

TITLE:

Dielectric RheoSANS – Simultaneous Interrogation of Impedance, Rheology and Small Angle Neutron Scattering of Complex Fluids

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SHORT ABSTRACT:

A procedure for the measurement of simultaneous impedance, rheology and neutron scattering from soft matter materials under shear flow.

LONG ABSTRACT:

A procedure for the operation of a new Dielectric RheoSANS instrument capable of simultaneous interrogation of the electrical, mechanical and microstructural properties of complex fluids is presented. The instrument consists of a Couette geometry mounted on a commercial rheometer that is contained within a modified forced convection oven and is available for use on the small angle neutron scattering beamlines at the National Institute of Standards and Technology (NIST) Center for Neutron Research (NCNR). The Couette geometry is machined to be transparent to neutrons and provides for measurement of the electrical properties and microstructural properties of a sample confined between the cylinders while the sample undergoes arbitrary deformation. Synchronization of these measurements is enabled through the use of a customizable LabVIEW code that monitors and controls the execution of predetermined experimental protocols. Described here is a protocol to perform a flow sweep experiment where the shear rate is logarithmically stepped from a maximum value to a minimum value holding at each step for a specified period of time while frequency dependent dielectric measurements are made. Representative results are shown from a sample consisting of a gel composed of carbon black aggregates within propylene carbonate. As the gel undergoes deformation, the carbon black network is mechanically deformed, which has implications for both the apparent microstructure and electrical characteristics of the sample. These results are discussed and potential future improvements to the instrument summarized.

INTRODUCTION:

Measurement of macroscopic properties are often used to gain fundamental insight into the nature of colloidal materials and self-assembled systems, usually with the goal of developing understanding in order to improve formulation performance. In particular, the field of rheology, which measures a fluid's dynamic response to an applied stress or deformation provides valuable insight into colloidal behavior both under equilibrium conditions, and also far from equilibrium, such as during processing.¹ Rheological tests of consumer and industrial fluids, gels, and glasses can also be used to measure rheological parameters, such as viscosity, attributes that are targeted by formulators. While rheology is a powerful probe of material properties, it is an indirect measurement of colloidal properties at the microscopic level, such that our understanding of fundamental colloidal behavior can be greatly enhanced by combining rheological measurements with complementary techniques.

One such orthogonal technique is impedance spectroscopy. Impedance spectroscopy is a bulk probe of dielectric relaxation behavior, which measures the response of a material to an applied oscillating electric field.² The impedance spectrum results from electrical relaxation modes that are active within the material including charge transport and polarization.^{3,4} These measurements provide additional evidence for colloidal behavior particularly when combined with rheology.⁵ Therefore, the combination of these techniques is especially relevant when probing charged colloidal dispersions, proteins, ionic surfactants, nanocomposites, and other systems.^{6,7}

Of fundamental interest in investigations of colloidal behavior is the material's microstructure. The microstructure of a colloidal fluid is thought to encode all of the information necessary to reconstitute both the rheological and electrical behavior of a complex fluid. Fundamentally, we seek to measure a snapshot of the nanoscale microstructural features that lead to a measured material response. Due to the complicated nature of many complex fluids' dependence on their process history, much of the effort on microstructural characterization has focused on making in situ measurements of the material as it undergoes deformation. This has

challenged experimentalists to devise methods to be able to make measurements of nano-sized particles under for example steady shear, where the velocities of the particles have made direct visualization intrinsically challenging. Direct measurement of material microstructure under flow has taken on many forms ranging from small rheo-optics, rheo-microscopy and even rheo-NMR.^{8–10} Small angle scattering methods and in particular small angle neutron scattering (SANS) techniques have proven themselves effective at measuring the time-averaged microstructure of samples at steady state in a bulk shear field including all three planes of shear.^{11–13} New data acquisition techniques have allowed structural transience to be capture with time resolution as affine as 10 ms.¹⁴ Indeed combining rheology with various in situ scattering methods has proven invaluable in hundreds of recent studies.¹⁵

An emerging engineering challenge is the use of colloidal suspensions as conductive additives in semi-solid flow battery electrodes.¹⁶ In this application, conductive colloidal particles must maintain an electrically percolated network while the material is pumped through an electrochemical flow cell. The performance demands on these materials require that they maintain high conductivity without detrimental effect on the rheological performance over a wide range of shear rates.¹⁷ It is therefore highly desirable to be able to make measurements of the colloidal behavior under steady and time-dependent shear conditions in order to quantify and characterize the underlying rheological and electrical response of these materials far from their equilibrium state. A significant complicating goal that has hindered further theoretical development in this regard is the thixotropic nature of carbon black slurries.¹⁸ Their history dependent rheological and electrical properties make experiments notoriously difficult to reproduce making it difficult to compare data sets measured using varying protocols. Also to date, there is no single geometry capable of performing all three, dielectric, rheological, and microstructural characterizations, simultaneously. Simultaneous measurement is important as the flow can change the structure, such that rest measurements of processed materials may not provide accurate indications of the properties under flow, which are more relevant for their use. Furthermore, as many of the measured properties of carbon black slurries are geometry dependent, there are complications with comparing data obtained from the same sample on different instruments.¹⁹

In order to meet this challenge in measurement science, we have developed a new Dielectric RheoSANS geometry at the NIST Center for Neutron Research and the University of Delaware capable of in situ impedance spectroscopy, rheology and small angle neutron scattering measurements of a material under arbitrary deformation on a commercial ARES G2 Rheometer. This is enabled by developing a Couette geometry capable of measuring the microstructural, electrical and rheological response of a material confined between the gap between two concentric cylinders. As the outer cylinder spins, torque imposed by the deformation of the sample is measured on the inner cylinder and the impedance measurement is made radially across the gap. The cylinders are machined from titanium so as to be transparent to neutrons and robust enough to withstand the shear stress experienced in the rheometer. We perform the SANS measurement through the radial position of the Couette, and have demonstrated that it is possible to measure high quality SANS patterns from the sample undergoing deformation. In this way, all three measurements are made on the same region of interest in the sample undergoes a well-defined deformation profile. The goal of this article is to describe the Dielectric Couette Geometry, its installation onto the ARES G2 RheoSANS instrument, and the successful executing of a simultaneous measurement. This rheometer is available at the NIST Center for Neutron Research at the National Institute of Standards and Technology. It has been designed to work on the NG-7 SANS beam line. We have provided drawings and a detailed description of the custom components that have been machined

and assembled in order to enable this measurement.

PROTOCOL:

1. Mounting the ARES G2 Rheometer onto the SANS Beamline. See Figure 1 for definitions of named components.
 - 1.1) Ensure that the power to the rheometer is off, the transducer is locked and the motor air bearing protector is installed.
 - 1.2) Ensure that the power to the rheometer is off, the transducer is locked and the motor air bearing protector is installed.
 - 1.3) Turn off the neutron beam.
 - 1.4) Close and Secure the Oven Door.
 - 1.5) Install large base plate onto Huber Table.
 - 1.6) Remove the snout and install the window.
 - 1.7) Secure 4 eyelets to mounting brackets for ARES G2 crane adapter such that the cables do not tangle and are not twisted.
 - 1.8) Using crane, lift Rheometer and maneuver it from the Rheometer table to rest centered on the Huber table with the LCD screen of the Rheometer facing outward taking care to guide the cables to minimize tangling.
 - 1.9) Send Huber Table to minimum Z position.
 - 1.10) Remove the ARES G2 crane adapter and lift off the platform using the crane.
2. Dielectric Cell Assembly – See Figure 2 for definitions of named components.
 - 2.1) Ensure that the power to the rheometer is off, the transducer is locked and the motor air bearing protector is installed.
 - 2.2) Clean Dielectric Cup and Bob Assemblies before use.
 - 2.3) Open the oven door and swivel it out of the way.
 - 2.4) Unlock Transducer and remove motor bearing lock.
 - 2.5) Mount Dielectric Geometry and Dielectric Bob Assembly onto the upper and tool mounts of the ARES G2 Rheometer.
 - 2.6) Loosen both set screws on the Dielectric Geometry using a 2 mm Allen key.
 - 2.7) Place the Dielectric Cup Assembly so that it is mounted on the Dielectric Geometry.
 - 2.8) Zero the gap using TRIOS software.
 - 2.9) With gap zeroed, apply 10 N Normal Force using TRIOS software and tighten screws using a 3 mm Allen key until Dielectric Cup Assembly is fully secured to Dielectric Geometry.
 - 2.10) Set the gap to the operating gap using TRIOS software.
 - 2.11) Swivel the oven into its closed position and ensure that the oven can fully enclose the Dielectric Cell with adequate vertical clearance on the top and bottom of the geometry.
 - 2.12) If height adjustment is needed, adjust the set screw so that the oven enclosure fits with adequate tolerance around the Dielectric Cell.
 - 2.13) Remove both the Dielectric Bob Assembly and the Dielectric Cup Assembly/Dielectric Geometry as one piece and replace with the Rheometer Alignment tool on the lower tool head.
3. Install the Slip Ring. See Figure 3 for Step by Step Summary.
 - 3.1) Install the Wire Baffle onto the shaft of the Dielectric Geometry.
 - 3.2) Connect the Dielectric Cup Connector to the Slip Ring Connector.

