

Recrystallization of DAN

Health and Safety:

Wear nitrile gloves, laboratory coat and safety glasses. The process should be done with fume hood ventilation.

Equipment and Materials:

Equipment

1. 2000 mL beaker,
2. hot plate with magnetic stirring,
3. 3 inch magnetic bar (Fisher, 14-513-68),
4. Buchner funnel set (diameter size from 60mm to 100mm, eg, Fisher



- FB-966-F),
5. 2000 mL filter glass,
6. filter paper,
7. 2000 Erlenmeyer flask,
8. spatula
9. balance,
10. vials (Fisher, 03-339-22C) for aliquot
11. Aluminum foil for cover the vials.

Chemicals

1,5-diaminonaphthalene, D21200-100G from Sigma/Aldrich; Acetonitrile, HPLC grade; MilliQ water

Procedure

1. Prepare 2000 mL 90% ACN and Prepare 200 mL 70% ACN
2. For 100 gram DAN: pour 1500 mL of 90% ACN in a 2000 mL beaker with a magnetic stirring bar and then place the beaker on the hot plate, A large petri dish of the size to cover the beaker should be placed on the top of the beaker to condense most of the solvent. Heat up the 90% ACN to 70°C. (The petri dish generally is not efficient, so, 100mL or 150mL of ACN may need to add during the heating to compensate the lost solvent). Then carefully add the DAN into the 90% ACN, heat up the mixture until about 85°C to obtain the clear solution of DAN. Normally, the dissolution process takes about 2 hours.
3. Set the stirring motion at a very high speed. Then turn off the heating. Wrap up the flask with aluminum foil and leave the flask on the hot plate with no heating.
4. The fast stirring and slowly cool down process combine to give a slow crystallization of DAN. During this process, the crystals tend to have a very small size and contain minimum impurity. This process will take couple hours for the crystals to start to form, and overnight to complete.
5. Filter the crystals of DAN, rinsed with about 200mL of ice cold 70% ACN and dry in a hood with a dimmed light.
6. Repeat the above procedures one more time with the obtained crystals.
7. Store the aliquot of DAN under -20 to 80 °C.

Expected outcome/data

When at highest purity, DAN crystals are dark brown color, and will be almost white color if the crystal size is very small. The color of very fine crystals will slowly change from white to light white/brown.

Reference: please see any basic organic laboratory training book for the recrystallization procedure and mechanism.

Note: sometimes commercial available matrices have low quality, so recrystallization may have to be done for twice or three times to obtain highest quality possible.
