Proposal: Company ABC 2020-00x (EXAMPLE QUOTE)

Title: Rheological Evaluation of xxxxx

Contact: XXXXXX

**Purpose:** Perform preliminary comparative rheological characterization on xxxx

#### **COMMON ASSAYS & COST ESTIMATES**

- •Below are the most commonly requested assays that can be performed with a broad array of parameters to meet your particular rheology needs. These and other assays not listed may be performed in various combinations.
- •Overview and application of proposed assays below can be found in more detail at <a href="https://www.rheologytestingservices.com/">https://www.rheologytestingservices.com/</a>. The homepage also contains a link to slides for "Rheology Principles and Applications" presentation.
- •Fees, Deliverables, and Considerations start on page 5.

Assay	Assay Description	# Samples Submitted	# Assay/ Sample	\$ Per Assay	\$ (USD) Total
	Suggested assay inputs may change depending on material and needs.	Submitteu	Sample	Assay	Total
1	ROTATION - Yield Stress Ramp ("flow curve")				
	<b>Description:</b> Shear stress controlled, <u>non-equilibrium</u> ramp. Measures				
	viscosity vs shear stress. Determine yield stress and yield viscosity to				
	quantify initiation of sample flow. Helpful to identify impact of stress				
	during manufacturing, application (spreadability, pumpability,				
	syringability) and performance, as well as for sample handling for testing.				
	Suggested Inputs:				
	Shear stress: 0-300Pa over 5min				
	Temperature: 25°C (working range 5-180°C)				
	Report: Yield stress and yield viscosity summary table, overlay plots.				
2	ROTATION – Increasing/Decreasing Shear Rate Ramp				
	<b>Description:</b> Shear rate controlled. Measures viscosity vs shear rate				
	increase, then decrease as a continuous ramp to screen for post-shear				
	(ir)reversibility (hysteresis). Helpful to identify impact of shear rate during				
	manufacturing, application (spreadability, pumpability, syringability) and				
	performance, as well as for sample handling for testing. For reference,				
	pouring and dripping are ≈1-10sec <sup>-1</sup> .				
	Suggested Inputs:				
	Increasing shear rate: 0.01* - 500sec <sup>-1</sup>				
	Decreasing shear rate: 500 - 0.01*sec <sup>-1</sup>				
	Temperature: 25°C (working range 5-180°C)				
	*Depending on sample, response at low shear rate may be noisy and may				
	be adjusted.				
	<b>Report</b> : Overlay plots, summary table of viscosity at several shear rates.				
3	ROTATION – <u>Stepwise</u> Increasing/Decreasing Shear Rate				

