

## Halide-Free Clay-free Invert Emulsion Fluids

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### Abstract

Conventionally, an aqueous solution of halide salt like calcium chloride is used as the internal phase in an invert emulsion fluid (IEF) to balance osmotic pressures for drilling in shales. After drilling the wells, the drilled cuttings are bioremediated or thermally treated to remove the oil. The drill cuttings are then mixed with soil for farming. However, if the residual salt content is high, the cuttings are not suitable for plant growth. The use of an organoclay-free IEF provides significant advantages over conventional clay-based IEFs, such as minimal seepage loss, low solids content, better equivalent circulating density (ECD) control, and faster rates of penetration (ROP). Consequently, a salt-free organoclay-free IEF was developed to provide improved technical and environmental performance.

The halide free organoclay-free IEF comprises a biodegradable and non-toxic internal phase to mitigate the potential negative effects of a brine-based IEF. The internal phase of the IEF, which achieves 70% biodegradation in 28 days, is environmentally friendly.

A novel suspension agent (SA) that is not a clay-based material was developed to stabilize this halide free IEF. The 12.0-ppg and 16.0-ppg halide free organoclay free IEFs were stable at temperatures greater than 300°F. The suspension agent is environmentally safe with a biodegradability of 71.4 % in 42 days and  $LC_{50} > 10\text{g} / \text{L}$  on the test organism. These new fluids exhibited rheology, fluid loss and shale erosion properties comparable to those of conventional IEFs. The salt free organoclay-free IEFs showed good tolerance to contaminants and static aging.

### Introduction

One of the primary reasons for the worldwide use of organoclay free invert emulsion drilling fluids is the improved wellbore stability and the increased drilling rate that it provides, thereby reducing the overall drilling cost. To provide this wellbore stability, invert emulsion drilling fluids usually employ aqueous solutions of inorganic salts, especially halide salts such as calcium-chloride solution for aqueous activity control.

Ionic salts dissolve in water by separating into positively-charged cations and negatively-charged anions. These ions attract associated water molecules that move with the ions. This attraction lowers the water activity of the solution, with the water activity decreasing with increasing salt concentration. The use of these salts prevents hydration of

troublesome shale formations by maintaining the drilling fluid aqueous activity less than, or equal to, the aqueous activity of the shale.<sup>1</sup>

However, the use of salts as aqueous activity controllers in the drilling fluid can pose challenges for the receiving environment, in the case of onshore drilling. There is no practical way to degrade the inorganic salts which are undegradable.<sup>2,3,4</sup> While such chemicals can be treated by diluting to below toxic limits or washing out of the waste, it does not necessarily eliminate the problem, as there is a limit to how much dilution can be done; thus, non-toxic levels can be difficult to reach.<sup>5,6</sup> This makes it desirable to replace these inorganic aqueous-activity controlling agents with something that is more environmentally friendly.

Stable organoclay-free halide-free IEFs have been formulated with aqueous solutions of a hygroscopic liquid which reduce the water activity. A comparison of the toxicity and biodegradation of  $\text{CaCl}_2$  and the hygroscopic liquid used in the present study is given in **Table 1**. A novel suspension agent (SA) that is not a clay-based material was developed to stabilize this halide-free IEF. The novel suspension agent in the absence of organophilic clay provides the necessary barite suspension thereby giving a stable halide-free organoclay-free IEF. The halide-free IEFs formulated with the novel SA are tolerant to contaminants and static aging.

### Methods and Materials

The fluids were formulated with commercially available invert emulsifiers, polymeric filtration control agent, barite, organic and inorganic rheology modifiers, and base oil composed of mixed paraffins. The internal phase of the halide-free IEFs was a 60% w/w aqueous solution of the hygroscopic liquid (HL). The concentration of products used to formulate the IEFs was estimated with a proprietary numerical simulator.

In a typical experiment, the fluids were first mixed on a multimixer Model #9B5 fitted with sine wave impeller blade no. 9B29X. The fluids were then hot-rolled at the desired temperature - 250°F for 12ppg / 300°F for 16ppg - in a roller oven (Model 705ES) for 16 hours. The fluids were then tested for rheology on the FANN® 35 viscometer at 120°F, as per API RECOMMENDED PRACTICE 13B-2; 6.3. The HPHT fluid loss was measured at - 250°F for 12ppg / 300°F for 16ppg - with 500 psi differential, as per API RECOMMENDED PRACTICE 13B-2; 7.2.

In the static aging studies, the hot rolled fluid was mixed on multimixer for 5 min and aged in HPHT SS cells in the

upright position in a mechanical convection oven at desired temperature and duration (Model MO1490SC-1 available from Thermoelectron Corporation). The sag performance of the fluid was assessed by determining the sag factor given in **Equation 4**, where the specific gravity of the top ( $SG_{top}$ ) and bottom ( $SG_{bottom}$ ) portion of the fluid in the aging cell were determined.

