Characterization of the Tribological Behavior of Graphene-Based Additives

Final CRADA Report

Energy Systems Division

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NON PROPRIETARY

FINAL CRADA REPORT

Date: 9-29-2014

CRADA Number: C1300301

CRADA Title: Characterization of the Tribological Behavior of Graphene-Based Additives

CRADA Start Date: 2-12-2013 End Date: 5-6-2014

Argonne Dollars: \$165k

Participant Dollars: \$10k funds in (cancelled after 1 year)

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Summary of Major Accomplishments:

XG-supplied nanocrystalline additives were extensively tested for friction and wear behavior. A technique for investigating the stability of additives was developed, and a report covered filtration and centrifugation methods for settling and filterable content. Friction and wear trends in partially and fully formulated oils were reported. Particle size analysis was performed and reported.

Summary of Technology Transfer Benefits to Industry:

The results reported in this document guide the industry toward selecting the most beneficial properties for this product, and aid in making a go/no-go decision regarding the introduction of this product to market. The settling properties act as an important guide to the industry for consumer acceptance.

Other Information/Results: (Papers, Inventions, Software, etc.)

Outline of Report CRADA 1300301

- Overview of DOE Project
- Goals/Objectives/Deliverables to DOE
- Discussion of Tasks
 - Friction and Wear Test Methodology
 - Summary of Early Friction Test Results for C500 Additions to Oils with Additives
 - Friction and Wear Maps for Tested Fluids
 - Viscosity Test Results for C300, C500 and C750 Mixtures
 - Particle Size DLS Results for C500 Mixtures
 - Mass Analysis Filtration Procedure
 - Mass Analysis Results for C500 Mixtures
 - Optical Sonication Results for C500 Mixtures

Project Overview

- Prior collaborations (with DOE support) examined the tribological performance of multiple grades of XG Science nanoplatelets.
- Results indicated nanoplatelets lowered friction and reduced wear – results obtained for base fluids.
- CRADA established to perform more detailed examination on the impact of nanoplatelets on tribological performance and to determine the mechanisms involved.

Goals/Objectives/Deliverables to DOE

1. Provide detailed data on the friction and wear properties of the Participant's nanoplatelet powders (in conjunction with molybdenum [Mo] and other transition metallo-organic friction modifiers and antiwear additives) blended into formulated and unformulated lubricant fluids.
2. Characterize the chemistry and structure of tribofilms generated by these enhanced lubricants under related vehicle tribological environments.
3. Characterize the dispersant nature, including particle size distribution, of the nanomaterials at variant loadings in lubricants, as well as viscosity change and suspension stability over time.
4. Quantify changes in surface metrology of automotive or industrial materials exposed to the participants' nanotechnology.
5. GO/NO-GO DECISION – At the end of the CRADA we will provide sufficient credible evidence to determine whether xGnP technology has potential to improve tribological performance of lubricants – decision to move forward on the development of fully formulated lubricant.
6. Deliverables at end of years 1 and 2
 1. Internal report to DOE sponsor and Parties" on the effect of test variables (nano-additive type, concentration, and surfactant/dispersant) on friction and surface morphology.

CRADA Tasks – Phase 1

- **Phase 1:** (Months 1–16): Evaluation of the effect of additive type (nanoplatelets/grade/BET), concentration, and surfactant (dispersant) on the tribological properties of base and formulated (semi-formulated) lubricants (mineral or synthetic).
 1. The Participant (XG Sciences) will prepare and blend the xGnP nanoplatelets into suitable carriers (mineral and/or synthetic oils) that will be combined into base and/or formulated lubricants for subsequent analysis.
 2. The Contractor (Argonne National Laboratory, hereafter Argonne) will evaluate the tribological properties (friction and wear) of base and formulated lubricants treated with the Participant's nano-additives (in suitable carrier fluids). Tests will include viscosity, dispersion, reciprocating pin-on-flat, cylinder-on-flat, ring-on-liner, skirt-on-liner, and rotating ball on disk configurations.
 3. The Contractor will blend MoDTC/ZDDP into non-treated and xGnP-treated solutions and will evaluate the tribological properties under conditions identical to those used in task 2. Tests will include viscosity, dispersion, reciprocating pin-on-flat, cylinder-on-flat, ring-on-liner, skirt-on-liner, and rotating ball on disk configurations.
 4. The Contractor will analyze the tests performed under tasks 2 and 3 to provide information on friction coefficient and wear properties.
 5. The Parties will perform detailed microscopy and 3-D profilometry of samples tested in task 2 and 3 to determine the effect of test variables on surface morphology and friction/wear.

Test Protocol for Friction Tests

Test parameters:

½" dia 52100 ball
Mirror polished 52100 flat
15.6 N load
20 mm reciprocating stroke
1 Hz reciprocating frequency

Dead weight
Ball and holder
Volt drop sensor
Specimen holder
Flat specimen
Temperature probe

Ball on Disk Tests

Predicted Hertzian contact pressure is
1 GPa maximum

Young's modulus E	213	GPa	213	GPa
Poisson's ratio ν	0.29		0.29	
Radius R (enter R=0! for a plain surface)	6.35	10^{-3} m	0	10^{-3} m
Load F prescribed			15.6	N
<input type="button" value="Solve"/> <input type="button" value="Reset"/> <input type="button" value="Print"/>				
Effective Young's modulus E'	232.6	GPa		
Effective contact radius R'	3.18	10^{-3} m		
Semi contact width r	0.09	10^{-3} m		
Indentation δ	1.17	10^{-6} m		
Contact stiffness	20.03	10^6 N/m		
Mean contact pressure p_m	0.67	GPa		
Maximum contact pressure p_{max}	1	GPa

Reciprocating tests were performed that consist of "ramps" at beginning and end of test with 1 hr of sliding between at 60 rpm

Beginning of CRADA Results

Comparisons:
1, 2, 3, 4 unadditized
1, 5, 9, 13 effects on base
2, 6, 10, 14 effects on DispOnly
3, 7, 11, 15 effects on FF_noFM
4, 8, 12, 16 effects on FF

