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LOMA LINDA UNIVERSITY School of Medicine in conjunction with the Faculty of Graduate Studies

Synthesis of HuaCat® Analogues as Novel Organocatalysts for the Formation of Asymmetric C-C Bonds

by

Kenneth Laboy

A Dissertation submitted in partial satisfaction of the requirements for the degree Master of Science in Biochemistry

September 2016

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Willie Davis, Associate Professor of Pharmaceutical and Administrative Sciences

Nathan Wall, Associate Professor of Biochemistry and Molecular Biology

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ABBREVIATIONS

Al	Aluminum
As	Arsenic
Boc	tert-butyloxycarbonyl - protecting group
Cl⁻	Chloride Ion
Cbz	Carboxybenzyl - protecting group
CDCl ₃	Deuterated chloroform
CDK	Cyclin dependent kinase
СоА	Coenzyme A
CO ₂	Carbon Dioxide
DBU	1,8-Diazabicyclo[5.4.0]undec-7-ene
DCM	Dichloromethane
DHAP	dihydroxyacetone phosphate
DIBAL	Diisobutylaluminium hydride, (Aluminum, hydrobis(2-
	methylpropyl)-)
DIPEA	N,N-Diisopropylethylamine
DMAP	4-dimethylaminopyridine
DMAPP	dimethylallyl pyrophosphate
DMF	Dimethylformamide
DMSO	Dimethyl sulfoxide
EDCI	1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide
EtOAc	Ethyl Acetate
Fmoc	9H-fluoren-9-ylmethyl ester (Fluorenylmethyloxycarbonyl)

Ga	Gallium
GAP	glyceraldehyde-3-phosphate
H_2O_2	Hydrogen Peroxide
H ₂ SO ₄	Sulfuric Acid
HATU	O-(7-Azabenzotriazol-1-yl)-N,N,N',N'-tetramethyluronium
	hexafluorophosphate
HBTU	O-(Benzotriazol-1-yl)-N,N,N',N'-tetramethyluronium
HMG	3-hydroxy-3-methylglutaryl
HOBt	N-Hydroxybenzotriazole (Benzotriazol-1-ol)
HWE	Horner-Wadsworth-Emmons
Hz	Hertz
Ir	Iridium
Li	Lithium
МСРВА	m-Chloroperoxybenzoic acid
Mg	Magnesium
MTBD	7-Methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene
NaH	Sodium hydride
NMR	Nuclear Magnetic Resonance
Pb	Lead
PP or IPP	isopentenyl pyrophosphate
Pro or P	Proline – Amino acid
Pybop	benzotriazol-1-yl-oxytripyrrolidinophosphonium
	hexafluorophosphate

ROS	Reactive Oxygen Species
SAR	structure-activity relationship
Sn	Tin
THF	Tetrahydrofuran
TMS	Tetramethylsilane
TBAHS	Tetrabutylammonium hydrogen sulfate
TLC	Thin Layer Chromatography
TiCl ₄	Titanium (IV) chloride
ZnCl ₂	Zinc chloride

ABSTRACT OF THESIS

Synthesis of HuaCat® Analogues as Novel Organocatalyst for the Formation of

Asymmetric Carbon-carbon Bonds

by

Kenneth Laboy

Master of Science, Graduate Program in Biochemistry Loma Linda University, September 2016 Dr. David J. Weldon, Chairperson

The synthesis of organic compounds with preferential stereochemistry is ubiquitous in the scientific community. A chemical reaction that asymmetrically induces one stereoisomer over another is achieved through the use of catalysts and various coupling reagents.¹ Inorganic catalysts, such as TiCl₄ and ZnCl₂, have been documented for many decades as effective agents in the synthesis of asymmetric bonds, however, there are environmental limitations to their use. First, the asymmetric reactions involving these metal catalysts require solvents that are not environmentally-friendly, especially when used on an industrial scale and second, the metal catalysts are recycled when possible, however, there is always metal waste that is equally unfriendly to the environment. One solution is to develop organocatalysts (non-metal catalyst) that will accomplish the same asymmetric reactions in aqueous reaction conditions. HuaCat® and HuaCatII® are organocatalysts developed by Dr. Rich Carter at Oregon State University that are able to achieve a single stereochemistry in 98% yield with greater than 95% diastereomeric excess.^{2, 3} These catalyst are not metal-containing and do work in aqueous conditions, but they still require the use of some solvent. The development of analogues of HuaCat[®] by substituting aromatic substituents with different electron

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withdrawing/donating groups will afford catalysts with increased yield and more predictable stereochemistry in aqueous conditions. One of the major challenges of the project is the formation of the key peptide bond of an amino acid and a sulfonamide containing a highly electronegative aromatic substituent in the ortho position, a feat that has minimal (and questionable) literature support to date. In this paper, we will detail the methods that have been attempted and the future plans for overcoming this unfavorable reaction to achieve what could be a new path to a unique organic structure for further synthetic manipulation.

CHAPTER ONE

INTRODUCTION

The Importance/Significance of Carbon-Carbon Bond Formation

Carbon-carbon (C-C) bond formation is the foundation to organic life here on earth.⁴ Any attempt to understand and expand upon the properties of organic life start with reactions of organic compounds and is further preceded by their chemical structure.⁵ Biomedical translational research hopes to convert what the scientist elucidates in the laboratory into practical application that can be employed in a clinical setting for organic life;⁶ therefore, it is explicit that the two fields have cohesion. The focus of this thesis will be intrinsic carbon-carbon bond forming activities in nature and the organic synthetic processes utilized to make novel structures with biological importance.

Nature affords a large number of structurally diverse compounds capable of regulating many biological processes, including cell division, differentiation and enlargement, chloroplast development and senescence. In order to access compounds with improved properties in comparison to those found naturally-occurring, it is essential to establish their structure-activity relationship (SAR). SAR research area in organic chemistry entails defining the role of the molecular features of any biologically-active chemical structure with respect to its target.⁷ SAR research involves the construction of a diverse and systematic library of derivatives that can then be compared to a pharmacophore model of the natural product to determine the role of the manipulated areas defined by the derivative library.⁸ A pharmacophore is defined by Yang as "the ensemble of steric and electronic features that is necessary to ensure the optimal supra-molecular interactions with a specific biological target and to trigger (or block) its

biological response."⁹ In other words, the pharmacophore is the minimal molecular features of any biological compound that are absolutely necessary to appreciably interact with the target. Defining a pharmacophore for a class of therapeutics is an early and essential step of translational research and modern drug discovery has an intense demand for stereoselective transformations of potential drug candidates.¹⁰ The search for potential pharmacophores has necessitated the use of catalysts that can facilitate the synthesis of natural products and their derivatives.¹¹ Leading to the rational observation that the pioneering of effective synthetic organic catalysts that can stereo- and regioselectively manipulate favorable chemical reactions with high product yields, minimal cost, and minimal environmental implications is fundamental to the scientific and health community.

Organic synthesis can be divided into various categories with two main types of reactions: C-C bond formations and functional group transformations.¹² C-C bond formation is the core of organic synthesis¹³ and furnishes the basis for producing more intricate organic compounds from simpler ones. The ability to manipulate the carbon backbone of various organic molecules through C-C bond formation makes it one of the cornerstones to transformation in organic synthesis.¹⁴ It is fundamental in medicinal chemistry to have the capability to expound and develop the carbon framework of organic molecules via a series of C-C bond forming reactions.¹⁵ Its function in nature for biosynthesis and metabolism is critical to the survival of organic lifeforms in order to maintain homeostasis.¹⁶ Moreover, it can be applied to organometallic¹⁷, agrochemical¹⁸, and biocatalytic organic synthesis.¹⁹

In nature, organic substances found in living organisms are conceived innately. Molecules from nature are essentially blueprints for organic chemists to develop novel techniques for determining structures, analyzing mechanisms of reactions, exploring the effects conformation and stereochemistry have on reactions, and pioneering challenging new targets to synthesize.²⁰ Harnessing the powerful techniques of organic molecules in their biological hosts, such as the enzymes that synthesize endogenous molecules and the complex processes that occur in a cell, is a valuable tool.¹⁵ In this congruent perspective, bioorganic chemistry hopes to understand the chemical mechanisms of enzyme-catalyzed reactions, to develop techniques to identify and to study how structure determines function in biosynthetic enzymes²⁰ with protein substrates obtained by organic synthesis through the use of catalyzed C-C bond formation.²¹

Expanding on the importance of C-C bond formation leads to a focus on biochemical enzymatic reactions, which are often overlooked as a method for organic synthesis of C-C bond formation. Biochemical reactions are generally catalyzed by an enzyme and are classified in one of these four broad categories: (1) group-transfer reactions, (2) oxidation and reductions, (3) eliminations, isomerizations, and rearrangements, and (4) reactions that make or break carbon-carbon bonds.²⁰ It is noted that elimination reaction mechanisms using dehydration resulting in the formation of a C=C double bond (e.g. enolase, fumarase) should be considered C-C bond formation;²² The focus of this research project is primarily C-C bond formation, the fourth category of biochemical enzymatic reactions.

There are many examples of catalysis in biological processes. One example are aldolases, a class of enzymes that act as catalysts for aldol condensation reactions.²³ An

aldol condensation is an organic chemical reaction in which an enol, or an enolate ion, reacts with a carbonyl compound to form a β -hydroxyaldehyde or β -hydroxyketone, followed by a dehydration step to yield a conjugated enone (**Figure 1**).²⁴



In the first step of the Krebs cycle, citrate synthase catalyzes the reaction of acetyl coenzyme A (acetyl CoA) and oxaloacetate to produce citrate through an aldol mechanism (**Figure 2**). In this specific reaction, it is not an aldehyde or a ketone that acts as the nucleophile as shown in **Figure 1**, but rather thioester Acetyl-CoA.²⁵ Generally speaking Biochemists refer to this reaction as an aldol condensation reaction describing the first step of the process, the addition initiated by base deprotonation of an α -Hydrogen from acetyl CoA forming an enolate nucleophile that subsequently attacks the electrophilic carbonyl carbon of Oxaloacetate ion. Protonation eventually leads to the final product with the generation of a new C-C bond and a new stero-center.²⁶



Another example of this particular reaction takes place in gluconeogenesis. The reaction between two 3-carbon containing sugars, specifically glyceraldehyde-3-phosphate (GAP) and dihydroxyacetone phosphate (DHAP), to form the six-carbon product fructose 1, 6-bisphosphate (F1,6-BP) (**Figure 3**). This reaction is catalyzed by the enzyme fructose 1,6-bisphosphate aldolase.²⁵ It must be noted that the aldol reaction is not formally a condensation reaction because it does not involve the loss of a small molecule, however, it is an important example where a catalyzed C-C bond formation is used in biochemistry, thereby alluding to its significance in nature.



Steroids present another example of C-C bond formation in nature. Steroid biosynthesis is an anabolic pathway which produces steroids from simple precursors. Following the biosynthetic pathway in animals is a common method for drug development of targets for antibiotics and other anti-infection drugs.^{9, 27} Steroid metabolism in humans is also the target of cholesterol-lowering drugs.^{9, 13} The biosynthesis of steroids flows through the mevalonate pathway, which uses acetyl-CoA as building blocks to make lanosterol, a precursor to steroids found in animals, fungi, and plants.^{25, 27} In the mevalonate pathway, or the HMG-CoA reductase pathway, isoprene units donated by intermediates, isopentenyl pyrophosphate (PP or IPP) and dimethylallyl pyrophosphate (DMAPP), that were formed by acetyl-CoA subunits are coupled into more complex compounds making squalene.²⁷ These compounds are then folded and rearranged into lanosterol for subsequent steroid biosynthesis (**Figure 4**). The coupling of these small subunits into larger more complex compounds is done through enzymatic catalysis of C-C bond formation.



While many processes in nature use organic-based catalysts known as organocatalysts, organometallic reactions leading to the formation of new C-C bonds are commonly used in synthetic organic chemistry. There are two classes of note that can be distinguished.¹⁷ The first is when the metal acts as a template that mediates the incidence of a peripheral reagent on a ligand without that reagent bonding within the coordination sphere. The reaction of electrophiles with iron acylate anions,²⁸ seen in **figure 5A**, and catalytic allylic alkylation utilizing palladium complexes,²⁹ seen in **figure 5B**, are a few

well-known examples. These correspondingly exhibit enriched reactivity and stereoselectivity via the contribution of organometallic complexes. In the second type, the new bond is fashioned amid ligated species and a supplementary subdivision is essential. The significant step may be cis-ligand movement, for which the most recognizable illustration is alkyl migration to coordinated carbon monoxide (CO) in catalytic hydroformylation. The C-C bond is established by coupling adjacent carbon-metal bonds associated with exclusion of the organic portion.¹⁷



Organometallics afford practical applications in many catalytic processes, most notably those applications involving CO and polymers originating from alkenes. Common manufacturing, such as polyethylene and polypropylene (types of rubber), are produced by means of organometallic catalysts, typically heterogeneously by the use of Ziegler-Natta³⁰ catalysis (**Figure 6**).³¹ The carbonylation of methanol catalyzed by iridium to acetic acid is an efficient process proclaimed by BP Chemicals using metal carbonyl catalysts as well.³²⁻³⁴ Generally, synthetic aldehydes are fabricated by means of hydroformylation. Additionally, organic lithium (Li), magnesium (Mg), and aluminum (Al) compounds are highly basic reducing agents that catalyze various polymerization reactions.³⁵ Furthermore, organometallic compounds are also used in the fabrication of semiconductors.³¹ In addition to the diverse applications, organocatalysis often produces reactions with desirable quantitative yields.^{29, 36} Although, in organometallic reactions, the conversion of product is obtained in respectable yield, generally a mixture of isomers are obtained.^{29, 36, 37} However these processes use volatile compounds such as trimethyl-gallium, trimethyl-indium, trimethyl-aluminum, as well as related nitrogen, phosphorus, arsenic, and antimony compounds. Although found in the environment, they can be toxically hazardous when used in these quantities in industrial purposes.^{38, 39}



Agricultural, as well as environmental chemistry, is also greatly impacted by organic chemical reactions. While nature has used monohydrogenase and other enzymes to functionalize alkanes in aqueous environments at ambient conditions,⁴⁰ alkanes are generally considered nonreactive in conventional organic chemistry.¹² The direct functionalization of alkanes is ubiquitously found in a variety of related fields including

the development of laboratory chemicals, energy, and medicine; therefore, chemical synthesis of alkanes has essential implications. Research in this area predominantly focuses on the conversion of a C-H bonds into a C-O bond. An example in **Figure 7** illustrates the coupling of methane with CO to produce acetic acid in aqueous conditions catalyzed by Ytterbium(III) acetate and sodium hypochlorite. ⁴¹ Moreover, the application of organic synthesis of the catalytic reaction of C-H bonds adjacent to heteroatoms such as nitrogen and oxygen.⁴²



Another area where C-C bond formation is applied is in the utilization of carbon dioxide (CO₂). CO₂ constitutes a natural source of carbon and a one-carbon (C₁) synthetic component with a capacity for the functionalization of substrates.³⁷ Unfortunately, incorporation of CO₂ into unsaturated hydrocarbons is difficult because of its molecular stability and therefore requires activation that can be imparted through the use of transition metal catalysts.⁴³ **Figure 8** illustrates the activation of dienes by palladium complexes and direct incorporation of CO₂ into alkynes.



This is one catalytic route in which unsaturated lactones, a common intermediate in many synthetic schemes, are made.⁴⁴ Additionally, vinyl-carbamates can be regioselectively produced in one-step from terminal alkynes, CO₂, and secondary amines with ruthenium catalysts (**Figure 9**).⁴⁵



The Significance of a Broad Selection of Catalysts and Reagents

There are a wide variety of organic (non-metallic) catalytic reagents for traditional C-C bond formations. For example, Wittig reactions use phosphonium salts and phosphonates are used for Horner-Wadsworth-Emmons (HWE) reactions.⁴⁶ Both of these reactions are utilized to create alkene-containing products.⁴⁷ However, synthetic methods accessible for C-C bond formation are continually advancing.

There are various contributing factors that affect innovation, such as the expansion of stout and dependable protocols for cross-coupling, augmented obtainability

of various organometallic reagents, and the continued development of stoichiometric reagents that facilitate the addition of a specific carbon-containing moiety. The last category, depends on a diverse selection of reagents that can be employed under mild conditions with a broad substrate scope. Therefore, further development of reagents that permit potent routes to carbon-carbon bond formation is necessary. Cross-coupling reactions make use of substrates with favorable yields in diverse reaction conditions, as well as metal catalysts for various reactions. Research success hinges on the ability of the researcher to obtain the desired product, which is aided by the availability of reagents that consistently yield high specificity, when the experiment calls for it.⁴⁷

Though enzymatic processes in nature are obligated to occur in an aqueous environment by necessity, water has been a solvent to be evaded for conventional organic reactions. The increased acknowledgment that organic reactions can move forward suitably in aqueous media and extend improvements over those taking place in organic solvents is widespread in the scientific community.⁴⁸ This assertion was preceded by studies into Diels-Alder reactions forged by Breslow⁴⁹ and hydroformylation reactions.⁵⁰⁻

The advent of the field of green chemistry has encouraged the expansion of research activities in this subject matter.⁵³ Initial curiosity has developed into a lucrative discipline that companies have invested in with profound returns.⁵⁴ This takes form by way of an increase in stereoselectivity that benefits the researcher when designing synthetic routes for experimental protocols, as well as the added bonus of milder reaction conditions.⁵⁵ Also, contributing to a decrease in consumable materials that leads to increased profit margins.⁵³ Moreover, this affords the added benefit of decreasing the

amount of waste produced when manufacturing large scale chemical products, thereby limiting harmful effects to the environment.^{56, 57} This contributed to advancement toward comprehending the reactions of organic compounds in high-temperature water, which has obvious implications to chemical synthesis that extends to energy and fuels.⁵⁸ Green chemistry seeks to reduce chemical related impact on human health and the environment by the use of alternative, environmentally friendly processes and reaction media.⁵³

The decrease in consumable materials affects two areas: Solvent and reagent usage. The selection of solvents, the chemicals used to dissolve substances into a solution, are a significant aim for Green Chemistry programs. The foremost source of industrial waste in industrial chemical production comes from solvents, but meticulous selection can also raise reaction rates and reduce reaction temperatures. Reduction in the amount of organic solvent consumed in the synthesis of Viagra[™] (Sildenafil Citrate) is a major triumph of Green Chemistry as well as shareholders at Pfizer in the UK.⁵⁹

Through innovation, organic chemists were able to make the synthesis of Sildenafil Citrate, the active ingredient in Viagra[™] and Revatio[™], more efficient. The improvements resulted in substantial environmental advantages, that brought about the decline in the amount of several organic solvents essential for the synthesis of sildenafil citrate.⁵⁵ The utter removal of all chlorinated and highly volatile solvents such as diethyl ether, dichloromethane, methanol and acetone. This innovation also allowed for the removal of tin (II) chloride from the synthesis, a known environmental polluter.⁵³ It was replaced by a catalytic hydrogenation reaction with water being the principal by-product. The new route was designed around a specific cyclisation reaction seen in the final step of the synthesis ensuring a higher quality product. The financial gains can be estimated

through the elimination of 30,000 tons of chemical solvent that are no longer needed and decrease in the chemical reagents and substrates necessary to produce the final product.⁵³ Essentially, the amount of product produced was increased with less reactants consumed.