The rheology of the fluid was characterized in terms of plastic viscosity (PV), yield point (YP), and low shear yield point (LSYP). The YP and PV are parameters from the Bingham plastic (BP) rheology model. The YP is determined by extrapolating the BP model to a shear rate of zero; it represents the stress required to move the fluid. The YP is expressed in the units of lb/100ft<sup>2</sup>. The YP indicates the cuttings carrying capacity of the IEF through the annulus or in simple terms its hole cleaning ability. The PV represents the viscosity of a fluid when extrapolated to infinite shear rate, expressed in units of centipoise (cP). The PV indicates the type and concentration of the solids in the IEF, and a low PV is preferred.

Both PV and YP are calculated using 300 revolutions per minute (rpm) and 600-rpm shear rate readings on a standard oilfield viscometer as given in **Equations 1** and **2**. The yield stress or  $\tau_0$  is a parameter from the Herschel Buckley (HB) rheology model which is the equivalent of the YP in the BP model. The  $\tau_0$  is determined by fitting the HB model to the shear stress vs. shear rate curve, which is the dial readings plotted against the corresponding rpm determined on the standard oil field viscometer. The  $\tau_0$  is expressed in the similar units as the YP. The  $\tau_0$  indicates the susceptibility of the IEF to barite sag, and a high  $\tau_0$  is proposed to deliver a sag resistant IEF. The  $\tau_0$  can be estimated reasonably by calculating the LSYP value from **Equation 3**.

$$PV = (600 \text{ rpm reading}) - (300 \text{ rpm reading})$$

**(Equation 1)**

$$YP = (300 \text{ rpm reading}) - PV$$

**(Equation 2)**

$$LSYP = [2 \times (3 \text{ rpm reading})] - (6 \text{ rpm reading})$$

**(Equation 3)**

$$SagFactor = \frac{SG_{bottom}}{SG_{bottom} + SG_{top}}$$

**(Equation 4)**

## Results and Discussions

### 1) Formulating stable organoclay free and halide free IEF

The halide-free organoclay-free invert emulsion fluids were formulated at 12 ppg and 16 ppg densities with 70/30 and 80/20 oil water ratio respectively. The base oil used for

the IEF was composed of mixed paraffins. The internal phase was a 60% w/w aqueous hygroscopic liquid (HL), free of any halide salts. The 60% w/w HL corresponds to the water activity provided by 250,000 ppm CaCl<sub>2</sub> as shown in **Fig. 1**. The IEFs of this paper were formulated with rheology and filtration properties expected of a typical drilling IEF as shown in **Table 3**.

#### (a) Stable organoclay free and halide free 12ppg IEF at 250°F.

A stable halide free and organoclay free 12ppg IEF was formulated with the novel suspension agent (SA). The formulation of the 12ppg halide free IEF is given in **Table 2**. The 12ppg IEFs were hot rolled for 16 hours at 250°F. The halide free IEFs were formulated at two concentrations – 2.5ppb and 5ppb – of the SA. A base halide free 12ppg IEF was formulated in the absence of the SA.

After hot rolling, the halide free and organoclay free “base” 12ppg IEF showed oil separation and barite settling at the bottom of the aging cell. This demonstrated the need for the SA in formulating a halide free and organoclay free IEF. Though the base was unstable it was still tested for its rheology and filtration properties which are shown in **Figure 2**. The YP and LSYP were 17 and 5 respectively whereas the PV of the base was low at 21. The HPHT fluid loss was low at 2ml.

In the presence of 2.5ppb SA, a stable halide free and organoclay free 12ppg IEF was obtained after hot roll. The rheology and filtration properties of the SA based 12ppg IEF were within the desired range given in **Table 3**. The YP and LSYP were 15 and 5 respectively whereas the PV was 22. The HPHT fluid loss was 2ml. Comparing the rheology of the base with the 2.5ppb SA based IEF shows that the addition of the SA negligibly changed the YP and LSYP whereas the PV increased roughly 30%. Thus the addition of SA improved the suspension of the halide free and organoclay free 12ppg IEF without impacting its overall rheology.

Stable halide free and organoclay free 12ppg IEFs were also formulated with 5ppb SA. These IEFs showed a YP and LSYP of 22 and 8 whereas the PV was 32. The high concentration of the SA effected an increase in rheology of the 12ppg IEF. The HPHT fluid loss of the 5ppb SA based IEF was still 2ml.

#### (b) Stable organoclay free and halide free 16ppg IEF at 300°F

Stable halide free and organoclay free 16ppg IEF was formulated with the novel suspension agent (SA). The formulation of the 16ppg halide free IEF is given in **Table 2**. The 16ppg IEFs were hot rolled for 16 hours at 300°F. The halide free IEFs were formulated at two concentrations – 3ppb and 4ppb – of the SA. A base halide free 16ppg IEF was formulated in the absence of the SA.

After hot roll, the halide free and organoclay free base 16ppg IEF showed oil separation and considerable barite

settling at the bottom of the aging cell. As with the 12ppg IEF, although the base was unstable it was still tested for its rheology and filtration properties which are shown in **Figure 3**. The YP and LSYP were 10 and 2 respectively whereas the PV of the base was 48. The HPHT fluid loss was 0ml.

