mPa.s @ 100°C. The inventors believe that  
10 a highly purified bitumen product has not heretofore existed.

11 Although the quality of the bitumen which is described in Example XI  
12 happened to have been achieved using the paraffin froth treatment process under  
13 the conditions outlined in Table 15 (natural gas condensate used at a solvent/froth  
14 ratio of 1.58 at 50°C), the inventors believe that bitumen of equivalent purity can be  
15 produced using other paraffinic solvents as outlined herein.

16 The inventors believe that the purity of the product bitumen obtained from the  
17 Paraffin Froth Treatment process will provide the following benefits in upgrading:

- 18 • In the conventional Froth Treatment process, employing Naphtha as a  
19 diluent, the product bitumen contains inorganic solids which end up as  
20 "ash" constituents in coke, as a fouling deposit on the inside of process  
21 equipment and piping, or as deposits on catalyst pellets. These  
22 deposits ultimately lead to shut-down for cleaning, frequent repairs, and  
23 the consumption of catalyst used in the hydrocracking process. The  
24 removal of virtually all inorganic solids from the product bitumen should  
25 reduce the rate of fouling and catalyst poisoning.
- 26 • Product bitumen which is free of inorganic solids can be channelled to

1 primary upgrading steps that previously were precluded. For example,  
2 processes such as \*Vega Combi-Cracking (VCC), which may be  
3 superior in yield and quality of products made from raw bitumen, cannot  
4 accept feeds containing significant solid impurities.

- 5 • The product bitumen is clean enough to meet the Bottom Sediment and  
6 Water (BS&W) specifications of pipeline operators. The product  
7 bitumen would therefore only need to be reduced in viscosity to meet  
8 pipeline specifications. This might be accomplished by the addition of a  
9 small quantity of light hydrocarbon diluent (such as natural gas  
10 condensate) or by the application of a mild "vis-breaking" technology  
11 such as heat-soaking or low severity catalytic treatments.
- 12 • If the product bitumen is produced under conditions which render it low  
13 in viscosity, such as in Example II, the product bitumen would be  
14 pumpable with none or very little dilution. This could eliminate the  
15 necessity of coupling extraction with upgrading facilities.

16 \* Trade Mark

Table 15

## Paraffin Froth Treatment Process Performance Data

## Using Natural Gas Condensates As Solvent

<u>Parameter</u>	<u>Value</u>
Solvent to Froth Ratio (wt/wt)	1.58
Solvent to Bitumen Ratio (wt/wt)	2.5
Mixer Temperature (°C)	50
Product Quality	
Hydrocarbon (%/wt)	99.92
Water (%/wt)	0.08
Solids (%/wt)	0.00
Bitumen Recovery (%)	87.6

Table 16

Comparison of +343°C Fractions of Feed, Product and Tailings Bitumen

Analysis	Feed Bitumen	Product Bitumen	Tailings Bitumen	Typical Coker Feed Bitumen
Carbon wt%	80	83	79	83
Hydrogen wt%	10.1	10.4	9.5	10.4
C/H ratio	7.9	8.0	8.3	8.0
% C in aromatic environment	31.0	28.3	32.2	
Sulphur wt ppm	52400	47500	56000	49700
Nitrogen wt ppm	5240	4770	6600	5870
Basic Nitrogen wt ppm	1520	1340	1860	
extraneous matter wt%	0.83	0.02	1.43	0.50
micro carbon residue wt% (not corrected for "extraneous matter"wt)	16.3	13.7	24.2	15.0
pentane insolubles wt%	21.4	12.7	39.2	
density gm/cc @60°C	1.030	1.007	1.071	
viscosity mPa.s @100°C	1880	324	49200	
% recovered @ 524°C	35.6	44.2	29.5	41.5



1           **THE EMBODIMENTS OF THE INVENTION IN WHICH AN**  
2 **EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS**  
3 **FOLLOWS:**

4  
5           1. A method for cleaning bitumen froth comprising hydrocarbon and aqueous  
6 phases and containing water and particulate solids contaminants dispersed in the  
7 hydrocarbon phase in the form of an emulsion, said froth having been produced by a  
8 water extraction process practised on oil sand, comprising:

9           adding a sufficient amount of paraffinic solvent to the froth to induce inversion  
10 of the emulsion;

11           mixing the froth and the solvent for a sufficient time to disperse the solvent in  
12 the froth;

13           subjecting the mixture to gravity or centrifugal separation for a sufficient period  
14 to separate substantially all of the water and solids from the bitumen to produce dry  
15 diluted bitumen; and

16           pumping the dry diluted bitumen through a pipeline to an upgrading circuit.