Another example comes from the scale up of the synthesis of pregabalin (LyricaTM), a medication utilized in the treatment of neuropathic pain.⁵⁴ Organic chemists remodeled the route of several enzymatic reactions to allow for chemical reactions to be performed in water instead of organic solvents. In this occasion, a synthetically produced, endogenous catalyst, lipase, was used. Thereby reducing the amount of organic materials consumed. Environmentally this enzymatic process was calculated to eliminate more than 3 million tons of CO₂ emissions relative to the previous industrial process. This equates to the removal 1,000,000 cars per year off the road in the United Kingdom (UK).⁵³

Various Methods of Carbon-Carbon Bond Formation

In the field of natural product synthesis, there are a number of methods at our disposal to make C-C bonds. Chronologically, five separate reactions in the field of organic chemistry have facilitated the practical use of synthetic organic reactions in the scientific community: Michael Additions, Mannich reactions, Diels-Alder reactions, HWE reaction, and Suzuki-Negishi (S-N) coupling reactions.

The Michael addition, introduced by Arthur Michael in 1887,⁶⁰ is a facile reaction for conjugate addition of a nucleophile and an activated olefin, or alkyne, for nucleophilic C-C multiple bond formation (**Figure 10**).⁶¹ The Michael addition has various advantages for the strategic planning of organic synthesis such as its use under mild reaction conditions and elevated functional group tolerance. It has a broad range of use for

polymerizable monomers and functional precursors as well as superior conversions with favorable reaction rates.⁶² Furthermore, its application in post-polymerization modification⁶³ and coupling of biological and synthetic polymers is well documented.⁶⁴ The Michael addition reactions versatility makes it well-suited to numerous types of laboratory disciplines to include biomedical applications such as gene transfection,⁶⁵ cell scaffolds,⁶⁶ and tissue replacements.⁶²

The convenience it affords over other reactions stems from facilitation of a wide range of polymers from assorted monomers, and analogous polymers are formulated in environments in which competing polymerization mechanisms will not function as well. This makes it valuable in biological applications, for instance, in protein derivitization the Michael additions mild reaction conditions are favorable since high temperatures, oxidizing radicals, and organic solvents are not possible.²¹

As aforementioned, the Michael addition encompasses the addition of a nucleophile, or 'donor,' to an activated electrophilic olefin, the 'acceptor,' resulting in a 'adduct.' It is generally considered in reactions where the addition of enolate nucleophiles to activated olefins is wanted; however, there are multiple functional groups that possess sufficient nucleophilicity to operate in the same capacity, as 'Michael' donors. Reactions containing non-enolate nucleophiles such as amines, thiols, and phosphines are classically referred to as 'Michael-type' additions. As long as the acceptor possesses an electron withdrawing and resonance stabilizing activating group that stabilizes the anionic intermediate, it can be considered a Michael addition.⁶⁷ There are many 'Michael-type' acceptors, because many functional groups have the electron withdrawing activating effect necessary to integrate seamlessly in Michael addition

reactions,⁶⁸ and are readily available commercially.⁴⁷ Examples are: Acrylamides, vinyl sulfones, α , β -unsaturated aldehydes, and azo compounds, to name a few.⁶⁷



The use of organic synthesis to produce natural endogenous compounds is quite prevalent and one such reaction employed to do so is the Mannich reaction, presented by Carl Mannich in 1912.⁶⁹ The synthesis of various compounds such as peptides,⁷⁰ nucleotides,⁷¹ antibiotics,⁷² and compounds that contain high concentrations of amines, such as alkaloids,¹¹ are all methods in which Mannich reactions are typically used.⁷³ Furthermore, it is commonly used in agrochemistry to synthesize endogenous auxins,¹¹ to establish quantitative structure-activity relationships, and analogous derivatives with improved properties to pioneer novel ligands compare pharmacophore model capacity.

The Mannich reaction is a condensation reaction considered to be a paradigm of nucleophilic addition of a primary or secondary amine to a carbonyl functional group, leading to subsequent dehydration to the Schiff base. The second step involves the electrophilic addition of the Schiff base with a compound possessing an acidic proton.⁷⁴ Essentially, it involves the addition of resonance-stabilized carbon nucleophiles to a substituted imines, or iminium salts. The carbonyl containing compound can undergo tautomerization to the enol form and subsequent attack of the protonated imine. The

Mannich reaction consists of three elements: (1) a primary or secondary amine; (2) an aldehyde; and (3) a methylene bridge,⁷⁵ a functional group that withdraws electrons by resonance.⁷¹ Condensation of the reactants takes place with concomitant discharge of H₂O. This leads to the production of a 'Mannich base', where the active hydrogen is supplanted by an amino-methyl group.⁷¹ A general reaction scheme can be seen in **figure 11**.



The Diels–Alder reaction is yet another applicable form in which C-C bond formation is fundament in research. It is the organic chemical reaction between a conjugated diene and a substituted alkene, ordinarily dubbed the dienophile, to form substituted cyclic systems.⁷⁶ In 1950, the Nobel Prize in Chemistry was awarded to Otto Diels and Kurt Alder for their introduction of this reaction in 1928.⁷⁷ Typically, the Diels–Alder reaction is acutely useful in synthetic organic chemistry as a dependable method for forming 6-membered systems with respectable management over regio- and stereochemical properties.⁷⁸ The underlying concept has also been applied to other π systems, such as carbonyls and imines, to furnish the corresponding heterocycles, known as the hetero-Diels–Alder reaction.^{79, 80} A general reaction scheme can be seen in **figure 12**.



Diels–Alder reactions are another valuable tool for the formation of C-C bond formation used in total synthesis of endogenous steroids, such as cortisone and cholesterol.⁸¹ Butadiene's reaction with quinone derivatives is essential to produce steroid skeleton rings with the sought after regiochemistry. Prostaglandins are another endogenous compound synthetically made that makes use of a Diels–Alder reaction early in its synthesis, thereby facilitating the relative stereochemistry of adjoining stereocenters on the prostaglandin core.⁸² The preceding applications justify the rational that C-C bond formation is scientifically fundamental and beneficial to the future development of laboratory research.

The HWE reaction is one of the tools of choice for olefination reactions in the field of organic chemistry. It stems from Leopold Horner's publication in 1958, of a unique Wittig reaction that uses carbanions stabilized by phosphine-oxide and carbonyl compounds.⁸³ Eventually W.S. Wadsworth and W. D. Emmons were able to modify the reaction further by applying phosphonates.⁸⁴ It is prevalently used for elaborating on complex synthetic precursors. However, it also used as a method to strategically couple preformed sections of a target molecule. Based on the developed route is may also be used to otherwise introduce a newly formed olefin when a defined stereochemistry is preferred. A general reaction scheme can be seen in **figure 13**.



Although the prevailing employment of this reaction is as a dependable means for E-alkene formation, it must be noted that the stereochemical outcome of the reaction depends on both the structure of the reactants and on the reaction medium. These factors encompass the base, solvents, and the inclusion (or exclusion) of any congruent additives, such as salts and crown ethers.⁸⁵ However it has been demonstrated that Z-selectivity can be accomplished as well using either Phosphonic acid bis(2,2,2-trifluoroethyl) ester established by Still⁸⁶ or bis(O-aryl)phosphonates offered by Ando.⁸⁷ Furthermore, it is well documented that the standard conditions originally in place can be modified as well with adequate success.⁸⁵

The palladium-catalyzed cross coupling reaction of boronic acid in the presence of organic halides is labeled the "Suzuki coupling" and is another means by which organic chemists are able to accomplish C-C bonds.⁸⁸ The originator of this method, was Akira Suzuki in 1979.⁸⁹ Palladium-catalyzed cross couplings in organic synthesis were pioneered by Suzuki, Ei-ichi Negishi, as well as Richard Heck, and they were jointly awarded the Nobel Prize in chemistry in 2005 for their efforts.⁹⁰ One of the differences between the Suzuki and Negishi coupling reactions is the use of either Nickel (Ni) or Palladium (Pd).⁹¹ Moreover, Negishi reactions are more commonly used in the synthesis of acyclic di, tri, and higher order terpenoid systems, over Suzuki reactions.⁹² Similar to HWE reactions, the Suzuki reaction is commonly employed to synthesize poly-olefins,⁸⁹ but with different substrate requirements. However, it is also used in reactions with styrenes, a precursor to plastic,⁹³ and substituted biphenyls are also coupled by this method.⁹⁴ It also shares the feature of its employment in the synthesis of complex compounds from smaller ones, which is advantageous for pharmaceutical agents in drug development.⁹⁵ For example, Caparratriene is an active compound isolated from the oil of Ocotea caparrapi tree, that is native to Colombia.⁹⁶ The oil is locally used in Colombia for remedies in treatment of a various ailments, and Cytotoxicity studies of the oil using human leukemia cells (CEM) showed considerable inhibition of growth at concentration when suspended in DMSO.⁹⁶ The key step in the synthesis of this compound uses the Suzuki reaction for coupling of the E-vinyl borane with E-2-bromo-2-butene that successfully generated isomerically pure caparratriene.⁹⁷ This infers the practicality of this reaction in chemical synthesis of C-C bond formation.

The general scheme for the Suzuki reaction (displayed in **figure 14**) employs a Pd catalyst, in the 0 oxidation state and a base, in the presence of a phosphine ligand (L_nPd^0) to facilitate the coupling of a organoboron species (RBR"₂) with a halide (R'X) to form a single carbon-carbon bond.⁹⁸ The reaction method is known to be less toxic and contain more functional group tolerance than its other organometallic predecessors. While having similar commercial availability options.⁹⁹ In addition, the Suzuki coupling is quite versatile with respect to the number of reaction conditions that it can be utilized in. For example, running the Suzuki coupling reaction in a micellar solution using TPGS-750-M, a designer surfactant, introduced by Bruce Lipshutz, enhances the yield and drastically diminishes the environmental contour it produces, or E factor, of the reaction.^{59, 100}



Figure 14 - General Suzuki reaction scheme with ligands below and catalytic cycle with Palladium
Stereochemistry and Regiochemistry

Although C-C bond formation is one of the foundations to organic chemical synthesis. It must be noted that physical and molecular properties can be expressed differently based on the stereochemistry of a compound. Furthermore how a compound participates in a reaction is a concept of regiochemistry.

A corollary of the tetrahedral arrangement of bonds to carbon is that two compounds may be different because the arrangement of their atoms in space is unalike. Stereoisomers have the same constitution but differ in their atomic spatial arrangement.¹⁰¹ For example, in **figure 15**, 2-bromo-3-chlorobutane have various confirmations in which it can assume, with some being enantiomers and others diastereomers.



Although the physical properties, such as density, melting point, and boiling point, can be relatively identical in most cases (*cis*- vs *trans*-2-butene being an exception¹⁰²). However, the difference in the relative spatial arrangement of atoms that

form the structure of molecules governs how molecules interact and their manipulation by other compounds in a reaction mixture.¹⁰³ The enantiomeric configurations of Carvone presents a comprehensible instance where this is made apparent (see **figure 16**).



The (S)-(+)-carvone, is a major component of caraway oil and it's enantiomer conformation, (R)-(-)-carvone, is the primary constituent of Spearmint oil.¹⁰⁴ Although structurally similar, except at its chiral center, have different odors. The disparity in expression of these two enantiomers is due to the divergence in behavior toward receptor sites in the nose.¹⁰⁵ These receptor sites are themselves chiral and are innately stereo-selective in the compounds that they will bind with.¹⁰¹ This illustration shows how the difference in stereoisomers modifies how two enantiomers accommodate to their environment.

The high degree of chiral recognition inherent in most biological processes has prompted a number of considerations ranging from safety and efficacy to synthetic methodology, thereby requiring more chiral synthetic drugs become available in enantiomerically pure form.¹⁰⁶ Most naturally derived drugs are chiral and are already extracted as a single enantiomer from the source instead of as a racemic mixture.¹⁰¹ Although the desired therapeutic activity resides in one of the enantiomers, many synthetically prepared drugs are administered as racemic mixtures. Although in some of these racemic drugs, the inert enantiomer is relatively benign. In the case of over the counter Ibuprofen, the (S) active form is responsible for the analgesic/anti-inflammatory effects and the (R) enantiomer is inert (see **figure 17** for structure). However, it is eventually metabolically converted through Lipase enzyme-catalysis in the body to the active (S) configuration.¹⁰⁷



However, this relatively benign effect is not always the case, as the frequently documented circumstance of thalidomide. Thalidomide is a pharmaceutical drug prescribed for the treatment of morning sickness in pregnant women¹⁰⁸ (see **figure 18** for structure). The drug was discovered to be teratogenic, producing harmful genetic modification to early embryonic development, leading to severe limb deformation in neonates.¹⁰⁹ There are various mechanisms proposed to explain the biological role for

both the (R)- and the (S)-thalidomide enantiomers.¹¹⁰ Pharmacokinetically thalidomide undergoes racemization: even if only one of the two enantiomers is administered as a drug, the other enantiomer is produced as a result of metabolism.¹¹¹ Thalidomide is currently used for the treatment of other diseases, most notably cancer.¹¹² This tragedy initiated the involvement of the Food and Drug Administration to develop strict guidelines requiring testing of drugs prior to making them accessible to the public.¹⁰⁶ These guidelines encouraged drug design that specifically focused on methods to synthesize the desired enantiomer, but left the option available for the approval of new drugs as racemic mixtures under special circumstances.¹¹³



The implications in synthetic organic chemical reactions is because during experiments, in the absence of other chiral molecules, reactions will generate achiral products or racemic mixtures.¹⁰¹ Functional groups such as Carbocations and alkenes, which are both planar, are attacked from either face in equal amounts generating racemic product mixtures.¹¹⁴ Since the faces of the systems are equivalent and there is nothing to distinguish them then the probability of interaction is generally the same. In order to

create non-racemic products, a chiral influence must be used in the form of the starting material, a reagent, a catalyst or even during purification.¹⁰¹ In either circumstance the general implication is that more reliable methods to produce higher yield of enantiomerically pure product is imperative to the organic synthesis.

Essentially, research into methods for executing syntheses of single stereoisomers is critical due to the obligation to produce optically pure pharmaceutical products, particularly where one enantiomer is more effective than the other.

In chemistry, regioselectivity is the process that favors bond formation at a particular atom over another possible atom.¹¹⁵ The concept concerns the various probable positions a reagent will affect a compound in a reaction procedure, such as which Hydrogen will be deprotonated by a strong base from an organic molecule, or the most favorable position of insertion of an additional substituent on an already substituted benzene ring.^{115, 116} This application of rules associated with reaction predictability based on the stability of a molecule relative to the point of incidence where two molecules possibly interact. The complexity of the topic is abstract and goes beyond the scope of this topic; therefore, the significance is better described by the description of what it implies in organic synthetic processes. Such as the potential to impact two adjacent carbons, like in addition reactions of C-C bond formation. In this instance, if two new single bonds are formed from two separate atoms, then the potential to form isomers exists and one isomeric configuration has priority over the other, or one form is the dominant form due to regioselectivity.

The most notable concept that applies is Markovnikov's rule¹¹⁷ that states, "With the addition of a protic acid (HX) to an unsymmetrical alkene, the acid hydrogen (H)

becomes attached to the carbon with fewer alkyl substituents, and the halide (X) group becomes attached to the carbon with more alkyl substituents." The basis for this rule is the formation of the most stable carbocation during the addition process¹⁰¹ (see **figure 19**). Essentially, a positive charge is produced on the opposing carbon when a hydrogen ion undergoes addition to one carbon atom of an alkene, forming a carbocation intermediate. The more substituted carbon element has more stability due to induction and hyperconjugation. The major product of the addition reaction is predicted to be the carbocation with the most stable intermediate. However, the other less substituted, or less stable, carbocation will still be formed, but at less appreciable yield.



An example can be ring-forming reactions using Baldwin's rules, which are notably familiar processes in organic chemistry. The application of a set of simple rules (or predesignated constraints) is useful in predicting the relative capability of different ring closures; therefore, suitable preparation can negate potential problems to organic chemists, especially in planning syntheses.¹¹⁸ Furthermore, these rules help to specify that certain experiments are favored over others; which may be helpful to define more precisely their limits. The rules are of a stereochemical nature, thus these designs benefit synthetic chemists in both planning syntheses and examination of unsuccessful constructs. Which is a common occurrence in all scientific disciplines, not solely limited to organocatalysis.

Regioselectivity extends to other disciplines as well. For example in molecular biology cyclin dependent kinases (CDK) are central for the processes of cell division and proliferation. Correspondingly, innovation into the development of synthetic CDK inhibitors for the treatment of cancer and other proliferative diseases is currently being done.¹¹⁹ More specifically, a report on the synthesis and inhibitory actions of purine derivatives against CDK₁ demonstrated that among these purines, the ortho hydroxyl substituted benzyl purine displayed greater activity towards the enzyme CDK₁ than its meta and para substituted counterparts.¹¹⁶ Therefore the synthetic approach was designed utilizing regioselective protection as the key step in the syntheses of the desired methyl benzoate.

This is shown in the reaction procedure by acylation of a compound predominating at hydroxyl groups that were meta and para to the carbomethoxy group and not the ortho substituted hydroxyl group of the benzoic acid substrate to be modified. Therefore, protecting groups¹²⁰ were introduced to the meta and para substituted hydroxyl groups to destabilize their reactive intermediates, ameliorating their regioselectivity in the reaction. The steric hindrance between the bulky protecting groups on the meta and para positions facilitated favorable conditions for O-methylation of the ortho-substituted hydroxyl group, converting it from the minor form to the dominant form. After this step the meta and para groups were deprotected yielding the desired methyl benzoate.¹²¹

Molecular regioselective predictability permitted for strategic planning to be used to ameliorate the inadequate yield of the desired product by revealing that the reaction conditions could be manipulated effectively to obtain the preferred construct. The ability to anticipate pitfalls in organic chemical synthesis in this approach is vital to generating the enantiomerically pure compounds required for pharmaceutical purposes and concomitant production of adequate quantitative yield of the preferred stereo- & regioselective product.¹²²

The Major Advantages of HuaCat®

A preponderance of chemical substances that are molded and fragmented in metabolic processes are optically active, and typically one specific enantiomer is shaped in these processes; therefore, affording their abundance in nature.¹²³ As a consequence of the stereoselectivity associated with these processes in biological systems, it is not mutually exclusive that it has also been observed on numerous circumstances that the physiological activity expressed of a particular compound resides almost exclusively in one of its optically active forms.¹²⁴ The scientific and the practical significance of methods for the research of specific optical isomers is relatively discernable.

