



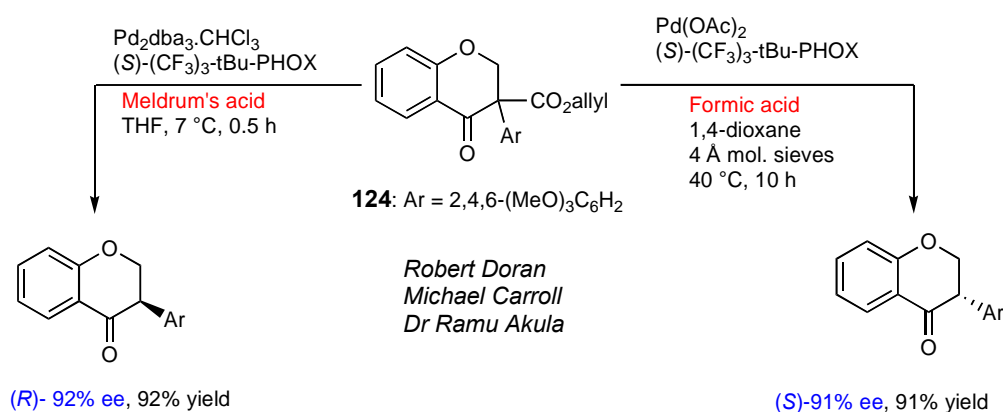
The Journal of the Institute of Chemistry of Ireland

Feature Articles:-

Research Review from UCD

Adventures In Asymmetric Catalysis, Natural Products and Medicinal Chemistry: Boyle-Higgins Medal Lecture 2014.

By Prof Patrick J. Guiry* UCD



Plus articles from



And much more in this issue.

President's 2013 – 2015 Final Address

As my term as President ends I will say a few words about the last two years as President. Firstly it has passed so quickly and I have thoroughly enjoyed the experience and I really grateful for the huge support I have got from our Council members, especially from Brian Murray Immediate Past President, John Keegan our hard pressed Hon. Treasurer, Philip Ryan our ever diligent Hon. Secretary and of course Vice President Margaret Franklin. I also had great support and efforts from Fergal Barry at LIT and his team organising last year's Congress and has many ideas about how to develop and finance the Institute. This year we had Pat Guiry and Eoghan McGarrigle organising Congress 2015 on the April 30th at UCD. We had Prof Dermot Diamond for this year's Boyle Higgins Award and I thank Dr Paraic James for his efforts in organising our event at DCU on April 16th.

When I was elected I indicated 3 areas in particular I wanted to focus on. These were:-

- The Membership Challenge
- Publication of ICN
- Embracing Communication Technology

Membership growth is slow but improving slightly but nowhere near what it should be. Any chance I get I urge chemists to join the Institute and get positive feedback but the actual uptake then falls far short of expectation. I was not at all happy with the gender balance on Council and we have been more successful in this endeavour with Patricia Cullen, Henkel, Mary Mullaghy ISTA, and Celine Marmion RCSI coming on Council. In addition we have Eoghan McGarrigle from UCD and Rob Elms NUIM on board. The effect of this group of 5 is to lower the age profile of Council which we really need to do.

A big issue we have is finance which is limiting what we can do. Part of this problem is members whose subscriptions are in arrears, in some cases for several years. This costs us in sending our reminders, and EuCheMS subscriptions we have to make for non-paying members on our accounts. We have started addressing this and will continue to do so over the coming months.

During the recession commercial sponsorship died off substantially but I happy to say we are seeing a return at last year's Congress and again at this year's upcoming Congress.

Publication of ICN is problematic mainly due to difficulty of getting academic papers on time. Due to work load and family commitments Bob Baker resigned as Editor after last Christmas's issue of ICN. I would like to thank him for his efforts over the last couple of years for his efforts in getting the issues out in spite of the workload. I have stepped in to the editor shoes and hope to increase the frequency of publication from 2 issues in 2014 to hopefully 3 this year. I really would like to push this up to 4 per year but getting the academic papers is a challenge. It's all electronic now with web links to interesting material which is an advantage over hard copy. I would like to improve the cover design so if you have suggestions please talk to me. I will be exploring if we can use an e-reader to turn pages like a magazine and the costs involved.

On the **Communication** front we are on LinkedIn, Facebook and Twitter. I had hoped this would result in much more engagement with chemists but not so. I myself am not very social media savvy and this in an area needing much more work. Perhaps our new younger members on council can help out here. Getting chemists involved with their professional body and to attend events is much more difficult than I expected.

EuCheMS We hosted the EuCheMS General assembly in 2012 while Brian was president and I attended the Budapest GA in 2013 and the Torun, Poland GA last year. EuCheMS is moving forward and are working on how to improve services and support to member societies after a brain storming session in Torun under the new president David Cole-Hamilton. The outcome will be communicated in due course.

Industrial Award.

We announced an Industrial Award for a Chemist last autumn, and we have selected a great candidate. The official announcement appears on a later page.

Briefly I want to wish Fergal Barry well as Vice President. Fergal is Vice President, Research, Development and Enterprise at LIT. We have been talking about how the Institute can be progressed and developed since 2013 and especially last year while organising the Congress. He has considerable experience working with partners including other colleges, Limerick City & County Councils, EI, IDA, County Enterprise Boards, Chambers of Commerce, and in raising finance. Raising finance is critical to our future and we will work with him to that end.

Our new President is **Margaret Franklin** whose enthusiasm and energy knows no bounds. Margaret is the third woman president of the Institute. She has been in the Institute for over 40 years, on Council for over 20 years and Vice President for the last 2 years. Former senior lecturer in Inorganic Chemistry at AIT. She is a freelance science writer, publishing articles in Science Spin Magazine and The Westmeath Independent. She has a great interest in astronomy and comes to UCD twice weekly for lectures in astronomy.

She has been a great support to me and I feel the Institute is in safe hands with her and I wish her every success in her role as President.

Patrick Hobbs MSc, FIFI, CChem, CSci, MRSC.

President April 2013 –April 2015

Introduction from the President 2015-2017

Dear Fellows, Members and Graduate Members,

It is with some trepidation that I accept the baton handed to me by my predecessor, Pat Hobbs, who has proved to be a dedicated and dynamic President over the past two years. He has worked tirelessly, at every opportunity, to recruit new members for our Institute, but in spite of this, membership numbers have been declining since they peaked in 2008. In that year, membership stood at a total of 742, but it was down to 655 in 2014. No doubt this has been due to the recession, so now that the economy has improved, it is my hope that we may be able to encourage more chemists to join our Professional Body. Ideally, I would like all practicing chemists in this country to be members. The more members we have, the better services we can provide, as membership fees are our main source of income.

Apart from expanding our membership, I am hoping to improve communications with our existing members. As you may have noticed, you are not getting as many postal mailings as heretofore. This is because the cost of stationery, printing and postage has become prohibitive, so we are moving towards electronic means of communication. Unfortunately, we do not have e-mail addresses for all of our members, but a number of requests have been sent out with the postal mail shots, asking members to give us their e-mails, so that we can provide you all with updates and news of coming events. I would also invite you to visit our website regularly, to keep up to date with our activities. You were sent a list of events for 2015 along with your subscription renewal notices and notice of the AGM. However, attendance at the AGM was disappointing, so in order to inform members of our activities over the past year, I decided to circulate the Honorary Secretary's Annual Report to all members on our e-mail list. Some of you may not be aware of the many events we organise and sponsor, or the awards we make, at various levels. We would like you to know of our various activities and encourage you to take an active part in the work of the Institute. We hope you found the annual report informative.

Already this year, Pat Hobbs, outgoing President, presented a medal to the candidate who gained the highest marks in the Honours Leaving Cert. Chemistry paper in the 2014 examination. This presentation was made during the Irish Science Teachers Association Annual Conference in Cork, just before Easter.

The Boyle-Higgins Award lecture was given by Professor Dermot Diamond of DCU on the day of the AGM (April 16th). Pat Hobbs presented him with his medal after what proved to be a fascinating presentation on chemical sensing with autonomous devices. After the AGM, my first official function was to present certificates to our two newly elected Honorary Fellows. They are Dr Noel Fitzpatrick, formerly of UCD, who was Editor of the Institute's journal 'Orbital' for many years and Dr Joe Eades, formerly of Teagasc, who served as Registrar for many years.

As I write, I am looking forward to two further events, which will have taken place by the time you read this. The first is our Annual Congress, which this year is being hosted by UCD, on Thursday, April 30th. The theme is Asymmetric Synthesis and the programme involves speakers from several of our Universities, as well as one from industry. This will be followed by the Congress Dinner, in the Stillorgan Park Hotel. We hope to publish the proceedings of the Congress in 'Irish Chemical News' later this year. The other event is a joint ICI/RSC Awards Symposium, which takes place on the very next day, May 1st, at Queen's University Belfast. The RSC will present a number of awards and I will have the pleasure of presenting the Annual Chemistry Award (Eva Philbin Lecture Series) to Professor Thorfinnur Gunnlaugsson of TCD.

This year, The Institute of Chemistry of Ireland has introduced a new award, the Industrial Chemistry Award 2015, which is generously sponsored by Henkel Ireland Ltd. The winner will have been formally announced during the Annual Congress on April 30th, so the name can now be revealed. The award goes to Donal Coveney of TopChem. We will be holding a major event later in the year, at which the award will be presented.

Our Young Chemists are the future of Chemistry in this country and over the past few years, I have endeavoured to engage recent graduates with the work of the Institute. Our Young Chemists' Group is affiliated with the European Young Chemists' Network (EYCN) and has represented us at meetings in different European countries, for which we were happy to be able to provide travel bursaries. The EYCN is the Young Chemists' division of the European Association of Chemical and Molecular Sciences (EuCheMS). Our young chemists have also been instrumental in engaging with social media on behalf of the Institute and have set up a Facebook page and a twitter account.

The Irish Universities Research Colloquium will be hosted by NUI Maynooth this year on June 24th * 25th. This event is sponsored by The Institute of Chemistry of Ireland. I hope to attend, as this is where we get the most up-to-date information on the research currently taking place in our university chemistry departments. It will also give me an opportunity to meet with a number of our young chemists and encourage at least some of them to join our Institute.

The Institute of Chemistry of Ireland remains affiliated with EuCheMS and I hope to represent the Institute by attending the General Assembly, which takes place in Vienna at the end of September.

As I look forward to the next two years as your President, I hope they will be good years for you and I do hope that many of you will attend our events and engage with our websites and social media.

Margaret Franklin, B.Sc., M.Sc., FICI

President, April 2015.

Main Website: <http://www.chemistryireland.org/>

LinkedIn: <https://www.linkedin.com/profile/view?id=310228693>

Facebook: <https://www.facebook.com/InstituteofChemistryofIreland>

Twitter: <https://twitter.com/irishchemistry>

Editorial

This is the first edition of Irish Chemical News (ICN) with me as Editor. First of all I would like to thank Dr Robert (Bob) Baker from Trinity College's Inorganic and Synthetic Materials Chemistry Department for his effort in publishing ICN over the last few years. He resigned his editorship in January this year due to the onerous work load in his department and family ties. Since taking on the task of editor I have come to realize how time consuming this task can be. When I became President in 2013 I stated I wanted to see ICN published more frequently in electronic format due to the cessation of the hard copy publication because of costs and a falloff in sponsorship during the recession.

To me publication of our Journal is a critical and important activity of the Institute. We have managed to have 2 editions per year in the last 2 years but I really want this become a quarterly publication over the next 2 years and then a bimonthly journal. In an ideal world a monthly publication would be best. For 2015 I aim to have 3 editions and 4 in 2016. This is ambitious as the rate limiting step is the availability of academic papers. Most academics have a heavy workload with teaching hours, research work and writing up funding applications. On top of that they need to prepare papers for publications in high prestige peer reviewed journals. That leaves little time for preparing articles for publication in non-peer reviewed journals like ICN.

Nevertheless I urge academics and especially post docs to make an effort to provide papers for ICN publications. This is an opportunity to inform a mainly Irish chemical community of the wide range and important chemistry research work being undertaken in Irish Universities, Institutes of Technology, and research institutions. After all a lot of Irish and European tax payers money goes into funding the research work.

Much the same can be said about the pharmaceutical and chemical industry here. Through the efforts of the IDA, Enterprise Ireland and SFI Ireland has an enviable success story to tell. I would then urge industrial chemists, and hopefully CEOs will encourage and support my call, to seriously consider telling us of some of the great successes you have. We don't need the release of confidential information but there are case histories which don't breach confidentiality which are worth publishing and sharing with your colleagues in the wider chemical community.

Apart from formal academic papers and successful stories I hope to include a range more general articles of interest to chemists, such as reports of events, job opportunities, articles on analytical techniques, experimental design, data acquisition and big data, contribution of women to chemistry, environment, EU legislation, and education.

I aim to have a September and December editions this year and will be contacting chemists in academia and industry etc. for articles so please give some thought to what you would like to submit.

Patrick Hobbs MSc, FIFI, CChem, CSci, MRSC.

Immediate Past President & Editor ICN.

Note: Opinions expressed in this Journal are those of the authors and not necessarily those of the Institute.

Congress 2015 UCD Report Dr Eoghan McGarrigle UCD

The 40th Annual Congress of the Institute of Chemistry of Ireland took place in the UCD O'Brien Centre for Science, on Thursday 30th April. The congress theme was '*Modern Approaches to Asymmetric Synthesis*'. The final programme with delegate list is now available ([click here](#)). We were happy to welcome almost 100 delegates from all over the island of Ireland. These included industrial delegates and academic delegates from University College Cork, University of Limerick, Limerick Institute of Technology, Queen's University Belfast, Maynooth University, Trinity College Dublin, the Royal College of Surgeons, Institute of Technology Tallaght, Dublin City University and, of course, University College Dublin.

We would especially like to thank our speakers who gave excellent talks and shared many new and unpublished exciting results. Pictured below are: (back row, left to right) Dr Gerard McGlacken, Dr Paul Evans, Dr Fintan Kelleher, Prof Declan Gilheany, (second row from back) Dr John Stephens, Prof Mauro Adamo, Prof Pat Guiry, (2nd row from front) Dr Eoghan McGarrigle (Chair organising Committee), Pat Hobbs (ICI Immediate Past President), (front row) Dr Francesca Paradisi, Prof Karl Hale and Dr Margaret Franklin (ICI President).



There was also a [poster session](#) with many high quality posters on all aspects of [Synthetic Chemistry](#). Based on these posters, one would have to say that the future of synthetic chemistry in Ireland looks bright. There were 3 poster prizes awarded. Two prizes of 100 Euro each sponsored by [Fluorochem](#) were awarded to Dr Bartosz Bieszczad (UCD) and Ms Catherine Tighe (UCD) by ICI President Dr Margaret Franklin.



The winner of the first prize in the poster session was Mr William Doherty (UCD) who won an iPad2 Air generously sponsored by [ChemGlass](#). The prize was presented by Dr Patrick Delaney from [ChemGlass](#).



The winner of the ICI Industrial Chemistry Award was also announced at the congress by Immediate Past President Pat Hobbs. Dr Donal Coveney of [TopChem](#) is the 2015 winner. An award lecture and event will be scheduled for later in the year.

In the intervals between talks there was a trade exhibition featuring new products and technologies from many of our sponsors: [Waters](#), [Agilent](#), [Sigma-Aldrich](#), [Lennox](#), [LabPlan](#), [GPE Scientific](#), [Chemglass](#), [Advion](#), [Fisher Scientific](#), [Lennox](#) and [Mason Technology](#). We would like to thank all our sponsors for supporting the event. It would not have been possible without them.

The congress closed with a reception and then the congress dinner. There was much discussion and healthy debate about the chemistry presented during the day and into the evening.



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We thank our Congress Sponsors



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Research Article

Adventures In Asymmetric Catalysis, Natural Products and Medicinal Chemistry: Boyle-Higgins Medal Lecture 2014.

Patrick J. Guiry*

School of Chemistry and Chemical Biology, Centre for Synthesis and Chemical Biology, University College Dublin, Belfield, Dublin 4, Ireland;
E-mail: p.guiry@ucd.ie

I wish to thank The Institute of Chemistry of Ireland for the award of the 2014 Boyle-Higgins Medal which I wish to dedicate to the members of my research group, past and present.

Introduction

The ability to synthesise new compounds for a range of scientific applications is one of the cornerstones of modern organic chemistry and science itself. As an undergraduate at UCD it was those courses on synthetic methodology, mechanism (how the reactions work) and applications in medicinal chemistry that were my favourite ones.

I was delighted to proceed to undertake a PhD degree with Professor Dervilla Donnelly at UCD where I significantly enhanced my laboratory skills to complement my theoretical understanding in order to perform cutting edge research. I was able to develop an interest in the application of organometallic reagents in synthetic organic chemistry, a recurring theme throughout the rest of my research career, and worked on applying aryllead triacetates as a source of aryl cation equivalents for a series of C- and N-arylation studies. This methodology was applied to a new synthesis of a range of natural products (3-aryl-4-hydroxycoumarins,¹⁻³ 2-arylbenzofuranones,⁴ neoflavanoids⁵⁻⁶ and neoflavenes).⁷ In addition, we developed a copper-catalysed N-arylation for the preparation of a range of secondary and tertiary amines with Dr Jean-Pierre Finet at the University of Marseille.⁸⁻⁹ Finally, we synthesised a series of sterically hindered triarylphenols, a study that was carried out in the laboratories of Professor Sir Derek Barton at Texas A&M supported by a Fulbright Award.¹⁰⁻¹¹

Thereafter, I undertook postdoctoral research with Dr John Brown FRS at the Dyson Perrins Laboratory, Oxford University. I chose his research area which was in asymmetric catalysis (mechanism and synthetic methodology development) as I had no background whatsoever in any asymmetric synthesis or catalysis and I felt these would be areas of increasing importance for the future. I was exposed to a range of projects, including (a) fundamental mechanistic studies employing low temperature multinuclear NMR spectroscopy on the bite angles of diphosphines and their importance on the rate of reductive elimination in palladium-catalysed cross-coupling reactions;¹² (b) probing soft versus hard Lewis acids in Diels-Alder reactions;¹³ (c) preparing ethane rhodium bisoxazoline complexes, crystallising them and comparing the solid state structures to those predicted by computational methods;¹⁴ (d) asymmetric reduction of benzylidenemalonates using rhodium complexes¹⁵ and (f) synthetic and mechanistic studies on a novel axially chiral, P,N ligand (Quinap) and its application in palladium-catalysed allylic alkylation.¹⁶

My independent research career began in 1993 with my appointment as a College Lecturer at UCD. It is always a strange transition in one's academic life from working with experienced researchers as supervisors, and I was particularly fortunate to have worked with Professor Dervilla Donnelly, Dr Jean-Pierre Finet, Professor Sir Derek Barton and Dr John Brown, to being the person responsible for the

choice of project, the direction of research and the nurturing of the future generation of researchers. I take great care to repay the trust that students place in me by choosing me as their PhD supervisor or postdoctoral colleague by training them to think for themselves, how to problem-solve, how to present their work (orally and written) and to enjoy research and the search for new knowledge. I have been fortunate to work (and continue to work) with some of the brightest and most enthusiastic students of their generation and together build a strong team ethic, work ethos and scientific reputation.

The answer to the question "What does it take to be a good PhD supervisor?" is easy – good students (and funding of course!). The following sections of the lecture will highlight selected examples of group research across the 21 years, divided into three sections (The Early Years, The Middle Years and the Later Years). The researchers who performed the work will be credited in the appropriate schemes / figures and apologies to those whose work does not appear – there remains a lot of research work performed that has still to be published, so watch this space!

UCD- The Early Years (1993-2000)

After my postdoctoral research and thinking of an academic career, I was still intrigued by the area of catalytic asymmetric synthesis as it employs many important aspects of the traditional disciplines of organic chemistry, inorganic chemistry and bioorganic chemistry, with a special emphasis on the preparation of compounds of use in biological, medicinal, agrochemical, and material / nanoscience-related research programmes.

Therefore, the specific area I began my career at UCD was in the design, synthesis and application of a series of P,N bidentate chiral ligands and their application in a series of important synthetic transformations. Initial examples from John Cahill's work included the *trans*-2,5-dialkylpyrrolidinylbenzylidiphenyl phosphines **1** and **2** and their application in palladium-catalysed allylic alkylations, Scheme 1,¹⁷⁻¹⁹ the intermolecular asymmetric Heck reaction²⁰ and iridium-catalysed imine reductions, Figure 1.²¹ We obtained enantiomeric excesses (ees) of up to 90% for allylic alkylation, 60% for the phenylation of 2,3-dihydrofuran and 52% for the reduction of the imine derived from acetophenone and aniline.

Annette Farrell extended the range of *trans*-2,5-dialkylpyrrolidine - containing ligands to include the planar chiral example **3** whose synthesis included a diastereoselective lithiation/phosphinylation protocol.²² The methyl-substituted example gave 38% ee for the alkylation of 1,3-diphenylpropylacetate.

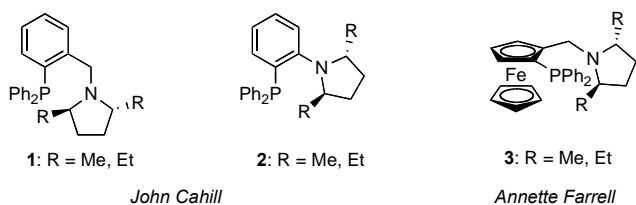
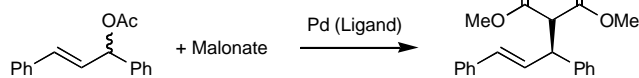


Figure 1.



Scheme 1. Asymmetric allylic alkylation (AAA) - standard test reaction of malonate with 1,3-diphenylpropenyl acetate.

We complemented this work with detailed NMR spectroscopic investigations of palladium allyl complexes and X-ray analysis of the palladium and iridium complexes of ligands **1** and **3**, Figure 2.

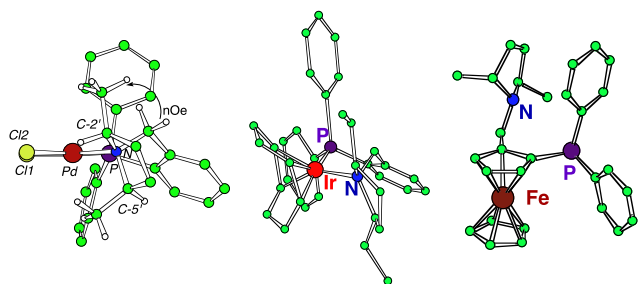


Figure 2.