17

18           2. The method as set forth in claim 1 wherein the dry diluted bitumen has a  
19 water plus solids content of less than about 0.5 weight percent.

20

21           3. The method as set forth in claim 1 or 2 wherein the solvent used comprises  
22 a mixture of low molecular weight alkanes having chain lengths from about C<sub>5</sub>-C<sub>16</sub>.

1           4. A method for delivering oil sand-derived bitumen through a pipeline,  
2 comprising:

3           a) subjecting oil sand to a water extraction process to obtain a bitumen froth,  
4 the bitumen froth comprising hydrocarbon and aqueous phases and containing water  
5 and particulate solids contaminants dispersed in the hydrocarbon phase in the form of  
6 an emulsion;

7           b) cleaning the bitumen froth to remove water and particulate solids  
8 contaminants, the cleaning process comprising:

9           adding a sufficient amount of paraffinic solvent to the froth to induce inversion  
10 of the emulsion,

11           mixing the froth and solvent for a sufficient time to disperse the solvent in the  
12 froth,

13           subjecting the mixture to gravity or centrifugal separation for a sufficient period  
14 to separate substantially all of the water and solids from the bitumen to produce dry  
15 diluted bitumen; and

16           c) pumping the dry diluted bitumen through a pipeline to an upgrading circuit.

17

18           5. A method for delivering oil sand-derived bitumen through a pipeline,  
19 comprising:

20           a) subjecting oil sand to a water extraction process to obtain a bitumen froth,  
21 the bitumen froth comprising hydrocarbon and aqueous phases and containing water  
22 and particulate solids contaminants dispersed in the hydrocarbon phase in the form of  
23 an emulsion;

24           b) cleaning the bitumen froth to remove water and particulate solids  
25 contaminants, the cleaning process comprising:

26           adding a sufficient amount of paraffinic solvent to the froth to produce a

- 1 solvent to froth ratio of at least about 0.5 (w/w);
- 2 mixing the froth and solvent for a sufficient time to disperse the solvent in the
- 3 froth;

1           subjecting the mixture to gravity or centrifugal separation to reduce its water  
2 plus solids content to less than about 0.5 weight percent; and

3           c) pumping the dry diluted bitumen through a pipeline to an upgrading circuit.  
4

5           6. The method as set forth in claim 1, 2, 3, 4, or 5 wherein the solvent is  
6 natural gas condensate containing more than 50% paraffins.  
7

8           7. The method as set forth in claims 1, 2, 3, 4, or 5 wherein the solvent is  
9 natural gas condensate containing more than 50% paraffins and the solvent is added  
10 in sufficient amount to produce a solvent to froth ratio of about 1.00 (w/w).  
11

12           8. The method as set forth in claim 4 or 5 wherein the solvent used comprises  
13 a mixture of low molecular weight alkanes having chain lengths from about C<sub>5</sub>-C<sub>16</sub>.  
14

15           9. The method as set forth in claims 1, 2, 3, 4, or 5 or wherein the solvent is  
16 natural gas condensate containing more than 50% paraffin and the solvent is added  
17 in sufficient amount to produce a solvent to froth ratio of about 1.0 - 1.5 (w/w).  
18

19           10. The method as set forth in claim 4 or 5 wherein the solvent used  
20 comprises a mixture of low molecular weight alkanes having chain lengths from about  
21 C<sub>5</sub>-C<sub>16</sub> and the solvent is added in sufficient amount to produce a solvent to froth ratio  
22 of about 1.0 – 1.5 (w/w).



