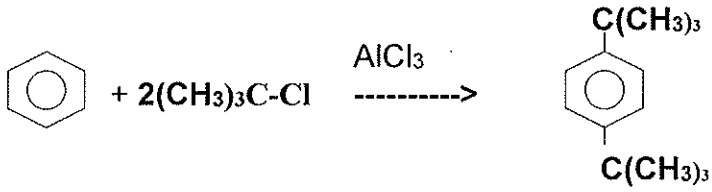
Sample lab notebook writeup :

Synthesis of 1,4-Di-tertbutylbenzene via Friedel-Crafts Alkylation

I. M. Student March 12, 2022

1.1. Purpose:

1,4-Di-tertbutylbenzene will be prepared from benzene via the Friedel-Crafts synthetic pathway shown below:



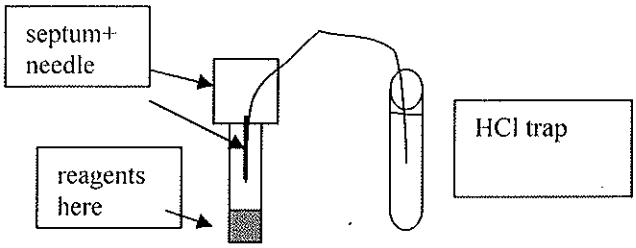
Write out the overall synthesis reaction in equation form. Use full sentences. Include name of mechanism governing reaction.

1.2 Procedure

NEED THIS HEADER !

Running the Reaction

Because of the exothermic nature of the reaction,1 ml (9 mmol) t-butyl chloride and 0.5 mL (4.5 mmol) dry benzene were mixed first and cooled to ice bath temperatures **prior** to adding a small quantity (~0.06 g) of $AlCl_3$. The reaction set-up used s illustrated in Figure 37.1 , page 401 of the handout (Taken from Williamson's "***Macro and Microscale Organic Experiments***" Heath Publishing (1989)). It is also sketched below The reaction started within a few minutes following addition of the $AlCl_3$. The reaction exhibited vigorous bubbling and evidence of HCl evolution.



$AlCl_3 + (CH_3)_3CCl + C_6H_6$

*Divide up **Procedure** into `**Running the Reaction**' (which describes how target compound is made. Include specific amounts used in g or ml and mmole) and **Workup** (which describes how the target is isolated and characterized.) Make sure wherever possible, to connect procedural steps with mechanisms. Use past tense and described what you actually did-not what the lab manual says you should have done.*

The initial mono alkylation produced an oily product. As the reaction proceeded the second substitution to make the ditert butyl product was signaled by precipitation of fine white dendrites . The success of the reaction hinged on judicious addition of the $AlCl_3$ Lewis acid. We had to try the reaction twice. There should be continuous bubbling for much of the reaction. The bubbling means HCl is being evolved, though wet benzene can produce a side reaction as shown below. Our first attempt was carried out with undried benzene. Our second attempt used benzene dried over $MgSO_4$ for 30 minutes. Drying the benzene was critical.

$3H_2O + AlCl_3 \rightarrow 3HCl + Al(OH)_3$ side reaction

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Workup

Workup of the product occurred through a series of solvent extractions. Once the white solid product formed and the reaction was allowed to run its course, 5 mL water was added to the reaction mix to extract residual inorganic components such as $AlCl_3$ and any HCl that remained in the mixture . The aforementioned mix was then washed three times with ~ 1 mL of ether, a solvent in which the product dissolvd easily. The ether extractions were combined in a large test tube. The remaining water-based mixture was discarded.

The ether extraction was dried over a small amount of $MgSO_4$, which was allowed to settle to the bottom of the testtube, after which the ether solution was transferred to a new, clean, dry testtube. The ether was allowed to evaporate-either by allowing it to stand overnight, or by gently blowing air over the surface of the testtube.

The resulting crude product (which appeared as a semi-solid, oily mass) was recrystallized by adding a minimum volume (0.4-0.5 mL) of methanol that re-dissolved it The resulting solution iwas finally cooled to ice bath temperatures to allow the purified crystals to form. The methanol was removed with a disposable pipet.