Still intrigued by the possibility of applying P,N ligands in catalysis, Mary McCarthy synthesised and resolved the axially chiral pyrazine- and quinazoline-containing ligands **4** and **5**, Figure 3.^{23,24} The use of ligand **4** in asymmetric catalysis was precluded as it racemised at room temperature whereas Pd complexes of ligand **5** (2-phenyl-Quinazolinap) gave 66% ee for the alkylation of 1,3-diphenylpropenyl acetate.²⁵ We coined the term 'Quinazolinap' by analogy with 'Quinap' as our ligands contained a quinazoline-naphthalene as the key biaryl unit, just as Quinap possessed a quinoline-naphthalene biaryl unit. It took Mary 6 (hard) months to resolve ligand **4** and this expertise allowed her to resolve ligand **5** in less than a week!

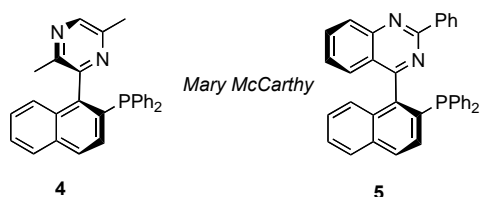
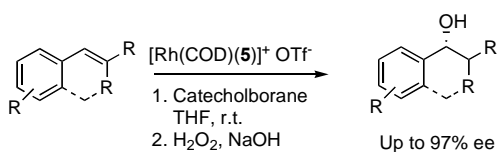


Figure 3.

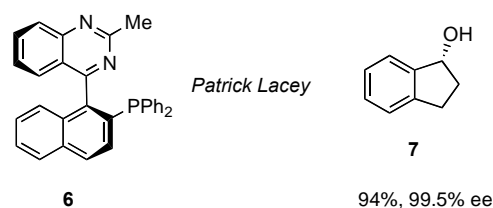
Initial applications of ligand **5** in the rhodium-catalysed hydroboration of alkenes, performed in Dr John Brown's laboratory, gave very high conversions and ees for the oxidation of a range of styrenes and stilbenes, with an optimal ee of 97% for the hydroboration/oxidation of *trans*- β -methylstyrene, Scheme 2.²⁶



Scheme 2.

During a mechanistic investigation of allylic alkylation with 2-phenyl-Quinazolinap **5**, we determined that the 2-phenyl group takes up a position in space leading to ligand-reactant steric interactions which have a major influence on the sense of the asymmetric induction observed. In light of this observation we wished to determine the

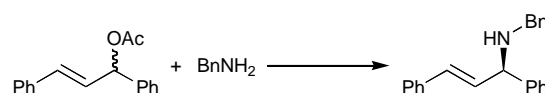
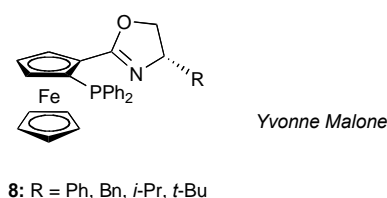
influence of substituents other than a phenyl group at the 2-position. Pat Lacey synthesised and resolved the 2-methyl-Quinazolinap ligand (**6**)²⁷ which afforded an optimal 94% yield and 99.5% ee for the Rh-catalysed hydroboration/oxidation of indene to form indanol (**7**), Scheme 3.



Scheme 3.

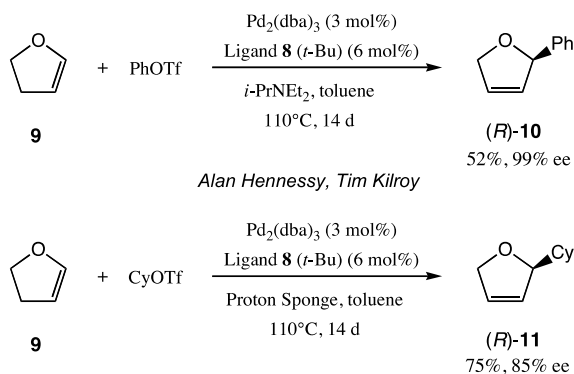
At this early stage we had also developed an interest in oxazoline-containing P,N ligands. Because of their ready accessibility, modular nature and applicability in a wide range of metal-catalysed transformations, compounds containing a chiral oxazoline ring have become one of the most successful, versatile and commonly used classes of ligands for asymmetric catalysis.²⁸⁻²⁹ The large majority of these ligands are derived from readily available chiral amino alcohols in a few high yielding synthetic steps. As a consequence, the enantiocontrolling stereocentre resides on the carbon atom neighbouring the coordinating nitrogen of the oxazoline ring and therefore in close proximity to the metal active site, thus having a direct influence on the stereochemical outcome of the reaction.

Yvonne Malone was the first PhD student in the group to work on oxazoline-derived ligands and indeed she was one of my first final year undergraduate project students in 1993. She developed a highly diastereoselective synthesis of the diphenylphosphinoferrocenyl oxazolines (**8**), Scheme 4. She applied their Pd π -allyl complexes to the enantioselective amination of the test substrate, 1,3-diphenylpropenyl carbonate, with benzylamine in moderate to high conversions with enantioselectivities of up to 72% for the (*S*)-valinol-derived oxazoline complex, Scheme 5.³⁰



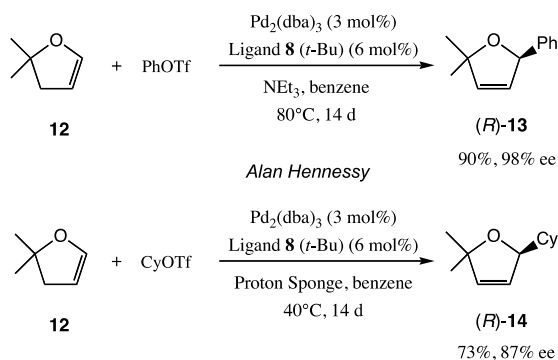
Scheme 5.

At this early stage we had also developed a keen interest in the asymmetric Heck reaction as the design and application of new chiral ligands was becoming pivotal to reaction enhancement of both the asymmetric inter- and intramolecular Heck reaction variants.³¹⁻³⁶ Alan Hennessy was the first PhD student to focus on this reaction and he and fellow PhD student Tim Kilroy exploited the range of P,N ligands prepared by the group to study their application in the asymmetric intermolecular Heck reaction, focusing on the arylation and cyclohexenylation of 2,3-dihydrofuran (**9**).³⁷ Palladium complexes of Yvonne Malone's diphenylphosphinoferrocenyl oxazolines (**8**, R = *t*-Bu), afforded the kinetic products (**10**) and (**11**) in 52% yield and 99% ee, and 75% yield and 85% ee, respectively, Scheme 6.



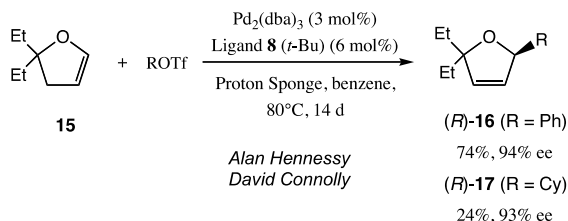
Scheme 6.

Whilst explaining the catalytic cycle of the intermolecular asymmetric Heck reaction using 2,3-dihydrofuran in a final year undergraduate lecture, it occurred to me that dihydrofurans disubstituted at the 2-position could be interesting new substrates as they could only form a single regioisomeric product, thus providing a true comparative test of enantioselectivity of a range of palladium complexes. Therefore, Alan Hennessy synthesised 2,2-dimethyl-2,3-dihydrofuran (**12**) and, with then 4th year project student David Connolly, they also prepared 2,2-diethyl-2,3-dihydrofuran and studied their application in the intermolecular asymmetric Heck reaction with both diphosphine and P,N ligands. For both the phenylation and cyclohexenylation of 2,2-dimethyl-2,3-dihydrofuran (**12**) the *t*-Bu substituted diphenylphosphinoferrrocenyloxazoline ligand (**8**) gave the best results of 90% yield and 98% ee for the phenylated product (**13**), and 73% yield and 87% ee for the cyclohexenylated product (**14**), respectively, Scheme 7.³⁸⁻³⁹



Scheme 7.

The use of 2,2-diethyl-2,3-dihydrofuran (**15**) as substrate demonstrated that the increased bulk at the 2-position had a deleterious effect on both the chemical yields and ees in phenylations and cyclohexenylations although enantioselectivities of 94% and 93% were obtained for products (**16**) and (**17**), respectively, Scheme 8.



Scheme 8.

In terms of a starting seven-year period, I could not have been more lucky with the PhD students and undergraduate students I had worked with. We managed to find a niche in the design, synthesis and application of a range of P,N ligands,⁴⁰⁻⁴¹ with a focus on organopalladium chemistry, then a field of growing importance in academia and industry. We had very limited research funding, which suited our work in catalysis as we performed our reactions on such a

small scale! The underpinning analytic techniques for % ee determination were still lacking as we did not have ready access to chiral HPLC or GC and many of my weekends were taken up measuring % ees on the 500 MHz NMR spectrometer using chiral shift reagents, especially for our allylic alkylation work. We relied on collaborations with John Brown (hydroboration) and Andreas Pfaltz (Heck reactions) to build our expertise in % ee measurement and bring that back to UCD. The excellent group members I had for the starting period of the group was instrumental in attracting further excellent students and we built a very solid foundation for future work.

UCD- The Middle Years (2001-2008)

We were very fortunate that the Irish Government, through its Programme for Research in Third Level Institutions (PRTL), decided to enhance the research infrastructure and capabilities across many areas of Science. The award of €26m to the Centre for Synthesis and Chemical Biology (CSCB) in late 2001 was incredibly significant to the development of the chemical sciences in UCD and our collaborating institutions, the RCSI and TCD. This led to the building and/or refurbishment of synthetic chemistry laboratories and the purchase of a suite of NMR spectrometers, mass spectrometric equipment (low and high resolution), X-ray diffractometers and a series of HPLC and GC and associated chiral columns. The award also supported a large number of PhD studentships and postdoctoral fellowships over a 6-year period to the benefit of a range of academic staff.

Having had considerable success with our first two Quinazolinap ligands (**5** and **6**), we wished to expand our work in the area of axially chiral P,N ligands for asymmetric catalysis.⁴²⁻⁴³ Cormac Saunders prepared the unsubstituted example **17**, Anne-Marie Carroll the benzyl-substituted analogue **18** and David Connolly the isopropyl and *tert*-butyl substituted ligands **19** and **20**, Figure 4. The synthesis of the latter two examples was made possible by the development of a facile and versatile route to 2-substituted-4(3*H*)-quinazolinones and quinazolines.⁴⁴⁻⁴⁵

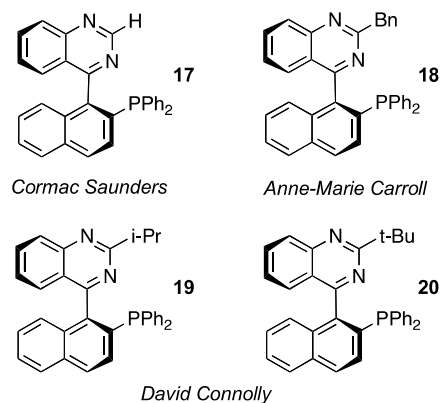


Figure 4.

These ligands were successfully applied to the rhodium-catalysed hydroboration of a series of styrenes, stilbenes and cyclic alkenes. The Quinazolinap catalysts were found to be extremely active, giving excellent conversions, good to complete regioselectivities and the highest enantioselectivities obtained to date for several members of the vinylarene class, including *cis*- β -methylstyrene (97% with ligand **19**), *cis*-stilbene (99% with ligand **6**) and indene (99.5% with ligand **6**).

Subsequently, Susan Flanagan prepared and resolved the 2-(2-pyridyl) and 2-(2-pyrazinyl)-Quinazolinap ligands **21** and **22**, Figure 5. These were designed in order to probe the importance of the second nitrogen donor atom as a potential hemilabile donor and X-ray crystal structures showed conclusively that these were bound in a tridentate manner. She applied these in Pd-catalysed allylic alkylation, Scheme 1, with moderate conversions and enantioselectivities of up to 81%.⁴⁸

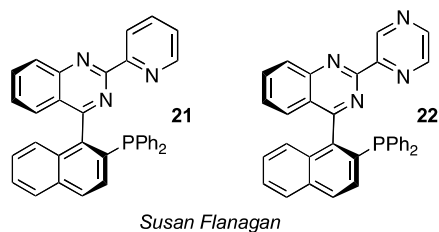


Figure 5.

The structures of the palladium cations of variously substituted 2-Quinazolins (H=green (**17**); isopropyl=red (**19**); pyridyl=yellow (**21**); pyrazinyl=white (**22**)) with the (*R*)-dimethyl[1-(1-naphthyl)ethyl]aminato ligand used for their resolution, are shown in Figure 6.

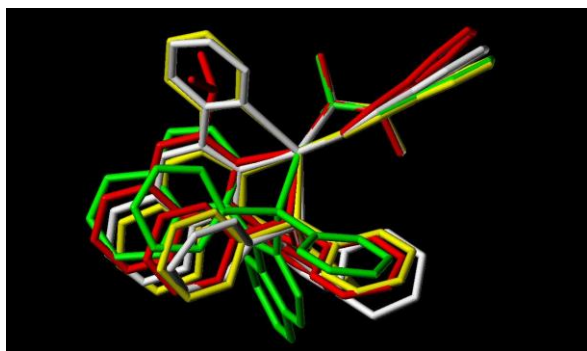


Figure 6.

These ligands (**21** and **22**) were successfully applied to the rhodium-catalysed hydroboration of alkenes and these induced high enantioselectivities using substrates such as indene (84% ee for **21**; 73% for **22**), and 1,2-dihydronaphthalene (91% ee for **22**).⁴⁹

Building upon our previous work on ferrocene-containing ligands **3** and **8**, we were interested in a series of planar chiral *N,O*-ferrocenyl pyrrolidines with varying substituents at the nitrogen and oxygen donor atoms, typified by examples **23** – **26**, Figure 7. This was work done by Theresa Ahern who introduced the oxygen donor atom via a diastereoselective *ortho*-metalation of *N*-methylpyrrolidinyl and *N*-allylpyrrolidinyl ferrocene intermediates followed by quenching with various ketones.⁵⁰

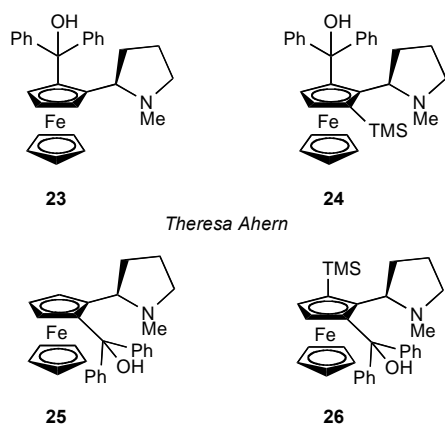
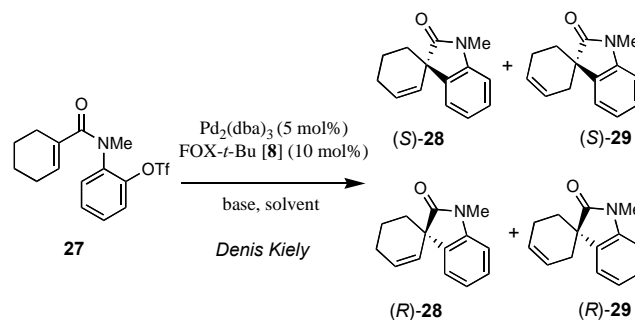


Figure 7.

The efficacy of these novel ligands was investigated in the enantioselective addition of diethylzinc and diphenylzinc to aromatic aldehydes. The ligands proved highly effective in the diethylzinc addition to benzaldehyde that resulted in high yields of up to 99% and ees of up to 95%. The role of planar chirality was explored and the results indicated that the planar chirality, and not the central chirality, of the ferrocenyl ligands was the dominant stereo-controlling element. Employment of a mixed ethyl-phenylzinc reagent in the phenylation of aromatic aldehydes led to a mixture of the two additional products, and the phenylated product was obtained in up to 37% ee.

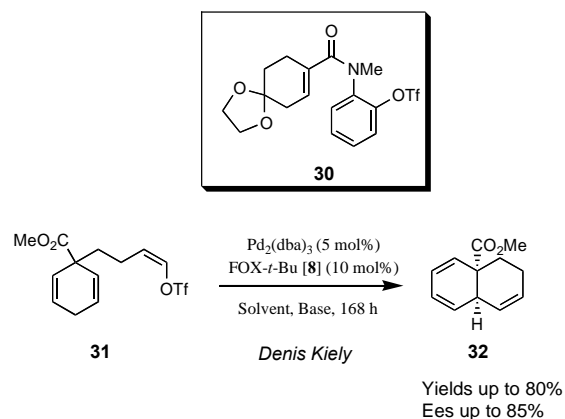
The intramolecular asymmetric Heck reaction process was investigated Irish Chemical News 2015 Issue No.1

by Denis Kiely who prepared a series of novel substrates and compared Pd complexes of well known ligands like BINAP to the range of P,N ligands prepared within our group. For example, the cyclisation of triflate **27** catalysed by Pd complexes of the diphenylphosphinoferrocenyloxazoline-containing ligand **8** prepared by Yvonne Malone gave **28** in 71% yield with a regioselectivity of >99:1 over **29**, and with a high enantioselectivity of 82%, Scheme 9.⁵¹



Scheme 9.

The related aryl triflate substrate **30** was tested and cyclisations proceeded in poor to moderate yields for all of the catalyst systems screened, with an optimal ee of 47% being afforded with Pd complexes of the diphenylphosphinoferrocenyloxazoline-containing ligands **8**, Scheme 10.⁵² He also studied the asymmetric intramolecular Heck cyclisation of alkenyl triflate **31** to form *cis*-decalins of type **32** with an optimal 85% ee, Scheme 10.⁵³



Scheme 10.

Building upon our work with axially chiral P,N ligands and the *N,O* ligands prepared by Theresa Ahern, Brian Sweetman proceeded to develop a high-yielding synthesis, and subsequent resolution via molecular complexation with *N*-benzylcinchonidium chloride salt, of the isoquinoline-containing tridentate ligand **33**, Figure 8.⁵⁴ The barrier to rotation about the central biaryl axis was evaluated *via* racemisation studies, and the absolute configuration assigned by X-ray crystallography.⁵⁵

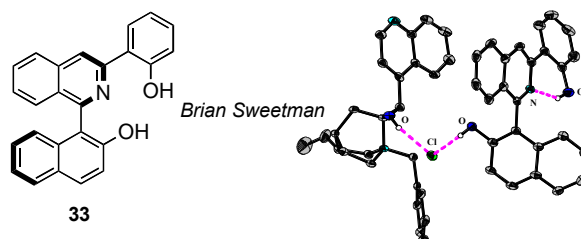
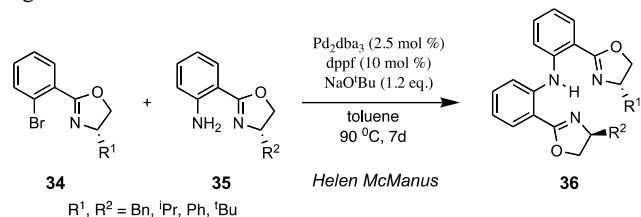


Figure 8.

Chiral tridentate ligands, believed to form a deeper chiral concave pocket around the metal center, have been less extensively used in asymmetric catalysis than their bidentate or tetridentate analogues. The potential for tridentate ligands became a focus of the work of Helen McManus who developed a convergent synthesis of a new class of

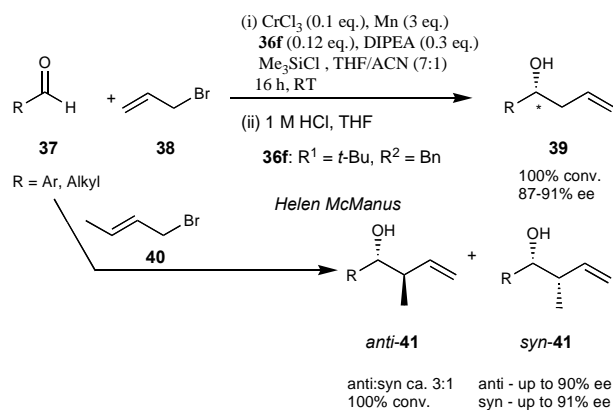
tridentate bis(oxazoline) ligands **36** for asymmetric synthesis, in which an *N*-phenylaniline unit links the two chiral oxazoline rings. The key step in her synthesis was the coupling of bulky, electron-deficient partners **34** and **35** in aryl amination which proceeded in 60–85% yields, Scheme 11.⁵⁶ This allowed for the preparation of C_2 -symmetric and non- C_2 -symmetric bisoxazoline ligands as we were keen to investigate the levels (and sense) of asymmetric induction levels employing such ligands.



Scheme 11.

Although initially designed for the metal-catalysed transfer hydrogenation of ketones, in which we envisaged the metal to chelate to the two oxazoline nitrogen donor atoms leaving the linking secondary amine free to hydrogen bond to the ketonic substrate, Rh and Ir complexes of **36** induced only moderate levels of enantioselectivity.

After some other disappointing results with ligand class **36** (and encouraging words of advice that “these ligands must be good for something”) we became aware of the Nozaki-Hiyama-Kishi (NHK) reaction. This is an important and versatile carbon-carbon bond forming transformation which has been utilised effectively in numerous total syntheses of complex natural products as a consequence of its pronounced chemoselectivity, distinct stereochemical preferences, and high tolerance of functionality in both reaction partners.^{57–59} Helen applied ligand class **36** in the NHK allylation and crotylation of benzaldehyde and found that both the magnitude and sense of the asymmetric induction depended strongly on the nature and combination of the oxazoline substituents.⁶⁰ She obtained allylic alcohols **39** with complete conversion and 87–91% ee, with the non- C_2 -symmetric ligand **36f** ($R^1 = t\text{-Bu}$, $R^2 = \text{Bn}$) affording the optimal enantioselectivities for both allylation and crotylation processes, Scheme 12.



Scheme 12.

In order to probe the potential for other oxazoline-containing ligands in the NHK reaction, Gráinne Hargaden synthesised sixteen members of a new ligand class of type **42** incorporating both an oxazoline ring linked to a chiral protected proline unit by an amide bond, Figure 9.⁶¹ The ligands were applied in the enantioselective NHK allylation of benzaldehyde and gave up to 57% ee. Diastereomeric ligand pairs were prepared in order to determine the role of each chiral centre on enantioselection. An X-ray crystal structure of one ligand is shown in Figure 9.

