

**Experimental Chemistry II, CH 463 & 463H**  
**“Chemistry in Action” ~ 25th annual Undergraduate Poster Session**  
**Friday, June 7, 2019 ~ GBAD 209**  
**Department of Chemistry ~ Oregon State University**

1. SYNTHETIC MYSTERY: UNKNOWN BYPRODUCTS OF FRIEDEL-CRAFTS ACYLATION. **T. J. E. Aldous**, Department of Chemistry, Oregon State University, Corvallis, Oregon 97331.

Synthesis of (4-chlorophenyl)(4-methoxyphenyl)methanone from chlorobenzene and 4-methoxybenzoyl chloride, using  $\text{AlCl}_3$  as a catalyst resulted in a mixture of three things: the product ketone and two unknowns. Determination of these two unknowns is of large interest because they represent unexpected products of various side reactions.

2. SYNTHESIS OF 4-ETHYL-4'-CHLOROBENZOPHENONE. **Evan Blaylock**, Department of Chemistry, Oregon State University, Corvallis, Oregon 97331. [blayloce@oregonstate.edu](mailto:blayloce@oregonstate.edu).

4-ethyl-4'-chlorobenzophenone was synthesized using ethylbenzene and 4-chlorobenzoyl chloride in a Friedel-Crafts acylation with aluminum chloride as an initiating catalyst. Exploitation of boiling point and solubility differences is used to purify the benzophenone.

3. PHOTOPHYSICAL STUDIES of (4-TERT BUTYLPHENYL) (4-CHLOROPHENYL) METHANONE BY SPECTROPHOTOMETRY. **Cindy Cedeno**, Department of Chemistry, Oregon State University, Corvallis, OR 97331.

Electronic transitions corresponding to  $n\text{-}\pi^*$  and  $\pi\text{-}\pi^*$  transitions of (4 tert butylphenyl) (4-chlorophenyl) methanone are studied via. Photophysical parameters are determined, as well as lifetime of emission.

4. OILING OUT: SOLVING A COMMON COMPLICATION IN THE RECRYSTALLIZATION OF (4-ETHOXYPHENYL)(PHENYL)METHANONE. **J.J. Conroy**. Department of Chemistry, Oregon State University- Corvallis, Oregon 97331. [Conroyjo@oregonstate.edu](mailto:Conroyjo@oregonstate.edu).

Attempts to recrystallize (4-ethoxyphenyl)(phenyl)methanone in an ethanol/water solvent system led to the formation of an oily substance rather than crystals. To resolve this issue, additional ethanol was added. Later analysis determined the melting point of the recrystallized product to be 38-41°C. In systems where the melting point of the product is lower than the boiling point of the solvent, oiling out is a common predicament that can often be solved with additional solvent.

5. SYNTHESIS. **Li Ding**, Oregon State University, Chemistry Department, 153 Gilbert Hall, Corvallis, OR 97331.

In this experiment, 4-Bromo-4-methoxybenzophenone was synthesized through Friedel craft reaction from ethoxy-benzene and 4-bromobenzyl chloride using aluminum chloride as a catalyst. The end product formed is impure; hence, an appropriate solvent is used for purification to obtain a pure 4-bromo-4-methoxybenzophenone crystal. An NMR test is then carried out to determine the purity of the crystals. The infrared (IR) spectrum showed that the crystal contained traces of by-products.

6. THE FORMATION AND THE ANALYSIS OF 4-FLUORO-4'-METHOXYBENZOPHENONE  
**Jessica A. Folliett**, Department of Chemistry, Oregon State University, Corvallis, OR 97331.

Synthesis of 4-fluoro-4'-methoxybenzophenone was attempted by means of Friedel-Crafts acylation using fluorobenzene and 4-methoxybenzoyl chloride as the reactants; however, the result was a mixture consisting mostly of 4-fluoro-4'-methoxybenzophenone (the desired product) and 4-fluoro-4'-hydroxybenzophenone. The mixture was inseparable due to near-identical melting points, so the yield had to be approximated by different means. The impurities within the mixture were pinned down by GCMS analysis and FTIR spectra. Overall, this synthetic method was determined to be inadequate for mass production of 4-fluoro-4'-methoxybenzophenone, as the results would not justify the cost.

