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TITANIUM DIOXIDE

DETERMINATION OF SCREEN PACK DISPERSION OF TITANIUM DIOXIDE
(70% Masterbatch, 500 Mesh Screen)

METHOD: T4400.583.03.DTT

1. Principle

Relative dispersion quality of titanium dioxide is determined by extruding a 70 WT % TiO₂/LDPE (low density polyethylene) concentrate through a screen pack and instrumentally rating screen blockage caused by undispersed TiO₂.

A 70 WT % concentrate of TiO₂/LDPE is prepared using a Batch Internal Mixer.

This concentrate is chopped into small granules and is extruded using a single screw extruder through a 500 mesh screen. A measurement of the undispersed TiO₂ particles retained on the screen is made using the X-ray fluorescence of titanium.

2. Applicability

2.1. General

This method can be used to measure the relative dispersion quality of titanium dioxide pigment in low density polyethylene (LDPE).

2.2. Specific

This method is used to measure the relative dispersability, primarily of plastic grade pigments designed for highly loaded masterbatch in Low Density Polyethylene (LDPE). The method is used for finished product customer specification testing. This method is not recommended for general purpose pigments.

Using XRF to rate screens, this test measures the relative amount of TiO₂ which remain on certain screens, but does not measure the rate of dispersion of the TiO₂ which goes through the screens.

3. Limitations

Only the undispersed TiO₂ is measured.

4. Sensitivity, Precision and Accuracy

4.1. Sensitivity

No data is available.

4.2. Precision

4.2.1. Single Operator

(This section will be populated with the analysis data as it becomes available.)

4.2.2. Multiple Operator

(This section will be populated with the analysis data as it becomes available.)

4.3. Accuracy

No study has been made: however, the method has proved to be satisfactory based on historical experience including customer experience.

5. Special Apparatus

Equivalent apparatus may be substituted and may have to be substituted due to model changes. However, substituted apparatus/equipment must be capable of passing Interlab Crosschecks (ILC). ILC's, for qualification of substitute equipment, must contain samples of both high and low range screen pack dispersion material.

Examples of equipment used are listed below:

5.1. Balance capable of weighing 2000 ± 5 g.

5.2. Batch Internal Mixer, Banbury Mixer model "BR", Farrell Corporation, Ansonia, CT.

- 5.3. Chopper, Equipped with a ¼" screen. Ramco 810 or equivalent.
 - 5.4. Extruder, Killion 3/4 inch, single screw, extruder, Feed Screw Master - 24 : 1 (std), material 4140 steel, Chrome. Killion Extruding Inc., 200 Commerce Road, Cedar Grove, NJ 07009, 201-239-0200.
(See T4400.583.02.WP for drawing of screw design).
 - 5.5. One inch screens for screen packs; 30, 60 and 500 mesh, QED inc., Charlotte, NC, 704-525-3053.
 - 5.6. Breaker plate. (See T4400.583.02.WP for drawing).
 - 5.7. Philips PW-1404 X-ray spectrometer (or equivalent), Philips Electronic Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430.
or a
9200 Series Portable Analyzer, Texas Nuclear Corp., Austin, TX.
6. Reagents (Reagent grade except as noted)
- 6.1. Petrothene NA-206-000 Low Density Polyethylene Resin, Equistar Chemical.
 - 6.2. DuPont 20 Series Polyethylene Resin.
 - 6.3. TiO₂
 - 6.4. DPE6611, DuPont Polyethylene Purge Resin 6611.
 - 6.5. Clear coating, Krylon 1301 clear spray coating, or equivalent.
7. Special Safety Considerations
- 7.1. Product Hazards
There are no unusual product hazards. However, titanium dioxide is regulated as an air contaminant and care should be taken to minimize product dusting. Wipe up all spills with a damp towel.

