Particle Characterization of Pigments

Mark Bumiller
Mark.bumiller@horiba.com

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Outline

- Why is particle size important?
- Organic pigments using laser diffraction
- Inorganic pigments using laser diffraction
- Ink pigments
- TiO_2 study using laser diffraction
- TiO_2 particle size and zeta potential using dynamic light scattering
- Conclusions

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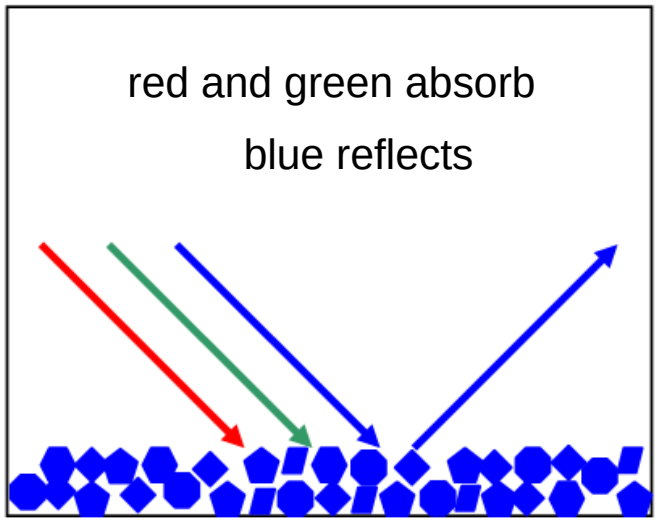
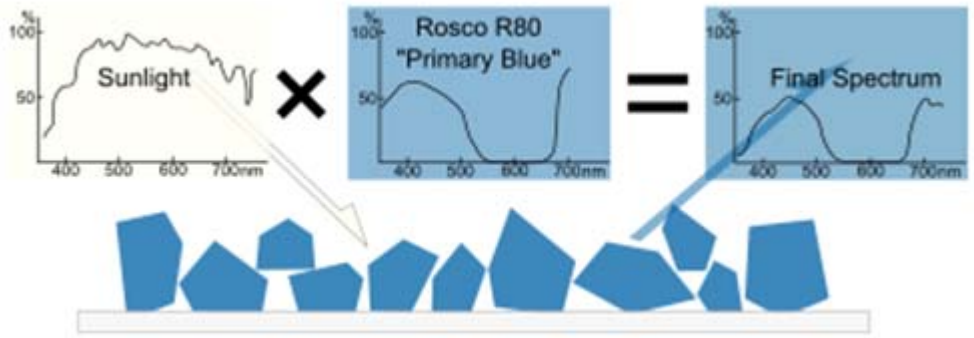
Pigments

A pigment is a material that changes the color of reflected or transmitted light as the result of wavelength-selective absorption





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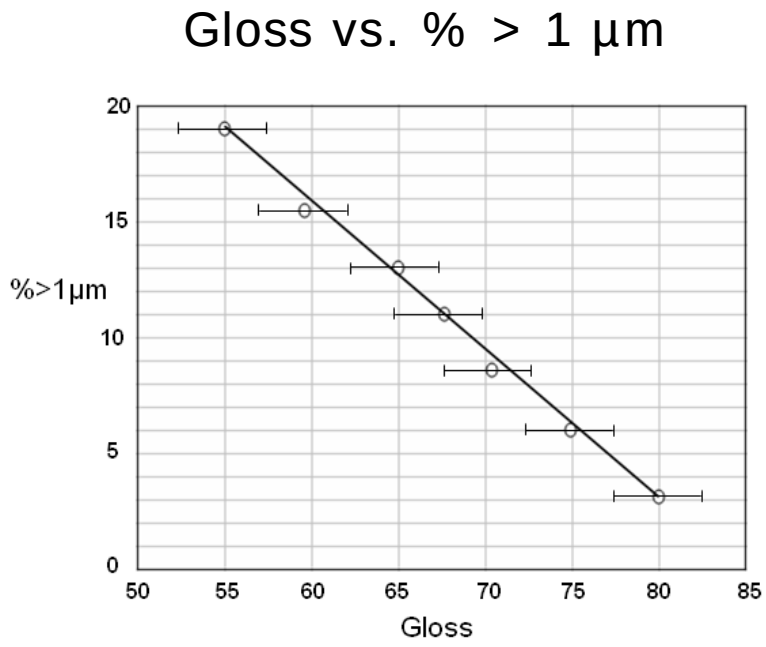
Properties Dependent on Particle Size

- Hue/Tint Strength
- Hiding/Transparency
- Gloss/Flatting and Film Appearance
- Flocculation
- Viscosity
- Stability
- Weather resistance



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Organic Pigments

- Commonly powder form
- Dry milling procedures monitored by dry laser diffraction method
- Particle size effects dispersibility of pigment in application media
- Wet laser diffraction method to monitor dispersion of organic pigments in low viscosity liquid media

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Organic Pigments Study

Samples*:

- 2583 Diarylide Yellow 83
- 1274 Hansa Yellow 74
- 2574 Hansa Yellow 74
- 5576 Phthalo Blue 15:3

1. Select the small, high-dispersion nozzle for the fine particle sizes expected.
2. Set the automatic measurement conditions to proper testing range (96-94%T for the laser).
3. Set dispersion air pressure to Low.
4. To prevent nozzle clogging, remove large agglomerates from sample using 1mm sieve
5. Load enough filtered sample onto feeder chute to perform multiple measurements.
6. Take 3 consecutive measurements using the Auto Measurement function.

*Thank-you to Lansco Colors, Warwick, RI for supplying the samples for this study. www.pigments.com

