

# Re: Gravimetric Analysis.

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- *From:* Uncle Al <[UncleAl0@xxxxxxxxxxxxxx](mailto:UncleAl0@xxxxxxxxxxxxxx)>
  - *Date:* Fri, 01 Jul 2005 09:52:04 -0700
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rain.on.moon@xxxxxxxxxx wrote:

>

> Dear All,

> I am in the middle of nowhere. I have had a reaction to synthesize  
> tributyl phosphate.

You buy it, \$28/liter.

> The reactants were phosphorus oxychloride, pyridine  
> and n-butanol. Now that the reaction is complete. I want to get the  
> organic layer separated in the separating funnel analysed. I want to  
> know how much quantities of tributyl phosphate, dibutyl phosphate,  
> monobutyl phosphate, water and butanol are present in the separated  
> organic layer.

Distill the  $\text{POCl}_3$  (aspirator vacuum) so it is clean. What precautions must you take during and after the distillation? Excess \*dry\* butanol (93 C azeotrope, 43% water) and do the reaction in ether so py.HCl precipitates to make workup easier. Filter; wash the filter cake. Rotovap.

TLC to monitor how things are going. Partially esterified phosphates will have some detergent character. Good luck, buddy.

> I have searched for all th available methods of analysis  
> but they are qualitative. I want to know if there is any method that  
> gives us the percentage of the above mentioned entities. Any sort of  
> help will be appreciated.

Now you're worried how things turned out? The time to worry is before you do it. If you do it right you don't worry about non-stoichiometric reaction channels – because you have been pro-active in preventing them them. NMR, proton and P-31.

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Uncle Al

<http://www.mazepath.com/uncleal/>

(Toxic URL! Unsafe for children and most mammals)

<http://www.mazepath.com/uncleal/qz.pdf>

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