APPLICATION NOTE

Sample Preparation Guide

Instrument: Microtrac Laser Diffraction Analyzers

Philip E. Plantz, PhD

SL-AN28_Rev. D

Purpose

The purpose of this document is to provide customers with a guide for proper sample preparation techniques when using the WET application mode of Microtrac Diffraction Particle Size Analyzer Systems.

How to Use This Document

1. Review the information on the next page to become familiar with abbreviations and comments on materials.
2. Learn what the chemical name is by requesting from the customer or from the MSDS. Synonyms are often located in the MSDS.
3. Observe the name and compare it to those in the first list of General Rules of Solubility.
4. If the name corresponds to the names in the solubility rules list for INSOLUBLE, then use water as the carrier medium.
5. If the name corresponds to the names in the solubility rules list for SOLUBLE, then use a solvent as the carrier medium.
6. If the name does not correspond to the types on the solubility rules list, ask the customer for solubility information. Also, the MSDS (see the section labeled Physical Properties on the MSDS) can be requested which has solubility information for water. The solubility should be less than 0.01% or solubility may show in the data.
7. Next use the list of specific compounds and materials to find specific chemical names. Be sure to use this step since the list has exception to the General Rules. For instance, carbonates are generally insoluble in water but Calcium Carbonate is slightly soluble and using IPA may be best. It is not possible to list all compounds that exist, but the list will be updated annually if a material seems to be of special importance.

.

Sample Preparation of Powders and Other Selected Materials

General comments:

1. Check MSDS for solubility or reactivity.
2. Specific compounds may have special chemical or solubility characteristics.
3. Many compounds have many synonyms. MSDS usually lists synonyms.
4. Hexane, heptane or toluene can usually be used if Isopar is not available.
5. Solvents can be very volatile and flammable. Solvents ending in “ane”, “ene” or “one” are often flammable.
6. IPA has surface active properties and may not need surfactants or dispersants as does water.
7. Liquid dish detergents can be substituted for Triton X100. These often contain a combination of anionic and non-ionic surfactants. Combination can aide wetting.
8. It is important to use the refractive of the material or a value that is closest. The instrument automatically takes into account that large particles do not transmit light. There is no need to select “Absorbing” for these large particles since, Mie scattering concepts are used over the entire range and takes this into account.

Special Comments:

1. Some clays, cellulose, starches and some polymers will swell in water. Understand use of the material to assist this. Baby diapers use high absorption polymer and will swell. Transmission may decrease as Li (loading index) increases as multiple measurements are made. Sometimes the loading screen will show this, but it depends on how fast swelling is occurring.
2. Some pharmaceutical slurries will have a mixture of compounds. Some compounds will dissolve, others may not. Transmission will increase, Li (loading index) may decrease as multiple measurements are made. Sometimes the loading screen will show this, but it depends on how fast dissolution is occurring.
3. Hygroscopic materials adsorb or absorb water. Particles will agglomerate due to capillary forces. Some minerals, foods, pharmaceuticals are included in this group. May be very difficult to completely disperse using dry powder system.
4. Materials very soluble in organic solvents usually can be run in water and vice versa.
5. Ultrasonic energy should be used with care. Be sure that the answer desired includes dispersed particles. It is not always the situation where completely dispersed particles are desired.
6. Representative sampling is extremely important to obtaining reproducible, meaningful data. See Microtrac User’s Course notes for suggestions.

Abbreviations:

Note: The numbers (1 – 5) below are used in the section showing acceptable surfactants and dispersants for specific materials and compounds.