The conventional chemical resolution system agonizes from the detriment of yielding theoretical maximums of approximately 50% of the required optically active isomer established by the racemic starting material. Treatment of the racemic mixture with reagents of biological origin such as, microbiological enzymes, is an alternate procedure that exists with relatively similar disadvantages.¹²⁴ Divergence from this regularity stems from the induction of an asymmetric synthesis that can theoretically

result in yields approaching 100% of simply one enantiomer.¹²⁵ Its significance is manifested through the copious amounts of journal articles and chemical literature demonstrating its magnitude.¹²⁶ The description of laboratory results with reactions producing one diastereoisomeric pair of a given structure is termed "stereoselective syntheses," and is sometimes referred to as, "asymmetric syntheses" in some circles.¹²⁴

In parallel with the aforementioned concept that stereoselective reactions are critically important in the field of organic chemistry. There are numerous accounts of pharmaceutical therapeutics that have complex stereochemistry as key parts of their pharmacophore.¹²⁷⁻¹³⁰ Researching methods for forming selective carbon-carbon asymmetric bonds can have a large impact on drug discovery¹³¹ and human health.⁹

Considering this rational and that organocatalysis has gained the notice of the synthetic pharmaceutical population. Research in this area has been stimulated by stereoselectivity¹³² that can be produced in mild reaction conditions,¹³³ with relative ease of execution,¹³⁴ and extensive array of feasible chemical transformations.¹³⁵ Proline (Pro) as an organocatalyst,¹³⁶ has drawn noteworthy consideration in this area, markedly in the role of facilitating aldol reactions.¹³⁷ Pro has been used in the development of broad scope aldolase antibodies that show very high enantioselectivities¹³⁸ and it has been postulated that they proceed via an enamine mechanism.¹³⁹ Asymmetric small-molecule aldol catalysts that use an enamine mechanism have great potential in the discipline of organic chemical synthesis.¹⁴⁰

Explicitly, the proposed mechanism for Pro catalysis follows the Zimmerman-Traxler model for aldol reactions.¹⁴¹ This model suggests that the transition states of certain aldol reactions have six membered intermediates that display chair confirmations

facilitated by intramolecular dipole-dipole bonding.¹⁴² **Figure 20** displays a general view of how the chair confirmation may look.



It is well documented that proline can undergo an assortment of reactions with aldehydes that are aliphatic¹⁴³ and aromatic¹⁴⁴ compounds. Although there are ketone side products this can be ameliorated with high concentration of a polar aprotic solvent, such as acetone or Dimethyl sulfoxide (DMSO);¹⁴⁵ however, this is problematic due to the high cost of solvents and the environmental difficulties that come with the removal of waste products.¹⁴⁶ Moreover, the yield is still relatively low at 68% as a racemic mixture.¹⁴⁵

The established reaction stereochemistry in the Zimmerman model, E-enolates (or trans for relative stereochemistry) have a tendency to generate anti-addition products with the two groups of higher priority added to opposite sides (or faces) of the newly formed carbon bond, essentially resulting in a reduction in bond order but a rise in the number of

substituents. In direct contrast, Z-enolates (cis) have a higher reaction propensity towards syn-addition products. The factors that control selectivity are the preference for placing substituents equatorially in six-membered transition states and the avoidance of synpentane interactions, respectively (See **figure 21** for proposed mechanism of interaction). Generally the use of metals such as lithium and boron are used for this enolate mechanism of aldol reactions and thereby reliably follow the Zimmerman–Traxler model.¹⁴⁷ However on occasion metals can be relatively unpredictable with respect to their stereochemical results.¹⁴⁸



Figure 21 - Proposed mechanism for Proline aldol reaction with transition state chair confirmation

The exploration of Pro as an organocatalyst was initiated due to: (1) its nature for being an optically active pyrrolidine derivative. (2) The asymmetric carbon atom is in the same molecule next to the functional groups. (3) The asymmetric carbon atom is in a five-membered ring. The cyclic system provides amplified rigidity that typically augments the optical rotatory power; therefore, concomitant rise in the stereoselectivity of the asymmetric reagent can be anticipated. (4) The facile addition of the optically active reagent on various positions of the reacting symmetrical construct facilitating differentiation of identical groups. This is facilitated by the addition of various functional group substituents to impart a transient disposition with respect to reaction conditions. (5) Furthermore, the isoelectric point of proline at pH 6.30 is ideal for limiting the formation of unwanted side products such as ketols.¹²⁴

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Notwithstanding Pro's effective enabling of organocatalytic activity, it has its limitations.¹⁴⁹ Accordingly, an assortment of Pro mimetics are concomitantly being designed to furnish enhanced reactivity profiles.¹⁵⁰ Two frequently commissioned classes of note are the tetrazoles,¹⁵¹⁻¹⁵⁴ shown in **figure 22A**, and sulfonamides¹⁵⁵⁻¹⁶⁰ in **figure 22B**. The use of 4-trans-hydroxyproline derivatives is prevalent in organocatalysis;¹⁶¹ however, only one enantiomer of trans-hydroxyproline is facilely accessible which poses

another obstacle to these substrates; therefore, more considerably diverse structures are required for facile use.



In addition, organocatalyzed reactions containing proline or proline surrogates utilize polar solvents such as DMF and DMSO.¹⁵⁷ Although universally employed in research laboratories, their polarity generates additional obstacles for product separation and purity. The industrial application of nonpolar solvents are valuable because they provide reliable phase splits with water and can be recycled to a limited scale.¹⁶²⁻¹⁶⁶

The use of solvents typically accounts for approximately 80% of the mass of a reaction in a conventional pharmaceutical batch chemical operation.¹⁶⁶ Furthermore, solvents are one of the principal players in the overall toxicity profile of a reaction procedure.⁵⁵ Yet, for synthetic organic chemists, solvents are classified as the medium in which a reaction takes place and are not typically included as reactants in the stoichiometric sense because the focus is in the formation of the anticipated compound, not in the instrument by which it is carried out.¹⁶⁶

It must be noted that the need for inexpensive materials is part of the bottom-line to any industrial and laboratory budget; therefore, proline mimetics should be reasonably priced and readily accessible in both enantiomeric forms. Thus, the development of a pragmatic solution for these tasks is persistently explored. However, there is a highly practical and readily available proline surrogate called HuaCat®, developed by Dr. Rich Carter of Oregon State University. It displays enhanced solubility properties in conventional nonpolar organic solvents.¹⁶⁷ HuaCat® is a proline mimetic sulfonamide-based catalyst.

HuaCat® has proven to be an effective catalyst for asymmetric aldol reactions (**A**), a reaction where the enolate of an aldehyde or ketone is alkylated at the α -carbon by a carbonyl of another molecule producing a β -hydroxy carbonyl compound with two newly formed stereocenters,¹⁶⁷ (**figure 23A**). HuaCat® can also mediate Mannich reactions (**B**), an organic reaction which consists of an amino alkylation of an acidic proton placed next to a carbonyl functional group by formaldehyde and a primary or secondary amine or ammonia,¹⁶⁸ (**figure 23B**). HuaCat® facilitates these reactions with high stereoselectivity in aqueous conditions. Additionally, intramolecular Michael additions (**C**), or cyclization of keto sulfones are also facilitated by HuaCat®, allowing for the synthesis of five and six-membered rings with good yield and stereoselectivity,¹⁶⁹ (**figure 23C**).



Since the goal of various research studies is to synthesize compounds with highly accurate stereo specificity through chiral affects from catalysts, reagents, and inexpensive solvent systems. It is prudent to discover more mechanistic avenues for selective addition of C-C Asymmetric bonds.

The major purpose of this experiment is to substitute select moieties on the Hua Cat catalyst with different electron withdrawing/donating groups and observe their Asymmetric induction of Carbon bonds in aqueous conditions. The hypothesis for the proposed research is that the substitution of the current alkyl moiety with a trifluoro, iodo, or dodecane functionality, in the Ortho position rather than the Para position, can offer another mechanism and increase catalytic yield of stereospecific compounds in aqueous conditions for a wider range of substrate starting materials. (see **figure 24** for product structures)



We hope to observe the effectiveness of the proposed catalyst through:

First, synthesizing the proposed compounds in high yield. Second, characterization of the compounds synthesized. Third, the use of the various catalysts in chemical reactions to observe their effectiveness relative to current compounds in use as reagents for chemical Asymmetric induction. The search for novel catalytic reagents is highly desired; with the aforementioned experiments we can preferentially formulate chemical reactions with one stereoisomer over another by broadening the chiral features that affect asymmetrically induced chemical reactions.

CHAPTER TWO

MATERIALS AND METHODS

General

All reactions involving air- and moisture -sensitive compounds were carried out under a dried argon (Ar) atmosphere with standard Schlenk and vacuum-lines. All glassware was oven dried at $\geq 110^{\circ}$ C for ≥ 6 hrs or flame dried under vacuum and purged with Ar three times. Reactions were performed in Teflon tape sealed round bottom flasks and pigment-free, filler-free septa sleeve stoppers with reactions mixtures ensuing agitation via oven dried magnetic stir bars. Chemicals and solvents were purchased from commercial suppliers at \geq 98.9% purity as certified ACS reagent grade from Fisher Scientific (Fair Lawn, NJ) and Sigma Aldrich (Milwaukee, WI), without further purification. Thinlayer chromatography (TLC), was performed on silica gel plates (Merck, silica gel 60 F_{254}). Concentration of compounds was accomplished using a Büchi Rotary Evaporator (Rotovap) at appropriate solvent conditions with reduced atmospheric pressure and water bath at an average temperature of 40° C. Flash column chromatography was carried out with Grace Discoveries Reveleris flash purification system with Reveleris® silica gel with an average particle size of 39.1 µm and a pore diameter of 65 angstrom. All compounds were visualized by irradiation with UV light and/or by treatment with a solution of phosphomolybdic acid (25 g), $Ce(SO_4)_2 \bullet H_2O$ (10 g), concentrated H_2SO_4 (60 mL), and H_2O (940 mL) followed by heating or by treatment with a solution of p-anisaldehyde (23 mL), concentrated H₂SO₄ (35 mL), acetic acid (10 mL), and ethanol (900 mL) followed by heating. ¹H NMR spectra were measured on a Bruker AM-300 spectrometer in $CDCl_3$ or acetone-d6 with tetramethylsilane (TMS) as

the internal standard, where J (coupling constant) values are estimated in Hz. Spin multiples are given as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broad).

CHAPTER THREE

SYNTHETIC STRATEGY, RESULTS, & DISCUSSION

Design Strategy

The main goal of the project is to make ortho-substituted HuaCat® derivatives with strong electron-withdrawing functional groups. Although a method has been established for making HuaCat® and other para-substituted HuaCat® derivatives, there is currently no reliable method for making the ortho-substituted analogous structures. The few relevant methods in the literature do not account for strong electron-withdrawing effects of the sulfonamide substrates rendering them much less nucleophilic than an unsubstituted sulfonamide. The development of the organocatalytic HuaCat® analogues began with the base proline structure and the attempted manipulation of the intramolecular forces that are displayed in the chair transition state model seen below in **figure 25**. The project had 3 different design phases based on the results of the respective previous designs.



Our initial experiments were based on the original synthetic route for a mainstream peptide coupling reaction with three marked regions of divergence that would serve to precede us through the design of further analogues after the initial brand originated from the master model of HuaCat®. The three building blocks, in **figure 26**, represent the areas where manipulation will occur. First, the substitution of an electron withdrawing/donating group at position 7 of building block 1. Second, is the rearrangement of the sulfonamide from para to ortho, or its movement from position 3 to position 5. Lastly, is the varying protecting groups at position 17 of building block 3.



In the first design phase, we conducted base catalyzed break down of a larger molecule (compound 1/saccharin) in preparation for subsequent nucleophilic substitution

of the ester moiety to an activated iodododecane electrophile. Eventually leading to a standard peptide coupling on the activated Nitrogen (N) of the sulfonamide moiety to the electrophilic carbonyl portion of a Carboxybenzyl-protected L-proline (Cbz-L-Pro-OH), as outlined in the second step of **figure 27**. However, after attempted characterization of the final product, we concluded that this method was not efficient due to a lack of appreciable yield; therefore this route was abandoned and we proceeded on to our next phase of synthetic design.



In the second design phase, although it was understood that an ortho-substituted version would be appreciably less nucleophilic than the para-substituted compounds like HuaCat I & II. Moreover, it was noted that the steric hindrance would also be problematic to nucleophilic activation. However, to inductively rule out all possible barriers to success, we proceeded with the initial attempt utilizing similar reaction conditions to couple analogous starting materials to make the desired target compounds. As can be seen in **figure 28**, standard peptide coupling conditions were attempted.



Essentially, this phase involved the purchase of sulfonamide, and corresponding carboxylic acid (Pro), starting materials to couple into the target compounds. Using NMR analysis of our product we concluded that the second design phase proved to be unsuccessful. We theorized that the CF₃ moiety created a heightened electron-withdrawing effect that deactivated the N portion of the sulfonamide nucleophile, thereby hindering its nucleophilic capability and preventing the reaction from moving forward. This lead to the use of various coupling agents under the same reaction conditions to rule out the effectiveness of the reagents. This also comprised alternating various types of organic bases to ensure nucleophilic activation through deprotonation. This ruled out the propensity for one base over another under certain reaction conditions. It eventually came to include manipulation of reaction conditions in the form of temperature control, protic vs aprotic solvent usage, and various other strategies outlined in **Tables 1 & 2** referencing **figure 30**.

Despite copious manipulation of reaction procedure support elements, we were unable to ascertain the desired product and therefore aborted our second phase of experimental design strategy. Speculating that the ortho-substituted sulfonamide was

much less nucleophilic than the para-substituted sulfonamide, we moved towards an alternate design strategy. One that utilized the sulfonamide substrate as the electrophile rather than the nucleophile. In this phase, Thionyl chloride substituted starting material was coupled with the corresponding amido-substituted Prolinamide (Cbz-L-Pro-NH₂), as seen in **figure 29**.



As aforementioned, we attempted many different ways to construct the target derivatives. Some of the attempts included substrate changes from sulfonamides as nucleophiles to amides as nucleophiles, activating carboxylic acids for nucleophilic attack with many different peptide coupling agents¹⁷⁰ (EDCI, HOBt, HATU, etc.) and converting them to acid chlorides, the use of many different bases from mild to very strong, and increased reaction temperature with expanding reaction times.¹⁷¹⁻¹⁷⁵ To date, we have not been successful in constructing the target compound in appreciable yield.

The low yield of the product led to our third experimental design phase that involved a more indirect route through the use of a thiol substituted Benzene ring. Taking advantage of the increased electrophilic nature of the Thio-amine, relative to the sulfonamide, we surmised that coupling the Carboxylic acid would be more feasible. Thereby allowing for higher yield of the target compound. After which we could oxidize the sulfur portion of the sulfo-amide converting it to the desired sulfonamide using a well-documented standard method with m-Chloroperoxybenzoic acid (MCPBA).^{176, 177}

We have attempted to synthesize HuaCat® ortho-substituted derivatives by reaction schemes outlined in **figure 30** following **Tables 1 & 2** reaction conditions. A more detailed explanation of each step will be presented in the experimental section.



Figure 30 - General scheme for the formation of the desired organocatalysts

R ₁	R ₂	R ₃	R ₄	Reaction Conditions
CF3	NH ₂	ОН	Cbz, Boc, Fmoc	A, B, C, D
CF3	Cl	NH ₂	Cbz, Boc, Fmoc	B, D
CF3	NH ₂	Cl	Cbz, Boc, Fmoc	D, E
I	NH ₂	ОН	Cbz, Boc, Fmoc	A, B, C, D
I	Cl	NH ₂	Cbz, Boc, Fmoc	B, D
I	NH ₂	ОН	Cbz, Boc, Fmoc	D, E
C ₁₂ H ₂₅	NH ₂	ОН	Cbz, Boc, Fmoc	А

 Table 1 - Sample of attempted reactions to achieve the desired final organocatalyst

Table 2 - Details of reaction conditions A-E

Reaction Condition A	Reaction Condition B	Reaction Condition C	Reaction Condition D	Reaction Condition E
EDC, DMAP, CH ₂ Cl ₂	ZnCl ₂ , Benzoic anhydride	H2SO4, Benzoic anhydride	NaH, THF	Oxalochloride, MTBD
rt, 72-120h	MeCN, rt, 24h	MeCN, rt, 24h	rt, 24h	Toluene, rt, 48h

Examination of Results & Discussion

The prevailing theory was that the common dynamics utilized to synthesize the original compound would again be applied to produce the analogue. Therefore, our initial protocol demonstrated as much; however, with different starting materials. Utilizing base catalysis that encompassed Saccharin as our starting material, with subsequent standard peptide coupling conditions¹⁷⁰ (EDC, DMAP, in DMF) of compounds that included Cbz-proline.¹⁷⁸ Followed by deprotection using activated charcoal as seen in **figure 31**.

However, this synthetic route was unsuccessful when we arrived at the third step that required the coupling of the Proline amino acid to the sulfonamide. Potentially due to the steric hindrance of the new position of the sulfonamide on the benzene ring. Rather than postulating the results with further experimentation we decided to explore other synthetic routes.

In light of this, we developed another unrelated synthetic scheme (**Figure 32**). First, a chemo-selective alkylation of a thiol using NaH followed by the formation of the thiol chloride and nucleophilic addition of ammonia should afford the benzenesulfamine.¹⁷⁹ The rationale behind this is that the thiohydroxylamine does not have the same acidic nature seen in the sulfonamide, potentially allowing for more reactivity that was hindered in previous experiments. We reasoned that the increased reactivity should tolerate the addition of the protected Proline-OH or Proline acid chloride peptide coupling.¹⁸⁰ Lastly, the oxidation of the thiol to the sulfone using m-chloroperoxybenzoic acid (MCPBA) yielding the desired product, before amino deprotection with Pd¹⁷⁸ on the Pro, is well documented.¹⁷⁶





Figure 32 - Newly identified synthetic route to overcome the limitations of previous synthetic attempts

Conclusion

Although a method has been established for making HuaCat® and other parasubstituted HuaCat® derivatives, there is currently no method for making the orthosubstituted analogous structures. The main goal of the project was to make HuaCat® derivatives with strong electron-withdrawing functional groups as a para-substituent. However we were unsuccessful at achieving this goal and are currently searching for other methods of obtaining this but are still deliberating on viable methods to accomplish this feat. The product can be synthesized via these routes however, the yield varies from 2-9 %. Unfortunately making it an undesirable amount relative to the cost, our test results vis-à-vis the desired standards for lucrative use in the laboratory.

Experimental Section



 Sodium hydride (85% suspension in mineral oil, 280 mg, 2.5 mmol) was added to a solution of 2-(trifloromethyl)benzenethiol (500 mg, 2.8 mmol, 1 equiv) in anhydrous THF (5 mL). The mixture was stirred at room temperature for 10 min and treated with benzyl bromide (480 mg, 2.8 mmol, 1 equiv) for 1 h. After treatment with acetic acid, the reaction mixture was diluted with EtOAc and brine. The organic phase was separated and dried over Na₂SO₄. The filtrate was concentrated in vacuo and the resulting residue was purified by flash column chromatography on silica gel to obtain product. Product weight after purification: <u>2.017</u> mg. 2.017/268.30=0.0075; 1.438/178.17=0.0081; 0.0075/0.0081=0.9259

Compound	MW (g/mol)	D (g/mL)	mmol	mg	mL	Equiv
2-(Trifloromethyl) benzenethiol	178.17	1.438	8.1	1.43	1.0	1
Sodium Hydride	23.9971	1.396		280.6		2.5
THF	72.11	0.948			5.61	0.5
Benzyl bromide	171.03	1.35	15.8	480.0	2	2

 Table 3 - Step one quantities

0.9259*100=92.59% yield.