	Oil	Friction Coefficient	Wear Vol (10 ³ μm ³)
1	BP429A_Base	0.144	1380
2	BP430A_DispOnly	0.113	23.7
3	BP428A_FF	0.092	12.1
4	BP427A_FF_NoFM	0.124	6.9
5	BP429A_Base + 1%C500	0.131	223
6	BP430A_DispOnly + 1%C500	0.123	3.1
7	BP428A_FF + 1%C500	0.091	6.4
8	BP427A_FF_NoFM + 1%C500	0.118	12.0
9	BP429A_Base + 1%ZDDP_1%Mo855	0.042	8.6
10	BP430A_DispOnly + 1%ZDDP_1%Mo855	0.034	0.97
11	BP428A_FF + 1%ZDDP_1%Mo855	0.104	5.2
12	BP427A_FF_NoFM + 1%ZDDP_1%Mo855	0.110	11
13	BP429A_Base + 1%ZDDP_1%Mo855 + 1%C500	0.066	12.7
14	BP430A_DispOnly + 1%ZDDP_1%Mo855 + 1%C500	0.047	23.3
15	BP428A_FF + 1%ZDDP_1%Mo855 + 1%C500	0.110	5.4
16	BP427A_FF_NoFM + 1%ZDDP_1%Mo855 + 1%C500	0.114	5.1

Comparison of Results

Wear Friction Map

Composite Image Showing the Appearance of Wear Scars for the Ball (left) and Flat (right)

Materials and Tests

CRADA (Blended at XG, received by Argonne; 0.5 wt% C500 in different BP SI Group IV oils)

1. **BP429A Base**
2. **BP429A Base + 0.5% C500 w/CA**
3. BP430A Disp Only
4. BP430A Disp Only + 0.5% C500 w/CA
5. BP427A FFnoFM
6. BP427A FFnoFM + 0.5% C500 w/CA
7. BP428A FF
8. BP428A FF + 0.5% C500 w/CA

Flat: hardened 52100 mirror polished, 1.5" x 2"
 Ball: hardened ½" dia 52100 ball
 Load: 15.6 N by deadweight application
 Stroke length: 2 cm
 Peak contact pressure: 1 GPa
 Temperature: 100°C actual temperature of flat

CRADA (Blended at Argonne by Demas and Erck; 1 wt% C500 in different BP SI Group IV oils)

BP429A Base

1. **BP429A Base**
2. **BP429A Base + 1% C500**
3. **BP429A Base + 1% ZDDP + 1% Mo855**
4. **BP429A Base + 1% ZDDP + 1% Mo855 + 1% C500**

BP430A Disp Only

1. BP430A Disp Only
2. BP430A Disp Only + 1% C500
3. BP430A Disp Only + 1% ZDDP + 1% Mo855
4. BP430A Disp Only + 1% ZDDP + 1% Mo855 + 1% C500

BP427A FFnoFM

1. BP427A FFnoFM
2. BP427A FFnoFM + 1% C500
3. BP427A FFnoFM + 1% ZDDP + 1% Mo855
4. BP427A FFnoFM + 1% ZDDP + 1% Mo855 + 1% C500

BP428A FF

1. BP428A FF
2. BP428A FF + 1% C500
3. BP428A FF + 1% ZDDP + 1% Mo855
4. BP428A FF + 1% ZDDP + 1% Mo855 + 1% C500

Blended at XG, Received by Argonne; 0.5 wt% C500 in Different BP SI Group IV Oils Blended at Argonne by Demas and Erck; 1 wt% C500

- Unlike C750, C500 alone (without capping agent) added in base oil does not show the same friction and wear reduction
- The capping agent (CA) in base oil lowers friction and wear over base oil alone, but 0.5% C500 w/CA offers significant friction and wear reduction

Goals

Oil for use in engines
contains additives

BASE

+

Friction Modifier

Detergent
Antioxidant
VI improver
Dispersant
Antifoam Agent
Pour Point Depressant
Anti-Wear Agent
Seals Agent
Rust preventative
Metal Deactivator

Procedure for Oils Received and Tested

Oils sent out and
received containing
C750: fully formulated
lacking friction modifier

Test

Analysis

Results

BP FF no CA CI
BP FF with CA CI
BP FFnoFM no CA SI
BP FFnoFM with CA SI

Afton FFnoFM no CA SI
Afton FFnoFM with CA SI

Diluted to
1 wt%

Preparation for All Fluids

Shake – 1 min

Stir – 1 min

Sonicate – 5 min

Dilute

Perform Test

Sonicate – 5 min

Stir – 1 min

Shake – 1 min

Nanoplatelets that Lack a CA Will Agglomerate

We discovered that a capping agent is necessary to prevent agglomeration. A thick residue was always present on the bottoms of jars produced with no capping agent, even after stirring and shaking. The residue was never present on bottom of jars that **included** a capping agent.

Afton FFnoFM SI 5% C750 **with** CA

BP FF SI 5% C750 **with** CA

BP FFnoFM CI 5% C750 **with** CA

Afton FFnoFM SI 5% C750 **no** CA

BP FF SI 5% C750 **no** CA

BP FFnoFM CI 5% C750 **no** CA

Friction Comparison of C750 as FM

The addition of the C750 as a FM causes negligible change in friction for Afton FFnoFM SI

Friction Comparison of C750 as FM

The addition of the C750 as a FM is adverse when added to BP FFnoFM SI

Friction Comparison of C750 as FM

The addition of the C750 as a FM causes negligible change in friction for BP FF CI
(Note: BP FF CI is for diesel engines and contains no FM, so that FF is effectively noFM)

Summary of Previous Results

The main thrust for “practical tribology” is whether the nanoplatelets can stand in for a FM in any of the three oils studied.

The addition of the C750 as a FM causes negligible change in friction for the mineral oil. There is an adverse (larger friction) effect when it is added to the BP version with no FM. Finally, there is negligible change when it is added to the diesel BP.

Description of Following Plan

In the absence of a clear benefit for the neat nanoplatelets in the previous study, a study was conducted to determine whether the addition of commercial antiwear and antifriction additives could be used to beneficially **augment** the use of nanoplatelets.