1

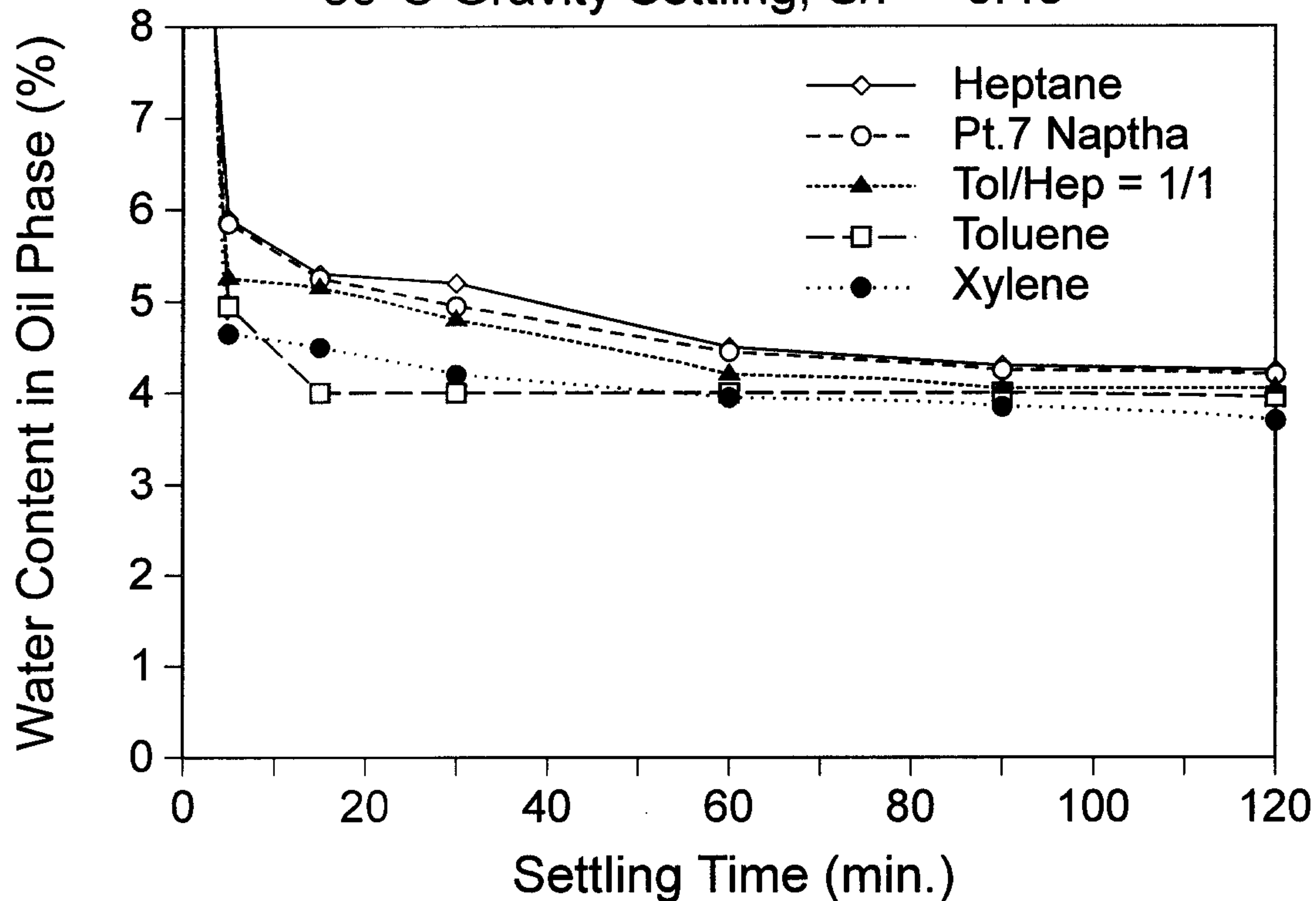
2           11. A dry, paraffinic solvent - diluted bitumen product having a water plus  
3 solids content of less than 0.5 weight %, said bitumen product having been produced  
4 by the method of claim 1, 3, 4, 5, 6, 7, 8, 9 or 10.