Refer to the Material Safety Data Sheets for any chemical used in the test with which you are not familiar.
 - 7.2. Procedure Hazards
 - 7.2.1. Observe all equipment special safety rules (for your individual pieces of equipment), including general site safety rules, when using any of the equipment listed in this procedure.
 - 7.2.2. The Batch Internal Mixer (Banbury), Extruder and Granulator all have moving parts and/or hot areas. Read and follow the manufacturers operating instructions and safety precautions for each piece of equipment.
 - 7.2.3. Wear gloves while handling warm/hot concentrate. DO NOT PERMIT HOT CONCENTRATE TO CONTACT ANY PART OF THE BODY AS A SERIOUS BURN WILL RESULT.
 - 7.2.4. The 9200 Series Portable Analyzer uses two radioactive sources, Iron 55 and Americium 214. Follow the Manufacturers and Site Safety and Operating Procedures/Instructions for the operation of the analyzer at your site.
 - 7.2.5. Follow the manufactures operating instructions, site safety rules and site Standard Job Procedure when operating the Philips PW-1404 (or equivalent) X-ray equipment.
 - 7.2.6. Individual site requirements for radiation monitoring and personal protective equipment must be followed when operating the equipment listed in #4 and #5 above.

8. Procedure

- 8.1. Operating Conditions This procedure consists of five separate operations.
OPERATION OF THE BATCH INTERNAL MIXER (BANBURY MIXER)
OPERATION OF THE RAMCO 810 STRAIGHT KNIFE GRANULATOR
OPERATION OF THE KILLION EXTRUDER
BURNING THE SCREENS
READING THE SCREENS

Batch Internal Mixer (Banbury), Granulator, Extruder, X-ray or Portable Analyzer
Follow the manufacturer's operating instructions.

Each individual site will have to establish their own SJP's and/or operating procedures including specific temperatures, pressures, speeds, times, etc., for their individual pieces of equipment and circumstances. Any changes in the method must not change the site's ability to pass ILC's.

- 8.2. Calibration

The Philips PW-1404 Spectrometer (or equivalent) is to be calibrated, at a frequency to be determined by each individual site using that instrument, following the site calibration procedure.

The Texas Nuclear Analyzer is to be calibrated at a frequency to be determined by each individual site using that equipment, according to the procedure in the manufacturer's operating manual.

- 8.3. Sampling

Samples are normally supplied by manufacturing.

- 8.4. Sample Analysis

Sample analysis consists of the following sections:

OPERATION OF THE BATCH INTERNAL MIXER (BANBURY MIXER)
OPERATION OF THE RAMCO 810 STRAIGHT KNIFE GRANULATOR
OPERATION OF THE KILLION EXTRUDER
BURNING THE SCREENS
READING THE SCREENS

- 8.4.1. OPERATION OF THE BATCH INTERNAL MIXER BANBURY MIXER

This equipment causes a compounding (mixing) of the TiO₂ and Petrothene low-density polyethylene resin into a 70% concentrate. The two materials are mixed at high shear conditions, to be determined by each site for their specific piece of equipment. The heat generated causes the polyethylene to melt and fuse with the TiO₂. A high level of housekeeping and cleanliness must be observed to prevent contamination between samples.

SAFETY:

Manufacturer's Safety Rules pertaining to the use of the Batch Internal Mixer (Banbury), along with Local Site Safety Rules (including Lock, Tag and Try) must be followed when working with this piece of equipment.

The equipment must be locked out before any clean-out work is started. Follow the "Lock, Tag and Try Procedure".

Use caution when handling "Hot Samples", always wear leather or similar type gloves.

Do not grab at concentrate when dumping.

The following may be used as a guide for the operation of the individual sites Batch Internal Mixer (Banbury).

- 8.4.1.1. Set water valves to obtain a nominal constant inlet temperature of 80 - 90°F. (Provide sufficient cooling to minimize resin sticking to metal surfaces).
- 8.4.1.2. Check the oil level in the lube oil reservoir if applicable. Add oil as needed per mfg.'s instructions.
- 8.4.1.3. Open the main air valve and turn on main power.
- 8.4.1.4. Set the Batch Internal Mixer air cylinder pressure to maintain required stock pressure during processing. (See Appendices 1 and 2 for information on calculating Stock Pressure and Fill Factor).
- 8.4.1.5. Weigh into an adequate sized paper bag (approx. 16#) 715 ± 5 g of NA-206-000 LDPE and 1670 ± 5 g of TiO₂ sample, standard or dummy sample. (A dummy sample is any R-104 type pigment that is used to preheat the mixer and then discarded).

