

QUALITY CONTROL AND ANALYTICAL CAPABILITIES

GELEST'S QUALITY POLICY

- Gelest is dedicated to providing quality specialty silanes, silicones, and metal-organic products and addresses the quality needs by market and by customer. The quality requirements are addressed by the analytical team with full support of management. Each employee understands the necessity and responsibility of quality. The process of quality is considered critical for the growth of both the customer and Gelest. The Gelest quality management system is compliant with ISO9001:2008 standard.

QUALITY CONTROL INSTRUMENTATION



GC-TCD


- Six GC-TCD instruments from Agilent Technologies
 - Dual Auto Injection-HP6890N
 - Auto Injection- HP6850 and HP6890
 - (3) Dual Auto Injection- HP7890
- Each instrument dedicated to a specific family of compounds
 - Preventing cross-contamination and reactions of certain compounds on the columns
- **Primary Use**
 - Determination of the purity of volatile silanes and metal organic compounds

GC-TCD

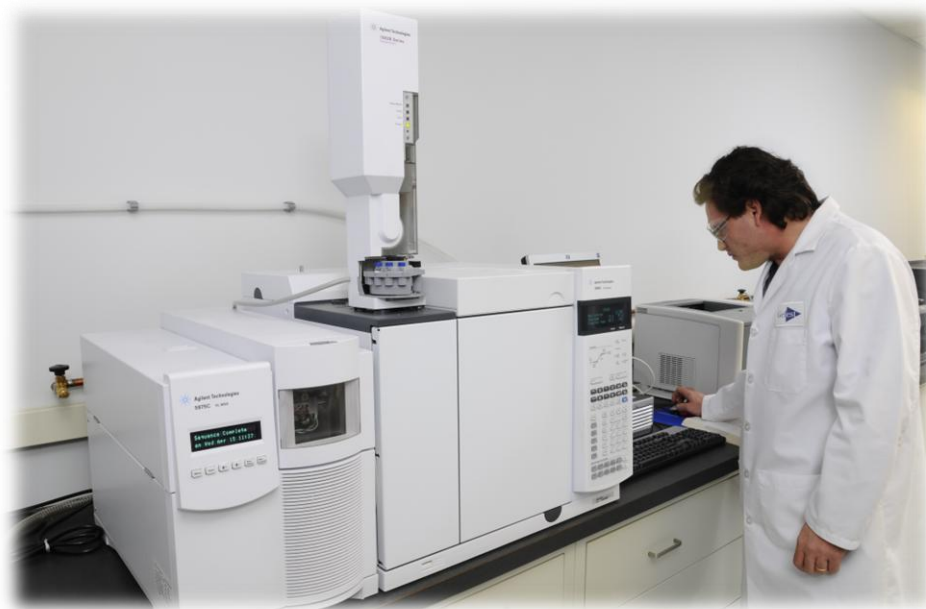
- Array of methods and columns is used to optimize separation and quantification
- Thermoconductivity Detector
 - Used for all purity determinations as it is the most compatible with the types of compounds analyzed
 - Produces no limiting problems
 - Combustion of organosilicon compounds builds up silica on the flame ionization detector (FID), decreasing the sensitivity of the instrument