2. Compound 1 (1.77 mg, 2 mmol) was dissolved in CH₂Cl₂ (2 mL) and SO₂Cl₂ (2 mL, 8 mmol) was added. The mixture was stirred at room temperature for 10 h and then concentrated to dryness. To the residue was added 7 M NH3 in MeOH 10 mL). The mixture was stirred 2 h at room temperature, then diluted with water, extracted with EtOAc, washed with brine, dried (MgSO₄), and concentrated to dryness. The residue was dissolved in a minimum amount of EtOAc and reprecipitated with hexane. The resulting solid was collected by filtration to afford the title compound as a light tan solid. Product weight after purification: 189.2 mg. 189.2/193.19=0.9793; 475/268.3=1.7704; 0.9793/1.7704=0.5532; 0.5532*100=55.32% yield.

Table 4 - Deep two quantities									
Compound	MW (g/mol)	D (g/mL)	mmol	mg	mL	Equiv			
Compound 1	268.3		1.77	475.0		1			
Sulfuryl chloride	118.97	1.631	8.85	1053.1	0.645	5			
Ammonia in MeOH	50.07		3.54	177.3	10	2			
anhydrous DCM	41.05	0.786	0.89		1.770	0.5			

Table	4	- Sten	two	quantities
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EDCI (176 mg, 1.14 mmol) and 4-dimethylaminopyridine (189 mg, 1.55 mmol) were dissolved in acetonitrile (5 mL). The solution was then added to a mixture of compound 2 (200 mg, 1.04 mmol) and Cbz-L-Proline (283 mg, 1.14 mmol) in a reaction flask cooled to 0° C. This reaction mixture was allowed to warm to room temperature and stir for 24 hours at which time it was diluted with EtOAc (50 mL) and washed with a saturated NaHCO₃ solution (3x30 mL). The combined aqueous layers were back extracted with EtOAc (2x30 mL). All organic layers were combined, dried over MgSO₄, and concentrated to give 0.9507 mg of crude product mixture which was purified via flash chromatography (silica gel, 1:1 hexane: EtOAc) to give compound 14 (mg, % yield) as yellow oil. Product weight after purification: <u>38.1 mg</u>. 38.1/424.44=0.0898; 200/193.19=1.0353; 0.089/1.0353=0.086; 0.086*100=<u>8.6 % yield</u>.

Compound	MW (g/mol)	D (g/mL)	mmol	mg	mL	Equiv
Cbz-L-Proline	249.27		1.14	283.9		1.1
EDCI	155.24		1.14	176.8		1.1
DMAP	122.17		1.55	189.7		1.5
Compound 3	193.19		1.035	200.0		1
Acetonitrile	41.05	0.786	0.57		1.13	0.5

Table 5 - Step three quantities

To a stirred solution of **Cbz-L-Proline** (500 mg, 2.01 mmol) in **Acetonitrile** (2 mL), were added **EDCI** (342 mg, 2.21 mmol), **DBU** (360 mg, 2.41 mmol), **HOBt** (338 mg, 2.21 mmol), and **2-(Trifloromethyl) benzenesulfonamide**) (451 mg, 2.01 mmol), then left to stir for 3 days respectively. Daily, **EDC** and **HOBt** were added again (about 60% of original equivalents). After TLC verification, 15 mL **1M Citric Acid** was introduced to the reaction mixture (to neutralize the base), and then washed with **Brine** (3 X ~40 mL). The dried (**MgSO4**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 5-10% **EtOAc / Hexane**.



4. To a solution of Compound 3 (500 mg, or 1 eq.) in CH₂Cl₂ (3 mL) at 0 °C was added m-CPBA (142 mg, or 1.8 eq.). After the mixture is stirred at 0 °C for 39 h, the reaction is quenched with saturated aqueous Na₂S₂O₃ and saturated aqueous NaHCO₃. The mixture was extracted with EtOAc, washed with saturated aqueous NaCl, dried over anhydrous MgSO₄, and concentrated to give the crude product mixture which is purified via flash chromatography (silica gel, 1:1 hexane: EtOAc) to give compound 14 (~59% yield) as a yellow oil.^{176, 177}



Figure 37 - Previous experimental procedures, 1-6 reactions



Figure 38 - Previous experimental procedures, 7-12 reactions



Figure 39 - Previous experimental procedures, 13-18 reactions
Typical procedure for the preparation of **Compound 2**:



General procedure:

A mixture of **saccharin** (0.997 g, 5.5 mmol.) and **sodium hydroxide** (**NaOH**) (0.5 g, 12.5 mmol.) in distilled water (25 ml) was heated to reflux (1 hr), cooled to room temperature and acidified to pH = 4 (15% **HCl**). The reaction mixture was subjected to cooling in freezer overnight and white flake-like product was filtered, washed with cold water and dried to yield 1.06 g of the title compound (5.3 mmol. 97%) which was recrystallized from a solution of **MeOH** and **CHCl3** (1:1) at 40° C to obtain colorless crystals.^{180, 181}

Typical procedure for the preparation of Compound 3a:



General procedure:

To a mixture of **Compound 2** (304 mg, 1.51 mmol) and N, N-Diisopropylethyl amine (**DIPEA**) (214 mg, 1.66 mmol) that were dissolved in 13 mL of **acetonitrile**, was added **1-iodododecane** (492 mg, 1.66 mmol) at 85° C. The mixture was stirred overnight, then the reaction mixture was allowed to come to Room temperature. The mixture was then concentrated in vacuo to remove acetonitrile and then diluted with **EtOAc** (20 mL) and washed with 10% **KHSO**₄ (2x5 mL), **NaHCO**₃ sat (2x5 mL) and **brine**. The organic extracts were dried over **Na₂SO**₄, filtered and concentrated in vacuo. The crude was purified by flash column chromatography (5-20% EtOAc-hexanes) to yield 72 mg (0.10 mmol, 81%) of **3a** as a white solid.^{*1*82}

Typical procedure for the preparation of Compound 4a:



General Procedure:

A mixture of **EDCI** (176 mg, 1.14 mmol) and **4-dimethylaminopyridine** (189 mg, 1.55 mmol) were dissolved in **acetonitrile** (5 mL). The solution was then added to a mixture of **compound 1** (200 mg, 1.04 mmol) and **Z-Pro** (283 mg, 1.14 mmol) in a

reaction flask cooled to 0° C. The reaction mixture was allowed to warm to room temperature and stir for 24 hours at which time it was concentrated in vacuo to remove acetonitrile, then diluted with **EtOAc** (50 mL) and washed with a saturated **NaHCO3** solution (3x30 mL). The combined aqueous layers were extracted with EtOAc (2x30 mL). All organic layers were combined, dried over **MgSO4**, and concentrated to give 0.9507 mg of crude product mixture which was purified via flash chromatography (silica gel, 10:1 EtOAc:hexane) to give compound 4 (38.1 mg, % yield) as yellow oil.¹⁸³ Product weight after purification: <u>**38.1**</u> mg.

Typical procedure for the preparation of **Compound 4b**:



General experimental procedure for N-acylation of sulfonamides with anhydrides:

To a mixture of **Cbz-L-Proline** (1.0 mmol) and **Benzoic anhydride** $(C_6H_5CO)_2O$ (1.5 mmol), 3 mol % of anhydrous **ZnCl**₂ (1M in ether) was added and the reaction stirred in 20 mL **DCM** to allow for anhydride to form, then add **Sulfonamide** to reaction mixture and was allowed to stir for the given time. After completion of the reaction (monitored by TLC), the reaction mixture was diluted with dichloromethane and washed with water and brine solution. The combined organic layers were dried over **K₂SO₄** and evaporated in vacuo. The crude compound was purified by column chromatography (hexanes and **EtOAc**) to afford the corresponding N-acylated product. General experimental procedure for N-acylation of **sulfonamides** with **carboxylic acids**:

To a stirred solution of 5 mol % anhydrous **ZnCl**₂ in anhydrous dichloromethane, carboxylic acid (1.2 mmol) was added followed by the addition of benzoic anhydride (1.2 mmol) under an Argon atmosphere at room temperature. After 10 min, a solution of sulfonamide in CH₂Cl₂ was added and the resulting reaction mixture was stirred for the given time. After completion, the reaction mixture was washed with water then brine and the combined organic layers were dried over Na₂SO₄ and evaporated in vacuo. The crude compound was purified by column chromatography (hexanes and EtOAc) to afford the corresponding N-acylated product.¹⁷⁴ Typical procedure for the preparation of Compound 4c:



Figure 44 – Experimental procedure for 4c.

General procedure:

Under an **Argon** atmosphere, a mixture of **Fmoc-Proline** and **Benzoic anhydride** $(C_6H_5CO)_2O$, in **THF** (~8 ml) was stirred at 60°C and treated with 96% **sulfuric acid** (5

mol%) was added and the reaction stirred to allow for anhydride to form. The solution was maintained at 60°C for 4 hours until reaction was judged complete by TLC. Then **Benzenesulfonamide** (300 mg, 1.33 mmol), was added to reaction mixture drop-wise and was allowed to stir overnight @ 60°C. After completion of the reaction (monitored by TLC), the solvent was removed by distillation and then the solution was cooled to 24°C. Water (10–15 ml) was added dropwise to form a precipitate. The resulting mixture was stirred for 1 h at 20°C and then filtered. The crude solid was washed with deionized water (5–10 ml) and then dried in vacuo under nitrogen at 50°C for 18 h. Weight of white crystalline solid was 238 g. The reaction mixture was diluted with **dichloromethane** and washed with water and brine solution (3x15 mL). The combined organic layers were dried over **Na₂SO₄** and evaporated in vacuo. The crude compound was purified by column chromatography (10:1, EtOAc and hexanes) to afford the corresponding *Nacylated* product.¹⁷³

Typical procedure for the preparation of **Compound 4d**:





General Procedure:

Boc-Proline-NH₂ (2.04 mmol, 438 mg) was dissolved in 50 mL of **pyridine**; to this solution was added **2-(Trifluoromethyl) benzene sulfonyl chloride** (2.04 mmol, 315 μ L). This mixture was stirred for 1–2 h and then left overnight without stirring. After completion of the reaction, **crushed ice** was added to this mixture and the mixture was stirred again to obtain a solid crude product; if the mixture was slightly acidified, we did not obtain any product. This crude product was washed with dichloromethane and ice cold water. The product was further purified using column chromatography to obtain crystals of compound.¹⁷²

Typical procedure for oxalyl chloride catalyzed preparation of **Compound 4b**:



General Procedure:

In a reaction mixture **Cbz-L-pro** (1.1 mg, 4.4 mmol) was suspended in dry **DCM** (10 mL) under Argon atmosphere. To this solution, **oxalyl chloride** (4.1 mL, 48 mmol) was slowly added and the mixture was stirred for 2 h (with TLC observation). Gas evolution was observed, and the system was allowed to stir at room temperature for 28 h. The system was connected to another flask of 6 M **NaOH** to react with the **HCl** gas that

is formed from the reaction of the Reactants. The **DCM** was removed under reduced pressure and the residue was suspended again in dry **CH₂Cl₂** (10 mL). After addition of **triethylamine** (0.74 mL, 5.2 mmol) to the reaction mixture, **2-(Trifluoromethyl) benzene sulfonamide** (1.2 mg, 5.2 mmol) in **DCM** (5 L) was slowly added. The reaction mixture was stirred at room temperature for 4 h and then the mixture was suspended in water to remove impurities. The organic phase separated and the solvent was removed under reduced pressure to obtain a brownish viscous oil.¹⁸⁴

Typical procedure for Amide peptide coupling reactions by various organocatalysts:



General Procedure:

To a stirred solution (after 4.0 hrs of stirring) of **Cbz-Pro-OH** (500 mg, 2.01 mmol) & **triethylamine** (0.300 mL, 2.21 mmol) in **THF** (2 mL), was added **HATU** (838 mg, 2.2 mmol). After 1hr a mixture of **2-(Triflouro) benzene sulfonamide** (677 mg, 3.01 mmol) with 60% **NaH** (136 mg, 3.3 mmol) in **THF** (2mL) was added and left to stir for 2hrs respectively. After TLC verification, 5 mL **1M HCl** was introduced to the reaction mixture (to neutralize the base), and then washed with brine (3 X ~40 mL) and

the organic layer was washed again with **1M NaOH**. The dried (**Na₂SO₄**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 1-50% **EtOAc/hexanes**.¹⁸⁵



General Procedure:

To a stirred solution of **Cbz-L-Pro** (0.166 g, 0.670 mmol) in **THF** (7 mL), were added **EDCI** (0.103 g, 0.670 mmol), **HOBT** (0.090 g, 0.670 mmol), and **2-(triflouro) benzene sulfonamide** (0.300 g, 1.33 mmol) in **THF** (7 mL), then left to stir for 5 days respectively. After TLC verification, 15 mL **1M HCI** was introduced to the reaction mixture (to neutralize the base), and then washed with **brine** (3 X ~40 mL). The dried (**Na2SO4**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 5-20% **EtOAc/hexanes**.¹⁸⁶



General Procedure:

To a stirred solution (after 1.0 hr of stirring) of **Cbz-Pro-OH** (200 mg, 0.8 mmol) & **DIPEA** (0.4 mL, 2.4 mmol) in **THF** (2 mL), was added **PyBOP** (440 mg, 0.89 mmol). After 1hr a mixture of **MTBD** (0.150 mL, 0.96 mmol) **2-iodobenzene sulfonamide** (216 mg, 0.96 mmol) was added. After 10 min another equivalent of **PyBOP** was added and left to stir overnight, respectively. After TLC verification, 5 mL **1M HCl** was introduced to the reaction mixture (to neutralize the base), and then washed with brine (3 X ~40 mL). The dried (**Na₂SO₄**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 10% **MeOH/DCM**.^{*187*}



General Procedure:

To a stirred solution (after 20 min of stirring) of **Boc-Pro-OH** (300 mg, 1.39 mmol) & **DIPEA** (0.728 mL, 4.18 mmol) in **THF** (2 mL), was added **HBTU** (581 mg, 1.53 mmol). After 1/2 hr, **2-(trifluoro) benzene sulfonamide** (313 mg, 1.39 mmol) was added and left to stir for 2hrs respectively. Reaction mixture was concentrated then extracted using **chloroform**. After TLC verification, 40 mL **1M HCl** was introduced to the reaction mixture (to neutralize the base), and then washed with **brine** (3 X ~40 mL). The dried (**Na₂SO₄**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 100% **EtOAc.** Product mass crude 976 mg.¹⁸⁸



General Procedure:

To a stirred solution of **Boc-L-Pro-NH2** (300 mg, 1.21 mmol) and NaOH (23 mg, 0.580 mmol) in **THF** (4 mL), were added tetrabutylammonium hydrogen sulfate (**TBAHS**) (40 mg, 0.121 mmol), and **2-(triflouromethyl) benzene sulfonyl chloride** (0.37 mL, 2.4 mmol) in **DCM** (2 mL), then left to stir for 2 days respectively. After TLC

verification, 5 mL **1M HCl** was introduced to the reaction mixture (to neutralize the base), and then washed with brine (3 X ~40 mL). The dried (**Na₂SO₄**) extract was concentrated in vacuo (after filtration) then purified by chromatography over silica gel, eluting with 10% **MeOH/DCM**.¹⁸⁹



General Procedure:

To a stirred solution of **THF** and **sodium hydride** (**NaH**) (50 mg, 1.22 mmo), **Cbz-L-Pro-NH**₂ (305 mg, 1.22 mmol) was added at 0° C. The solution was allowed to stir for **1 hr** at **rt** and then **2-(trifluoromethyl) benzene sulfonyl chloride** (315 μ L, 2.0 mmol) was added drop-wise at 0° C, then allowed to stir for **1 hr**. The Reaction mixture was allowed to come to **rt** and left overnight. Then concentrated to remove **THF** and rediluted with **EtOAc**. It was then separated using **brine**, **water**, and **EtOAc**. The organic layer was dried using **Na₂SO**₄ anhydrous, then concentrated in vacuo. The product was weighed and purified using flash chromatography.¹⁹⁰

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APPENDIX A

FLASH CHROMATOGRAPHER PURIFICATION REPORTS





Method Name: Run Name: 2014-08-11_h-b2-c Run Date: 2014-08-11 10:34

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 38.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Camer: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	0.0	AB	2
4	4.0	AB	2
5	0.0	AB	5
6	5.0	AB	5
7	0.0	AB	10
8	5.0	AB	10
9	0.0	AB	15
10	5.0	AB	15
11	0.0	AB	20
12	4.0	AB	20
13	0.0	AB	25
14	3.0	AB	25
15	5.0	AB	25
16	0.5	AB	25
17	5.0	AB	25
18	0.5	AB	25

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray:Vial	
1	1.2	1:4	
2	1.5	19	
3	1:10	1:11	



Method Name: Run Name: 2014-07-30_KL-H-B5-A Run Date: 2014-07-30 15:37

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 33.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	4.0	AB	1
3	5.0	AB	2
4	5.0	AB	4
5	5.0	AB	6
6	5.0	AB	10
7	4.0	AB	12
8	3.0	AB	14
9	2.0	AB	16

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray Vial	
1	12	1.2	
2	13	13	
3	1:4	1.5	



Method Name: Run Name: KL-H-B2-H Run Date: 2015-11-16 11:42

Column: Generic Silica 40g Flow Rate: 40 mL/min Equilibration: 6.0 min Run Length: 17.8 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chosen> Solvent D: <No solvent chosen>



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.5	AB	0
3	0.0	AB	1
4	0.5	AB	1
5	0.0	AB	2
6	1.0	AB	2
7	0.0	AB	4
8	1.0	AB	4
9	0.0	AB	6
10	1.0	AB	6
11	0.0	AB	10
12	1.0	AB	10
13	0.0	AB	20
14	1.0	AB	20
15	0.0	AB	30
16	1.0	AB	30
17	0.3	AB	51
18	1.3	AB	51
19	0.4	AB	81
20	3.5	AB	81
21	0.3	AB	100
22	5.0	AB	100

1.2
1:4
1.8



Method Name: Run Name: KL-H-B2-H Run Date: 2015-11-16 11:42

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray.Vial	
4	19	1:11	
5	1:12	1:12	
6	1:13	1:13	



Method Name: Run Name: KL-H-B2-H_2 Run Date: 2015-11-17 07:33

Column: Generic Silica 40g Flow Rate: 40 mL/min Equilibration: 6.0 min Run Length: 11.1 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chosen> Solvent D: <No solvent chosen>



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.5	AB	0
3	0.0	AB	10
4	0.5	AB	10
5	0.0	AB	20
6	1.0	AB	20
7	0.0	AB	30
8	1.0	AB	30
9	0.0	AB	40
10	1.0	AB	40
11	0.0	AB	50
12	1.0	AB	50
13	0.0	AB	60
14	1.0	AB	60
15	0.5	AB	81
16	4.1	AB	81
17	0.5	AB	100