Afton FFnoFM + 0.5% Molyvan 855

Afton FFnoFM + 0.5% ZDDP

Afton FFnoFM + 0.5% Molyvan 855 + 0.5% ZDDP

Afton FFnoFM + 1% ZDDP

Afton FFnoFM + 1.0% Molyvan 855 + 1.0% ZDDP + 1.0% C-750

Afton FFnoFM + 0.5% Molyvan 855 + 0.5% ZDDP + 0.5% C-750

Afton FFnoFM + 0.5% Molyvan 855 + 0.5% C-750

Afton FFnoFM + 1.0 % Molyvan 855 + 1.0 % ZDDP

Optical Images of Flats

Afton FFnoFM SI
131113e

BP FFnoFM SI
131113c

BP FF CI
131113a

Afton FfnoFM SI + 1 wt%
XGC750 with CA
131216a

BP FFnoFM SI + 1 wt%
XG C750 with CA
131218a

BP FF CI + 1 wt%
XGC750 with CA
131218b

Profilometric Images of Balls with Au Film

Afton FFnoFM SI
131113e, 312 loss

BP FFnoFM SI
131113c, 3821 loss

BP FF CI
131113a, 3267 loss

Afton FfnoFM SI + 1 wt%
XGC750 with CA
131216a, 4342 loss

BP FFnoFM SI + 1 wt%
XG C750 with CA
131218a, 1849 loss

BP FF CI + 1 wt%
XGC750 with CA
131218b, 1946 gain

Comparison of Ball Wear and Friction for Commercial FMs and Anti-Wear, Including C750

		Ave COF	Ball Wear (10 ³ µm ³)
1	0.5% Croda Perfad 3000	0.11	-1516
2	0.5% Croda Perfad 3050	0.11	-1192
3	0.5% Molyvan 822	0.12	-3518
4	0.5% Molyvan L	0.08	-8524
5	0.5% Molyvan 807	0.12	-5268
6	0.5% Molyvan 2000	0.11	-3707
7	0.5% Vanlube 289	0.11	-2271
8	0.5% Molyvan 855	0.05	-6691
9	0.5% Molyvan 855 repeat	0.05	-1711
10	1.0% Molyvan 855	0.10	-8027
11	0.5% ZDDP	0.12	-4723
12	1.0 % ZDDP	0.12	-4767
13	1.0% GMO	0.085	-4461
14	0.5% Molyvan 855 + 0.5% ZDDP	0.07	-8929
15	0.5% Molyvan 855 + 0.5% ZDDP repeat	0.07	-5914
16	1.0% Molyvan 855 + 1.0% ZDDP	0.05	-2352
17	0.5% Molyvan 855 + 0.5% C-750	0.09	-46291
18	0.5% Molyvan 855 + 0.5% ZDDP + 0.5% C-750	0.08	1477
19	1.0% Molyvan 855 + 1.0% ZDDP + 1.0% C-750	0.08	-42317

Tests of the C750 with and without Commercial Friction Modifiers Show Large Difference Depending on ZDDP and C750 Concentration

0.5% Molyvan 855 + 0.5% C-750

Wear -46291 µm³

0.5% Molyvan 855 + 0.5% ZDDP + 0.5% C-750

Wear 1477 µm³

1.0% Molyvan 855 + 1.0% ZDDP + 1.0% C-750

Wear -42317 µm³

Goals and Questions Related to Technique to Measure Mass Loading in Nanoplatelet Loaded Fluids

- Find an effective procedure to accurately determine the mass of nanoparticle additives in mineral/synthetic oils
- Use ASTM D4055 as a starting point
- Predict, test, and confirm systematic and random error in all segments of procedure apparatus
- These factors may be errors/offsets/anomalies and need to be assessed and controlled:
 - Humidity effect on mass measurement
 - Washing effect on mass measurement
 - Drying/cooling cycle effect on mass measurement
 - Does 2-minute limit affect our concentrations?
 - Oil remaining on funnel, annulus, or lost from filter surface
 - Mettler balance reproducibility
 - Drying cooling cycles on a filter that is loaded with oil particles
 - Fingerprint mass (for comparison)
 - Residue and remnants left behind on the funnel
 - Does filtering produce a “null” result when we know there are no particles present? (clear oil)

Filtration Diagram

Concept of Filtration Process

Note: weighing is done after each step

Wash filter with 100 mL Pentane

Weigh Filter

Place Filter in Oven at 90°C for 30 min.

Mix oil with 100 mL pentane in beaker

Measure out desired amount of oil in small bottle

Place filter in desiccator for 30 min.

Filtration Process (continued)

Place filter onto filtration apparatus

Attach funnel and clamp
Pour oil mixture down funnel

Wash around the funnel

Weigh filter

Place in desiccator for 30 min.

Place in oven for 30 min.

Spray pentane to wash annulus

Argonne Procedure

1. Agitate oil per preparation procedure (details elsewhere). Measure 1 ± 0.01 g into small bottle. Note that sonication was used only for the oils with no CA.
2. Wash the filter with 100 mL pentane.
3. Place filter in oven at $90 \pm 5^\circ\text{C}$ for 30 minutes. Remove filter from oven and place in the desiccator for a minimum of 30 minutes.
4. Weigh filter after removing from desiccator.
5. Fill the small bottle containing the oil sample with pentane and pour mixture into a 500 mL beaker. Repeat this several times to make sure all of the oil in the bottle is transferred to the beaker. Continue to add pentane to the beaker until it reaches the 100 mL mark. Mix the oil and pentane well.
6. Using forceps, place the filter onto the filtration apparatus. Assemble funnel and clamp.
7. Turn on the vacuum and pour the oil and pentane mixture down the funnel. The mixture must fully pass through filter in <120 seconds, if not, repeat with smaller oil sample size.
8. Wash the sides of the beaker several times with the wash bottle (~10 mL) and pour down funnel.
9. Rinse the walls of the filter funnel with a gentle stream of pentane from wash bottle (~10 mL). (We will refer to this as a “funnel wash.”)
10. Remove the clamp and funnel and gently wash the filter, particularly the edges, with pentane, using the wash bottle (~10 mL). (We will refer to this as an “annulus wash.”)
11. Release vacuum and remove the filter using the forceps.
12. Place filter in the oven for 30 minutes.
13. Remove the filter from oven and place in desiccator for 30 minutes. Weigh the filter.
14. Scrape off remnants and weigh them.

Oil Residue Estimation

- Question: Does filtering clear synthetic motor oil produce a “null” result when we know there are no particles present?