NOTE: If a Batch Internal Mixer different than a Banbury "BR" model is used, refer to Appendix 1 for calculation of the appropriate batch charge.

- 8.4.1.6. Close the paper bag and shake or mix by inverting 7-8 times.
- 8.4.1.7. Batch Internal Mixer Purge
Prewrite four 1150 ± 5 g charges of NA-206-000 into paper bags. Two of these will be used with the dummy sample to clean and preheat the mixer. The remaining two will be used to clean the mixing chamber after compounding is finished.

NOTE: Use Appendix 1 to calculate charge size for Batch Internal Mixers other than Banbury "BR" models.

- 8.4.1.8. Turn on the potentiometer and digital temperature output.
- 8.4.1.9. Turn on hood exhaust.
- 8.4.1.10. Start Up:
Follow the manufacturers and site startup and operating procedure for the Batch Internal Mixer at your site.
- 8.4.1.11. Start the Batch Internal Mixer by pressing the forward button. Adjust the mixer to low speed (70 - 100 rpm) using the potentiometer.
- 8.4.1.12. Run two batches of the preweighed Petrothene polyethylene resin (1150 ± 5 grams) and the dummy sample/polyethylene through the Batch Internal Mixer to heat it up before running the standard or samples. Check for correct ram air cylinder pressure at this time. Discard these three samples. Then compound the reference standard and samples. (Follow Steps 8.4.1.13 - 18 for all material).
- 8.4.1.13. Close the sides and with the ram in the up position, add the resin, dummy sample, standard or sample. Brush down any pigment which may have collected on the ram or feed chute area.
- 8.4.1.14. Close the feed chute door and increase the Batch Internal Mixer speed to maximum. Lower the ram (weight).

- 8.4.1.15. Continue to run the Batch Internal Mixer at maximum rpm the temperature display indicates $235 \pm 5^{\circ}\text{F}$ for dummy, standard or sample charges. For resin, temperature display should indicate $225 \pm 5^{\circ}\text{F}$. Once this temperature is reached, reduce the speed to ~116 rpm. Raise the ram, open the door and vacuum the ram and feed chute area of the Batch Internal Mixer.
 - 8.4.1.16. Close the door and lower the ram.
 - 8.4.1.17. Run the Batch Internal Mixer at ~116 rpm for one minute. At the end of the minute, reduce the mixer speed to low speed (70 - 100 rpm).
 - 8.4.1.18. Dump the sample into the aluminum pan located under the side. To dump the sample from the mixing chamber, open the front swing side. **DO NOT GRAB AT THE CONCENTRATE WITH YOUR HANDS.** Place the compounded sample on the counter top to cool and pull apart chunks ~1" to 2" in diameter. Put these chunks into a labeled paper bag and save for granulating. Concentrate can also be granulated while still warm or hot to decrease the handling. Gloves must be worn to protect the hands.
 - 8.4.1.19. Run the two remaining 1150 gm batches of Petrothene from Step 8.4.1.7 through the mixer to clean the sample chamber.
 - 8.4.1.20. Follow manufacturer's and site's operating procedures to shut down Batch Internal Mixer.
 - 8.4.1.21. Clean the feed chute, the chute back plate, mixing chamber, etc., as appropriate. **NOTE:** Ensure local site safety rules are met and followed before and while working on the mixer.
 - 8.4.1.22. Close mixer and turn off main air supply.
- 8.4.2. **USE OF THE GRANULATOR**
Final Size Reducing (Mixer Discharge)
The following may be used as a guide for the operation of the individual sites Granulator.

This equipment shreds or chops the lumps of concentrate into small pieces, which may be fed into the extruder. The cooled 70% concentrate which is fed into the granulator must not exceed ~2" in diameter. Larger pieces may cause an overload on the motor causing the equipment to shut down. When using warm concentrate, larger pieces may be used. The granulator must be opened after each series of samples is completed and cleaned thoroughly.

SAFETY:

Hearing protection may be required while operating this equipment. Follow individual site safety rules for requirements.

The main disconnect switch must be locked out or the machine disconnected when the granulating chamber is open.