- 3.3) Hold the Slip Ring so that it is concentric with the shaft of the Dielectric Cup Assembly/Dielectric Geometry but above the flange on the Dielectric Geometry.
- 3.4) Place the Slip Ring Adapters (x2) such that their nobs insert into the holes drilled into the Dielectric Geometry and their base rests on the Dielectric Geometry flange.
- 3.5) Gently slide the Slip Ring Over the Slip Ring Adapters. The Slip Ring should slide effortlessly around the Slip Ring Adapters holding them in place.
4. Alignment of the Rheometer. See Figure 4 for Schematic of Beam Path.
 - 4.1) Install the Slip Ring.
 - 4.2) Close the oven around the Rheometer Alignment tool.
 - 4.3) Install the truncated snout and the sample aperture (1mm wide \times 8 mm tall)
 - 4.4) Using TRIOS, set the angle to 0.49 rad.
 - 4.5) Using the NICE software, ensure that all the neutron guides are removed. Open the oven door so that the laser is visible.
 - 4.6) Perform a rough alignment of the rheometer by maneuvering the it so that the beam passes through the oven and crosses through the slit in the center of the Rheometer Alignment Tool.
 - 4.7) Using NICE Software, adjust the height of the Huber table and its rotation to optimize laser alignment. Note the rheometer is aligned when the laser beam passes through the slit in the Rheometer Alignment Tool set at an 0.49 rad angle without impinging on its walls and the beam passes through the center line in the oven.
5. Calibrate SANS instrument
 - 5.1) Perform configurations alignment
 - 5.2) Run Open Beam, Empty Cell, and Dark Current Corrections
6. Connecting the Electric Components
 - 6.1) Set the gap using the LCD screen to 100 mm.
 - 6.2) Remove the Rheometer Alignment Tool from the bottom tool flange.
 - 6.3) Reinstall the Dielectric Bob Assembly on the upper tool head and the Dielectric Cup Assembly/Dielectric Geometry/Slip Ring Assembly onto the lower tool head as one piece and re-zero the gap.
 - 6.4) Ensure that the Carbon Brush Assembly is secured to the Carbon Brush Adapter using screws.
 - 6.5) Secure the Carbon Brush Adapter and Carbon Brush Assembly to the rheometer using screws.
 - 6.6) Ensure that the carbon brushes on the Carbon Brush Assembly mate with the grooved metal rings of the Slip Ring. This ensures maintenance of electrical contact.
 - 6.7) Connect the female pin connectors on the Carbon Brush Assembly and the Dielectric Bob Assembly to the male pin connectors of the top and bottom bus bars respectively.
 - 6.8) Ensure that the labeled shielded BNC cables connected to the bus bars and terminating at the LCR meter are installed in their corresponding BNC connectors.
 - 6.9) Connect the BNC cable labeled "TO SANS" to the BNC cable connected to the DAQ card labeled "AO0".
 - 6.10) Connect the BNC cable labeled "FROM SANS" to the BNC cable connected to the DAQ card labeled "AI0".
 - 6.11) Connect the BNC cable labeled "TRIGGER" to the BNC cable connected to the DAQ card labeled "AO1".

- 6.12) Connect the BNC cable connected to the 15 pin connector on the back of the Rheometer to the BNC cable labeled "AI3".
- 6.13) Ensure the LCR meter and Rheometer are communicating with control computer.
7. Connecting the Electric Components
 - 7.1) Open Oven.
 - 7.2) Set the Gap to 100 mm.
 - 7.3) Load 4 mL of sample into the temperature equilibrated Dielectric Cup Assembly taking care to minimize sample left on the Cup Wall.
 - 7.4) Lower the Geometry to 40 mm using the Front LCD Screen.
 - 7.5) Set the velocity on TRIOS motor control settings to 1 rad/s.
 - 7.6) Using the slew option on the ARES G2, lower the Dielectric Bob Assembly until the gap distance is at 0.5 mm.
 - 7.7) Using TRIOS, go to geometry gap.
 - 7.8) Set the velocity on TRIOS motor control settings to 0 rad/s.
 - 7.9) Ensure that the sample level fills all the way up the Couette wall without overfilling.
 - 7.10) Install the Solvent Trap by filling the inner Dielectric Bob Assembly Wall with the desired solvent and place the solvent trap on the rim of the Dielectric Cup Assembly.
8. Running the Dielectric RheoSANS Experiment
 - 8.1) Configure LabVIEW Code Labeled "TA_ARES_FlowSweep.vi"
 - 8.1.1) Specify path for log file to and the base name of log file.
 - 8.1.2) Run the LabVIEW code by pressing the "Run" arrow button on the menu bar.
 - 8.1.3) Select Rheological Parameters
 - 8.1.4) Select Temperature
 - 8.1.5) Select Preshear Conditions (if desired, enable radio button to "ON")
 - 8.1.6) Specify Time per Shear Rate and Collection Rate
 - 8.1.7) Enable Handshaking radio button
 - 8.1.8) On Test Parameters Tab Select Logarithmic or Linear Sweep – if radio button is green, a list of N number of points will be logarithmically spaced from min shear rate to max shear rate.
 - 8.1.9) Discrete Shear Rates and Times can also be specified via the "Discrete Values" tab.
 - 8.1.10) Select number of frequency points, frequency minimum and frequency maximum default.
 - 8.1.11) Set Time Dependent Frequency – specifies the desired time dependent frequency for all shear rates.
 - 8.1.12) Set Time for Steady State – sets the amount of time that the code will measure dielectric parameters as a fixed frequency as a function of time for each shear rate.
 - 8.1.13) Specify the signal type and amplitude.
 - 8.1.14) Specify the number of cycles to average and the measurement time.
 - 8.2) Turn AutoLogging on SANS computer.
 - 8.3) Set SANS Configuration
 - 8.3.1) Select configuration and specify run time to be at least 1 minute longer than the total time contained within the shear rate list in the LabVIEW Code.
 - 8.3.2) When the configuration is achieved VIPER should read "dio stat 16" which indicates that it will be waiting for the Analogue Signal from the LabVIEW code to change.

8.4) Configuring TRIOS

8.4.1) In the experiment tab, Press “Open Procedure File” in the “Procedure” drop down menu.

8.4.2) Navigate to the IGOR Procedure File Labeled “Dielectric RheoSANS Script File”

8.4.3) Ensure that Rheometer is ready to execute experiment.

8.5) When the SANS is ready, LabVIEW is configured and Trios script file is open, Press “Parameters Set”.

9. End of Experiment

9.1) Turn off the neutron beam and disable AutoLogging.

9.2) Unload the sample and remove the Dielectric Cup and Bob Assemblies from the rheometer.

9.3) Install the motor air bearing protector and lock the transducer.

9.4) Power down the computer, LCR Meter, and rheometer power supplies.

9.5) Disconnect the air line.

9.6) Disconnect all BNC Cables and reinstall the crane lift onto the rheometer.

9.7) Uninstall the truncated snout.

9.8) Reinstall the ARES G2 crane adapter.

9.9) Lift the rheometer from the Huber table and place onto the rheometer table ensuring that the cables remain untangled.

[Insert Figure 1 here]

Figure 1: a.)-e.) Pictures of Components of the SANS beamline and the ARES G2 Rheometer necessary to Install rheometer on the beamline that are labelled and defined below.

[Insert Figure 2 here]

Figure 2: a.)-e.) Pictures of components Dielectric RheoSANS geometry with labels defining terms below.

[Insert Figure 3 here]

Figure 3: a.-d.) Pictures of Procedure for Installing the Slip-Ring onto the Dielectric RheoSANS geometry.

[Insert Figure 4 here]

Figure 4: Schematic of Beam Path through Oven Geometry and Dielectric RheoSANS Geometry.

REPRESENTATIVE RESULTS:

Representative results from a Dielectric RheoSANS experiment are shown in Figure 5 and 6. These data are taken on a suspension of conductive carbon black in propylene carbonate. These aggregates flocculate due to attractive interactions at relatively low solids loadings forming gels that are electrically conducting. The rheological and conductivity responses of such suspensions are an active area of research and current investigations seek to understand the microstructural origins of the rheology and conductivity. The Dielectric RheoSANS instrument is a tool uniquely suited to address this question as it probes simultaneously the electrical and mechanical properties of a material as it undergoes deformations similar to those found in an application such as in a semi-solid electrochemical flow cell. In such a cell the carbon black forms the conducting additive that provides volumetric conductivity to the flowing electrodes.

The experiment outlined in the procedure is designed to test a conductive material as it undergoes a flow sweep test, where the shear rate is stepped logarithmically from a maximum value to a minimum value holding at each shear rate for a specified period of time. Rheology, dielectric data and neutron scattering are measured continuously during the course of this experimental sequence. Upon completion of a Dielectric RheoSANS experiment, the data is stored in three independent formats. The SANS data is stored as an event mode file that is a binary file generated by the detector containing the list of the time of arrival of each neutron on the detector and the x, y position of the pixel on which it was detected. The rheology data is stored with a TRIOS data file and can be exported within the TRIOS software. Finally, the dielectric data is contained within the log file written by the LabVIEW interface to the specified folder. The first post-processing task is to synchronize and then sort the raw data. A detailed description of this process is published elsewhere, but briefly, the synchronization is made possible by a data acquisition card that monitors a digital signal from the ARES G2 rheometer and uses an analogue triggering protocol to encode measurement condition transitions into the SANS detector clock time. Using this approach, the raw measured signals from the SANS, rheometer and LCR meter can be reconstructed as a function of both shear rate and time.

After the raw signals are sorted, they are corrected. The dielectric data correction and analysis procedure is shown in Figure 5a after removing the open and short circuit measurements at each frequency and shear rate. Once corrected the dielectric signals are converted to the real and imaginary components of the impedance versus frequency. In figure 5a, there is a plot of Nyquist representation of dielectric measurement of an 0.08 weight fraction Vulcan XC72R sample undergoing steady shear averaged over the last 900s of the acquisition. In the Nyquist representation the real and complex components of the impedance are plotted parametrically against one another. On the top left plot, the data points are logarithmically colorized by the frequency at which the measurement is taken with yellow representing the highest frequency (20 MHz) and black representing the lowest accessible frequency (20 Hz). In the middle plot, the sample admittance, Y^* , or the inverse of the complex impedance, Z^* , is plotted against the frequency. It is normalized by the known cell constant, λ , and the sample conductivity and electrical susceptibility are defined as the imaginary and real components of the admittance. This normalized sample response can be converted to the complex permittivity, ϵ^* , by dividing the admittance by $2\pi f\epsilon_0$. Finally, we fit the complex permittivity of the sample response using the dielectric response model as a sum of a Havriliak-Nagami Relaxation and a Constant Phase Element that accounts for the effects of electrode polarization.²⁰

[Place Figure 5 here]

Figure 5: a.) Summary of Dielectric Data Analysis; *left* Nyquist Representation, *middle*: Conductivity and Susceptibility vs frequency, *right*: Complex Permittivity versus Frequency – Dielectric Model account for electrode polarization and Havriliak-Negami Relaxation shown overlaid on top of data, b.) Summary of SANS Data Analysis; *left*: $I(Q)$ from 0.08 weight fraction Vulcan XC72R at 1 rad/s averaged for last 900s of shear rate, *middle*: scaled model fit to dilute sample $P(Q)$, *right*: sample structure factor, $S(Q)=I(Q)/(A \cdot P(Q))$ – red circle denotes Q -position position where data is averaged to obtain the minimum structure factor depth, S_0 .

The raw event mode data is histogrammed with respect to time onto the two-dimensional SANS detector representing $I(Q_x, Q_y)$. This raw signal intensity is then corrected for the empty cell, blocked beam, and transmission and converted to absolute scale with units cm^{-1} . After these

corrections, the normalized absolute intensity can be plotted as a function of shear rate and time. In Figure 5b, on the left, the two-dimensional reduced scattering intensity versus Q_x and Q_y is plotted. In the middle we plot the scaled form factor, $P(Q)$ model fit for the dilute sample over an identical Q -range. We then divide $I(Q)$ by $A \cdot P(Q)$ to obtain $S(Q)$ which represents an apparent structure factor for the interactions between the fractal carbon black aggregates that comprise the sample. Next the two-dimensional $S(Q)$ plot is integrated at the minimum accessible Q value $= 0.0015 \text{ \AA}^{-1}$ to calculate S_0 , which is an estimate of the apparent repulsive interaction between fractal aggregates. This result is then converted to an equivalent hard sphere volume fraction.

Using this approach, the data can be analyzed at each shear rate and the extracted parameters that result from both the structural analysis and the dielectric analysis can be plotted as a function of the applied shear rate and rheological shear stress as shown in Figure 6. Also plotted are the two-dimensional $S(Q)$ plots for several shear rates of interest that mark important microstructural transitions. Because these values are all measured at the same time from the same region within the Couette, they can be directly compared and correlated. This is emphasized by the fact that transitions in conductivity, κ_{LF} , and effective volume fraction, ϕ_{HS} , correspond with the increase of stress when the shear stress exceeds the yield stress marked by the transition from region I-II. In this transition, both ϕ_{HS} and κ_{LF} decrease which is associated with the yielding of the macroscopic gel. As the shear rate is further increased, the sample shear thickens as indicated by the apparent increase in the viscosity and the κ_{LF} increases while ϕ_{HS} continues to decrease. This transition is marked by region II-III. For concentrated colloidal suspensions, shear thickening is associated with the formation of large structures that form as result of hydrodynamic interactions imposed by the flow of the bulk fluid around the primary carbon black particles. These hydrodynamic forces draw the aggregates together resulting in an abrupt increase in conductivity and viscosity.

[Place Figure 6 here]

Figure 6: *top:* two-dimensional $S(Q)$ plots at shear rates that represent important microstructural transitions in the sample, *bottom:* summary of rheological (shear stress and viscosity), dielectric (static permittivity and low frequency conductivity) and SANS parameters (scale factor and effective excluded volume fraction) as a function of applied shear rate. The regions of interests are marked as I-III. In region I, creep maintains an interconnected network structure. In region II, the gel macroscopically yields leading to a decrease in the overall conductivity. In region III, there is an apparent shear thickening resulting in clustering and an increase in conductivity. Error bars represent one standard deviation of the average.

The technical schematics of the key components of the Dielectric RheoSANS geometry shown in Figure 2 are provided in Figures 7-14, such that this geometry can be reproduced on any commercial ARES G2 rheometer.

Figure Legends:

[Place Figure 7 here]

Figure 7: Technical Schematic of the Dielectric Cup Adapter.

[Place Figure 8 here]

Figure 8: Technical Schematic of the Dielectric Cup Wall.

[Place Figure 9 here]

Figure 9: Technical Schematic of the Dielectric Bob Wall.

[Place Figure 10 here]

Figure 10: Technical Schematic of the Dielectric Bob Shaft.

[Place Figure 11 here]

Figure 11: Technical Schematic of the Dielectric Bob Cap.

[Place Figure 12 here]

Figure 12: Technical Schematic of the Dielectric Bob Assembly.

[Place Figure 13 here]

Figure 13: Technical Schematic of the Slip Ring Adapter.

[Place Figure 14 here]

Figure 14: Technical Schematic of the Carbon Brush Adapter.

DISCUSSION:

A Dielectric RheoSANS experiment measures simultaneously the rheological, electrical and microstructural responses of a material as it undergoes a predefined deformation. The example shown here is electrically conductive carbon black suspension that forms the conductive additive used in electrochemical flow cells. The Dielectric RheoSANS instrument enables the interrogation of the radial plane of shear within a narrow gap Couette cell without compromising the fidelity of any of the measurements. In the experiment outlined in this procedure, a flow sweep is performed where the shear rate is logarithmically stepped from a maximum value to a minimum value while the time-dependent and shear rate dependent rheo-electro-structural properties are recorded. Synchronization of all three measurements is facilitated by the implementation of an analogue triggering protocol.

The results provided by this new instrument open up a new path to interrogate electrically active colloidal materials as they undergo deformation. This technique has relevance not only to electrochemical flow cells but the development of fuel cell electrodes and other electronic devices where materials are processed from the solutions state and subject to macroscopic shear. The instrument also has relevance to the study of materials whose mechanical properties can be actuated via an applied electric field. All these applications can potentially be studied by virtue of the flexible design of this instrument and the methodology for synchronizing the execution of each testing protocol.

Work is ongoing to improve the protocols for executing a Dielectric RheoSANS and creating new testing methodologies for a wide range of materials. Additionally, improved atmospheric control will be enabled with the improvement of the oven design and upcoming replacement of the window material within the oven environment. This will include an improved solvent trap design that will make long duration experiments on volatile fluids feasible. Upcoming oven designs promise access to the tangential plane of shear which has been demonstrated in operating RheoSANS instruments, but is not currently a tested and proven capability of the Dielectric RheoSANS instrument.

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DISCLOSURES:

The authors have nothing to disclose.

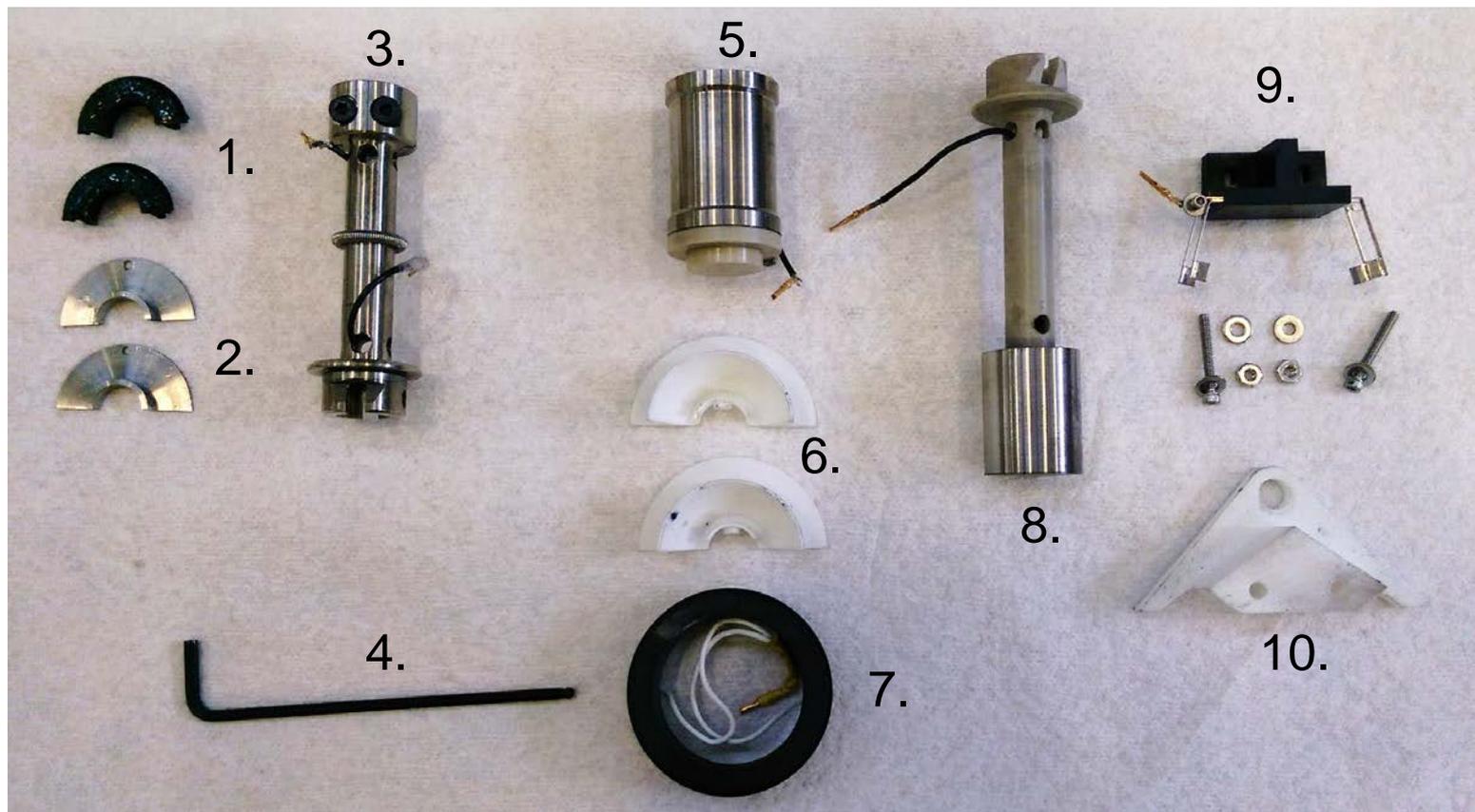
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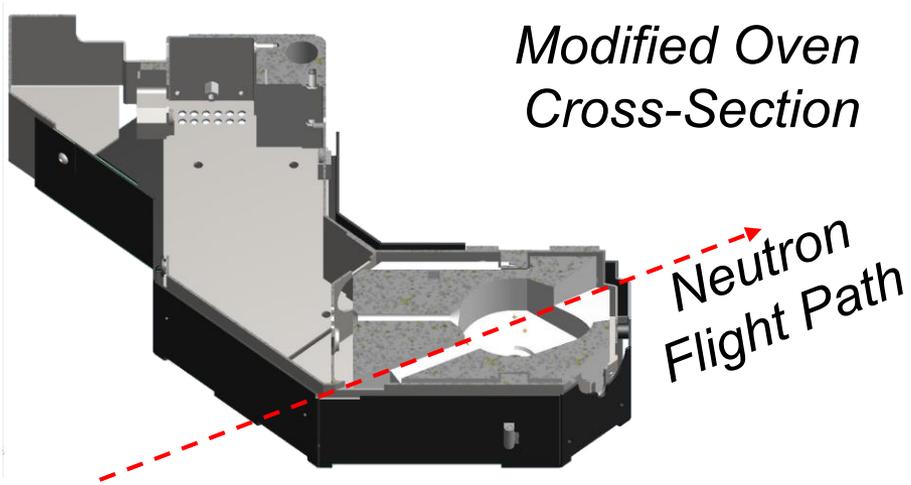
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|--------------------|----------------|-------------------|
| 1. Transducer Lock | 5. Window | 9. Neutron Guides |
| 2. Motor Lock | 6. Crane Mount | 10. ARES G2 |
| 3. Snout Mount | 7. Crane Hook | 11. Detector Tube |
| 4. Huber Table | 8. Cables | |



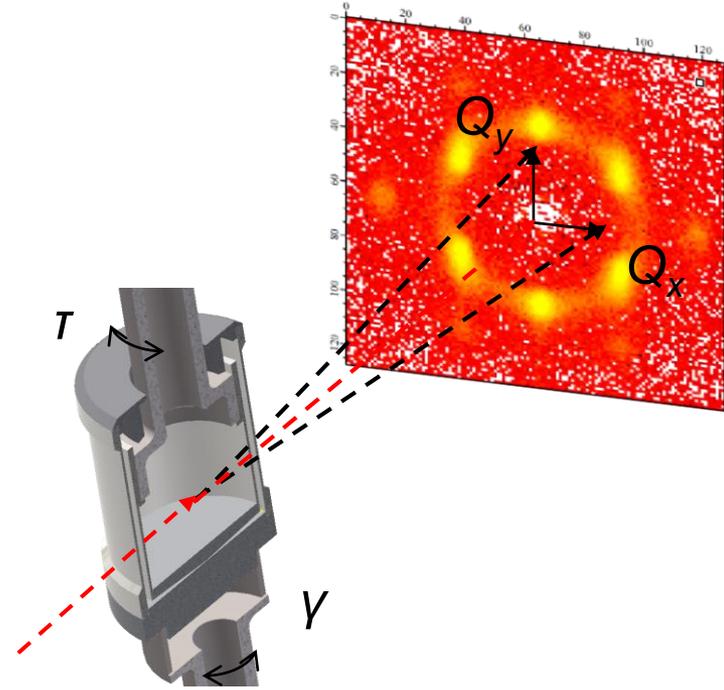
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|-------------------|----------------------|------------------|
| 1. Solvent Trap | 5. Dielectric Cup | Assembly |
| 2. Wire Baffle | Assembly | 9. Carbon Brush |
| 3. Dielectric | 6. Slip Ring Adapter | Assembly |
| Geometry | 7. Slip Ring | 10. Carbon Brush |
| 4. 3 mm Allen Key | 8. Dielectric Bob | Adapter |



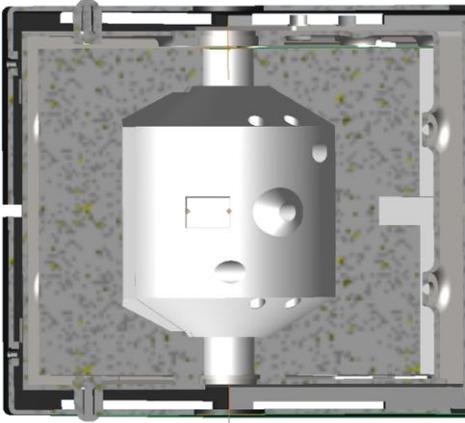
*Modified Oven
Cross-Section*



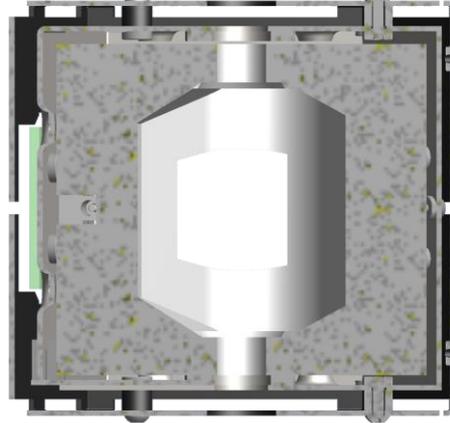
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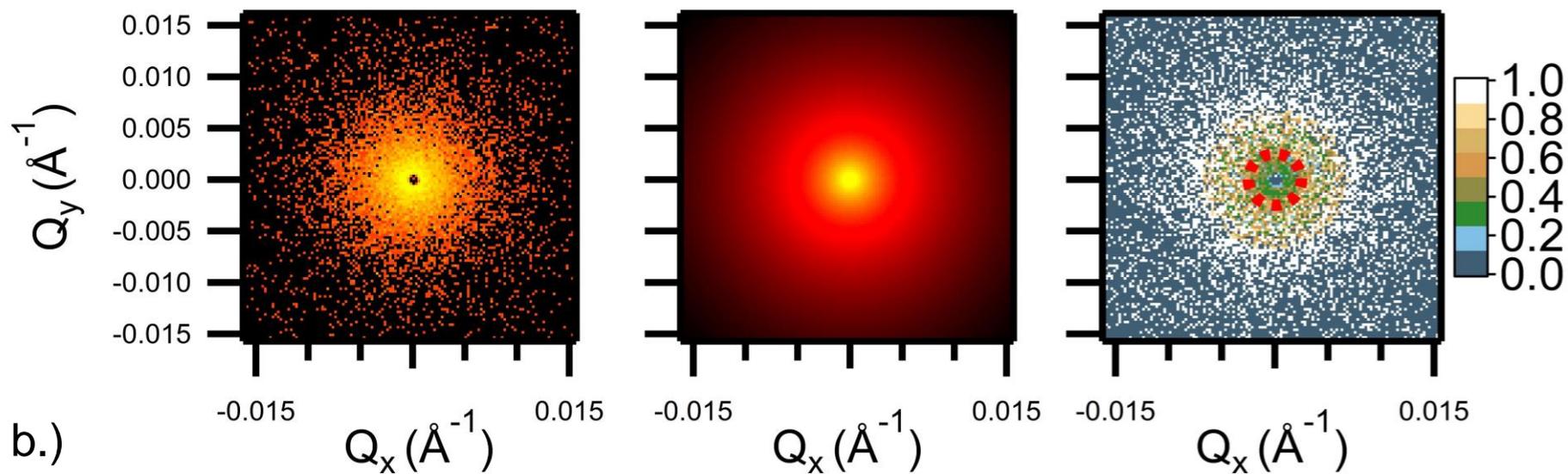
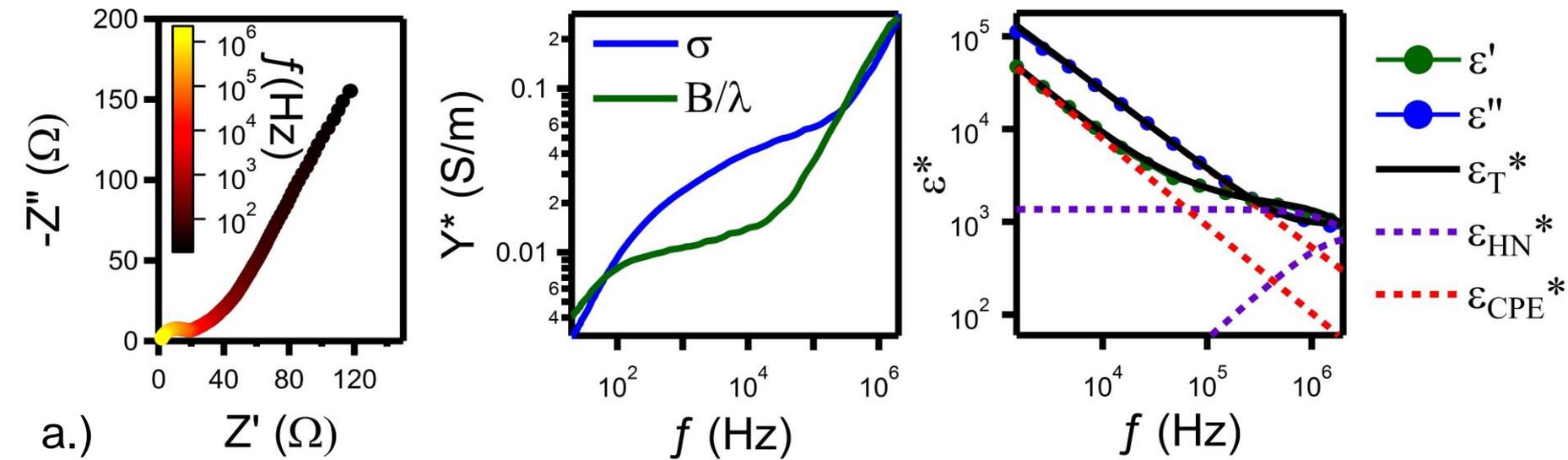


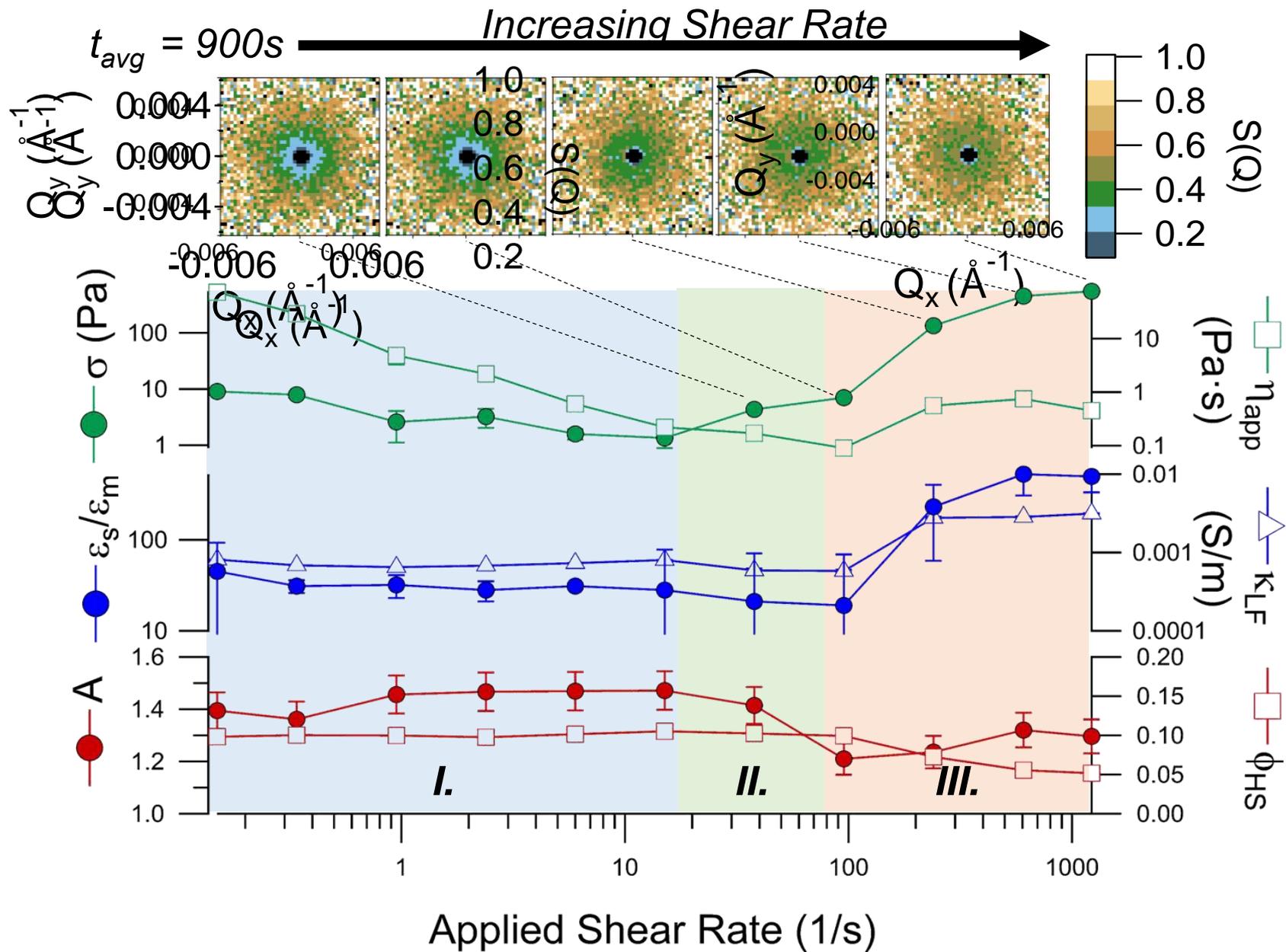
Beam Entry Cross-Section



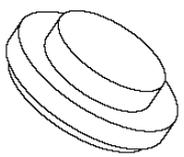
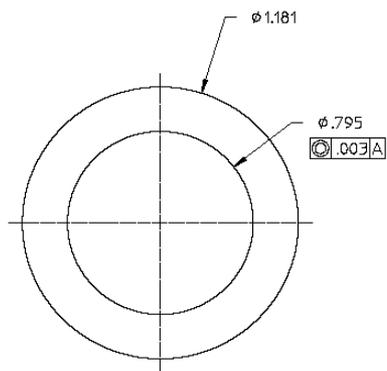
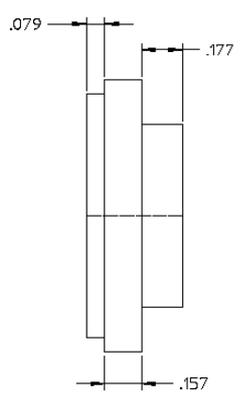
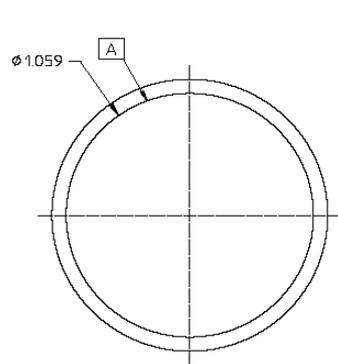
Beam Exit Cross-Section





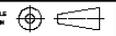


REVISIONS			
Nb.	ZONE	ECN	CHANGE
1			



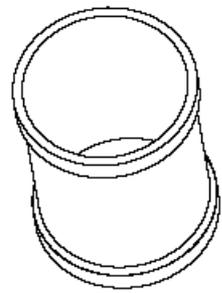
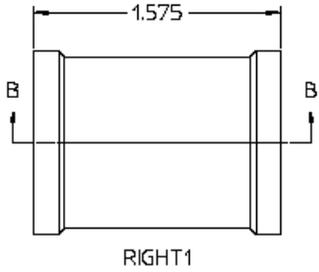
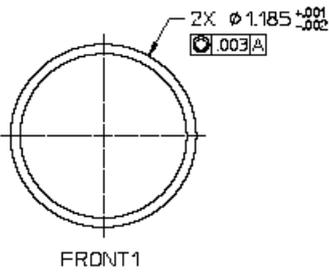
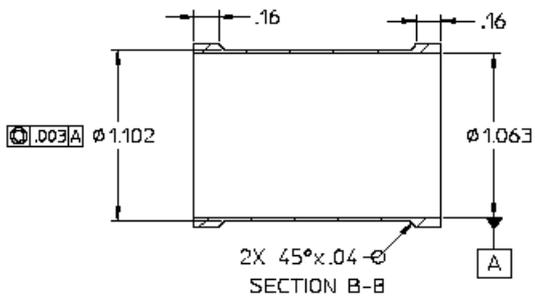
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MATERIAL PEEK	TEMPERATURE RT	DATE	DATE	DECIMALS XXX	FRACTIONS A/32	PEEK ADAPTER - ARES			
FORM B&D ROUND	SIZE	DATE	DATE	XXX X/16	XXX X/32	DRAWING DATA			
MODEL NAME ADAPTER		DATE 12/8/15 3:08 PM		DRAWING NUMBER 3010975-2D20		SCALE 3:1			
CREATOR GAGNEP	DATE 12/8/15 5:23 PM	APPROVED BY GAGNEP	DATE 12/8/15 3:08 PM	FABRICATOR Codic Gagnon (3010975-2D20)		RELEASE DATE		SHEET 1 of 1	
THIRD ANGLE PERSPECTIVE		DATE 12/8/15 3:26 PM		EMAIL ADDRESS codic.gagnon@nist.gov		CALC. BY		DIM. & TOL. PER ANSI Y14.5M-1982	

NOTES:



ns_bdr

REVISIONS			
No.	DATE	BY	CHANGE
2	08/2		ADDED SHOULDER AND CHAMFERS



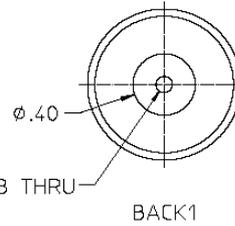
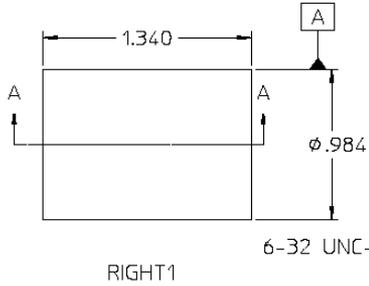
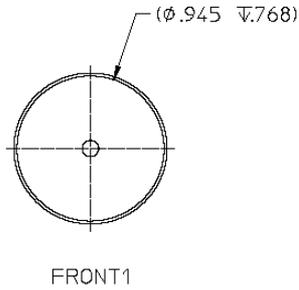
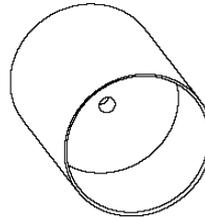
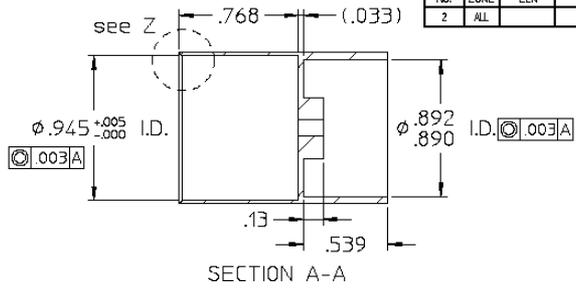
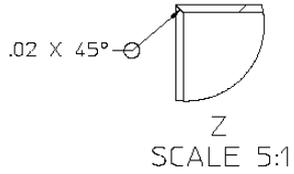
NOTES:
1-REMOVE ALL BLURS AND SHARP EDGES.

MATERIAL SPECIFICATION PART: 014-1934 QTY: 1 DATE: 08/2/10		APPROVALS DESIGNED BY: [Signature] CHECKED BY: [Signature] DATE: 08/2/10		UNCLASSIFIED PORTION CONTROLLED BY: [Signature] TELEPHONE NO.: [Number] ADDRESS: [Address] CITY: [City] STATE: [State] ZIP: [Zip]		CONTROL FOR BUSINESS PUBLICATION NATIONAL BUREAU OF STANDARDS and TECHNOLOGY U.S. DEPARTMENT OF COMMERCE 100 BUREAU BLVD. GAITHERSBURG, MD 20899	
PROCESS DATA MFG. PROC: [Process] DATE: [Date] QTY: [Quantity]		QUANTITY DATA QTY: [Quantity] DATE: [Date]		DATE DATE: [Date]		REV. NO. REV. NO.: [Revision]	
REVISIONS REV. NO. 1: [Description] REV. NO. 2: [Description]		DATE DATE: [Date]		BY BY: [Signature]		DATE DATE: [Date]	
DATE DATE: [Date]		BY BY: [Signature]		DATE DATE: [Date]		BY BY: [Signature]	
DATE DATE: [Date]		BY BY: [Signature]		DATE DATE: [Date]		BY BY: [Signature]	

NSI National Institute of Standards and Technology U.S. DEPARTMENT OF COMMERCE		CONTROL FOR BUSINESS PUBLICATION NATIONAL BUREAU OF STANDARDS and TECHNOLOGY U.S. DEPARTMENT OF COMMERCE	
CAN, CUP - APRES D14-1934			
REV. NO. REV. NO.: 014-1934		REV. NO. REV. NO.: 2	
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DATE DATE: 08/2/10		DATE DATE: 08/2/10	

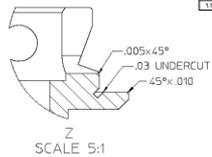
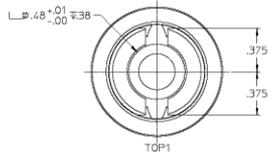
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REVISIONS				
No.	ZONE	ECN	CHANGE	DATE
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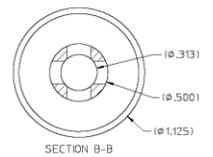
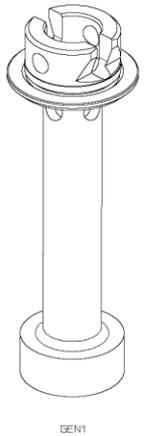
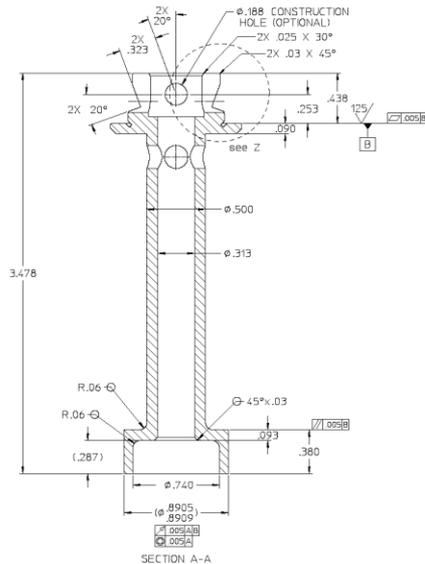
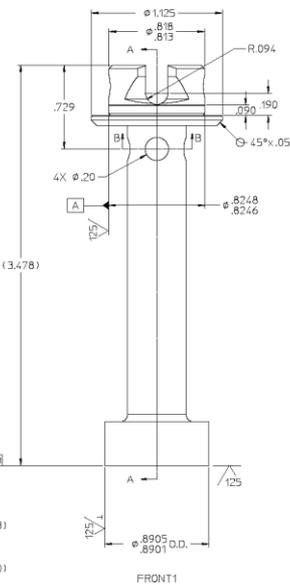


NOTES:
1-REMOVE ALL BURRS AND SHARP EDGES.

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MATERIAL: TITANIUM GRADE 2, R50400	DATE:	DATE:	DATE:	DECIMALS:	FRACTIONS:	NIST		TO BUREAU OF METROLOGY	
FORM: BAR, ROUND	DATE:	DATE:	DATE:	XXX, .XXX:	XXX/16:	National Institute of Standards and Technology		BETHESDA, MD 20815	
SIZE:	DATE:	DATE:	DATE:	XXX, .XXX:	XXX/16:	U.S. Department of Commerce			
ADDITIONAL DATA:		DRAWING DATA:		DO NOT SCALE DRAWING		CAN, BOB - ARES			
DESIGNER:	DATE:	DATE:	DATE:	PART NUMBER:		D14-1928			
CREATOR:	DATE:	DATE:	DATE:	PART NAME:		B DWG No. 014-1932			
DATE:	DATE:	DATE:	DATE:	PART NUMBER:		SCALE: 2:1 RELEASE DATE:			
DATE:	DATE:	DATE:	DATE:	PART NUMBER:		CALC. BY:			
DATE:	DATE:	DATE:	DATE:	PART NUMBER:		DIM. & TOL. PER ANSI Y14.5M-1992			
DATE:	DATE:	DATE:	DATE:	PART NUMBER:		SHEET 4 of 1			