The final, recrystallized product weighed 0.47 grams (~ 57 % yield). Both the mp ,GC & IR spectrum were recorded.

1.3.) Table of Pertinent Material Properties

compound	CRC ref	mp, °C	bp,°C	d(g/mL)	grams/(mL)	mmoles	notes
$AlCl_3$ (MW=133)	20 p4-37	190	na		0.06 (a pinch)		reacts with water **catalyst for rxn**
benzene (MW=78)	867 p3-26	5.5	80	0.8765^{20}	(0.5 mL)	5.6	**substrate**
t-butyl chloride (MW=92.6)	9863 p3-270	-26	51	0.8420^{20}	(1.0 mL)	9.1	**limiting reagent** (2:1 vs benzene)
ether (MW=74.12)	5608 p3-156	-116	34.5	0.7138^{20}	(~5 mL)		**solvent extracts product**
methanol (MW=32)	7581 p 3-208	-97.6	64.6	0.7914^{20}	(~1 mL)		**recrystallizes product**
tert-butylbenzene (MW=134)	1459 p3-42	-58	169	0.8665			**intermediate** (sol in ace, ether)
1,4-Di-tertbutyl benzene (MW=190)	990 p3-29	79.5	238	0.985	~0.87 g	4.55	**desired product @ 100% yield vs limiting reagent sol in et, etoH,**

***76th edition**

You will need to predict the grams or mL of product at 100% based on limiting reagent. (t- butyl chloride limits here)

1.4) Results

Grams of product: 0.48
% Yield: {0.48/0.84 (theory based on t-butyl chloride)} * 100 = 57.1 %
Observed mp 76-78°C
appearance white, dendritic crystals

Table of Observed IR bands (in CCl_4) {see attached spectrum)
(see also, attached IR)

Observed for synthesized material		Observed for reference 1,4 ditertbutyl benzene	
cm^{-1}	**band shape**	**cm^{-1}**	**bandshape**
3050	mw, sharp	3045	weak, sharp
2950-2800	s, sharp multiplets	2945-2800	s, sharp, multiplets
2100-2220	vw, multiplet	2100-2220	vvw multiplets
1600	mw	1601	m singlet
1510,1470	med strong, sharp doublets	1515,1485	ms, sharp doublet

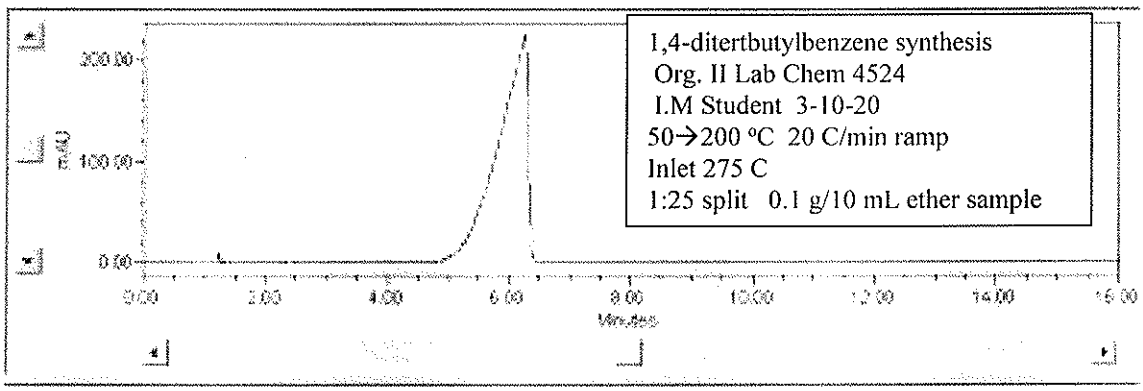
Table of IR trace conditions...as in Instrumentation lab