In the presence of 3ppb SA, a stable halide free and organoclay free 16ppg IEF was obtained after hot roll. The rheology and filtration properties of the SA based 12ppg IEF were within the desired range given in **Table 3**. The YP and LSYP were 15 and 6 respectively whereas the PV was 48. The HPHT fluid loss was 3ml. Comparing the rheology of the base with the 3ppb SA based IEF shows that addition of the SA increased the YP and LSYP marginally from 10 to 15 and 2 to 6 respectively whereas the PV remains unchanged.

To investigate the effect of SA on rheology a 4ppb SA based 16ppg IEF was formulated. This IEF showed a YP and LSYP of 19 and 6 whereas the PV was 48. Increasing the concentration of the SA did not substantially increase the rheology of the 16ppg IEF. The HPHT fluid loss of was 3.8ml which was with the desired range in **Table 3**.

It was concluded that with the novel suspension agent stable organoclay free halide free IEFs could be formulated. The novel SA provided suspension to barite without any substantial increase in the rheology of the IEF.

## 2) Static aging studies on 12ppg and 16ppg halide-free organoclay free IEFs

After hot rolling the 12 and 16ppg IEFs at 250°F and 300°F respectively, both IEFs were then subjected to static aging for 16 hours and 48 hours at 250°F (12ppg) and 300°F (16ppg) respectively.

The rheology and filtration properties of the 2.5ppb based 12ppg IEF after static aging for 16 and 48 hours are shown in **Figure 4**. The PV, YP, LSYP and 10 min gel strengths were approximately 26, 15, 5 and 12 respectively. The HPHT fluid loss at 250°F for the 48 hour static aged IEF was 2.8ml which was near the fluid loss of 2ml with the unstatic aged IEF. Thus the rheology and filtration of the organoclay free and halide free SA based 12ppg IEF remain similar even up to 48 hours of static aging. The sag factor determination on the 16 and 48 hours static aged IEF was 0.51. A drilling fluid with sag factor of less than 0.53 has good suspension characteristics and is considered sag-resistant.<sup>7</sup>

The static aging on 16ppg IEF was performed for 16 hours with SA concentration of 3ppb and for 48 hours with SA concentration of 4ppb at 300°F. The rheology and filtration properties for the 16 hours and 48 hours static aged 16ppg IEF are shown in **Figures 5 and 6** respectively.

After 16 hours of static aging the PV, YP, LSYP and 10 min gel strengths were 50, 12, 5 and 11 respectively whereas the HPHT fluid loss at 300°F was 2ml. The unstatic aged IEF gave PV, YP, LSYP and 10 min gel strengths of 48, 15, 6 and 10 respectively whereas the HPHT fluid loss at 300°F was 1.8ml. Thus the rheology and filtration properties of the 16ppg IEF changed negligibly after 16 hours of static aging. The sag factor on static aging for 16 hours was 0.52

After 48 hours static aging of the 16ppg IEF, the YP decreased from 19 to 8, the LSYP decreased from 6 to 4 and the gel strengths decreased from 11 to 7. The HPHT fluid loss at 300°F changed little from 2.8 to 3ml. This decrease in rheology of the 16ppg IEF though did not significantly affect the suspension of the IEF, since a sag factor of 0.527 was obtained.

From the static aging studies it was observed that halide free organoclay free IEFs formulated with the novel SA were resistant to barite sag. Even a decrease in rheology after static aging did not lead to barite sag.

## 3) Contamination studies

Tolerance to contamination is the primary requisite of any good drilling fluid. Contamination studies were performed on organoclay free and halide-free 12ppg IEF with 2.5ppb SA concentration. The contamination study was divided into two parts as shown in **Figure 7**:

- Part A: Effect of the contaminant on the rheological and the filtration properties of the IEF.
- Part B: Effect of treatment on the contaminated IEF to restore its rheological and filtration properties within 20% of its original values (uncontaminated state).

The contaminants used in the study were as follows:

- 40.0 ppb of artificial drilled solids,
- 10% seawater,
- 2.0 ppg weight up with barite, and
- 5.0 ppb lime.

The results of contamination studies are presented in **Fig. 7a and 7b**. The contamination of the halide free 12 ppg IEF with 2.0 ppg weight up with barite and 40ppb drilled solids addition resulted in negligible changes in the YP, LSYP and HPHT fluid loss of the IEF relative to the uncontaminated IEF. These changes did not warrant any treatment.

The contamination of 12ppg IEF with 10% v/v sea water caused the YP to increase from 14 to 34 and the LSYP to increase from 5 to 10. The addition of lime as treatment to the sea water contaminated IEF decreased the YP from 34 to 10. This shows that the halide free IEF was sensitive to lime.

Thus when the halide free IEF was contaminated with 5ppb lime the YP decreased from 14 to 7 and the LSYP decreased from 5 to 3. The addition of 2ppb of commercially available organic rheology modifier as treatment to the lime contaminated IEF increased the YP from 7 to 12.

The HPHT fluid loss of both the contaminated and the treated IEF changed negligibly.

The results demonstrate that the organoclay free and halide free IEFs formulated with SA are tolerant to contaminants and any change in the rheological properties of the IEFs was easily treated with conventional additives.