	<b>Description:</b> Shear rate controlled. Measures viscosity with stepwise shear			
	rate change as next step criteria are met to provide a more stabilized			
	viscosity result rather than acquiring data across a continuous ramp.			
	Quantify viscosity vs shear rate and determine post-shear (ir)reversibility			
	(hysteresis). Helpful to model impact of shear rate during manufacturing,			
	application (spreadability, pumpability, syringability) and performance, as			
	well as for sample handling for testing. For reference, pouring and			
	dripping are ≈1-10sec <sup>-1</sup> .			
	, the Q			
	Suggested Inputs:			
	Increasing shear rate: 0.01* - 500sec <sup>-1</sup>			
	Decreasing shear rate: 500 - 0.01*sec-1			
	Temperature: 25°C (working range 5-180°C)			
	*Depending on sample, response at low shear rate may be noisy and may			
	be adjusted.			
	be adjusted.			
	<b>Report</b> : Overlay plots, summary table of viscosity at several shear rates.			
4	ROTATION: 3-Step Thixotropy (post-shear rebuilding)			
'	<b>Description:</b> Step 1 measures viscosity at low shear (baseline). Evaluate			
	rebuild time and extent (Step 3) after exposure to shear thinning (Step 2).			
	Helpful to identify impact of shear forces during sample handling for			
	testing as well as for product manufacturing, delivery and performance.			
	For reference, pouring and dripping are ≈1-10sec <sup>-1</sup> .			
	Suggested Inputs (may change based on response to shear forces):			
	Step 1: 0.01*sec <sup>-1</sup> , 3min			
	Step 2: xxsec <sup>-1</sup> , 10sec			
	Step 3: 0.01*sec <sup>-1</sup> , 3-10min			
	Temperature: 25°C (working range 5-180°C)			
	*Response at low shear rate may be noisy and may be adjusted.			
	Bounds Our developed to the series Channel 2 and 2 arranged to be of			
	<b>Report:</b> Overlay plots showing Steps 1, 2, and 3, summary table of			
	averaged viscosity for each step.			
5	ROTATIONAL: Heat/Cool Ramp Cycle at Single Shear Rate			
	(single sample loading per cycle)			
	<b>Description:</b> Measure viscosity vs increasing/decreasing temperature			
	cycles while typically applying a low as possible fixed shear rate having			
	acceptable signal/noise. Helpful for process development and stability			
	screen by evaluating potential for (ir)reversible changes (hysteresis).			
	Suggested Inputs:			
	-Shear rate: TBD* sec <sup>-1</sup> .			
	-Temperature: up/down temperature ramps over n cycle(s)			
	$(T_0 - T_1 - T_0)_n$ at $10^{\circ}$ C/min with 2min hold at $T_1$ and 2min hold at final $T_0$ .			
	(working range 5-180°C)			
	Report: Overlay plots, summary table of viscosity at several			
	temperatures.			
	temperatures.			
	*If a suitable shear rate is not determined, can perform as a single			
	frequency (typically 1Hz) rather than based on fixed shear rate.	-	-	
6	ROTATION: Creep-Recovery			

	<b>Description</b> : Evaluate viscoelastic properties by measuring sample			
	recovery over time following an applied, then released shear stress often			
	over multiple stress-release cycles.			
	Suggested Inputs:			
	Shear stress: TBD Pa (select from pre-yield values from Assay 1)			
	Temperature: 25°C (working range 5-180°C)			
	<b>Report</b> : Overlay plots, summary table of compliance at several times.			
7	OSCILLATION: Amplitude (Strain) Sweep			
,	<b>Description:</b> Apply an increasing strain (force) at a constant frequency			
	until sample rheologically "breaks" to determine upper LVER (Linear			
	Viscoelastic Region) as % strain. Using %strain within the LVER is a critical			
	input for frequency assay to ensure rheological integrity. Upper LVER limit			
	is defined as 5% decrease in G' (elastic modulus) indicating start of			
	structural breakdown with increasing amplitude. LVER tends to correlate			
	with physical stability and strength of intermolecular interactions.			
	Suggested Inputs:			
	Frequency: 1Hz (default)			
	% strain: 0.1 to 100% strain			
	Temperature: 25°C (working range 5-180°C)			
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	Report: LVER determination, overlay plots, summary table at selected			
	%strains			
8				
٥	OSCILLATION: Single Frequency over Time (typically strain controlled)			
	<b>Description:</b> Measure changes in G' (elastic modulus), G" (viscous			
	modulus), $\delta$ (phase angle), $\eta^*$ (complex viscosity), and G* (complex			
	modulus) vs over time at a constant frequency (typically 1Hz) and % strain			
	within the LVER determined from amplitude sweep. Identify potential for			
	rheological change over time and temperature used for other assays.			
	Unaccounted for changes may confound interpretation results of assays.			
	Suggested Inputs:			
	Frequency: 1Hz (typical default)			
	%strain: xx (determined from amplitude sweep)			
	Temperature: 25°C (working range 5-180°C)			
	Time: TBD (typically 30min)			
	Time. Tob (typically softlin)			
	Report: Overlay plots and summary table at selected frequencies for			
	measurables noted in "Description".			
9	OSCILLATION: Frequency Sweep (typically strain controlled)			
	<b>Description:</b> Measure G' (elastic modulus), G" (viscous modulus), $\delta$ (phase			
	angle), $\eta^*$ (complex viscosity), $G^*$ (complex modulus), $G'$ - $G''$ crossover (if			
	observed, typically not observed for gels) vs frequency at a constant %			
	strain determined from an amplitude sweep. Generates a rheological			
	"fingerprint". Helpful to evaluate properties to support Q3. Quantifies			
	solid/liquid nature of material to model processes having short- and long-			
	timeframes corresponding to high and low frequency, respectively.			
	Suggested Inputs:			
	Frequency: 20-0.1Hz			
	%strain: xx (determined from amplitude sweep)			
	Temperature: 25°C (working range 5-180°C)			
	Temperature. 23 C (working range 3-100 C)	l	L	