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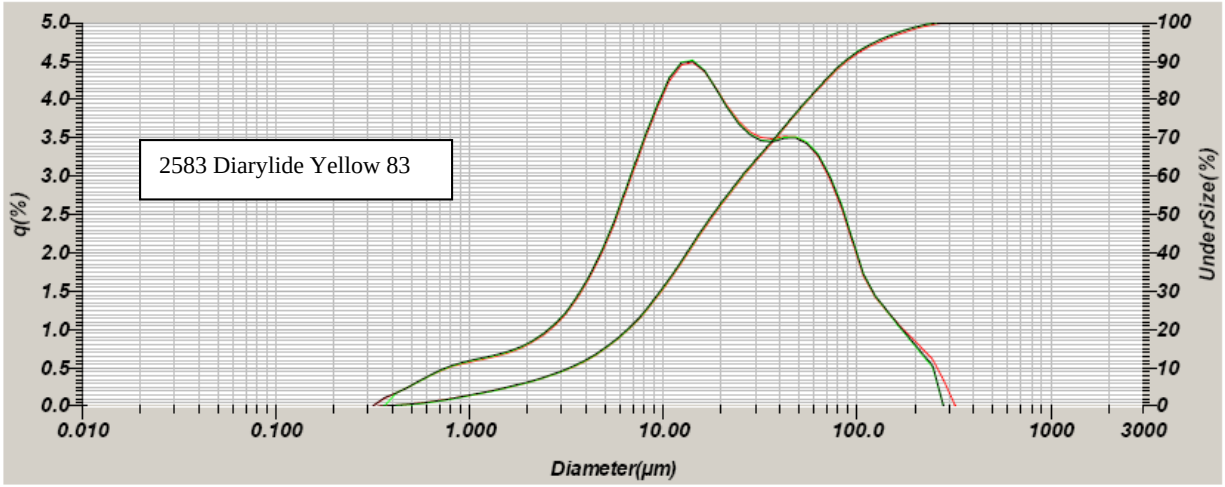
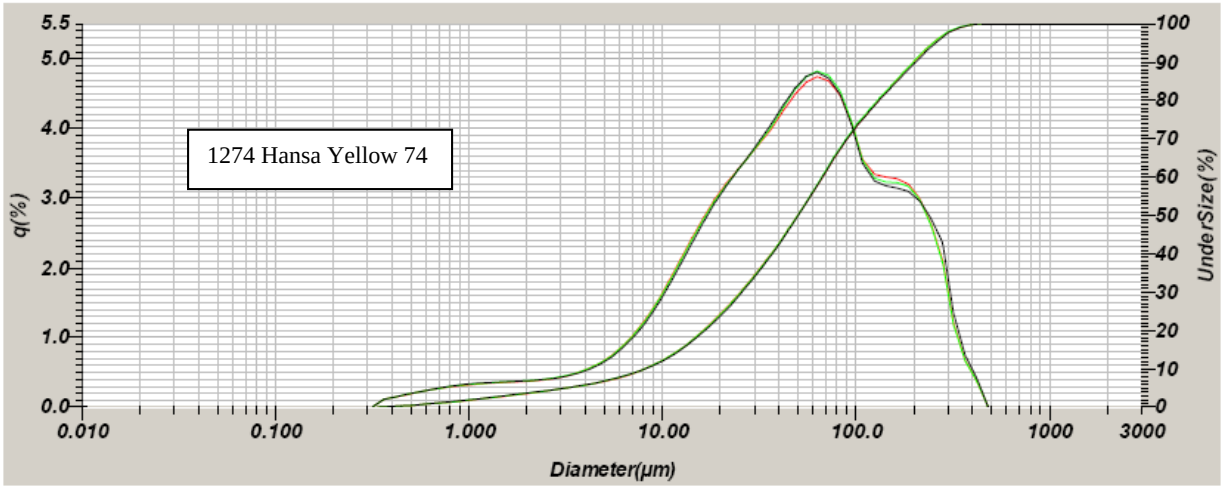
Dry Organic Pigment Results

2583 Diarylide	Yellow 83
Sample ID	Results
Run 1	18.386
Run 2	18.082
Run 3	18.032
Mean	18.17
COV %	1.05



1274 Hansa	Yellow 74
Sample ID	Results
Run 1	50.125
Run 2	50.096
Run 3	50.442
Mean	50.220
COV %	0.38



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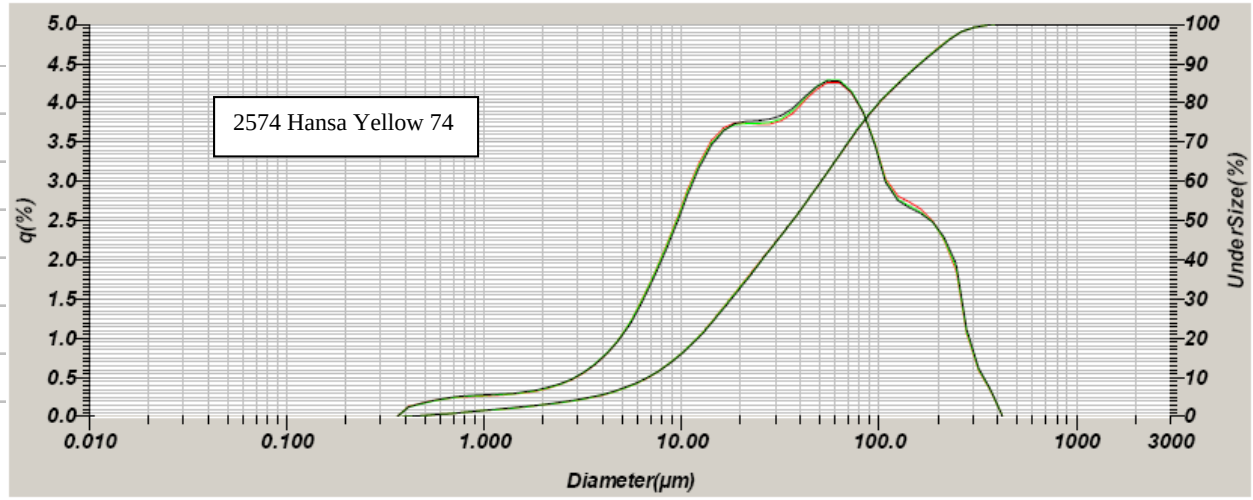
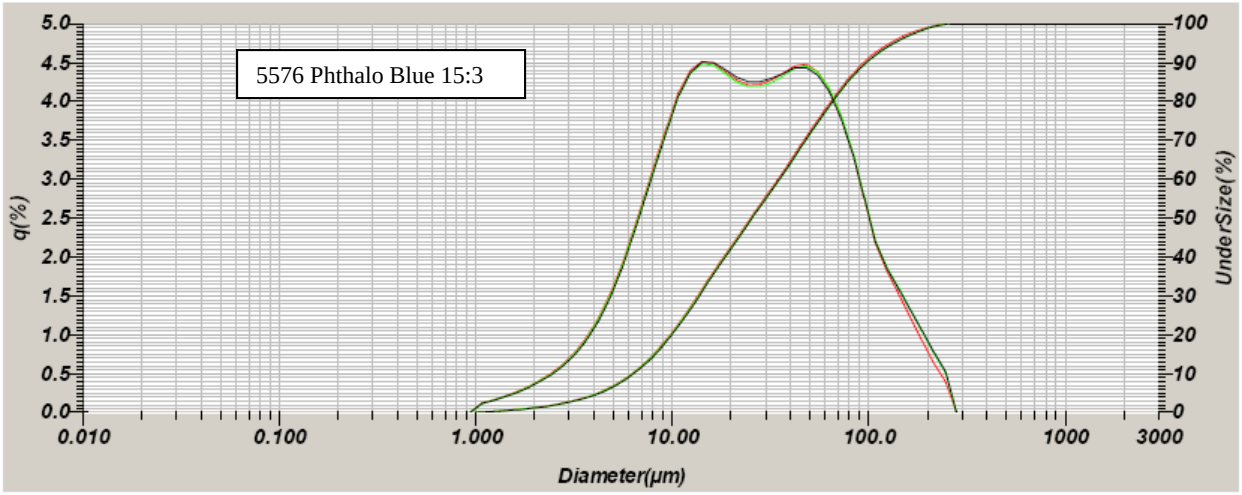
Dry Organic Pigment Results