## 4) Shale erosion studies

Since the conventional  $\text{CaCl}_2$  brine was replaced with the

halide free internal phase it was imperative to determine its effect on shale erosion. In a typical shale erosion experiment, an initial quantity (about 30gm) of sized shale cuttings (sieved between US# 5 and US# 10) is hot rolled in the IEF for 16 hours at 150°F. After hot roll, the final quantity of shale cutting remaining on US# 10 is determined after drying. The difference between initial and final quantity of shale cuttings is a measure of shale erosion. The initial moisture content of the shale cuttings is taken into account when determining the shale erosion.

The shale erosion studies were performed on London clay outcrop in 12ppg and 16ppg organoclay free and halide free IEFs. The X-Ray diffraction composition of the London clay outcrop is shown in **Table 4**. The London clay contained 70% total clay of which 49% was illite that disintegrates in water and 20% is smectite that swells in water. The shale erosion studies were conducted in organoclay free  $\text{CaCl}_2$  based 12ppg and 16ppg IEFs for purpose of comparison. The formulations for these  $\text{CaCl}_2$  based IEFs are given in **Table 5**. The water phase salinity of  $\text{CaCl}_2$  brine was 250,000 ppm that gives water activity similar to 60% w/w HL aqueous solution. The rheology, filtration and shale erosion values of the halide-free and  $\text{CaCl}_2$ -based IEFs are given in **Table 6**. The 12-ppg halide-free IEF gave a shale erosion of 3.5% on comparison the  $\text{CaCl}_2$  based IEF gave shale erosion of 4.2 %. The 16-ppg halide free IEF gave shale erosion of 3.8% on comparison the  $\text{CaCl}_2$  based IEF gave a shale erosion of 5.9 %.

Thus the organoclay free and halide-free IEFs provided similar shale recovery when compared to the  $\text{CaCl}_2$  based IEF.

## 5) Biodegradability and eco-toxicity studies of SA

To check the applicability of SA in environmentally stringent locations, SA was subjected to biodegradation and eco-toxicity studies. The SA was assessed for marine biodegradation by the BODIS method where the biodegradation was recorded every week, up to 42 days. The eco-toxicity study of SA was performed with marine juvenile fish *Cyprinodon variegatus* in seawater with OECD 203 guidelines for marine testing of offshore chemicals. The results are given in **Table 7**. The results demonstrate the good environmental profile of the suspension agent with potential applications in the North Sea.

## Conclusions

1. Stable 12 and 16ppg organoclay free and halide free IEFs were formulated with the novel suspension agent (SA).
2. The novel SA was able to impart barite suspension without an increase in the rheology of the IEF.
3. The static aged organoclay free and halide free IEFs were resistant to barite sag.
  - a. The rheology for the 12ppg IEF did not change much on static aging up to 48 hours.
  - b. The rheology of the 16ppg IEF decreased on 48 hours of static aging.
  - c. The filtration properties of 12ppg and 16ppg

IEF changed negligibly.

4. The organoclay free and halide free IEFs were tolerant to the effect of contaminants, and any deviations were easily treated with conventional additives.
5. The shale erosion of the organoclay free and halide free IEF was comparable to the  $\text{CaCl}_2$  based IEF.

## Acknowledgements

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## Nomenclature

SA	= Suspension agent
YP	= Yield point
LSYP	= Low shear yield point
PV	= Plastic viscosity
IEF	= Invert emulsion fluids
ppg	= Pounds per gallon
ppb	= Pounds per barrel
LGS	= Low gravity solids
ECD	= Equivalent circulating density
OWR	= Oil water ratio
WPS	= Water phase salinity
NOEC	= No observed effect concentration
LC50	= lethal concentration, median

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**Table 1: Comparison of biodegradation and toxicity profiles of CaCl<sub>2</sub> and hygroscopic liquids used in the study**

	Biodegradation (Method OECD 301)	Toxicity against Rats LD50	Aquatic Toxicity (Fish) LC50	Invertebrate toxicity (Daphnia Magna) LC50
CaCl <sub>2</sub>	Non-biodegradable	1g/kg	0.1g/kg	0.76–3g/kg
HL	60% in 10 days	12.6g/kg	5g/kg	15.3g/kg

**Table 2: Mixing order and time of products for 12ppg and 16ppg halide-free IEFs**

Products			Mixing time (min)
Mud weight (ppg)	12	16	–
OWR	70:30	80:20	–
BASE OIL, bbl	As required	As required	–
Primary emulsifier, ppb	11	15	2
Lime, ppb	0.65	0.65	2
Rheology Modifier, ppb	3	3	
Filtration control agent, ppb	4	4	5
S.A., ppb	2.5/5	3/4	
60% w/w HL	As required	As required	5
Inorganic rheology modifier, ppb	5	5	
Drilled solids, ppb	20	20	5
Barite, ppb	As required	As required	10
Hot roll temperature	250°F / 16 hours	300°F / 16 hours	

**Table 3: Conventional IEF specification**

Mud Properties	
Plastic viscosity cP	Less than 35 for 12ppg
	Less than 55 for 16ppg
Yield Point lb/100 ft <sup>2</sup>	7–18
LSYP	5–12
HPHT filtrate	Less than 10 ml