EXAMPLE OF A GC-TCD METHOD

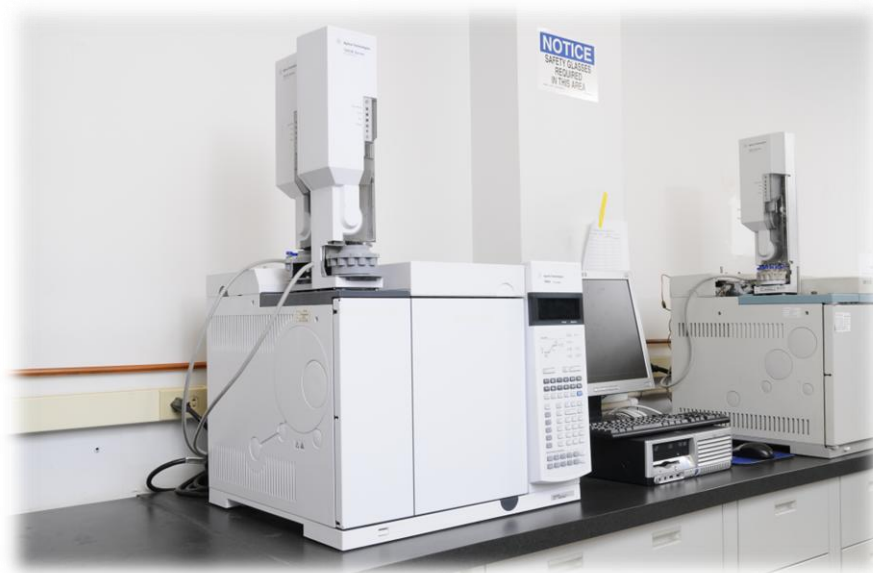
	Gelest Method#1025 HP6850 Standard Method	Gelest Test Methods
		Rev. 0
		Released Date: 06/22/2006
<p>Detector: TCD (thermal conductivity) 350°C</p> <p>Gas type: Helium</p> <p>Column: Capillary d=0.32 mm, L=30 m</p> <p>Additional GC information: <i>PEAKS BELOW 0.014% ARE EXCLUDED FROM INTEGRATION.</i> Injection Source: GC Injector Injection Location: Back</p> <p>OVEN Initial temp: 75°C Maximum temp: 325°C Initial time: 2.00 min Equilibration time: 1.00 min Ramps: # Rate Final temp Final time 1 30.00 290 11.00 2 0.0(Off) Post temp: 0°C Post time: 0.00 min Run time: 20.17 min</p> <p>INLET (SPLIT/SPLITLESS) Mode: Split Initial temp: 290°C (On) Pressure: 9.70 psi (On) Split ratio: 100:1 Split flow: 181.3 mL/min Total flow: 186.2 mL/min Gas saver: Off Gas type: Helium</p>		

ELECTRON IONIZATION GC-MSD



- **GC-MSD from Agilent Technologies**
 - GC- 7890A
 - MSD- G3170A
 - Libraries
 - NIST- 191,000 spectra and 163,000 structures
 - Gelest-constructed of compounds sold by Gelest
- **Primary Uses**
 - Structure elucidation and identification of volatile silanes and metal organics
 - Problem solving tool used in characterization and identification of unknown peaks
 - Compound confirmation via Gelest library search
 - Development of customer specific methods

GAS ANALYSIS: GC-TCD



- **Gas Analysis GC-TCD from Agilent Technologies**
 - GC-HP6890
 - Customized gas sampling manifold-allows gas to be injected without contamination
 - Able to accurately test to 99.999% purity level
 - Cryogenic Chamber: Allows analysis to start at -80°C
- **Dedicated Use**
 - In-house analysis of specialty, high-purity gases

GEL PERMEATION CHROMATOGRAPHY (GPC)



- **Model number: TDA Model 301**

- Column: Mixed bed (single column)
- Mobile phase: THF
- 4 detector system
 - UV
 - Refractive Index
 - Viscosity
 - Light Scattering

- **Model number: Waters 2695 Separations**

- Columns: 2 mixed bed, 1 single pore (in series)
- Mobile phase: THF
- 2 detector system
 - UV
 - Refractive Index

