8-2: Benzene Nitration – 2

For this assignment, the target compound that you should synthesize is **1-methyl-4-nitro-benzene.** This is an electrophilic aromatic substitution reaction. Examine the product carefully and determine the substitution pattern. Which group will already be present in the substrate? Keep in mind the mechanism and how that will control the selectivity of the process. Remember, you can easily separate ortho and para isomers.

Synthesis Procedures

1. Start *Virtual ChemLab Organic* and select *Benzene Nitration – 2* from the list of assignments. After entering the synthesis laboratory, use the available reagents on the stockroom shelf and identify the appropriate starting materials required to synthesize the target compound and add them to the round bottom flask. Select the appropriate solvent and drag the flask to the *Stir Plate* on the lab bench.

2. The round bottom flask containing the starting materials should now be on the stir plate, and the contents of the flask should be visible on the chalkboard. From the group of reagents found on the lab bench, select the correct reagent to synthesize the target compound and add it to the flask on the stir plate. Now attach the heater, condenser, and N_2 gas to the round bottom flask so the reaction mixture can be heated.

3. Start the reaction by clicking on the *Stir* button on the front of the stir plate. You should be able to observe the reaction mixture stirring in the flask. Monitor the progress of the reaction using TLC measurements as necessary until the product has formed and the starting materials have been consumed (if you have not previously completed activity 1-1: Using Thin Layer Chromatography, please see the note at the bottom of that assignment regarding TLC in Beyond Labz). You can advance the laboratory time using the clock on the wall. With the electronic lab book open (click on the lab book on the stockroom counter), you can also save your TLC plates by clicking *Save* on the TLC window.

4. When the reaction is complete, "work up" your reaction by doing a separatory funnel extraction. Drag and drop the separatory funnel on the flask and then add the appropriate solvent to the funnel. Remember that the addition of any aqueous solvent also adds diethyl ether, although this is not shown (see note at the bottom of assignment 1-2: Performing a Separatory Funnel Extraction). Either the organic or the aqueous layer can be removed by clicking and dragging it to the bench. Your target compound should be in one of these layers. The other layer can be discarded into the red bin.

5. The layer in the round bottom flask may contain multiple products. To separate them, you must carry out a distillation. Drag the flask back to the stir plate. Drag and drop the distillation apparatus onto the flask and attach flowing N_2. Start the distillation by clicking on the *Stir* button. The temperature of the distillation flask can be monitored by hovering your mouse over the thermometer. As the temperature increases, products will evaporate and then distill into the collection flask at the back of the apparatus according to their boiling points. Products that are not needed can be discarded until the desired product is isolated.

   List the starting materials, solvent, reagent, and products formed: _____

   _____

   How long did it take to finish the reaction? _____

   What are the TLC values (R_f) for (a) Starting Materials: _____ (b) Products: _____

Write a mechanism for this reaction:

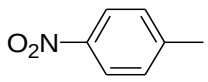
FTIR and NMR Spectra

After completing a reaction and working up the products, it is still necessary to confirm that the correct product was formed. The most common tools used for this analysis are Fourier Transform Infrared (FTIR) and Nuclear Magnetic Resonance (NMR) spectroscopy. In the virtual laboratory, ^{1}H and ^{13}C NMR spectra are available. Details on interpreting FTIR and NMR spectra are found in your textbook. Your instructor may or may not ask you to perform this section depending on how your class is structured.

6. To collect an FTIR spectrum of your product, click on the FTIR spectrometer located to the right of the lab bench and drag the salt plate icon to the flask on the lab bench. A window containing the FTIR spectrum for your product should now open. Identify the relevant peaks in the FTIR spectrum and record the position and associated functional group for each in the FTIR table below. The FTIR spectrum can also be saved to the lab book for later analysis.

FTIR **List position (cm^{-1}) & functional group**	4.
1.	5.
2.	6.
3.	7.

7. To collect a ^{1}H NMR spectrum of your product, click on the NMR magnet and drag the NMR sample tube to the flask on the lab bench. A window containing the NMR spectrum for your product should now open. You can zoom into various portions of the NMR spectrum by clicking and dragging over the desired area. The *Zoom Out* button is used to zoom back out to view the full spectrum. Identify all of the peaks in the NMR spectrum and record the chemical shift, the splitting, and the number of hydrogens for each peak in the NMR table below. The NMR spectrum can also be saved to the lab book for later analysis. If necessary to confirm the structure of your product, you can measure the ^{13}C NMR for the product and record the chemical shifts for the peaks. Mass spectrometry is also available if needed.

^{1}H NMR Structure:  1-Methyl-4-nitro-benzene	**Peak**	**Chemical Shift (δ)**	**Multiplicity†**	**H‡**	**Peak**	**Chemical Shift (δ)**	**Multiplicity†**	**H‡**
	1				7			
	2				8			
	3				9			
	4				10			
	5				11			
	6				12			

† Specify the multiplicity as a singlet (s), doublet (d), triplet (t), quartet (q), or multiplet (m).
‡ Specify the number of hydrogens associated with each peak.

8. *Do the FTIR and NMR spectra you measured and recorded in the tables above confirm that you synthesized the assigned target compound? Explain.* _____

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