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“The palest ink will endure beyond the memories of man”

— Tan Twan Eng, *The Garden of Evening Mists*

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The Editors

Isotopologues Synthesis for KIE Studies

By Matthew Sayre

Lay Abstract

The utilization of isotopic labeling is still growing in its use among chemists; the use of hydrogen isotope labeling allows for direct comparisons between two molecules undergoing a catalyzed reaction. An understanding of how the molecules behave when undergoing catalysis can be used when designing medication. Thus the understanding and prediction of chemical behavior at the molecular level can lead to a number of scientific advances; such as catalyst synthesis, inferences in biochemical reactions, and even possible mathematical systems that can model these reactions. Overall the reactions described are commonly used in this isotopic labeling technique, allowing for multiple measurements of reactions of our choosing.

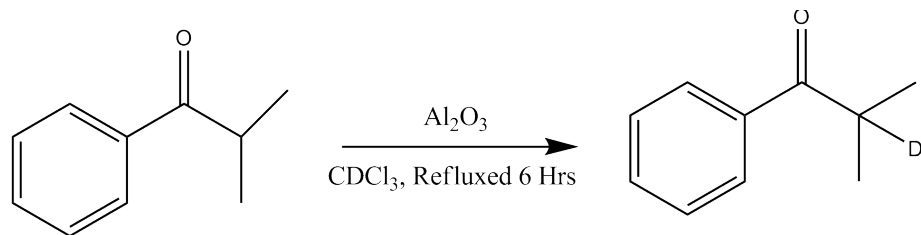
These reactions can help scientists gain an understanding of molecule interaction in a catalyst. Protein catalysts lower the amount of energy needed for a reaction to take place in the human body and are what are responsible for all physiological capabilities that humans possess, this understanding is the basis of drug synthesis in medicine and pharmacology; the ability to create a catalyst or a molecule and have its behavior predicted entirely to minimize side products produced in the reaction, and subsequently minimize side effects at a larger scale. Our research describes techniques on how we gain that complete understanding of certain catalysts, and the reactions I describe in the technical abstract are how we can probe those reaction pathways, using hydrogen isotopes.

Technical Abstract

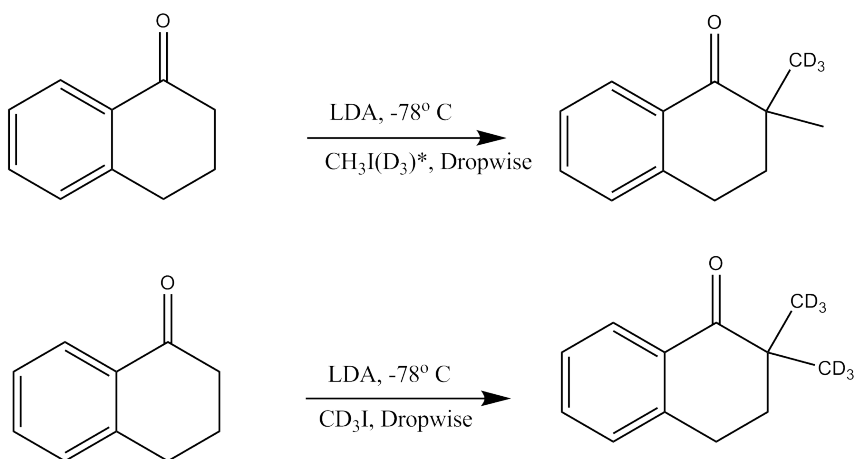
Isotopically labeled compounds are a key component to mechanistic studies in chemical research. They are used extensively in kinetic isotope effect (KIE) studies, which compare the reaction rates of isotopologues to elucidate the mechanism of a reaction. For example, KIE measurements can be used to study the physiochemical origins of enantioselectivity, transition state modeling or to determine the rate or product determining steps in a reaction. Here, a variety of deuteration reactions are used on various substituted alkyl aryl ketone substrates. We describe three reactions that are highly enantioselective and result in high yields, with little to no side products. The first is a deuteration at the alpha carbon position of isobutyrophenone with Al_2O_3 refluxed over six hours in deuterated chloroform (CDCl_3), with relatively no workup and a 98% yield (Scheme 1). Then lithium diisopropylamide (LDA), which is derived from n-butyllithium and isopropylamine, and d_3 -methyl iodide are used to alkylate alpha-tetralone and acetophenone derivatives in approximate 85% yield (Scheme 2,4). This synthesis can be used to produce D_3 and D_6 isotopologues via consecutive alkylations, with D_3 production utilizing non-deuterated iodomethane in the first alkylation step (Scheme 2,4). Finally, a Friedel-Crafts acylation is used to synthesize compounds with pre-isotopically labeled methyl substituents or alkyl group substituents on 2,5-dimethyl-isobutyrophenone with a 97% yield (Scheme 3). A key advantage to using isotopologues to probe reaction mechanisms is that the system is perturbed without changing the potential energy surface of the reaction. Isotopic perturbation is a powerful phenomenon which can be exploited to elucidate important physiochemical aspects of a reaction such as hyperconjugation, electrostatic effects, induction, steric repulsion and conformational and hybridization changes. These reaction rate-determining processes can be exploited to develop finely tuned catalysts, transition state analogs for increased drug effectiveness or explain certain aspects of quantum mechanics.

Figures

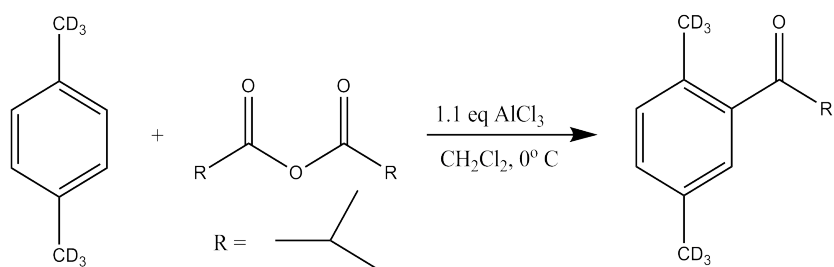
Scheme 1.



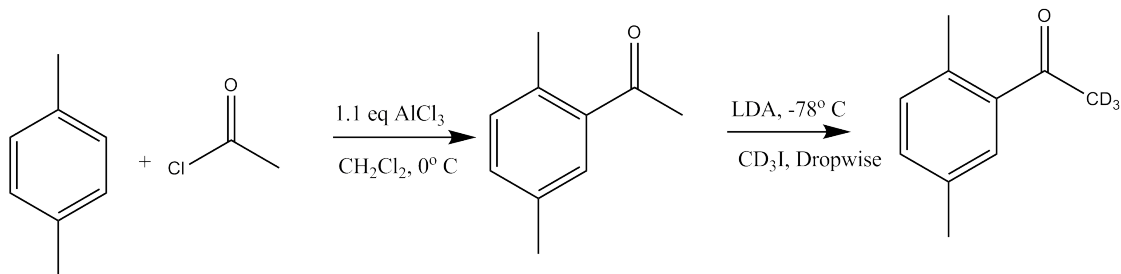
Scheme 2.



Scheme 3.



Scheme 4.





Matthew Sayre is from Santa Clarita, CA. He is currently a fourth year Human Biology major at the University of California, Merced. Matthew has always enjoyed both the biological and the chemical sciences, which is why he chose research that encompasses both disciplines; physical organic chemistry. His paper describes the interactions of molecules within catalytic reactions and how those rates are affected. After graduation he hopes to gain more research experience, moving towards medical/pharmacological research with his ultimate goal of attending medical school and pursuing a career in cardiology.