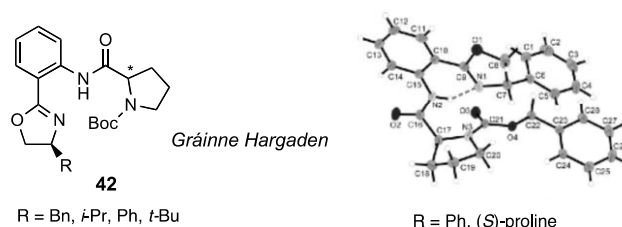
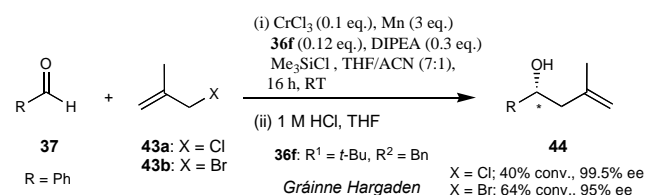


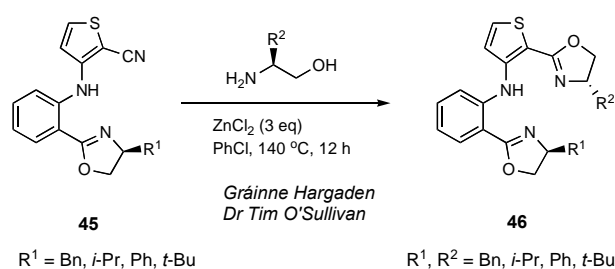
Figure 9.

Gráinne expanded the scope of the application of our bisoxazoline ligands to include the methylation of a range of aldehydes, Scheme 13.⁶² The previously successful non-symmetrical ligand **36f** (with *tert*-butyl/benzyl substituted oxazolines) provided the highest enantioselectivity of 99.5% for the methylation of benzaldehyde using methylaldehyde **43a** although the % conversion was low at 40%, whereas methylaldehyde **43b** was more reactive (64% conversion) but led to lower enantioselectivity (95% ee).



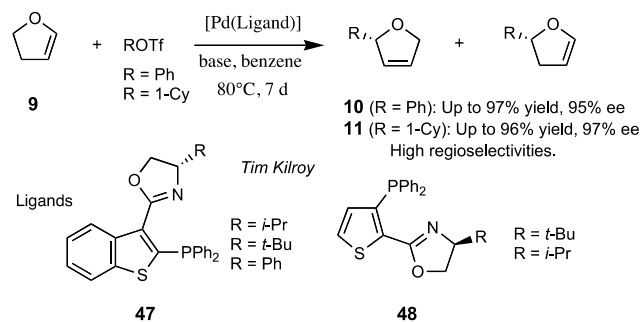
Scheme 13.

As it appeared that we were obtaining our best levels of asymmetric induction with non- C_2 -symmetric ligands we began to investigate other means of desymmetrising the tridentate ligand framework. Gráinne and Dr Tim O’Sullivan developed a high-yielding three-step synthesis of non-symmetrical bis(oxazoline)-containing ligands of type **46** possessing an *N*-thienylaniline unit.⁶³ Their convergent synthesis employed a palladium-catalysed aryl amination of 2-(2'-bromothiophene) nitrile **45** as the key step, with sixteen ligands prepared in total, Scheme 14. These ligands were subsequently applied in the chromium-catalysed enantioselective NHK allylation of benzaldehyde with an optimal enantioselectivity of 73% (88% conv.) afforded with ligand **46** (with $R^1 = t\text{-Bu}$ and $R^2 = \text{Bn}$ substituted oxazolines) whereas the ligand with the reverse substitution pattern ($R^1 = \text{Bn}$ and $R^2 = t\text{-Bu}$) afforded 62% ee.



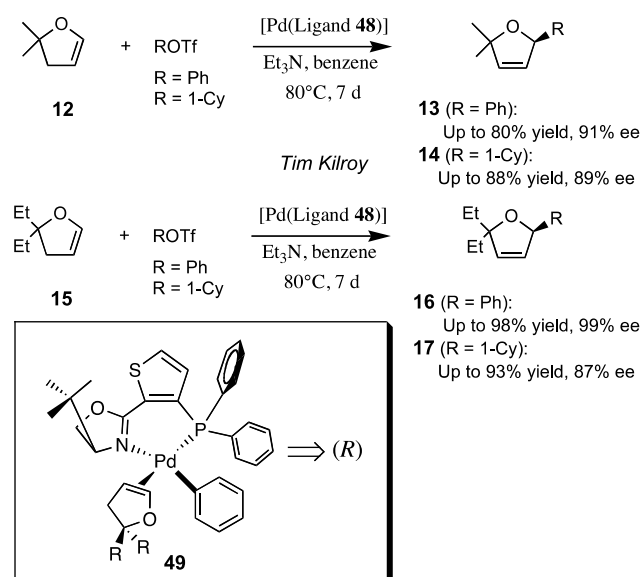
Scheme 14.

Tim Kilroy extended the range of P,N oxazoline-containing ligands by synthesising a range of heterocyclic phosphinoxazolines (HETPHOX) derived from thiophene and benzothiophene of type **47** and **48**, respectively, and applied them in the intermolecular asymmetric Heck reaction, Scheme 15.⁶⁴ Phenylation of the standard substrate 2,3-dihydrofuran **9** with the *t*-butyl-substituted thiophene ligand **48** gave (*R*)-2-phenyl-2,3-dihydrofuran (**10**) highly regioselectively with excellent enantioselectivity (91–95% ee) and in good yields (70–97%). In addition, the reaction of 2,3-dihydrofuran **9** with cyclohexenyl triflate proceeded with up to 97% ee in up to 97% yield.



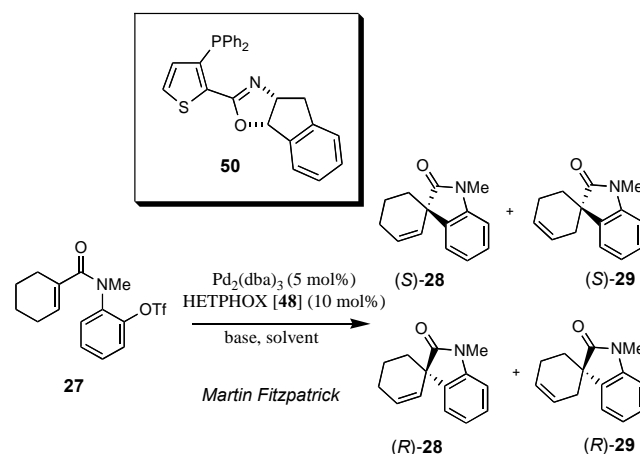
Scheme 15.

Tim also tested 2,2-dialkyl-2,3-dihydrofurans as substrates and the phenylation and cyclohexenylation of 2,2-dimethyl-2,3-dihydrofuran (**12**) proceeded in high yields and ees up to 91% and 89%, respectively, Scheme 16.⁶⁵ The phenylation and cyclohexenylation of the 2,2-diethyl analogue (**15**) proceeded in excellent yields and ees up to 99% and 87%, respectively. For each substrate palladium complexes formed from the *t*-butyl-substituted ligand **48** gave the highest yields, regioselectivities and enantioselectivities over the broad range of reaction conditions studied. 2,2-Diisopropyl-2,3-dihydrofuran was prepared but was found to be unreactive in the intermolecular Heck reaction thus providing insight into the steric limits for 2,3-dihydrofuran substrates.⁶⁶ For the phenylation of these dihydrofurans we proposed that when the alkene approach is *trans* to phosphorus, intermediate (**49**) does not suffer steric repulsion and this route for migratory insertion would lead to a high ee of the (*R*) product and this is what we observed experimentally.



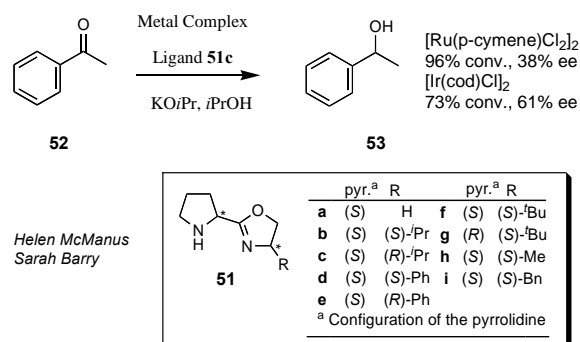
Scheme 16.

Martin Fitzpatrick prepared a novel thiophene-oxazoline P,N ligand **50** derived from *cis*-aminoindanol and tested it and a range of analogous HETPHOX ligands **47-48** in the intramolecular Heck reaction, Scheme 17.⁶⁷ The enantioselectivity obtained was 76% employing the *t*-butyl substituted HETPHOX ligand **48** with an aryl triflate spirooxindole precursor **27**. The isomer distributions of the product spirooxindoles were high (up to 99:1) favouring **28** over **29**.



Scheme 17.

Helen McManus and Sarah Barry continued work on oxazoline-containing ligands and prepared nine members of a new ligand class (**51a-i**) incorporating both an oxazoline ring and a pyrrolidine unit were prepared in an efficient four-step synthesis starting from readily available chiral amino alcohols and proline. A study of these ligands in the asymmetric transfer hydrogenation of acetophenone showed that the catalysts formed from ligand **51c** and [Ir(cod)Cl]₂ were the most active while those derived from [Ru(*p*-cymene)Cl]₂ gave the highest enantioselectivities (up to 61% ee), Scheme 18.⁶⁸



Scheme 18.

Building upon our expertise in axially chiral P,N ligands and oxazoline chemistry, Dr Tom Fekner developed a practical synthesis of potentially tridentate P,N,N-ligands of type **54** containing two stereogenic elements incorporated into the axially-chiral Quinazolinap and centrally-chiral 2-oxazoline sub-units, Figure 10.⁶⁹ These ligands possess an in-built resolution motif obviating the need for the use of the expensive chiral palladacycle approach for resolution. The application of these novel hybrid ligands in Pd(0)-catalyzed asymmetric allylic alkylation, Scheme 1, revealed the matched and mismatched diastereomer, dominant stereogenic element, as well as the effect of the oxazoline R substituent on the level of enantioselectivity (ee up to 81%).

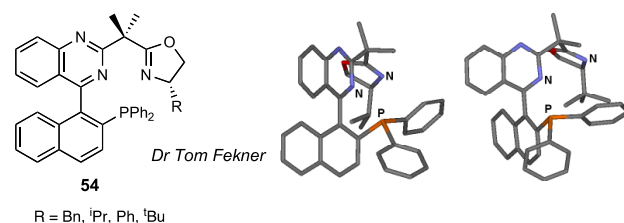


Figure 10. X-ray structures of ligands (*S*,*S*)-(*i*-Pr)-**54** (left) and (*S*,*S*)-(*t*-Bu)-**54** (right). Hydrogen atoms omitted for clarity.

Inspired by the advent of novel phosphite and phosphoramidate ligands and the considerable success of their metal complexes in asymmetric catalysis, Dr Raymond Bronger further exploited our background in oxazoline chemistry through his synthesis of a novel series of aminophosphine-oxazoline **55** and phosphoramidite-oxazoline **56** ligands, Figure 11.

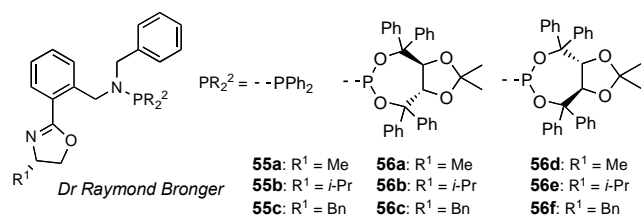
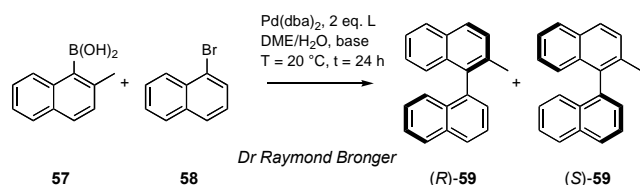


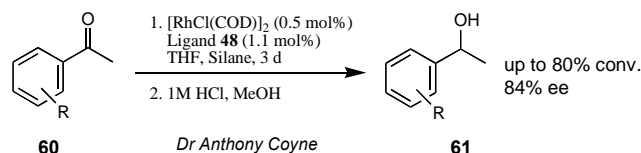
Figure 11.

The efficacy of the aminophosphine-oxazoline ligands **55** was investigated in the Pd-catalysed asymmetric allylic alkylation of 1,3-diphenylprop-2-enyl acetate, Scheme 1, leading to a maximum of 38% ee at 64% conversion. Phosphoramidite-oxazoline ligands **56**, however, gave ees of up to 87% at 71% conversion in the same reaction and also proved to be effective in the important Pd-catalysed asymmetric Suzuki coupling between 2-methylnaphthylboronic acid (**57**) with 1-bromonaphthalene (**58**) leading to a maximum of 46% ee (*R*) in 54% isolated yield of biaryl **59** at room temperature, Scheme 19.⁷⁰



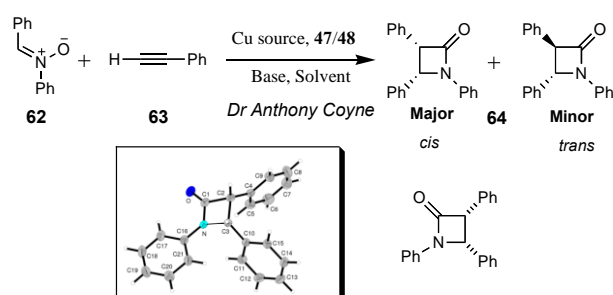
Scheme 19.

The breadth of reactions to which we applied literature and novel phosphorus/oxazoline ligands continued to be a research focus.⁷¹ To that end, Dr Anthony Coyne applied the HETPHOX ligand class to the Rh-catalysed asymmetric hydrosilylation of a range of substituted acetophenones of type **60**, Scheme 20.⁷² Enantioselective hydrosilylation of acetophenone with the *t*-butyl substituted thiophene oxazoline **48** gave (*R*)-phenylethanol **61** in excellent enantioselectivity (84% ee) and in good conversion (80%) which is comparable to that obtained using other P,N ligands.



Scheme 20.

Dr Coyne also studied the reaction of nitrones **62** with terminal alkynes **63**, catalysed by a range of copper complexes of HETPHOX ligands of type **47** and **48**, which afforded β -lactams **64** in moderate to good conversions with ees up to 55%, Scheme 21.⁷³ High levels of diastereoselectivity, dependent on the alkyne, were obtained. For example, the reaction is highly *cis*-diastereoselective with phenylacetylene (>9:1), while an unexpected reversal of diastereoselectivity was observed with the 3,5-trifluoromethylphenylacetylene, which is highly *trans*-selective (1:9) with an ee of 53%. An X-ray crystal structure of the major, *cis*-product **64** is illustrated in Scheme 21.



Scheme 21.

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The design of our Quinazolinap ligands relied primarily on variation of the steric bulk at the 2-position and this was extended by Dr Tom Fekner who developed an expedient, seven-step synthesis of two new members of the Quinazolinap ligand family, 2-cyclobutyl- and 2-(1-adamantyl)-Quinazolinaps **65** and **66**, respectively, Figure 12.⁷⁴ The enantioenriched ligands provide good levels of enantioselection (ee's up to 89%) in a prototypical Pd(II)-catalysed allylic alkylation reaction, Scheme 1. 2-Cyclobutyl-Quinazolinap was further functionalised at the 2-position *via* metalation with a superbase followed by reaction with a range of electrophiles. X-ray crystal structures of Pd(II) complexes of **65** and **66** are shown in Figure 13, along with the phosphine oxide of **66**.

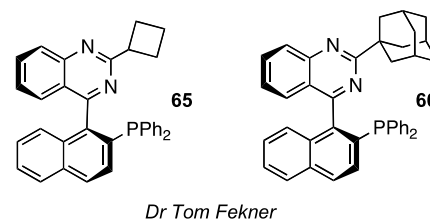


Figure 12.

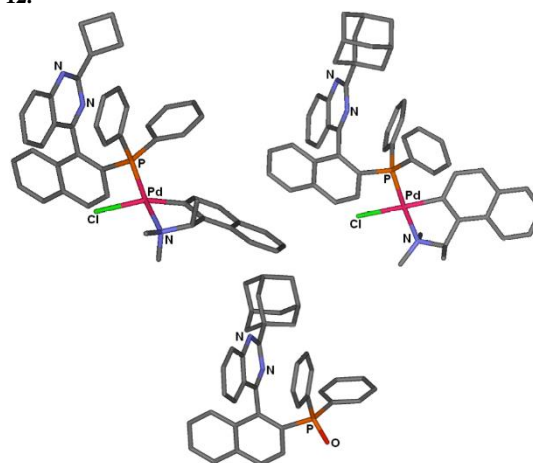


Figure 13. X-ray structures of Pd(II) complexes (*S_sR*)-**65** (top left) and (*R_sR*)-(+)-**66** (top right), and phosphine oxide (*S_s*)-**65**-O (bottom).

As the Director of the Conway Institute of Biomedical and Biomolecular Science at UCD, I hosted a visit in 2004 by the Scientific Advisory Board and Professor Charlie Serhan (Harvard University) delivered an impressive lecture on lipoxins, an important class of biologically active mediators derived from arachidonic acid. The major lipoxins, LXA₄ **67** and LXB₄ **68**, are conjugated tetraene-containing eicosanoids, Figure 14, which act to mediate inflammatory responses by interfering with neutrophil and eosinophil adhesion and migration. However, the accumulation of LXA₄ at the site of inflammation is short lived as it is rapidly metabolised *in vivo* into inactive metabolites. Their instability is a major obstacle to the application of these compounds as important pharmacological agents.

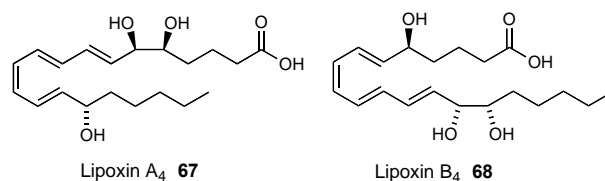
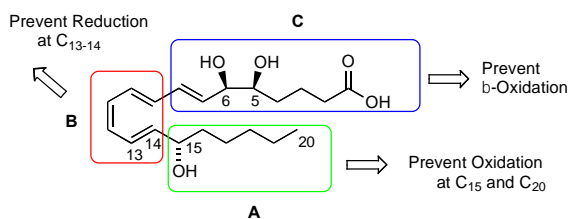


Figure 14.

Therefore, considerable synthetic efforts have gone into mimicking the core structure of LXA₄ **1** by replacing certain functionalities with chemically stable motifs, sub-divided into 3 distinct categories (**A-C**) based on the target area being modified, Scheme 22, in order to retain the potent biological activity.⁷⁵



Key: (A) structural modifications of the C₁₅–C₂₀ chain; (B) replacement of the triene with aromatic / heteroaromatic systems; and (C) modifications of the C₁–C₈ unit.

Scheme 22.

Thus began our foray into medicinal chemistry and I was particularly fortunate to have at that time in the group four excellent postdoctoral fellows, Drs Tim O'Sullivan, Karl Vallin, Jerome Fakhry and Tasadaque Ali-Shah, who helped to build up our expertise in this new field. Our starting point was the design of the benzo-LXA₄ analogue **69** and the related benzo-LXB₄ analogue **70**, Figure 15.

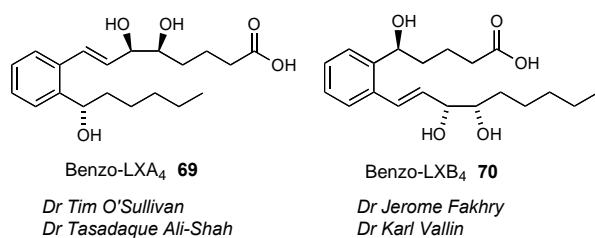
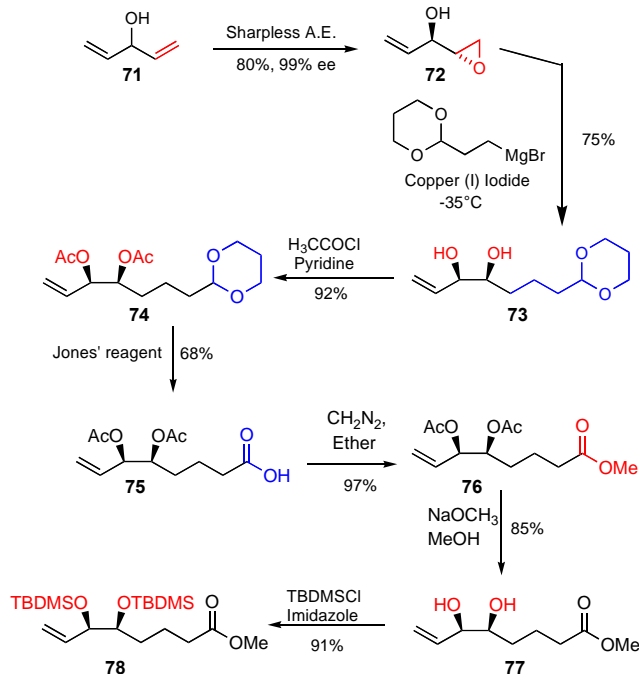


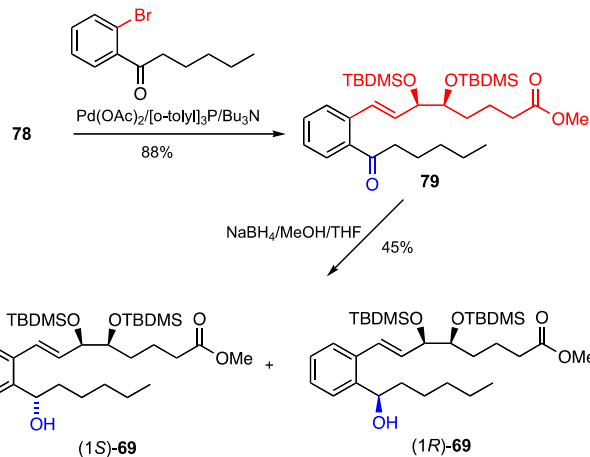
Figure 15.

After much hard work, which included the investigation of a variety of synthetic routes to **69** and **70**, we developed a modular, stereoselective synthesis of benzo-LXA₄ and LXB₄ analogues by employing Sharpless epoxidation, Pd-mediated Heck coupling and diastereoselective reduction as the key transformations. As an example, the alkene coupling partner (**78**) for the Heck coupling, is shown in Scheme 23.



Scheme 23.