Procedure:

- Wash filter (**0.8 μm**) with 100 mL pentane (off rig), dry for 15 minutes in oven, weigh filter (**72.70 mg**) (baseline)
- Mix about 1 g (**1006.57 mg**) of oil (**DELO5W40FF**) and 100 mL pentane and run through the filter. Dry the filter in oven for 15 minutes. Weigh the filter (**76.44 mg**) (gain 3.74 mg due to unwashed oil).
- Wash 10 mL pentane down funnel while filter is inside the apparatus (funnel wash). Dry in oven for 15 minutes. Weigh filter (**74.04 mg**) (gain is down to only 1.34 mg after little rinsing).
- Place filter back onto the apparatus. Wash 100 mL pentane around the inside of the funnel (funnel wash). Dry in oven for 15 minutes. Weigh filter (**73.97 mg**) (gain is marginally down to 1.27 mg after large rinsing).
- Place filter back on but remove the funnel. Wash 100 mL pentane over the filter (annulus wash). Dry filter for 15 minutes in oven. Weigh filter (**73.40 mg**) (gain is 0.7 mg after full rinse everywhere).
- **Conclusion:** The mass gain from filtering a clear oil is a **0.7 mg**. The annulus wash is necessary to attain this low value. This value (0.7 mg) is small in comparison to mass gain from either the fluid measurements (~10–30 mg) or the centrifuge tube measurements (~10–20 mg).

Oil Residue Summary and Filter Properties

Clear oil type	Delta mass after washing
Delo synthetic FF (0.8)	0.7 mg
Afton mineral base (0.8)	0.08 mg
Afton mineral base (0.45)	0.29 mg
PAO10 (0.8)	-0.15 mg
Repeat	-0.11
PAO10 (0.45)	-0.19
PAO10 (0.45) annulus wash	-0.11

Conclusion: uncertainties are a fraction of a mg

Filter	Thickness	Mass (avg.)	Density
0.8 µm Pore	0.16 mm	72.18 mg	0.26 g/cm ³
0.45 µm Pore	0.15 mm	76.52 mg	0.29 g/cm ³
0.025 µm Pore	0.10 mm	116.16 mg	0.67 g/cm ³

Appearance of Known Concentrations of Particles Prior to Centrifugation

1:100 (0.5 g oil,
49.5 g PAO10)

1:1000

1:10000

Appearance of Known Quantities of Particles on Filter Paper

- The oils shown on the previous slide were filtered. Data from this filtration process is shown on the next slide.

1:100

1:1,000

1:10,000

Clear Afton Based Oil

With a starting material close to 11.5% solids (this data is shown later in the presentation), the 1:100 should be 0.11% = 57.5 mg, the 1:1,000 should be 0.011% = 5.75 mg, and the 1:10,000 should be 0.0011% = 0.575 mg.

Filtration of Known Dilutions

- Each sample shown had an oil mass of 50.0 g (0.50 g BP Oil, FF, CA, C.I + 49.50 mg PAO10)

	1:100	1:1000	1:10000
Untested Filter	68.10 mg	68.69 mg	68.41 mg
Wash with 100 mL Pentane	68.58 mg	68.78 mg	68.62 mg
30 min Oven	68.02 mg	68.38 mg	68.02 mg
30 min Desiccator	68.02 mg	68.34 mg	68.05 mg
Post Test	118.42 mg	72.78 mg	70.25 mg
30 min Oven	117.75 mg	72.65 mg	70.06 mg
30 min Desiccator	117.71 mg	72.62 mg	70.04 mg
Measured Particle Mass	49.69 mg	4.28 mg	1.99 mg
Predicted Particle Mass	57.5 mg	5.75 mg	0.575 mg
Particle Mass/Oil Mass	0.100%	0.009%	0.004%
Predicted Mass/Oil Mass	0.11%	0.011%	0.0011%

These results are consistent with our expectations that were mentioned on the previous slide. This confirms our accuracy of the testing.

Conclusion of Technique

- Pentane filtering method is useful for determining concentration of filterable particles with an Error Estimation as follows:
 - Humidity: ± 0.04 mg
 - Scale Reproducibility: ± 0.02 mg
 - Remnants: Collected solid masses range from 0.00 mg to 1.91 mg (most fall between 0.00 mg and 1.00 mg)
 - Residue (Afton Oils only): Collected solid masses range from 0.31 mg to 6.55 mg
- Syringe filtering method is useful for determining approximate size of solid particles suspended in the oils being tested. This gives us a good idea of what filter sizes to use for the pentane filtering method.
- We find that the residue and remnants that form on the funnel and annulus are significant. It is important to collect and weigh them when collecting filtration data.
- 0.025 μm filters are too small to use for this testing.
- The scale reproducibility is ± 0.03 mg and has a standard deviation of 0.0095.
- Fingerprints do have a significant mass. Gloves must be worn.
- From the wash tests, we can see that the mass gain from filtering clear oils (Afton based and PAO10 in our case) is very small compared to the results that we get from filtering the nanoplatelet oils.

5 Hour Centrifuge Syringe Filter Testing BP Oil, FF, CA, C.I.

BP

Conclusion: small difference in particle size due to centrifugation

Syringe Filter Estimation of Particle Size

BP Oil, FF, CA, C.I.

BP

BP Oil, FFnoFM, CA, S.I.

BP

Afton Oil, FFnoFM, CA, S.I.

Afton

BP Oil, FF, No CA, C.I.

BP

BP Oil, FFnoFM, No CA, S.I.

BP

Afton Oil, FFnoFM, No CA, S.I.

Afton

Conclusion: Estimated particle size is between 0.45 and 1.0 μm for oils with CA, but particle size is smaller for oils without CA.

Thermal Effects on Reaction Products or Volatilization

- Here we investigate whether it is necessary to test to see if the 90°C heat causes evaporation of volatile fractions (mass loss), reacts with ambient air to form reaction products (mass gain), or whether no mass is lost or gained. The results are shown below.

		30 min Heat	30 min Heat	5 hr Heat	5 hr Heat
BP Oil, FF, CA, C.I. (Cook Only)	Before Centrifuge	After Centrifuge (Top)	After Centrifuge (Bottom)	After Centrifuge (Top)	After Centrifuge (Bottom)
Untested Filter	67.36	67.95	68.28	68.52	69.12
1st Wash 100 mL Pentane	67.65	68.08	68.43	68.78	69.24
30 min Oven	67.22	67.75	68.1	68.22	68.82
30 min Desiccator	67.31	67.86	68.21	68.22	68.83
Oil Mass	10.00 g	10.00 g	10.00 g	10.00 g	10.0 g
Post Test	84.85	87.56	84.39	78.49	80.56
30 min Oven	84.49	87.31	83.98	78.05	80.28
30 min Desiccator	84.52	87.34	83.94	78.05	80.69
Particle Mass	17.21	19.48	15.73	9.83	11.86
Particle M/Oil M	0.175%	0.198%	0.159%	0.983%	0.119%
Remnants	0.3	0.36	0.14	0.00	0.00

This test does not show any measurable changes due to heating.

(KBA58A) BP Oil, FF, CA, C.I.