**Table 4: XRD of London clay outcrop cuttings**

London clay	
Quartz, wt %	26.00
Smectite, wt %	20.00
Illite, wt %	49.00

**Table 5: Mixing order and time of products for 12ppg and 16ppg CaCl<sub>2</sub>-free IEFs**

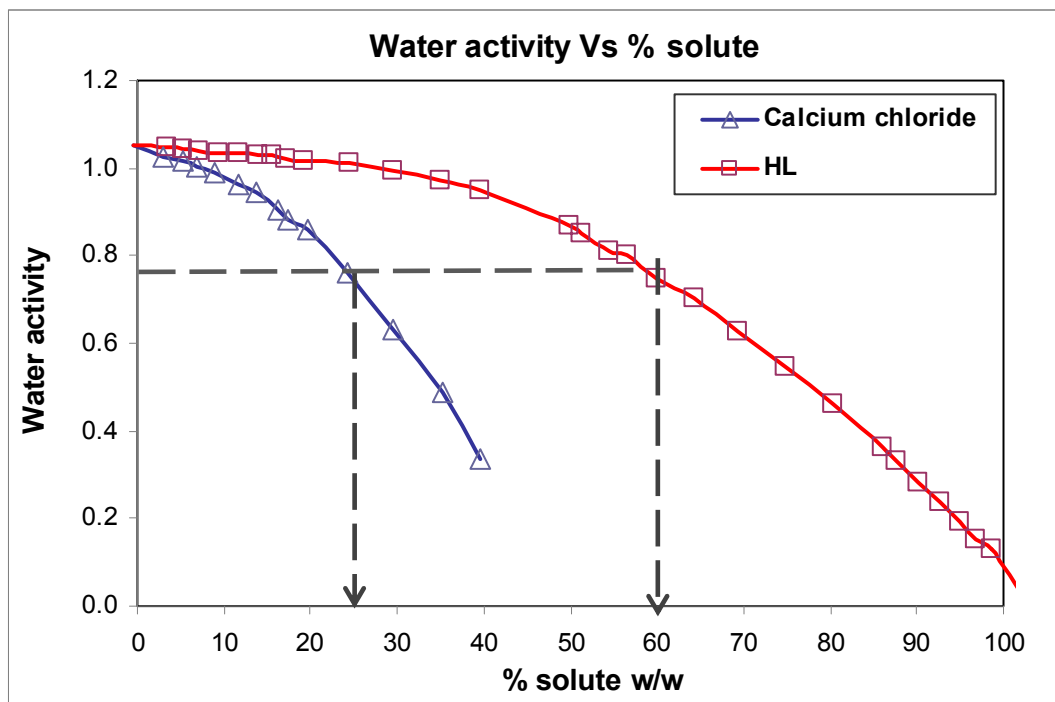
Products			Mixing time (min)
Mud weight (ppg)	12	16	–
OWR	70:30	80:20	–
BASE OIL, bbl	As required	As required	–
Primary emulsifier, ppb	12	16	2
Secondary emulsifier, ppb	-	1	2
Lime, ppb	1.3	2.5	2
Rheology Modifier 1, ppb	3	3	
Filtration control agent, ppb	2	2	5
CaCl <sub>2</sub> solution for 250K WPS, ppb	As required	As required	5
Inorganic rheology modifier, ppb	-	5	
Drilled solids, ppb	20	20	5
Barite, ppb	As required	As required	10
Micronized CaCO <sub>3</sub> , ppb	50	50	
Rheology Modifier 2, ppb	-	2	
Hot roll temperature	250°F / 16 hours	300°F / 16 hours	

**Table 6: Rheological properties of 12ppg and 16ppg CaCl<sub>2</sub> and halide-free IEFs for shale erosion studies**

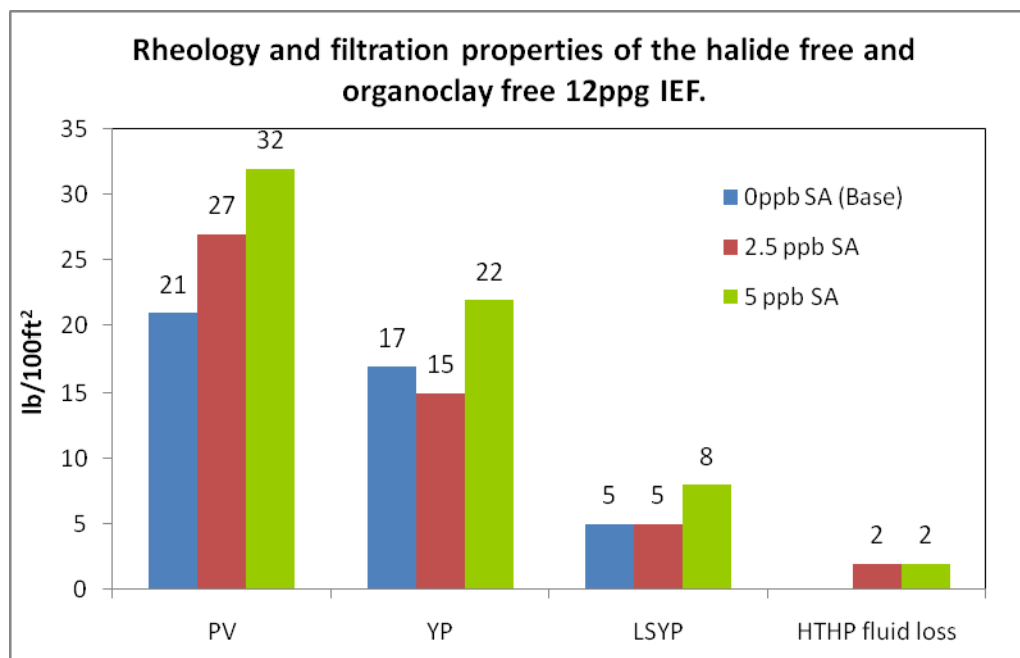
	12ppg CaCl <sub>2</sub>	12ppg halide-free	16ppg CaCl <sub>2</sub>	16ppg halide-free
PV	38	32	51	48
YP	30	22	24	15
LSYP	11	8	12	6
HTHP fluid loss, ml	2.8	2	2.8	3
Shale erosion %	4.2	3.5	5.9	3.8