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	Report: Overlay plots and summary table at selected frequencies for			
	measurables noted in "Description".			
10	VERTICAL: Squeeze Pull-Away			
	<b>Description:</b> Compress sample between lower and upper plates, then pull			
	plate apart at define rate. Quantifies the force (stickiness, adhesion,			
	cohesion) required to vertically separate sample between plates. These			
	values can correlate with human sensory panel results.			
	Report: Overlay plots, summary table of peak pull-away force (Newtons),			
	area under the curve (N-sec), time force is reduced 90% of peak (sec).			
11	VERTICAL: Surface Tension (requires 4mL minimum)			
	<b>Description:</b> A DuNouy ring is slowly raised through the sample to			
	quantify the liquid-liquid or liquid-air interface tension due to the			
	attraction force (Newtons) exerted from asymmetric intermolecular			
	interactions that can appreciably differ from the more balanced			
	interactions within the sample.			
	<b>Report</b> : Individual and average surface tension for n=6 replicates.			
12	ROTATIONAL: Tribology (friction, lubricity)			
	<b>Description:</b> Measures resistance as torque (N-m) as sample is exposed to			
	a bearing rotating with increasing velocity under a defined normal force.			
	Suggested Inputs:			
	Start and End Velocity: 0.0001 to 100 radians/sec			
	Normal Force: 1N (upper limit 40N (4,040g)			
	Temperature: 25°C (working range 5-180°C)			
	Report: Overlay of Stribeck curves (coefficient of friction vs angular			
	frequency), summary table of coefficients of friction at selected angular frequencies.			
	Report (included in fees and defined in "Deliverables" below)	 	0	0
		 TOTAL (xx assays)		\$xxxx

#### Fees for most assays listed above\* (back to top)

\$500 minimum per submitted set since involves running performance standards and report.

1-10 assays \$175/assay 11-25 assays \$150/assay >26 assays \$125/assay

#### **Deliverables**

- -Summary of results and experimental details
- -Overlays plots
- -Summary tables listing primary results to highlight trends.
- -Excel files of results
- -Follow-up discussion to review results and address questions as needed