- 1 = SMP = Sodium (hexa) meta phosphate; Use 2% in water
- 2 = TSPP = Tetrasodium pyrophosphate; Use 2% in water
- 3 = TX100 = Triton X100; Non-ionic surfactant. Use 5% in water
- 4 = IPA = Isopropyl alcohol; Use 99% IPA
- 5 = Lec = Lecithin; Soy bean, liquid: Use 2% in IPG
- IPG = Isopar G
- NN = No surfactant or dispersant needed

General Comments About Transferring Sample to the Circulation System

To be sure that there is no dynamic situation occurring, allow the software to remain in the Loading Screen unless there is assurance that solubility or swelling cannot occur. For samples where there might be questions, allow circulation for ½ - 1 minute. During that time period the transmission can change by 0.1-0.2 but not much more. If that happens then measuring time should be shortened from the normal 30 seconds to 15 -20 seconds or a new fluid must be used.
If Triton X100 or other high foaming surfactant is being used, be sure that the sample is "run down" the side of the dispersion bowl of the SDC or FlowSync. Transferring the sample from a pipette directly into the fluid residing in the bowl, will likely cause bubbles to form. Place the tip of the pipette onto the side about ½ inch above the fluid surface. Gently expel the sample suspension down the side
If suspensions are sampled and large particles are present in the material, they can sediment at the bottom of the container. When sampling with a pipette, this can lead to sampling errors! Be aware of this when you observe particle settling. In this case, stir the liquid well, e.g. with a stirring plate. Immerse the pipette up to approx. 1/3 above the floor and remove the amount of liquid you need for the analysis. Transfer this amount completely into the instrument (sedimentation also occurs in the pipette).

Soluble Sample Particles

Use of saturated solutions for measuring materials soluble in water is an approach that can avoid the dissolution of particles. Particle size measurement requires that sample particles are insoluble in the carrier medium used to transport the particles to the cell through which the laser passes and where the light scattering occurs. Isopar will avoid dissolution in most situations, but often requires a surfactant. One possible surfactant is lecithin which is a food grade oil. Soybean lecithin is a liquid and is available from health-food stores. Its yellow color does not interfere with measurements or set zero. Microtrac has used this solution for many years with considerable success.

In some situations, the use of an organic solvent is not desirable. Local disposal and use regulations can cause excessive costs to be born for transport and safe disposal as well as storage of organic solvent supply not presently in use. One approach is to measure the samples dry using the Turbotrac or TurboSync accessory. A potential issue is that capillary forces that develop between and among the particles require considerable physical energy to overcome agglomeration resulting from such forces. Application of highest energy may "break" or fracture the particles rather than separate the agglomerate into separated particles. Fumes hoods that expel solvent fumes may not be available to allow safe use of solvents as well.

A third approach to measuring water soluble particles is to prepare a solution of the compound in water that will not sustain further dissolution of particles. This type solution is known as being saturated. It is prepared by adding sample to water and stirring until no further sample will dissolve. It can also be prepared based upon known solubility data for the compound. There are a couple of comments that are necessary. Saturated solutions represent a dynamic condition where particle molecules that have already dissolved can return to the solid state as a solid molecule dissolves. Thus, the system is not perfectly stable. Slight increases or decreases of temperature can cause molecules to assemble and precipitate or crystallize when temperature drops.

Most likely temperature will increase, and the saturated solution may demand more sample to remain saturated. As a result of this possibility, it is important that the circulating solution being used as the carrier fluid not be circulated very long before transfer of actual sample. Transfer of clean fluid, alignment and set zero should be performed immediately if circulation so to continue and sample particles should be transferred immediately upon completion of the set zero. This will minimize or totally avoid dissolution for sample particles due to clean fluid temperature increase. The loading index and transmission values should be monitored for a decrease and increase respectively that will indicate dissolution of sample. The opposite effect may indicate re-crystallization or return of the sample particles to the circulating sample. Depending upon the amount of sample returning or dissolving, the data may be affected by reporting incorrect information on the distribution. Loading values should be nearly constant during the loading procedure to be sure particles are not dissolving during the sample loading process. Note that small particles will disappear before larger particles because they are smaller. Thus, the presence of fines can diminish resulting in larger than expected size.

General Rules for Solubility of Compounds in Aqueous Systems

The solubilities listed below are general and exceptions may exist. If material is soluble use an organic solvent as the carrier fluid or use dry powder measurement. If material is insoluble in water, use water as the carrier fluid.