5

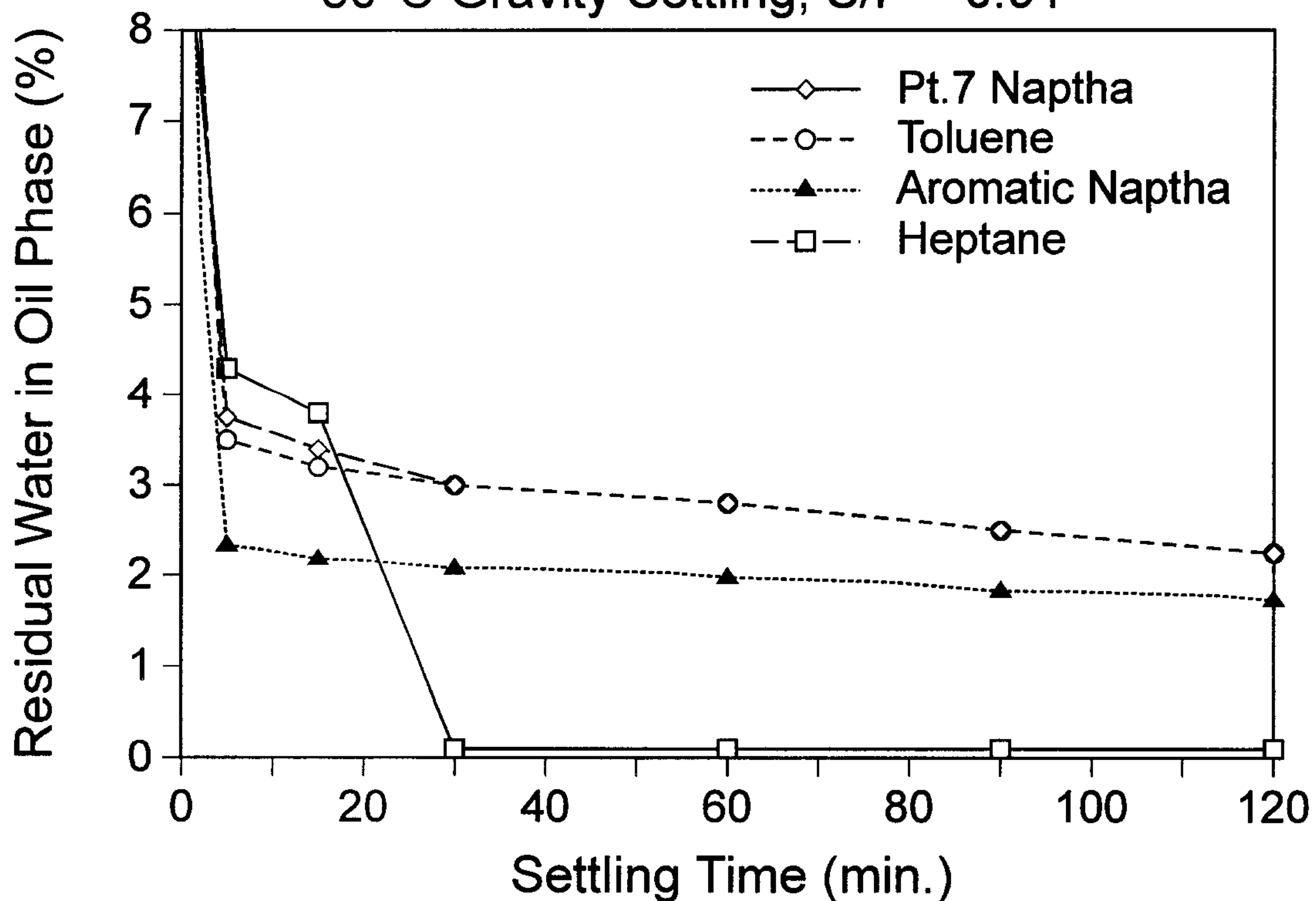
6           12. A dry, paraffinic solvent - diluted bitumen product having a water content  
7 of less than about 0.1 weight %, a solids content of less than about 0.1 weight %, and  
8 an extraneous matter content of less than about 0.1 weight %, said bitumen product  
9 having been produced by the method of claim 1, 3, 4, 5, 6, 7, 8, 9 or 10.