2574 Hansa	Yellow 74
Sample ID	Results
Run 1	36.490
Run 2	36.618
Run 3	36.603
Mean	36.570
COV%	0.19



5576 Phthalo	Blue 15:3
Sample ID	Results
Run 1	25.114
Run 2	25.546
Run 3	25.531
Mean	25.397
COV %	0.97



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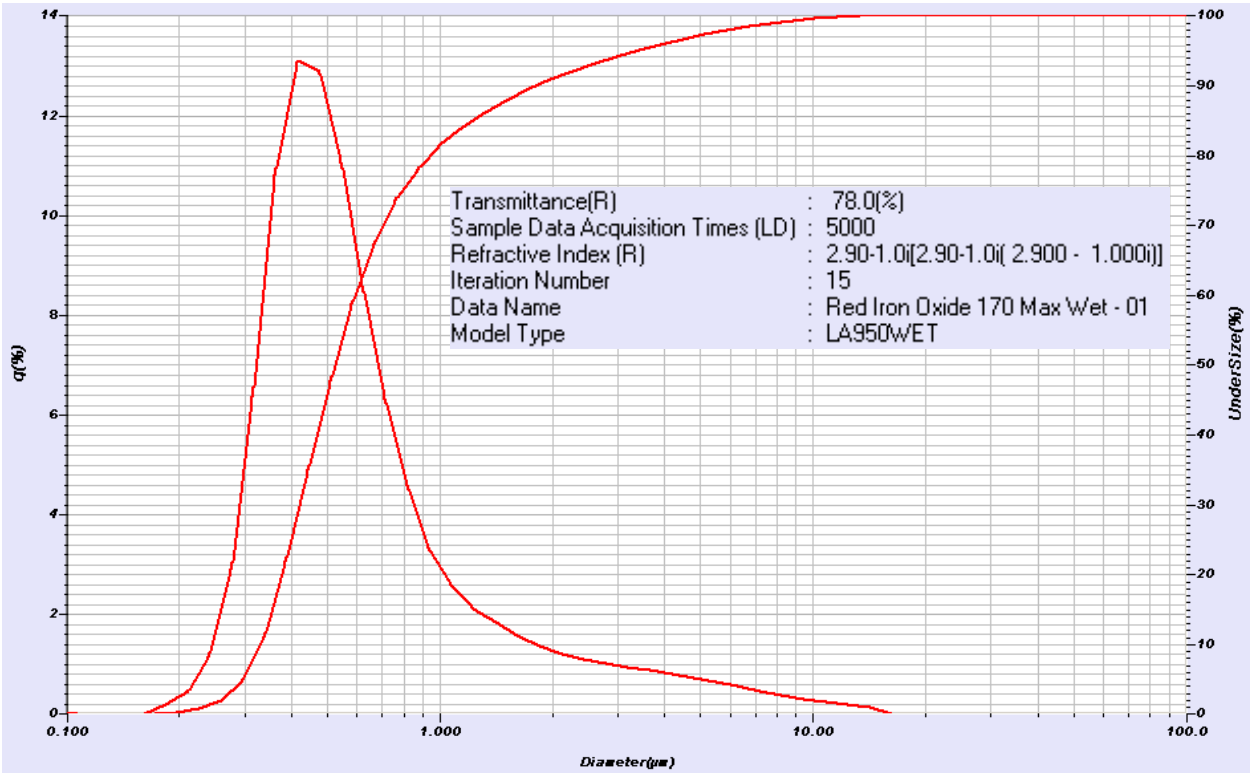
Inorganic: Iron Oxide, LA-950 Wet



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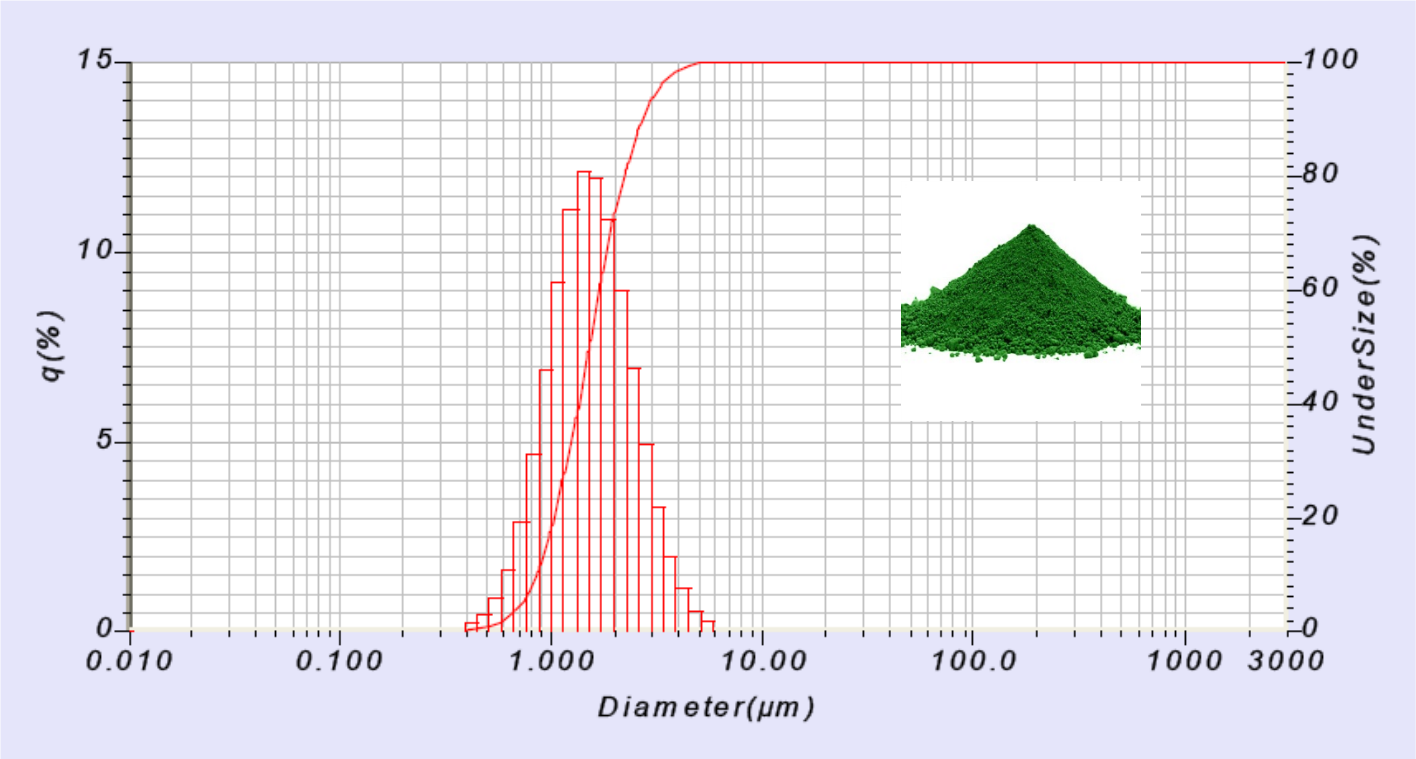
Inorganic: Chromium Oxide Green