The Heck coupling of **78** to afford ketone **79** allowed for the non-stereoselective reduction with NaBH₄ to give an epimeric mixture of alcohols which were separable by column chromatography, Scheme 24. Subsequent asymmetric ketone reductions were performed using (–)-β-chlorodiisopinocampheyl borane or CBS-reduction protocols using oxazaborolidines. More recently we have required the use of asymmetric transfer hydrogenations and metal-catalysed reductions using molecular hydrogen in order to achieve both high chemical yields and diastereoselectivities.



Scheme 24.

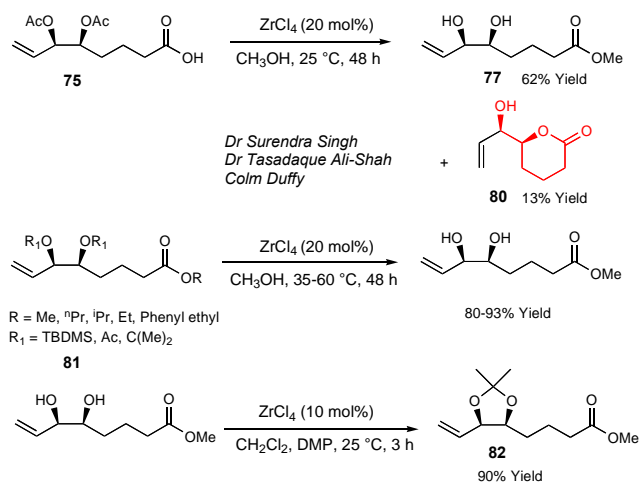
Subsequent biological testing, in an interesting and fruitful collaboration with Professor Catherine Godson at the UCD Conway Institute, has shown that these analogues display potent biological activities.⁷⁶ Both LXA₄ analogues (1*R*)-**69** and (1*S*)-**69** were found to result in a significant increase in phagocytosis of apoptotic polymorphonuclear leukocytes (PMN), with comparable efficacy to the effect of native LXA₄, albeit with greater potency, while the LXB₄ analogue **70** also stimulated phagocytosis with a maximum effect observed at 10⁻¹¹ M. LX-stimulated phagocytosis was associated with rearrangement of the actin cytoskeleton consistent with that reported for native lipoxins.

In terms of a middle seven-year period, I again could not have been more fortunate with the PhD students and undergraduate students I had worked with. The big change during this period was the number of excellent postdoctoral fellows who joined the group as their expertise allowed us to branch out into new research areas and definitely led to a more professionalised research group approach. We became far more international also with postdoctoral fellows coming from Sweden, France (2), the Netherlands, Poland and Pakistan. We continued to work in the design, synthesis and application of a range of P,N ligands, with a focus on organopalladium chemistry, most notably in allylic substitutions and inter- and intramolecular Heck reactions. We also had begun to extend the range of catalytic asymmetric transformations that we studied and applications of these methodologies in natural product synthesis became more of a focus.

UCD- The Later Years (2008-2014)

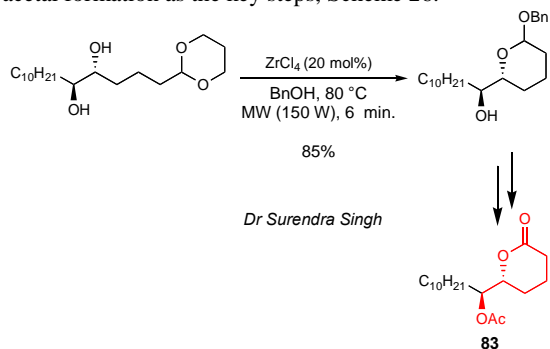
The Lipoxin medicinal chemistry effort also led to some unexpected discoveries which brought our research down some new avenues of synthetic methodology development and exploitation in natural product synthesis. For example, in our synthesis, we prepared (5*S*,6*R*)-methyl-5,6-dihydroxyoct-7-enoate (**77**) as a key intermediate in a two step procedure from (5*S*,6*R*)-5,6-diacetoxyoct-7-enoic acid (**75**) involving conversion of the acid to the methyl ester (**76**) using diazomethane and subsequent diacetate deprotection under basic conditions at low reaction temperatures, Scheme 23. In light of the ability of ZrCl₄ to catalyse a wide range of transformations, including the esterification of acids, and its potential to promote acetate deprotection, Dr Surendra Singh, Colm Duffy and Dr Tasadaque Ali-Shah investigated its use in a one-pot conversion of (5*S*,6*R*)-5,6-diacetoxyoct-7-enoic acid (**75**) to (5*S*,6*R*)-methyl-5,6-dihydroxyoct-7-enoate (**77**). They developed an efficient one-pot method for the esterification and deprotection of diacetates **75** using ZrCl₄ as a catalyst, Scheme 25.⁷⁷

They noted an interesting lactone by-product (**80**) and also found that ZrCl₄ can be used for the deprotection of different protecting groups such as acetonide, bis-TBDMS and diacetate giving excellent yields of the corresponding diols (**77**). They found that ZrCl₄ is an efficient catalyst for the trans-esterification of different esters (**81**) and was also a novel catalyst for the protection of 1,2-diols as an acetonide **82**.



Scheme 25.

The lactone by-product (**80**) contains the key structural unit in the mosquito attractant pheromone (–)-(5*R*, 6*S*)-erythro-6-acetoxy-5-hexadecanolide (**83**). Dr Surendra Singh developed a stereoselective synthesis of **83** which was completed over seven steps in 28% overall yield using Sharpless asymmetric epoxidation and ZrCl₄-catalysed cyclic acetal formation as the key steps, Scheme 26.⁷⁸



Scheme 26.

Dr Surendra Singh then improved upon this ZrCl₄-catalysed acetal formation by a microwave-assisted process (typically 5-10 min. reaction time at 150 W) as a key step in the asymmetric synthesis of substituted tetrahydropyrans, exemplified by the short and efficient synthesis of *endo*-(+)- and *exo*-(+)-brevicomin (**84**, **85**), respectively,⁷⁹ (–)-*exo*-isobrevicomin (**86**) and the volatile contributor of beer aroma (**87**), Figure 16.⁸⁰

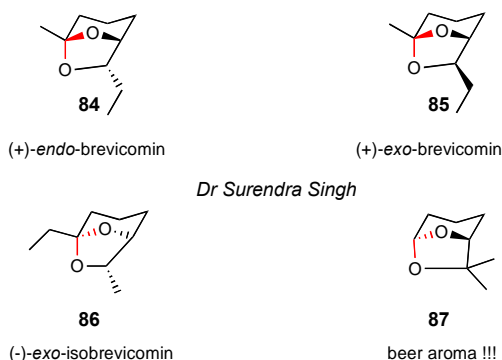
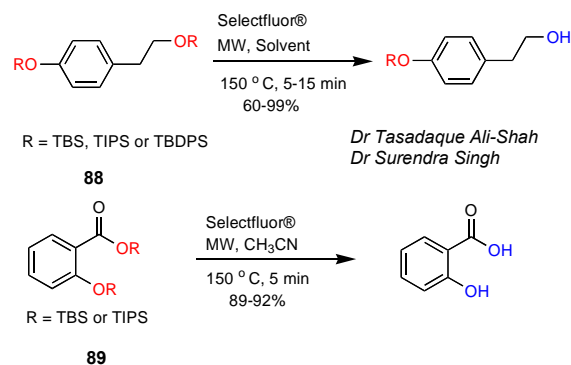


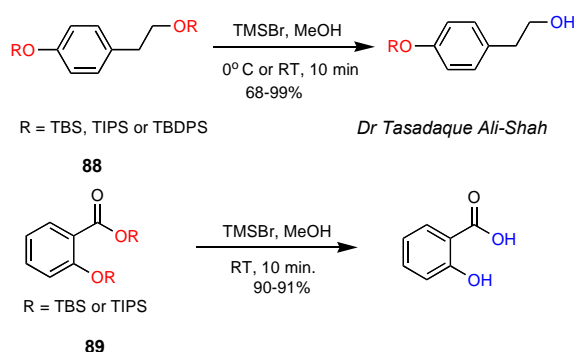
Figure 16. The bonds formed in the ZrCl₄-catalysed acetal formation are shown in red.

Again arising from our Lipoxin work, Drs Tasadaque Ali-Shah and Surendra Singh developed a novel microwave-assisted, chemoselective and efficient method for the cleavage of silyl ethers (aliphatic and aromatic) catalysed by Selectfluor®, Scheme 27.⁸¹ A wide range of TBS-, TIPS-, and TBDPS-protected alkyl silyl ethers of type **88** could be chemoselectively cleaved in high yield in the presence of aryl silyl ethers. The chemoselective deprotection of phenolic TBS ethers **89** and not the TIPS-, or TBDPS-protected phenolic ethers and the deprotection of silyl esters were also achieved under these reaction conditions.



Scheme 27.

Tasadaque further investigated protection group chemistry and developed an efficient and chemoselective cleavage of silyl ethers of type **88** (primary, secondary and aromatic) by using catalytic quantities of trimethylsilyl bromide (TMSBr) in methanol, Scheme 28.⁸² A wide range of alkyl silyl ethers such as TBS, TIPS, and TBDPS were chemoselectively cleaved in high yield in the presence of aryl silyl ethers. The deprotection of silyl esters of type **89** was achieved employing catalytic quantities of TMSBr.



Scheme 28.

Our modular synthesis of the Quinazolinap P,N ligand framework allowed Aoife Maxwell and Dr Celine Franc to probe electronic effects⁸³ by synthesising and resolving electronically varied ligands of type **90** which bear different aryl groups on the donor phosphorus atom, Figure 17.⁸⁴ Their resolution again relied upon the fractional crystallisation of the diastereomeric complexes derived from (*R*)-dimethyl(1-(1-naphthyl)ethyl)amine and in these cases the resolution was not as straightforward as the corresponding diphenylphosphine ligands. The X-ray structure of the (*S_a*,*R*)-palladacycle **91** formed from ligand **90a** is also shown in Figure 17. The application of these ligands in the rhodium-catalysed hydroboration of vinylarenes resulted in enantioselectivities of up to 92%. Rhodium complexes derived from ligands **90a–b** exhibited a decrease in activity and selectivity compared to the parent 2-isopropyl-Quinazolinap **19**. Application of ligands **90a–b** in the palladium-catalysed allylic alkylation of 1,3-diphenylprop-2-enyl acetate, Scheme 1, resulted in conversions of up to 99% and enantioselectivities of up to 94%, the highest ee observed in this reaction to date using the Quinazolinap ligand series.

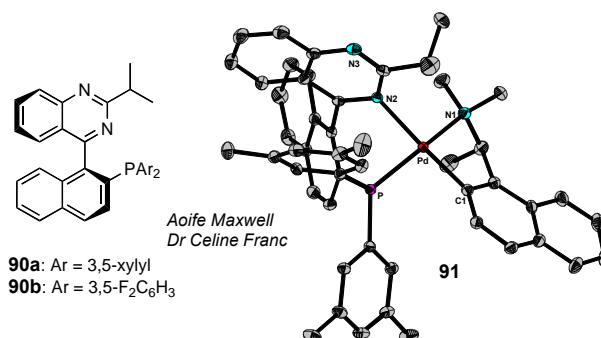


Figure 17. Novel Quinazolinap ligands **90** and X-ray structure of Pd(II) complex (*S_a*,*R*)-**91** isolated during the resolution of ligand **90a**.

Billy Fleming extended our work on Quinazolinap ligands through his synthesis and resolution of the new 7-chloro-substituted Quinazolinap **92**, Figure 18.⁸⁵ This was the first ligand in the Quinazolinap series which allowed for the study of electronic variation at the 7-position. It was also envisaged that post-resolution modification at this position would provide an attractive route to a range of diverse Quinazolinap ligands.

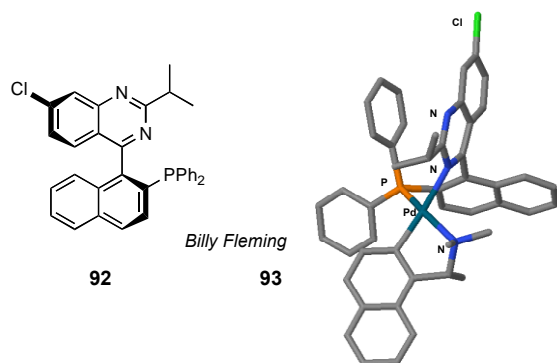
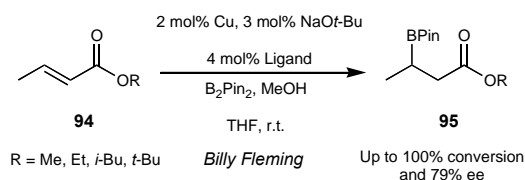


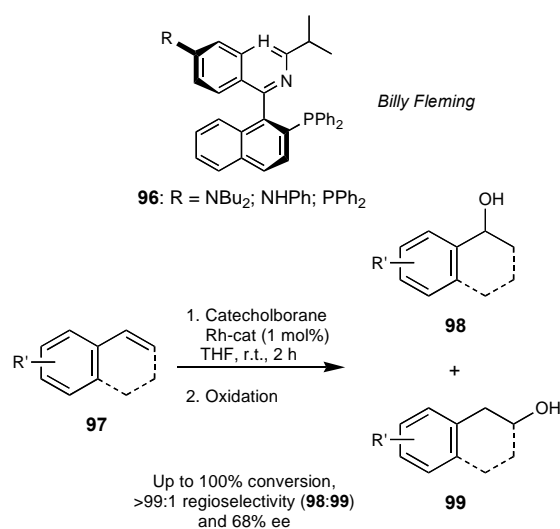
Figure 18. Novel Quinazolinap ligand **92** and X-ray structure of Pd(II) complex (*S_a,R*)-**93** isolated during its resolution.

Billy applied ligand **92** and the related P,N ligands (Quinap and Quinazolinaps **6** and **19**) to the copper-catalysed β -borylation of α,β -unsaturated esters (**94**) resulting in conversions of up to 100% and ees of up to 79% for the borylated products **95**, Scheme 29.



Scheme 29.

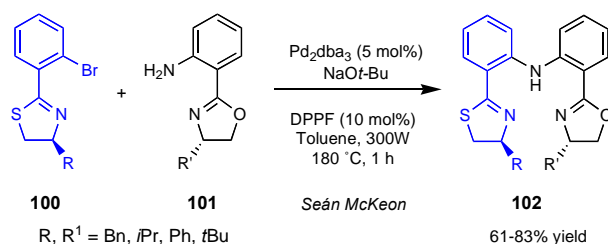
Billy subsequently prepared three further ligands of type **96** from (*R*)-7-chloro-2-isopropylquinazolinap (**92**), an expedient route as it precluded the need for resolution of each ligand prepared.⁸⁶ (*R*)-7-Chloro-2-isopropylquinazolinap was applied to the rhodium-catalysed hydroboration of vinylarenes with regioselectivities of up to >99:1 and ee values of up to 68%, Scheme 30. Each of the Quinazolinap ligands prepared were applied to the palladium-catalysed allylic alkylation of 1,3-diphenylprop-2-enyl acetate, Scheme 1, resulting in conversions of up to 100% and ee values of up to 85%.



Scheme 30.

Building upon our research on oxazoline-containing ligands and exploiting our modular synthesis of the tridentate examples already prepared (e.g. **34** and **46**), Seán McKeon prepared six members of a novel non-*C*₂-symmetric ligand class of type **102** incorporating an oxazoline and thiazoline unit, Scheme 31.⁸⁷ This was a four step, high yielding and convergent synthesis, in which the key step was a

microwave-assisted palladium-catalysed aryl amination between aryl bromide **100** and anilinoxazoline **101**. This synthetic approach allowed for the synthesis of non-symmetric ligands, as with ligand classes **34** and **46**, and in addition gave us the opportunity to selectively place different substituents on either the oxazoline or thiazoline rings.



Scheme 31.

Interestingly, one of the ligands, **102**: R = Bn, R¹ = *t*-Bu, was amenable to structure confirmation by X-ray crystallography, Figure 19. The three nitrogens are in plane with one another forming a concave pocket and it appears as though the ligand is already in a position whereby it could easily bind in a tridentate fashion to a metal centre forming two six-membered rings.

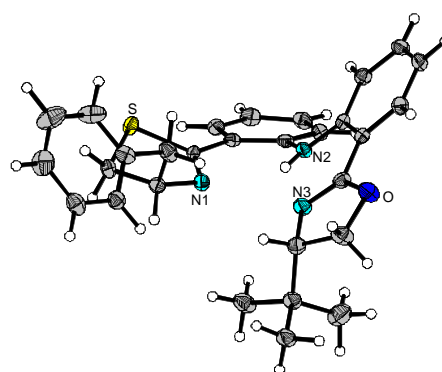
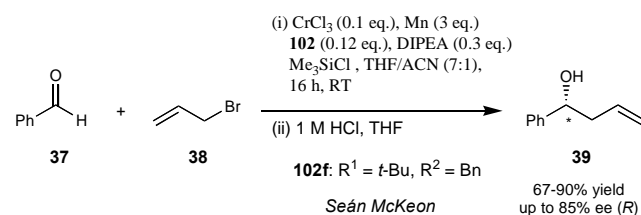


Figure 18. X-ray crystal structure of novel thiazoline-oxazoline ligand **102** (R = *t*-Bu, R¹ = Bn).

Ligands **102** were applied by Seán to the zinc-catalysed enantioselective Friedel-Crafts alkylation of indole with the R = *t*-Bu, R¹ = Bn -substituted oxazoline ligand providing the highest enantioselectivity of 71% when nitrostyrene was employed as the substrate. We then applied this ligand to the alkylation of indole with a range of nitroalkenes with ee values up to 76%.

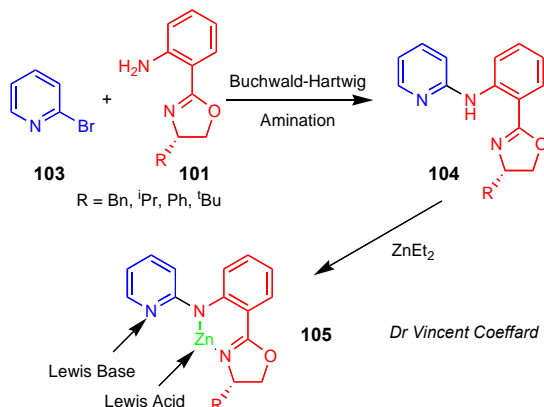
Seán expanded this work by preparing a further eleven novel ligands of type **102** incorporating an oxazoline and thiazoline unit.⁸⁸ The complete series of ligands was applied to the chromium-catalysed Nozaki-Hiyama-Kishi (NHK) allylation of benzaldehyde, affording enantiomeric excesses of up to 85%, Scheme 31, with the R = *t*-Bu, R¹ = Bn-substituted oxazoline ligand providing the highest enantioselectivity. The isomeric R = Bn, R¹ = *t*-Bu-substituted oxazoline ligand afforded allylated product **39** in 55% ee demonstrating the very subtle effects of substitution patterns with these ligands.



Scheme 32.

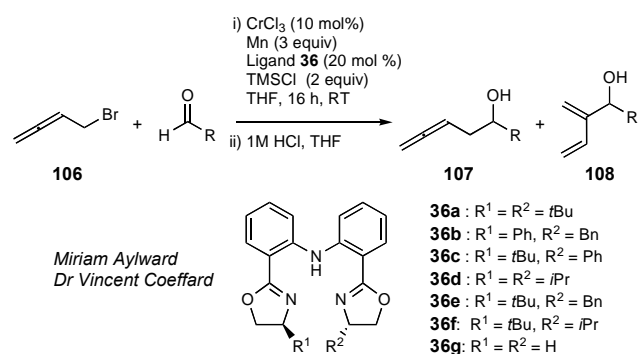
As part of our programme on tridentate bisoxazoline ligands, we had employed 2-(*o*-aminophenyl)oxazolines **101** as key synthetic intermediates. Dr Vincent Coeffard exploited this work as he developed a straightforward preparation of new modular oxazoline-containing bifunctional catalysts of type **104** employing a microwave-assisted Buchwald-Hartwig aryl amination of pyridine **103** as the key step, Scheme 33.⁸⁹ Covalent attachment of 2-(*o*-aminophenyl)oxazolines

and pyridine derivatives generated in good-to-high yields a series of ligands in two or three steps in which each part was altered independently to tune the activity and the selectivity of the corresponding catalysts. These catalysts prepared *in situ* were subsequently applied in the asymmetric addition of diethylzinc to various aldehydes, producing the corresponding alcohols with enantioselectivities of up to 68%.



Scheme 33.

Building upon our work in the NHK reaction we noticed that one of the least well developed C-C bond-forming processes was the reaction between homoallyl bromide **106** with aldehydes. Literature precedent showed the process to be non-regioselective with the formation of the β -allenol and butadiene products **107** and **108**, respectively, in roughly a 3:1 ratio and no enantioselective variants had been reported. Miriam Aylward and Dr Coeffard tested the range of C₂-symmetric and non-C₂-symmetric tridentate bisoxazolines **34** originally prepared by Helen McManus. They successfully developed the first regio- and enantioselective homoallynylation of aldehydes in which it is critical to have access to the non-C₂-symmetric ligands, Scheme 34.⁹⁰ Ligand **34** (R = *t*-Bu, R¹ = *i*-Pr) gave the highest yield (51%) and enantioselectivity (96%) in the homoallynylation of benzaldehyde. This study showed just how important it was to have access to non-C₂-symmetric bisoxazoline ligands as otherwise we would not have been able to develop this transformation to such a high level.



Ligand	107/108	Conv. [%]	Yield [%]	ee [%] (Conf.)
36a	71/29	80	39	8 (S)
36b	74/26	93	33	11 (R)
36c	84/16	85	40	49 (R)
36d	100/0	35	16	84 (R)
36e	100/0	45	23	91 (R)
36f	100/0	87	51	96 (R)
36g	69/31	83	43	-

Scheme 34.

The homoallynylation was also carried out using a range of aromatic and aliphatic aldehydes with moderate yields (40-63%) and excellent enantioselectivities (91-98%), with the best substrate being *meta*-chlorobenzaldehyde, which afforded the β -allenol in 63% yield and

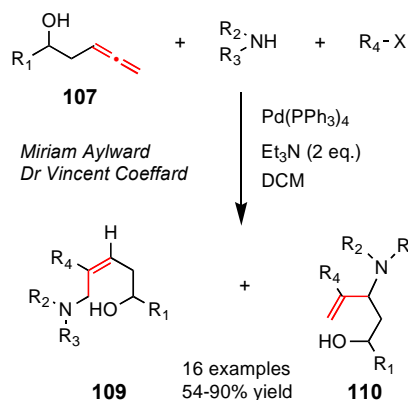
98% ee. The process was completely regioselective, apart from when 1-naphthylaldehyde was employed when a 97:3 ratio of β -allenol:dienylated product was obtained.