100 mL pentane dilution	0.8 µm (1)	0.8 µm (2)	0.8 µm (3)	0.8 µm (4)	0.8 µm (5)
Untested Filter	73.54	76.1	72.85	72.69	71.93
First Wash with Pentane	73.74	76.38	73.05	72.74	71.81
30 min Oven	73.21	75.74	72.23	72.01	71.31
Oil Mass	1003	1008	254	259	243
Post-test	191.67	191.59	101.32	101.47	99.11
30 min Oven	190.25	190.1	100.91	101.03	98.66
Dried Particle Mass	117.04	114.36	28.68	29.02	27.35
Particle Mass over Oil Mass	11.67%	11.53%	11.36%	11.32%	11.44%
Collected Remnants		1.91	0.18	0.3	0.45
Remnants over Total Particle Mass		1.64%	0.62%	1.02%	1.62%

This test was done using the given procedure shown previously.

BP Oil, FF, CA, C.I. <10 mL dilution pentane (minimum amount to transfer oil)	Sample 1 0.8 µm filter
Untested Filter	68.27 mg
1st Wash with 100 mL Pentane	68.35 mg
30 min Oven	67.74 mg
30 min Desiccator	67.91 mg
Oil Mass	249.09 mg
Post Test	95.44 mg
30 min Oven	95.2 mg
30 min Desiccator	95.21 mg
Particle Mass	27.3 mg
Particle Mass/Oil Mass	11.37%
Remnants	1.01 mg
Remnants/Total Particle Mass	3.57%

This test is done using only the minimum amount of pentane to transfer oil from beaker to filter.

- Capping agent suspends nanoparticles in oil.
- With 0.8 µm membrane, no visible particles in filtered pentane. The fluid coming through was fairly clear.
- With 1 g oil mass, test took 4+ minutes. With 250 mg oil mass, test took 1 min 40 sec.
- Including remnants caught in annulus, solids represent 11.3%–11.8% of oil mass, ±<0.2%

BP

*All units are in mg

For this test, the oil sample that is being filtered is mixed with only 10 mL pentane prior to filtration instead of 100 mL as the procedure described.

BP Oil, FF, CA, C.I. 10 mL dilution Pentane	Sample 1 0.8 µm
Pretest	67.01 mg
1st Wash with 100 mL Pentane	6.2 mg
30 min Oven	66.96 mg
30 min Desiccator	67 mg
Oil Mass	255.33 mg
Post Test	95.02 mg
30 min Oven	94.71 mg
30 min Desiccator	94.75 mg
Particle Mass	27.75 mg
Particle Mass/Oil Mass	10.97%
Remnants	0.26 mg
Remnants/Total Particle Mass	0.93%

(KBA57A) BP Oil, FFnoFM, CA, S.I.

BP

*All units are in mg

100 mL dilution pentane	0.8 µm (1)	0.8 µm (2)	0.8 µm (1)	0.45 µm filter (1)	0.45 µm filter (2)
Untested Filter	75.07	72.31	72.03	76.55	76.72
1st Wash with Pentane	75	72.63	72.12	76.6	76.84
30 min Oven	74.77	72.37	71.81	76.24	76.4
30 min Desiccator	74.77	72.47	71.92	76.3	76.51
Oil Mass	253.56	148.02	103.31	105.11	59.7
Post Test	100.55	87.96	82.42	86.77	82.86
30 min Oven	100.06	87.62	82.21	86.43	82.53
30 min Desiccator	100.25	87.67	82.31	86.44	82.52
Particle Mass	25.48	15.2	10.39	10.14	6.01
Particle Mass/Oil Mass	10.07%	10.28%	10.16%	10.17%	10.64%
Collected Remnants	0.05	0.02	0.06	0.55	0.34
Remnants/Total Particle Mass	0.20%	0.13%	0.57%	5.15%	5.35%

BP Oil, FFnoFM, CA, S.I. 10 mL dilution Pentane	Sample 1 0.8 µm
Pretest	67.73 mg
1st Wash with 100 mL Pentane	67.89 mg
30 min Oven	67.59 mg
30 min Desiccator	67.59 mg
Oil Mass	105.72 mg
Post Test	78.13 mg
30 min Oven	77.93 mg
30 min Desiccator	78.16 mg
Particle Mass	10.57 mg
Particle Mass/Oil Mass	10.33%
Remnants	0.35 mg
Remnants/Total Partical Mass	3.21%

- It took over 2 minutes for filtering to be completed for 0.8 µm (1), 0.8 µm (2), and 0.45 µm (1).
- With the 0.8 µm filter, the fluid coming through the filter into the collection flask was fairly clear with some black particles swirling in the flask liquid. With the 0.45 µm filter, the fluid coming through still had a few black particles, but visibly fewer particles than the 0.8 µm filter.

(KBA54A) Afton Oil, FFnoFM, CA, S.I.

*All units are in mg

Afton	0.8 μ m (1)	0.8 μ m (2)	0.8 μ m (3)	0.8 μ m (4)	0.45 μ m (1)
Untested Filter	72.34	72.81	72.47	75.88	76.03
1st Wash with Pentane	72.42	73.97	72.69	75.96	76.14
30 min Oven	72.28	72.74	72.25	75.63	75.76
30 min Desiccator	72.32	72.74	72.1	75.68	74.82
Oil Mass	249.71	245.45	246.16	244.17	247.42
Post Test	94.99	93.89	94.15	98.28	99.61
30 min Oven	94.49	93.36	93.64	97.66	99.39
30 min Desiccator	94.65	93.06	93.73	97.65	99.18
Particle Mass	22.33	20.32	21.63	21.97	24.36
Particle Mass/Oil Mass	8.94%	8.28%	8.91%	9.52%	9.85%
Residue Mass	*	*	0.31	1.28	*
Residue Mass/Particle Mass	*	*	1.41%	5.51%	*
	*	*			*
Kimwipe Mass	NA	NA	2747.58	3077.84	NA
Wipe and residue	*	*	2747.89	3079.12	*
Residue Mass	*	*	0.31	1.28	*

- With the 0.8 μ m filter, the fluid coming through the filter into the collection flask was fairly clear with some visible black particles floating swirling in the flask liquid. With the 0.45 μ m filter, the fluid coming through still had a few black particles, but visibly fewer particles than the 0.8 μ m filter.
- This oil left a residue behind in the funnel. This residue was weighed using a kimwipe. The mass reading of the kimwipe fluctuates greatly when on the scale, which makes it difficult to accurately measure the residue.

