

Experimental Chemistry II, CH 463 & 463H Poster Abstracts for 2009
Department of Chemistry – Oregon State University
June 3, 2009 1:30 – 3:30
Gilbert Addition 313

1. DESIGN AND CHARACTERIZATION OF 4-CHLORO-4'-METHOXYBENZOPHENONE TO DESCRIBE ITS PHOTOPHYSICAL PROPERTIES. **Blake Gerttula**, Department of Chemistry, Oregon State University, Corvallis, OR 97331

4-chloro-4'-methoxybenzophenone was synthesized via the Friedel-Crafts acylation with anisole and 4-chlorobenzoyl chloride as reagents and recovered with a 22% yield after recrystallization from isopropyl alcohol. The product was then characterized using ¹H and ¹³C NMR, melting point, MS, and IR. The photophysical parameters for 4-chloro-4'-methoxybenzophenone were tested in a non-polar solvent methylcyclohexane, and a more polar solvent, EPA. The oscillator strengths for similar transitions in the polar and the non-polar solvent indicate that the absorption and excitation transitions were equally allowed. However, for the T1-S0 emission the transition was more allowed in the non-polar than the more polar solvent.

2. PHOTOPHYSICAL PROCESSES; UV ABSORPTION SPECTROSCOPY, EMISSION, AND EXCITATION OF 4-CHLORO-4'-ETHOXYBENZOPHENONE IN METHYLCYCLOHEXANE AND EPA. **Jamie Muong**, Department of Chemistry, Oregon State University-Corvallis, Oregon 97331.

A quantitative solvent study was completed on 4-chloro-4'-ethoxybenzophenone in the UV to determine the photophysical parameters for the electronic transition states. Two solvents were used including a more polar solvent, EPA, and more non-polar solvent, methylcyclohexane. UV absorption spectroscopy was used to measure the wavelength and absorbance for both a concentrated and diluted solution for each solvent. Curve fitting using GRAMSAI software led to a value for the Integrated Absorption Coefficient (IAC). The IAC led to calculation of S0 - S2 and S0 - S3 for polar EPA, S0 - S1 for concentrated non-polar methylcyclohexane, and S0 - S2, S0-S3, and S0 - S4 for diluted nonpolar methylcyclohexane. Excitation from S0-S1 and emission from T1 - S0 and the lifetime of the emission was measured at 77K (LN2) for the concentrated methylcyclohexane solution. This information is presented on a Jablonski energy diagram. Both solvents showed a pi-pi* peak but the n-pi* peak was only present in the methylcyclohexane.

3. THE PHOTOCHEMICAL REDUCTION OF 4-METHYLBENZOPHENONE, **Shane W. Monares**, Corvallis, OR 97331

When exposed to high levels of ultraviolet radiation 4-methylbenzophenone forms ketyl radicals which react to form a benzopinacol. The aim of this experiment was to determine the quantum efficiency, or conversion efficiency, of MBP to its benzopinacol form. The quantum efficiency, Φ_{red} was found to be 0.0204 mol benzophenone reduced/mol photons absorbed. A qualitative experiment with a concentrated benzophenone solution in isopropyl alcohol appeared to have formed a benzopinacol after a seven day irradiation period.

4. CHARACTERIZATION AND ELUCIDATION OF (2,4-DIMETHYLPHENYL)(4-METHYLPHENYL)METHANONE. **Nicole R. Tanguileg**, Department of Chemistry, Oregon State University, Corvallis, OR 97331

The compound (2,4-dimethylphenyl)(4-methylphenyl)methanone is a rare compound and has been minimally characterized. This seeks to elucidate characteristics of (2,4-dimethylphenyl)(4-methylphenyl)methanone and show a unique and interesting way to synthesize this compound. Data such as clean ¹³C and ¹H NMR spectra will be shown to capture wholly the basic characteristics of the compound and 2D NMR will confirm the structure.

5. DETERMINATION OF THE PHOTOREDUCTION QUANTUM EFFICIENCY OF 4-FLUOROBENZOPHENONE **Ashley A.P. Fulleton** Oregon State University Department of Chemistry Corvallis, OR 97331

The photoreduction quantum efficiency was found of 4-fluorobenzophenone by radiation and testing of samples using IR. Four samples were exposed to mercury lamps for different periods of time. These samples were then tested with the IR to find the change in concentration to plot against number of photons exposed to. From this the photoreduction quantum efficiency was found to be 0.3226.

6. SYNTHESIS AND CHARACTERIZATION OF 4-CHLORO-4'-FLUOROBENZOPHENONE. **Christopher D. Schmitz**, Department of Chemistry, Oregon State University, Corvallis, OR 97331

Synthesis of 4-chloro-4'-fluorobenzophenone was achieved via Friedel-Crafts acylation, using chlorobenzene, 4-fluorobenzoyl chloride, and an aluminum chloride catalyst. Workup and purification of the product was done by steam distillation, separatory funnel extraction, rotovap, and finalized by recrystallization with petroleum ether. Purity of the product was confirmed initially by melting point test, which was at its known value of 114 °C. Analysis of 1D and 2D NMR (including COSY, HSQC, and HMBC), IR, and MS, confirmed the structure of the product.