When we submitted this paper to *Angewandte Chemie* it was necessary to come up with a catch phrase to describe the work as part of the abstract. It was Friday July 3, 2009 and the semi-final of Wimbledon with Roger Federer playing Tommy Haas. I had the scoreboard up on my computer as I followed the match intermittently and Vincent, Miriam and I were keen to submit that day. As we had developed a high-yielding, regioselective and enantioselective process, this was in tennis terms 'Game, Set and Match'! After acceptance, Dr Peter Goltz asked us to design a cover image and we managed to finally bring together two of my important interests – tennis and chemistry, Figure 19. The chiral tennis racquet interacts with the dienylchromium (tennis ball) intermediate and approaches the *re*-face of the aldehyde net to afford the β -allenol product **107** and the 'match scoreboard' shows the relevant numbers for yield, regio- and enantioselectivity. The chromium-homoallene complex remains out of the game and the dienylated product **108** loses!



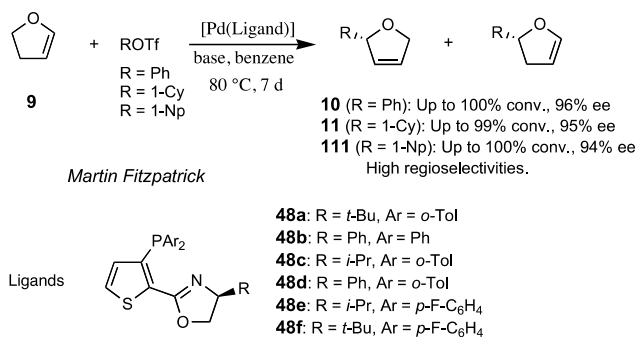
Figure 19.

It is often the case for researchers in asymmetric catalysis to focus on the levels of enantioselectivities and yields obtained in the key asymmetric transformation with little attention paid to the synthetic utility of the products formed. We had no background in allene chemistry but we felt the products of our recently developed NHK homoallynylation, β -allenol product **107**, should be amenable to further functionalisation. Therefore, Miriam and Vincent again developed an interesting palladium-catalysed three-component transformation of enantioenriched homoallenols with aryl halides and amines to selectively afford (*Z*)-configured 1,5-amino alcohols **109** in good-to-excellent yields without any epimerisation, Scheme 35.⁹¹ This reaction possesses the ability to generate valuable chiral building blocks **109**, merging an allene, an amine and an alcohol into the same molecule in an atom-economic fashion.



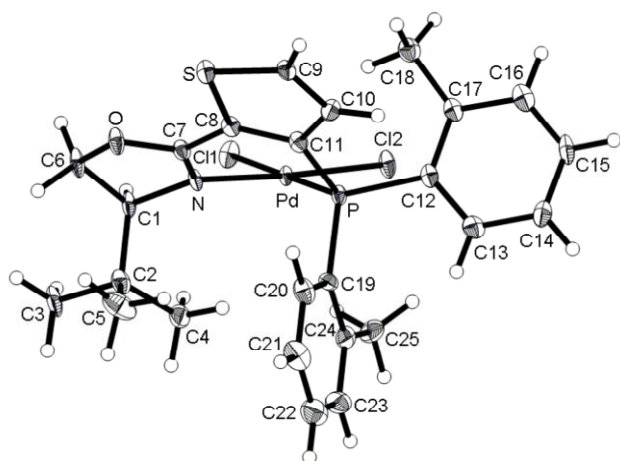
Scheme 35.

Martin Fitzpatrick continued our studies on HetPhox ligands and prepared a further six members of the HetPHOX ligand class and applied them to the palladium-catalysed intermolecular asymmetric Heck reaction, Scheme 36.⁹² The *tert*-leucinol derived ligands proved the most enantioselective, with palladium complexes of these ligands affording ees of up to 96, 95 and 94% in the phenylation, cyclohexenylation and naphthyllation respectively of 2,3-dihydrofuran.



Scheme 36.

As an aid to the explanation of the observed selectivity, a palladium complex of the most enantioselective ligand was prepared and an X-ray crystal structure obtained, Figure 20.

Figure 20. X-ray crystal structure of the PdCl₂ complex of HetPhox ligand **48a**.

Our Lipoxin work was taken up by Colm Duffy, the first PhD student in our medicinal chemistry efforts. Our original exciting results with the benzo-LXA₄ analogue **69**, in addition to the potential for an enhanced pharmacological profile, prompted him to investigate the synthesis of epimeric heteroaromatic LXA₄ analogues of type **112** for biological evaluation, Figure 21, as replacing benzene by heteroaromatic bioisosteres is a well known and successful strategy in medicinal chemistry. Colm's synthesis of these pyridine-containing Lipoxin A₄ analogues had employed an asymmetric reduction of a ketone, a palladium-mediated Heck reaction, a Sharpless asymmetric epoxidation, and a regioselective pyridine lithiation as the key synthetic

steps.⁹³ The pyridine-containing Lipoxin analogues induced a greater increase in phagocytosis of apoptotic polymorphonuclear leukocytes (PMN) by macrophages compared to both the endogenous Lipoxin A₄ and the novel stable aromatic Lipoxin analogue **69**.

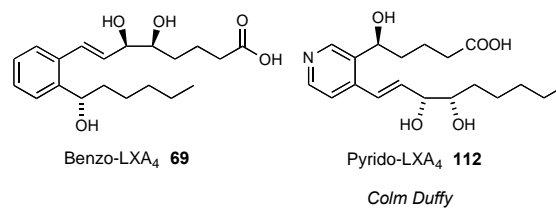


Figure 21.

In the meantime, our benzo-LXA₄ analogue **69** was subjected to further biological evaluation by the groups of Professor Catherine Godson (UCD) and Professor Jesper Haeggstrom (Karolinska Institute, Stockholm). Catherine studied native LXA₄ **67** and **69** in renal fibrosis in a 3 day unilateral uterine obstruction (UUO) model - both compounds showed a trend towards reduced epithelial apoptosis, Figure 22.^{94, 95} LXA₄ and our analogue significantly blunted UUO-induced INF- γ expression and benzo-LXA₄ **69** significantly increased expression of the anti-inflammatory cytokine IL-10, suggesting an active shift towards pro-resolution.

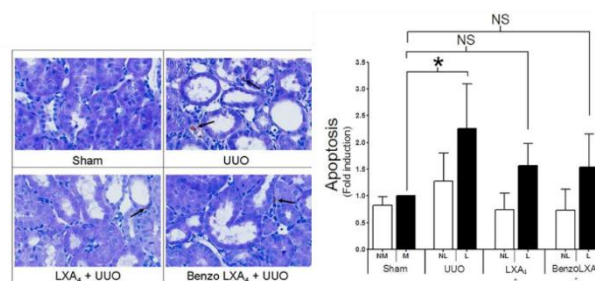
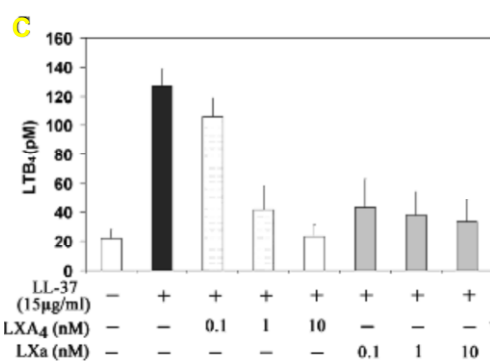


Figure 22.

In humans, the antimicrobial peptide LL-37 and LTB₄ are important pro-inflammatory mediators, whereas LXA₄ possesses anti-inflammatory, pro-resolving properties. Jesper and his group determined that incubation of polymorphonucleocytes (PMNs) with 15 $\mu\text{g/ml}$ LL-37 and different concentrations of LXA₄ or our analogue **69** for 20 min significantly reduced the production of LTB₄, with our analogue being more potent at 0.1 nM, Figure 23.⁹⁶

Figure 23. Lipoxins inhibit LTB₄ release from human PMNs with LL-37 peptide.

Caroline Barth aimed to synthesise a set of novel examples of stable Leukotriene B₃ (LTB₃)/Leukotriene B₄ (LTB₄) analogues of type **113** employing an approach similar to the work on LXB₄ analogues, Figure 24.⁹⁷

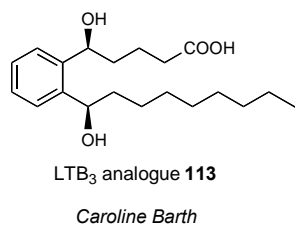
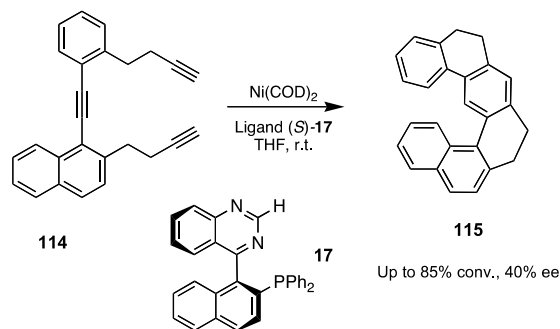


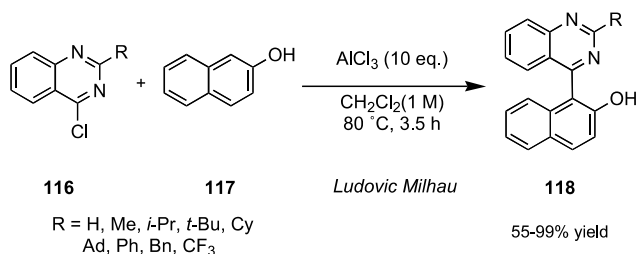
Figure 24.

In a collaboration with Professor Ivo Stary in Prague, they tested our parent Quinazolinap ligand **17** in the Ni(0)-catalysed enantioselective cycloisomerisation of alkyne **114** to afford the chiral helicene **115** in 85% yield and 40% ee, Scheme 37.⁹⁸



Scheme 37.

The original Quinazolinap ligand class synthesis was improved by Ludovic Milhau who investigated and developed a novel, short synthetic route for a key precursor of Quinazolinap ligands. The key step was a Friedel-Crafts type reaction between 4-chloroquinazoline **116** and 2-naphthol **117**, with moderate to quantitative yields of biaryl **118** being observed, Scheme 38.⁹⁹ Overall, five steps were removed from the original Quinazolinap synthesis as the nucleophilic component of the Suzuki reaction, the boronic acid is no longer required as the biaryl is formed directly from 2-naphthol **118**.



Scheme 38.

The design and synthesis of new LXA₄ stable analogues and their biological evaluation continued to be a growth area for the research group. The asymmetric synthesis of 1,3 and 1,4-disubstituted aromatic LXA₄ analogues **119** and **120** was accomplished by Gavin Haberlin, with help from an undergraduate student, Robert Doran, Figure 25. A total of eight analogues were synthesised in a convergent approach utilising Sharpless asymmetric epoxidation, asymmetric reduction and intermolecular Heck reactions as the key steps, Scheme 39.¹⁰⁰ The set of analogues were assessed for their effect on the production of cytokines IL-12p40, IL-23, IL-6, IL-1b and TNF α . A positive inhibitory result was observed for the production of IL-6 and IL-1 β .

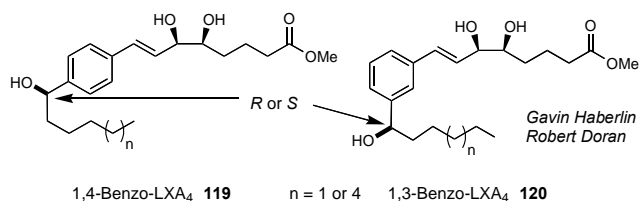
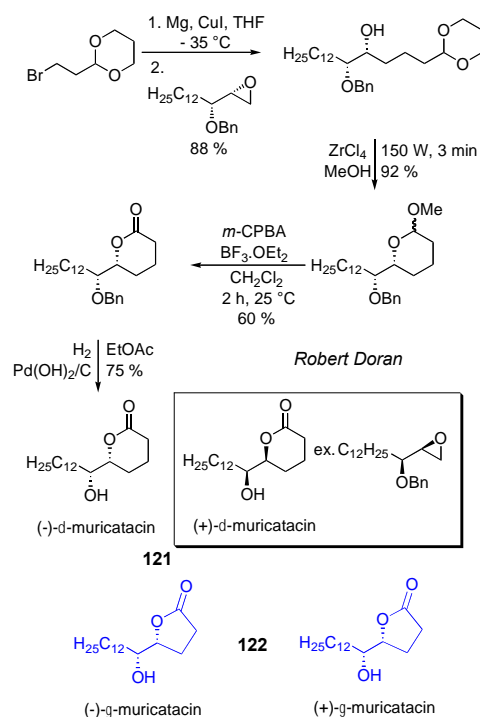


Figure 25.

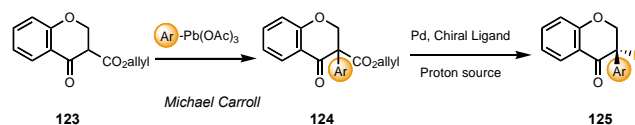
Robert performed the asymmetric synthesis of both enantiomers of the Irish Chemical News 2015 Issue No.1

δ -lactone analogue **121** of the anti-tumoral natural product γ -lactone muricatacin **122**.¹⁰¹ Initial attempts to also synthesise the natural product proved unsuccessful due to the poor reactivity of the Grignard reagent derived from 2-(bromomethyl)-1,3-dioxolane. The designed synthetic route enabled us to increase the ring size to generate the δ -lactone analogue employing Sharpless asymmetric epoxidation and ZrCl₄-catalysed intramolecular acetalisation as the key steps, Scheme 39.



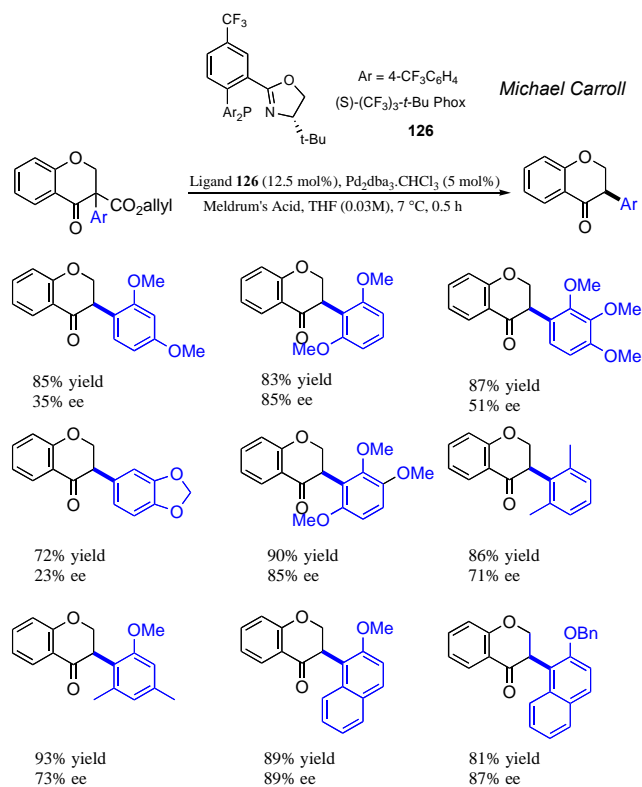
Scheme 39.

Isoflavanones are an important class of natural products containing an α -aryl tertiary stereocenter. We followed the work of Stoltz on palladium-catalysed decarboxylative protonation with interest and we wished to combine our background in P,N chiral ligands,¹⁰²⁻¹⁰⁴ lead mediated arylations¹⁻¹¹ and natural product synthesis in developing a catalytic asymmetric approach to isoflavanones. Michael Carroll investigated a two-step approach from the β -keto ester **123**, using arylead triacetates to α -arylate to prepare the key substrates **124** for his asymmetric catalysis studies in the synthesis isoflavanones **125**, Scheme 40.



Scheme 40.

Michael tested the sterically hindered 2,4,6-trimethoxyphenyl substrate with the *t*-Bu Phox ligand and observed a 78% ee, which was subsequently optimised with the electron-deficient (CF₃)₃-*t*-Bu Phox ligand **126** to a 92% ee using Meldrum's acid as the proton source. Michael illustrated the scope of this catalytic asymmetric approach to isoflavanones (9 further examples) using this ligand with good to excellent yields (72-93%) being obtained under mild conditions in 30 min, Scheme 41.

**Scheme 41.**

Michael's work represents an important addition to the asymmetric preparation of tertiary α -aryl ketones and complements existing methods where sterically hindered examples are generated with reduced yields and enantioselectivities. He was able to determine the absolute configuration of the 2,4,6-trimethoxyphenyl isoflavanone derivative by X-ray crystallography, Figure 26.

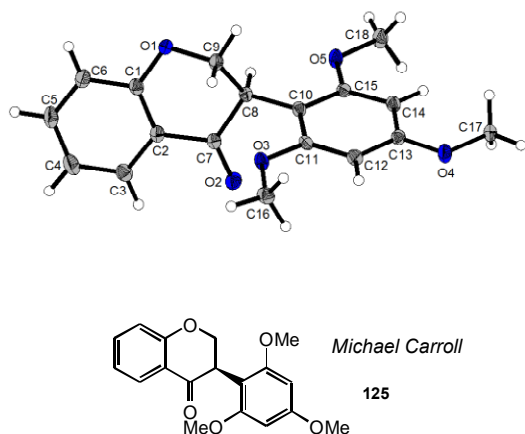
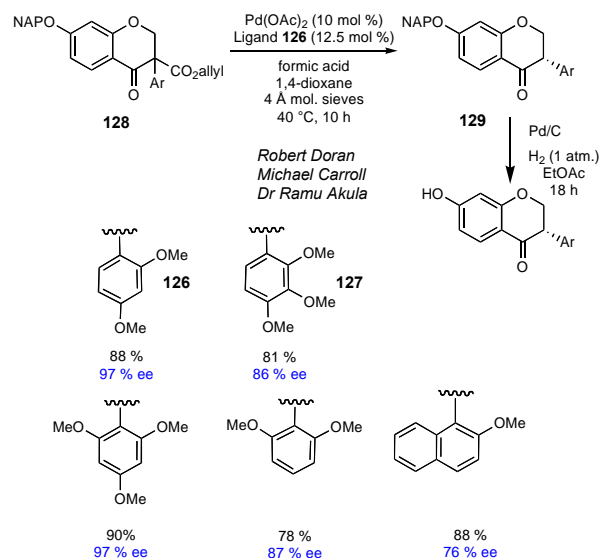
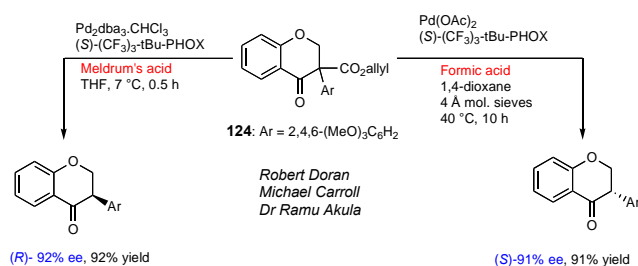


Figure 26. X-ray crystal structure of the 2,4,6-trimethoxyphenyl-isoflavanone **125**.

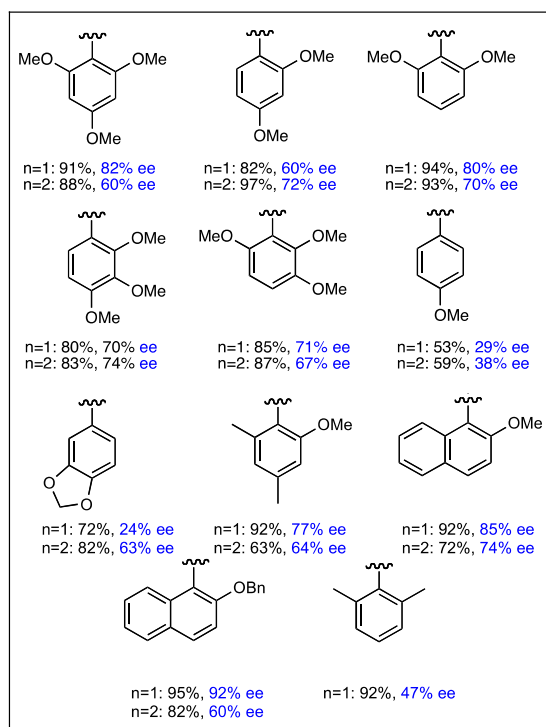
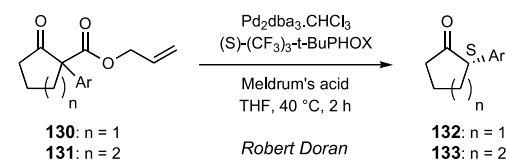
Michael had a short time at the end of his PhD to apply these optimised reaction conditions to the preparation of some naturally occurring examples, in particular sativanone and 3-*o*-methylviolanonone, **126** and **127**, respectively. Unexpectedly the levels of enantioselectivity for their preparation were low, at 2% and 25% ee, respectively. Robert Doran and Dr Ramu Akula took up the challenge to improve upon these results. After careful reaction optimisation, including testing a range of proton sources, they developed a highly enantioselective synthesis of isoflavanones in excellent enantioselectivities from 76-97%, Scheme 42.¹⁰⁵ A switch in the sense of stereoinduction was observed when different H⁺ sources were employed as formic acid afforded the enantiomeric product, showing the first example of dual stereocontrol in an asymmetric protonation reaction.

**Scheme 42.**

Intrigued by this finding, Robert went back and investigated the original β -keto aryl ester **124** using formic acid as the H⁺ source, Scheme 43. This confirmed that the enantiodivergence of this process was retained as we observed the formation of (*R*)-**125** in 91% ee and 91% yield. This finding shows a remarkable switch in enantioselectivity from 92% *R* to 91% *S* as a result of changing the H⁺ source.

**Scheme 43.**

Robert proceeded to extend the substrate scope of the catalytic asymmetric synthesis of a series of tertiary α -aryl cyclopentanones and cyclohexanones using the same Pd-catalysed transformation, Scheme 44.¹⁰⁶ Enantioselectivities of up to 92% ee and 74% ee were obtained for cyclopentanone and cyclohexanone substrates, respectively. The route described gives access to these important structural motifs in moderate to high levels of enantioselectivity. In particular, this is only the second direct approach for the preparation of tertiary α -aryl cyclopentanones. The synthetic approach allows for simple modification of the aryl group and, significantly, substrates containing sterically hindered aryl groups gave the highest levels of enantioselectivity.