1/3

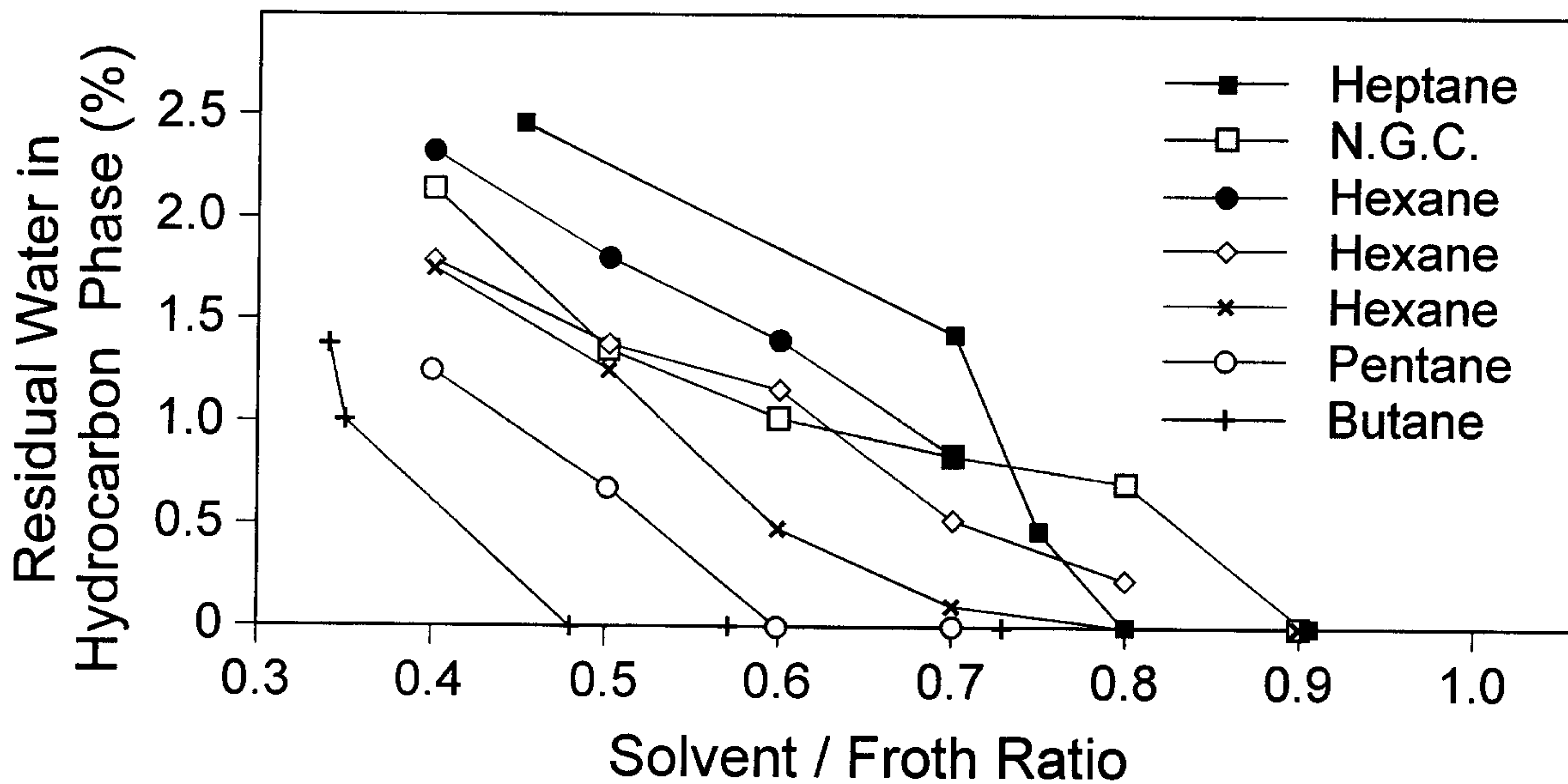
**FIG. 1.** Solvent Effect on Water Removal;  
80°C Gravity Settling; S/F = 0.45