Example data

Median:	1.51 μm
Mean:	1.73 μm
D(10%):	0.89 μm
D(90%):	2.87 μm

- Micro 90 surfactant to wet powder
- 0.1% sodium pyrophosphate

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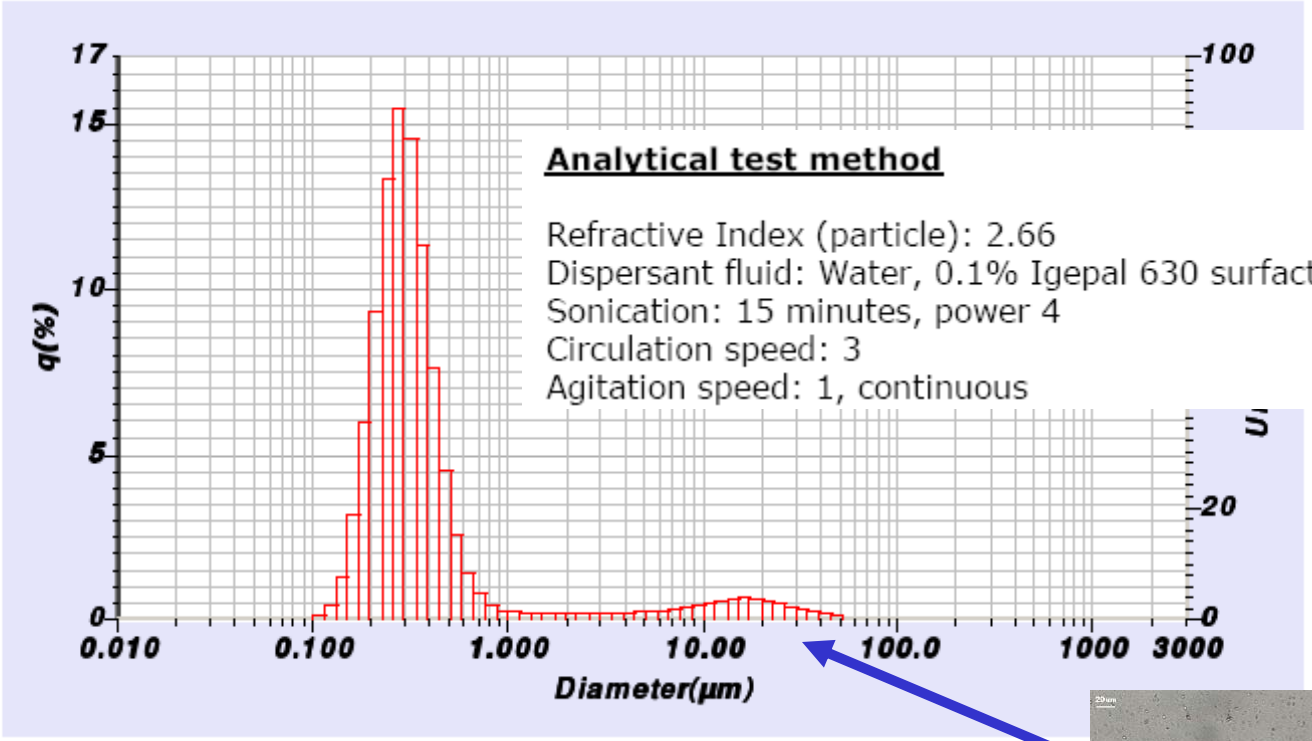
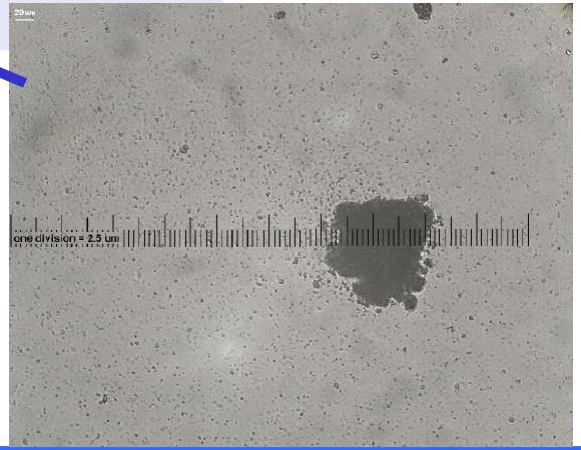
Inorganic: Alumina Al_2O_3



Analytical test method

Refractive Index (particle): 2.66
Dispersant fluid: Water, 0.1% Igepal 630 surfactant
Sonication: 15 minutes, power 4
Circulation speed: 3
Agitation speed: 1, continuous



Example data

File Name:	6472B
Median:	0.300 µm
Mean:	1.481 µm
S. D.:	4.950 µm
D(10%):	0.194 µm
D(90%):	0.643 µm

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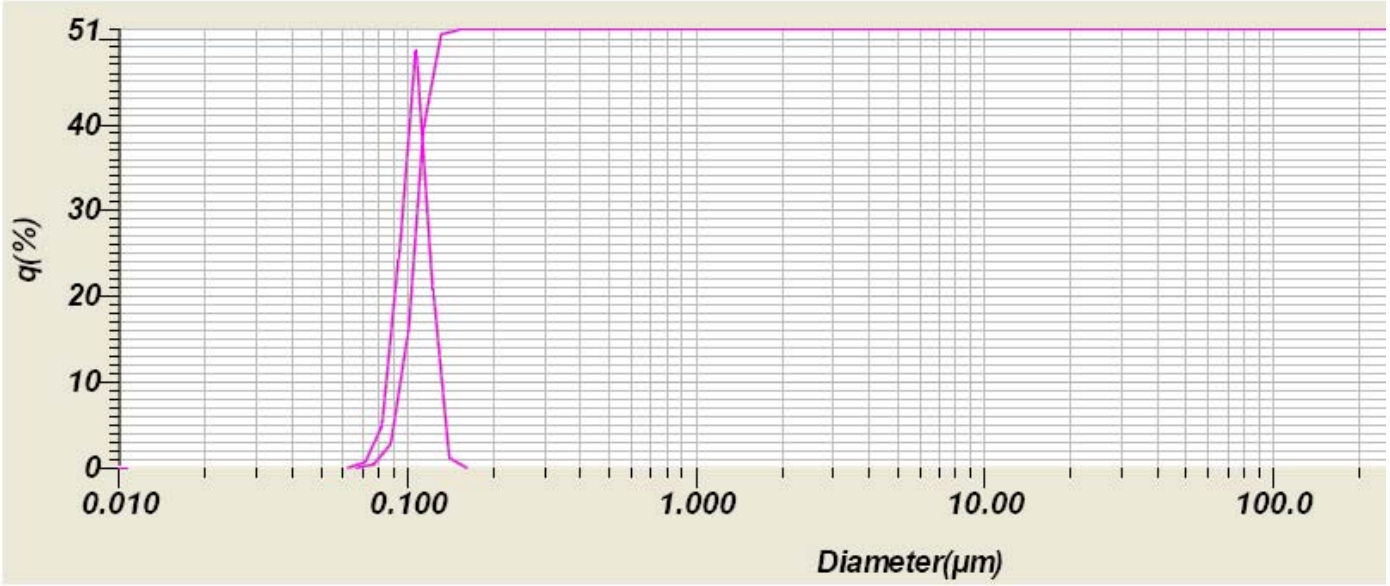
Ink Pigment



Data Name	Graph Type	Sample Name	Median Size	Std.Dev.
200511011226001		COLOR WONDER SPRAY INK	0.10583(μm)	0.0120(μm)
200511011227002		COLOR WONDER SPRAY INK	0.10583(μm)	0.0120(μm)
200511011227003		COLOR WONDER SPRAY INK	0.10574(μm)	0.0120(μm)

