

GILES CHEMICAL CORPORATION		
COMPANY PROCEDURE		
Standard Operating Procedure	Page : 1 of 4	Revision : Date : 3/27/06
Reviewed: Carl Mooney	Title: Crystal Size Determination - Wet Samples from Crystallizer	

QA-LAB-03

Safety: Wear safety glasses and/or goggles when working in the lab

Purpose or Objective: Crystal Size Determination - Wet Samples from Crystallizer

Procedure:

Background Information:

For purposes of product and process development 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 an aqueous matrix. Crystals and gross liquid can be separated by filtration but residual brine causes adherence and clumping if thermal drying is attempted, and the classification of crystals by means of the standard laboratory sieve is hampered.

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.

Scope :

A sample is drawn or obtained at the desired process location and taken to the laboratory. If the sample contains liquid brine the excess brine is discarded, and most of the remaining brine removed by vacuum filtration. The semi-dried crystals are then rinsed in acetone, the residual acetone allowed to evaporate, and the dry crystals classified for size using the shaker sieve assembly.

Equipment

Plastic Container of about 1000 ml. capacity
 2 - 1000 ml Pyrex Glass Beakers
 Weighing Balance -- B440 Sartorius
 Small Spatula
 Filter Funnel -- Porcelain Buchner 100 mm
 60 / 40 mesh inserts
 Filter Flask -- Pyrex Glass 500 - ml
 Tumble Dryer -- This is an in-house accessory assembled from locally obtained components (See working model)
 Small Chain -- 24" long
 Sieves and Sieve shaker -- CSC Meinzner #S1232 - 1
 Timer (for tumble dryer)
 Timer (for sieve shaker) -- S / P C6510 - 1 3 Channel
 Sheet of 8 1/2" x 11" office letter paper

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- 1 Separating crystals from mother liquor
 - a. Vacuum filter equipment is assembled using the 40 / 60 mesh inserts in place of filter paper in the filter funnel.
 - b. The vacuum pump is turned on and the heterogeneous mixture is poured into the filter funnel
(**No supernatant is to be sucked into the vacuum pump itself**)
 - c. After all supernatant has been removed the crystals are to be moved around the on the filter screen using the spatula to remove as much liquid as possible

(**the filtration process should continue for about five minutes**)

$$\frac{\text{Weight of Crystals}}{\text{Weight of Total Sample}} \times 100 \% = \text{Percent Population}$$

2. Drying crystals after separation of mother liquor
 - a) 50 grams of wet crystals are transferred to the tumble dryer
canister, along with the chain (for agitation)
 - b) The tumble dryer is completely assembled and the warm air source is swung into the operating position.
 - c) Using the installed timer, the apparatus is allowed to run for 15 to 20 minutes
 - d) When drying is complete, the chain is removed and crystals are transferred from the tumble dryer to the sieve shaker

(**completion of drying is evident by the chain being free of adhering**)

- e) the stack cover is placed on the top pan and tightened down
by means of the turn screws provided

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- f) The vibration mechanism is activated and allowed to run for two minutes, using the timer provided
- g) When vibration has stopped, remove top sieve pan, lid attached, invert pan, tap or scrap the screen lightly to remove lodged salt particles.
- h) The pan is then lifted from the cover and all the crystals will remain on the inverted cover
- I) Transfer the crystals to the paper and weigh
- j) The crystal size determination is reported as the percentage retained on each sieve and the bottom of the pan

$$\frac{\text{Crystal Weight on Each Mesh}}{\text{Total Weight of Sample}} \times 100 \% = \text{Percent Crystal Size}$$

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TRAINING DOCUMENTATION

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