The granulator knife blades are very sharp. Leather gloves must be worn when cleaning the granulator chamber or chute.

- 8.4.2.1. Follow the manufacturers and site operating procedure for start up and operation of the Granulator at your site.

- 8.4.2.2. Using a 1/4-inch hole diameter screen, granulate material into a suitable size plastic trash bag to help prevent contamination of the sample.
- 8.4.2.3. Check feed chute and collection drawer for cleanliness before granulating any concentrate.
- 8.4.2.4. Feed sample concentrate into granulator. When the grinding chamber is clear, turn off the granulator, remove the granulated concentrate and collection bag and identify the sample.
- 8.4.2.5. Granulate all samples in the series following the above procedure.
- 8.4.2.6. At the end of the series, clean the granulator chamber and chute. Use Leather gloves when working around the cutter blades.

NOTE: Follow local site safety rules.

- 8.4.2.7. Using a top loading balance, weigh 1000 ± 5 grams of each granulated concentrate into a labeled container. This 1000 gram sample will be the feed for the extruder. Weigh 1000 ± 5 grams of excess concentrate to be used as the initial purge for the extruder.

8.4.3. OPERATION OF THE EXTRUDER

The extruder is used to force the concentrate through a pack of screens. The barrel of the extruder is heated to melt the concentrate. The undispersed pigment, or grit, is retained on a 500 mesh screen, which is part of the pack of screens. This TiO_2 grit and/or other elemental material are measured on a Philips X-Ray Spectrometer. TiO_2 only, may be measured on the Texas Nuclear Elemental Analyzer.

SAFETY:

Use heavy leather gloves while handling hot material or equipment.

Be sure the extruder rupture disc is in place and positioned in the lower port on the bottom of the barrel.

Follow manufactures recommendations of 5500 - 7500 PSI for the rupture disc.

Maximum number of barrel ports not to exceed four and no less than two.

Protect wiring of barrel heaters and thermocouples from hot plastic should the rupture disc be activated.

"Cool-Press" instant cool packs must be readily available while the extruder equipment is in use.

The exhaust hood over the extruder must be in operation while testing is in progress.

Do not permit the hot waste extrudate, to contact any part of the body. It will tend to stick, causing severe burns.

Follow Manufacturer's and site start up and operating procedure for Operation of the Extruder at your site.

- 8.4.3.1. Energize unit, assuring that extruder speed control is at zero. (Caution: Energizing Extruder with speed control at a position other than zero can damage the extruder).

- 8.4.3.2. Turn on the feed throat cooling water. (Failure to maintain cooling water to the feed throat will cause the concentrate to plug the feed section of the extruder).
- 8.4.3.3. Set extruder and die heater zones to $400 \pm 10^{\circ}\text{F}$, ($204 \pm 5^{\circ}\text{C}$). Ensure all zones attain this temperature before rotating extruder screw.
- 8.4.3.4. Insert a breaker plate and pack of screens into the end of the extruder barrel. NOTE: Prepare a screen pack and breaker plate as follows; Breaker plate, 60 mesh, 60 mesh, 60 mesh, 500 mesh, 60 mesh and 30 mesh screens. The breaker plate should be to the outside and the 30 mesh screen to the inside next to the extruder barrel. Mount the die nut on the extruder and tighten with the spanner wrench. **CAUTION: HOT EQUIPMENT.**
- 8.4.3.5. Check that the temperatures of all zones are at $400 \pm 10^{\circ}\text{F}$. Add the purge concentrate to the feed hopper. Start the screw motor and raise the speed to between 100 and 120 rpm. Feed the entire purge concentrate through the extruder until the screw is visible. Then add approximately 1 liter of DuPont 20 series LDPE resin to flush the extruder.

NOTE: Quantity of clear LDPE needed to flush the extruder will vary with different models of extruders. For accurate results, sufficient LDPE must be used to have the polymer leaving the screenpack be basically clear.

NOTE: Extremely high screenpack results have been encountered on the first sample run through the extruder. Deposits of TiO_2 and LDPE will build up on the extruder screw as batches of material are processed. When the extruder is heating up, the thermal expansion of the screw tend to break this scale loose, causing high results in the first sample. If excessive screw build up and scale is a concern, initially use 500 grams of DPE6611 which is designed to clear this scale from the extruder. Always follow DPE6611 with Dupont 20 LDPE resin. At frequencies, determined by each site, the screw will need to be pulled from the unit and cleaned.

