Selective Synthesis: New Reagents for Specific Transformations
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The chairmen of the European Research Councils’ Chemistry Committees (CERC3) elected organic synthesis as this year’s topic for their annual workshop, and Switzerland as the host country. The conference chairman, Prof. Philippe Renaud made the excellent choice of Champery for hosting the workshop; the combination of his enthusiasm, efficient organization and the beautiful alpine scenery made this meeting a really unique event. Its success clearly arose from the scientific program, which was particularly exciting. Thirty-one half-hour lectures from young chemists, mostly at the beginning of their independent research careers were complemented by three plenary lectures from prominent, established professors in Europe.

The first lecture was given by Bernd Goldfuss (University of Heidelberg, Germany), focusing on the use of modular fencholates as probes for studying the enantioselective addition of organozinc reagents to benzaldehyde. High-level computational modeling of the transition structure revealed that subtle changes of substituents on the ligand, even remote from the chiral centers, had a strong impact on the geometry of the dimeric catalyst; these effects could be so strong that similar catalysts varying only by one substituent gave opposite enantiomers [1].

The afternoon session was concluded by Petri Pihko (The Scripps Research Institute, La Jolla, USA), on the synthesis of Azaspiracid (Fig. 1), a very potent toxin, isolated from Irish mussels. The FGHI spirocyclic portion proved to be a very challenging pattern to assemble, particularly with the presence of acid-sensitive ketal groups. After several attempts at redesigning the route without success, Pihko showed how the azaspiro moiety was eventually prepared efficiently. The 'northern part' ABCDE proved to be just as challenging; despite intensive efforts, to date, it has only been partially prepared. These synthetic problems show once again that total synthesis remains an art, and that it should not be taken for granted that any molecule can now be easily synthesized [2].

The day was concluded with the first plenary lecture, by Professor Varinder Aggarwal (University of Bristol, UK). He explained the asymmetric epoxidation reaction, in which, instead of using the classical strategy (i.e. oxidation of an alkene), the key step is a carbon-carbon bond forming process (the so-called Corey-Chaykovsky epoxidation). The beauty of this reaction, very simple in its experimental realization, is revealed by the intricate catalytic cycles orchestrated like clockwork. Detailed mechanistic studies, including catalyst structure/activity relationships and computational modeling were presented to rationalize the origin of the enantioselectivity. In the last part of his lecture, Aggarwal showed that this reaction was not limited to epoxidation, but can be applied also to aziridination or cyclopropanation [3].

Transition metal asymmetric catalysis was central to the first part of the Thursday morning session, starting with Giuseppe Mantovani (ISSEC Firenze, Italy). The methoxycarbonylation of styrene (i.e. the reaction of styrene with carbon monoxide and methanol) was investigated under Pd catalysis with various phosphorous ligands. It was found that small changes in the ligand structure induced dramatic changes in the reaction outcome. For example, the classical Pd(dppe)(MeCN), yielded exclusively methyl cinnamate (the expected product), whereas a dimeric analogue of 2,3-dppd (1,3,4,5 tetrakis(diphenylphosphino) cyclobutane) led mainly to the formation of dimethyl phenylsuccinate [4].
The field was then expanded by Simon Breeden (National University of Ireland, Galway), showing the versatile activity of the chiral ESPHOS (Fig. 2) and semi-ESPHOS ligands. They were used in Rh-catalyzed asymmetric olefin hydroformylation, Pd-catalyzed asymmetric allylic substitution and amination, and in Cu-catalyzed conjugate addition of organozinc compounds to enones [5].

![Fig. 2](image)

The first part of the Thursday morning session was concluded by an interesting lecture from Peter Langer (University of Göttingen, Germany), focusing on the regio- and stereoselective cyclization of diazomium ions with dielectrophiles. The elegance of the concept resides in its apparent simplicity. However, making it a regio- and stereoselective reaction is by no means trivial. For example, the diazium of ethyl acetacetate cleanly reacted with the bis-Weinreb amide derived from oxalic acid, to give an α-alkylidene-α-hydroxybutenolide with a geometric purity greater than 98% (E). On the other hand, the related bis-silyleneether reacted with diacetyl in the presence of titanium tetrachloride to give a highly functionalized cyclopentenone (Scheme 1). Langer went on to describe the reaction of 1,1-diazo-1-methylene-2-aminobenzene with the same type of 1,2-dielectrophiles leading to complex heterocyclic structures [6].

