**Materials and Methods**

Protocol was titled *Synthesis and application of a radical trapping agent* was obtained from CHEM 3880-A1. For the synthesis of N-Benzylidene-tert-butylamine N-Oxide (2) in a 50 mL Erlenmeyer flask along with a flea sized stir bar 0.91 mL of N-tert-buyl benzylamine , 0.07 g of Na2 WO42H2O and 10 mL of methanol was placed. The solution was cooled in an ice bath and 1.5 mL of 30% aqueous hydrogen peroxide was added. The mixture was removed from the ice bath and was left to stir for one hour on a stir plate. Upon completion of an hour, the flask was stoppered and it was placed in our draw until the next lab period 10/24/13. At the next lab period the mixture was poured into a 25 mL round bottom flask and placed on the rotary evaporator to dry. Once dried 25 mL of methylene chloride was added to it and then was poured into a separatory funnel. It was then washed twice with 10 mL portions of saturated aqueous sodium chloride. Then the mixture was separated and the bottom layer was collected in a 25 mL Erlenmeyer flask. Then the methylene chloride solution was dried over anhydrous sodium sulfate. The drying agent was removed by filtration and the methylene chloride was evaporated on a hot plate. The residue crystallized upon cooling and using a glass rod to scratch the bottom of the flask. The product was recrystallized for a small amount of hexane. The product was collected by filtration and the dry product was weighed to be 0.6010 g. Melting point was taken and found to be 72.5-75.0 and H NMR was taken in Chloroform. Once our nitrone 2 was made we placed 10 mg of nitrone and 15mg of benzoyl peroxide in a vial and added 1 mL of benzene and then carefully sparged the solution with nitrogen for 3 minutes. Then about 0.5 mL of the solution was transferred to a serum-capped, nitrogen flushed ESR sample tube. The tube was heated in a 45 degree Celsius water bath for 5 minutes then the ESR spectrum was immediately taken.

**Results**

An 1H-NMR spectrum of the compound was run in deuterated chloroform (CDCl3). An ESR spectrum of the compound was run. The 1H-NMR and ESR spectrum peaks are tabulated in table I. The 1H-NMR and ESR spectrum integrated values are tabulated in table II. The melting point was determined to be 72.5 – 75.0oC.

Table I. Peaks of 1H-NMR in chloroform and ESR

|  |  |  |
| --- | --- | --- |
|  | 1H-NMR (ppm) | ESR |
|  | 8.25, 7.60, 7.40, 7.25, 5.25, 3.90, 3.75, 3.50, 1.60, 1.50, 1.25, 1.00 |  |
|  |  |  |

Table II. Integration values of 1H-NMR in chloroform and ESR.

|  |  |  |
| --- | --- | --- |
|  | 1H-NMR | ESR |
|  | 1.00, 0.43, 1.46, 0.61, 9.16, 4.53, 5.56, 7.43, 14.56, 4.69 |  |
|  |  |  |

Peak assignments: 1.60 (9H, tBu–H), 7.25 (m, 3H, Ph–H), 7.60 (1H, C–H), 8. 25 (2H, Ph–H). These compare well with the literature values of 1.62, 7.35, 7.55, and 8.29 (Give the Andrade reference here). The other peaks are from solvent and perhaps unreacted reactants – 7.25 (CDCl3), 1.00 (TMS). If you want to assign the other non-product peaks, look at the NMR of the reactants and see if they match up. The melting point from literature is 72oC and use the Andrade reference here as well.

**Have #1 and #4 listed below been answered in the information you provided**

**1.** **Calculation of percent yield of nitrone 10** \_\_\_\_\_

2. m. p. of nitrone (compare with literature value; incl. reference) 10 \_\_\_\_\_

H NMR spectrum\* of nitrone

3. quality of spectrum 10 \_\_\_\_\_

**4.** **assignment of resonances** 10 \_\_\_\_\_

5. comparison with literature spectrum (incl. reference) 5 \_\_\_\_\_

ESR spectra\*

6. quality of spectrum 10 \_\_\_\_\_

7. interpretation of spectrum

8. determination of hyperfine coupling constant values 10 \_\_\_\_\_

9. calculation of g value 10 \_\_\_\_\_

10. comparison of spectral parameters with literature data (incl. reference) 5 \_\_\_\_\_

Formatting and appearance of report 10 \_\_\_\_\_