7. SYNTHESIS AND CHARACTERIZATION OF 4-NITRO-4'-METHYLBENZOPHENONE. **Adam L. Silbernagel**, Department of Chemistry, Oregon State University-Corvallis, OR 97331

4-methyl-4'-nitrobenzophenone was produced via a Friedel-Crafts reaction using excess toluene and 4-nitrobenzoyl chloride. Recrystallization had to be done using a polar solvent (ethanol), and then recrystallized again with a non-polar solvent (DCM). The resulting product was pale, yellow needle shape crystals. The product was characterized with a melting point, thin layer chromatography, IR spectroscopy, NMR, and GC/MS. The final percent yield was 17%.

8. QUANTITATIVE PHOTOCHEMICAL REDUCTION OF 4-BROMOBENZOPHENONE. **KJ Wright**, Department of chemistry, Oregon State University, Corvallis, OR 97331.

4-bromobenzophenone prepared via Friedel Crafts acylation from benzoyl chloride and bromobenzene was quantitatively photoreduced. Samples were subjected to UV irradiation at 254 nm on a given time scale. The rate of the disappearance of the benzophenone was measured via infrared spectroscopy versus quantitative dilutions of 0.0550M. 4-bromobenzophenone in methylcyclohexane and the reduction efficiency will be presented.

9. CHARACTERIZATION OF 3,4,4'-TRIFLUOROBENZOPHENONE USING NMR. **M. Martin**, Department of Chemistry, 110 Gilbert Hall, Corvallis, OR, 97331.

3,4,4'-trifluorobenzophenone was synthesized using a Grignard reaction and characterized using ¹HNMR, ¹³CNMR, and ¹⁹FNMR. The three fluorines in the compound produce a splitting in the NMR spectra which complicate structure elucidation. The complications introduced by the fluorine splitting are overcome by the use of 2d coupling spectra.

10. CHARACTERIZATIONS OF 4-METHOXYBENZOPHENONE. **Nick A. Boyles***, Nathan Collett, Emile Firpo, Somnath Jana, Chris Pastorek and Johanna Schwartz, Oregon State University Gilbert Hall Addition, 2101 SW Campus Way Corvallis, OR 97330

Benzophenones are aromatic, diphenyl ketones that are important in organic synthesis, photochemistry and have been used in many commercial applications. Once the synthesis of the benzophenone was complete the correct structure of 4-methoxybenzophenone was determined through mass spectroscopy, IR and 2-Dimensional NMR spectroscopy including; HSQS, HMBC, COSY as well as proton and carbon NMR's. Through detailed mapping of the compound through these characterizations the structure was corroborated with the HyperChem software.

11. PHOSPHORESCENT PROPERTIES OF (4-BROMOPHENYL)(4-METHYLPHENYL)METHONE. **Joseph E. Price**, Department of Chemistry, Oregon State University, Corvallis OR 97331

The emission, excitation and lifetime for the phosphorescence of (4-bromophenyl)(4-methylphenyl)methone was analyzed using two different sampling methods. First it was dissolved in methylcyclohexane and frozen in liquid nitrogen and analyzed on the Aminco Bowman Series2. This revealed an emission lifetime of 4.94 ms. Second, the compound was dissolved in methmethacrylate which was polymerized using AIBN initiator and allowed to set in a dry-block heater and a vacuum oven to cure. The measured emission lifetime was 0.6 ms.

12. PURIFICATION AND CHARACTERIZATION OF 4-CHLOROBENZOPHENONE, **Jeremy Unrau** Department of Chemistry, Oregon State University.

The Friedel-Crafts Acylation involving the reaction of chlorobenzene, benzoyl chloride, and aluminum chloride produced the desired ketone, 4-chlorobenzophenone, along with the isomer 2-chlorobenzophenone. The isomers were separated through a mixed solvent recrystallization of ethanol/water, and the 4-chlorobenzophenone was further purified through a recrystallization in hexane. The characterization of the product included melting point, mass spectrometry, IR spectroscopy, and ¹H and ¹³C NMR (COSY, HSQC, and HMBC) compared against known literature values to confirm the structure of the benzophenone.

13. CHARACTERIZATION OF 4-CHLORO-4'-ETHYLBENZOPHENONE. **Marissa N. Katz**, Department of Chemistry, Oregon State University, Corvallis, OR 97331

In order to ensure that the product formed was 4-chloro-4'-ethylbenzophenone characterizations were performed. First, two melting temperatures were done to check purity and to compare to a reference value. Second, a ¹H NMR was run to see if the hydrogens corresponded to the target molecule. An infrared spectrum was taken to examine distinguishing peaks. Mass spectrometry was used to determine the molecular weight of the molecule as well as to see if the correct fragmentation occurred. ¹³C-NMR data was examined to determine the corresponding carbons as well as to second check the positions of the hydrogens.

14. SYNTHESIS AND CHARACTERIZATION OF 4-CHLORO-4'-METHYLBENZOPHENONE. **Nicole Jones**, Department of Chemistry, Oregon State University, Corvallis, OR 97331.

