A Personal Account from Inside Keith Fagnou's Research Laboratory

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It was an early September day in 2003. I had just returned from a trip with my partner to New York City (my first visit) which we planned as part of celebrating the completion of our undergraduate degrees; her's in accounting, mine in biopharmaceutical sciences. As I climbed the long linear staircase that spans all 4 floors of D'Iorio Hall, the building which houses the chemistry department at the University of Ottawa, I didn't appreciate how formative the next 4 years working with Prof. Keith Fagnou would end up being.

I first heard about Keith from a close friend and future lab-mate who 6 months prior had decided to pursue graduate studies with this new professor. Shortly after this, another friend, who I shared a lab with during my honors thesis project, declared they were going to join his lab as well. I was intrigued and decided to go meet with him myself. I didn't start my senior year with the intent to pursue graduate studies in chemistry, but after my initiation to research through my honors thesis work, I had toyed with the idea of continuing my studies. Keith explained what joining a brand-new lab would be like. He had no concrete independent research program to speak of yet, just a bunch of ideas, and made it clear that if interested, I would need to wait until September to start as he had already committed to two new graduate students who would start over the summer. Yet, I was immediately struck by his genuine excitement for research and his enthusiasm for building something from the ground up. We just clicked. I walked out of his office genuinely excited about the prospects of joining his lab. When I was admitted to graduate school later in June, my letter came with an offer to join Keith's laboratory, and I was thrilled! It's not every graduate student who gets to have their first choice of Ph.D. advisor, or to join at the very beginning of a new lab. It's even rarer to join with two friends from undergrad, which made this experience extra special (Figure 1).

Figure 1 First Fagnou research group picture, circa September 2003. Pictured from left to right: Louis-Charles Campeau, Keith Fagnou, Mathieu Parisien, Marc Lafrance.

On my first day, Keith and I talked about project ideas. I was 'interested in synthesis', though thinking about it retrospectively, I'm not sure that I really appreciated what that meant. He proposed a ligand design project for 'Direct Arylation' reactions. These were biaryl-forming reactions where one of the pre-functionalized coupling partners used in traditional palladium-catalyzed methods, usually the organometallic one, is replaced with an unfunctionalized arene or heteroarene. This would entail phosphine synthesis with application in existing and new reactions, which could lead to application to natural product synthesis down the line. The world of palladium-catalyzed reactions had just been completely transformed by advances in phos-

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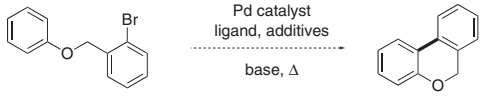
phine ligand design by Stephen Buchwald and others.[1] The idea was that perhaps by studying ligands in the context of direct arylation reactions, we would identify key insights that would enable new reactions and ligands. Accepted wisdom at the time was that the 'C–H functionalization' step of any catalytic cycle would be the most difficult, therefore much of the precedent focused on studying aryl iodides and activated bromides with electron-rich heteroarenes, or simple arenes bearing Lewis basic directing groups. These designs being aligned with the fact that oxidative addition would be easier and binding of the oxidative addition complex to a Lewis basic group or electron-rich heteroarene would accelerate the C-H functionalization step. *Side note: Keith disliked the use of the term 'C–H activation' to describe these types of reactions. To him this implied a fundamental mechanistic understanding which had not been explicitly proven.* Keith asked that I survey the literature and gave me a pile of printed papers, half a foot thick, he had on his desk, and suggested I pick three reactions to use as my model transformations for study.

This was often his approach as a supervisor. He would get you excited about a topic, send you on a direction where there was some limited pre-work, and then circle back once you had established a knowledge base and started your experimental plan. Later, as his group grew, he would pair junior graduate students with more senior ones to get them started this way, probably on an offshoot of an existing project. They could also help in building out scope tables for existing projects at first. This provided basic lab training for new students whilst also creating mentoring responsibilities for senior students. This happened pretty organically. However, looking back on it now, I realize how deliberate he was to create space for peer mentors in the group. He encouraged new students to engage their peers as well as providing coaching for the mentors too. I was very fortunate to get to develop these skills as one of the first graduate students in the lab; both as a mentor to undergrads (4) and to new graduate students (6) who later joined. These were certainly more formative leadership experiences than I appreciated at the time, and certainly ones that served me well in the professional environment that I entered into after graduation. More importantly, I think this created the kind of collaborative group culture and atmosphere that Keith felt would lead to success.