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray Vial	
1	1:2	13	
2	1:4	1.5	
3	1.6	1.8	
4	19	1:10	
5	1:11	1:11	


Method Name: Run Name: 2014-08-01_KL-H-B5-B Run Date: 2014-08-01 06:21

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 34.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	5.0	AB	1
3	5.0	AB	2
4	5.0	AB	4
5	5.0	AB	6
6	5.0	AB	8
7	4.0	AB	10
8	3.0	AB	12
9	2.0	AB	14

Vial Mapping Table		
Peak#	Start Tray Vial	End Tray.Vial
1	12	1.2
2	13	1:4



Method Name: Run Name: 2014-08-01_KL-H-B5-C Run Date: 2014-08-01 07:10

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 33.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	4.0	AB	1
3	5.0	AB	2
4	5.0	AB	4
5	5.0	AB	6
6	5.0	AB	8
7	4.0	AB	10
8	3.0	AB	12
9	2.0	AB	14

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray Vial	
1	12	1.3	
2	1:4	1:5	
3	1.6	1.6	



Method Name: Run Name: 2014-08-04_H-B5-D2 Run Date: 2014-08-05 12:33

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 23.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	5.0	AB	5
4	5.0	AB	10
5	5.0	AB	15
6	5.0	AB	20

Vial Mapping Table			
Peak# Start Tray Vial End Tray Via			
1	1.2	1.2	



Method Name: Run Name: 2014-08-11_h-b2-d1 Run Date: 2014-08-11 14:03

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 32.5 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	0.0	AB	2
4	4.0	AB	2
5	0.0	AB	5
6	5.0	AB	5
7	0.0	AB	10
8	5.0	AB	10
9	0.0	AB	15
10	5.0	AB	15
11	0.0	AB	20
12	4.0	AB	20
13	0.0	AB	25
14	3.0	AB	25
15	5.0	AB	25
16	0.5	AB	25

	Vial Mapping	Table
Peak#	Start Tray: Vial	End Tray Vial
1	1.2	1.8



Method Name: Run Name: 2014-08-11_h-b2-d2 Run Date: 2014-08-11 15:01

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 32.5 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradie	dit 1 abie	
	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	0.0	AB	2
4	4.0	AB	2
5	0.0	AB	5
6	5.0	AB	5
7	0.0	AB	10
8	5.0	AB	10
9	0.0	AB	15
10	5.0	AB	15
11	0.0	AB	20
12	4.0	AB	20
13	0.0	AB	25
14	3.0	AB	25
15	5.0	AB	25
16	0.5	AB	25

Peak#	Start Tray.Vial	End Tray Vial
1	1.2	1:4
2	1.5	1.5
3	1.6	1.6



Method Name: Run Name: KL-H-B6-E Run Date: 2015-03-19 11:14

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 10.5 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	1	T-	
	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.5	AB	0
3	1.0	AB	30
4	7.0	AB	30
5	1.0	AB	100

Vial Mapping Table		
Peak#	Start Tray Vial	End Tray Vial
1	1.2	1.2
2	13	13
3	1:4	1:4



Method Name: Run Name: 2014-08-12_h-b7-a Run Date: 2014-08-12 07:23

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 26.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent choser Solvent D: <No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	0.0	AB	2
4	3.0	AB	2
5	0.0	AB	5
6	5.0	AB	5
7	0.0	AB	10
8	4.0	AB	10
9	0.0	AB	15
10	5.0	AB	15
11	0.0	AB	20
12	5.0	AB	20
13	0.0	AB	25
14	3.0	AB	25

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray.Vial	
1	12	1:2	
2	13	1.3	



Method Name: Run Name: 2014-08-14_h-b7-b Run Date: 2014-08-14 11:03

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 35.0 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexans Solvent B: Ethyl acetate Solvent C: <No solvent choser Solvent D: <No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	0.0	AB	2
4	4.0	AB	2
5	0.0	AB	4
6	5.0	AB	4
7	0.0	AB	6
8	5.0	AB	6
9	0.0	AB	8
10	5.0	AB	8
11	0.0	AB	10
12	4.0	AB	10
13	0.0	AB	12
14	4.0	AB	12
15	0.0	AB	14
16	3.0	AB	14
17	0.0	AB	16
18	2.0	AB	16

Peak#	Start Tray Vial	End Tray Vial
1	1:2	1.2
2	13	1:4
3	1.5	1.5



Method Name: Run Name: 2014-08-29_h-b7-c Run Date: 2014-08-29 14:53

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 21.9 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent choser Solvent D: <No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.3	AB	0
3	0.0	AB	1
4	0.1	AB	1
5	0.0	AB	2
6	0.3	AB	2
7	0.0	AB	4
8	0.3	AB	4
9	0.0	AB	6
10	0.3	AB	6
11	0.7	AB	6
12	0.2	AB	6
13	0.0	AB	8
14	0.5	AB	8
15	0.0	AB	10
16	0.3	AB	10
17	1.3	AB	10
18	0.2	AB	10
19	0.0	AB	12
20	0.5	AB	12
21	0.0	AB	14
22	0.5	AB	14
23	0.0	AB	16
24	0.5	AB	16
25	0.0	AB	18
26	0.4	AB	18
27	0.0	AB	20
28	0.3	AB	20



Method Name: Run Name: 2014-08-29_h-b7-c Run Date: 2014-08-29 14:53

	Min	Solvents	% 2nd
29	0.5	AB	25
30	0.5	AB	30
31	0.1	AB	30
32	0.3	AB	30
33	0.1	AB	31
34	2.4	AB	31
35	0.3	AB	35
36	0.5	AB	40
37	0.5	AB	50
38	5.0	AB	50
39	5.0	AB	50

Vial Mapping Table

Peak#	Start Tray. Vial	End Tray Vial
1	1:2	1:2
2	13	13
3	1:4	1:4
4	1.5	1:6
5	1:7	1:10
6	1:11	1:11
7	1:12	1:12



Method Name: Run Name: 2014-08-29_h-b7-d Run Date: 2014-08-29 15:35

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 18.1 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent choser Solvent D: <No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.3	AB	0
3	0.0	AB	1
4	0.1	AB	1
5	0.0	AB	2
6	0.3	AB	2
7	0.0	AB	4
8	0.2	AB	4
9	0.4	AB	4
10	0.1	AB	4
11	0.0	AB	6
12	0.5	AB	6
13	0.0	AB	8
14	0.5	AB	8
15	0.0	AB	10
16	0.5	AB	10
17	0.0	AB	12
18	0.3	AB	12
19	0.9	AB	12
20	0.2	AB	12
21	0.0	AB	14
22	0.5	AB	14
23	0.0	AB	16
24	0.1	AB	16
25	0.4	AB	16
26	0.4	AB	16
27	0.0	AB	18
28	0.1	AB	18



Method Name: Run Name: 2014-08-29_h-b7-d Run Date: 2014-08-29 15:35

	Min	Solvents	% 2nd
29	0.5	AB	18
30	0.3	AB	18
31	0.0	AB	20
32	0.1	AB	20
33	0.5	AB	20
34	0.2	AB	20
35	0.5	AB	25
36	0.5	AB	30
37	0.5	AB	35
38	0.5	AB	40
39	0.4	AB	47
40	3.4	AB	47
41	0.1	AB	50
47	5.0	AR	50

Vial Mapping Table

Peak#	Start Tray.Vial	End Tray.Vial
1	1:2	12
2	13	13
3	1:4	1:4
4	1.5	1:6
5	1.7	1.7
6	1.8	1:8
7	1.9	1.9



Method Name: Run Name: KL-H-C3-B; 10-13-2014 Run Date: 2014-10-13 16:02

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 31.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 3 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect All Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Methylene chloride Solvent B: Methanol Solvent C: <No solvent chosen> Solvent D: <No solvent chosen>



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	1
2	0.5	AB	1
3	0.0	AB	2
4	1.0	AB	2
5	0.0	AB	3
6	2.0	AB	3
7	0.0	AB	4
8	3.0	AB	4
9	0.0	AB	5
10	4.0	AB	5
11	0.0	AB	6
12	5.0	AB	6
13	0.0	AB	7
14	4.0	AB	7
15	0.0	AB	8
16	3.0	AB	8
17	0.0	AB	9
18	2.0	AB	9
19	0.0	AB	10
20	1.0	AB	10
21	5.0	AB	10
22	0.5	AB	10

Peak#	Start Tray:Vial	End Tray:Vial
1	1:3	1:3
2	1:4	1:4
3	1:5	1:6
		Page 1



Method Name: Run Name: KL-H-C3-B; 10-13-2014 Run Date: 2014-10-13 16:02

Vial Mapping Table					
Peak#	Start Tray:Vial	End Tray:Vial			
4	1:7	1:7			
5	1:8	1:8			
6	1:15	1:15			
7	1:16	1:16			
8	1:17	1:17			
9	1:23	1:24			
10	1:25	1:25			



Method Name: Run Name: 2014-09-08_h-c1-a,#2 Run Date: 2014-09-08 15:50

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 10.2 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 15 mV UV Threshold: 0.04 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: «No solvent choser Solvent D: «No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.2	AB	0
3	0.0	AB	1
4	0.3	AB	1
5	0.0	AB	2
6	0.3	AB	2
7	0.0	AB	4
8	0.2	AB	4
9	0.6	AB	4
10	0.1	AB	4
11	0.0	AB	6
12	0.3	AB	6
13	0.0	AB	8
14	0.3	AB	8
15	0.0	AB	10
16	0.3	AB	10
17	0.0	AB	12
18	0.3	AB	12
19	0.0	AB	14
20	0.3	AB	14
21	0.2	AB	17
22	0.6	AB	17
23	0.1	AB	20
24	0.2	AB	25
25	0.2	AB	25
26	0.1	AB	30
27	0.1	AB	30
28	0.6	AB	30



Method Name: Run Name: 2014-09-08_h-c1-a,#2 Run Date: 2014-09-08 15:50

	Min	Solvents	% 2nd
29	0.2	AB	30
30	0.3	AB	40
31	0.0	AB	40
32	2.0	AB	40
33	0.1	AB	40
34	0.8	AB	40
35	0.2	AB	40
36	0.3	AB	50
37	0.2	AB	50
38	0.8	AB	50
39	0.2	AB	50

Vial Mapping Table

Peak#	Start Tray. Vial	End Tray: Vial
1	1:2	1:2
2	13	13
3	1:4	1:4
4	1.5	1.5
5	1.6	1:6



Method Name: h-c2-a Run Name: 2014-09-09_h-c1-a; #'s 4, 5, & 6 Run Date: 2014-09-09 17:45

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 8.8 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 10 mV UV Threshold: 0.03 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: «No solvent choser Solvent D: «No solvent choser



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	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.3	AB	0
3	0.2	AB	2
4	0.3	AB	7
5	0.3	AB	10
6	0.3	AB	15
7	0.0	AB	20
8	0.5	AB	20
9	0.0	AB	25
10	0.3	AB	25
11	0.0	AB	30
12	0.5	AB	30
13	0.0	AB	35
14	0.2	AB	35
15	4.0	AB	35
16	0.2	AB	35
17	0.0	AB	40
18	0.5	AB	40
19	0.0	AB	45
20	0.3	AB	45
21	0.0	AB	50
22	0.3	AB	50
23	0.0	AB	60
24	0.3	AB	60
25	0.3	AB	70



Method Name: h-c2-a Run Name: 2014-09-09_h-c1-a; #'s 4, 5, & 6 Run Date: 2014-09-09 17:45

Vial Mapping Table			
Peak#	End Tray.Vial		
1	1.2	12	
2	13	1.3	
3	1:4	1:4	
4	1.5	1:5	
5	1.6	1.7	



Method Name: Run Name: 2014-09-08_h-c2-a Run Date: 2014-09-08 10:21

Column: Reveleris® Silica 4g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 13.1 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 20 mV UV Threshold: 0.05 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent choser Solvent D: <No solvent choser



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.3	AB	0
3	0.3	AB	1
4	0.5	AB	1
5	0.8	AB	1
6	0.0	AB	1
7	0.2	AB	2
8	0.3	AB	2
9	0.3	AB	4
10	0.2	AB	4
11	0.3	AB	5
12	0.5	AB	5
13	0.3	AB	5
14	0.1	AB	5
15	0.0	AB	6
16	0.3	AB	6
17	0.3	AB	8
18	0.3	AB	8
19	0.3	AB	10
20	0.2	AB	10
21	0.9	AB	10
22	0.1	AB	10
23	0.3	AB	11
24	1.0	AB	11
25	0.0	AB	12
26	0.3	AB	12
27	0.3	AB	14
28	0.3	AB	14



Method Name: Run Name: 2014-09-08_h-c2-a Run Date: 2014-09-08 10:21

	Min	Solvents	% 2nd
29	0.0	AB	16
30	0.3	AB	20
31	0.3	AB	20
32	0.2	AB	30
33	0.1	AB	30
34	0.3	AB	50
35	0.1	AB	50
36	2.9	AB	50
37	0.2	AB	50

Vial Mapping Table

Peak#	Start Tray: Vial	End Tray Vial
1	1:2	1.2
2	13	13
3	1:4	1:4
4	1.5	1:5
5	1.6	1:6



Method Name: Run Name: KL-H-D4-D Run Date: 2015-03-25 09:01

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 19.5 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradient Table				
Min Solvents %2					
Ī	1	0.0	AB	0	
	2	1.5	AB	0	
	3	1.0	AB	45	
	4	5.0	AB	45	
	5	0.0	AB	75	
	6	1.0	AB	75	
	7	1.0	AB	100	
	8	5.0	AB	100	
	9	5.0	AB	100	

Peak#	Start Tray. Vial	End Tray Vial
1	1.2	1.2
2	13	13
3	1:4	1:4
4	1:5	1.5
5	1.6	1.6
6	1:7	1:10



Method Name: Run Name: KL-H-D4-E; 2 Run Date: 2015-03-04 10:31

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 13.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Oracient rable					
	Min	Solvents	% 2nd			
1	0.0	AB	0			
2	1.0	AB	0			
3	1.5	AB	25			
4	5.9	AB	25			
5	0.3	AB	25			
6	0.1	AB	25			
7	1.0	AB	99			
8	3.2	AB	99			
9	0.0	AB	100			

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray Vial	
1	12	1.2	
2	13	13	
3	1:4	1.5	
4	1.6	1:6	
5	1:7	1.7	
6	1.8	1.9	
7	1:10	1:10	
8	1:11	1:11	
9	1:12	1:13	
10	1:14	1:14	
11	1:15	1:15	
12	1:16	1:18	



Method Name: Run Name: KL-H-D4-E; 3 Run Date: 2015-03-04 13:59

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 16.1 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	0.3	AB	4
4	1.6	AB	4
5	1.2	AB	20
6	6.0	AB	20
7	1.0	AB	100
8	5.0	AB	100

Peak#	Start Tray Vial	End Tray Vial
1	12	1.2
2	1.3	1:4
3	1.5	1.8
4	19	1.9



Method Name: Run Name: KL-H-D2-A Run Date: 2015-01-29 15:28

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 7.4 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.5	AB	0
3	0.2	AB	5
4	0.3	AB	10
5	0.5	AB	20
6	1.0	AB	30
7	0.6	AB	36
8	0.4	AB	36
9	0.4	AB	40
10	0.8	AB	50
11	0.6	AB	60
12	0.4	AB	70
13	0.1	AB	71
14	1.1	AB	71
15	0.2	AB	30
16	0.2	AB	90
17	0.1	AB	100

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray Vial	
1	1:2	12	
2	13	13	
3	1:4	1:4	
4	1.5	1.6	
5	17	1.8	



Method Name: Run Name: KL-H-D3-A Run Date: 2015-02-01 15:47

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 3.0 min Run Length: 12.9 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 20 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.5	AB	0
3	0.3	AB	5
4	0.1	AB	5
5	0.9	AB	5
6	0.3	AB	10
7	0.4	AB	18
8	0.5	AB	18
9	0.1	AB	20
10	1.0	AB	30
11	0.2	AB	32
12	0.3	AB	32
13	0.8	AB	40
14	1.0	AB	50
15	0.5	AB	60
16	0.4	AB	70
17	0.3	AB	80
18	0.2	AB	90
19	0.1	AB	100
20	5.0	AB	100

Vial Mapping Table Peak # Start Tray Vial End Tray Vial 1 12 13 2 1:4 15 3 1.6 1.6 4 17 19 1:10 1:10 Page 1



Method Name: Run Name: KL-H-D2-C Run Date: 2015-03-17 10:33

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 20.9 min Air Purge Time: 0.5 min Slope Detection: Off ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	1.0	AB	0
3	1.0	AB	30
4	4.9	AB	30
5	79	AB	30
6	0.1	AB	30
7	1.0	AB	100
8	5.0	AB	100

Peak#	Start Tray Vial	End Tray Vial
1	12	1:3
2	1:4	1.5
3	1:6	1:10
4	1:11	1:11
5	1:12	1:19
6	1:20	1:20
7	1:21	1:21
8	1:22	1:22
9	1:23	1:23
10	1:24	1:24
11	1:25	1:30
12	1:31	1:31



Method Name: Run Name: KL-H-D2-C; #2 Run Date: 2015-03-24 08:05

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 13.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



Gradient Table Min Solvents % 2nd 0.0 AB 0 23 1.0 AB 0 1.0 AB 40 4 5.0 AB 40 100 5 1.0 AB 6 5.0 AB 100

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray Vial	
1	1.2	13	
2	1:4	1:4	
3	1.5	1:6	
4	1:7	1.7	
5	1.8	1.8	
6	19	1.9	
7	1:10	1:10	
8	1:11	1:11	
9	1:12	1:14	
10	1:15	1:15	
11	1:16	1:17	



Method Name: Run Name: KL-H-E1-C Run Date: 2014-11-21 11:13

Column: Reveleris® Silica 12g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 18.7 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 10 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	0.0	AB	10
4	2.0	AB	10
5	0.0	AB	20
6	2.0	AB	20
7	0.0	AB	30
8	0.9	AB	30
9	0.0	AB	40
10	1.7	AB	40
11	0.0	AB	50
12	1.7	AB	50
13	0.0	AB	60
14	0.9	AB	60
15	0.0	AB	70
16	0.7	AB	70
17	0.0	AB	80
18	0.5	AB	80
19	0.0	AB	90
20	0.3	AB	90
21	5.0	AB	90

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray.Vial	
1	12	12	
2	13	1.5	
3	1:6	1.7	
4	1.8	1.8	



Method Name: Run Name: KL-H-El-C Run Date: 2014-11-21 11:13

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray.Vial	
5	19	1:10	
6	1:11	1:11	
7	1:12	1:12	
8	1:13	1:14	
9	1:15	1:18	