(KBA60A) BP Oil, FF, No CA, C.I.

BP	0.8 μ m (1)	0.8 μ m (2)	0.8 μ m (3)	0.45 μ m (1)	0.45 μ m (2)	0.45 μ m (3)	0.45 μ m (4)
Untested	*	*	*	76.49 mg	76.31 mg	76.48 mg	76.53 mg
1st Wash	*	*	*	76.72 mg	76.45 mg	76.9 mg	76.88 mg
30 min Oven	*	*	*	76.35 mg	76.09 mg	76.41 mg	76.5 mg
30 min Cool	72.75 mg	72.72 mg	75.93 mg	76.42 mg	76.12 mg	76.47 mg	76.47 mg
Oil Mass	252 mg	218 mg	141 mg	208.63 mg	103.78 mg	51.51 mg	43.38 mg
Post Test	*	*	*	92.34 mg	87.32 mg	92.21 mg	80.07 mg
30 min Oven	*	*	*	92.02 mg	86.87 mg	91.85 mg	79.79 mg
30 min Cool	99.03 mg	94.62 mg	85.74 mg	92.04 mg	87.01 mg	91.92 mg	79.84 mg
Particle Mass	26.28 mg	21.9 mg	9.81 mg	15.62 mg	10.89 mg	15.45 mg	3.37 mg
Particle Mass/Oil Mass	10.43%	10.05%	6.96%	7.60%	10.78%	30.60%	9.94%
Collected Remnants	*	*	*	0.22 mg	0.3 mg	0.31 mg	0.94 mg
Remnants/ Total Particle Mass	*	*	*	1.39%	2.68%	1.97%	21.81%
Filtration Time				30 min	25 min	25 min	2 min

- With the 0.8 μ m filter, the fluid coming through the filter into the collection flask was a lighter gray/black color but was still slightly darker than full transparency. With the 0.45 μ m filter, the fluid coming through was still slightly gray, but had fewer particles

(KBA59A) BP Oil, FFnoFM, No CA, S.I.

All units are in mg unless otherwise stated.

BP	0.8 μm (1)	0.8 μm (2)	0.8 μm (3)	0.8 μm (4)	0.45 μm filter (1)	0.45 μm filter (2)	0.45 μm filter (3)
Untested Filter	67.37	66.59	66.95	67.48	76.54	76.6	75.77
1st Wash with Pentane	67.47	66.69	67.2	67.7	76.85	76.53	76.11
30 min Oven	67.16	66.5	66.85	67.34	76.42	76.14	75.71
30 min Desiccator	67.15	66.48	66.91	67.39	76.47	76.14	75.83
Oil Mass	242.62	156.16	109.99	104.2	115.04	58.5	37.94
Post Test	82.95	76.4	74.93	76.23	86.59	81.53	79.55
30 min Oven	81.31	76.19	74.65	76.23	86.18	81.2	79.2
30 min Desiccator	81.37	76.24	74.67	76.37	86.3	81.25	79.23
Particle Mass	14.21	9.76	7.76	8.98	9.83	5.11	3.4
Particle Mass/Oil Mass	6.01%	6.26%	7.01%	8.93%	9.13%	8.97%	9.12%
Collected Remnants	0.49	0.01	0.2	0.32	0.67	0.14	0.06
Remnants/Total Particle Mass	3.33%	0.10%	2.51%	3.44%	6.38%	2.67%	1.70%
Time Taken to Complete	10 min	3 min	2 min	2 min	10 min	3 min	1 min

- With the 0.8 μm filter, the fluid coming through the filter into the collection flask had a light gray/black color but still had reasonable transparency. With the 0.45 μm filter, the fluid coming through the filter was not quite as dark as the 0.8 μm filter but still had some visible black particles .
- It is easily seen from the data that the 0.45 μm filter caught more of the solid particles than the 0.8 μm filter.

(KBA56A) Afton Oil, FFnoFM, No CA, S.I.

Afton	0.8 μm (1)	0.8 μm (2)	0.8 μm (3)	0.45 μm filter (1)	0.45 μm filter (2)
Untested Filter	67.43 mg	67.87 mg	66.62 mg	76.72 mg	76.24 mg
1st Wash with Pentane	67.65 mg	68.06 mg	66.81 mg	77 mg	76.64 mg
30 min Oven	67.39 mg	67.81 mg	66.55 mg	76.62 mg	76.13 mg
30 min Desiccator	67.5 mg	67.89 mg	66.59 mg	76.63 mg	76.1 mg
Oil Mass	192.73 mg	248.39 mg	253.6 mg	236.64 mg	254.96 mg
Post Test	78.74 mg	83.35 mg	80.95 mg	93.85 mg	94.44 mg
30 min Oven	78.45 mg	82.88 mg	80.78 mg	93.33 mg	93.91 mg
30 min Desiccator	78.48 mg	82.88 mg	80.83 mg	93.42 mg	93.93 mg
Particle Mass	10.98 mg	14.99 mg	14.24 mg	16.79 mg	17.83 mg
Particle Mass/Oil Mass	7.87%	6.48%	6.90%	9.86%	8.07%
Collected Residue	4.19 mg	1.1 mg	3.25 mg	6.55 mg	2.74 mg
Remnants/Total Particle Mass	27.62%	6.84%	18.58%	28.18%	13.32%

- All runs were completed in under 2 minutes.
- With the 0.8 μm filter, the fluid coming through the filter into the collection flask was fairly clear with some visible black particles swirling in the flask liquid. With the 0.45 μm filter, the fluid coming through still had a few black particles, but visibly fewer particles than the 0.8 μm filter.

Images of Results of Centrifugation in Parent Oil 5 Hour

- The samples with capping agent became semi-transparent while the samples without capping agent remained opaque. Consistent with previous tests in PAO 10.

BP

BP

BP

BP

(KBA56A) Afton Oil, FFnoFM, No CA, S.I.