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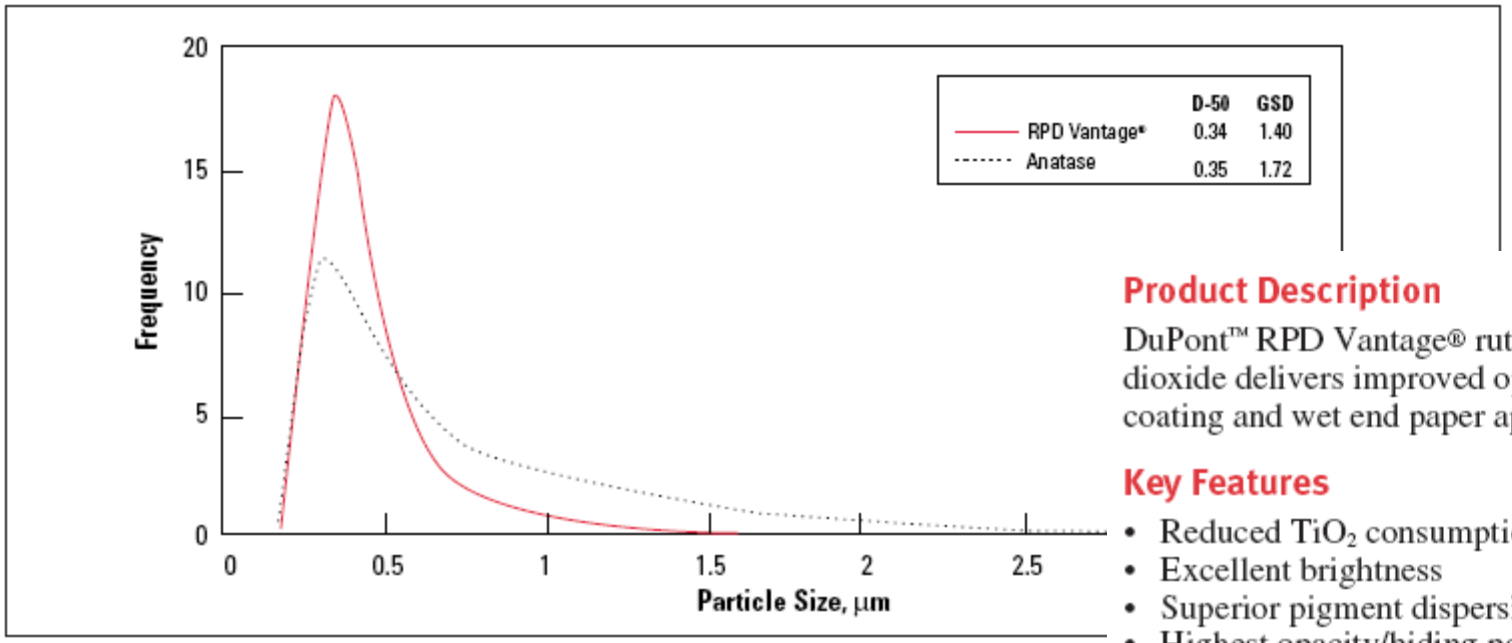
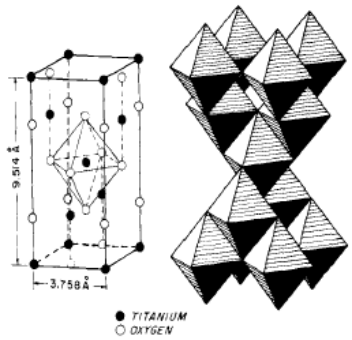
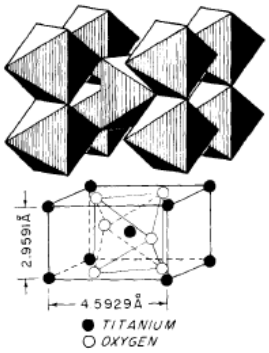
TiO_2 Commercial Data Sheet

Figure 1. Particle Size Distribution (Horiba Laser)





Crystal structure of Anatase titanium dioxide



Crystal structure of Rutile titanium dioxide

Product Description

DuPont™ RPD Vantage® rutile paper dry titanium dioxide delivers improved opacifying value for coating and wet end paper applications.

Key Features

- Reduced TiO_2 consumption
- Excellent brightness
- Superior pigment dispersion
- Highest opacity/hiding power

Particle Size

Particle size and distribution are important contributors to light scattering by diffraction. **Figure 1** shows the RPD Vantage® particle size distribution compared against anatase. The lower median particle size (D50) and the narrower geometric standard deviation (GSD) of RPD Vantage® combined with the higher refractive index of rutile result in the highest scattering efficiency.

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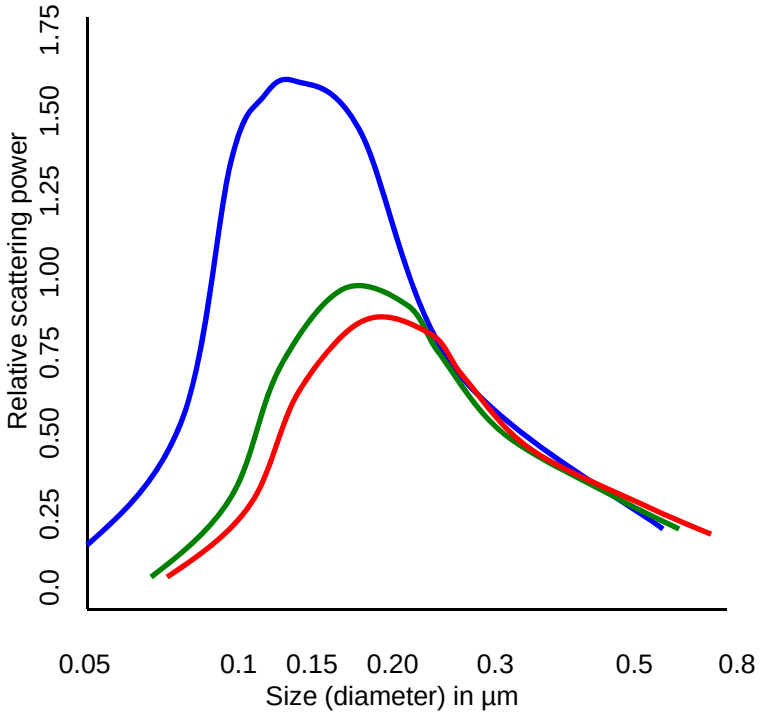
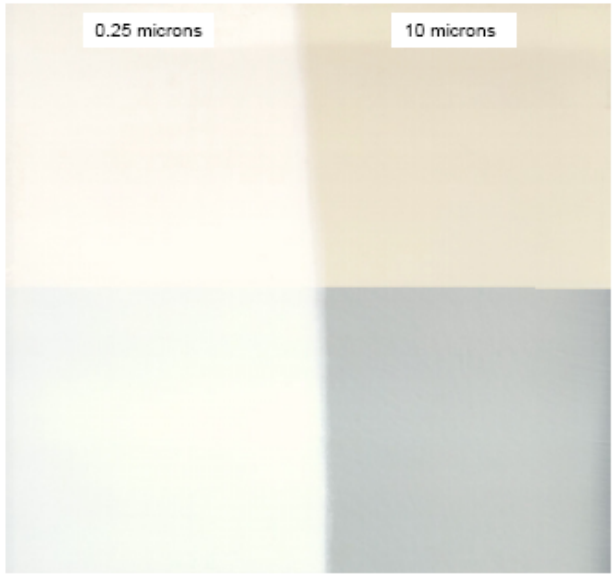
TiO_2 : Effect of Size