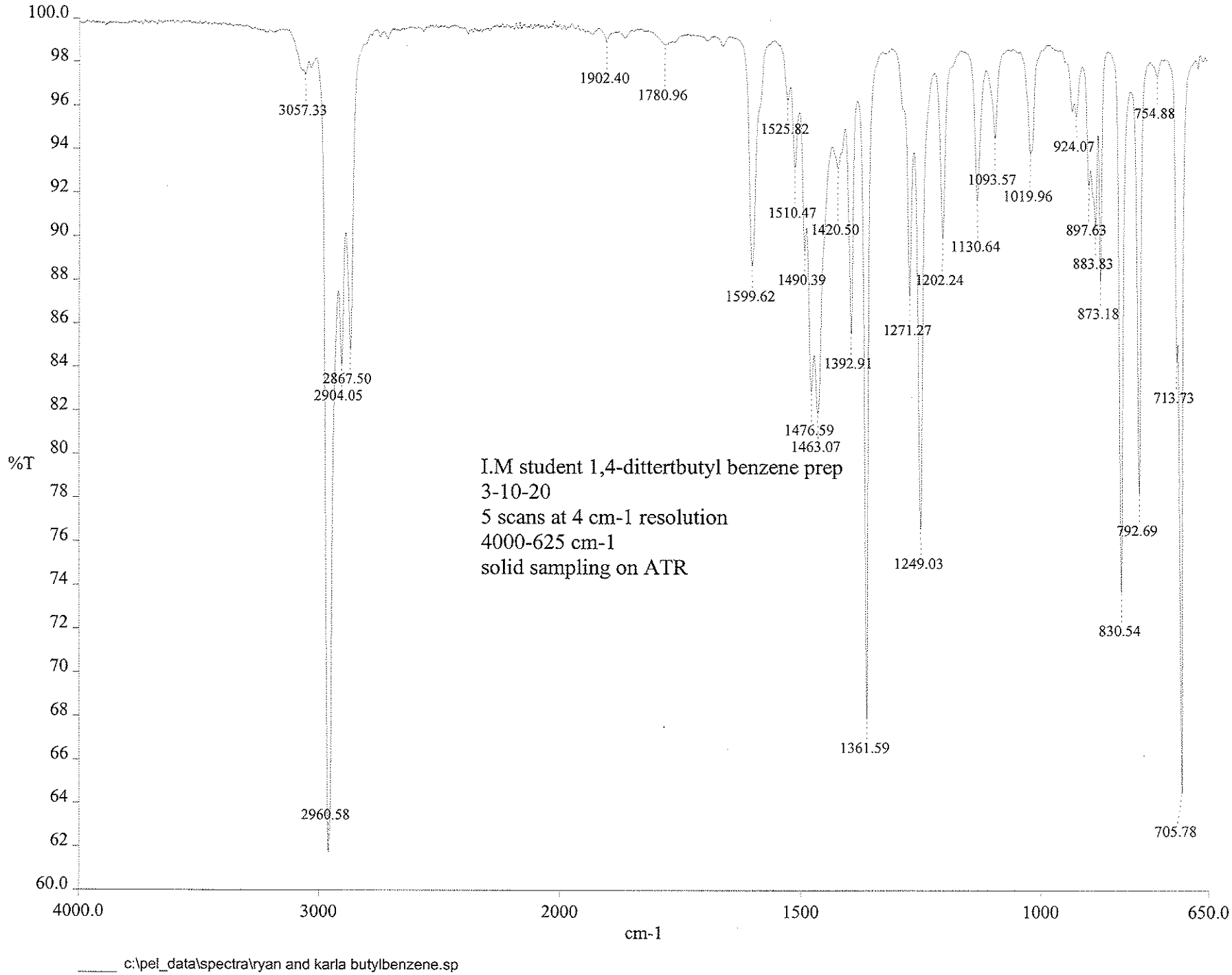
Make tables of main IR bands, GC peaks and peaks as we did in Instrumentation lab and Practiucm. Don't just refer the reader to the spectra and chromatograms without analysis.

Table of observed GC results

Sample	t_r(min)	peak area	
Reference 1,4-ditertbutyl benzene (1000 ppm in ether/wet needle)	6.01	4.0 uA*s	
Synthetic product (0.1 g/10 mL ether)	6.09	6.8	file C☺HPCHEM\1\DATA\CHEM4524\ORGSYN0210.D

Table of GC run conditions ...as in Instrumentation lab annotated directly on GC plot

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I.M student 1,4-dittertbutyl benzene prep
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5 scans at 4 cm-1 resolution
4000-625 cm-1
solid sampling on ATR
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