Effects of Horn Sonication on Agglomeration

- Horn sonication at 55% power, pulsed: on 10 seconds/off 2 seconds, 1/8 inch horn
- KBA59A 0.1% diluted in parent BP FFnoFM
- Images at 100X

No Sonication

Sonication 45 min

- Little to no effect from either sonication

Effects of Bath Sonication on Agglomeration

- Bath sonication 37 kHz for 1 hour, KBA59A 0.1 wt% diluted in PAO10

No Sonication

Sonication

Syringe Filter Weighing Results

- Each weight represents the mass gain in the filter filtered from 10g of oil
- Both oils repeated to see if consistency could be found

		No Syringe Filter	5 μm Filter	1 μm Filter	0.45 μm Filter
BP	KBA59A	10.15 mg	6.66 mg	4.14 mg	N/A
BP	KBA57A	11.56 mg	4.77 mg	6.8 mg	N/A
BP	KBA59A	8.05 mg	4.02 mg	5.88 mg	0.02 mg
BP	KBA57A	11.00 mg	4.51 mg	6.88 mg	0.30 mg

- The results show that the filters remove substantial quantities of filtrate causing the weight to be less. The 0.45 μm filter very effective but the larger filters are only modestly effective

Optical Images of Syringe Filtered Samples

Visual representation of what is passing through each syringe filter.

KBA57A (BP S.I. capping agent) diluted in PAO 2 to roughly 1 wt%.

All optical images taken in a wedge of fluid. Particles in the plane of focus have sharp edges and particles above or below the plane of focus appear as rings.

The syringe filters progressively remove the larger particles from the mixture leaving only small particles.

No Syringe Filter

Cameo 5 μm

Whatman 1 μm

Target 0.45 μm

Qualitative Results of Centrifugation

- The following slides are comparative images for the purpose of drawing qualitative conclusions.
- These samples were diluted in the parent oil prior to centrifugation.
- Nominal 0.1 wt% nanoplatelet, 0.5 g of sample diluted in 49.5 g of parent oil

Qualitative Results of Centrifugation

- Diluted in parent oil after 30 min
- Nominal 0.1 wt% nanoplatelet, 0.5 g of sample diluted in 49.5 g of parent oil

Qualitative Results of Centrifugation

- Diluted in parent oil after 5 hours
- Conclusion: Samples with capping agent settle faster
- Nominal 0.1 wt% nanoplatelet, 0.5 g of sample diluted in 49.5 g of parent oil

Qualitative Results of Centrifugation

- Diluted in PAO 10 after 5 hours
- Conclusion: nanoplatelets settle faster in PAO 10 than in the parent oils
- Nominal 0.1 wt% nanoplatelet, 0.5 g of sample diluted in 49.5 g of PAO 10

Particle Size Measurement

- Test results of particle size were obtained using dynamic light scattering

Calibration of DLS Is Correct

- Multiple polymer microsphere standards tested to see if the Nanotracer is operating correctly
- Tested in water at room temperature
- Viscosity of water: 0.797 cP @ 30°C, 1.002 cP @ 20°C

22 nm Poly

- Summary -	
Data	Value
MV(nm):	17.31
MN(nm):	13.95
MA(nm):	16.05

100 nm Poly

- Summary -	
Data	Value
MV(nm):	93.00
MN(nm):	84.30
MA(nm):	90.00

220 nm Poly

- Summary -	
Data	Value
MV(nm):	200.8
MN(nm):	183.2
MA(nm):	194.6

- When looking at the number weighted sizes they appear to be consistently 10–27% smaller than their known size

Particle Size Measurement

- Test results of particle size were obtained using dynamic light scattering

BP SI Samples 30 Minutes of Centrifugation

BP CI Samples 30 Minutes of Centrifugation

CI Capped Before Centrifugation:

30 min Top:

30 min Bottom:

BP_{CI}

BP_{CI}

CI Uncapped Before Centrifugation:

30 min Top:

30 min Bottom:

BP SI Samples 5 Hours of Centrifugation

SI Capped Before Centrifugation:

BP_{SI}

5 Hour Top:

5 Hour Bottom:

BP_{SI}

SI Uncapped Before Centrifugation:

5 Hour Top:

5 hour Bottom:

BP CI Samples 5 Hours of Centrifugation

CI Capped before Centrifugation

BP_{CI}

BP_{CI}

CI Uncapped Before Centrifugation

5 Hour Top

5 Hour Top

5 Hour Bottom

5 Hour Bottom

Conclusions from Centrifugation and DLS

- The capped particles appear to be diametrically larger than the uncapped when measured using DLS. This is consistent with the capped particles possessing an outside “shell.” For equal density, a larger particle will settle faster; therefore if capped particles are larger than the uncapped it would lead one to believe that they would settle faster and clarify first. If the shell density were much less than the core, then the settling properties could reverse. The size distributions are insignificantly different, and thus no conclusions about the shell properties are warranted.
- After 30 min of centrifugation most of the size distributions of the remaining particles are smaller, suggesting that the larger particles are indeed settling first.
- This can be seen to an even greater extent in the 5 hour tests. Longer centrifugation times produce increasingly smaller particle size measurement as the large particles fall out. In the BP CI capped sample some particles smaller than 100 nm exist that were not detected at all prior to centrifugation.

Filtration Conclusions

- Unquantifiable variations are:
- Scale: small
- Heating: 0.22 mg
- Desiccation of filter: small
- Desiccation of loaded filter: 0.4 mg
- Errors when filtering clear oil: +0.3 to -0.2 mg
- Six oils that had nominally 5 wt% XG C750 loading had a measured loading of 8–11%; this is reproducible
- The quantity of pentane use for dilution or rinsing was not important
- If stirred, sonication of the oils was not important
- Visually, the tip quantity gave some indication of the amount of particles spun down into the tip and out of the oil
- Any mass measurement on the order of a few mg was of questionable validity
- The centrifuge top- and bottom-particle mass measurements made no sense despite measuring dozens of mg of material
- At 90°C, masses of large particles would settle into the tube tip with no centrifugation
- With centrifugation at 90°C, oil would be largely clarified after 5 hr but not after 30 min
- Highly diluted oil gave correctly small mass measurements before centrifugation, but gave large mass measurements after centrifugation despite the filters being practically “white”

Limitations of DLS for Small Loading

- On the previous slide, an example was shown of how the Nanotracer will force from whatever it is detecting, meaning it could output useless data from erroneous background noise.
- There are two ways to tell if the Nanotracer is actually detecting a particle.
- First, the user must pay attention to the “loading” portion of the test. The loading index should be at a reasonable value (ideally in the green zone above 0.1). Anything below 0.01 is not a strong enough signal to indicate with certainty that the results are valid. Furthermore, one can look at the loading image. If a clear distribution shape like the one shown below is seen, then one can be fairly certain the Nanotracer is detecting a distinct particle.

- Another way to make certain the data is not being forced from background noise is by comparing the measurement “raw” data to the “setzero” data. In order to be considered valid data, the signal strength of the raw data must be on a much higher order of magnitude than the setzero data.