Relative scattering power
rutile TiO_2 vs. size



Opacity of TiO_2
mean 0.25 and 10 µm



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TiO_2 Measurement Procedure

- Step 1: Buy a HORIBA LA-950
- Decide wet or dry analysis
- Wet: disperse sample using surfactant and ultrasound
- Method development using Method Expert
  - RI, concentration, ultrasound
- Dry: Use smallest nozzle and highest air pressure

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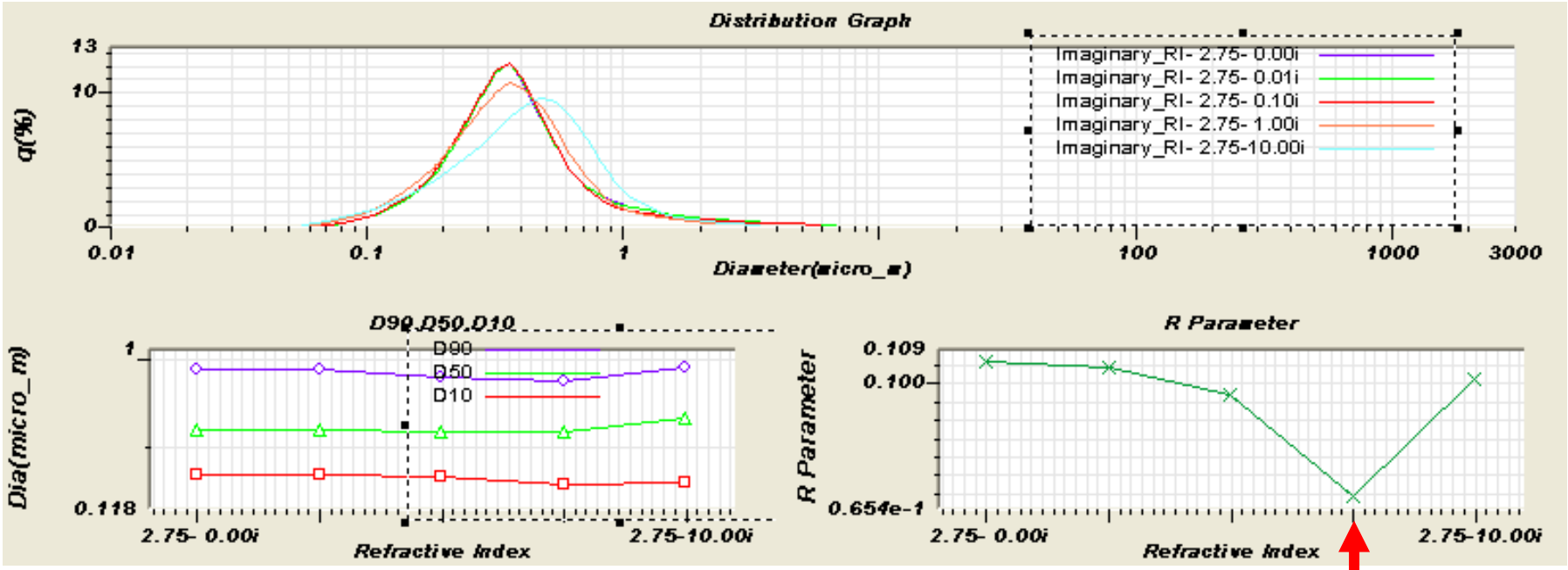
TiO_2: Effect of Refractive Index

Measure sample once
Fix real RI, vary imaginary
Minimize R parameter (error calculation)

partica LA-950
Method Expert



Use 1.0 for imaginary RI

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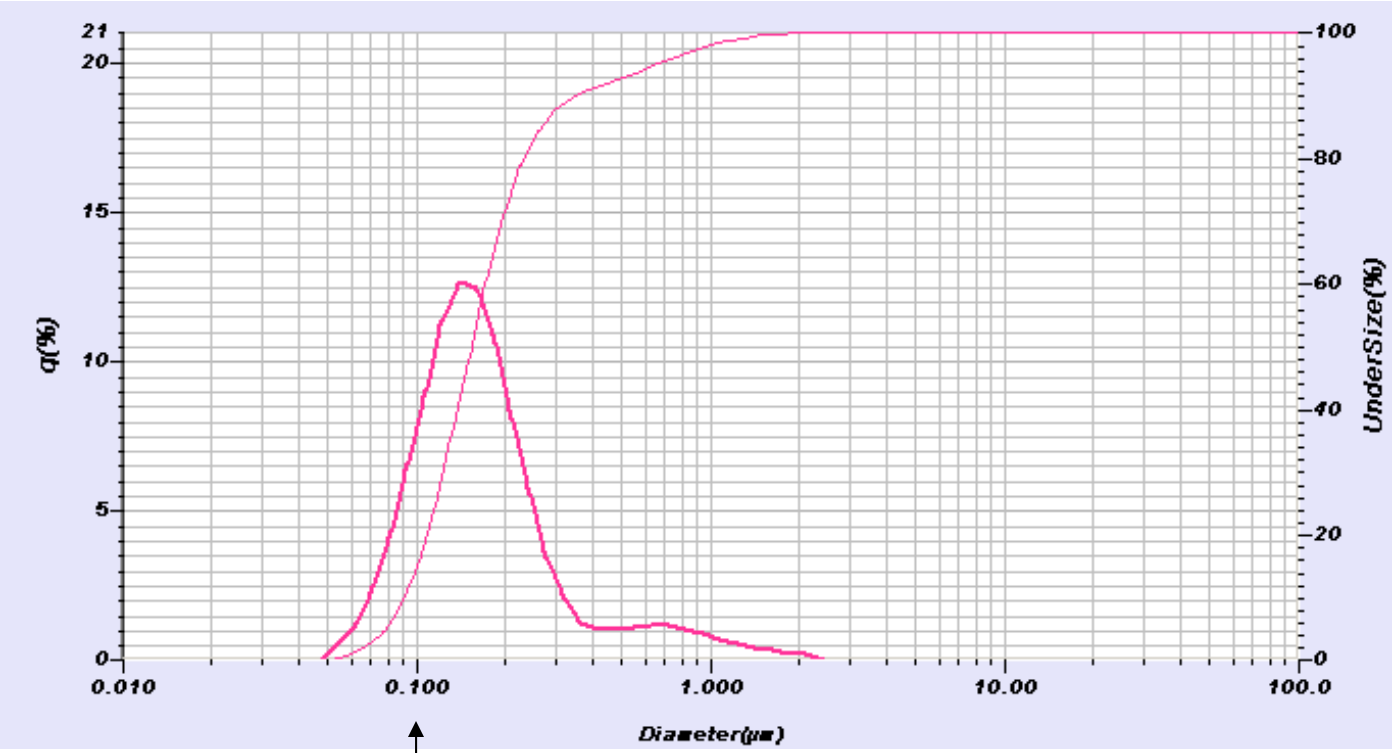
TiO_2 for Cosmetics



% under 100 nm
"nanoparticles"

Mean Size	:	0.21941(μm)
Median Size	:	0.15503(μm)
R Parameter	:	0.2560
D(v,0.1)	:	0.09093(μm)
D(v,0.5)	:	0.15503(μm)
D(v,0.9)	:	0.36061(μm)
Cumulative % on Diameter	:	(1)0.100 (μm)- 14.473(%)