**Table 7: Biodegradability and Eco-Toxicity data Of SA**

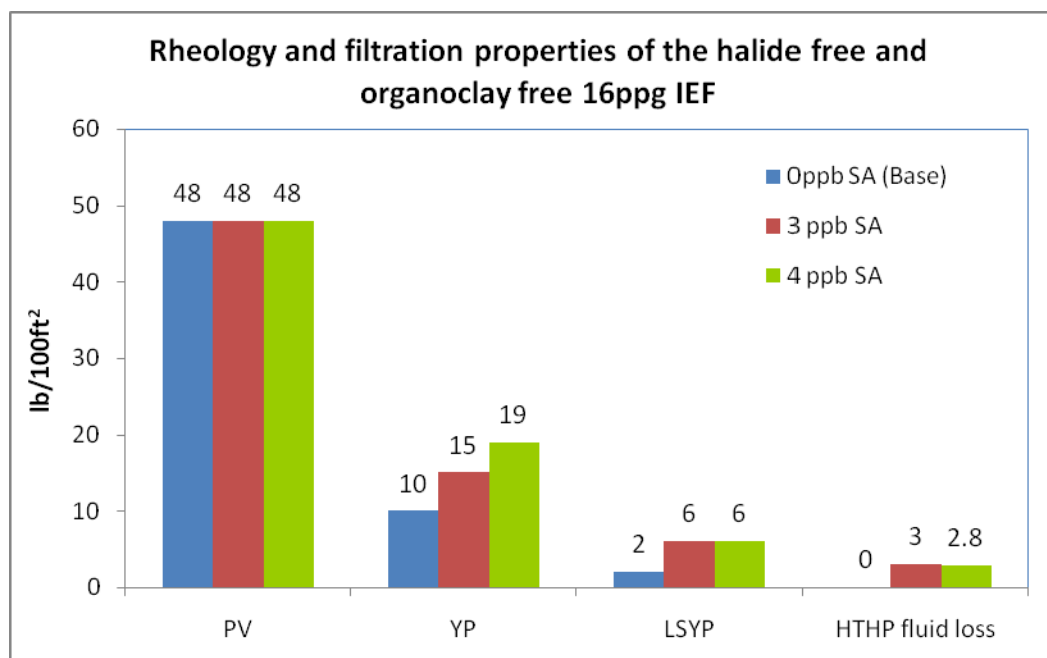
Tests		
Biodegradation	28 days	38.2%
	42 days	71.4%
Cyprinodon variegatus	48-hr LC50	>10g/l
	96-hr LC50	>10g/l
	96-hr NOEC	10g/l



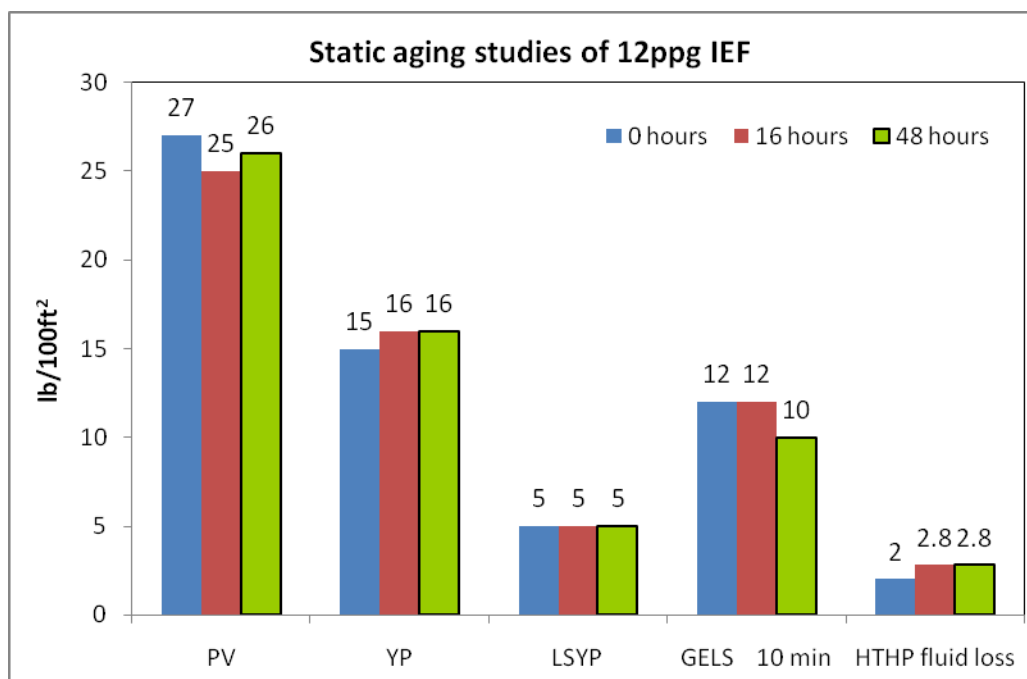
**Fig. 1: Relationship of water activity and percent solute of  $\text{CaCl}_2$  and HL**