The second part of the morning was dedicated to more multidisciplinary aspects of organic synthesis, in particular with the contribution of Mario Smet (University of Leuven, Belgium), dealing with the preparation of heterocyclic analogues of rubrene. These molecules were shown to exhibit interesting photophysical and electronic properties, and are potentially useful as dyes in laser technology. Smet also discussed the use of diketopyrrolopyrroles as fluorophores for calcium ion sensors [7].

Also interesting was the work presented by Reko Leino (Abo Academy, Finland). Solid support chemistry is usually carried out on polymeric matrices (e.g. polystyrene beads), prepared by grafting or co-polymerizing with the molecule of interest. The drawbacks of this approach are poor thermal stability and low mechanical resistance. Other types of solid supports lacking these drawbacks are the fibrous polymers. These fibers (of length up to 2 mm) can be exposed to an electron beam generating radicals able to react with the suitable modifier. This technology is already routinely used for textile processing. A spectacular example was the immobilization of a TADDOL-ligand by this technique. This solid catalyst was then tested on the enantioselective addition of diethylzinc to benzaldehyde in the presence of Ti(IV). The enantiomeric ratio of 97:3 was constant, even after several cycles [8].

Christian Bochet (University of Geneva) closed the morning session, in a lecture dealing with the selective activation of functional groups using monochromatic light. In this approach, the selectivity does not arise from elements contained in the substrate or the reagent, but from the wavelength of a monochromatic light beam. The clean and smooth orthogonal deprotection of photosensitive protecting groups was shown as an example [9].

The afternoon session was opened by John Storey (University of Kingston, UK). He presented his recent results on the preparation of oxindoles and spirooxindoles by radical cyclization. The classical approach, which involves the cyclization of α-bromoanilides, has been shown to give poor yields of oxindoles. Storey was able to use a very elegant radical translocation, where the halide is located on the aromatic ring (e.g. in a α-bromoanilide). Upon initiation, the aryl radical abstracts the hydrogen on the amide, forming a new radical, which is then capable to cyclize. Excellent yields were obtained in these cases [10].

Andreas Gansäuer (University of Bonn, Germany) showed how epoxides could be very efficient radical precursors when treated with a titanocene dichloride catalyst, in the presence of a stoichiometric reductant. Such β-alkoxy radicals can then add to olefins or α, β-unsaturated esters. Tethering the radical acceptor to the epoxide allowed the formation of oxygen- and nitrogen-containing polycyclic heterocycles [11].

Titanium complexes were also central to the research presented by Olivier Riant (Université Catholique de Louvain, Belgium), but this time for asymmetric catalysis, as salen-type complexes. These easily prepared, air-stable complexes were able to catalyze the asymmetric pi-nacol coupling of aldehydes, in excellent diastereoselectivity (90%), and encouraging enantioselectivity (up to 77% ee). Riant then went on to discuss the hydroisofloration of ketones with triphenylsilane, in the presence of copper fluoride. When a binaphthyl-derived chiral ligand was used, enantioselectivities went up to 80% ee. Such a transformation was found to be very practical, and no protection from air and moisture is required.

After the coffee break, Göran Hilmersson (University of Göteborg, Sweden), detailed the addition of chiral amine-noethers to butyllithium, forming C7-symmetric amide dimers in diethylether solution. The addition of THF broke these aggregates, and only monomers were detected by Li-NMR. Semi-empirical calculation at the PM3 level, NMR study and structure/activity analysis complemented these studies. In a second part, Hilmersson examined the properties of 1-lithioacetoniirile. Indeed, these compounds are known to show an imide character, where the metal is mainly located on the nitrogen atom. So the question arose: why a chiral lithium modifier (e.g. an aminoether) can lead to significant asymmetric induction when located so far away from the reacting center? Hilmersson embarked on a program to answer this question [12].

Asymmetric catalysis was also at the center of the lecture of Henri Doucet (Université de Marseille, France). Indeed, palladium-catalyzed reactions frequently suffer from a quite low turnover number, because of the precipitation of
colloidal zerovalent palladium (so-called 'black out'). Strongly bound phosphine ligands can potentially prevent this event occurring, but also hinder the efficiency of the catalysis. Doucet solved this problem by preparing a cyclic tetraphosphine (Tedycip) (Fig. 3). When used in Pd-catalyzed reactions (Heck reaction, Suzuki couplings, malonate allylation), extraordinary turnover numbers were observed, up to 10 billion [13].