Halide = Chloride, bromide, iodide, fluoride
Alkali metals = Li, Na, K, Rb, Cs (Shown left-hand column on the periodic chart)
Ammonium = NH4+, Ag= silver, Hg = mercury, Pb=lead

Rule – Soluble in Water – Do Not measure in Water	**Exceptions – Measure in Water**
Ammonium (contains NH_4^+)	None
Li, Na, K, Rb, Cs compounds (These are alkali metals)	
Nitrates	
Acetates	
Bromides	Halides of Ag(I), Hg(II), Pb(II)
Chlorates	
Chlorides	Halides of Ag(I), Hg(II), Pb(II)
Fluorides	Halides of Ag(I), Hg(II), Pb(II)
Iodides	Halides of Ag(I), Hg(II), Pb(II)
Perchlorates	
Sulfates	Sulfates of Ag, Ca, Sr, Ba, Pb(II), Hg(II)

Rule – *In*soluble or Not Soluble in Water – Measure in Water	**Exceptions – Do Not measure in Water**
Carbonates	Alkali metal compounds, Ammonium salts
Chromates	Alkali metal compounds, Ammonium salts
Hydroxides	Calcium, strontium and barium $Ca(OH)_2$ Alkali metal compounds, Ammonium salts
Oxalates	Alkali metal compounds, Ammonium salts
Oxides	Alkali metal compounds, Ammonium salts
Phosphates	Alkali metal compounds, Ammonium salts
Sulfides (S)	Alkali metal compounds, Ammonium salts CaS, SrS, BaS
Silver, Pb(II) and Hg(I) compounds	

Material Chemical	Chemical Formula	Carrier Fluid	Surfactant Dispersant	Comments
Alloys		Water	1, 2 or 3**	
Aluminosilicates	CaAl2Si2O8 or AlNa12SiO5	Water (caution) or IPA	1, 2 or 3	Some aluminosilicates can be slightly soluble in water
Barium Sulfate	BaSO4	Water	1, 2 or 3	very slight dissolution in water
Bentonite (clay) NON-swelling	Ca (calcium) Bentonite	Water	1, 2 or 3	
Bentonite (clay) swelling	Na (sodium) Bentonite	IPA or other organic solvent	NN	swells in water
Bicarbonate		IPA or other organic solvent	3, 5	Some very soluble e.g. Mn, Na, Li, K, Na, Rb, Cs, ammonium
Bismuth trioxide		Water	1, 2 or 3	
Calcium carbide		Isopar G	5	Incompatible with IPA and water
Calcium carbonate	CaCO3 powder	IPA		slight dissolution in water
Calcium carbonate	Slurry	Water	1, 2	Saturated solution will precipitate in IPA
Calcium fluoride		Water	1, 2	
Calcium fluoroapatite		Water	1, 2	
Calcium oxide		IPA or Isopar G	4, 5	
Calcium sulfate		IPA or Isopar G	4, 5	
Carbide		Isopar G	5	As a class assume incompatible with water and alcohols
Carbon Black		Water or IPA	WATER use 3 IPA use NN	
Carbonates		Water or IPA	WATER use 3 IPA use NN	Some very soluble in water
Cement		Water (caution) or IPA or dry	WATER use 1 or 2, IPA use NN dry powder	some dissolution in water
Ceramics		Water	1, 2 or 3	
Charcoal		Water or IPA	WATER use 3 IPA use NN	

Material Chemical	Chemical Formula	Carrier Fluid	Surfactant Dispersant	Comments
Chlorides		IPA	NN	
Chocolate		Low MW oil or Isopar	Food grade oil use NN IPG use 5	Typical food grade oil is NeoBee M5, low to medium molecular weight fatty acid esters
Chromate		Water	1,2, 3	Some are soluble such as Li, Na. K, Rb and ammonium
Clay		Water or IPA	1, 2	
Coal		Water or IPA	WATER use 3 IPA use NN	
Diamond		Water		
Diamond		Water		
Diatomaceous earth		Water or IPA	WATER use 3	
Dolomite (MgCO3)		Water (caution) or IPA	WATER use 1,2, or 3 IPA use NN	Very slight dissolution in water
Emulsion oil in water		Water	NN	Dilute as little as possible
Emulsion water in oil		Isopar G or original oil	NN	Be careful of viscosity of original oil - dilute as little as possible
Feldspar		Water	1, 2 or 3	
Fly Ash water insoluble		Water	1, 2 or 3	
Fly Ash water solubles present		IPA	NN	
Garnet		Water	1, 2 or 3	
Hydrides		See MSDS		Potential reactions with water to liberate hydrogen gas
Hydroxides, e.g. aluminum hydroxide		Water (caution) or IPA	1, 2 or 3	May be slightly sol in water ammonium, Sr, Ba, Ca, Li, Na, K, Rb
Iron carbonate	FeCO3	Water	1 or 2	
Iron Oxide as Hematite, magnetite and other iron oxides	Fe2O3, FeO, Fe3O4	Water	1, 2 or 3	