7. PHOTOCHEMISTRY OF BENZOPHENONE DERIVATIVES: PHOTOREDUCTION OF (4-CHLOROPHENYL)(4-ETHOXYPHENYL)METHANONE. **Paige M. Foster**, Department of Chemistry, Oregon State University, Corvallis, Oregon 97331.

Benzophenone derivatives are widespread in research and applications. Essential to their interest is their photochemistry: the ability to generate reactive species upon UV irradiation. This experiment studied the photochemistry of the benzophenone derivative (4-chlorophenyl)(4-ethoxyphenyl)methanone, which has competing substituent effects in photoreduction. The appearance of light-absorbing transients, additional bands on TLCs, and the products predicted GC-MS fragmentation pattern suggest the production of the associated benzopinacol.

8. ANALYZING PINACOL FORMATION BY PHOTOREDUCTION OF 4-CHLORO-4'-T-BUTYL-BENZOPHENONE. **A.R. Frank**, Department of Chemistry, Oregon State University, Corvallis, OR 97331. franka@oregonstat.edu

4-chloro-4'-t-butyl-benzophenone was reduced in a photoreactor for three weeks. The crystals that formed were analyzed by thin-layer chromatography, IR spectroscopy, and gas chromatography-mass spectroscopy. TLC indicated the presence of at least two compounds other than the benzophenone. IR analysis indicated the loss of a ketone and presence of a hydroxyl group. GC-MS failed to provide conclusive data about the presence of a pinacol.

9. PHOTOCHEMICAL REDUCTION OF 4-METHOXYBENZOPHENONE TO 1,2-BIS(P-METHOXYPHENYL)-1,2-DIPHENYL-1,2-ETHANEDIOL. **Zachary Fried**. Department of Chemistry, Oregon State University, Corvallis, OR, 97331-4003

1,2-Bis(p-methoxyphenyl)-1,2-diphenyl-1,2-ethanediol has previously been synthesized from 4-methoxybenzophenone via iron and zinc catalysis in moderate yield at room temperature. This research sought to synthesize the product via a photochemical reaction with acetic acid catalyst. Completion of the reaction by this method was confirmed by FTIR and TLC results.

10. BENZOPINACOL FORMATION STUDY OF DISUBSTITUED DIPHENYL KETONE. **D.D. Fuller**, Department of Chemistry, Oregon State University, Oregon 97330.

The formation of Benzopinacol involves a photochemical radical reaction where UV light acts as a catalyst. The formation of a radical creates the possibility of many by-products. Presented herein, the product of the reaction of (4-ethylphenyl)(4'-fluorophenyl)methanone with UV light was analyzed via GC-MS and TLC. Benzopinacols are useful intermediates and catalysts in organic synthesis when properly obtained.

11. GCMS/FTIR ANALYSIS TO VERIFY THE SYNTHESIS OF A DI-SUBSTITUED BENZOPHENONE. **Alexander T. Green**, Department of Chemistry, Oregon State University, Corvallis Oregon 97331. [green.alexandert@gmail.com](mailto:green.alexandert@gmail.com)

Chemistry 463 students at Oregon State University were tasked synthesizing and characterizing a unique di-substituted benzophenone. GCMS (Gas Chromatography-Mass Spectrometry) and FTIR (Fourier-Transform InfraRed spectroscopy) are two analytical techniques that can be used to verify its synthesis. GCMS provides information about different substances that make up a sample, as well as the chemical makeup of each of these substances. FTIR provides information about a sample's functional groups. Together, they can be used to verify that synthesis resulted in a pure benzophenone with the intended substituents.

12. **Madeleine Marie Hatch**, Oregon State University, Chemistry Department, 153 Gilbert Hall, Corvallis, OR 97331.

13. DESIGNING A DISUBSTITUTED KETONE: 4-BROMO-4'-FLUOROBENZOPHENONE.

**Nicholas A. Hogan.** Department of Chemistry, Oregon State University, Corvallis Oregon 97331. [hogann@oregonstate.edu](mailto:hogann@oregonstate.edu)

4-bromo-4'-fluorobenzophenone was synthesized via Friedel Crafts reaction using a mixture of carbon disulfide (solvent), fluorobenzene (substrate), of 4-bromobenzoyl chloride (reagent) and of aluminum chloride (catalyst). Products were isolated through steam distillation and solvent extraction. The product was purified via recrystallization in boiling hexane yielding crystalline (87.6% yield). Characterized via MP 111°C, TLC: R<sub>f</sub> = 0.60 (hexane), IR mass spec: 278 (m/z), GCMS: 10.7 minute elution time, NMR 1H and 13C. Then photochemical UV studies were begun. Excited state phosphorescence measurements for absorptivity and work up of photoreduced pinacol from the benzophenone.