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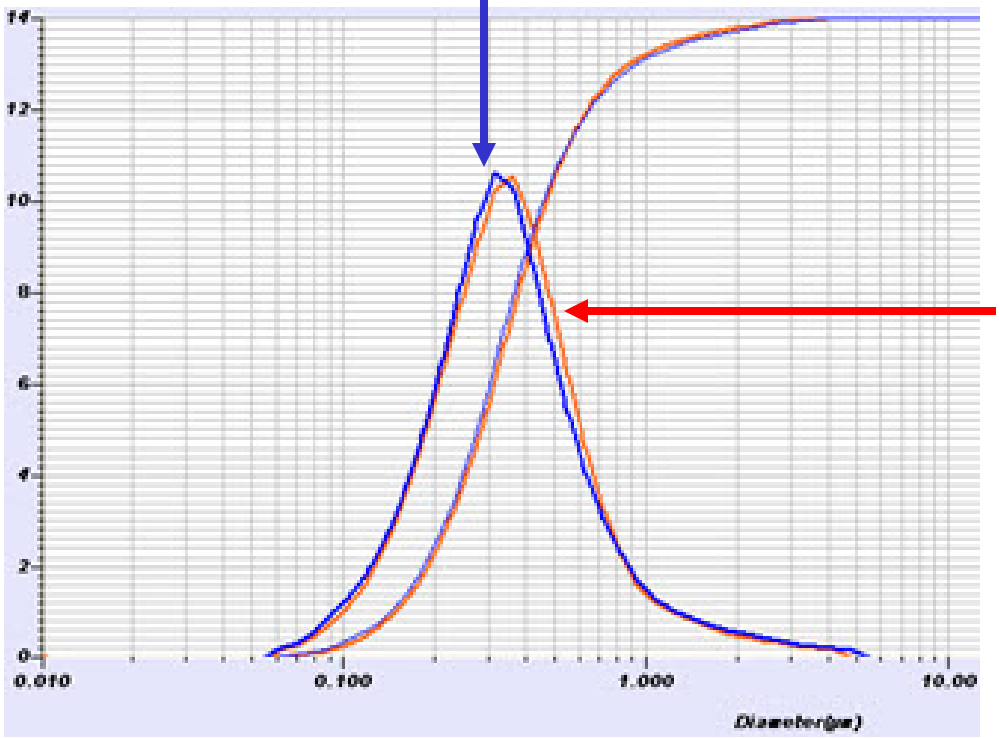
TiO_2: Wet vs. Dry

Mean Size	:	0.42307(µm)
Mode Size	:	0.3635(µm)
Geo.Std.Dev.	:	1.7940(µm)
R Parameter	:	6.8993E-2
D(v,0.1)	:	0.16831(µm)
D(v,0.5)	:	0.35496(µm)
D(v,0.9)	:	0.71036(µm)

Wet

Only 8 nm difference!
Dry dispersion below 350 nm median



Mean Size	:	0.44614(µm)
Mode Size	:	0.3615(µm)
Geo.Std.Dev.	:	1.8683(µm)
R Parameter	:	1.3147E-1
D(v,0.1)	:	0.16820(µm)
D(v,0.5)	:	0.34653(µm)
D(v,0.9)	:	0.74838(µm)

Dry

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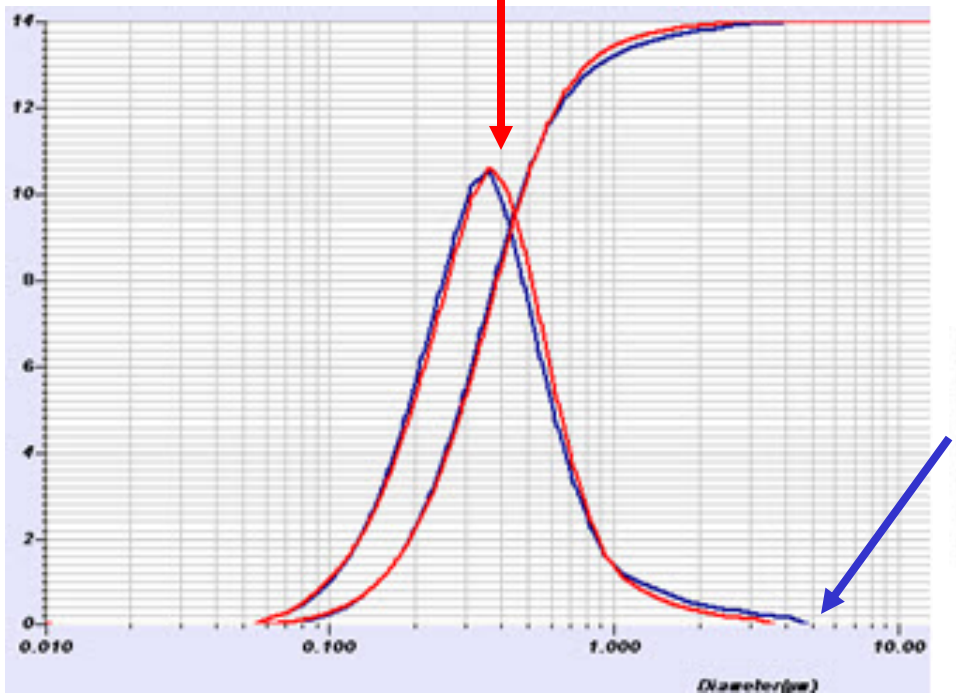
TiO_2: Dry Analysis of 2 Samples

Mean Size	:	0.44614(µm)
Mode Size	:	0.3615(µm)
Geo.Std.Dev.	:	1.8683(µm)
R Parameter	:	1.3147E-1
D(v,0.1)	:	0.16820(µm)
D(v,0.5)	:	0.34653(µm)
D(v,0.9)	:	0.74838(µm)

Able to reproducibly distinguish 2 samlpes with 12 nm difference @ D(v,0.5)



Mean Size	:	0.45286(µm)
Mode Size	:	0.3183(µm)
Geo.Std.Dev.	:	1.9285(µm)
R Parameter	:	8.0177E-2
D(v,0.1)	:	0.16169(µm)
D(v,0.5)	:	0.33394(µm)
D(v,0.9)	:	0.77890(µm)