It's noteworthy that most of Keith's publications as a principal investigator feature groups of students and postdoctoral researchers as co-authors, rather than individual students. This was, in part, a result of this collaborative approach to problem-solving as well as his recognition of contributions made by early trainees working with senior students. Keith was famously quoted as saying that fostering a positive, creative, and collaborative group atmosphere would lead to greater overall productivity than dictating long work hours. That was certainly my experience in my 4 years in his laboratory. He encouraged us to "learn to be productive in a regular workday, because you will need to be able to do that when you get a real job later" (particularly in industry). He set a very good example for a work-life fit integration. He expected us to be in lab by 9 am, his usual start time, and was rarely around past 5–6 pm as he attended to his young family. I took it for granted at the time, but I later realized how much of a strong example this was and certainly something that was counter to the prevailing culture in synthetic organic chemistry labs in North America at the time. This doesn't mean that students, me included, sometimes didn't work in the lab outside of these core hours, but it wasn't because the principle investigator (PI) demanded it, and we learned how to be more productive during the day. Through his mentorship, we became early adopters of 'high-throughput' experimentation to get data more quickly. We bought aluminum blocks and worked with the machine shop to drill 12 holes in them the size for 2 mL reaction vials. One could set up 24 reactions on one stir plate with 2 of these blocks. He acquired autosamplers for our GCs and HLPC to maximize our productivity. We were deliberate about selecting model substrates that could facilitate analysis. He encouraged us to "think about an experiment long enough to convince yourself to try it, but not so long that you'll convince yourself not to." He loved to recount a story about his Ph.D. where he always set up a crazy experiment right before seminar which was based on a new idea, and one of these led to a great paper.[2] This 'never talk yourself out of an experiment' mindset stuck with me, and I later adopted it as a mantra throughout my career.

I never did make a single ligand in my Ph.D.! Of the three reactions I chose to study, we discovered that one of these reactions, which was designed to be a negative control, did yield significant product under the right conditions with a new ligand that had been published since the original report (Scheme 1). Keith encouraged me to quickly pivot, and I ended up spending the rest of my Ph.D. following the science and uncovering new reactions in the universe of direct arylation reactions. He was so excited for this result. As he used to say, "When you get those initial results in the morning, you are probably the first person in the universe who's uncovered this new knowledge. That's amazing!" I get goosebumps thinking about this even now.



Scheme 1 Initial reactions studies on the direct arylation of simple arenes

The lab's first paper was published in JACS in April 2004,[3] describing the development of optimized reaction conditions for this 'negative control' substrate class; related substrates were initially reported as negative controls in a prior publication.[4] Studying these in an intramolecular

fashion enabled us to gain an understanding of what would be required to enable direct arylation at 'simple' arenes, without the use of anionic or Lewis basic directing groups, which may alter the electronics of the organometallic species at play. We discovered that iodide is in fact often a poison for many direct arylation reactions and devised solutions to expand into this substrate class.[5] We expanded reactivity to more challenging aryl chloride substrates,[6] benefiting from more than a decade of innovations with these substrates for other palladium-catalyzed processes. These early successes[7] led to applications toward small polycyclic biaryl natural products like mukonine, allocolchicines[8] and aporphine alkaloids such as nuciferine[9] (Figure 2), as well as cascade reactions involving other palladium-catalyzed reactions.[10]

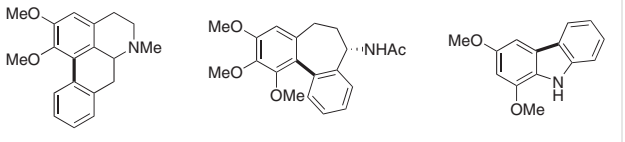


Figure 2 Application of intramolecular direct arylation in the synthesis of natural products

It's through studying longer tethers and observing reactivity differences with varying electronics of substrates that we identified certain critical attributes warranting further study. For example, the unique reactivity of Pd-carboxylate complexes pointed to the importance of these additives in the reaction.[11] The lack of bias for electron-rich arenes, as was observed by us[5] and others,[12] directed us challenge typically proposed S_EAr mechanistic pathways and implicated a potential concerted Pd–C-forming and C–H-breaking step. We were further urged to consider different options given the surprising regiochemistry observed with unsymmetrical substrates as well as some of our first intermolecular reactions, like in the case of benzodioxole.[5] Taken together, this pointed to a ternary mechanistic framework for these types of reactions inspired by mercuriation literature.[13] This was the inception in our thinking, along with others,[14] that led to what would later be called concerted metalation–deprotonation or CMD[15] (Figure 3).