14. FRIEDEL-CRAFTS ACYLATION SYNTHESIS OF (4-BROMOPHENYL)(4-METHOXYPHENYL)METHANONE: A CASE STUDY ON A POOR NUCLEOPHILE AND ACYL CHLORIDE COMBINATION. **L. S. X. Huffman;** Department of Chemistry, Oregon State University, Corvallis, OR 97331. [lucysuxiaohuffman@gmail.com](mailto:lucysuxiaohuffman@gmail.com)

The attempted Friedel-Crafts acylation synthesis of (4-bromophenyl)(4-methoxyphenyl)methanone (4B4MM) is discussed, with a focus on side reactions and extraction as explanations of minimal yield. 4-methoxybenzoyl chloride proved too reactive in the beginning stages of synthesis and its combination with bromobenzene, a poor nucleophile, proved an ineffective method of creating 4B4MM. Probable side reactions made it difficult to extract the organic product. Additionally, multiple organic products were produced.

15. PHOTOCHEMICAL REDUCTION OF 4-CHLOROBENZOPHENONE TO BENZOPINACOL, 1,2-DIPHENYL-1,2-BIS-(4-CHLOROPHENYL)-ETHANEDIOL. **Michael Koester,** Department of Chemistry, Oregon State University, Corvallis, OR 97331.

A qualitative experiment of 4-chlorobenzophenone to a benzopinacol was studied through a photochemical reduction reaction which yielded 1,2-diphenyl-1,2-bis-(4-chlorophenyl)-ethanediol. The benzopinacol was characterized via melting point analysis (170-172°C), and Thin Layer Chromatography (TLC)(R<sub>f</sub>=0.86) to confirm identity of compound. 4-chlorobenzophenone was irradiated by UV light for 9 days causing the benzopinacol to form.

16. INTRIGUING PROPERTIES OF 4-CHLORO-4'-N-PROPYLBENZOPHENONE. **Kenneth Y Koga,** Department of Chemistry, Oregon State University

4-chloro-4'-n-propylbenzophenone currently has no commercial or industrial applications. However, the photophysical properties of the compound suggest that it may be integrated into processes or products as a photosensitizer or a photoinitiator.

17. SYNTHESIS AND CHARACTERIZATION OF 4-BROMO-4'-TERT-BUTYLBENZOPHENONE VIA FRIEDEL-CRAFTS ACYLATION REACTION. **Martin Liao**, Department of Chemistry, Oregon State University, Corvallis, OR 97331-4003, United States, [Liaom@oregonstate.edu](mailto:Liaom@oregonstate.edu)

4-Bromo-4'-tert-butylbenzophenone was synthesized via Friedel-Crafts Acylation reaction using AlCl<sub>3</sub> as the catalyst, excess bromobenzene as the Aromatic Substrate and 4-tert-butylbenzoyl chloride as the Acyl Chloride.

18. PHOTOPHYSICAL PARAMETERS FOR (4-FLUOROPHENYL)(4-PROPYLPHENYL)METHANONE. **Hari G. Menon**, Department of Chemistry, Oregon State University-Corvallis, Oregon 97331.

The values for the photophysical parameters for (4-fluorophenyl)(4-propylphenyl)methanone are not known. They will be presented here from the [phosphorescence] spectroscopy done on this compound.

19. FRIEDEL-CRAFTS AS AN ALTERNATIVE TO PALLADIUM CATALYZED SUZUKI CROSS-COUPLING FOR SYNTHESIZING HIGH PURITY BENZOPHENONES. **Alexi R. Renquist**, Department of Chemistry, Oregon State University-Corvallis, Oregon 97331

Friedel-crafts is used as an alternative to the expensive and generally low yield Suzuki method due to the complexity of the Suzuki reaction and opportunity for side reactions to occur when reaction conditions are not heavily controlled. Alternatively, Friedel-crafts has a much more straightforward reaction that is simpler and less expensive to perform, while still maintaining high purity (>90%). Due to the desired Benzophenone only having been synthesized via the Suzuki reaction in literature, a brief explanation of the conceived method will be presented among the characterizing proof of high purity.