Scheme 44.

Miscellaneous Collaborations

We have had a long-standing collaboration with the groups of Professor Alan Keenan, Dr Gethin McBean and Professor Alan Baird in the UCD Conway Institute on the chemistry and biology of MDMA and related compounds. As examples of our work, we have investigated the comparative potencies of 3,4-methylenedioxyamphetamine (MDMA) analogues as inhibitors of [H-3] noradrenaline and [H-3]5-HT transport in mammalian cell lines,¹⁰⁷ studied the *in vitro* neuronal and vascular responses to 5-hydroxytryptamine: modulation by 4-methylthioamphetamine, 4-methylthiomethamphetamine and 3,4-methylenedioxyamphetamine,¹⁰⁸ and *in vitro* neuronal and vascular responses to 5-HT in rats chronically exposed to MDMA.¹⁰⁹ We worked with Professor Kevin O'Connor on the asymmetric synthesis of a chiral alcohol, 1-(3,4-dihydroxyphenyl)ethanol, to help with the identification of its production by mushroom tyrosinase.¹¹⁰

Concluding Remarks and Future Outlook

This article describes much of our published work in asymmetric catalysis, medicinal chemistry and natural product chemistry. It has been interesting to write this article and see how our research programme has developed over the years. We gained a very good grounding in asymmetric catalysis, from ligand design, synthesis and applications in the early years. We built upon this through the study of a variety of interesting synthetic transformations catalysed by a range of transition metal complexes. With enhanced funding, infrastructure and analytical instrumentation we were able to tackle new areas and return to some of my original interests in chemistry - synthetic methodology, mechanism and applications in medicinal chemistry. Our natural products / medicinal chemistry efforts have grown in the past few years and we are also currently working on a variety of new asymmetric transformations with many interesting results obtained recently (even a 99.99% ee for the formation of a quaternary chiral centre!) and these and other results will be reported in due course. Ultimately, the success of our research programme is down to the dedication and hard work of

the students and postdoctoral fellows with whom it has been a real pleasure to work with. I follow their careers with interest and pride and look forward each year to our 'Guiry Group Christmas Drinks' in McDaid's 23rd December at 8pm.

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Pat Guiry, from Co. Tipperary, graduated with an Honours BSc. degree in Chemistry from University College Dublin (UCD) in 1986. He stayed at UCD for his PhD working under the supervision of Professor Dervilla Donnelly on the application of aryllead triacetates to the synthesis of natural products. During his PhD he also worked in Marseille in 1988 under the supervision of Dr Jean-Pierre Finet (Cu-catalysed N-arylation) and at Texas A&M in 1989 with Professor Sir



Derek Barton (mechanistic studies of arylation /phenol arylation). He received his PhD degree in 1990 and moved to the group of Dr John Brown FRS at the Dyson Perrins Laboratory, Oxford University for postdoctoral studies in the area of asymmetric catalysis. He returned to UCD as a College Lecturer in 1993 where he started his independent research. His research interests are the design and preparation of chiral ligands and their application in a broad range of asymmetric catalytic transformations. He was a visiting researcher in the group of Professor Andreas Pfaltz at the MPI at Mülheim an-der-Ruhr in 1996. He was the recipient of a President's Research Award in 1996 and a President's Teaching Award in 2000 from UCD. He was promoted to Senior Lecturer in 2002, to Associate Professor of Synthetic Organic Chemistry in 2003 and to Professor of Synthetic Organic Chemistry in 2006. He was the Merck Frosst Visiting Professor at the University of Toronto in early 2004. He was appointed as the Chief Executive of the Conway Institute of Biomolecular and Biomedical Sciences at UCD 2004-5, Director of the Synthesis and Chemical Biology since 2002 and was Head of the UCD School of Chemistry and Chemical Biology 2011-2014. He is currently a member of the UCD Governing Authority and of the Senate of the National University of Ireland. He was elected as a Member of the Royal Irish Academy in 2013. He has supervised 38 PhD and 2 MSc students to completion and worked with 14 postdoctoral fellows and ca. 80 final year project students. His current group comprises 13 PhD students, 4 postdoctoral fellows and 5 final year project students. A keen tennis player, he will represent Ireland in 2015 in the Fred Perry Cup (ITF World Team Competition) in La Baule, France.

INSTITUTE OF CHEMISTRY OF IRELAND.

CALENDAR OF EVENTS 2015

Date	Venue	Event
March 5 th	TCD (Supported by ICI) Bioscience Inst. Tercentenary Hall	3 rd Inaugural Cocker Lecture Prof. Karl Wieghardt Max Planck Institute.
April 27 th	NUI Maynooth	Forensic Science Lecture. S. O'Muircheartaigh
March 28 th	UCC	Presentation of L.C. Medal At ISTA Annual Conference
April 6 th		Easter Bank Holiday Monday
April 16 th	DCU	Boyle-Higgins Award Lecture Dermot Diamond
April 16 th	DCU	AGM
April 16 th	NUI Galway	Eva Philbin Lecture 2 (2014/15) At 4pm
April 17 th	UCC (ICI/RSC)	Synthesis of Bioactive Molecules Prof. Ian Paterson
April 30 th	UCD	Congress 2015: Modern Approaches to Asymmetric Synthesis
May 1 st	QUB	Eva Philbin Lecture 3 (2014/15)
May 4 th		May Bank Holiday
June 1 st		June Bank Holiday.
June 12 th –15 th	NUIG	International Symposium on Applied Bioinorganic Chemistry.
June 25 & 26 th	NUIM	Research Colloquium
Sept. 28/29	Vienna	EuCheMS General Assembly
November	Dublin/Sligo	Science Week Industrial Award (TBD)

The Institute is active so make sure you attend some of the career enhancing events we organise or sponsor and contribute to your Continuing Professional Development (CPD)

Events held so far this Year

Events kicked off with a very interesting lecture supported by ICI by Prof Karl Wieghart of the Max Plank Institute at the Bioscience Institute, Tercentenary Hall, TCD, and the 3rd Inaugural Cocker Lecture about Alfred Werner (1866-1919), a Swiss chemist and winner of the Nobel Prize for Chemistry in 1913 for his research into the structure of coordination compounds.

On April 27 Sean O’Muircheartaigh gave an informative talk with demonstrations about the use and misuse of forensic science in the case of the Birmingham Six in Britain. His work helped clear them and have then released. Sean will write a paper for ICN later this year.

We had our Boyle Higgins Annual Award Lecture by Professor Dermot Diamond at DCU on April 16. The topic was *“Chemical Sensing with Autonomous Devices in Remote Locations: Why is it so difficult and how do we deliver revolutionary improvements in performance?”* Delivered with his usual good humour and flair he covered many applications and problems encountered with remote sensors and when place in the human body.

This was followed by a wine reception and then by the AGM. A new Council was elected with Patrick Hobbs stepping down after his two year term as President and Margaret Franklin elected as President, the 3rd woman to be so elected.

April 16th was a busy day for the Institute with an event conflict as the 2nd Annual Award Lecture by Prof Thorri Gunnlaugsson from Trinity took place in NUI Galway and facilitated by Professor Niall Geraghty.

Another event took place the next day in UCC sponsored by ICI/RSC the Synthesis of Bioactive Molecules with 9 speakers including Prof Ian Paterson. The event was coordinated by Dr Gerard McGlacken.

Then came our Congress 2015 on April 30th in UCD facilitated by Prof Patrick Guiry and the Chairman Dr Eoghan McGarrigle of the organising committee ably and efficiently did a fantastic job at putting into place all the facilities, sponsors, reception and dinner to make the even a great success.

Again a busy next day as we had an Awards Symposium in cooperation with the RSC at Queens for Thorri Gunnlaugsson to deliver his 3rd Annual Award Lecture and Margaret Franklin to represent his Award plaque. This event was coordinated by Dr Mark Muldoon of Queens who organised lunch, a wine reception and a dinner in the Ulster Reform Club curtesy of Prof Duncan Thorburn Burns who has a longstanding relationship with the Institute.



67th Irish Universities Chemistry Research Colloquium

2015 will see the Department of Chemistry at Maynooth University hosting the 67th Irish Universities Chemistry Research Colloquium on **June 25th & 26th**.

As always the colloquium will highlight the outstanding research being conducted by the best and brightest of Ireland’s young chemists at third level institutions across Ireland.

The schedule will feature a mix of innovative oral and poster presentations from graduate students as well as exciting plenary lectures from leading researchers based at home and abroad.

- See more at: <https://www.maynoothuniversity.ie/chemistry/chemistry-colloquium-2015#sthash.rPsPL1on.dpuf>

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DUBLIN

Catalysis and Sensing for our Environment

The Catalysis and Sensing for our Environment Symposium 2015 will be held in Dublin on 9-10th July, jointly hosted by Trinity College Dublin, Maynooth University and the Royal College of Surgeons in Ireland. This year's symposium will be held in the vibrant city of Dublin, with a half-day pre-Symposium meeting on Supramolecular Chemistry being held on the afternoon of the 8th of July in nearby Maynooth University. This year's programme will bring together researchers from China, Ireland, the UK and other countries in high profile and dynamic research areas with plenary lectures delivered by Professor Eric Anslyn (UTA, USA) and Professor Yun-Bao Jiang (Xiamen, China).

Please email the organising committee if you wish to attend or present a poster.

Local Organising Committee:

Thorrfinnur Gunnlaugsson (gunnlaut@tcd.ie)

Donal O'Shea (donalfoshea@rcsi.ie)

Robert Elmes (robert.elmes@nuim.ie)

Aisling Hume (ahume@tcd.ie)



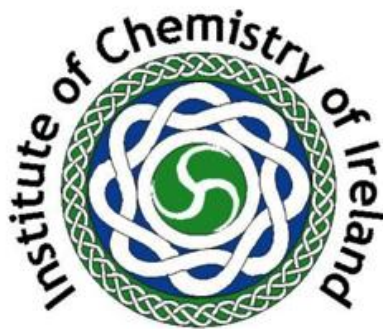
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Announcement of the Winner of the ICI Industrial Award 2015

As part of its ongoing campaign to enhance the chemistry profession in Ireland, along with the status and standing of Chemists working in Ireland, the Institute of Chemistry of Ireland last autumn announced a new award for Industrial Chemists.

The award is called “[Industrial Chemistry Award 2015](#)”. All chemists who are members of the Institute are eligible. The prize is €1000. The award is sponsored by Henkel Ireland Ltd.

We initiated this award when Dr Brian Murray was President, continued during my Patrick Hobbs Presidency and with help from Dr Ray Leonard a former President got sponsorship from Henkel Ireland Ltd curtesy Dr Patricia Cullen, Director Product Development.

This is our first step with this award and it was aimed at an individual chemist. Some feedback indicates a group might be more appropriate in the multinational pharmaceutical sector where significant projects tend to be completed by teams rather than individuals and that the format of the prize might be reviewed. We will be working with indigenous industry and multinationals on ways to improve this award.

I am delighted to announce we have a very deserving winner ratified by Council on April 16th and officially announced at Congress 2015 at UCD on April 30th. The winner is Donal Coveney of TopChem Pharmaceuticals Ltd an indigenous Irish company and based in Sligo.

Donal is the founder and managing director. He established TopChem a manufacturer to develop & manufacture API's for supply to the global pharmaceutical industry. He has led the company to FDA & EU GMP certification. The company has filed 4 Drug Master Files & secured business from global players Mylan, Sandoz and Perrigo. He currently employs 15 chemists.

To date more than 80 chemists, Grads and Post-Grads have been employed since inception in 2007 and many have gone on to find employment in the multinational sector. The Company also offers technical services to the Pharma sector including process development, technical support and troubleshooting.

Donal is a graduate of UCC, and obtained his PhD in Organic Chemistry under Prof Dervilla Donnelly in 1987. He has also been a President of the Institute from 2007-9. He has published a number of papers and is holder of 6 patents. We will be having a special event in the autumn in Sligo IT, probably during Science Week to present the prize. Longer term we would hope to make this a prestigious annual event with support from the Irish pharmaceutical and chemical industry to recognise the contribution chemists in Ireland make to Ireland and our economy.

Patrick Hobbs MSc. FICI. CChem. CSci. MRSC

Immediate Past President & Editor Irish Chemical News

Institute of Chemistry of Ireland

Irish Chemical News 2015 Issue No.1



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Athena SWAN Charter

Recognizing commitment to advancing women's careers in science, technology, engineering, maths and medicine (STEMM) employment in higher education and research.

Tackling gender inequality in STEMM

Gender inequality in STEMM subjects, including chemistry, is an issue facing the third level education sector. Dr Ruth Gilligan, Athena SWAN Adviser, looks at how some universities and institutes of technology are trying to change the demographics.

Figures published by the Higher Education Authority in December 2014 highlighted gender inequality as an issue for the whole higher education sector. Across Irish universities, only 19% of professors are women. In institutions of technology, women make up 45% of academic staff but just 29% of senior academic staff.

When we drill down into subject differences, the gender balance drops even further for many science, technology, engineering, mathematics and medicine (STEMM) subjects. This includes physical sciences as well as mathematics, ICT and engineering.

The Institute of Chemistry Ireland has also found that there are few women chemists among their membership. Writing in a previous issue of *Irish Chemical News*, ICI president Patrick Hobbs called for more women to join from third level institutions, and industry.

A sector-wide issue

Achieving gender equality is high on the agenda for the Irish higher education sector, and for government. Third-level institutions, and individual chemistry departments have a role to play in meeting these objectives.

Tom Boland, Chief Executive at the Higher Education Authority, has affirmed that the body is 'absolutely committed to the promotion of gender equality among students and staff across the Irish higher education system'.

Minister for Education and Skills, Jan O'Sullivan has also stressed the importance of gender equality in STEMM for both the scientific community and Irish society as a whole.

'It is important that our higher level education sector fairly represents the diversity and innovation that are at the heart of Irish society. In the area of STEMM women play a key role in teaching, cutting-edge research and building links with industry and the wider community.'

Actively promoting gender equality is an important goal. Gender equality should be central to how all public organisations operate. The principal of equality demands nothing less. Ensuring the fair representation and career progression of female academics is also important in retaining Ireland's international reputation for the quality and impact of our scientific community.'

Making a change

On 5 February 2015 seven universities, 14 institutes of technology and the Royal College of Surgeons officially signed up to a charter committing them to advance women's careers in STEMM employment in academia.

These institutions are taking part in a major national initiative that will see a successful UK scheme, ECU's Athena SWAN Charter, introduced to Ireland as a framework for tackling gender inequality for staff.

Participants will be able to submit for an Athena SWAN award in April 2015, which involves a robust process of self-assessment and peer review.

Speaking about the launch of this three-year pilot, Tom Boland said that HEA is 'eager to see real and substantial progress in addressing gender imbalance in the immediate years ahead'.

Introducing Athena SWAN

The Athena SWAN Charter, run by UK higher education equality body Equality Challenge Unit (ECU), is a programme that has had a proven impact on gender equality in higher education and research.

Initially developed out of two academic women's forums (the Athena Forum and the Scientific Women's Academic Network), ECU's Athena SWAN Charter launched in 2005 with ten members. There are now over 125 members including UK higher education institutions, medical schools, and research institutes. A number of UK research funders have linked achievement of an Athena SWAN award to funding, resulting in widespread action. The Australian higher education sector is currently developing a pilot scheme to introduce the charter there.

The awards

The awards are granted at institutional or departmental level, and are available in three progressive levels:

- = **Bronze award:** recognises a solid foundation for eliminating discrimination and developing an inclusive culture that values all staff
- = **Silver award:** recognises a significant record of activity and achievement in promoting equality and in addressing challenges across the whole institution
- = **Gold award:** recognises sustained progression and achievement in addressing challenges and acting as a champion for gender equality

To progress to the next level award, or to renew at their current level, organisations will need to show evidence of progress against the action plan, and the impact this has had on women's careers and the organisational culture.

Institutions and departments taking part in the first round of awards in Ireland will be submitting detailed and rigorous self-assessments and action plans in April. Far from a box-ticking exercise, they will need to critically analyse and reflect on the realities of their organisation and departments, identifying barriers to women's careers, and the reasons behind underrepresentation and attrition rates at higher levels. The submissions will go through a stringent peer-review process.

Alongside institution-wide submissions, we are expecting a number of applications in the pilot from individual departments, including chemistry. In the UK, 26 chemistry departments hold ECU Athena SWAN awards. Currently there are only 7 gold department awards across the UK – three of these top awards are held by chemistry departments.

Impact

Independent evaluation of the Athena SWAN Charter found considerable evidence of the positive impact of participating both on improving gender diversity and equity within institutions and on the career development and satisfaction of women - and men - working in STEMM.

Membership has a proven impact as a catalyst for change, leading to cultural and organisational transformation that makes a real difference for women and enables all staff to achieve their maximum potential. The framework allows you to identify areas for positive action as well as recognise and share good practice.

Responding to the evaluation, ECU CEO David Ruebain stated that "Athena SWAN Charter has had a lasting impact on gender equality and women's careers in STEMM, and we are delighted that the Higher Education Authority and the Irish HE sector have invited us to run the programme here for the first time. Through this commitment to addressing cultures and attitudes, the participants are setting off on a path to real change."

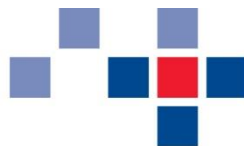
The Athena SWAN Charter itself is currently evolving, and will expand in the UK to cover gender equality in arts, humanities, social science, business and law departments later this year. Once the first year of the pilot has been reviewed, we will assess if we can also expand the scope in Ireland.

Find out more: ECU's Athena SWAN: www.ecu.ac.uk/equality-charter-marks/athena-swan

Independent evaluation of Athena SWAN: [*Advancing women's careers in STEMM: evaluating the effectiveness and impact of the Athena SWAN Charter*](#)

Contact Dr Ruth Gilligan if you are interested in taking part in the Athena SWAN Charter:

E: Ruth.gilligan@ecu.ac.uk T: +44 20 7269 6541



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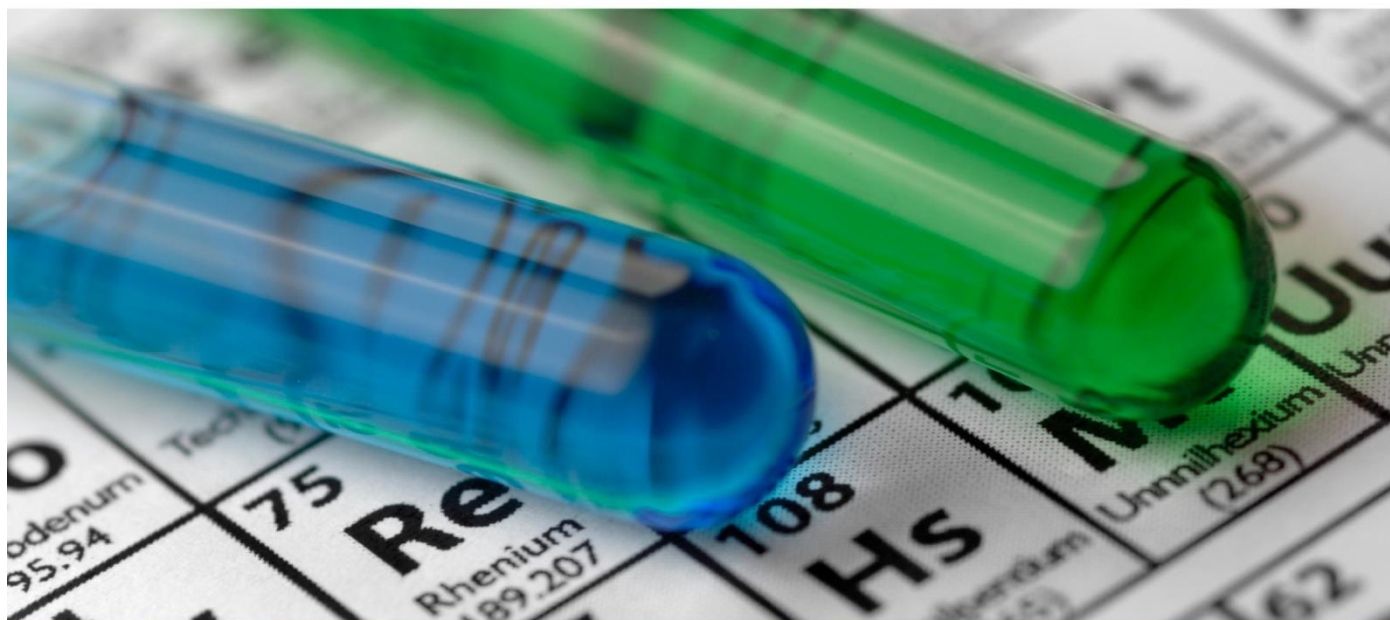
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The Synthesis and Solid State Pharmaceutical Centre (SSPC)

The Synthesis and Solid State Pharmaceutical Centre (SSPC), a Global Hub of Pharmaceutical Process Innovation and Advanced Manufacturing, funded by Science Foundation Ireland (SFI) and industry (€42 million investment), is a unique collaboration between 22 industry partners, 9 research performing organisations, and 12 international academic collaborators.



Figure 1 SSPC Partners

The SSPC, hosted at the University of Limerick, within the 15,100m² Bernal (a €52 million strategic initiative)/Materials and Surface Science Institute (MSSI) complex, transcends company and academic boundaries, and is the largest research collaboration in Ireland, and one of the largest globally within the pharmaceutical area.



