APPLICATION NOTE

MICROTRAC

Sample Preparation Guide

Instrument: Microtrac Laser Diffraction Analyzers

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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.
- 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.
- 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:

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

1ICROTRA

- 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.

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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 NH4 ⁺) | 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 – Insoluble 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 | Carrier Fluid | Surfactant | Comments |
|----------------------------------|-----------------------------|----------------------------------|---|---|
| Chemical | Formula | | Dispersant | |
| | · · · | | - | · · · · · · · · · · · · · · · · · · · |
| 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 | Carrier Fluid | Surfactant | Comments |
|----------|----------|---------------|------------|----------|
| Chemical | Formula | | Dispersant | |

| 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 | Carrier Fluid | Surfactant | Comments |
|----------|----------|---------------|------------|----------|
| Chemical | Formula | | Dispersant | |

| 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 | Carrier Fluid | Surfactant | Comments |
|----------|----------|---------------|------------|----------|
| Chemical | Formula | | Dispersant | |

| 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 | Carrier Fluid | Surfactant | Comments |
|----------|----------|---------------|------------|----------|
| Chemical | Formula | | Dispersant | |

| Uranium Dioxide | Water or IPA | WATER use 1, 2, 3 | |
|--------------------|--------------------|-------------------|---------------------------|
| | | IPA use NN | |
| Vanadium pentoxide | IPA or Isopar | IPA use NN | Slightly soluble in water |
| | | IPG use 5 | |
| Wax Furniture | IPG | | |
| Wax Floors | Water | 3 | |
| Zinc Carbonate | Water (caution) or | 1, 2 or 3 | Slightly soluble in water |
| | IPA | IPA use 3 | |
| 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