- **Primary Use**

- Identification of polymer molecular weight and distribution



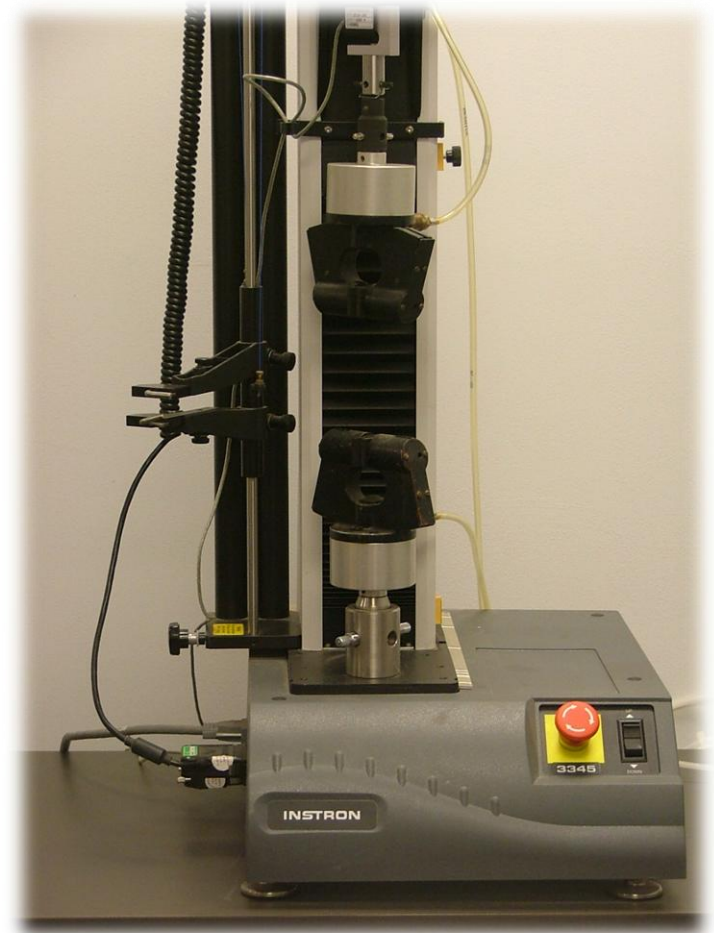
RHEOMETER

- TA Instruments-Model number AR2000
 - 25 mm parallel plates
 - Operated from ambient to 390°C
- **Primary Uses**
 - Measuring the stress and strain on fluids and elastomers via parallel plate geometry
 - Performing cure tests on platinum catalysts and formulated products



INSTRON AND TEXTURE ANALYZER

- **Instron**
 - Purchased from Instron:
 - Model number 3345
 - 5kN capacity
 - 1kN loading cell
 - **Primary Use**
 - Performing tensile compression tests on elastomers
- **Texture Analyzer**
 - TA Instruments
 - Model XT plus Texture Technology
 - Bench top version of the Instron
 - **Primary Use**
 - Performing tensile tests on gels and elastomers



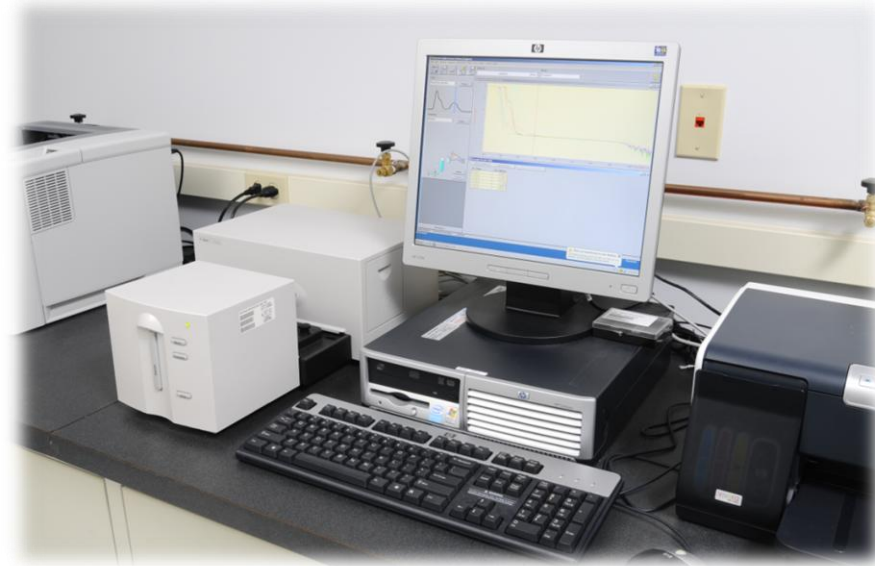
Instron

NUCLEAR MAGNETIC RESONANCE (NMR)

- 400MHz JEOL NMR
 - Various settings allow for multiple analyses:
 - ^1H
 - ^{13}C
 - ^{29}Si
- **Typical Uses**
 - Purity evaluation of non-volatile, air sensitive and solid compounds
 - R&D tool used in structure elucidation, compound characterization, and impurity determination
 - Customer specific requests



UV-VISIBLE SPECTROSCOPY



- **UV-Vis from Agilent Technologies**
 - Model number: 8453
 - Deuterium and Tungsten lamps: 190nm-1100nm
 - Can be used for quantification and/or qualification
- **Primary Uses**
 - Determination of the wavelength(s) of absorbance of UV-active silanes and treated inorganic substrates
 - Pre-HPLC analysis to determine absorbance of compounds
 - Allowing the HPLC's UV detector to be set at an optimal wavelength

INFRARED SPECTROSCOPY (IR)

- **Model Number: Nicolet iS10**
- **Primary Uses**
 - Qualitative
 - Characterization of compounds via their IR fingerprint
 - Problem-solving tool used to determine the presence of certain functional groups
 - Quantitative
 - Determining the percentage of various functional groups in silicones



INFRARED SPECTROSCOPY

- Various sampling techniques & methods of analysis

- NaCl plates
 - Solution cell
 - Diamond ATR
- Liquid Samples**

- KBr pellet
 - Diffused Reflectance (Accessory)
 - Diamond ATR
- Solid Samples**

- Gas Cells
- Gas Samples**

DIFFUSED REFLECTANCE

- **Theory**

- Spectrophotometer beam is focused into sample, where it's reflected, scattered, and transmitted.
- The part of the beam which is scattered within the sample and returns to the surface as reflection is collected as diffuse reflectance.