Material Chemical	Chemical Formula	Carrier Fluid	Surfactant Dispersant	Comments
Latex aqueous suspension		Water	NN	Customer suggestion may be better
Latex powder		Water	3	Customer suggestion may be better
Liposomes		Water	NN	Customer suggestion may be better
Magnesite (Magnesium Carbonate)	$MgCO_3$	Water (caution) or IPA	1, 2 or 3	Slight dissolution in water
Magnesium aluminum silicate		Water or IPA	1, 2 or 3	
Metal powders		Water	1, 2 or 3	
Mica		Water	3	
Microsilica		Water	1, 2 or 3	
Milk powder		Water (caution) or IPA	NN	
Nickel		Water	1, 2 or 3	
Nitride		Water	1, 2 or 4	Also, check MSDS
Nylon powder		IPA or Isopar	IPA use NN IPG use 5	
Ophthalmic suspension		Water (caution)	NN	
Oxides		Water or IPA	1, 2 or 3	
Peptides		Water or dry	WATER insoluble use 1, 2 or 3 IPA insoluble use NN	
Perchlorates		Isopar	5	
Pharmaceuticals water insoluble		Water	1, 2 or 3	
Pharmaceuticals water soluble powders		Isopar G	5	
Pharmaceuticals water soluble suspension		Water (caution)	NN	Load quickly and measure 10-20 seconds

Material Chemical	Chemical Formula	Carrier Fluid	Surfactant Dispersant	Comments
Pigments - inorganic		Water	1, 2 or 3	
Pigments - organic		IPA, Isopar , isobutyl ketone,	5	
Plastic powders		Water or IPA	3	
Potassium perchlorate		Isopar G	5	
Proteins		Isopar G or IPA	IPG use 5	Some proteins soluble in IPA
Pyrites		Water	1, 2 or 3	
Rubber		Water or IPA	WATER use 3 IPA use NN	
Salt (table salt)		IPA, Isopar, hexane, heptane, toluene	5	
Sand (quartz)	SiO_2	Water	1, 2 or 3. Most often NN	
Silane emulsions		Water or IPA	NN	
Silicic Acid		Water or IPA	1, 2 or 3	
Silicates		Water	1, 2 or 3	
Silicon nitride	SiN_3	Water or IPA	1, 2 or 3	
Sodium carbonate		IPA or Isopar		
Sodium chloride		IPA	NN	
Soil/Sediment		Water	1, 2 or 3	
starch		IPA or Isopar G	IPA use NN IPG use 5	
sucrose		IPA or Isopar G		
Sugar		IPA or Isopar G	IPA= NN IPG use 5	
Sulfates		See Solubility rules		
Sulfides		See Solubility rules		
Talc (talcum powder)		Water or IPA	3	
Teflon		IPA or water	3	

Material Chemical	Chemical Formula	Carrier Fluid	Surfactant Dispersant	Comments
Uranium Dioxide		Water or IPA	WATER use 1, 2, 3 IPA use NN	
Vanadium pentoxide		IPA or Isopar	IPA use NN IPG use 5	Slightly soluble in water
Wax Furniture		IPG		
Wax Floors		Water	3	
Zinc Carbonate		Water (caution) or IPA	1, 2 or 3 IPA use 3	Slightly soluble in water
Zinc phosphate		Water	3	
Zinc sulfide		Water / IPA	3	Slightly soluble in water
Zirconia		Water	1, 2 or 3	
Zirconium dioxide		Water	1, 2 or 3	

.

For further information please contact us at:

www.microtrac.com