

Swim just found these posted over at scimadness as a revision to that sodium eth sulfate synth both ripped from the hive swim believes maybe an older bee can clarify as well as give either one a thumbs up or thumbs down based on experience

The following was added in the original article by moo.

Preparation of Nitroethane
by Gerard Desseigne & Henri Giral

The reaction flask is charged with:

26.5 g (0.0625 * 3moles) of tech grade K₂CO₃ dissolved in 137 g of water

320 g of 97% tech sodium nitrite

6 ml of cetyl-oleic alcohol or oleic alcoholm(antifoaming agent)

Vol of the mixture is about 420 mls

The mixture is heated on a bath at 130C with stirring. The addition funnel is charged with 750 ml of an aqueous solution of 444 g (3moles) sodium ethyl sulfate. The solution is added to the reaction mixture during 50-60 mins at a suitable rate to keep the reaction mixture at 125-130C, with vigorous stirring.

The distillation of the nitroethane begins when the addition is started.

When addition of NaEtSO₄ is complete 100 mls of water is added during 10 mins.

The distillate separates into 2 layers. Can be distilled at 760mmHg with a distillation column. Distillation starts at 30C. All below 74C is discarded. Temp should settle at 87C & distillation is stopped at 99.8C.

Yield of nitroethane:96 g (1.28 moles), 42.6% based on NaEtSO₄

and this was posted by Evil_Lurker:

Nitroethane, that substance which is so cheap at any chem supply house, but so damn hard to get. Making the stuff ain't very much easier than buying it from the recipes on the net.

The two OTC easiest ways, using sodium ethyl sulfate and diethyl sulfate as reagents are very hard and dangerous to manufacture. The problem lies in the water molecule the EtOH creates when it forms the ester. The apparatus looks plain scary to operate, and creates diethyl ether as a side product and the yields suck ass!

But now that problem has been fixed. Sodium ethyl sulfate can be easily prepared in the home laboratory from common sodium bisulfate found in pool pH down and 95% pure grain alcohol, and baking soda or sodium carbonate found in pH up.

113 parts by weight of sodium bisulfate and 20 parts by weight of pure grain alcohol are combined in a flask and brought to boiling upon which time the sodium bisulfate crystals disappear and sodium sulfate forms.

When the reaction has been completed, the flask was is immersed in an ice bath and vigorously stirred until the temperature rapidly goes below 32.3C.

The formed sodium sulfate is thus filtered out leaving an anhydrous ethyl hydrogen sulfate/ethanol mix.

The excess ethanol is distilled off, and the ethyl hydrogen sulfate is neutralized with an appropriate quantity of sodium carbonate leaving sodium ethyl sulfate.

Simple... thats all there is to it! The secret to how it works is the excess