Method Name: Run Name: KL-H-E1-C Run Date: 2014-11-21 11:13

Column: Reveleris® Silica 12g Flow Rate: 15 mL/min Equilibration: 2.4 min Run Length: 18.7 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 10 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradient Table			
	Min	Solvents	% 2nd	
1	0.0	AB	0	
2	3.0	AB	0	
3	0.0	AB	10	
4	2.0	AB	10	
5	0.0	AB	20	
6	2.0	AB	20	
7	0.0	AB	30	
8	0.9	AB	30	
9	0.0	AB	40	
10	1.7	AB	40	
11	0.0	AB	50	
12	1.7	AB	50	
13	0.0	AB	60	
14	0.9	AB	60	
15	0.0	AB	70	
16	0.7	AB	70	
17	0.0	AB	80	
18	0.5	AB	80	
19	0.0	AB	90	
20	0.3	AB	90	
21	5.0	AB	90	

Arge receipting 1 gore		
Peak#	Start Tray: Vial	End Tray Vial
1	12	1.2
2	13	1.5
3	1:6	1.7
4	1:8	1.8



Method Name: Run Name: KL-H-El-C Run Date: 2014-11-21 11:13

Vial Mapping Table		
Peak#	Start Tray Vial	End Tray: Vial
5	19	1:10
6	1:11	1:11
7	1:12	1:12
8	1:13	1:14
9	1:15	1:18



Method Name: Run Name: KL-H-F1-A2 Run Date: 2015-06-30 10:27

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 18.3 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.9	AB	0
3	10.4	AB	0
4	0.1	AB	0
5	0.0	AB	5
6	2.0	AB	5
7	0.0	AB	10
8	3.0	AB	10
9	5.0	AB	100

Vial Mapping Table		
Peak#	Start Tray: Vial	End Tray Vial
1	1.2	1.2
2	13	1:10
3	1:11	1:11
4	1:12	1:21
5	1:22	1:22
6	1:23	1:23
7	1:24	1:24
8	1:25	1:25
9	1:26	1:26
10	1:27	1:28
11	1:29	1:29
12	1:30	1:30



Method Name: Run Name: KL-H-F1-A4 Run Date: 2015-07-03 08:22

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 6.1 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



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Min Solvents % 2nd 1 0.0 AB 0 2 2.0 AB 0 3 5.0 AB 100

Vial Mapping Table		
Peak#	Start Tray Vial	End Tray.Vial
1	12	12
2	13	1.3
3	1:4	1:4
4	1.5	1.7
5	1.8	1.8
6	1.9	1.9



Method Name: Run Name: KL-H-F1-B Run Date: 2015-07-21 10:32

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 7.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradie	ent Table	
	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	0.0	AB	1
4	1.0	AB	1
5	0.0	AB	2
6	0.2	AB	2
7	0.0	AB	2
8	0.8	AB	2
9	0.0	AB	4
10	1.0	AB	4
11	1.0	AB	100

Vial Mapping Table		
Peak#	Start Tray: Vial	End Tray: Vial
1	12	12
2	13	13
3	1:4	1:4
4	1.5	1.7



Method Name: Run Name: KL-H-F1-C Run Date: 2015-11-03 07:55

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 8.9 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



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	Min	Solvents	% 2nd
1	0.0	AB	0
2	5.0	AB	0
3	0.0	AB	5
4	0.5	AB	5
5	1.0	AB	95
6	2.4	AB	95
7	0.1	AB	100

Peak # Start Tray Vial End Tray V		
1	12	13
2	1:4	1:4
3	1:5	1:5
4	1.6	1.7



Method Name: Run Name: KL-H-F2-A Run Date: 2015-07-28 07:28

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 7.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



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	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	0.0	AB	1
4	1.0	AB	1
5	0.0	AB	2
6	1.0	AB	2
7	0.0	AB	4
8	1.0	AB	4
9	1.0	AB	100

Vial Mapping Table		
Peak#	Start Tray: Vial	End Tray Vial
1	12	1.2
2	13	13
3	1:4	1:4
4	1.5	1.5
5	1.6	1.6
6	1:7	1.7
7	1.8	1.8
8	1.9	1.9
9	1:10	1:11
10	1:12	1:12


Method Name: Run Name: KL-H-F2-B Run Date: 2015-08-10 16:57

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 10.0 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradient Table				
	Min	Solvents	% 2nd		
1	0.0	AB	0		
2	1.2	AB	0		
3	3.0	AB	0		
4	1.8	AB	0		
5	0.0	AB	1		
6	1.0	AB	1		
7	0.0	AB	2		
8	1.0	AB	2		
9	0.0	AB	4		
10	1.0	AB	4		
11	1.0	AB	100		

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray.Vial	
1	12	12	
2	13	13	
3	1:4	1:4	
4	1.5	1.5	
5	1.6	1.6	
6	1:7	19	



Method Name: Run Name: KL-H-F2-B2 Run Date: 2015-08-25 14:27

Column: Reveleris® Silica 40g Flow Rate: 40 mL/min Equilibration: 7.1 min Run Length: 11.4 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	2.7	AB	0
3	4.9	AB	0
4	0.3	AB	0
5	0.0	AB	1
6	1.0	AB	1
7	0.0	AB	2
8	1.0	AB	2
9	0.0	AB	4
10	0.5	AB	4
11	1.0	AB	100

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray: Vial	
1	1.2	13	
2	1:4	1:4	
3	1.5	1:5	
4	1.6	1.6	
5	1:7	1.7	
6	1.8	1.8	
7	1.9	1.9	
8	1:10	1:10	
9	1:11	1:14	



Method Name: Run Name: KL-H-F2-B3 Run Date: 2015-08-27 11:12

Column: Reveleris® Silica 40g Flow Rate: 40 mL/min Equilibration: 7.1 min Run Length: 7.9 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.4	AB	0
3	1.9	AB	0
4	0.6	AB	0
5	0.0	AB	1
6	1.0	AB	1
7	0.0	AB	2
8	0.5	AB	2
9	0.5	AB	100

Vial Mapping Table			
Peak#	Start Tray: Vial	End Tray Vial	
1	12	1.2	
2	13	13	
3	1:4	1:4	
4	1.5	1.5	
5	1.6	1.6	
6	1:7	1.8	



Method Name: Run Name: KL-H-F2-B4 Run Date: 2015-08-27 11:30

Column: Reveleris® Silica 40g Flow Rate: 40 mL/min Equilibration: 7.1 min Run Length: 7.4 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	6.0	AB	0
3	0.0	AB	1
4	0.5	AB	1
5	0.0	AB	2
6	0.5	AB	2
7	0.4	AB	100

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray Vial	
1	12	1.2	
2	13	13	
3	1:4	1:4	
4	1.5	1.5	
5	1.6	17	



Method Name: Run Name: KL-H-F3-A Run Date: 2015-07-13 10:49

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 8.8 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradient Table			
	Min	Solvents	% 2nd	
1	0.0	AB	0	
2	2.8	AB	0	
3	1.8	AB	0	
4	0.2	AB	0	
5	0.0	AB	1	
6	1.0	AB	1	
7	0.0	AB	2	
8	1.0	AB	2	
9	0.0	AB	4	
10	1.0	AB	4	
11	1.0	AB	100	

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray.Vial	
1	12	12	
2	13	1:3	
3	1:4	1:4	
4	1.5	1:6	



Method Name: Run Name: KL-H-F4-B Run Date: 2015-11-03 07:36

Column: Reveleris® Silica 12g Flow Rate: 30 mL/min Equilibration: 4.8 min Run Length: 14.3 min Air Purge Time: 0.5 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Dry ELSD Carrier: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradient Table				
ſ		Min	Solvents	% 2nd	
ľ	1	0.0	AB	0	
l	2	13.3	AB	0	
l	3	1.0	AB	100	

Vial Mapping Table			
Peak#	Start Tray Vial	End Tray: Vial	
1	12	12	
2	13	13	
3	1:4	1:4	
4	1.5	1.6	



Method Name: Run Name: KL-H-F2-E Run Date: 2015-10-25 08:27

Column: Reveleris® Silica 40g Flow Rate: 40 mL/min Equilibration: 7.1 min Run Length: 15.5 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 5 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer. Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chose Solvent D: <No solvent chose



1-7B19



	Gradie	ent Table	
	Min	Solvents	% 2nd
1	0.0	AB	0
2	3.0	AB	0
3	4.9	AB	0
4	0.0	AB	0
5	1.0	AB	5
6	1.0	AB	10
7	0.1	AB	14
8	2.3	AB	14
9	2.1	AB	75
10	0.3	AB	75
11	0.8	AB	100

	Vial Mapping	Table
Peak#	Start Tray: Vial	End Tray:Vial
1	1.2	12
2	13	13
3	1:4	1:5
4	1:6	1.6
5	1:7	1.7
6	1.8	1.8
7	1.9	1.9
8	1:10	1:10
9	1:11	1:11
10	1:12	1:12
11	1:13	1:13
12	1:14	1:14
13	1:15	1:15
14	1:16	1:16



Method Name: Run Name: KL-H-F2-E Run Date: 2015-10-25 08:27

Peak#	Start Tray.Vial	End Tray: Vial					
15	1:17	1:17 1:18					
16	1:18	1:18					
17	1:19	1:19					
18	1:20	1:20					
19	1:21	1:21					
20	1:22	1:22					
21	1:23	1:23					
22	1:24	1:24					
23	1:25	1:25					
24	1:26	1:26					
25	1:27	1:27					
26	1:28	1:28					
27	1:29	1:29					
28	1:30	1:30					
29	1:31	1:31					
30	1:32	1:33					
31	1:34	1:35					
32	1:36	1:36					



Method Name: Run Name: KL-H-F2-E2 Run Date: 2015-10-26 06:25

Column: Generic Silica 40g Flow Rate: 40 mL/min Equilibration: 6.0 min Run Length: 19.7 min Air Purge Time: 1 min Slope Detection: High ELSD Threshold: 20 mV UV Threshold: 0.02 AU UV1 Wavelength: 254 nm UV2 Wavelength: 280 nm Collection Mode: Collect Peaks Per-Vial Volume: 25 mL Non-Peaks: 25 mL Injection Type: Manual ELSD Camer: Iso-propanol Solvent A: Hexane Solvent B: Ethyl acetate Solvent C: <No solvent chosen> Solvent D: <No solvent chosen>



1-7B19



	Min	Solvents	% 2nd
1	0.0	AB	0
2	0.5	AB	0
3	0.9	AB	0
4	2.5	AB	0
5	0.0	AB	5
6	2.0	AB	5
7	0.0	AB	10
8	1.6	AB	10
9	7.7	AB	10
10	0.4	AB	10
11	2.8	AB	94
12	1.2	AB	94
13	0.2	AB	100

	Vial Mapping	Table
Peak#	Start Tray Vial	End Tray Vial
1	1:2	1:2
2	13	13
3	1:4	1:4
4	15	1.5
5	1.6	1.6
6	1:7	1.7
7	1.8	1.8
8	19	19
9	1:10	1:10
10	1:11	1:11
11	1:12	1:14
12	1:15	1:15



Method Name: Run Name: KL-H-F2-E2 Run Date: 2015-10-26 06:25

Peak#	Start Tray. Vial	ial End Tray: Vial					
13	1:16	1:16					
14	1:17	1:17					
15	1:18	1:18					
16	1:19	1:19					
17	1:20	1:20					
18	1:21	1:21					
19	1:22	1:22					
20	1:23	1:23					
21	1:24	1:29					
22	1:30	1:30					
23	1:31	1:31					

APPENDIX B

NMR ANALYSIS REPORTS

Carbon NMR



		KL-H-F2-A			
Agilent Technolog	ies	Sample Name KL-HFE2.0 Date collected 2015-07-28	Pulse sequence CARBON Solvent cdcl3	Temperature 28 Spectrometer agilent-NMR-vnmrs400	Study owner weldon Operator weldon
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Study owner weldon Operation weldon

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Study owner weldon Operator weldon

Temperature 28 Spectometer aglient NMR-wmro400

Putse sequence CARBON Solvent adal3

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Study owner weldon Operator weldon

Temperature 28 Spectometer aglient MMR-ummro400

Pulse sequence CARBON Solvent adal2

Sample Name KL-H-F2-E Date collected 2016-10-29

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Tiels         Tiels <th< td=""><td>R8     H     H     H     H       R8     H     H     H     H       S88.75     S88.75     G     G       S88.75     S88.75     G     G       S88.75     G     G     G       S88.75     G     G     G       S88.75     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     &lt;</td><td>Turn         In         Turn           H         H         H         H           H         H         In         In           RB/2         R         In         In           SB/2         R         In         In           SB/2         R         In         In           SB/2         R         In         In           SB/2         R         S         S           SB/2         R         S         S           SB/2         R         S         S           In         S         S         S         S           ML         Note         S         S         S         S           S1         N         S         S         S         S         S           S1         N         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S&lt;</td><td>82</td><td>= 1</td><td>=  </td><td></td><td></td><td></td><td></td></th<>	R8     H     H     H     H       R8     H     H     H     H       S88.75     S88.75     G     G       S88.75     S88.75     G     G       S88.75     G     G     G       S88.75     G     G     G       S88.75     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     G     G     G       G     <	Turn         In         Turn           H         H         H         H           H         H         In         In           RB/2         R         In         In           SB/2         R         In         In           SB/2         R         In         In           SB/2         R         In         In           SB/2         R         S         S           SB/2         R         S         S           SB/2         R         S         S           In         S         S         S         S           ML         Note         S         S         S         S           S1         N         S         S         S         S         S           S1         N         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S<	82	= 1	=				
TER     Procession       H1     1       R8.767     R0.428100       888.767     Contract       888.767     Contract       888.767     Contract       889.767     Contract       99.168     80.8       10     0       10     0       10     0       10     10       10     23.4       10     10       10     23.4       10     10       10     23.4       10     10       10     10	RR     RS     R     N       RR     H     H     H     H       RR     H     H     H     H       RR     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R     R       R     R     R     R   <	FROCERNIKO         M           RR.767         RR.767         Intucat           SR8.767         Intucat         SR8.767           SR8.767         Intucat         SNA.77	000.L	⊆ 문	c :				
TER         Processing           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           68         560           68         563           68         564.2           68         564.2           61         848.3           7         48.3           7         48.3           84.2         28.4           84.2         28.4           84.2         28.4           94.2         94.2           10         10           10         10           10         10           10         28.4           10         28.4           10         10           10         10           10         10           10         10	FROCERING         PROCERING           388.77         0.0 FLAY           389.77         0.0 FLAY           56.00         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           6.60         9           7         9           7         9           8         9           8         9           8         9           8         9           8         9           8         9           8         9           8         9           8         9	PROCERTINO           888.75         100 motored           888.75         00 FLAY           888.75         00 FLAY           888.75         00 FLAY           660         29           661         29           661         29           662         29           663         29           664         200.2           664         200.2           664         200.2           7         200.2           7         20.4           7         20.4           84         00           84         00           84         00           84         00           84         00           84         00           84         00           84         00           84         00           85         28           85         28           85         28           85         28           85         26           85         56           85         56           85         56           85         56	9 69	7 2	. 5				
TTER         Processino           388.75         388.75           388.75         388.75           388.75         388.75           388.75         388.75           66         66           68         508.2           68         508.2           68         508.2           6.660         59           6.660         59           70         78.4           71         208.2           71         208.3           71         208.4           72         72           73         44           75         24.4           75         24.4	FROCERINO           RR. 14 88.757 88.757 88.757 88.757 88.757 66         1           RR. 16 66         8           88         9           88         9           88         9           88         9           88         9           88         9           88         9           88         9           9         9           9         9           19         9           19         9           19         9           19         10           19         10           19         10           19         10           19         234           2412         234           10         10           10         10           10         10           10         234           10         24           10         24           10         10           10         10           10         24           10         24           10         24	PROCENSING         Processing           11         1         nrtuced           288.7         288.7         Captured           288.7         288.7         Captured           288.7         0471.4         nrtuced           6.660         9         308.3           6.661         9         308.3           6.661         9         308.3           11         208.4         208.4           11         208.4         0           11         28.3         0           11         9         28.4           28.1         0         28.4           28.1         0         28.4           28.1         0         28.4           28.2         0         28.4           28.2         0         28.4           11         10.1         10.1           11         4.4         4.4           11         11.1         4.4           11         11.1         4.4           11         11.1         4.4           11         11.1         4.4           11         11.1         4.4							
H         1         1           387.57         287.57         287.57           889         389.5         100.000           899         399         508.2           660         39         508.2           6.60         39         508.2           6.61         39         508.2           6.62         39         408.8           7         41         308.4           8         41         38.4           9         9         48.8           9         9         48.8           9         9         48.8           9         9         48.8           1         9         1           1         9         1           84.1         9         28.4           9         9         28.4           9         55         38.4           1         1         1           1         1         1           1         1         1           28.4         9         28.4           1         1         1           1         1         1           1 <t< td=""><td>H1         1         notuced           88         556         39         500.2           69         500.2         500.2         500.2           69         500.2         500.2         500.2           69         500.2         500.2         500.2           70         500.2         500.2         500.3           70         1         200.2         200.3           70         1         200.3         200.3           71         1         200.3         200.3           71         1         200.3         200.4           73         24.1         200.1         200.4           74         25.4         25.4         25.4           74         26.5         20.4         20.4</td><td>H1         1         netword           288.757         288.757         288.757           889         288.7         288.7           889         388.4         208.2           6.660         39         508.2           6.61         39         508.2           6.61         39         508.2           6.61         39         648.8           19         818.8         818.8           19         48.2         0           19         48.2         0           284.1         000         284           284.2         000         284           34.1         000         284           31.5         000         284           31.5         000         284           31.5         000         284           31.5         000         284           31.5         44         44           280.000         1.1_5104.01         44</td><td>BMITTER</td><td>PROC EE BINO</td><td></td><td></td><td></td><td></td><td></td></t<>	H1         1         notuced           88         556         39         500.2           69         500.2         500.2         500.2           69         500.2         500.2         500.2           69         500.2         500.2         500.2           70         500.2         500.2         500.3           70         1         200.2         200.3           70         1         200.3         200.3           71         1         200.3         200.3           71         1         200.3         200.4           73         24.1         200.1         200.4           74         25.4         25.4         25.4           74         26.5         20.4         20.4	H1         1         netword           288.757         288.757         288.757           889         288.7         288.7           889         388.4         208.2           6.660         39         508.2           6.61         39         508.2           6.61         39         508.2           6.61         39         648.8           19         818.8         818.8           19         48.2         0           19         48.2         0           284.1         000         284           284.2         000         284           34.1         000         284           31.5         000         284           31.5         000         284           31.5         000         284           31.5         000         284           31.5         44         44           280.000         1.1_5104.01         44	BMITTER	PROC EE BINO					
38/15/ 88/2         38/15/ 88/2           88/1         08/15/ 88/1           89/1         08/15/ 1           89/1         08/15/ 1           1         208/1           1         208/1           1         208/1           1         208/1           1         208/1           1         201           1         201           1         201           2         40           2         234           1         1           2         234           1         234           1         234           2         234           2         234           1         234           1         234           1         234           1         234           1         234           2         234           3         3           3         3           3         3	387.57         388.7         388.7           888.7         389.4         380.2           6.660         39         300.2           10         39         300.2           10         10         200.2           11         200.3         300.4           12         1         200.3           24.1         0         1           24.1         0         234.4           24.3         234.4         234.4           13         101         101           14         5.5         2           15         5.5         5.4           16         23.4         23.4           16         23.4         23.4           16         23.4         23.4	387.57         388.75           389.7         08.14           389.7         08.14           389.7         08.14           6.66         59         308.3           6.66         59         308.3           1         10         308.4           1         10         308.3           1         10         308.4           1         10         48.3           1         10         48.3           1         10         48.3           34         10         10           34         10         10           34         10         234           34         10         234           35         1         44           1         1         44	Ŧ	¢	notu ced				
88.7         0.8 P.I.A.V           6.6         4           6.5         5           6.6         4           1         20.3           1         8.00.3           1         8.00.3           1         8.00.3           1         8.00.3           1         1           1         8.00.4           1         1           1         1           2         4           24.13         4           24.14         1           24.15         1           24.15         1           24.15         1           25.4         23.4           1         1           1         1           1         1           1         1           1         2           1         2           1         2           1         2           1         2           1         2           2         2           2         2           2         2           2         2           3	288.7         CIBFLAV           6660         5	288.7         GEPLAV           66         29         208.2           6.60         29         208.3           1         1         208.4           1         208.4         408.8           1         1         208.4           1         208.4         408.4           1         1         208.4           1         1         208.4           1         1         20.4           1         1         20.4           1         1         20.4           24.1         10         22.4           24.2         10         23.4           24.2         10         23.4           25         25         25           1         10.1         4.4           25         25         25           1         10.5         4.4           26.0         27.4         4.4           26.0         27.4         4.4           26.0         27.4         4.4           26.0         27.4         4.4           27.5         27.5         27.5           27.5         27.5         27.5	389.767						
66         30.8.2           6.60         00         30.8.2           7         7         30.8.2           7         7         30.8.2           7         7         30.8.2           7         7         30.8.3           8         1         20.8.3           9         9         40.5           84         9         48.3           84         9         9           84         9         9           84         9         9           84         9         1           84         9         1           84         9         1           84         9         234           9         1         1           1         1         1           1         1         1           1         1         1	660         201.2           1         201.2           1         201.3           1         201.4           1         201.4           1         1           2         2           2         3           2         3           2         3           3         4           3         4           3         4           3         4           3         4           3         4           3         4           3         4	6.60         20         2014.3           0.11         0.11         2014.3           0.12         0.11         2014.3           0.13         0.11         21.4           0.14         0.11         21.4           0.12         0.12         23.4           0.14         0.11         23.4           0.12         0.12         23.4           0.12         0.12         23.4           0.12         0.12         23.4           0.12         0.12         23.4           0.12         0.12         23.4           0.12         0.12         23.4	288.7	CIBPLAY					
Fill         500 and 200.2           C18         10         400.3           C18         10         800.4           0         1         20.4           0         1         -         -           0         1         -         -         -           0         1         -         -         -           0         1         -         -         -           24         0         -         -         -           24.1         0         -         -         -           24.2         0         -         -         -           24.1         0         -         -         -           24.2         0         -         -         -           24.3         0         -         -         -           24.5         5         -         -         -           1         -         -         -         -         -           24.1         -         -         -         -         -           1         -         -         -         -         -           1         -         -	Mathematical         Mathematical         Mathematical           01         0         6401.8         6401.8           01         0         6401.8         6401.8           01         0         6401.8         6401.8           01         0         6401.8         8401.8           01         0         6401.8         8401.8           01         0         6401.8         853.8           02         34         0         833.8           034.13         0         6401.8         0           11         1         1         1           12         1         1         1           13         1         1         1           14         1         1         1	Field         SP         -408.2           1         9         -408.3           1         9         -408.4           1         9         -408.4           1         9         -408.4           1         9         -408.4           1         9         -48.3           1         9         -48.3           1         9         -48.3           2         -48.3         -44           2         -28.4         -28.4           2         -28.4         -28.4           3         -00         -28.4           5         -28.4	89						
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Study owner w**eldon** Operator w**eldon** 