Figure 2 SSPC headquarters at the 15,100m² Bernal/MSSI Complex, University of Limerick

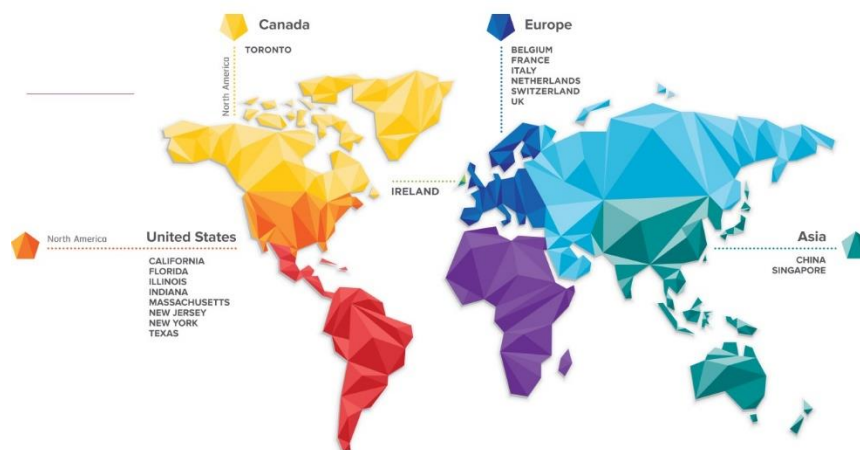


Figure 3 SSPC's Global Reach

The SSPC, formerly known as the Solid State Pharmaceutical Cluster (2007-2013), leads the way for next generation drug manufacture and carries out research ranging from molecule, to material, to medicine, with the objective of gaining a better understanding of mechanisms, controlling processes, and predicting outcomes for the efficient and environmentally sustainable production of safe medicines. The SSPC is designed to link scientists and engineers in partnerships across academia and industry to address crucial research questions. The SSPC aims to deliver industry relevant solutions, which result in job growth and retention within the pharmaceutical industry in Ireland.

The initial phase, the Solid State Pharmaceutical Cluster (2007-2013), was funded by Science Foundation Ireland's Strategic Research Cluster programme. The Cluster focussed exclusively upon the crystallisation stage of the active pharmaceutical ingredient (API) in the manufacturing process. Within the pharmaceutical industry, this stage is a particularly challenging aspect of manufacturing, as there is a significant lack of fundamental understanding of the science and engineering challenges at this stage of the process. Significant breakthroughs made by the Cluster led to the establishment of the Synthesis and Solid State Pharmaceutical Centre (SSPC) in 2013. Due primarily to the unprecedented success of the Cluster, the SSPC's research remit logically expanded from crystallisation, upstream into wet chemistry, including synthetic organic chemistry and biocatalysis, and downstream into advanced formulation of the API into a drug product.

Next Generation Drug Manufacture

The SSPC's research programme is organised into three interconnecting strands, which actively reflect the three distinct steps in the manufacture of modern medicines.



Figure 4 SSPC research remit - Molecule, Material, Medicine

Strand 1 - New Frontiers in Pharmaceutical Synthesis (Molecule)

Strand 1 - New Frontiers in Pharmaceutical Synthesis focuses on enantioselective and efficient synthetic routes and processes, i.e. developing better and more environmentally sustainable ways to make APIs. Major themes include:

- New catalysts and systems for cleaner production with excellent selectivity in asymmetric synthesis, elimination of hazardous reagents and operation in benign conditions
- Innovative process technologies operating in flow conditions and where appropriate achieving multiple transformation steps in a single reactor
- New chemical methods for the efficient removal of impurities and side products

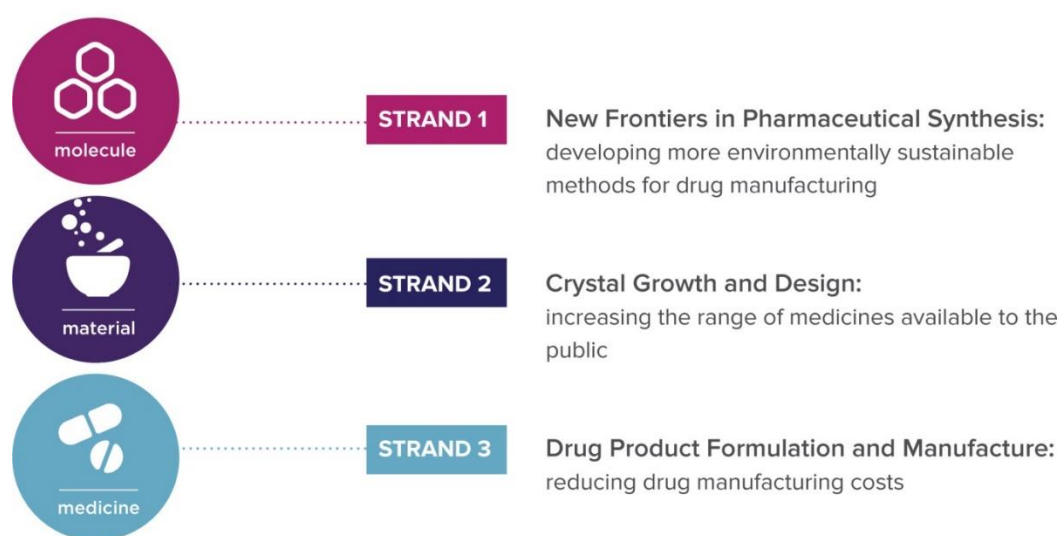


Figure 5 SSPC's three strands of research

Strand 2 - Crystal Growth and Design (Material)

Strand 2 - Crystal Growth and Design focuses on science and process engineering underpinning the crystallisation of complex organic molecules with conformational flexibility and a multitude of functional groups, i.e. investigating optimal ways to produce APIs in order to increase the range of medicines available to the public. Impurities and solvent selection are central to this strand. Major themes include:

- Study of the underlying molecular interactions in supersaturated solutions and at interfaces
- Understanding the mechanisms that control product crystal properties such as crystal structure, purity, shape and size
- Exploiting these mechanisms to tailor and control crystal properties, to scale up and scale down processes, to develop model based control for improved product quality in traditional batch crystallisations as well as in emerging technologies like continuous processing, cocrystallisation, nanocrystallisation and crystallisation into excipient matrices

Strand 3 - Drug Product Formulation and Manufacture (Medicine)

Strand 3 - Drug Product Formulation and Manufacture focuses on reducing drug manufacturing costs, by bringing stronger scientific and process engineering principles and knowledge into the domain of Solid State Pharmaceutics, which up to now has essentially been empiric. This improved approach is demanded by the Quality by Design (QbD) knowledge based approach to develop new products and formulations. Major themes include:

- Understanding the nature and strength of interactions between APIs and excipients
- Identification of currently unknown critical attributes in APIs and excipients that lead to failures during formulation
- Development of new materials and technologies for the generation and stabilisation of the amorphous state, which is one approach to realising the potential of BSC Class II poorly soluble drugs

In total, the SSPC supports 20 state of the art research projects across these three strands of research, which are divided into platform and targeted projects.

Platform Research

The SSPC supports 9 platform projects, which are targeted towards the progression of scientific state of the art, driven by scientific challenges of the area and aimed at achieving scientific excellence.

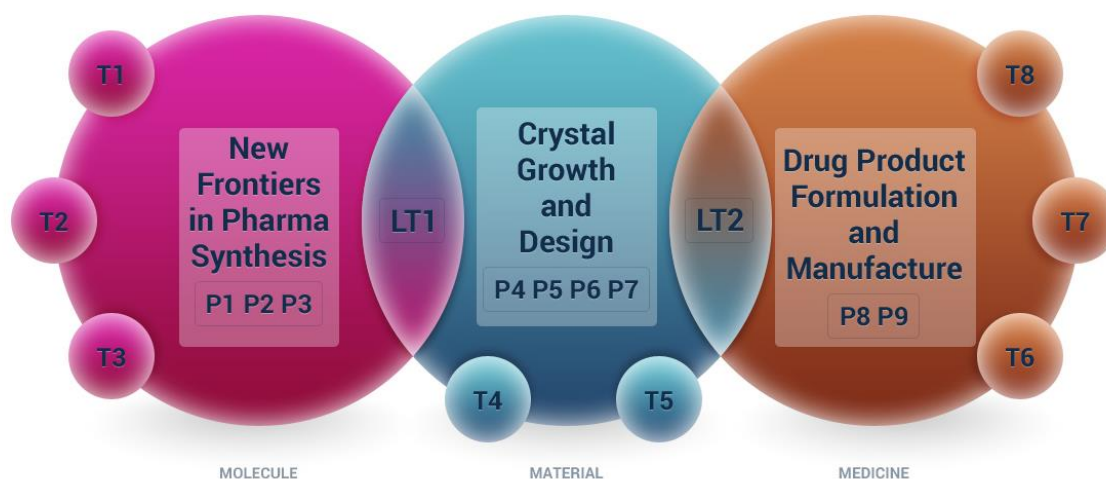


Figure 6 SSPC Platform and Targeted Projects

Targeted Projects

The SSPC supports 9 targeted projects, which are driven by scientific challenges of specific industrial needs. Under targeted projects, the SSPC also supports 2 linker projects, which are the interactions between the three strands. Linker projects are vital in order to make progress in this area, as they concentrate effort at interfaces where the

most important developments need to take place. The programme has been designed to pay particular attention to how attributes from each strand can impact upon each of the other strands.

One of SSPC's latest developments in targeted research extends into the BioPharma area, via a collaboration with seven industry partners: Allergan Pharmaceuticals Ireland, BioMarin International Limited, Eli Lilly and Company, Genzyme Ireland Ltd - A Sanofi Company, Janssen Biologics, MSD and Pfizer Ireland Pharmaceuticals and three research performing organisations; the National Institute for Bioprocessing Research and Training (NIBRT), Trinity College Dublin (TCD) and Dublin City University (DCU). This latest collaboration received over €1m in funding from the Department of Jobs, Innovation and Enterprise (DJEI) through Science Foundation Ireland's (SFI) Spokes programme, coupled with €450,000 in cash contributions from industry partners. The manufacturing process of biotherapeutic drugs is complex and costly, with problems relating to formulation and protein instability often affecting the biological performance of these therapeutics. As a result of this Advanced Biopharmaceutical Technologies collaboration, Ireland's leading scientists and engineers are working with industry partners to develop innovative single use bioprocessing solutions and to examine the effects of extractable and leachable agents on product quality. This research will generate significant intellectual property for Irish universities and gains commercial advantage for Irish-based biopharma companies.

To find out more please go to www.sspc.ie

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SSPC National Crystal Growing Competition Winners

Announced! Submitted by [admin](#) on Thu, 03/19/2015 - 15:44



Studying crystals after the event with SSPC Education and Outreach Officer Dr Sarah Hayes was Youbeel Hagi, Galway Community College, Galway who was awarded joint first place in the competition. Photo: Alan Place

“Crystals are all around us and in every aspect of our lives, from chocolate to medicine to paints and plastics”
Prof Kieran Hodnett, SSPC Scientific Director.

The Synthesis and Solid State Pharmaceutical Centre (SSPC) today (19th March 2015) announced the winners of its first SSPC National Crystal Growing Competition, which was launched as part of Science Week in November 2014, to celebrate the International Year of Crystallography. The SSPC National Crystal Growing Competition was open to primary and post-primary schools from across Ireland.

At today’s award ceremony, Professor Michael Zaworotko, one of the world’s top 20 chemists, announced the winners of the SSPC National Crystal Growing Competition as Youbeel Hagi, Galway Community College, Galway and Jason Folan, Colaiste Bhaile Chlair, Galway. Due to the exceptional quality of the entries, Professor Zaworotko also announced two runners up as Oisín Tobin, Colaiste Bhaile Chlair, Galway and Clare McKernan, St Aloysius’ Secondary School, Cork. The competition judging criteria centred on the quality of the single crystals that were grown by students, considering elements such as the definition of the crystal faces, along with the clarity and size of the crystal, which are important factors that need to be taken into consideration when controlling crystal growth within the pharmaceutical industry when making medicines.

Judging panel members for the SSPC National Crystal Growing Competition paid tribute to the high standard and excellent quality of entries. Professor Kieran Hodnett, SSPC Scientific Director said:

“The standard and quality of the crystals submitted for the SSPC National Crystal Growing Competition was outstanding. It is very encouraging to see such a high level of enthusiasm by the students and their teachers in growing the crystals over the last number of months”.

Professor Mark Ferguson, Director General of Science Foundation Ireland and Chief Scientific Adviser to the Government of Ireland said:

“As one of Science Foundation Ireland’s twelve research centres, SSPC has a critical role supporting Ireland’s pharmaceutical sector. Ireland’s first SSPC National Crystal Growing Competition is an important element of SFI’s public engagement remit and SSPC’s education and outreach programme, which aims to inspire young minds as to the possibilities and application of science in our everyday lives. Congratulations to the deserving winners.”

Jon O'Halloran, SSPC General Manager, said:

“The SSPC National Crystal Growing Competition is an active part of the SSPC’s education and outreach programme, which is dedicated to developing the next generation of scientists and crystallographers in Ireland. The SSPC look forward to engaging with Ireland’s schools on future initiatives”.

The crystals submitted to the SSPC National Crystal Growing Competition will be kept on public display at SSPC headquarters at the University of Limerick.



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To mark World Accreditation Day on 9th June 2015, Eurachem Ireland will host a TrainMiC workshop on Metrology in Chemistry, to include the estimation of Uncertainty of Measurement, at the State Laboratory.

Eurachem Ireland is an organisation for people working in chemistry in Ireland, with a focus on analytical chemistry. Chemistry students are welcome too. Eurachem Ireland promotes the objectives of Eurachem (www.eurachem.org) in Ireland including good quality practices. Other objectives of Eurachem Ireland include, but are not limited to:

- Facilitate networking among Irish analytical chemistry laboratories from the public sector, private sector and education sector;
- Provide a forum for the discussion of common issues;
- Encourage Irish participation in Eurachem working groups;
- Increase awareness of opportunities for organisations to participate in research;
- Contribute to the development of chemistry students to meet the needs of Irish employers.

To be informed of the activities of Eurachem Ireland, including the TrainMiC workshop on 9th June 2015, you are invited to:

- 1) Join the mailing list - To request to join, email eurachem@statelab.ie. Information will also be available on: www.statelab.ie/eurachem.html.
- 2) Join the LinkedIn group 'Eurachem Ireland'.

What is Eurachem?



Eurachem is a network of organisations in Europe, having the objective of establishing a system for the international traceability of chemical measurements and the promotion of good quality practices. It provides a forum for the discussion of common problems and for developing an informed and considered approach to both technical and policy issues.

Members



Membership of Eurachem is open to countries within the European Union and the European Free Trade Association, the European Commission and European countries recognised by the EU and EFTA as accession states. Other European countries and organisations with an interest in quality of analytical measurements may participate in Eurachem as Associate or Observer members. Eurachem currently has [32 member countries](#)

What does Eurachem do?

Eurachem promotes best practice in analytical measurement by producing authoritative guidance within its [expert working groups](#), publishing [guides on the web](#) and supporting [workshops](#) to communicate good practice. Guidance covers technical issues such as measurement uncertainty evaluation, method validation and proficiency testing.

Eurachem also works through [liaisons](#) with accreditation bodies and organisations with interests in measurement quality to help ensure practical accreditation policies and promote sensible technical provisions in regulation.

Eurachem - History and Impact - The first ten years

Eurachem was founded in 1989, and on the occasion of the tenth General Assembly, Alex Williams, the convenor of the initial meetings set up to consider establishing a forum for traceability in analytical chemistry in Europe, presented a history of the formation and the first ten years of Eurachem. This presentation has since been documented as a personal view of Eurachem, and provides an insight into the early development of the organisation and the reasons for its establishment. It is part of Eurachem's history.

Eurachem's 25th Anniversary - a perspective

On the occasion of the 25th anniversary, Eurachem was invited to submit review articles to the journal Accreditation and Quality Assurance (AQUAL) covering Eurachem's formation and impact in its first 25 years.

The first of these articles, is "Eurachem's 25th Anniversary: Two members' perspective" and covers some key developments in the development of an infrastructure for metrology in chemistry.

Impact of Eurachem: 25 years of activity

A second paper, "Impact of Eurachem 25 years of activity", presents collated evidence of the impact of Eurachem's activity up to the 25th anniversary in 2014.

These papers are available on the Eurachem's web site www.eurachem.org

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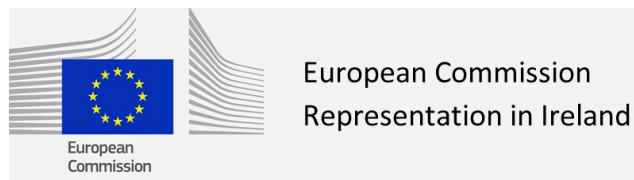
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European Commission Representation in Ireland. Lunchtime briefing: Career Opportunities in the EU Institutions held on 10 March 2015.

Barbara Nolan, Head of the European Commission Representation in Ireland, invited a representative of the Institute to a lunchtime briefing on career opportunities in the EU institutions. Speakers were the Minister of State for European Affairs, Dara Murphy TD and Aoife Lyons of the Public Appointments Service. The briefing was designed to provide information on the opportunities available to Irish graduates in the EU institutions and the selection and recruitment process. The audience consisted of representatives of professional organisations and university alumni associations.

Background:

In March 2015, the European Personnel Selection Office published details of two open competitions to recruit graduates to work in the EU institutions, as Administrators or 'AD' officials. Applicants must have a good command of 2 official EU languages.

The first competition is open to graduates of any discipline. Positions will be primarily Brussels-based. Details were published on eu-careers.eu on 19th March.

The second competition is the field of Audit and is open to graduates of Accountancy, Finance, Economics, Public Administration, Project Management, IT, Audit or Law. Positions will be based in the European Court of Auditors in Luxembourg. Details were published on eu-careers.eu on 26th March.

Successful candidates will be recruited at the graduate entry grade of AD5.

A third competition to recruit lawyers with a qualification in Irish law to work in The Court of Justice of the European Union in Luxembourg has been launched. Successful candidates will be recruited both at the graduate entry grade of AD5 and at the more senior position of AD7 for which at least 6 years' relevant professional experience is required. Candidates will need to have a thorough knowledge of English or Irish (C1) and a good knowledge of French (B2). Details: http://europa.eu/epso/apply/jobs/perm/2015/national-law/index_en.htm .

As well as providing support and assistance to new staff for relocation, the EU institutions also offer a comprehensive remuneration package, including pension and health insurance.

While this round of job opportunities is not aimed at chemists specifically but they could apply under the first competition. From time to time jobs for chemists do arise so if interested you can log on to the EU web site for alerts. Be aware that this is a competitive process and there are strict requirements. Periodically the European Commission Representation in Ireland organise training events for prospective candidates. Below is an example of such a training event held in Dublin, in early April in Dublin.

Following that are the slides presented, by Aoife Lyons of the Public Appointments Service, during the meeting. It outlines in some detail the whole process involved in the recruitment process and the requirements that prospective candidate must meet.



EU Jobs Training: How to Succeed in the EU Recruitment Process

What: Expert guidance on how to succeed in the EU Recruitment Process – focusing in particular on the online application form and the Computer Based Tests

When: 17.30 – 19.30 Wednesday 8 April

Where: Government Buildings, Merrion Street, Dublin

Register: Email eujobs@taoiseach.ie by Friday 3 April with subject line '8 April', including your name & the organisation to which you're affiliated (employer or third level institution)

Effective preparation is the key to success in the EU recruitment process. [EU Jobs Ireland](#), an initiative coordinated by the Department of the Taoiseach, is holding a training session on Wednesday 8 April to provide Irish citizens and residents with expert advice on the EU's competition processes, designed to maximise prospects of success.

The session will focus on three competitions launched by the [European Personnel Selection Office](#) (EPSO) this March (see below) offering advice on how to complete the online application form and how to prepare effectively for the computer based aptitude tests which follow. Presentations will also look at other avenues to an EU career, drawing from the experience of Irish EU officials.

To register, please email eujobs@taoiseach.ie **no later than Friday 3 April**, confirming your name, the organisation to which you're affiliated (employer or third level institution, as relevant) and, if relevant, any EU competition(s) you have previously applied for. If there are any specific issues you would like to see covered in the session, you are encouraged to raise these also by email – it will help ensure the session is as useful as possible to all concerned. A full programme and related information materials will be circulated to registered candidates closer to the date. Capacity is strictly limited and places will be allocated on a first come, first served basis so early registration is advised.

Current & Upcoming EU Graduate Recruitment

This March, the [European Personnel Selection Office](#) (EPSO) launches three new competitions seeking to recruit graduates for executive administrator positions in the EU institutions.

Up to 150 graduates are sought from any academic background to serve as [generalist administrators](#) (AD5) within the institutions, with a closing date for applications of 21 April. 80 graduates with a background in law, accountancy, public administration, finance and economics or project management are sought to serve as [auditors](#) (AD5) in the European Court of Auditors, with a closing date for applications of 28 April. Finally, a smaller number of candidates with a qualification in law and detailed knowledge of the Irish legal system are sought to serve as [legal researchers](#) (AD5 and AD7) in the European Court of Justice, with a closing date for applications of 31 March. Full details of all of these competitions are available on the [EPSO website](#).

Upcoming Competitions and How to apply



Aoife Lyons, Public Appointments Service



Why an EU Career?

Interesting and challenging work	International working environment	
Training languages skills		Variety of career options and roles
	Travel	Make a difference for Europe



Who are we looking for?



	Graduates + Non-graduates	Speaking EN, FR or DE
Highly skilled Resilient	Communicative Motivated	Result driven Analytical Potential to lead
And speaking... at least another official EU language?	Enjoying working in multicultural teams	Ready to move to Brussels or Luxembourg



Profiles

Law	Public Administration (EPA)	Interpretation
Translation	Economics	Communication
Audit	Language editing	Lawyer-linguist
Assistants	Administrator	Many other Profiles..



Common features of Competitions

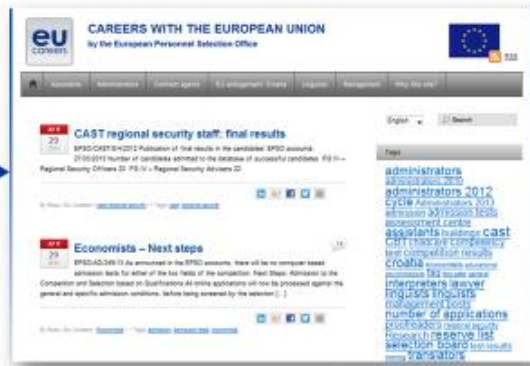
- Need to Register with EPSO to create your own account;
- Notice of Competition or NOC sets out all the stages in the process;
- EPSO Competencies used for all competitions with an assessment matrix provided;
- Useful “competition blogs” where questions are asked and answered;
- Post Assessment, the individual institutions will carry out the actual “recruitment”.



Your EPSO Account



Stay up to date



Upcoming Competitions

- | | Opening Date | Closing Date |
|---|--------------|--------------|
| • Administrators - Generalists (AD 5) | 19/03/15 | 21/04/15 |
| • Auditors (AD 5) | 26/03/15 | 28/04/15 |
| • Administrators specialising in Legal Research – including Ireland | 26/02/15 | 31/03/15 |



Administrator - Generalist (AD 5)

- Involved in Policy Formulation, Operational Delivery and Resource Management
- Duties include:
 - Devising, implementing, monitoring and control of programmes and action plans;
 - Managing resources including staff, finances and equipment;
 - Drafting Policy analysis briefings
 - Relations with External stakeholders and the Member States;
 - Drafting contracts, calls for tender etc.