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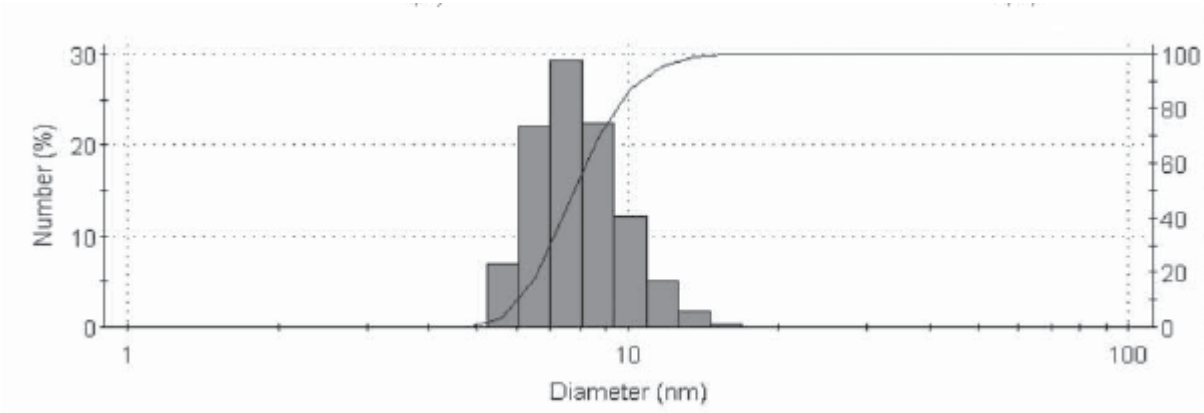
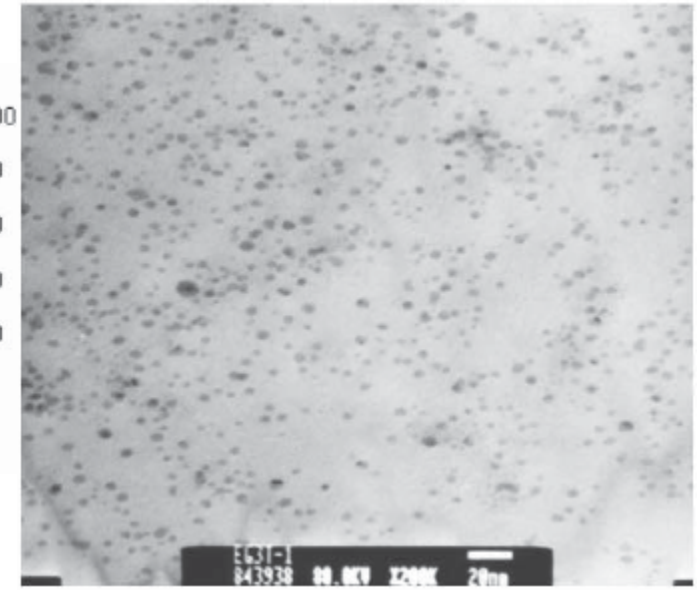
Nano TiO_2*



Data from HORIBA LB-500



* **PREPARATION AND UV CHARACTERIZATION OF TiO_2 NANOPARTICLES SYNTHESIZED BY SANSS**

Ching-Song Jwo[1], Der-Chi Tien[2], Tun-Ping Teng[3], Ho Chang[4] ,
Tsing-Tshih Tsung[4], Chih-Yu Liao[5] and Chi-Hsiang Lin[1]

Rev.Adv.Mater.Sci. 10 (2005) 283-288

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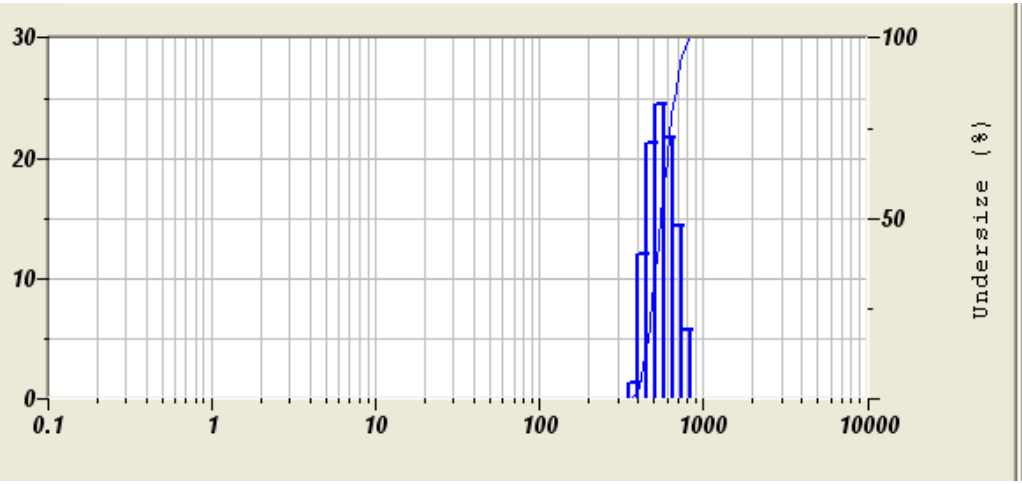
TiO_2 Size & Zeta Potential on SZ-100



Date	: mardi 13 mars 2012 13:40:23
Measurement Type	: Particle Size
Sample Name	: TiO2 P2
Scattering Angle	: 173
Temperature of the holder	: 24.8 °C
Run duration	: 60 Seconds
Representation of result	: Scattering Light Intensity
Count rate	: 1766 kCPS

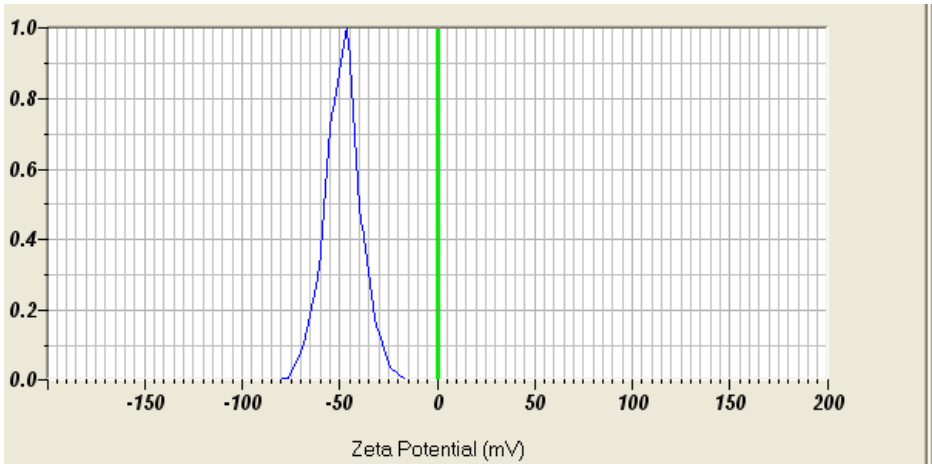
Calculation Results

Peak No.	S.P.Area Ratio	Mean	S. D.	Mode
1	1.00	567.1 nm	99.9 nm	546.6 nm
2	---	--- nm	--- nm	--- nm
3	---	--- nm	--- nm	--- nm
Total	1.00	567.1 nm	99.9 nm	546.6 nm

Cumulant Operations

Z-Average	: 566.7 nm
PI	: 0.047

Size, note: Intensity distribution



Measurement Results

Date	: jeudi 23 février 2012 10:50:08
Measurement Type	: Zeta Potential
Sample Name	: TiO2 pH 7
Temperature of the holder	: 25.0 °C
Viscosity of the dispersion medium	: 0.895 mPa·s
Conductivity	: 0.105 mS/cm
Electrode Voltage	: 3.4 V

Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	-48.4 mV	-0.000375 cm2/Vs
2	--- mV	--- cm2/Vs
3	--- mV	--- cm2/Vs

Zeta Potential (Mean)	: -48.4 mV
Electrophoretic Mobility mean	: -0.000375 cm^2/Vs

Zeta potential @ pH = 7

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Conclusions

- Particle size important physical parameter requiring analysis for control
- Laser diffraction excellent tool down to 30 nm
- Dynamic light scattering for sub micron particle size + zeta potential
- HORIBA has products and support for your application requirements

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HORIBA

Thank you

ありがとうございました

ขอบคุณครับ

谢谢

اشكُرْ

Gracias

Grazie

Σας ευχαριστούμε

धन्यवाद

நன்றி

Tacka dig

Danke

Merci

Obrigado

감사합니다

Большое спасибо

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