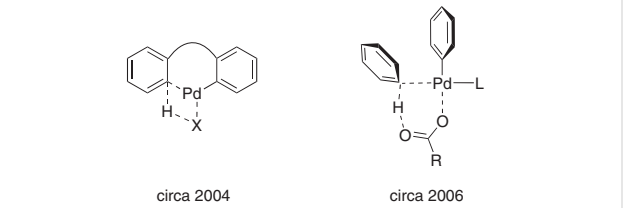


Figure 3 Evolution of mechanistic proposals leading to the concerted metalation–deprotonation model

This mechanistic framework inspired explorations into new intermolecular reactions with electron-neutral and electron-deficient characteristics, which were underrepresented substrates in direct arylation reactions at the time. An emblematic first example was pyridine *N*-oxides,[16] followed by perfluoroarenes[17] and nitrobenzenes,[18] and other diazine and azole *N*-oxides (Figure 4).[19] With lots of students focused on the reactivity of arenes, we naturally expanded exploration into oxidative biaryl coupling reactions of arenes lacking any aryl halide or organometallic prefunctionalization, both for intramolecular[20] and intermolecular reactions (Figure 5).[21] An analogous approach to the discovery and development of direct arylations of alkanes was also developed.[22]

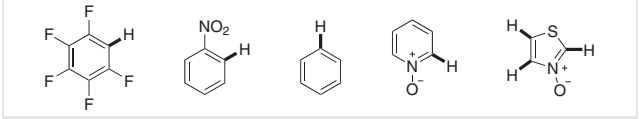


Figure 4 Substrates for intermolecular direct arylation with aryl halides and triflates

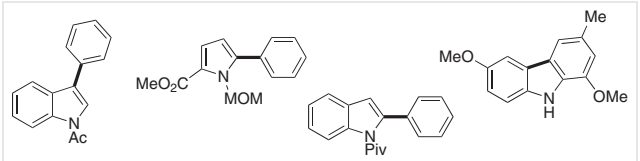


Figure 5 Typical products accessible via oxidative biaryl formation

By this point, around 2006, the research group had more than tripled in size and explorations of several new areas of research were ongoing. Keith's travel schedule increased significantly as his reputation in the field was significant despite his short tenure. Through it all, he was still very accessible, in his very informal style, stopping by the lab to chat with every student whenever possible. The 'group activities' we planned at the beginning, like pool and bowling, had morphed into curling and golf events. The annual golf tournament and BBQ of the lab, aptly named the 'Mathieu Parisien Invitational Golf Tournament' after the group's first graduate became quite the event. While very few of the lab members were golfers, it was something that Keith really wanted to establish as a tradition and even had trophy made for it (Figure 6). The event was always about having fun together, not really the golf, and all skill level participants were welcome. Alumni often returned to participate, and it was another way for Keith to foster a strong sense of collaboration and fun with our team.

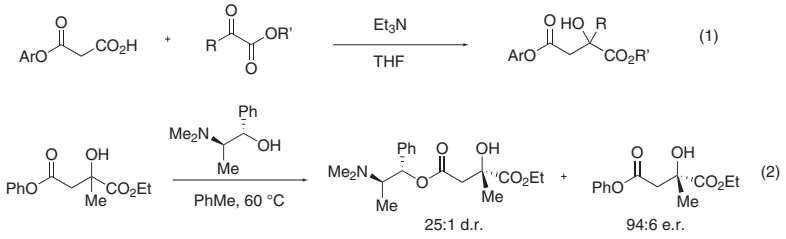
Ever curious, Keith encouraged students to pursue many off shoots from these initial projects which led to new areas of research, or in some cases completely new directions for the research group. For example, it was through the pursuit of a side reaction during the allocolchicine total synthesis that team members became interested in decarboxylative

Figure 6 Keith Fagnou and Mathieu Parisien at the annual golf outing (left). The golf tournament trophy (right).