Concluding the session, Richard Grainger (King's College, London, UK), exposed his efforts to harness the power of sulfur monoxide in Diels-Alder reactions. Although sulfur dioxide is very well mastered, sulfur monoxide is extremely unstable, and has to be generated in situ. Grainger systematically explored all the possible ways to generate it. Organometallic catalysis, and photochemistry were attempted, generally with some success, but never in quantitative yields. It was finally by thermolysis of a novel trisulfide-oxide that Diels-Alder trapping of SO occurred in up to 100% yield. This work nicely showed that perseverance coupled with careful mechanistic studies and X-ray analysis could solve apparently insoluble problems [14].

The evening plenary lecture was brilliantly delivered by Samir Zard (Ecole Polytechnique, Palaiseau, France). He started with the total synthesis of a tetracyclic alkaloid, the 13-deoxy-serratine. His approach (twelve steps) was three times shorter than the only existing synthesis, with an overall yield about a thousand times greater. Zard continued with the observation of the strange conversion of a trisubstituted olefin into an internal acetylene upon treatment with sodium nitrite and acetic acid. Careful examination of the mechanism and preparation of some of the intermediates led to the development of a general synthesis of acetylenes, reacting isoxazolinones with sodium nitrite and acetic acid in the presence of iron sulfate. The lecture was concluded with a new procedure for preparing esters, under totally neutral conditions (Scheme 2). Indeed, simple heating of an acid and a propargyl xanthate allowed the clean formation of esters; in fact, any HX was found to work, from hydrofluoric acid to phenol [15].

The sunny Friday morning session, chaired by Varinder Aggarwal, was opened by a most interesting lecture from Martin Hiersemann (Technical University of Dresden, Germany), on the metal-catalyzed Claisen rearrangement. The thermal rearrangement of allyl-vinyl ethers (achiral substrate), cleanly gives a single diastereoisomer of a chiral compound, but in its racemic form. Clearly, this was an opportunity for asymmetric catalysis. Precedents from literature showed that Lewis-acidic transition metals were able to promote the reaction, even enantioselectively, but always by using stoichiometric amounts of chiral ligands. In order to solve this problem, Hiersemann first studied the factors governing the selectivity, and in particular the influence of the double bonds on the diastereoselectivity. He then found that the use of copper triflate in the presence of a chiral bis-oxazoline, allowed the reaction to proceed with up to 88% e.r., even using catalytic amounts. Interestingly, changing the substituents on the bis-oxazoline could reverse the major enantiomer obtained [16].

The catalytic activity of copper/bis-oxazoline complexes was further developed by Jacob Thorhauge (University of Aarhus, Denmark), in a very lively lecture. These complexes were capable of catalyzing the hetero Diels-Alder condensation of electron-rich alkenes with \( \alpha \)-ketoenones, with an extremely high asymmetric induction (routinely greater than 90% ee). Thorhauge went on to describe the enantiomeric selective Friedel–Crafts alkylation. As an example, he showed that indoles could react with \( \alpha \)-ketoenones in the presence of the Cu-bis-oxazoline catalyst. In the last part of his talk, Thorhauge discussed a strange reversal in the sense of asymmetric induction in the Diels-Alder reaction, similar to the one described in the previous lecture. High-level calculation and screening of metallic cations shed some light on the nature of the reversal. Although still at a preliminary stage, this work showed that the \( \beta \)-Bu-substituted bis-oxazoline/Cu(II) complex is fairly rigid (but neither tetrahedral nor square-planar), whereas the phenyl-substituted analogue is more flexible [17].

The world of antitumor alkaloids was then explored by Maria-Joao Queiroz (University of Minho, Portugal), showing all the efforts of her team to assemble the skeleton of pyrocarbazoles, such as ellipticine or olivacine. These structures owe their potent activity to their ability to intercalate in DNA and their interaction with DNA-topoisomerase II. An original combination of reductive or Heck-type intramolecular cyclizations and transition-metal catalyzed C–N bond-forming reactions was used to access the thienocarbazole system, another interesting and potentially active analogue [18].

The second part of the morning continued with a breathtaking plenary lecture by Erik Carreira (ETH Zürich). He focused on the zinc triflate-catalyzed addition of terminal alkynes to nitrones and aldehydes. The elegance and efficiency of this process is the underlying C–H activation, leading to an atom-economical transformation (i.e., all the atoms of the reactants are included in the products); this reaction does not generate any waste. This is a major goal for the incoming century. Not satisfied with this achievement however, Carreira rendered the process asymmetric. By using N-methylphedrine as a ligand, very high enantiomeric excesses could be obtained [19] (Scheme 3).