#### **GENERAL CONSIDERATIONS** (may not be applicable to all sample types)

- Overview and application of proposed assays can be found at https://www.rheologytestingservices.com/.
- Assays are performed under non-GMP.
- Multiple assays per sample may be helpful depending on desired precision.
- Since this study is a basic comparative screen, specific test parameters are not optimized for each sample and are based on best estimates from experience.
- Ideally <u>at least</u> 2mL per sample per assay is requested, especially for low viscosity samples. If sample is scarce, many assays can be performed with 200uL (transferrable) using a small plate with small gap between plates although accuracy and precision may decrease. <u>Having extra samples would be very helpful in case method refinement and repeat analyses are necessary.</u>
- Unless requested otherwise, samples will be stored at ambient conditions prior to and after analysis. Please communicate any special hazard, handling, preparation or storage needs prior to finalization of the quote.
- Please note if sample contains volatiles at assay temperatures. Loss of volatiles may affect results if not accommodated with use of a solvent trap to saturate the sample test environment.
- Assays can be performed with sample under low-flow N<sub>2</sub> to reduce potential for degradation. Request if needed.
- Samples will be handled and applied to the rheometer with a high level of consistency and care so as to minimize potential shear force that could bias results.
- Unless requested, samples will <u>not</u> be pre-sheared. Pre-shearing can be helpful to rheologically normalize samples that may undergo shear thinning during sample handling. However, depending on the material sensitivity to pre-shearing, some rheological properties may be erased. It is recommended to consider preshearing if deemed necessary based on observed rheologicial sensitivities of sample.
- Lower viscosity samples may generate variable results at suggested lower shear rates and may necessitate adjusting if poor signal to noise response is observed.
- Unless specified, assays are typically performed with 25mm diameter roughened parallel plates to reduce potential for slippage and minimize sample usage. Low viscosity samples or samples requiring highly accurate viscosity measurements will be assayed with a 40mm 4° cone (smooth) assuming adequate sample is provided.
- Assays performed with high shear rates may require using a flat plate with narrow sample gap (200um) to retain sample within plates. Low viscosity samples are more prone to spin out from plates at high shear rate.

<sup>\*\$250</sup> for longer assays such as temperature cycling and single frequency sweep 1-2hrs, then \$100/hr for assays >2hrs.

- While a cone may provide better rheological resolution, it is not suggested for working for wide ranging temperature ramp due to potential sample expansion.
- Unless otherwise stated, all analysis will be performed with samples equilibrated at 25.0°C.
- A draft report will be submitted for review. The report will be considered finalized after 1 week with no response and invoice for payment will be submitted (30 days net upon receipt of invoice).

#### **SAMPLES**

Indicate any safety, handling, or disposal considerations <u>prior to sample shipment</u> in case Rheology Testing Services cannot properly accommodate.

- Samples are to be clearly labeled with unique IDs and accompanied by appropriate Safety Data Sheets (SDS), along with any special handling and storage information.
- A screw-top glass or plastic vial, bottle or jar is preferable to crimp-top container/closures. If samples are submitted in crimp-top containers, please provide additional several caps per sample to reseal.
- Arrange for sample delivery to occur on a weekday since no one may be present to receive on weekends.
- Please forward shipment tracking information. You will be notified when samples arrive.

#### Ship samples to:

Mark Patrick 103 Nettle Ridge Chapel Hill, NC 27517

#### Please note:

- Unless specifically requested otherwise, samples will be retained for 30 days after report is approved and then properly disposed according to local Environmental requirements.
- If unused samples are to be returned to client, unless prepaid by requestor or is local (RTP area North Carolina) sample return will incur a pass-through cost plus \$50 for handling.

#### **Payment Terms**

Prepayment of 0% of the total estimated project cost will be required to initiate the study. Net 30 upon receipt of invoice for the remaining balance at the study completion. Electronic funds transfer can be arranged. Otherwise, payment by check should be made and sent to:

Pharma Developability Solutions Consulting, LLC 103 Nettle Ridge Chapel Hill, NC 27517 (c/o Mark Patrick)

#### **Duration**

With coordination of sample shipment with Rheology Testing Services, work is typically initiated within 2 business days upon receipt of samples and approved purchase order with a signed quote <u>or</u> via email response with quote stating approval. Approximate duration of the project is typically 3-5 days depending on number of samples and type of testing.

**Note:** This proposal is based on information provided by the client. If the information is inaccurate or incomplete, this proposal is subject to change.

Submitted By:	Approved By:
Mark Patrick, Ph.D.	Name:
Owner, Rheology Testing Services	Title:
Preparation Date: dd-mmm yyyy	Date:
Quote Valid Until: dd-mmm yyyy (2 months)	PO #: