



Dispersion Technology Inc.

Characterization of Concentrated Dispersions and Emulsions, Liquids and Porous materials

Model DT-100 Acoustic spectrometer: Particle sizing in concentrates.

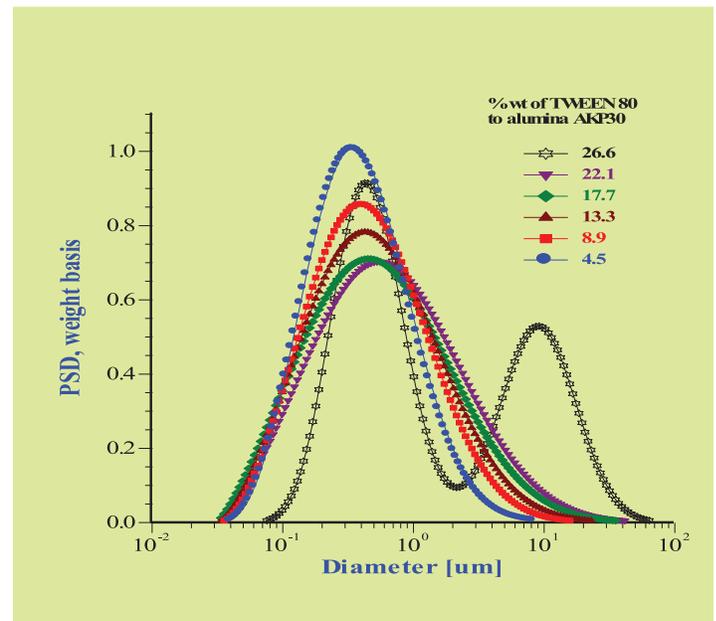
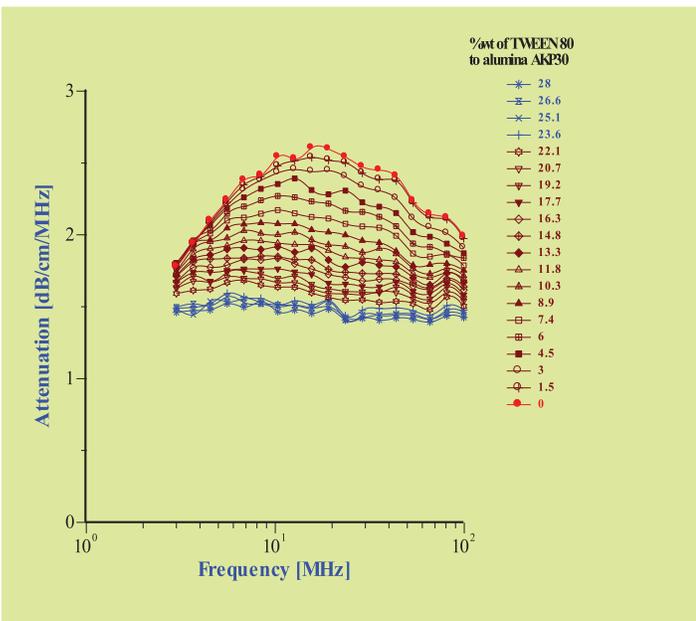


Model DT-100 has a unique Acoustic sensor for characterizing *particle size distribution* by measuring ultrasound attenuation at set of frequencies from 1 to 100 MHz, as well as sound speed. This method can be referred to as “*ultrasound scattering*”.

Application of ultrasound instead of light allows characterization of concentrated opaque sample as is, with no dilution or other sample preparation.

Calculation of particle size distribution from the measured attenuation spectra takes into account particle-particle interaction, which is imperative in concentrated systems.

The same ultrasonic raw data can be used for calculating compressibility, elastic modulus and longitudinal viscosity of any liquid sample, when Rheological option is installed.



Available Options:

Titration option with one or two burettes allows conducting complicated experiments involving modification of chemical composition of the liquid medium according to a certain protocol. There are several different types of protocols available: “pH ramp”, “pH stat”, “surfactant titration”, “temperature titration”. Titration “pH ramp” allows scanning of a certain pH range in single or multiple sweeps. Titration “pH stat” monitors amount of a particular reagent that is required for maintaining given pH. Surfactant titration reflects changes in particle size distribution with incrementally increasing surfactant concentration. It is used for determining optimum surfactant dose. Temperature titration requires installation of “heating control option”, which would allow performing T sweeps within a range from room T up to 50 C.

Conductivity aqueous option for measuring electric conductivity of aqueous systems within a range from 10^{-3} to 10 S/m. This probe functions at MHz range and,

consequently, is not affected by electrode polarization.

Conductivity non-aqueous option for measuring conductivity of various solvents including non-polar liquids within the range from 10^{-11} up to 10^{-4} S/m. This option is identical in function to the DT-700 model. This option requires installation of “non-aqueous option”, which is important for protecting instrument sensor from aggressive solvents if they are intended to be used.

Rheological option allows calculation of high frequency (MHz range) longitudinal rheological parameters such as compressibility, elastic modulus, viscosity, and performs test on Newtonian nature of the liquid sample.

External pump option is required when rather viscous samples are monitored continuously, which can serve as laboratory prototype for on-line characterization.

N o m i n a l S p e c i f i c a t i o n s :

Calculated parameters		Sample volume, minimum [ml]	
Mean size [microns]	0.005-1000	No sedimentation, no mixing	15
Weight fraction	±0.1%	Mixing with magnetic mixer	70
Compressibility E10[1/Pa]	±0.003	Mixing with peristaltic pump	150
Bulk viscosity [cP]	±0.01		
Measured parameters		Sample requirements	
Temperature [C 0]	0 to 100, ±0.1	Volume fraction, % (1)	0.1-50
pH	0.5-13.5, ±0.1	Conductivity	none
Frequency range [MHz]	1-100	pH	0.5-13
Ultrasound attenuation [dB/cm/MHz]	0 to 20, ±0.01	Temperature [C0]	<50
Sound speed [m/sec]	500 to 3000, ±0.1	Viscosity of media [cP] (2)	<20,000
Conductivity [S/m]	10^{-11} to 1, ±1%	Viscosity of sample [cP]	<20,000
Measurement time [min]	0.5-10	Particle size [microns]	0.005-1000

(1) Instrument can measure attenuation well above 50% vl, but verification of the theory for computing particle size is not possible above this limit.

(2) The “micro-viscosity” is important for theoretical calculation. It might be different than “macroscopic” viscosity for gels and other structured systems measured with conventional rheometers.

Physical Specifications. Electronic unit: weight 20 kG, sensor unit 30 kG. Power: 100-250 VAC, 50-60 Hz, <300 W. Software: embedded Windows HP, MS Office optional.

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Characterization of Concentrated Dispersions and Emulsions, Liquids and Porous materials

Zeta Potential Probe: Model DT-300 and Model DT-310 (titration included)

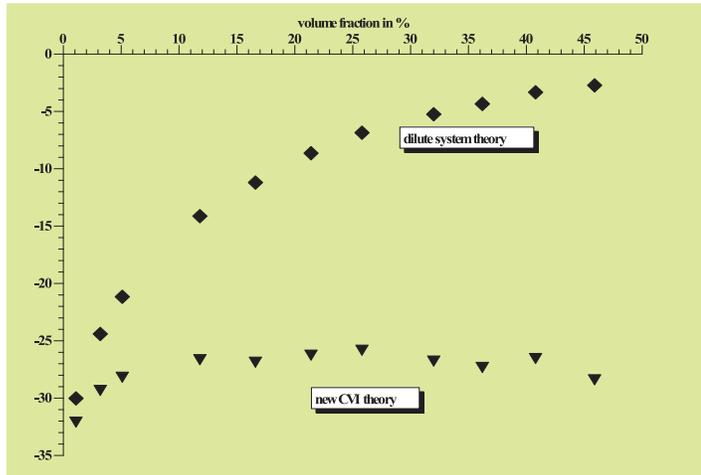


Models DT-300 and DT-310 have a unique **Electroacoustic sensor**, which is built as a probe (see on the right) for measuring ζ -potential in concentrates without dilution. The same probe can be used for monitoring sedimentation kinetics.

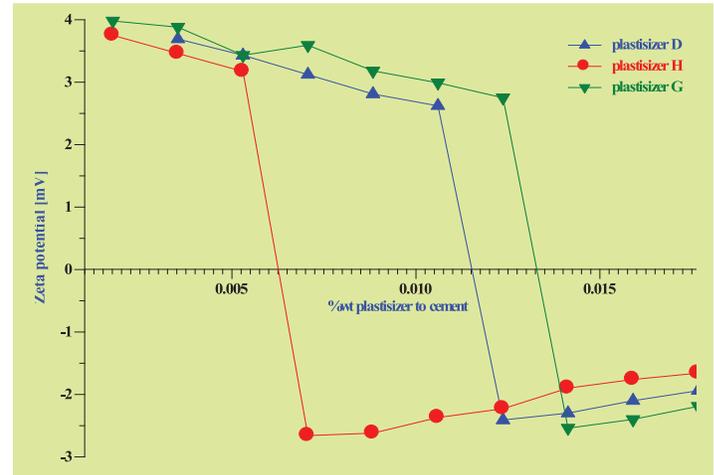
There is a piezo-crystal inside of the probe that generates sound pulse of certain frequency. These pulses propagate through the sample via the gold central electrode. Ultrasound moves particles relative to the liquid, which displaces Double Layers and generates an electric field. This field, in turn, changes the electric potential of the gold electrode. The electric potential of the steel cylinder remains zero because it is outside of the electric field. The Electronics measures AC current flowing between the gold and steel. This Colloid Vibration Current predicted by Debye in 1933 is proportional to electrophoretic mobility, which is in turn proportional to ζ -potential. DTI has verified theory that takes into account both particle-particles hydrodynamic and electrodynamic interactions when calculating ζ -potential from the measured electroacoustic signal.



Equilibrium dilution of rutile dispersion for electroacoustic theory verification in concentrates – ζ -potential must be independent of volume fraction if dilution maintains surface-bulk equilibrium.



Example of ζ -potential surfactant titration: three cement samples at 72% wt with incremental additions of three different superplasticizers.



Available Options:

Conductivity aqueous option for measuring electric conductivity of aqueous systems within a range from 10^{-3} to 10 S/m. This probe functions at MHz range and, consequently, is not affected by electrodes polarization.

Conductivity non-aqueous option for measuring conductivity of various solvents including non-polar liquids within the range from 10^{-11} up to 10^{-4} S/m. This option is identical in function to the

DT-700 model. This option requires installation of “non-aqueous option”, which is important for protecting instrument sensor from aggressive solvents if they are intended to be used.

External pump for when viscous samples are monitored continuously, which can serve as laboratory prototype for on-line characterization.

Heating control option for temperature titrations.

N o m i n a l S p e c i f i c a t i o n s :

Calculated parameters		Sample volume, minimum [ml]	
Zeta potential [mV]	$\pm(0.5\%+0.1)$	No mixing	0.1
Debye length [nm]	± 0.1	Mixing with magnetic mixer	20
		Mixing with peristaltic pump	100
Measured parameters		Sample requirements	
Electroacoustic signal [$mV(s/g)^{1/2}$]	$\pm 1\%$	Volume fraction, % (1)	0.1-50
Temperature [C 0]	0 to 100, ± 0.1	Conductivity	none
pH	0.5-13.5, ± 0.1	pH	0.5-13
Frequency range [MHz]	1-10	Temperature [C0]	<50
Conductivity [S/m]	10^{-11} to 1, $\pm 1\%$	Viscosity of media [cP] (2)	<20,000
Measurement time [min]	0.5-2	Viscosity of sample [cP]	<20,000
		Particle size [microns]	0.005-1000
		Zeta potential [mV]	none

- (1) Instrument can measure electroacoustic signal well above 50% vI for dispersions and ionic current for pure liquids. However, verification of the theory is possible only for specified range.
- (2) The “micro-viscosity” is important for theoretical calculation. It might be different than “macroscopic” viscosity for gels and other structured systems measured with conventional rheometers.

Physical Specifications. Electronic unit: weight 20 kG, Power 100-250 VAC, 50-60 Hz.
Software: embedded Windows HP, MS Office optional.

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Characterization of Concentrated Dispersions and Emulsions, Liquids and Porous materials

Model DT-1202 Acoustic and Electroacoustic spectrometer: Particle sizing and Zeta potential measurement in concentrates.



Wide Range of Applications:

- Nanotechnology
- Colloid stability
- Ceramic slurries
- Cement slurries
- Battery slurries
- CMP slurries
- Cosmetics
- Paints and pigments
- Non-aqueous systems
- Clays and minerals
- Food emulsions
- Mixed dispersions
- Structured systems
- Photo materials

Model DT-1200 has two unique sensors: Acoustic and Electroacoustic.

Acoustic sensor characterizes *particle size distribution* by measuring ultrasound attenuation at set of frequencies from 1 to 100 MHz and sound speed. The same ultrasonic raw data can be used for calculating compressibility, elastic modulus and longitudinal viscosity of any liquid sample (when “Rheological option” is installed).

Electroacoustic sensor is built as a probe for measuring ζ -potential in concentrates without dilution. The same probe can be used for monitoring sedimentation kinetics and for characterizing porous materials (when “Porous materials option” is installed).

These sensors can function either separately for individual measurements, or together, providing certain synergism in sample characterization.

Available Options:

Titration option with one or two burettes allows conducting of complicated experiments involving modification of chemical composition of the liquid medium according to a certain protocol. There are several different types of protocols available: “pH ramp”, “pH stat”, “surfactant titration”, “temperature titration”. Titration “pH ramp” allows scanning of a certain pH range in single or multiple sweeps and usually performed for determining iso-electric point. Titration “pH stat” monitors amount of a particular reagent that is required for maintaining given pH. Surfactant titration reflects changes in ζ -potential, particle size distribution, or both, with incrementally increasing surfactant concentration. It is used for determining optimum surfactant dose. Temperature titration requires installation of “heating control option”, which would allow performing T sweeps within a range from room T up to 50 C.

Conductivity aqueous option allows for measuring electric conductivity of aqueous systems within a range from 10^{-3} to 10 S/m. This probe functions at MHz range and, consequently, is not affected by electrode polarization. The same probe is used for measuring porosity of a porous material if Porous materials option is installed.

Conductivity non-aqueous option allows for measuring conductivity of various solvents including non-polar liquids within the range from 10^{-11} up to 10^{-4} S/m. This option is identical in function to the DT-700 model. This option requires installation of “non-aqueous media option”, which is important for protecting instrument sensor from aggressive solvents if they are intended to be used.

Rheological option allows calculation of high frequency (MHz range) longitudinal rheological parameters such as compressibility, elastic modulus, viscosity, and performs test on Newtonian nature of the liquid sample.

Porous materials option allows characterization of porosity using the aqueous conductivity probe, as well as pore size and zeta potential of a porous material with electroacoustic probe. Characterization of these last two parameters would require calibration.

External pump option is required when viscous samples are monitored continuously, which can serve as a laboratory prototype for on-line characterization.

N o m i n a l S p e c i f i c a t i o n s :

Calculated parameters		Sample volume, minimum [ml]	
Mean size [microns]	0.005-1000	Size only	15
Zeta potential [mV]	$\pm(0.5\%+0.1)$	Zeta only	0.1
Weight fraction / porosity	$\pm 0.1\%$	Both, no sedimentation	15 +0.1
Compressibility E10 [1/Pa]	± 0.003	Both with mixing	70
Bulk viscosity [cP]	± 0.01	Both with titration	110
Debye length [nm]	± 0.1	Both with pumping	150
Measured parameters		Sample requirements	
Temperature [C 0]	0 to 100, ± 0.1	Volume fraction, % (1)	0.1-50
pH	0.5-13.5, ± 0.1	Conductivity	none
Frequency range [MHz]	1-100	pH	0.5-13
Ultrasound attenuation [dB/cm/MHz]	0 to 20, ± 0.01	Temperature [C 0]	<50
Sound speed [m/sec]	500 to 3000, ± 0.1	Viscosity of media [cP] (2)	<20,000
Electroacoustic signal [mV(s/g) ^{1/2}]	$\pm 1\%$	Viscosity of sample [cP]	<20,000
Conductivity [S/m]	10^{-11} to 1, $\pm 1\%$	Particle size [microns]	0.005-1000
Measurement time [min]	0.5-10	Zeta potential [mV]	none

(1) Instrument can measure attenuation well above 50% v1, but verification of the theory for computing particle size and zeta potential is not possible above this limit.

(2) The “micro-viscosity” is important for theoretical calculation. It might be different than “macroscopic” viscosity for gels and other structured systems measured with conventional rheometers.

Physical Specifications. Electronic unit: weight 20 kG, sensor unit 30 kG. Power: 100-250 VAC, 50-60 Hz, <300 W. Software: embedded Windows HP, MS Office optional

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Characterization of Concentrated Dispersions and Emulsions, Liquids and Porous materials

Model DT- 700: Non-aqueous Conductivity Probe.



Model DT-700 is a stand alone instrument for measuring electric conductivity of low polar (alcohols) down to completely non-polar liquids (toluene, hexane, etc) within the range of 7 orders of magnitude. This measurement can be used for studying ionic composition of such liquids, which affects many properties of the liquids, emulsions and dispersions. Consequently, this instrument is suitable for the quality control of these properties.

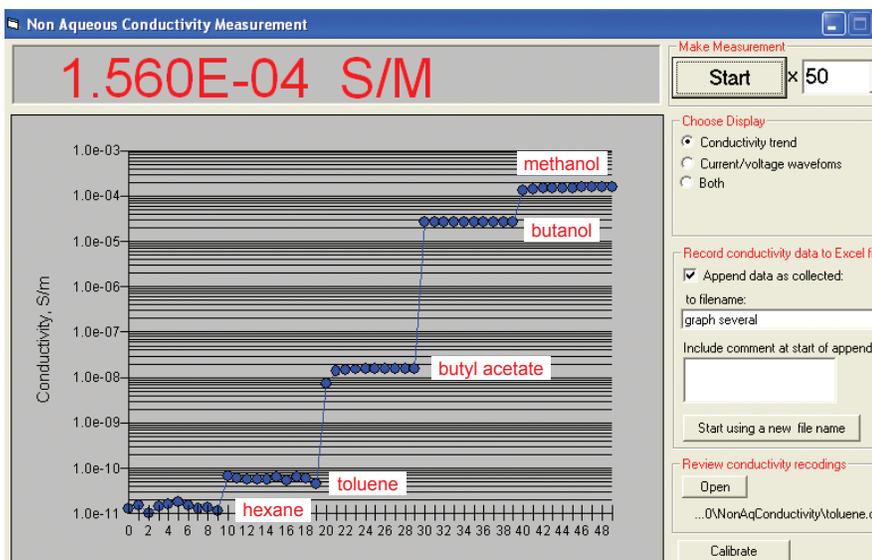
This instrument design eliminates several problems existing in previous instruments of the same purpose.

First of all it works within the full range of the conductivity without using different sub-ranges as previous designs.

Secondly, conductivity probe by itself can be easily taken apart and then re-assembled, which simplifies cleaning.

Thirdly, user has ability to calibrate probe by himself using measurement of toluene, liquid with well defined dielectric permittivity.

Finally, Windows based software allows displaying results of multiple measurements, which is important for tracking time dependences. All results can be saved to Excel files.



Principles of Operation:

The measuring cell consists of an outer and inner coaxial cylindrical electrode, as well as a guard electrode to eliminate leakage paths between the two. The outer electrode can be unscrewed from the body of the probe to facilitate cleaning of messy samples as shown in the figure below.

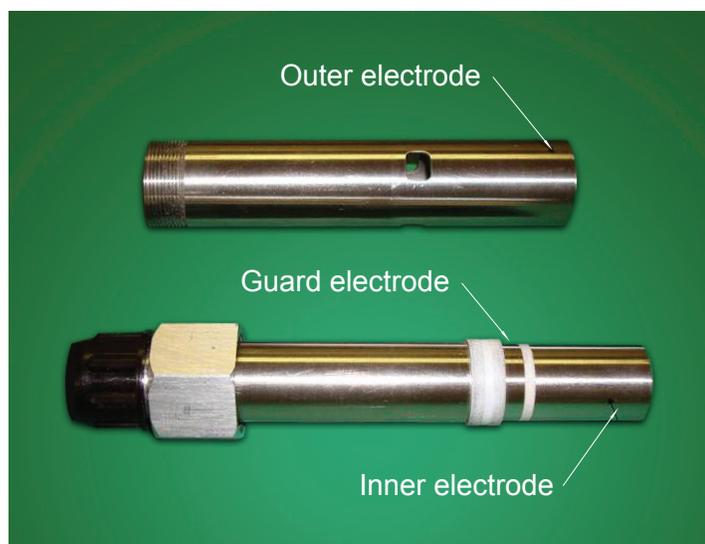
During a measurement the instrument applies a sinusoidal voltage to the outer electrode and measures the current that flows through the sample to the inner electrode. The frequency of this applied voltage is changed depending on the measured conductivity. The lowest conductivity samples are measured at 1 Hz, the highest ones at 10 Hz. The required sine wave voltage values are computed vs. time and converted to an actual voltage by a Digital to Analog converter. This voltage, after suitable filtering, is then connected to the outer electrode, simultaneously measured with an Analog to Digital converter, and optionally displayed in the application window on the Display.

The current is measured with a log amplifier, so no “range selection” is required by the user as was common in older designs. At the same time that the voltage waveform is being applied to the outer electrode, the output signal from the log amp is captured by a second channel of the Analog to digital converter. The desired

current waveform is calculated from the captured logamp values by performing an inverse log calculation. This log/ inverse log technique allows measurements to be made over many decades of current without the need for any range selection switches. The amplitude and phase of both the voltage and current waveforms is then computed from the first order Fourier coefficients of the respective waveforms. The Complex Conductance of the cell contents is then computed from the current voltage ratio. Finally the specific conductivity of the sample material is determined by computing the real part of the complex conductance and then multiplying by a cell constant that is determined by the geometry of the cell.

Cell constant is determined with calibration that employs toluene as a liquid with well known dielectric permittivity 2.38. It is assumed that cell constant for dielectric permittivity measurement of toluene is identical to the cell constant that is required for correcting for geometry of electric field lines during conductivity measurement.

Results of multiple measurements for several liquids are shown on front page Figure illustrating DT-700 software screen.



Nominal Specifications:

Range available for conductivity measurement is 7 orders of magnitude: from 10E-11 to 10E-4 S/m.

Precision equals to $\pm (1\% + 10E-11 \text{ S/m})$ over the complete range.

Physical Specifications.

Weight: electronic unit 5 kg, Power: 100-250 VAC, 50-60 Hz, <300 W. Software: embedded Windows XP.

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