REVISIONS	
No.	DATE
1	11

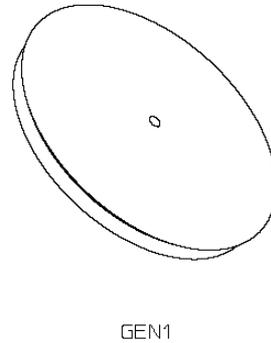
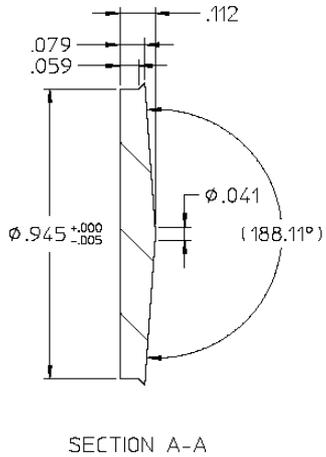
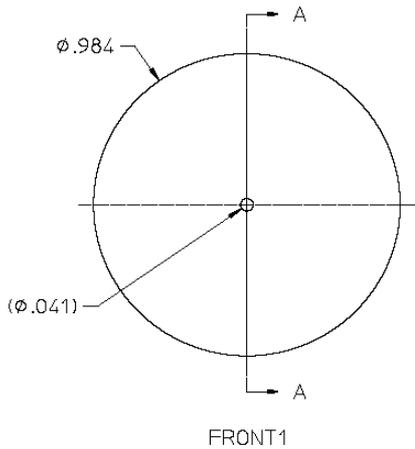


REVISIONS		REVISIONS	
No.	DATE	No.	DATE
1	11		

DATE	11/11/11	DESIGNER	Electric	PROJECT	014-1931
DRAWN	11/11/11	CHECKED	11/11/11	SCALE	1:1
APP'D		DATE	11/11/11	BY	

NOTE:
1 REMOVE ALL BURRS AND SHARP EDGES.

REVISIONS			
No.	ZONE	ECN	CHANGE
1			



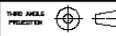
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SECTION A-A

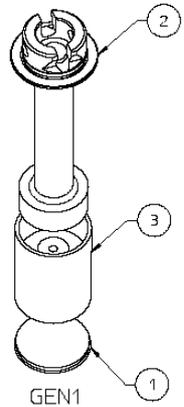
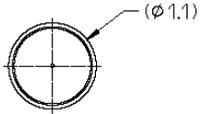
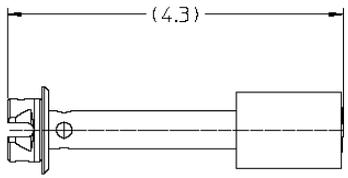
GEN1

NOTES:
1-REMOVE ALL BURRS AND SHARP EDGES.

MATERIAL SPECIFICATION MATERIAL: PEEK PART: BWR ROUND SIZE:		APPROVALS COR: DATE: ENR: DATE:		UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE: DECIMALS FRACTIONS .005 .010 .015 .020 .030 .040 .050 .060 .070 .080 .090 .100 .125 .150 .175 .200 .250 .300 .375 .450 .500 .625 .750 .875 1.000 ANGLES .5 .6 .7 .8 .9 1.0 1.5 2.0 3.0 4.0 5.0 6.0 8.0 10.0 15.0 20.0 25.0 30.0 45.0 60.0 90.0 DO NOT SCALE DRAWING		NIST National Institute of Standards and Technology U.S. Department of Commerce CENTER FOR NEUTRON RESEARCH 10 BUREAU DRIVE SALT SPRING, MD 20688	
MODEL DATA FILE NAME: PEEK_bwr.cad DATE: 8/3/15 12:21 PM CREATOR: GAGNE DWGID:		DRAWING DATA TITLE: CAP, BOB - ARES SHEETS: 1 DATE: 8/18/15 11:47 AM DWGFR: GAGNE DATE: 8/10/15 2:30 PM CDR: GAGNE		PART NUMBER: Cotic Gagnon (301975-2D20) EMAIL ADDRESS: cotic.gagnon@nist.gov		CAP, BOB - ARES D14-1928 DWG No. 014-1930 SCALE: 4:1 SHEET: 4 of 1 DIM. & TOL. PER ANSI Y14.5M-1992	



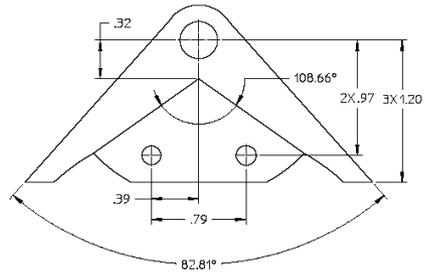
REVISIONS				
No.	ZONE	ECN	CHANGE	DATE
2				



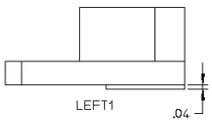
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1	D14-1931	N/A	OPEN MARKET	SHAFT, BOB - ARES	PEEK	2
1	D14-1930	N/A	OPEN MARKET	CAP, BOB - ARES	PEEK	1

MATERIAL SPECIFICATION		APPROVALS		UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES UNLESS NOTED OTHERWISE		NIST		CENTER FOR NEUTRON RESEARCH	
ASSEMBLY	DATE	DATE	DATE	DECIMALS	FRACTIONS	NIST		TO BUREAU DRIVE	
PER ASSEMBLY				XXX.XXXXX	XX/XX	National Institute of Standards and Technology		BETHESDA, MD 20815	
PER ASSEMBLY				XXX.XXX	XX/XX	U.S. Department of Commerce			
MATERIAL DATA		DRAWING DATA		DO NOT SCALE DRAWING		ARES DIELECTRIC BOB ASSEMBLY			
ITEM NAME	DATE	DATE	DATE			D14-1927			
CREATOR	DATE	DATE	DATE			REV B		DWN No. 014-1928	
DATE	DATE	DATE	DATE			SCALE 1:1		SHEET 4 of 1	
THIRD ANGLE PROJECTION		DRAWN BY		DATE		CALC. BY		DATE	
		DATE		DATE				DIM. & TOL. PER ANSI Y14.5M-1982	

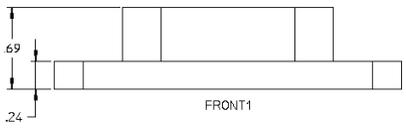
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NO.	ZONE	ECN	CHANGE	DATE
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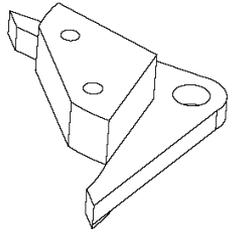
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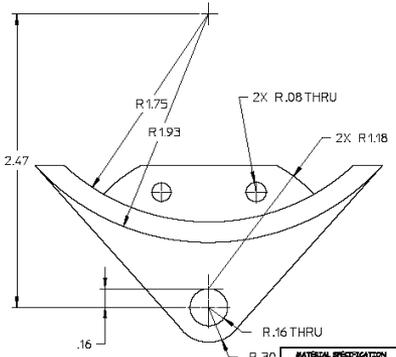
LEFT1



FRONT1



GEN1



BOTTOM1

NOTES:
1. REMOVE ALL BURRS AND SHARP EDGES.

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ACBIL DATA SPEC: 303 MAT: 303 SUP:		DEVIATION DATA SPEC: 303 MAT: 303 SUP:		DID NOT SCALE DRAWING		CARBON BRUSH ADAPTER - ARES			
THIS DRAWING IS THE PROPERTY OF NIST. IT IS TO BE KEPT IN THE DRAWING OFFICE AND NOT REPRODUCED OR DISTRIBUTED WITHOUT THE EXPRESS WRITTEN PERMISSION OF NIST.		DATE: 7/2/16 15:00 PM BY:		DATE: 7/2/16 15:00 PM BY:		DATE: 7/2/16 15:00 PM BY:		DATE: 7/2/16 15:00 PM BY:	
014-2144		1 of 1		1 of 1		1 of 1		1 of 1	