- 8.4.3.6. Decrease the screw speed to zero and stop unit.
- 8.4.3.7. Remove the breaker plate. **Caution: Hot Equipment.** Loosen the die nut with the spanner wrench. It may be necessary to turn the motor on, adjust the speed up slowly to force the breaker plate out of the extruder barrel. Clean the breaker plate using a spatula and stainless steel brush. Remove any excess DuPont 20 Series LDPE resin from the end of the extruder barrel by use of a spatula. Discard the pack of screens from the flushout concentrate.
- 8.4.3.8. Prepare a screen pack and breaker plate as in Step 8.4.3.4.
- 8.4.3.9. Insert the screen pack and breaker plate into the end of the extruder barrel, 30 mesh first.
- 8.4.3.10. Replace the die nut and tighten using the spanner wrench. **CAUTION: HOT EQUIPMENT.**
- 8.4.3.11. When all four temperature zones indicate they are at $400 \pm 10^{\circ}\text{F}$, pour a granulated sample or standard into the feed hopper.
- 8.4.3.12. Start the extruder by pushing the start button (make sure speed control is on "0" first) and adjust screw speed to between 100 and 120 rpm.

- 8.4.3.13. **The operator must keep the sample feeding.** The concentrate granules tend to bridge over. Use a polyethylene rod to move the sample into the extruder feed throat.
- 8.4.3.14. When the extruder screw becomes visible, add approximately one liter of DuPont 20 Series LDPE resin to the feed hopper. Continue to run the extruder until just before the screw becomes visible, adjust rpm to zero.
- 8.4.3.15. Remove the breaker plate and screen pack from the extruder barrel. **Caution Hot Equipment.** Again it may be necessary to start the extruder and adjust it to run slowly until the screw pushes the screen pack out of the extruder barrel. Reduce the speed to zero and turn the screw off.
- 8.4.3.16. Separate the breaker plate from the screen pack. **Caution Hot Equipment.** Scrape any excess resin from the breaker plate with a spatula.
- 8.4.3.17. Cool the screen pack and place it into an envelope. Mark the envelope with the identification of the sample.
- 8.4.3.18. Repeat Steps 8.4.3.8 - 17 for each sample and standard.
- 8.4.3.19. After all the samples have been extruded, turn off the power to the heaters and main unit. Place "HOT" sign on extruder.

8.4.4. BURNING THE SCREENS:

The pack of screens must be burned in order to remove the excess resin and separate the 500 mesh screen from the others for analysis.

SAFETY:

Very hot open flame, use care.

Work in a ventilation hood.

Hot, melted plastic will drip, avoid dripping on your body.

Do not have combustible materials where the flaming, melted drops of plastic, will contact them.

Spray screens in a hood; avoid spraying near the burner flame. Aerosol spray in contact with an open flame can cause a fire and/or explosion and serious injury. Depending on the size and configuration of the hood being used and local site safety rules, the Burner may or may not have to be extinguished when spraying screens.

Aerosol cans of spray must be stored in the paint cabinet in the solvent storage area or in an area assigned by local site safety rules.

- 8.4.4.1. Set up a blast burner in a lab hood. Place a tripod with a wire gauze pad over the burner.
- 8.4.4.2. Light the burner and establish a cherry-red flame.
- 8.4.4.3. Remove pack of screens from the envelope and using a pair of tongs, place it with 30 mesh screen (feed side) up on the "HOT" wire gauze. As the plastic is burned off, you will be able to remove the screens. Remove and discard the 30 and 60 mesh screens. Carefully remove the 500 mesh screen, keeping the feed side up. While holding it over the flame, burn away any excess resin. Always

work with the feed side of the screen up to avoid the loss of any pigment grit. Lay screen aside until cool, feed side up.

- 8.4.4.4. Spray the feed side of the 500 mesh screen with Krylon to adhere the pigment to the screen.

NOTE: Liquid latex may be used to coat the screens rather than spray, at the discretion of the individual site.