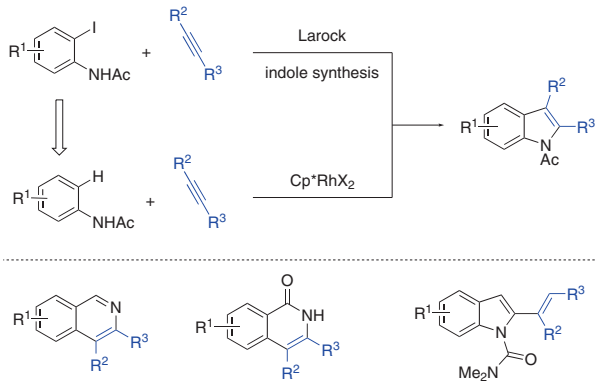
aldol reactions[23] and the kinetic resolution of these products[24] (Scheme 2). We even dabbled in hydrogen generation from ammonia-borane reservoirs.[25]

It was while attempting to extend the concept of replacing a pre-functionalized coupling partner in another classic palladium-catalyzed process, the Larock indole synthesis, that the lab's research pivoted to study another transition-metal catalyst: rhodium. When Keith first started his research group, he had expressed his desire to not delve into rhodium catalysis. Having spent his Ph.D. focused on the development of rhodium(I)-catalyzed ring-opening reactions of bicyclic alkenes,[26] he felt it important to cast his focus elsewhere. A decade later, the science led him back to rhodium. Many attempts to accomplish a similar reaction with palladium complexes were unfruitful, but based on precedent from Satoh and Jones,[27] Rh(III) complexes were examined and proved very productive in this direct annulation reaction.[28] For two more years, these major thrusts of research continued yielding important findings: (1) new observations on the regiochemistry[29] and reactivity[30] of palladium-catalyzed reactions, and (2) exploring the reactivity of Rh(III)-mediated processes,[31] including the discovery of internal catalyst turnover with hydroxyl-imine or hydroxamic ester directing groups (Scheme 3).[32]

I left the lab in the summer of 2007. It was through Keith's network that I was first introduced to scientists at Merck Process. Through my career development conversation with Keith, he knew that my dream job was to be a process chemist at Merck, and he made every effort to connect me with recruiters and senior leaders in the organization whenever the opportunity presented itself. This network afforded me the opportunity to interview in the fall of 2006 for a position. When I got the job offer right before Christmas that year, Keith was incredibly supportive in en-



Scheme 2 Decarboxylative aldol reactions and resolutions



Scheme 3 Development of Rh(III)-catalyzed direct annulation reactions inspired by the Larock indole synthesis

abling my transition to my new career on my preferred timeline. After I started my career, Keith and I continued to talk regularly as we wrapped up writing papers related to my Ph.D. work and connecting at the graduation ceremony in spring 2008. He continued to mentor me as a young professional and our relationship deepened into an important friendship for me. The last time I saw Keith in person was at the 2008 golf tournament and BBQ at his home. It was amazing to see him continue the tradition of building and sustaining a culture with his large team that he had fostered with me and my peers when we were only a few students in the lab. It was a tight-knit group of folks which had 'fire in the belly' for discovering new things and had each other's best interest at heart. One's success was everyone's success. It felt like returning home and I have fond memories of this day.

The legacy of any chemistry professor is both the direct scientific contributions of their research team and the scientists that they train in their laboratory. Given the research highlights described above and the amazing citation record Keith achieved in his short career, it is indisputable that his research contributions were significant for the field.[33] However, it is his legacy as a mentor that I will cherish most. It is common for alumni of Keith's research team, we've playfully referred to ourselves as the #FagnouFactory since 2004 (Figure 7), to encounter peers of Keith's who point to an amazing track record in mentorship. While the Ph.D. cohort who graduated from his lab was small (2 graduated before his passing and 6 total graduated with Ph.D.'s mostly accomplished in his group), three have careers as professors running their own labs, and the three others, including myself, have had successful careers in pharma. More broadly, of the other scientists who had their start or a sojourn in Keith's laboratory, 9 more obtained Ph.D.'s following their initial stint. The Canadian graduate school system is also renowned for providing highly skilled M.Sc. trainees, which are sought after by the best pharmaceutical companies in Canada, the US and abroad. In total, >15 found careers in research across pharma and government laboratories after further studies. It is also noteworthy that over a third of the original research from Keith's research group was published posthumously (24 of 63 papers) by students and post-docs who completed the work.[34] This is a testament to the loyalty and dedication he cultivated in his students who supported each other through his tragic death, alumni included, to keep the laboratory going to accomplish this feat. It should also be noted that Keith cultivated amazing relationships with his peers who stepped in and supported the students who were still in the lab at that time, notably Professors Andre Beauchemin and Louis Barriault and the department chair at the time, Professor Tito Scaiano.