- **Sample Prep**

- Particulates, powders, and crystalline material mixed with an IR transparent salt (KBr)

ATTENUATED TOTAL REFLECTANCE (ATR)

- **Attenuated Total Reflectance (ATR)**
 - **Theory**
 - Solid sample (or liquid) is pressed in contact with an optical crystal.
 - IR radiation enters the crystal and reflects through the crystal.
 - With each reflection, the evanescent wave penetrates the sample a finite amount.
 - At the end of the crystal, the beam is directed to the detector.
 - **Sample Prep**
 - Sample (solid or liquid) placed on the crystal (diamond).
 - If solid, the crank is lowered to provide sufficient contact between the solid and the crystal (diamond).

INFRARED SPECTROSCOPY (IR)- RESEARCH

- **Nicolet iS50 ABX w/ Automated Beam Splitter Exchanger**
 - Beam splitters
 - CaF₂ (Near IR)
 - KBr (Mid IR)
 - Solid Substrate (Far IR)
 - Sources
 - Polaris
 - White Light
 - Detectors
 - DLaTGS with KBr window (Near and Mid IR)
 - DLaTGS with PE window (Far IR)
 - MCT-A (Near and Mid IR)
 - InGaAs (Near IR)
 - DLaTGS with proprietary window (Mid and Far IR)



INFRARED SPECTROSCOPY (IR)- RESEARCH

○ **Accessories**

○ Smart SAGA (Smart Aperture Grazing Angle)

○ Use: Examines thin films on reflective substrates; provides sub- μm layer detection

○ Praying Mantis

○ Use: Couples with DRIFT accessory for applying programmable heating/cooling to the sample pre/during/post analysis; monitors sample changes as a function of temperature

○ PAC 300 (Photoacoustic)

○ Use: Measures acoustic (pressure) waves as a light source is pulsed on solid samples; controllable depth penetration allows for selective surface layer probing on multi-layer films

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)



- Agilent Technologies-1200 Series
 - Quaternary pump (400 bar limit)
 - Auto sampler (100 vial tray)
 - Temperature controlled column compartment
 - Detectors
 - Variable Wavelength
 - Refractive Index
 - Capability to do both reverse and normal phase LC

- Agilent Technologies-1260 Infinity Series
 - Quaternary pump (600 bar limit)
 - Auto sampler (100 vial tray)
 - Temperature controlled column compartment
 - Detectors
 - Diode Array
 - Capability to do both reverse and normal phase LC

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

- **Primary Uses**
 - Purity determination of various silanes and silicones
 - Determination of the inhibitor content (BHT, PTZ, and MeHQ)
 - Determination of capping efficiency, degree of polymerization (Dp), and MW for various silicones
 - Customer specific methods
 - Method development tool for purity of non-volatile compounds

VARIOUS BENCH TOP INSTRUMENTS

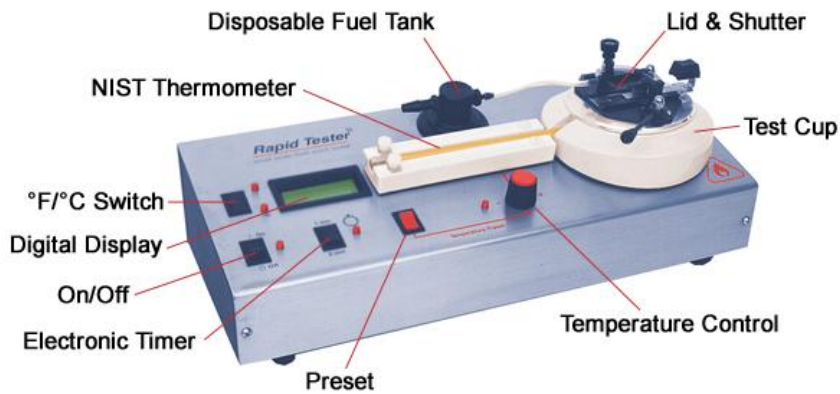
- **Karl Fischer-Water Titrator**
 - Mettler Toledo V20
- **Autotitrator (Potentiometric)**
 - Metrohm 916 Ti Touch
- **Viscometers**
 - Brookfield DV-I+
- **APHA and Gardner Color Standard Apparatus**
 - Orbeco-Hellige Aqua Tester (APHA)
 - Orbeco-Hellige Comparator Illuminator (Gardner)
- **Turbidimeter**
 - LaMotte 2020
- **Melting Point Apparatus**
 - Barnstead Electrothermal IA9300
- **Refractometer**
 - American Optical ABBE
- **pH Meter**
 - Mettler Toledo SevenEasy