- 8.4.4.5. After the coating is dry, return the 500 mesh screen to its envelope for grading at a later time.

- 8.4.4.6. Follow Steps 8.4.4.3 - 5 for each sample and standard.

8.4.5. READING SCREENS:

- 8.4.5.1. Philips PW-1404 X-ray Spectrometer (or equivalent)

SAFETY:

Individual site requirements for radiation monitoring and personal protective equipment must be followed.

- 8.4.5.1.1. For specific operation of this equipment, consult the "X RAY SPECTROMETER" Standard Job Procedure.

- 8.4.5.1.2. Use your specific sites X-ray Program to obtain Ti.

- 8.4.5.1.3. Place the screen feed side down into the sample holder. Make sure the Mylar film, if used, is clean and intact. Center the screen and place the weight, if used, on top.

- 8.4.5.1.4. Analyze using your sites specific program. Record and log values for Ti per site requirements.

NOTE: A calibration must be developed for each individual x-ray fluorescence unit between its titanium fluorescence intensities and the assigned Screenpack Dispersion values of approved DTT calibration standards. These standards will be approved and controlled by the TTC. X-ray units should be recalibrated with the DTT standards whenever new tubes or sources are installed and/or as determined by the individual sites.

- 8.4.5.2. Texas Nuclear Elemental Analyzer:

NOTE: The Texas Nuclear Elemental Analyzer may be used (for TiO₂ analysis only) if a Phillips PW-1404 Spectrometer (or equivalent) is not available or is out of service, The unit must be calibrated to the TTC standards.

SAFETY:

This piece of equipment contains radioactive sources. It is shielded, but incorrect use could expose your eyes to radiation. The shutter operating button must not be depressed, except by lowering the labstand lid.

Site requirements for radiation monitoring and personal protective equipment must be followed.

- 8.4.5.2.1. The THRESHOLD and UPPER LEVEL controls should be set to manufacturer's specifications.

- 8.4.5.2.2. Push POWER on button - BATTERY meter should read above the line.

- 8.4.5.2.3. Wait at least one minute for instrument to stabilize.
- 8.4.5.2.4. Raise the labstand lid and place the instrument standard over the source. Lower the lid and latch the arm. Wait ten seconds for the instrument to restabilize. The shutter actuator pin should be completely depressed by the handle of the labstand lid.
- 8.4.5.2.5. Press the RESET button to set the counter to 0000.
- 8.4.5.2.6. Rotate the filter tray to the UP position.
- 8.4.5.2.7. Push COUNT UP switch up; counter starts counting.
- 8.4.5.2.8. At the end of the count, move the filter tray to the down position. Do not reset.
- 8.4.5.2.9. Push COUNT DOWN switch down. At the end of the count record the displayed net counts.
- 8.4.5.2.10. Repeat Steps 8.4.5.2.5 - 9 three times and average the results. The average should be within the limits stated on the instrument standard. If it is not, adjust the delta-T pot until it is, clockwise to raise and counter-clockwise to lower.
- 8.4.5.2.11. Place the screen, feed side down, into the aluminum sample holder. Place the aluminum plug on top of the screen.
- 8.4.5.2.12. Raise the labstand lid, place the sample holder with the sample over the source in the measuring head and close the labstand lid, making sure the actuator pin is completely depressed by the handle of the lid.
- 8.4.5.2.13. Press the RESET button to set the counter to 0000.
- 8.4.5.2.14. Rotate the filter tray to the UP position.
- 8.4.5.2.15. Push COUNT UP switch up; counter starts counting.
- 8.4.5.2.16. At the end of the up count, move the filter tray to the DOWN position. Do not reset.
- 8.4.5.2.17. Push COUNT DOWN switch down. At the end of the count record the displayed net counts.
- 8.4.5.2.18. Repeat Steps 8.4.5.2.13 - 17 twice more and average the results. The white line is the decimal point.
- 8.4.5.2.19. Using the calibration developed versus the DTT Calibration Standards, convert reading to Screenpack value.

8.5. Calculations
Not applicable. See section above, Reading Screens.

9. Quality Control

9.1. A reference standard will be run at a frequency to be determined by each site, and the results will be used for statistical process control, "CUSUM".