Figure 7 Members of the #FagnouFactory (taken shortly after Keith's death in November 2009)

Keith and I had a wager dating back to my 2nd year of grad school. He often liked to have small wagers (a beer or a coffee) with us on reaction outcomes; kinetic isotope effect magnitude was a favorite – *I lost one of those, once*. He never let us bet against ourselves; you couldn't bet a reaction wouldn't work or that you couldn't accomplish a goal. At the end of my second year, we wagered a more significant prize, a bottle of scotch. At some point early in his tenure, Keith decided he wanted to become a scotch aficionado and he became enthusiastic about collecting and savoring different scotches. The wager was that by the time it was all said and done, I would publish as many papers with him from my Ph.D. work as he had during his with Mark Lautens. This became true in the summer of 2009 two years after I left the lab. I couldn't make the golf tournament that year because of the birth of my first son. Keith and I resolved that he would settle the wager when I hosted him to give a seminar at Merck. We set a date for November 9th, 2009. The week before his visit, I received a voicemail from Keith on my office phone informing me that we would need to postpone his visit given he was quite sick with the flu. This is the last time I heard his voice. Keith passed away No-

vember 11th, 2009, from complications related to an H1N1 infection. My friend and former lab mate, David Stuart, called me to share the news—we were devastated. As I write this now, my heart is in my throat, my eyes are watering, just recalling this 2-minute conversation. I saved the voicemail from Keith on my office phone until I relocated to New Jersey more than a year later. In my office now hangs my favorite picture of Keith and I (Figure 8) with a copy of his final hand-written note to me. A note he wrote on the acknowledgements section of my thesis which my wife framed for me after his passing. It thanks me for joining his group "at a time when it didn't exist" and promises that there "will always be scotch in his office" when I stop by. It reminds me of the great times we had together and that through his mentorship and example, he had a tremendous impact on me as a scientist, a father, and a husband. It reminds me of the privilege I had to share those 6 years with him. I am so lucky!

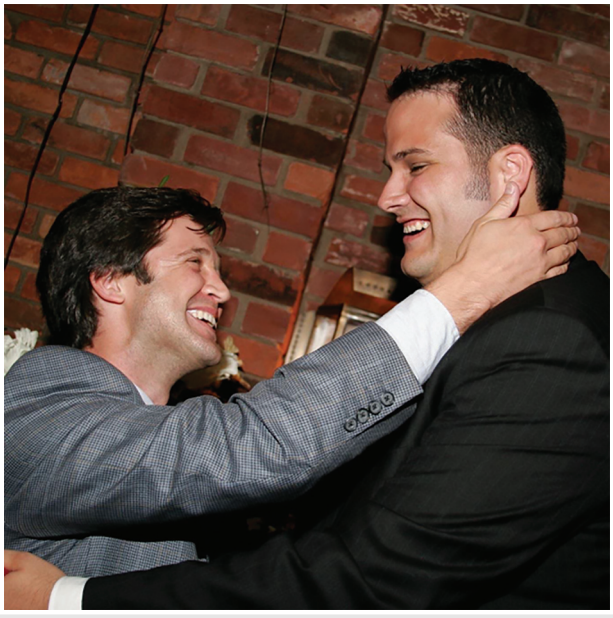


Figure 8 Keith Fagnou and L.-C. Campeau at L.-C.'s wedding in summer 2006

Conflict of Interest

The author declares no conflict of interest.

Acknowledgment

I am thankful to all my #FagnouFactory colleagues over the years, who formed an amazing work environment and helped me grow as a scientist and a leader. I'm particularly thankful to Marc Lafrance and Mathieu Parisien, who talked me into this journey in the first place and for their friendship and support. Thank you also to Sophie Rousseaux and David Stuart for helpful feedback during the drafting of this account.

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(33) In 2019, nine years after his death, Keith Fagnou was still listed in the Clarivate Highly Cited Researchers report; see: https://recognition.webofscience.com/awards/highly-cited/2019/ (accessed Apr. 16, 2024)
(34) Keith Fagnou – Eternal Authorship, In Just Like Cooking Blog; 2012; https://justlikecooking.blogspot.com/2012/02/keith-fagnou-eternal-authorship.html (accessed Apr. 16, 2024)