4-chloro-4'-methylbenzophenone was synthesized via Friedel-Crafts Acylation from 4-chlorobenzoyl chloride and toluene with the slow addition of AlCl₃ as the reaction catalyst and was heated to reflux until completion, determined by TLC, yielding 76.3% benzophenone product. After purification a melting point at 129 °C supported product purity. The synthesized product was then characterized further through analysis of infrared spectroscopy, 1D and 2D NMR and mass spectrometry, all confirming the intended product.

15. QUANTITATIVE PHOTOCHEMISTRY OF 4-BROMO-4'-CHLOROBENZOPHENONE. **Abby E. Floeter**, Department of Chemistry, Oregon State University, Corvallis OR 97330

The reduction of 4-bromo-4'-chlorobenzophenone into its respective benzopinacol, 1,2-(4-bromophenyl)-1,2-(4-chlorophenyl)ethanediol, was studied and the rate of photoreduction was determined. The solvent system used was 60% (v/v) isopropyl alcohol in acetonitrile. Glacial acetic acid was used as the reaction catalyst. A timed photoreactor experiment was performed on the prepared benzophenone samples and the reacted products were analyzed using quantitative infrared spectroscopy (IR) measurements. The photoreduction quantum efficiency, $\phi_{\text{Reduction}}$, was determined to be 0.36.

16. SOLVENT STUDY. **Jenaya Hoffman**, Department of Chemistry, Oregon State University, Corvallis OR 97330

Weak and strong electronic transitions of 4,4'-dibromobenzophenone were studied using a polar solvent and a non-polar solvent. When dissolved in a polar solvent a blue shift occurs for λ_{max} for the S₀ → S_{1,2} transitions and for the T₁ → S₀ transition in relation to the non-polar solvent.

17. SYNTHESIS OF 4,4'-DIFLUOROBENZOPHENONE BY FRIEDEL-CRAFTS ACYLATION AND CHARACTERIZATION BY NUCLEAR MAGNETIC RESONANCE AND INFRARED SPECTROSCOPY. **Ben D. Taucher**, Emile J. Firpo, Christine Pastorek. Chemistry Department, Oregon State University, 153 Gilbert Hall, Corvallis, OR 97331-4003

In a Friedel-Crafts acylation, 4-fluorobenzoyl chloride and fluorobenzene reacted with AlCl₃ in CS₂ solvent to produce 4,4'-difluorobenzophenone with a yield of 30.5%. Through characterization methods including IR, NMR, MS, and melting point determination, the product was determined pure.

18. SYNTHESIS AND COMPARISON OF EXCITED STATE MEASUREMENTS BY DOUBLE SOLVENT STUDY OF 4-FLUORO-4'-N-PROPYL BENZOPHENONE **N.K. O'Donnell**
Department of Chemistry, Oregon State University-Corvallis, Oregon 97331

The preparation of 4-fluoro-4'-n-propyl benzophenone was done by Friedel-Crafts acylation method using 4-fluorobenzoyl chloride and n-propylbenzene utilizing DCM as the solvent. Isomers were made along with an unknown that was isolated using flash chromatography on a silica gel column. Using the polar solvent EPA and non-polar solvent methylcyclohexane (MCH), UV absorbance, excitation, emission and lifetime data was acquired using a UV spectrophotometer and a luminescence spectrometer. The $n \pi^*$ and $\pi \pi^*$ transitions were observed for possible differences in transition energies.

19. SYNTHESIS AND CHARACTERIZATION OF 4-BROMO-4'-FLUOROBENZOPHENONE **Brian R. Barley**, Department of Chemistry, Oregon State University, Corvallis OR

The product 4-bromo-4'-fluorobenzophenone was synthesized via Friedel-Crafts Acylation. The reagents 4-bromobenzoyl chloride and fluorobenzene were reacted in the presence of heat and an aluminum chloride catalyst. The percent yield was 40.51% and its melting point was determined to be 110-111 $\hat{\text{A}}^{\circ}\text{C}$. Analysis of IR spectroscopy, mass spectrometry, HNMR, ^{13}C NMR, HSQC and HMBC are presented below supporting the formation of 4-bromo-4'-fluorobenzophenone.

20. A COMPARISON OF PHOSPHORESCENCE IN 4-FLUORO-4'-METHYLBENZOPHENONE IN A RIGID GLASS AT LOW TEMPERATURE AND IN POLYMETHMETHRACYLATE **Caitlin C. Rering**, Department of Chemistry, Oregon State University- Corvallis, OR 97330

The phosphorescence of 4-fluoro-4'-methylbenzophenone was analyzed with a fluorimeter using two methods: 1) The ketone was dissolved in methylcyclohexane and then frozen using liquid nitrogen to form a rigid, optically clear sample. 2) A polymethmethracylate (PMMA) complex was formed using methyl methacrylate and the initiator azobisisobutyronitrile (AIBN). Emission, excitation and lifetime analyses were collected using both methods. The agreement between the methodology was good, though the glass had a higher resolution.