Safety: Wear the appropriate lab PPE when working in the LAB.

Purpose: FREE MOISTURE AND SIZE DETERMINATION - Wet Crystals

Procedure:
Introduction

For purposes of process development and product control it is desirable to determine the size of crystals being produced at any of a number of specific points in the production procedure. Very often this may involve crystals still in a slurry with mother liquor. Crystals and gross liquid can be separated to a degree by decantation or filtration but liquor will still adhere to the surface of the crystals. If drying is attempted at that point the liquor causes clumping which interferes with the classification of the crystals by means of the standard laboratory sieve set.

MgSO4 is virtually insoluble in acetone, and since acetone is hydrophyllic it is possible to remove surface moisture from the crystals by rinsing in that solvent. This procedure outlines the steps for achieving dry crystals in this manner.

A sample is obtained at the desired process location and taken to the laboratory. If the sample contains visible liquid brine the excess brine is discarded by decantation and vacuum filtration. A weighed amount of filtered crystals are then rinsed in acetone, the residual acetone allowed to evaporate, and the dry crystals weighed. Comparison with the weight before acetone gives the free moisture content of the sample. Crystals are then classified for size using the shaker sieve assembly.

Equipment.

Sampling Vessel - Plastic cup. 250 mL

Laboratory Spatula

Buchner Perforated Filter Funnel – 10 cm Ceramic

Vacuum Filter Flask – 200 mL Pyrex

Filter Paper – Baxter 90 mm. S/P Grade 361 Qualitative

Vacuum Pump – Boekel Hy-Vac 30 liter, Direct Druve. Capacity 26 " Hg.

Crystallizing Dish – 50x70 mm.

Laboratory Scale – Sartorius Basic

Acetone - S/P Grade

Plastic Ptpette – 1-3 mL Sampling

Sheet of office paper

Shaker Seive Assembly– CSC Scientific Shaker. USA Standard Screens. 12, 16, 20, 60, 120. mesh plus bottom pan.

Method

1. Obtain sample of product in process at the desired location using the 250 mL plastic laboratory cup **(no glass in the production area).** Take the sample to the laboratory immediately. If supernatant brine is present pour off and discard as much as possible without losing any crystals. Do this soon to avoid further crystal formation and growth.
2. Prepare the vacuum filter assembly and place the crystals in the cup on the filter paper.
3. Start the filter pump and stir the crystals lightly with the spatula until no more liquid drops from the tip of the funnel

   Crystals out of the centrifuge will not require Step 3.
4. Set the laboratory scale to O.
5. Place the Crystallizing dish on the scale and record the weight.
6. Using the spatula gently mix the crystals in the cup to assure that the mixture is homogeneous. and then transfer about 25 grams of crystals to the crystallizing dish. Record the weight.
7. Add enough Acetone to the dish to cover the crystals, about 30 mL.
8. Using the spatula stir the crystals and aceteone to assure that all of the crystals have been exposed.
9. Thrust the pipette to the bottom of the dish, pull out and discard as much of the acetone as possible.
10. Repeat steps 7, 8, and 9 two more times.
11. Dump most of the contents of the crystallizing dish on to the clean sheet of office paper and smooth the pile out to a thin layer to allow the residual acetone to evaporate. If a few crystals remain in the dish they will dry out there.
12. Allow the airing-off to proceed until the scent of acetone can no longer be detected. (A few minutes).
13. Check to see that the scale is reading zero.
14. Return the crystals on the paper to the crystallizing dish, place the dish on the scales and record the weight.
15. Subtract the weight at step 14 from the weight at step 5 and divide the difference by the weight at step 5. **This figure is the free moisture content of crystals out of the production centrifuge**.
16. Proceed to the Shaker Seive Assembly and determine the crystal size range.

TRAINING DOCUMENTATION

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Revision Number	Revision Date	Revision Author	Revision Description
00	3/28/2006	PLO	Original document
01	7/6/2009	PLO	Updated format and corrected spelling errors