**Fig. 2: Formulation of 12ppg halide-free IEF at 250°F**

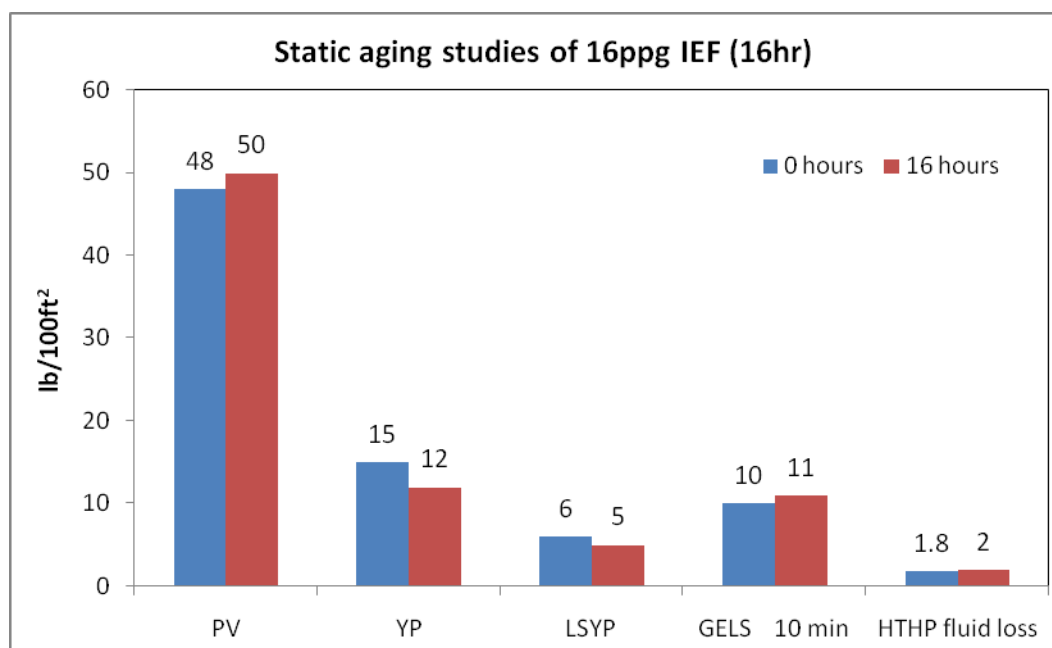


**Fig. 3: Formulation of 16ppg halide-free IEF at 300°F**

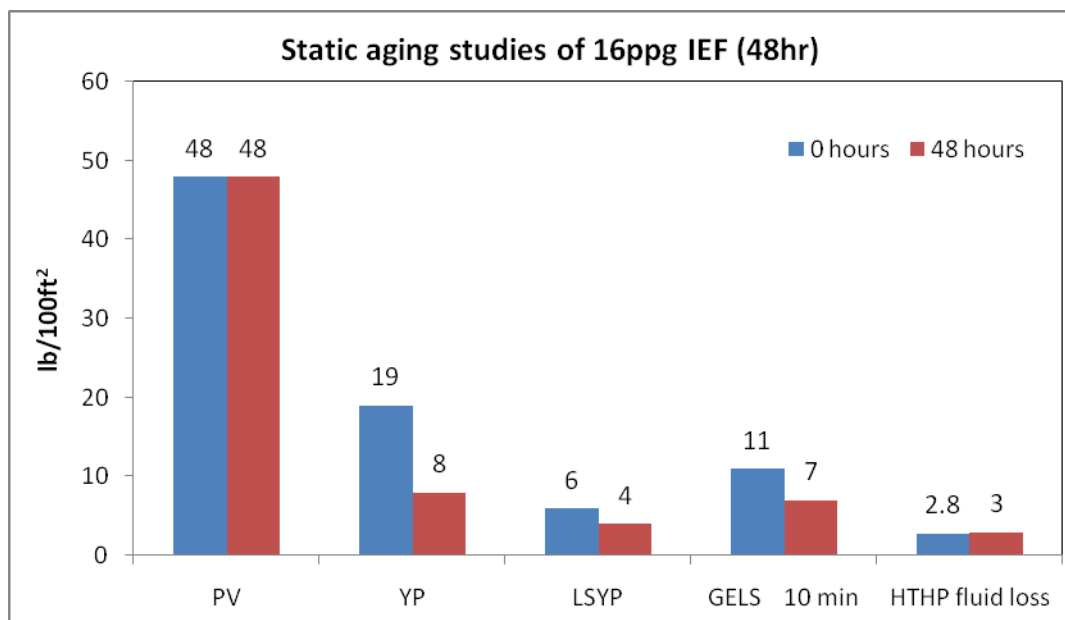


**Fig. 4: Static aging studies of 12ppg halide-free IEF with 2.5ppb S.A.**

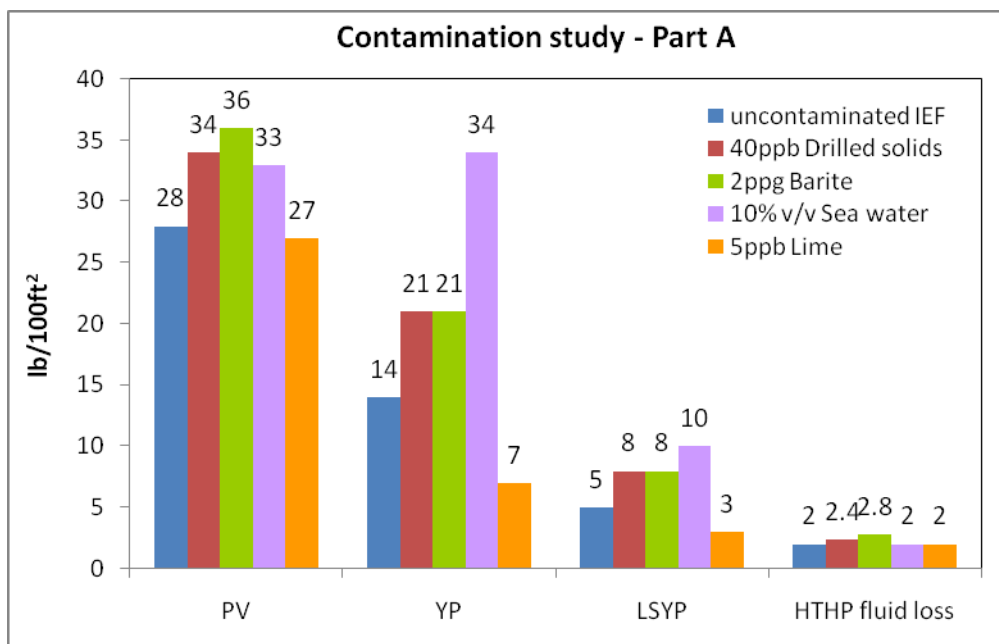