Chan	Raw
1	1.34E-06
2	1.27E-06
3	1.24E-06
4	1.29E-06
5	1.32E-06

Chan	Raw
1	1.24E-01
2	1.37E-01
3	1.54E-01
4	1.78E-01
5	2.06E-01

Signal strength of setzero data is typically on the order of 10^{-6} while good raw data for a measurement should be around 10^{-1} to 10^{-4} .

Viscosity Data Obtained under the CRADA

Fluid	40C Visc (cSt)	100C Visc (cSt)	VI	-40C Visc (cP)	-35C Visc (cP)	-30C Visc (cP)	-25C Visc (cP)	-20C Visc (cP)
BP 427A FFnoFM syn	56.02	10.74	187	7085	3861	2263	1419	
BP 427A FF no FM+0.5% XG C500				6853	3762	2212		
BP 428A FF syn	54.22	10.35	184	7137	3894	2286	1443	
BP 428A FF+0.5t% XG C500				7095	3884	2255		
BP 429Al base syn	23.10	5.04	152	3313	1883	1117	713	
BP 429A base+0.5% XG C500				3318	1897	1108		
BP 430A disp only syn	30.66	6.12	152	4926	2776	1650	1024	
BP 430A disp only+0.5% XG C500				4806	2733	1632		
Delo 400 0W30 syn	49.9	10.09	195	8061	4383	2536	1588	994
Delo 400 0W30+0.5% XG C750	48.28	9.27	178					
Delo 10W30	78.97	11.7	141					
Delo 10W30+0.5% XG C300	201.1	11.42	-17					
Delo 10W30+0.1% XG C300	79.9	11.70	139					
Delo 10W30+0.5% C750	72.35	10.05	122					
Mobil1 10W30	64.43	10.36	149	33500	14595	6635	3389	2071
Mobil1 10W30+0.1% XG C300	65.2	10.45	149					
Mobil1 10W30+0.1% XG C300	59.0	9.50	144					
Mobil1 10W30+0.5% XG C300	63.8	10.21	147					
Mobil1 10W30+0.5% XG C750	71.3	11.1	147					
PAO10	70.03	10.44	136	38014	18561	9865	5561	
PAO10+ 0.1% XG C300	70.6	10.28	131					
PAO10+0.5% XG C300	70.5	10.20	129					
PAO10+0.5% XGC750	90.5	14.07	160					

Summary

The viscosity was either obtained at 40°C and 100°C, or was obtained at subzero temperature. In any case, any large deviations due to additive interactions would be apparent.

- It is apparent that there is some scatter in the baseline data so that only large trends can be relied upon.
- **C300** was mixed with Delo10W30. The effect is modest (0.1%) for 0.1 and is anomalous for 0.5% because thickening at 40°C is not consistent with an absence of an effect at 100°C.
- **C300** was mixed with Mobil1 10W30 and with PAO10. There seems to be no systematic effect or significant effect with either.
- **C500** was mixed into the BP oils. The effect is a tiny lessening of subzero viscosity.
- **C750** was mixed with Delo 0W30. At 0.5% there is no discernable effect.
- **C750** was mixed with Delo 10W30. At 0.5% there seems to be significantly less viscosity.
- **C750** was mixed with Mobil110W30. At 0.5% there seems to be a slightly higher viscosity.
- **C750** was mixed with PAO10. There was a distinctive increase in viscosity.

Overall Summary

- Friction and wear of C500 mixtures
 - C500 additions to a variety of commercial oils that were additized with commercial friction and wear modifiers were not effective in reducing friction, and wear reduction was mixed. It is likely that C500 particles compete with additives in performing their functions. In base oils (p. 8) the effect is positive. Optical images show mixed results.
- Oils obtained lacking friction modifiers – friction
 - The emphasis of the CRADA was shifted to “Practical Tribology” to examine the use of nanoplatelets as friction reducers by blending with commercial oils that lacked friction modifiers. This would specifically measure their friction-reducing properties in the presence of the full chemical package.
 - The capping agent is necessary to prevent agglomeration.
 - The addition of the C750 as a FM causes negligible change in friction for the mineral oil. There is an adverse (larger friction) effect when it is added to the BP version with no FM. Finally, there is negligible change when added to the diesel BP, which has no natural FM.
- Oils obtained lacking friction modifiers – augmentation of commercial friction modifiers
 - The addition of C750 has no benefit when added as an augmentation to the additized nonFM oils.
- Wear – FFnoFM or with augmentation
 - The addition of the C750 has no clear influence on wear when added to commercial nonFM oils, and had mixed influence on wear when augmenting commercial AW and FM added to commercial nonFM oils.
- Filtration mass measurement
 - A procedure was developed for filtration mass measurement. The procedure and sources of error were identified.
 - It was necessary to undertake additional experiments involving large dilutions to understand the behavior of the provided mixtures because they did not behave in a simple manner.
 - The six provided oil mixtures that were blended to be a nominal 5 wt% loading had a measured loading of 8–11%. This quantity was repeatable with good accuracy.
 - It was found that the quantity of pentane used for dilution or rinsing was not important, and if stirred, sonication of the oils was not important to the mass measurements.
 - Any mass measurement that was a few mg or less was of questionable validity because unquantifiable variations for heating, dessication, and oil adsorption had a maximum of 0.4 mg.
 - At 90°C, masses of large particles would settle into the tube tip with no centrifugation and with centrifugation at 90°C, oil would be largely clarified after 5 hr but not after 30 min.
- Syringe filters
 - Regardless of fluid and treatment, the filtration-method particle sizes were generally in a size band between 0.2 μm and 1 μm .
- Sonication
 - For the mixtures studied, sonication using a horn sonicator or a bath sonicator produced no obvious redistribution of particles to more uniformly homogeneous sizes; the reverse occurred, optically showing increased agglomeration with sonication.
- Particle Size
 - Particle size as measured using dynamic light scattering was consistently near a diameter of several hundred nm. Trends toward smaller retained particle size were observed for centrifuged mixtures (on top and on bottom), but the effect was not large. Capped and uncapped showed only slightly different sizes.
- Viscosity
 - The viscosity results, both below 0°C and at the standard 40°C and 100°C test temperatures, show that the fluid viscosity is remarkably unaffected by the introduction of nanoplates. There was no significant viscosity effect, and in some instances the viscosity decreased at low temperatures, which would provide increased pumpability.

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