Temperature 28 Spectometer aglient NM R-ummr400

Putse sequence PROFION Solveni adal3

Sample Name KL-H-F4-B-6 Date collected 2016-11-03

KL-H-F4-B4



0ab 1c mome/LW AmmisysAbabAvebon/KL-H-F+8-6_20151100_01/PR0 T0 N_01:M

KL-H-F4-B4

**bab 1**2 from eVLUI /htmnsys/diable/web/on/KL-H-1++8+6_20151100_01/PR0_T0_N_01.4d



Data is mome/LUI Ammisys Adata Wethon/KL-H-Fi-O-3_20151102_01/FROTON_01.1d

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Temperature 28 Bauty correr weiden Spectrometer aglient KMR-wimmradig Operator weiden

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KL-H-F1-C-3 Sample Name KL-H-F1-C-3 Dale oxiected 2016-11-03



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Ci     Fig     Nin       FAMEMITTER     FeoceBBINO       Name     BB       Name     BB       Sin     288,757       Lin     288,757       Lin     288,757       Lin     10       Sin     39       Lin     39       Lin     310       Lin     310       Lin     310       Lin     310.43       Lin     11       Lin     310.43       Lin     11       Lin     200.43       Lin     11       Lin     11       Lin     200.43       Lin	I         R         R         N         N           RAM BMITTER         PROCESSING         PROCESSING         N           RAM SMITTER         PROCESSING         PROCESSING         N           R         SS         CIRPLAY         PROCESSING           N         SS         CIRPLAY         N           N         SS         CIRPLAY         SIR           N         SS         SIR         SIR           N         SS         SIR         SIR           N         SS         SIR         SIR           N         CIRPLAY         SIR         SIR           N         SIR         SIR         SIR           N         <	
Texemitter         Peocelation           h         h         h         Peocelation           h         stars         stars         h         h         hourd           h         stars         stars         contract         contract         contract         contract           h         stars         contract         contract         contract         contract         contract           h         contract         contract <td< td=""><td>FARBUITTER         PROCERBINO           1         1         1         1           1         288.75         CHEPLAY         1           1         286.7         CHEPLAY         2           1         286.7         CHEPLAY         2           1         286.7         CHEPLAY         2           1         286.7         2         2           1         286.2         2         2           1         2         2         2           1         2         2         2           1         1         2         2         2           1         1         2         2         2           1         1         2         2         2         2           1         1         2         2         2         2           1         1         1         1         1         2           1         2         2         2         2         2           1         2         2         2         2         2           1         2         2         2         2         2           1         <td< td=""><td>Culo 0000 0011100 000</td></td<></td></td<>	FARBUITTER         PROCERBINO           1         1         1         1           1         288.75         CHEPLAY         1           1         286.7         CHEPLAY         2           1         286.7         CHEPLAY         2           1         286.7         CHEPLAY         2           1         286.7         2         2           1         286.2         2         2           1         2         2         2           1         2         2         2           1         1         2         2         2           1         1         2         2         2           1         1         2         2         2         2           1         1         2         2         2         2           1         1         1         1         1         2           1         2         2         2         2         2           1         2         2         2         2         2           1         2         2         2         2         2           1 <td< td=""><td>Culo 0000 0011100 000</td></td<>	Culo 0000 0011100 000
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STR         288.75         DBPLAY           bur         6.60         29         203.1           put         6.60         29         203.1           DBCOUPLER         11         203.2         203.1           dn         C13         11         203.3           dn         C13         11         203.4           dn         C13         11         203.4           dn         C13         12         203.4           dn         C13         12         46.0           dn         C13         12         46.0           dn         C14         0         0           dn         C15         23.4         23.4           dn         23.4         23.4         23.4           dn         23.4         23.4         23.4           dn         C17         14         44.1           dn         C17         23.4         23.4           dn         C17         23.4         23.4           dn         C17         23.4         23.4           dn         C17         24.4         24.4           dn         C17         24.4         24.4	TI         288.75 288.75         CIGPLAY           0         68         200.4           0         0         9         400.8           0         0         9         400.8           0         0         9         400.8           0         0         9         400.8           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         1         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0         0         0           0         0	H H notuced
MI         SEC.         ULLIAN           PU         5.66         20.2           PDCOUPLER         M         200.2           PDCOUPLER         M         40.0           PDC         M         1           PDC         M         1           PDC         284.2         284.2           PDC         284.2	M         BB         MILLIN           W         6.60         3p         -203.2           MODULER         11         -203.2         -203.2           MODULER         11         -203.2         -203.2           MODULER         11         -203.2         -203.2           MODULER         11         -203.2         -203.4           MODULER         12	
With State         State         State           DECOUPLER         UP         400.8           UP         200.2           UP         20.1           UP         4.4           UP         4.4	M         6.660         5	
DECOLIFIER         UP         640.8           In         1         200.4           In         1         200.4           In         1         200.4           In         1	HOULPLER         Up         640.8           n         1         20.4           n         1         20.4           n         1         20.4           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         341         1           n         342         1           n         343         1           n         1         4           n         1         4           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1         1           n         1	v 6.660 so 308.2
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Tendents     Method     Method     Method     Method     Method       Image: State in the		<u> </u>	Sample Name KL-H-B2-H_2 Date collected 2016-11- 7	Pulse sequence PROTON Soluent adaits	Temperature 28	Study owner weldon
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4L         PRENT DENT OF	All         Medical Induction           1         Induction <t< td=""><td>IT         REBAY LENAL           IT         REBAY LENAL           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1</td><td>PE         PERKIT BALTON           PL         PERKIT BALTON</td><td>Result (MI (MI (MI (MI (MI (MI (MI (MI (MI (MI</td><td></td><th></th><td><u>ب</u></td><td></td><td></td><td></td><td></td><td></td></t<>	IT         REBAY LENAL           IT         REBAY LENAL           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.734           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1)         0.0.744           (1	PE         PERKIT BALTON           PL         PERKIT BALTON	Result (MI			<u>ب</u>					
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IE         PREAK LEARING           1         PREAK LEARING           1         In or (19) (6)           1         In or (19) (10)           1         In or (10) (10)           1         In or (10) (10)           1         In or (10) (10) (10) (10) (10) (10) (10) (10)	IE         FEENT LENT ON           1         Detext LENT ON           1         Detext LENT ON           Annon (LLT) and Annon	Intelline         Present Intelline           Intelline         Present Intelline           Intelline         Intelline	FE         PREMIX LENTION           PIE         PREMIX LENTION           (nonvirtuo)         manoni (1,0,munuo)           (nonvirtuo)         man	IE         PREMI LIGATON           1         No 0' 13 (6)         PREMI LIGATON           1         10 (0) (1)         PREMI LIGATON           1         10 (0) (1)         PREMI LIGATON           1         10 (0) (1)         PREMI LIGATON           1         10 (0)         PR								
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101         1.000         1.000</td> <td>Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt       Initial Jurgeorou Litt     Initial Jurgeorou Litt     Initial Jurgeorou Litt</td> <td>Introduction     Introduction       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Matrix         Matrix         Matrix           2,5413         2,5413         1,1100           2,5413         2,541         1,100           2,5413         1,100         1,100           1,100         1,100         1,100           1,100         1,100         1,100           1,100         1,100         1,100           1,100         1,100         1,100           2,241         Procession         2,043           2,2413         1,00         2,043           2,100         2,043         2,043           2,100         2,043         2,043           2,101         1,01         2,043           2,101         1,01         2,043           2,101         1,01         2,043           2,101         1,01         2,043           2,101         1,01         2,043           2,111         1,01         2,043           2,111         1,01         2,043           2,111         1,01         2,043           2,111         1,01         2,043           2,111         1,01         2,043           2,111         1,01         2,043           <	Million         Million         Million         Million           2,543         2,543         1,100           2,543         2,543         1,100           2,543         1,100         1,100           2,543         1,000         1,100           4401         1,000         1,100           2,243         1,000         1,000           2,243         1,000         1,000           2,243         1,000         1,000           2,243         0,000         2,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000           2,100         1,000         1,000	Control         Picol         1, cont           2.646         1, cont         1, cont           2.640         1, cont         1, cont           2.640         1, cont         1, cont           2.640         2, cont         2, cont           2.640         2, cont         2, cont           2.640         1, cont         2, cont           2.640         1, cont         2, cont           2.641         1, cont         2, cont <td< td=""><td>Maintonic         Part (a) (b) (b) (c) (c) (c) (c) (c) (c) (c) (c) (c) (c</td><td>Introv         PCI         Lun           2,566         F,106         1,100           2,566         F,106         1,000           4000         1,000         1,000           4000         1,000         1,000           4000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000</td><td>CITIE III</td><th></th><td>- hel</td><td></td><td></td><td></td><td></td><td></td></td<>	Maintonic         Part (a) (b) (b) (c) (c) (c) (c) (c) (c) (c) (c) (c) (c	Introv         PCI         Lun           2,566         F,106         1,100           2,566         F,106         1,000           4000         1,000         1,000           4000         1,000         1,000           4000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000         1,000         1,000           1,000	CITIE III		- hel					
9413         3560         1000           2660         1         0           2010         1         0           2010         1         0           2010         1         0           2010         1         0           2010         1         0           2010         1         0           2011         1         0           2011         10         0           2012         2014         0           2014         1         0           2014         1         0           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014           2014         2014         2014 <td>440.3         Three         Three         Three   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2718         FLAG         0.00           2718         FLAG         0.00           2718         FLAG         0.00           2100         1         1           1000         10         1           1000         10         1           280,7         FLAG         1           1000         10         1           280,7         FLAG         1           281,8         FLAG         1           281,7         FLAG         1           281,7         FLAG         1           281,7         FLAG</td><td>Meta         Meta         Mata           2.568         FLAGE           2.568         FLAGE           2.568         FLAGE           2.000         II         II           2.000         II         II           2.000         II         III           2.000         II         IIII           2.001         IIIIII         IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII</td><td>440.3         Mode Signation (100         Mo</td><td></td><th></th><td>101</td><td></td><td></td><td></td><td></td><td></td></td>	440.3         Three         Three         Three           2568         11         0         1           200         10         1         0           201         1         0         1           202         1         0         1           203         1         0         1           203         1         0         0           203         1         1         0           203         1         1         0           203         203         203         203           203         203         203         203           203         203         203         203           203         203         203         203           203         203         203         203           204         0         0         203           204         0         0         203           204         0         0         203           204         0         0         204           204         0         0         204           204         0         0         204           204         0 </td <td>M102         M102         M101           2168         FLAG         0.00           2718         FLAG         0.00           2718         FLAG         0.00           2718         FLAG         0.00           2100         1         1           1000         10         1           1000         10         1           280,7         FLAG         1           1000         10         1           280,7         FLAG         1           281,8         FLAG         1           281,7         FLAG         1           281,7         FLAG         1           281,7         FLAG</td> <td>Meta         Meta         Mata           2.568         FLAGE           2.568         FLAGE           2.568         FLAGE           2.000         II         II           2.000         II         II           2.000         II         III           2.000         II         IIII           2.001         IIIIII         IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII</td> <td>440.3         Mode Signation (100         Mo</td> <td></td> <th></th> <td>101</td> <td></td> <td></td> <td></td> <td></td> <td></td>	M102         M102         M101           2168         FLAG         0.00           2718         FLAG         0.00           2718         FLAG         0.00           2718         FLAG         0.00           2100         1         1           1000         10         1           1000         10         1           280,7         FLAG         1           1000         10         1           280,7         FLAG         1           281,8         FLAG         1           281,7         FLAG         1           281,7         FLAG         1           281,7         FLAG	Meta         Meta         Mata           2.568         FLAGE           2.568         FLAGE           2.568         FLAGE           2.000         II         II           2.000         II         II           2.000         II         III           2.000         II         IIII           2.001         IIIIII         IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	440.3         Mode Signation (100         Mo			101					
3610         3710         4010           2569         F.4.04         0           4000         1000         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         10         1           1000         10         1           100         10         1           100         10         1           100         10         1           100         10         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1           10         1         1 </td <td>36103         313         313         4000           25783         FLAGE         1         1      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    4001         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1         1           100         1         1         1         1         1         1           100         1         1         1         1         1         1           100         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1         <t< td=""><td>5413         513         1100           2,518         FLAGE         1           2,001         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000</td><td>5413         343         1000           2,6413         10         100           4003         10         1           1001         1         1         1           1001         1         1         1         1           1001         1         1         1         1         1           1001         1         1         1         1         1         1           1001         10         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1</td><td></td><th></th><td>namd</td><td></td><td></td><td></td><td></td><td></td></t<></td>	36103         313         313         4000           25783         FLAGE         1         1           1000         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1000         1         1         1           1000         1000         1000         1000         1000           100         1         1         1         1         1           100         1         1         1         1         1         1           100         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1         1           100         1         1         1         1         1	34013         313         1010           2,508         F.4068         0           4001         10         1           4001         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1         1           100         1         1         1         1         1         1           100         1         1         1         1         1         1           100         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1         1         1           100         1         1         1         1         1         1         1 <t< td=""><td>5413         513         1100           2,518         FLAGE         1           2,001         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000</td><td>5413         343         1000           2,6413         10         100           4003         10         1           1001         1         1         1           1001         1         1         1         1           1001         1         1         1         1         1           1001         1         1         1         1         1         1           1001         10         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1</td><td></td><th></th><td>namd</td><td></td><td></td><td></td><td></td><td></td></t<>	5413         513         1100           2,518         FLAGE         1           2,001         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000         1         1           1,000	5413         343         1000           2,6413         10         100           4003         10         1           1001         1         1         1           1001         1         1         1         1    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   1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1           1.01         1         1	2168 400 100         F.Ade           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101    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2018         FLAOB           1         1         1           1         1         1         1           1         1         1         1         1           1         1         1         1         1         1           1         1         1         1         1         1         1           1         1         1         1         1         1         1           1         1         1         1         1         1         1           288.77         081.4         1         1         1         1         1           288.76         081.4         1         1         1         1         1           288.77         081.4         1         1         1         1         1           288.76         081.4         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1 <td>4000 200         FLoole           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1     &lt;</td> <td>4001         FLAOB           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100</td> <td>2018         F.A.064           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           101         1         1         1           101         1         1         1           101         1         1         1           101         1         1         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1</td> <td>4010 101         FL008           100         10         1           100         10         1           100         10         1           100         10         1           100         10         1           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         <td< td=""><td></td><th>2.668</th><td></td><td></td><td></td><td></td><td></td><td></td></td<></td>	4000 200         FLoole           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         200.2           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1     <	4001         FLAOB           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100	2018         F.A.064           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           101         1         1         1           101         1         1         1           101         1         1         1           101         1         1         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1           101         1         4         1	4010 101         FL008           100         10         1           100         10         1           100         10         1           100         10         1           100         10         1           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10         10           100         10 <td< td=""><td></td><th>2.668</th><td></td><td></td><td></td><td></td><td></td><td></td></td<>		2.668						
400         1         1           1000         1         1         1           1000         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100         1         1         1         1           100<	400         1         1           1.000         1         1           1.000         1         1           1.000         1         1           1.000         1         1           1.000         1         1           1.000         1         1           1.000         1         1           1.001         1         1           1.001         1         1           1.001         1         1           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2           1.001         1         200.2	400         1         0           1.000         1         1         1           1.000         1         1         1           1.000         1         1         1         1           1.000         1         1         1         1         1           1.000         1         1         1         1         1         1           1.000         1         1         1         1         1         1         1           1.000         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1 <t< td=""><td>400         1         0           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           102         1         200.2         200.2</td><td>400         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1</td><td></td><th>22782</th><td>FL AGB</td><td></td><td></td><td></td><td></td><td></td></t<>	400         1         0           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           1002         1         1         1           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           101         1         200.2         200.2           102         1         200.2         200.2	400         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1           1000         1         1		22782	FL AGB					
1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         100         1000           1000         100         1000           1000         100         1000           1000         100         1000           1000         100         1000           1000         100         1000           1000         100         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000           1000         1000         1000	100         1         1           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        1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1</td> <td>1000         1         0         0           1000         10         0         1           1000         10         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           1000         1         1         1         1           100         1         5         1         1           100         1         5         1         1         1           100         1         5         1         1         1         1           100         1         5         1         5         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1</td> <td>1000         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           1001         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           101         1         1           102         2         2           103         1         1           104         1           10</td> <td></td> <th>884</th> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	1000         10         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1         1           100         1    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1000     1     1       14111ER     1     1       18111ER     PROCEIBINO       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1       18111     1	100         1,100         1,100         1,100           1411TTER         PROCERINO         n         n           1814TTER         PROCERINO         1         n           1814TER         1         1         n         n           1814TER         1         1         1         n         n           1914TER         1         1         1         1         1         1           1914TER         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1	100         1,0         1,0         1,0         1,0           KIMITTER         POCCERINO	100         1.0         1.0           4MITTER         PROCERIMO           4MITTER         PROCERIMO           3R8.767         REPLAY           SR8.767         CRELAR           SR8.8         CRELAR	100         10         1           IMITTER         PROCERIMO         7           IMITTER         PROCERIMO         7<		22	=	=				