10. Comments

10.1. General

The procedure described in section 8.4 above will have to be written to be site and equipment specific for each individual site using this method.

10.2. Specific
None

11. References

11.1. General
None

11.2. Specific
T4400.583.02.WP Determination of 50% Screen Pack Dispersion 500 Mesh.

12. Appendix

12.1. Calculation of optimum Batch Internal Mixer (Banbury) charge. (P.A. Tooley March 25, 1993)
Revised 8/04 for 70% masterbatch.

12.2. Calculation of Stock Pressure or Containment Pressure and Fill Factor. (D.A. Holtzen March
29, 1993) Revised 8/04 for 70% masterbatch.

APPENDIX 1
CALCULATION OF OPTIMUM BATCH INTERNAL MIXER (BANBURY) CHARGE

The mixing chamber of the model "BR" Banbury has a charge volume of 1165 cc. To insure a good blend and proper fluxing of the resin, the final concentrate volume must equal the charge volume. If a mixer with a different charge volume is used, the following standard formula is used to solve for the required weight of ingredients.

Formula

$$\text{Total Weight} = \frac{\text{Charge Volume}}{\left(\frac{\text{Weight Fraction TiO}_2}{\text{Density TiO}_2} \right) + \left(\frac{\text{Weight Fraction LDPE}}{\text{Density LDPE}} \right)}$$

Example:

To prepare the 70 wt% concentrate of TiO₂ in NA-206-000 LDPE for a mixer with a charge volume of 1250 cc:

1. Insert the 1250 cc. into the formula as the charge volume.
2. Insert the 0.70 weight fraction (70 Wt%) into the formula for the Wt Fraction of TiO₂ and 0.3 for LDPE.
3. Insert the Specific Gravity of TiO₂ as 4.2 gm/cc and Specific Gravity of LDPE as 0.92 gm/cc.

Gives:

$$\text{Total Weight} = \frac{1250}{\left(\frac{0.7}{4.32} \right) + \left(\frac{0.3}{0.92} \right)} = 2561 \text{ g}$$

Solves to total weight = 2561 g.

Therefore the required weights of both the TiO₂ and LDPE are their Wt Fraction of the Total Weight or 0.70 x 2561 g. For the 1250 cc mixer charge volume, 1793 g of TiO₂ and 768 g of NA-206 LDPE are needed to run the 70 wt% Screen Pack Dispersion test.

APPENDIX 2 CALCULATION OF RAM AIR PRESSURE REQUIREMENTS

The Stock Pressure in the mixer needs to be maintained at about 55 psig in order for adequate mixing and fluxing to occur during processing. Stock pressure is set by the ram force. For each mixer, the required air cylinder pressure to the ram must be determined based on the area of the ram face in the chamber and the area of the air cylinder powering the ram.

$$\text{Required Air Pressure} = \frac{\text{Stock Pressure} \times \text{Ram Face Area}}{\text{Air Cylinder Area}}$$

For a Model "BR" Banbury, the Ram Face Area is 14.25 sq. inches. For a "BR" Banbury having a 5 inch diameter air cylinder, the air cylinder area would be 19.7 sq. inches. To determine required air pressure, the equation becomes:

$$\text{Required Air Pressure} = \frac{55 \text{ psig} \times 14.25 \text{ sq. in.}}{19.7 \text{ sq. in.}} = 40 \text{ psi}$$

If the mixer is equipped with a different size ram or air cylinder, the above equation should be used to determine the required air pressure to maintain the 55 psig stock pressure.

CALCULATION OF CHARGE VOLUME:

Most commercial internal mixers are run at fill factors of ~0.7, where fill factor is the ratio of the charge volume to the total mixer free volume. For the "BR" Banbury with 4 wing rotors, we have a free volume of 1574 cc. For this procedure, our charge volume is 1165 cc. Therefore, we run our "BR" Banbury mixers at a fill factor of 0.74

If a different style or size mixer is used, the required charge volume should be calculated based on that mixer's total free volume and a fill factor of 0.69 to 0.7. This calculated charge volume is used in Appendix 1 to set the weights needed of TiO₂ and NA-206 LDPE.