VARIOUS BENCH TOP INSTRUMENTS

- **Flashpoint Apparatus**

- **Rapid Flashpoint**



- **Closed Cup Flashpoint**



WET CHEMISTRY TECHNIQUES

- **Titration to determine concentrations of:**
 - Acids/Bases
 - Grignard reagents
 - Hydrolyzable chloride
 - Vinyl in a silicone fluid
 - Hydroxyl Number
 - Amines
 - Epoxides
 - Quaternary ammonium chlorides
- **Specific Gravity**
 - Silanes @20°C
 - Silicones @25°C
- **Cure Tests**
 - Silicones and vinyl fluids
- **Solids/Volatile Testing**
 - Oven and Rotary Evaporator
- **Freeze and Boiling Points Analysis**
- **Cloud and Solubility Testing**



OUTSIDE ANALYTICAL

Accredited outside laboratories are used for those analyses that are not possible within Gelest.

- Outside testing includes:
 - ICP: Specific metals
 - Sn, Fe, Pt, Ti, Ge, etc
 - ICP: 60 trace metal scan
 - CHN Analysis
 - ICP-MS: Specialty metals testing of high-purity gases
 - Microbial Analysis on cosmetic products
 - Other tests as required by the customer

THERMAL GRAVIMETRIC ANALYSIS (TGA)

- **TA Instruments: Q50**
 - Analysis Options
 - Temperature Ramp
 - Temperature Ramp with Gas Switch
 - Heat and Hold
 - Stepwise Isothermal
 - Temperature limit of 1000°C
 - Analysis Gas Options: Air and N₂
- **Typical Uses**
 - LOD/LOI Analysis on treated micro-powders
 - Primary Metal Analysis on various organometallics
 - Volatile Analysis
 - Decomposition Analysis



DIFFERENTIAL SCANNING CALORIMETER (DSC)

○ **TA Instruments: Q20**

- DSC is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and a reference is measured as a function of temperature
- Analysis Options:
 - Temperature Ramp
 - Isothermal
 - Cycle
- Temperature Range: -40°C to 400°C

○ **Typical Uses**

- Physical Polymer Characterization:
 - Glass Transition
 - Melting Point
 - Crystallization Temperature
- Chemical Process Characterization
 - Curing Temperature



GC-FID

GC-FID

- Agilent Technologies
 - Auto Injection- HP7890
- Flame Ionization Detector (FID): The effluent from the GC column passes through the flame, which breaks down organic molecules and produces ions. The ions are then collected via a biased electrode and produce an electronic signal.
- Pro:
 - FID are much more sensitive than their TCD counterparts
- Con:
 - Constant analysis of silanes will cause the formation of sand in the FID jet (maintenance required and down time of the instrument)
- **Primary Uses**
 - Customer Specific Method Development
 - Customer Issues and Complaints

PARTICLE SIZE ANALYSIS

Microtrac: S3500

- Laser Diffraction Particle Size Analysis
- Measuring Range: 0.02-2800 microns
- Wet measurements only
- Tri-laser (red), multi-detector, multi-angle optical system
- Proprietary Modified Mie calculations allow users to measure complex particles

○ **Primary Uses**

- Particle Size analysis on various treated micro-particles, cosmetic pigments, silica, and silicon nanoparticles



ZETA POTENTIAL

Brookhaven Instruments: ZetaPlus

- Zeta Potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle
- Significance of zeta potential measurement is that the value can be related to the stability of a colloidal solution
 - High Zeta Potential: Stable Solution (electrically stable)
 - Low Zeta Potential: Attraction exceeds repulsion and the dispersion will flocculate
- **Primary Uses**
 - Surface Characterization of charge potential on various treated micro-powders
 - R&D and Tech Service Tool



COLORIMETER

SpectroColorimeter

- Lovibond
 - Model: PFXi-195/1

- **Theory/Application**
 - Comprised of a standardized light source (tungsten halogen) and collimator, sample chamber, light detector, monochromator and processor board
 - Designed for objective color measurement of clear liquids or transparent solids
 - Provides color data according to a full range of established industry scales (e.g. APHA, Gardner, Iodine, and CIE)
 - Ability to compare samples to stored references or to build up a customized scale from a series of reference samples

- **Primary Uses**
 - Objective and reproducible color measurements:
 - APHA
 - Gardner
 - Iodine number