8         10         1           10111ER         POC EBINO         1           11         1         1         1           288.15         288.15         201         201           288.15         288.15         201         201           888.16         388.16         201         201           888.16         201         201         201           88         66         306.2         306.2           90         308.2         308.2         308.2           91         1         201         201.2           1         1         201.2         201.2           21         2         2         2           21         1         1         1           21         1         1         1           21         1         2         2         2           21         1         2         2         2           21         1         1         1         1           21         1         1         1         1           21         1         1         2         2           21         1         2	BINITER         Procession         N           IBINITER         Procession         N           BINITER         BINITER         N           BINITER         BINITER         N           BINITER         BINITER         BINITER	2         00         7           MBNITER         Processino         7           MBNITER         Processino         7           SB, H         Processino         200.2           CIS         Processino         200.2           ML, Holding         Processino         200.2           ML, Holding         Processino         200.2           ML, Holding         Processino         200.2           ML, Holding         Processino         200.2	2         20         0         7           IMITTER         PROCERNINO         PROCERNINO         PROCERNINO           SRR.XF         PROCERNINO         PROCERNINO         PROCENCINO           SRR.MF         PROCENCINO         PROCENCINO         PROCENCINO           SRR.MF         PROCENCINO         PROCENCINO         PROCENCINO	2         00         7           IMITTER         Procession         7           IMITTER         Procession         7           SRF57         Excession         7           SRF57         Custon         5           Coston         Custon         5           SRF57         Custon         5           Custon         Custon         5           SRF57         5         5		1.000	Ē	=				
R         R         N           IMITTER         PROCERNIKO         PROCERNIKO           SR87.57         SR87.57         N           SR87.57         SR87.64         PROCERNIKO           SR87.7         SR87.64         M           SR87.7         SR87.64         SR87.64           SR87.64         SR87.64         SR87.64           SR87.7         GR81.44         SR87.64           SR87.7         GR81.45         SR87.64           SR87.64         SR8.75         SR87.74           SR87.7         GR81.84         SR87.84           OT         SR8.8         SR8.84           OT         SR8.8         SR8.84           OT         SR8.8         SR8.84           OT         SR8.8         SR8.84           SR8.8         T         SR8.84           OT         SR8.8         SR8.84           SR8.8         T         SR8.84           OT         T         SR8.8           SR8.8         T         SR8.8           SR8.8         T         SR8.8           OT         T         SR8.8           SR8.8         T         SR8.8           SR	R         N         N           IMITTER         PROCEEDING         PROCEEDING           SRF/SF         DEFLAY         A           SRF/SF         DEFLAY         DEFLAY           SRF/SF         DEFLAY </td <td>2         2         2         2         3         3         3         4         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         <th1< th=""> <th1< th=""> <th1< th=""> <th1< th=""></th1<></th1<></th1<></th1<></td> <td>2         12         10           IMITTER         PROCERNINO         PROCERNINO           SRR, SF         PROCERNINO         SRR, SF           SRR, SF         SRR, SF         SRR, SF           SRR, SRR, SRR, SRR, SRR, SRR, SRR, SRR,</td> <td>R         In           IMITTER         PROCEIBINO           IMITTER         PROCEIBINO           SBR/75         Recentation           SBR/75         CEPLAV           SBR/75         SBR/75           SBR/75&lt;</td> <td></td> <th>60</th> <td>dр</td> <td></td> <td></td> <td></td> <td></td> <td></td>	2         2         2         2         3         3         3         4         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1         1 <th1< th=""> <th1< th=""> <th1< th=""> <th1< th=""></th1<></th1<></th1<></th1<>	2         12         10           IMITTER         PROCERNINO         PROCERNINO           SRR, SF         PROCERNINO         SRR, SF           SRR, SF         SRR, SF         SRR, SF           SRR, SRR, SRR, SRR, SRR, SRR, SRR, SRR,	R         In           IMITTER         PROCEIBINO           IMITTER         PROCEIBINO           SBR/75         Recentation           SBR/75         CEPLAV           SBR/75         SBR/75           SBR/75<		60	dр					
IMITTER         PROCERINO           887,57         1           887,57         389,7           887,7         389,7           887,7         389,7           887,7         389,7           887,7         389,7           887,7         389,7           887,7         389,7           887,7         389,7           68         306,2           69         306,2           901LER         308,3           61         308,3           61         308,3           61         6           61         0           61         0           61         0           74,4         54,4           74,4         54,4           74,5         54,5           74,6         54,6           74,6         54,7           74,6         54,7           74,6         54,7           74,6         54,7           74,6         54,7           74,7         54,7           74,7         54,7           74,7         54,7           74,7         54,7           74,7	IMITTER         PROCERING           H         1           H         1           R8.167         288.167           S88.167         288.167           S88.167         288.167           S88.167         288.167           S88.167         288.167           S88.167         288.167           S88.167         288.16           S88.167         288.2           S88.167         288.2           101         288.2           111         111           111         111           111         111           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           111         211           1	MBMITTER         PROCERNIKO           888.57         1           888.57         0.8 PLAY           888.75         0.8 PLAY           888.75         0.8 PLAY           6.60         39           6.61         30.2           6.62         39           6.63         39           6.64         30.2           6.65         39           6.66         30.2           6.67         30.2           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           7         30.4           8         90.7           8         90.7           8         90.7           8         90.7           8         90.7           9         90.7           9         90.7           9         90.7           9         90.7 <t< td=""><td>Image: Figure Figure</td><td>IMITTER         PROCERINO           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1</td><td></td><th>60</th><td>2</td><td>2</td><td></td><td></td><td></td><td></td></t<>	Image: Figure	IMITTER         PROCERINO           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1         1           11         1		60	2	2				
Immitter         Procession           888.74         1           888.74         1           888.74         1           888.75         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           888.76         1           98.8         3           1         1           1         1           1         2           1         1           1         1           1         1           1         1           1         1           1         2           2         3           1         2           1         2           1         2           2         2           2         3           1         2           1         2           2         2	Immiliary         Proclamic           100001164         11         11         11           10001167         288.75         100014         288.75           10001167         288.75         100014         200.2           10001167         11         200.2         200.2           1001167         11         200.2         200.2           1011167         11         200.2         200.2           1011167         11         200.2         200.2           1011167         11         200.2         200.2           1011167         11         200.2         200.2           2011167         11         200.2         200.2           201117         11         200.2         200.2           201117         11         200.2         200.2           201117         11         200.2         200.2           201117         11         200.2         200.2           201117         11         200.2         200.2           201117         11         200.2         200.2           201117         201.2         200.2         200.2           201117         201.2         200.2         200.	MANUTER         PROCESSIO           388.75         1           88.75         389.75           88.75         389.75           88.75         389.75           88.75         389.75           88.75         389.75           88.75         389.75           88.75         389.75           88.75         389.75           68         408.8           68         408.8           68         408.8           69         448.8           71         304.2           71         304.2           71         304.2           71         304.2           71         304.2           71         304.2           71         21.4           72.4         22.4           73         22.4           74         31.4           74.4         44.4           75.7         234.5           75.7         234.5           75.7         234.5           75.5         234.5           75.5         234.5           75.5         234.5           75.5         24.5 <tr< td=""><td>HILL         Tradicating           388.55         18 PLAY           388.56         388.56           388.56         388.56           388.56         388.56           388.56         388.56           388.56         388.56           56.60         38           56.60         39           56.60         39           56.60         39           56.60         39           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           69</td><td>Immitter         Processor           1987         1           1987         1           1987         1           1987         1           1987         1           1987         1           1987         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           &lt;</td><td></td><th></th><td></td><td></td><td></td><td></td><td></td><td></td></tr<>	HILL         Tradicating           388.55         18 PLAY           388.56         388.56           388.56         388.56           388.56         388.56           388.56         388.56           388.56         388.56           56.60         38           56.60         39           56.60         39           56.60         39           56.60         39           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           68         30.2           69	Immitter         Processor           1987         1           1987         1           1987         1           1987         1           1987         1           1987         1           1987         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           198         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           199         1           <								
H1         1         notuced           288.757         CBFLAY           88         6           6         9           6         9           6         9           6         9           10         9           11         218.2           11         218.2           11         218.3           11         218.4           11         218.4           11         218.4           11         218.4           11         218.4           11         218.4           11         218.4           12         1           21         1           21         21           22         24           12         24           12         24           13         4           14         4	H1         1         network           287.57         CHELAY         200           89.7         CHELAY         200           89.7         CHELAY         200           6.60         59         308.2           0.01LER         59         308.2           0.01LER         70         308.4           0.01LER         70         308.4           0.01LER         70         208.4           0.01         10         57.8           0.01         10         57.8           384.5         76.4         208.4           384.7         wc         284.4           10         10         10           10         10         10         10           10         10         284.4         284.4           10         10         10         10           10         10         10         10         10           10         10         10         10	H1         1         notuced           88.757         88.757         88.757           88.7         98.7         98.7           88.7         18.14         99.2           66.6         59         508.2           66.7         99.8         508.2           66.8         99.8         508.2           0.11.16         17         308.8           0.11.16         17         308.8           0.11.16         17         308.8           0.12         17         308.8           0.13         19         10           19         19         20.4           24.1         96.7         264           11         26.5         26.4           11         44.4         44.4           11         44.4         44.4	H1         1         notuced           387,67         08PLAY           88         08PLAY           88         0.8PLAY           88         3.9           86         3.9           5660         3.9           6.660         3.9           6.660         3.9           6.660         3.9           8.660         3.9           8.660         3.9           8.660         3.9           8.661         3.9           8.662         3.9           8.663         9.9           8.663         9.9           8.664         9.9           8.643         9.0           8.643         9.0           8.643         9.0           8.643         9.0           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6           8.65         5.6 <td< td=""><td>H1         1         notuced           288,57         CHFLAY         E461           86         59         5462           66         59         5462           66         59         5462           6.66         59         5462           6.66         59         5462           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           3412         0         47           3413         0         204           3413         0         204           3413         0         204           0         0         0         1           1         0         1         1           3413         0         204         204           0         0         20         204           0         0         0         204           0         0         0         1      &lt;</td><td></td><th>2</th><td></td><td></td><td></td><td></td><td></td><td></td></td<>	H1         1         notuced           288,57         CHFLAY         E461           86         59         5462           66         59         5462           66         59         5462           6.66         59         5462           6.66         59         5462           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           0         1         2046           3412         0         47           3413         0         204           3413         0         204           3413         0         204           0         0         0         1           1         0         1         1           3413         0         204         204           0         0         20         204           0         0         0         204           0         0         0         1      <		2						
R88.67         Interval           889.7         10           889.7         10           66         39         200.4           10         39         200.4           11         200.4         200.4           11         200.4         200.4           11         200.4         200.4           11         200.4         200.4           11         200.4         200.4           11         200.4         200.4           11         200.4         200.4           201.4         10         10           201.5         54         54           201.7         54         54           11         4.4         4.4	288.17 BB.7         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10         10          10         10	388.75 88.75         388.75 88.75         10000000           88.75         388.75         388.75           88.75         388.75         388.75           88.75         388.75         388.75           88.75         388.75         388.75           660         39	SBF/FI BBF/F         Introduce           SBF/F         DBFLEX           SBFFLEX         DBFLEX           SBFFLEX         DBFLEX           SBFFLEX         DBFLEX           SBFFLEX         SBFFLEX           SBFFLE	Rev H         Introduct           289.51         CHERAY           289.57         CHERAY           660         20           6.60         20           6.61         20           6.61         20           6.61         20           10         20           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         21           11         22           11         21           11         21           11         21           11         21           11         21           11         21		3	÷					
S88.1/2         Client view           889         Client view           68         Client view           68         S104.2           66         S104.2           66         S104.2           0         S104.2           0         S104.2           0         S104.2           0         C13           0         S104.2           0	Status         Cliffler           68         Cliffler           68         Status           68         Status           68         Status           66         Status           11         Status           12         Status           13         Status           14         Status           11         Status           12         Status           13         Status           14         Status           14         Status	288.70 68         08 PLAY           68         0 B PLAY           68         10 B PLAY           68         308.2           68         308.2           68         308.2           68         308.2           68         308.2           68         308.2           68         308.2           68         308.2           68         308.2           68         308.4           68         308.4           61         0           61         0           61         0           62         23.4           7         32.4           7         34.4           62         54           7         34.4           63         44.4           64         32           7         44           7         44.4           7         44.4	SEC. 70 68         SEC. 70 68         SEC. 70           10FLER         0         208.2           660         39	Set / 66         CIB-LAY           66         20           66         20           66         20           66         20           66         20           67         20           11         20           11         20           11         20           11         20           11         20           11         20           11         20           11         20           11         20           11         20           23         24           24         24           24         24           25         24           26         23           12         24           25         24           26         23           12         24           25         24           26         23           26         23           27         44           28         26           29         20           20         23           26         23           27 <td></td> <th>Ŧ</th> <td>F</td> <td>notu 😎 d</td> <td></td> <td></td> <td></td> <td></td>		Ŧ	F	notu 😎 d				
Marrier         Marrier           10 FLER         00         500         500           00         500         500         500         500           01         0         500         500         500         500           01         0         0         500         500         500         500           01         0         0         -67.8         0         0         0         0           01         0         -67.8         0         0         0         0         0           01         0         -67.8         0         0         0         0         0           02         1         -67.8         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0	Matrix         Matrix           0.0PLER         58.60           0.0PLER         58.82           0.0PLER         58.82           0.0PLER         78.82           38.473         44           1.0PLER         28.4           1.0PLER         28.4           1.0PLER         28.4           1.0PLER         28.4           1.0PLER         28.4           1.0PLER         28.4           1.0PLER         28.5           1.0PLER         4.4           1.0PLER         4.4	Bit         Cultures         Cultures <thcultures< th="">         Cultures         C</thcultures<>	68         000000000000000000000000000000000000	Marrier         Marrier           68         50         518.2           6.660         50         518.2           10         1         1         218.2           11         1         1         218.2           11         1         1         218.3           11         1         1         218.4           11         1         1         218.4           11         1         1         218.4           284.2         1         218.4         218.4           284.2         1         1         1           284.2         1         1         1           284.2         1         214         214           284.2         1         214         214           284.2         1         214         214           11         1         1         1           11         1         1         1         1		200.000	001002					
LiPLER         5.60         301         3012           UIPLER         1         3012         3012           UPLER         1         2014         3012           UPLER         1         2014         3014           UPLER         1         2014         3014           UPLER         1         2014         3014           UPLER         1         457         3014           UPLER         1         457         3014           2014         1         1         1           2014         1         1         1           2014         1         1         1           2014         1         1         1           2014         1         2         2           2015         1         4         1	6.60         3         300.2           UPLER         1         200.2           0         1         200.2           0         1         200.4           0         1         200.4           1         1         200.4           1         1         200.4           1         1         200.4           2         24.4         200.4           2         3         3           1         10         1           1         10         1           2         3         3           1         3         3           1         44         4           1         44         4	6.60         29         -30.3           OUPLER         0         94.3           0         0         94.3           0         0         20.4           0         0         94.3           0         0         47.8           0         0         47.8           0         0         47.8           0         0         47.8           0         0         47.8           0         0         47.8           0         0         23.4           284.10         000         23.4           1         000         23.4           1         4.4         4.4           1         4.4         4.4           1         4.4         4.4           1         1         1           1         4.4         4.4	6.60         50         200.2           10PLER         10         300.2           10         10         300.6           10         10         300.6           10         10         440.8           10         10         47.8           10         10         47.8           10         10         -47.8           10         10         -47.8           234         10         -47.8           23412         10         -47.8           23413         10         -47.8           11         10         -47.8           23413         10         -47.8           11         10         -47.8           12         234         -47.8           13         44.9         -47.8           14         10         -47.8           15         10         -47.8           16         -47.8         -44.8           16         -47.8         -44.8           17         -44.8         -44.8           18         -44.8         -44.8	Side         200         200           11PLER         11         200.2           11         200.2         200.2           11         200.2         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2           11         1         200.2								
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## **APPENDIX C**

## VITA

Kenneth Laboy was born in Washington, DC on September 22, 1979. In 1998 Ken Graduated from Springbrook High School. He chose to enter the United States Navy and Served as a US Navy SEAL at SEAL Team 2. While in the SEAL Teams he deployed OCONUS to various regions. In 2004 He left the military in pursuit of a college degree. However, he was unable to complete this venture due to being involuntarily recalled to active duty once again in 2006. Here he became a plank owner of SEAL Team 18 and was deployed OCONUS to various combat zones.

In 2011, he officially discharged honorably from the US Navy and enrolled at La Sierra University in Riverside, CA. While at La Sierra, he worked as a Laboratory Technician under Dr. Marvin Payne and also worked as a Teacher's Assistant for the department of Physics under Dr. Ivan E. Rouse. He eventually earned his degree in Biochemistry (Bachelor of Science) and applied to a laboratory position under Dr. Michael Malerick working in the field of organometallics, where he worked on the total synthesis of chemotherapeutic pharmacophores based off Platinum derivatives using Molybdenum as the base cofactor for the pharmacophore. He was eventually granted admission into Loma Linda University School of Medicine Basic Sciences program. Here he was given the opportunity to work under Dr. David Weldon as a Graduate student in his laboratory transitioning into the field of Medicinal Chemistry.