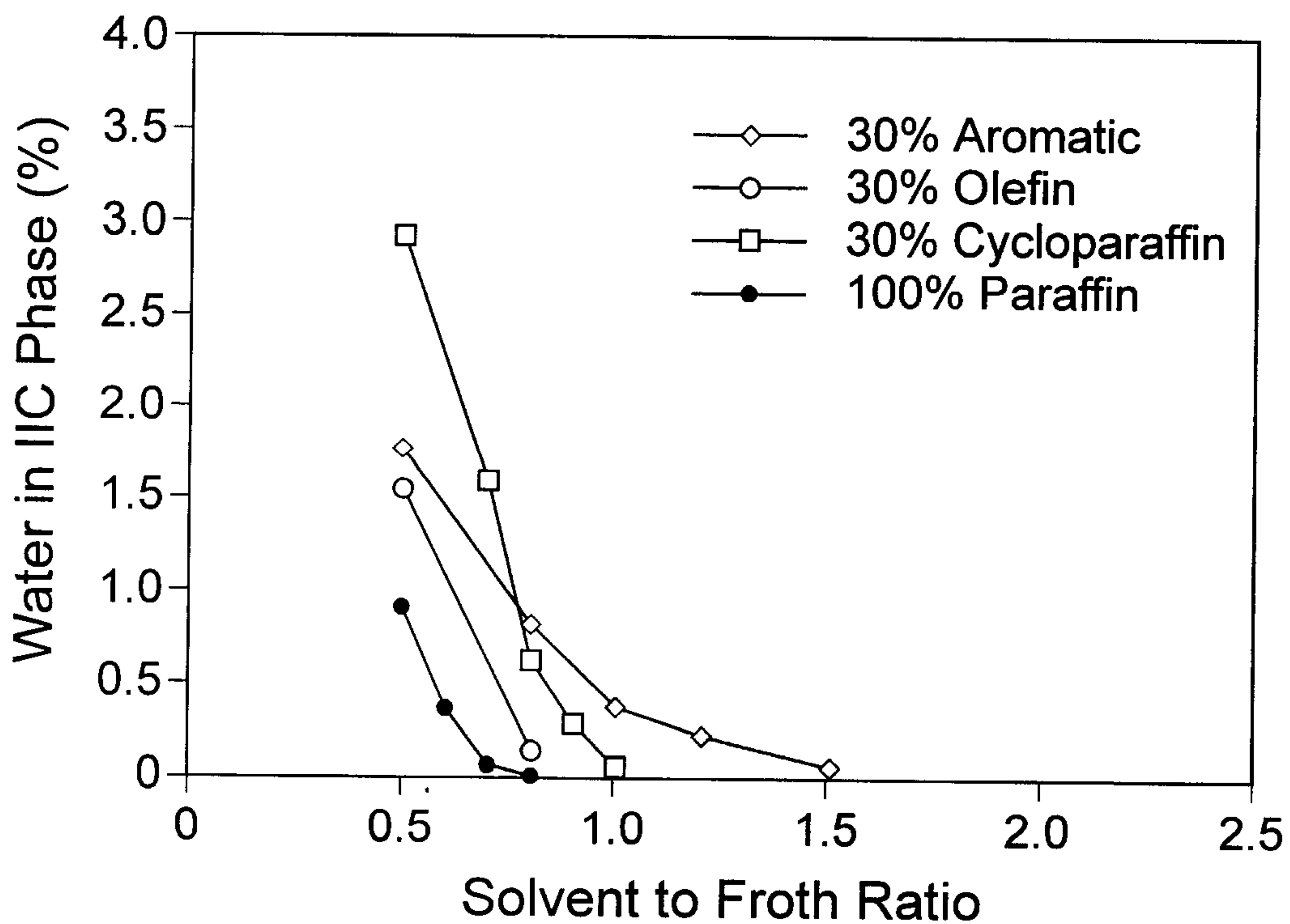
**FIG. 2.** Solvent Effect on Water Removal;  
80°C Gravity Settling; S/F = 0.91