20. STRUCTURAL CHARACTERIZATION OF 4-FLUORO-4'-METHOXYBENZOPHENONE BY NMR. **Tyler A. Sherwin**, Department of Chemistry, Oregon State University, Corvallis, OR. 97331. [Sherwint@Oregonstate.edu](mailto:Sherwint@Oregonstate.edu)

Benzophenones have many industrial applications from pharmaceuticals to cosmetics. Title molecule was synthesized via Friedel-Crafts acylation and characterized using IR, TLC, MP, GCMS, and 1&2D NMR. This poster focuses on assignment of aromatic carbons and hydrogens using NMR data.

21. SYNTHESIS AND CHARACTERIZATION OF (4-BROMOPHENYL)(4-TERT-BUTYLPHENYL)METHANONE. **Hannah R Smith**, Department of Chemistry, Oregon State University, Corvallis, Oregon, 97331 [smithh5@oregonstate.edu](mailto:smithh5@oregonstate.edu).

The purpose of this experiment was to synthesize and characterize (4-bromophenyl)(4-tert-butylphenyl)methanone. This benzophenone is useful in the process of creating organic light-emitting diodes (OLEDs). OLEDs is an ongoing subject of research to improve full color large display applications. The synthesis of this compound was difficult because its color was black. When recrystallizing, it became clear that the black coloring was from impurities, and that impurity is shown in the characterization of the compound from GC-MS, IR, and NMR. A

second benzophenone (4-chlorophenyl)(4-tert-butylphenyl)methanone was determined to be the contaminant.

22. CHARACTERIZATION OF SYNTHESIZED 4,4'-DIFLUOROBENZOPHENONE. **Nakayla A. Williamson**, Department of Chemistry, Oregon State University, Corvallis, OR 97331. [Willinak@oregonstate.edu](mailto:Willinak@oregonstate.edu)

Previously synthesized 4,4'-difluorobenzophenone was characterized and analyzed via CNMR, HNMR, HSQC, IR (C=O stretch  $1646\text{ cm}^{-1}$ , C=C stretches  $1590, 1500, \text{ and } 1408\text{ cm}^{-1}$ ), and GC-MS (218 g/mol) to confirm correct synthesis and determine purity.

23. ELECTRON IMPACT FRAGMENTATION OF 4-BROMO-4'-ETHYLBENZOPHENONE **Henry R. Wise**, Oregon State University Chemistry Department, 153 Gilbert Hall, Corvallis, OR 97331. [wisehe@oregonstate.edu](mailto:wisehe@oregonstate.edu)

4-bromo-4'-ethylbenzophenone was analyzed via gas chromatography electron ionization mass spectrometry. Fragments corresponding to peaks in the spectrum were proposed. The structures of the product ions were explored to propose reasoning for their relative abundances.

24. DETERMINATION OF PHOTOCHEMICAL REDUCTION PRODUCTS OF 4-CHLORO-4'-METHYLBENZOPHENONE USING GC-MS. **J. S. Yang**, Department of Chemistry, Oregon State University, Corvallis OR 97330. [yangiasm@oregonstate.edu](mailto:yangiasm@oregonstate.edu)

The photochemical reaction of 4-chloro-4'-methylbenzophenone and isopropyl alcohol yielded substituted tetraphenylethylene compounds. GC-MS data suggests that a majority of the compounds in solution were the starting benzophenone (RT=10.944 min, m/z=230), but the yellow reaction mixture signified reaction progress. The parent ion of the tetrasubstituted tetraphenylethylene was weakly detected (RT = 12.855 min, m/z=428.9). Other detected compounds were 4,4'-dimethylbiphenyl (RT= 9.370 min, m/z = 182), 4-chloro-4'-methylbiphenyl (RT= 9.730 min, m/z = 202) and 4,4'-dichlorobiphenyl (RT = 10.205 min, m/z = 222).

25. STRUCTURAL DETERMINATION OF 4-BROMOBENZOPHENONE BY GCMS. **Gunnar Thomas Resch**, Oregon State University, Chemistry Department, 153 Gilbert Hall, Corvallis, OR 97331.

4-Bromobenzophenone was synthesized in poor yield and poor quality. Multiple byproducts persisted through attempts at purification via recrystallization. Without access to column chromatography, NMR was messy and without use. GCMS was then used to determine the major product structure.