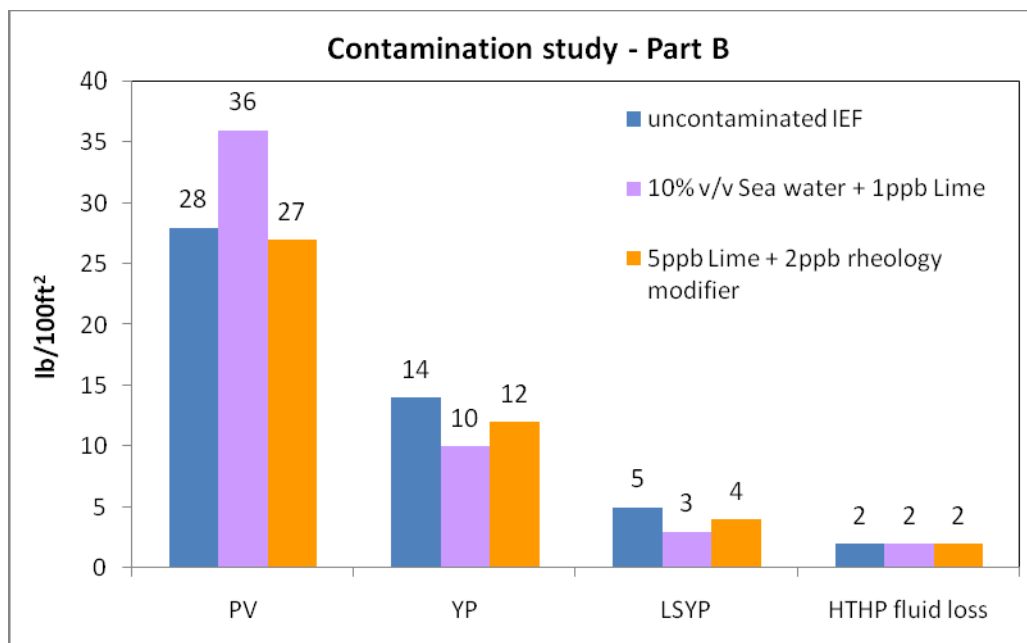
**Fig. 5: Static aging studies of 16ppg halide-free IEF with 3ppb S.A.**



**Fig. 6: Static aging studies of 16ppg halide-free IEF with 4ppb S.A.**



**Fig. 7a: Part A - Effect of contaminants on 12ppg halide-free IEF**



**Fig. 7b: Part A - Effect of treatment on contaminated 12ppg halide-free IEF**