


Preparing Internal Standard Solution Containing 5mM DSS-d6 (IS2)		
S001		
Version: 1.0	Date: 25-Sep-2018	

Preparing Internal Standard Solution Containing 5mM DSS-d6 (IS2)


Purpose This procedure provides information on how to prepare 5 mM DSS-d6 solution in D₂O. This stock solution will be used to prepare internal standard (IS2).

(Materials)

Reagents	Supplies	Equipment
<ul style="list-style-type: none"> • 3-(Trimethylsilyl)-1-propanesulfonic acid-d6 sodium salt (DSS-d6) • Deuterium oxide (D₂O) • Sodium Azide (NaN₃) 	<ul style="list-style-type: none"> • 50 mL falcon tube • 100 mL volumetric flasks • Weighing paper/tray • 125 mL amber storage bottle • Spatula 	<ul style="list-style-type: none"> • Analytical balance • pH meter • Vortex

Procedure Record the lot numbers and other relevant tracking information about the reagents used in your laboratory notebook.

Step	Details
1.	Review the Material Safety Data Sheets for DSS, NaN ₃ and D ₂ O.
2.	Calibrate the analytical balance.
3.	Using a weighing boat, weigh $0.112 \pm 0.01\text{g}$ of DSS-d6 and $0.1 \pm 0.01\text{g}$ of sodium azide and quantitatively transfer into a 50 ml Falcon tube. Dissolve them in 30-40 ml D ₂ O. Rinse the weighing dish with D ₂ O into the tube. By mixing and turning upside down and back, make sure everything is completely dissolved. Use vortex or sonicator if needed.
4.	After thoroughly mixing, adjust the pH the solution between 6.5 and 7.5 by using 1M HCl and 1M NaOH to adjust the pH (lower and higher, respectively). NOTE: Due to the lack of salts in this solution there will be very little buffering effect. Do NOT add very much acid or base when adjusting the pH. Record the initial pH of the solution in your laboratory notebook. Record the amount of acid/base needed to adjust the pH of the solution in your laboratory notebook.
5.	Transfer the solution to a 100 ml volumetric flask. Add D ₂ O to bring the level of the flask ~ 1-2 cm below the mark. Re-check the pH and re-adjust if necessary
6.	Bring up to volume with D ₂ O (meniscus should be aligned with the mark when viewed directly at eye level).
7.	Stopper the flask and mix by turning upside down and back several times.
8.	Transfer the resulting solution into a 125mL amber storage bottle. Label with the following information: name, log book tracking number, concentration, date prepared, and initials of the individual who had prepared the solution.
9.	Parafilm the lid on the container and store at 2-8°C (in the refrigerator). This stock solution will be used to prepare internal standard (IS2).

Preparing Internal Standard Solution Containing 5mM DSS-d6 (IS2)		
S001		
Version: 1.0	Date: 25-Sep-2018	

Procedural Notes

1. The following calculation is used to determine the mass required:

m = mass
C = concentration
MW = molecular weight
V = total volume
P = Purity

2. The following calculation is used to determine the concentration:

m = mass
C = concentration
MW = molecular weight
V = total volume
P = Purity

References Not applicable

Related Documents

Weighing Compounds on the Mettler Toledo Analytical Balance, LP005, current version
Calibrating the Mettler Toledo Analytical Balance, LP008, current version
Tracking Solution Preparation, LP038, current version

SOP prepared by: Nazanin Assempour

SOP approved by: Neil Taylor