**FIG. 2A.** Effect of Solvent Molecular Weight on Water "Inversion"

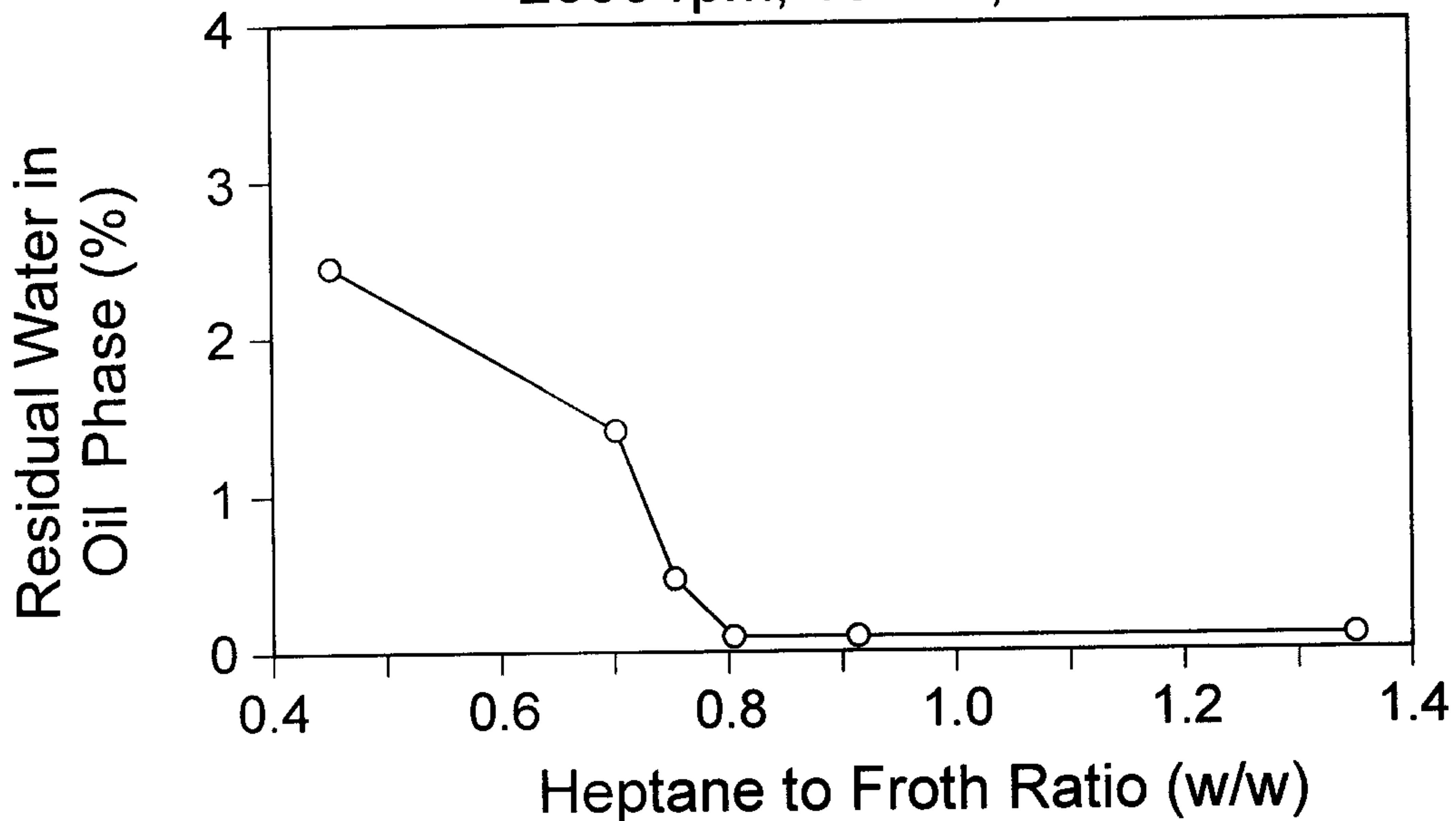


**FIG. 2B.** Effect of Adding Aromatics, Olefins and Cycloparaffins to a Pure Paraffin



3/3

**FIG. 3.** Effect of Solvent / Froth Ratio on Water Removal; Heptane as Diluent; Centrifuging 2000 rpm, 10 min, 80°C



**FIG. 4.** Water Removal at Different Solvent to Froth Ratios; 40°C Gravity Settling; NG Condensate