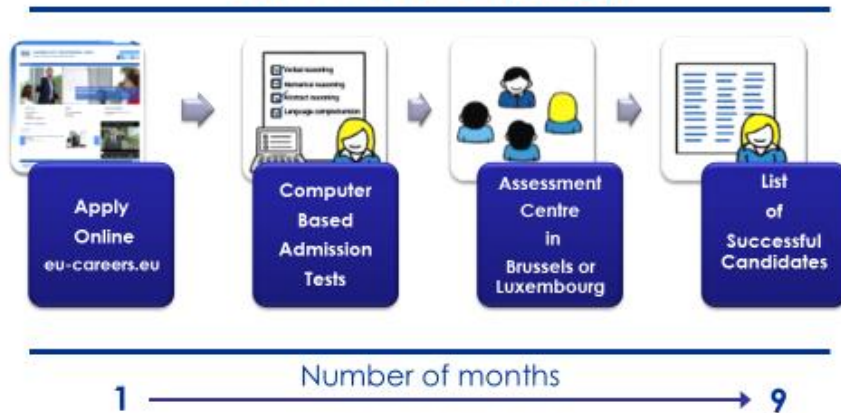


Administrators (AD 5) Requirements

- Have completed a university degree of at least 3 years;
- Be an EU Citizen;
- Thorough knowledge of an Official EU Language;
- Satisfactory knowledge of a second language (English, French or German);
- Salary – €4,384.38 per month for a 40 hour week.
- 137 candidates sought.



Selection Procedure Administrator (AD 5)



Computer Based Tests (CBT)



Assessment Centre

Administrator

1,5 day
in your 2nd language



Case
Study

Oral
presen-
tation

Structured
interview

Group
exercise

Auditors (AD 5) Requirements

- Have completed a university degree of at least 3 years relevant to duties or Professional Training/ Qualification of an equivalent level;
- Citizenship/Language Requirements/ Salary the same as AD;
- 40 candidates sought
- Slight differences at Assessment Centre:
 - Case study "in the field";
 - European Court of Auditors is based in Luxembourg so the Assessment Centres take place there.

Auditors (AD 5) Role

- Duties include:
 - External Audit, Financial Audit, Compliance Audit and sound financial management
 - Documentary and on-the-spot checks and control, analysis, assessment and improvement of audit systems and project and programme management
 - Internal Audit, methodological support, advice and training
 - Inter-service coordination and consultation on audit issues
 - Drafting of opinions, advice or recommendations.

Administrators Specialising in Legal Research (AD 5 and AD7)

AD 5

- Analysis of case law of the three courts
- Preliminary analysis of new cases
- Research work, in particular as regards their national law
- Monitoring national and Union Law

AD 7

- Co-ordination and revision, involving analysis of judgements of the courts of the EU, preliminary analysis of new cases, research and monitoring in the fields of national, EU, comparative and international Law
- Steering Projects relating to law and monitoring major IT projects that require high level legal expertise
- Defining and/or refining legal research tools and tools for disseminating laws analysed by the Directorate

Administrators Specialising in Legal Research (AD 5 and AD7)

AD 5

- Completed an Irish law degree of at least 3 years;
- Salary the same as AD 5; 2 people sought

AD 7

- Completed an Irish law degree of at least 4 years;
- At least 6 years relevant professional experience;
- Salary €5,612.65 per month: 2 people sought

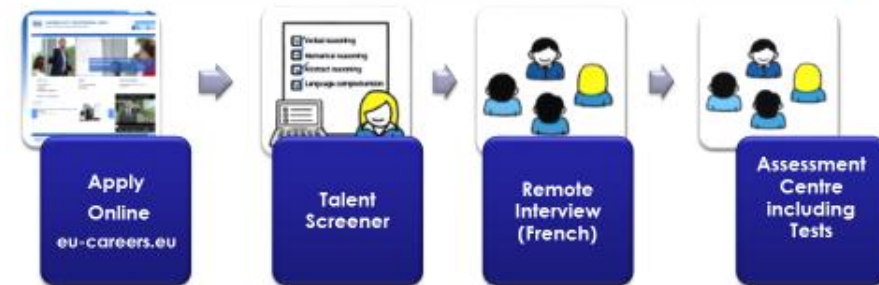
Both

- Citizenship & a thorough knowledge of the language of the competition: Irish/English and a good knowledge of French

Talent Screener

- Answer questions concerning your qualifications and experience.
- Information provided will be used to rate your application and identify the best to be invited to the AC.
- Important that you are completely honest in your answers as they will be validated
- You must reply yes or no to each question by clicking on the appropriate button.
- If you reply yes, you must provide text underneath. If you reply no to a question you will not be able to enter anything in the text box.
- Typically, you cannot exceed four thousand characters when responding to each question

Selection Procedure AD Specialising in Legal research



Last Thoughts

- EPSO Process designed to discourage unsuitable candidates or those lacking in motivation;
- If you apply you have to give 100%
- The balance of opportunity is much better in the specialist streams
- Monitor the EU Careers website but also register on publicjobs.ie





Dear colleagues,

You are kindly invited to the workshop **(Re)searching for Jobs**, which will take place in Brussels on the 2nd of June 2015 at EuCheMS Brussels Offices.

The main purpose of this workshop is to present the first European Employability Survey for Chemists and Chemical Engineers and to discuss the many barriers that young researchers face when entering the labour market. Part of the survey results have already been published at [Analytical and Bioanalytical Chemistry](#), while the article written by R. Salzer, P. Taylor, N. Majcen, F. De Angelis, S. Wilmet, E. Varella, I. Kozaris, featuring the final results, has been accepted for publication by Chemistry-A European Journal.

(Re)searching for Jobs

2nd June, 2015

EuCheMS Brussels offices - Rue du Trône 32, **7th floor**, Brussels

Chaired by Catherine Stihler, MEP

Registration is available at <http://bit.ly/1GfOCax>

Leaflet with abstracts is available [Here](#).

Poster is available [here](#).

9:30 Coffee and Registration

10:00 **Setting the Scene** - Catherine Stihler, MEP

10:15 **Helping Students into the Job Market** - David Cole-Hamilton, EuCheMS President, University of St Andrews

10:30 The Professional Status of European Chemists and Chemical Engineers - Reiner Salzer, Dresden University of Technology

11:00 Employability for Graduates and the Role of the EU - Margaret Waters, European Commission, DG Education and Culture

11:15 A View of a Young Scientist - Cristina Todasca, University Politehnica of Bucharest

11:30 Industrial Employability: Soft Skills Enhancing Scientific Know-How - Viviana Fluxa, CSL Behring

11:45 Employment Trends among Chemists in the US - Elizabeth C. McGaha, American Chemical Society (video presentation)

12:00 Panel Discussion and Conclusions

12:20 Closing Remarks - Catherine Stihler, MEP

12:30 Light Buffet Lunch

Please feel free to distribute this invitation as you find appropriate.

Best regards,

Nineta H. Majcen, PhD

General Secretary

European Association for Chemical and Molecular Sciences (EuCheMS)

62 Rue du Trône, 6th floor

1050, Brussels

Tel: [+32 \(0\) 2 289 26 90](tel:+3222892690) | Mail: nineta.majcen@euchems.eu



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Building Collaborative North South Partnerships in MSCA

The Institute of Chemistry attended 'Building Collaborative North South Partnerships in MSCA' a networking and information event for research centres and multidisciplinary groups across the island which was organized by the Irish Marie Skłodowska-Curie Office and InterTradeIreland and held at the Printworks, Dublin Castle on the 24th March 2015.

This article contains a brief synopsis of the Marie Skłodowska-Curie Actions and InterTradeIreland supports for all island H2020 applications.

The **Marie Skłodowska-Curie Actions (MSCA)** support researchers at all stages of their careers, across all research disciplines & in all employment sectors. The Actions reinforce cooperation between academia & industry in particular through cross-border & cross-sector mobility of researchers. They focus on excellent & innovative research training, career development & knowledge exchange.

The **Irish Marie Skłodowska-Curie Office** is operated by the Irish Universities Association (IUA), with the support of the Irish Research Council (IRC) and Science Foundation Ireland (SFI). The office provides advice and support on preparing applications for Marie Skłodowska-Curie funding and the management of Marie Skłodowska-Curie awards.



Dr. Jennifer Brennan and Dr. Suzanne Miller-Delaney, Irish Marie Skłodowska-Curie Office.

MSCA Annual Calls:

Strict mobility rules and the following definitions apply to all MSCA funding calls:

- MS/AC: the 28 EU Member States and Associated Countries
- Academic: consists of public or private higher education establishments awarding academic degrees, public or private non-profit research organisations whose primary mission is to pursue research, and international European interest organisations
- Non-Academic: includes any socio-economic actor not included in the academic sector and fulfilling the requirements of the Horizon 2020 Rules for Participation. Examples (not-exhaustive) include: industry (SMEs etc.), charities, NGOs, government/public bodies, national archives, libraries, etc.

Full details of all the MSCA calls can be found at:

<http://www.iua.ie/irish-marie-curie-office/funding-calls/>

For Individual Researchers - Individual Fellowships (IF)

Individual researchers with a PhD or >4 years research experience can apply for a **European fellowship** to move to another MS/AC for 1-2 years. **Career Restart** and **Reintegration** panels also exist for those not active in research in the last 12 months or for those reintegrating into Europe following time spent elsewhere. Alternatively researchers can apply for a **Global Fellowship** to be based outside of Europe for 1-2 years, returning to a MS/AC for 12 months. In all cases, applications are made with the support of a proposed supervisor.

Next IF call deadline: 10 September 2015

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For Organisations – Co-funding of regional, national and international funding programmes (COFUND):

The COFUND call partially funds organisations to run new or existing fellowship or doctoral programmes. Any organisation in a MS/AC that can manage a fellowship programme can apply. The financial support offered by the action is approximately 40% of the total costs of the fellowships programme, up to a maximum of €10 million per programme. Multiple calls for fellowships must be held during the lifetime of the project and all programmes must have an element of transnational mobility (either incoming, outgoing or reintegration).

Next COFUND call deadline: 10 October 2015

For Principal Investigators and Organisations – Research and Innovation Staff Exchange (RISE)

The RISE call funds the exchange of research staff and students in order to implement a joint research project between academic & non-academic organisations throughout Europe & the world. Consortia must consist of at least 3 independent participants in 3 different countries (2 of these must be MS/AC). If all participants are from MS/AC, at least 1 must be academic and 1 non-academic. MS/AC secondments must be inter-sectoral and cross-border. Staff and student salaries are not funded.

Next RISE call deadline: 28 April 2015

For Principal Investigators and Organisations - Innovative Training Networks (ITN)

European Industrial Doctorate (EID): Funds partnerships between at least one academic and one non-academic organisation in another MS/AC to deliver PhD programmes where student(s) spend at least 18 months working within academia and 18 months working in the non-academic sector.

European Training Networks (ETN): **Funds** pan-European inter-sectoral consortia implementing a research training programme for Masters &/or PhD students. Consortia must consist of a minimum of 3 beneficiaries in 3 MS/AC.

European Joint Doctorate (EJD): Funds the creation of joint doctoral programmes delivering joint, double or multiple doctoral degrees. Consortia must consist of a minimum of 3 doctoral degree awarding beneficiaries in 3 MS/AC.

Next ITN (EID, ETN & EJD) call deadline: January 2016

More information on the 'Building Collaborative North South Partnerships in MSCA' event including the full programme and presentations, please visit:

<http://www.iaa.ie/building-collaborative-north-south-partnerships-in-msca/>

Building Collaborative North South Partnerships in MSCA

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The Institute of Chemistry of Ireland attended The Irish Marie Skłodowska-Curie Office and InterTradeIreland networking and information event for research centres and multidisciplinary groups across the island.



Horizon 2020 Support The Key To Innovation Success

Collaborate to Innovate

Horizon 2020 Support Activities



Horizon 2020

InterTradeIreland is helping companies and researchers from Ireland and Northern Ireland to collaborate in [Horizon 2020](#), the European Commission's 7 year, €80billion, Research and Innovation programme designed to boost jobs and growth across Europe.

With a specific objective to increase the number of North South applications to Horizon 2020, InterTradeIreland has developed a support programme to help facilitate building this type of partnership. The **InterTradeIreland** Horizon 2020 support programme is open to companies, researchers, academia and other organisations that are working on a North South basis and the type of support available includes, information and advice, help with finding partners, travel support and all-island events. Full details are available online www.intertradeireland.com/horizon2020 or by emailing horizon2020@intertradeireland.com

InterTradeIreland Travel Schemes

- Cross-Border Collaboration Voucher: Up to £500 or euro equivalent towards travel and accommodation costs when developing and establishing H2020 partnerships in the opposite jurisdiction.
- EU Travel Scheme: Up to £350 or euro equivalent financial support for existing North South partnerships towards the cost of attending H2020 related events, consortium meetings or EU Commission meetings taking place in Europe.

Advisory Service -

InterTradeIreland offers H2020 participants:

- Free advice and online guides
- Help with identifying partners
- Advice related to project ideas
- Signposting to the relevant supports

InterTradeIreland Horizon 2020 App

The InterTradeIreland Horizon 2020 App promotes North South collaboration in Horizon 2020 by bringing together innovative SMEs, researchers, academic institutes and other organisations. The free App can be accessed via the InterTradeIreland website or it is available to download onto mobile devices at the Apple, Windows and Android App stores.

Events

InterTradeIreland has developed the 'Focus On' event series which concentrates on specific opportunities around topics in the Horizon 2020 Work Programmes. These invitation only events bring together the relevant stakeholders to explore the potential for North South collaboration. Details of upcoming events can be found on our website or using our app.

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Sigma-Aldrich is committed to accelerating customer success through innovation and leadership in Life Science and High Technology.

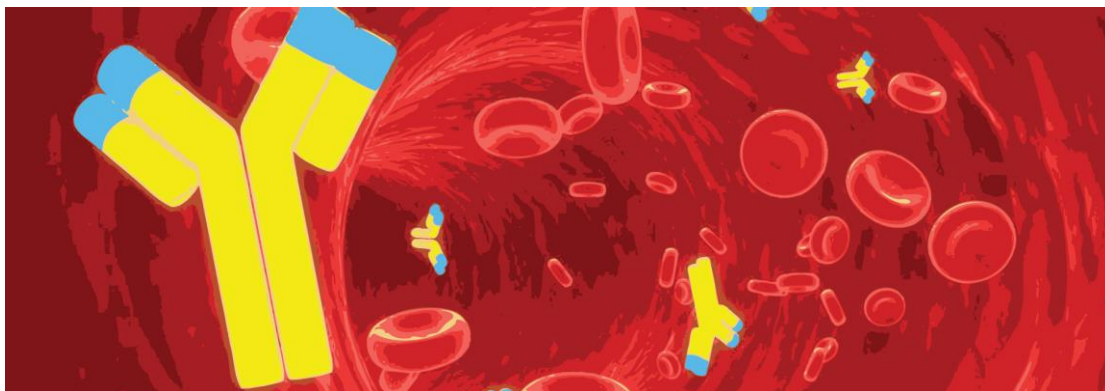
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Your local contact:

Andreina Moran
Account Manager
Sigma Aldrich Ireland Ltd

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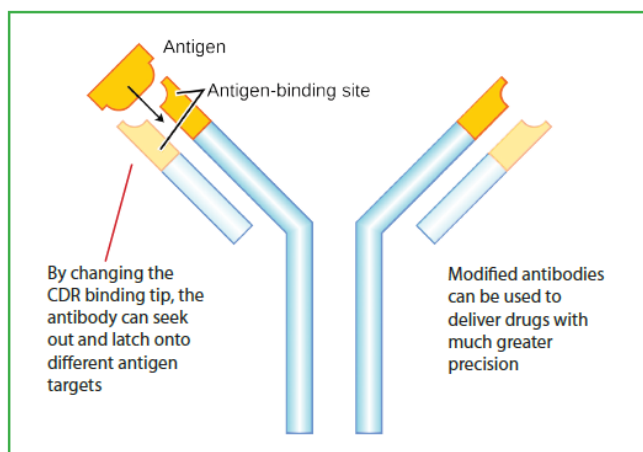
Antibodies giving Irish Industry a Healthy Boost



Report, Tom Kennedy, Science Spin Magazine.

We are constantly under attack. Surrounded by germs, all waiting to invade, yet good health lasting from infancy to old age is thought to be normal. Without our billions of tiny antibodies, leading the attack on invaders, life as most of us enjoy it, would be impossible.

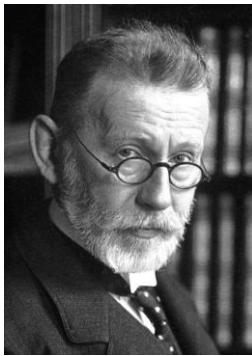
One of the remarkable things about antibodies is that so many different types exist, each equipped to seek out a particular foreign body and tag it for destruction. There are millions of different types, said Prof Richard O’Kennedy, who happily admits to loving antibodies. Not only does he find them fascinating, but as he pointed out to members of the Institute of Chemistry of Ireland when they met for their annual congress in Limerick, he also sees them as representing a huge opportunity to boost Ireland’s biopharma industry. At DCU’s Biomedical Diagnostics Institute, Richard and a large team of researchers have been looking at how the ability of antibodies to seek out invaders can be employed for disease diagnosis and more effective drug delivery.



Although there are probably as many antibodies as there are diseases, they all have a similar structure. Richard described them as a bit like himself, two legs and two arms, and it is the tip of an arm that makes them all different. This tip, known as the Complementary Determining Region (CDR), is the part that latches on to the target, known as the antigen. This might, for example, be a distinctive molecule on the surface of a cancer cell, and once the binding occurs, the antibody gets to work. The antibody, explained Richard has a few weapons in its armoury. It can attack directly, it can target the supporting blood vessels or it can get other proteins or cells to come in for the kill.

As long ago as 1897 the ability of antibodies to seek out their targets was known, and Richard said that the
 Irish Chemical News 2015 Issue No.1

Nobel Prize winner, Paul Ehrlich, was the first to suggest that they might be used in medicine as “magic bullets” for delivery of drugs. However said Richard, it was not until the 1990s that this possibility began to become a reality through the production of monoclonal antibodies.



More than a century ago Paul Ehrlich suggested that antibodies could be used as ‘magic bullets’

In the 1970s, César Milstein and others discovered that it is possible to produce antibodies that are specific to just one type of antigen by fusing previously challenged spleen cells from a mouse with cancer cells. The mouse cells contributed the antibodies and the cancer cells enabled the hybrid to keep on dividing. These highly specific antibodies could be used for diagnosis of diseases and scientists could now think of producing potential carriers for piggy-backing drugs directly to the intended targets.

The Nobel Prize in Physiology or Medicine 1984 http://www.nobelprize.org/nobel_prizes/medicine/laureates/1984/



César Milstein
Prize share: 1/3



Niels K. Jerne
Prize share: 1/3



Georges J.F. Köhler
Prize share: 1/3

The Nobel Prize in Physiology or Medicine 1984 was awarded jointly to Niels K. Jerne, Georges J.F. Köhler and César Milstein *"for theories concerning the specificity in development and control of the immune system and the discovery of the principle for production of monoclonal antibodies"*.

Richard said he had been lucky enough to meet César before he got the Nobel Prize, and when asked about why he had undertaken this research the scientist had replied that it was because he was curious. César had not been thinking of possible applications, and at the time, the Medical Council funding the research took the view that the findings had no value. The discovery, said Richard, was never patented, yet antibody drugs will shortly be the most valuable one's ever developed.

Whenever a case to be made for supporting basic research, said Richard, this is a good example, but, as he added, it took a century to go from concept to application.

Five of the top drugs worldwide are now based on antibodies, and as Richard pointed out, this is just the beginning, and Ireland is in a very strong position to benefit from this. The knowledge is in place as is the industry, he said. Nine of the top ten pharma companies are already in Ireland and of the 83 manufacturing Irish Chemical News 2015 Issue No.1

plants 33 are FDA (Federal Drug Administration) approved.

At the Biomedical Diagnostic Institute, said Richard, researchers from different disciplines come together to work on antibodies. As Richard explained, his own knowledge of biology would not be of much use unless there are chemists involved. “Chemists are very good at making things stick together,” he remarked. Much of the research involves playing around with molecular structures, and Richard said researchers can produce antibodies with specific binding properties, they can make derivatives and get them to glue killer toxins onto specific cells.



At the Biomedical Diagnostics Institute, DCU, a large team experts from different disciplines work together on research

Richard loves antibodies, and he also has a fondness for chickens. Chickens, he said have a high ambient temperature, so their antibodies are more stable than ours. They lay eggs full of antibodies, which can be harvested, and from these all sorts of derivatives can be synthesised, including those with human inclusions. The all-important CDRs, he said are amino acids, and chemical engineers can do a lot with them to make them work in different ways. “It’s a bit like playing with Lego,” said Richard, and the more they understand about their molecular structures, the more they can do to match them up with particular targets.

This approach to drug delivery is highly effective, and as Richard explained, many drugs, such as those used against cancers, have such dreadful side effects that patients often do not want to use them. This is because the drugs are not specific to one target, and for example, a drug that kills tumours is also likely to kill other fast-growing cells. Once a marker is identified, and a complementary CDR attached to the arm of an antibody, precise targeting to the diseased cells is possible.

“Antibodies are good, but we want them to be better,” said Richard, and there is a big opportunity to match them up with existing drugs, making them more effective while giving them a new lease of life. “You can do a lot,” he said, “one end of the antibody can bind onto a drug and the other end can bind onto something like a toxin, so we get a combination of actions.” With this approach, he said, dosage goes down and you get a thousand fold better targeting.

Using antibodies in diagnostics is already well established, with lots of start-ups in Ireland, and Richard said we can expect to see big improvements in this field. As reported elsewhere in this issue, it is possible to automate testing for a variety of diseases by incorporating antibodies into the channels of a chip or disk. Because chemists, engineers, physicists and biologists are working together, said Richard, this is a fast developing field. The day is not far off, he said, when all we will need is an app on our phone to take and send a reading from your skin to a GP who can then make an initial diagnosis.

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Congress Photos from UCD





Some more Photos



President Margaret Franklin Opening Congress 2015



President Margaret Franklin at Queens presents Annual Award to Prof Thorri Gunnlaugsson TCD at 3rd in series of Annual Lecture



President Patrick Hobbs presents the 2015 Robert Boyle Gold Medal to Prof Dermot Diamond at DCU