



SUBJECT SILICATE DETECTION IN SULFATE SOL'NS  
Process Specifications

SUPERSEDED DATE 4/6/48

1. PURPOSE

One of the causes of holes in finished fluorescent screens has proven to be premature coagulation of silicate. This is due to the intermixing of the silicate binder and the sodium or potassium sulfate electrolyte previous to screen dispensing.

It is proposed to detect the presence of silicate in the purified sodium or potassium sulfate solutions (S108B, P69B, or P72B) immediately after its preparation according to a predetermined sampling plan as a period control. The method herein described is similarly applicable for checking stock solutions or at the dispensing operation.

Such silicate contamination may occur due to inadequately cleaned vessels or equipment or by human error in handling the respective solutions.

2. EQUIPMENT

- a. Erlenmeyer flask, 250 cc. capacity.
- b. Graduated cylinder suitable for measuring 20 cc. sample.
- c. Capped bottles fitted with medicine dropper (2 required).
- \*\*d. 1000-cc. Pyrex beaker.
- \*\*e. 50-cc. graduated cylinder.
- \*\*f. 1000-cc. volumetric measuring flask.
- \*\*g. Hot plate or bunsen burner.

3. MATERIALS

- a. Ammonium molybdate, 10% solution (A631 - reagent).
- b. Sulfuric acid, 50% solution by volume (S22 - reagent).
- \*\*c. Ammonium nitrate (A632 - reagent).
- \*\*d. Ammonium hydroxide (A9 - reagent).
- e. Blue litmus paper.

AMMONIUM HYDROXIDE HANDLING PRECAUTIONS - See S.N. 33-2-8A.

SULFURIC ACID HANDLING PRECAUTIONS - See S.N. 33-2-7C.

\*\* DANGER

\*\*Note:

- Prepare the 10% ammonium molybdate solution as follows:
- (1) Measure 600 cc. of distilled water into a 1000-cc. beaker.
  - (2) Add 15 cc. ammonium hydroxide.
  - (3) Add and dissolve 65 g. ammonium molybdate.
  - (4) Heat gently and add, while stirring, small portions of ammonium nitrate until 225 g. have been dissolved.
  - (5) Filter twice.
  - (6) Dilute to 1000 cc. in a 1000-cc. volumetric flask.

4. PROCEDURE

- a. Using clean graduated cylinder, draw a 20 cc. sample of the sulfate solution, after first rinsing the cylinder with the same material.
- b. Transfer 20 cc. to the clean, 250-cc Erlenmeyer flask.
- c. Add a piece of blue litmus, 1/2" square, to this alkaline sample.
- d. Add 5 drops of the sulfuric acid solution which will normally make the litmus paper turn red (indicating change to acid condition). The acid should be mixed by swirling. If the litmus does not turn red, add more sulfuric acid drop by drop to obtain the acid condition.

(Cont. on page 2.)

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★ CHANGE  
★★ ADDITION  
★★★ DELETION

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4. PROCEDURE (Cont.)

- e. Add 15 drops of the 10% ammonium molybdate solution.
- f. The presence of silicate is indicated by a yellow discoloration of the solution to any degree. A colorless solution conversely indicates absence of silicate in the solution. However, it is advisable occasionally to test both the reagents and the technique by deliberately adding a few cc. of silicate solution (10%) upon which the yellow discoloration should appear, provided the solution has remained acid as shown by the red litmus.

ENGINEERING SECTION  
STANDARDIZING

4-498-9-60 PCL11500-121/bwr

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