The morning session was concluded by Philippe Bertus (University of Reims-Champagne-Ardenne, France), who talked about an interesting ketone cyclopropanation reaction, in which the addition of a zirconacyclopropane complex to enones and subsequent treatment with sulfuric acid (or a Lewis acid) cleanly afforded vinylcyclopropanes. The versatility of this functional group in reacting further in a number of different reactions...
makes this process particularly desirable [20].

The afternoon session was chaired by Samir Zard, and started with the presentation by Alison Franklin (University of Exeter, UK), focusing on the preparation of nitroalkenes. Nitroalkenes are valuable building blocks, due to their Michael-acceptor properties, and by the possibility of reducing the NO₂ function down to an amine. Franklin found that a Horner-Emmons type reaction of a nitrophosphonate to benzaldehyde efficiently yielded substituted nitroalkenes, and systematically optimized the parameters. She then examined their hydrogenation to yield alkylamines, first using Wilkinson’s catalyst, then switching to the asymmetric version. The use of a ruthenium-BINAP catalyst gave an enantiomeric excess of 25%. Interestingly, this quite sluggish reaction was greatly accelerated by microwaves [21].

Carsten Schmuck (University of Cologne, Germany) continued with the synthesis of new artificial amino acids as building blocks for peptide receptors. By studying the ability of Vancomycin to specifically recognize the L-Lys-D-Ala-D-Ala sequence in bacteria proteins with molecular modeling, it became apparent that the assembly of a cyclotribenzylene aminoacid, D-valin and a guanidinium aminoacid could behave similarly. Hence, Schmuck detailed his efforts towards the preparation of the unusual cyclotribenzylene. Whereas the unsubstituted parent ring was relatively unproblematic, the preparation of a monosubstituted cyclotribenzylene proved to be a tough challenge.

The next lecture was delivered by Adriaan Minnaard (University of Groningen, Netherlands), who exploited the efficient enantioselective addition of diethylzinc to enones under Cu catalysis to prepare building blocks for asymmetric synthesis. The well-known Feringa ligand was used in nice examples of desymmetrization of meso-dienes. The powerful asymmetric induction was also exemplified by the reaction with chiral racemic enones, in kinetic resolution. In both cases, very high ees were obtained [22].

Dario Bassani (University of Bordeaux, France) opened the final session of the day with a completely different field, in an exciting lecture on supramolecular photochemistry. It is known that the photochemical dimerization of cinnamates is not very efficient, and not particularly selective (many of the possible stereoisomers are obtained). On the other hand, by ordering the reactants through hydrogen-bonding with a suitable template, real supramolecular catalysis occurs. A ten-fold acceleration factor was observed, with perfect stereoselectivity [23].

Jean-Michel Brunel (University of Marseille, France) continued, by showing the preparation and the use of new chiral o-hydroxyarylphosphine oxides in asymmetric catalysis (Fig. 4). This ligand was very effective, not only in the addition of diethylzinc to benzaldehyde, but also in the asymmetric formation of benzaldehyde cyanohydrin. The free phenol could be methylated, leading to a new ligand, which was particularly effective in the asymmetric opening of epoxides by tetrachlorosilane [24].

Bernd Schmidt (University of Dortmund, Germany) closed this afternoon session, in a lecture focusing on the use of Grubbs-type catalysts to form cyclic ethers by ring-closing metathesis. The residual double bond was shown to be a versatile handle to introduce further hydroxyl groups and to prepare C-glycosides. A broad variety of these structures could hence be accessed in a very efficient process [26].

The final day was opened by Floris van Delft (University of Nijmegen, Netherlands) showing his recently initiated work, at the interface of organic chemistry and biochemistry.

Gilles Alcaraz (University of Rennes, France) was next, and he showed the applications of resin-capture-release strategy in the immobilization of boronates. In this approach, an aryloboronic acid is captured by an ammonium resin. This ion-pair is stable, and shows no leaching, even after continuous extraction with THF. On the other hand, the treatment of this resin with an aryl halide and a Pd-catalyst allowed the clean formation of biphenyls via a Suzuki coupling. Very nice examples of macrocyclic biarylates were prepared by this method [27].

We remained in the world of transition-metal catalyzed cross-coupling reactions with Amadeu Brigas (University of Algarve, Portugal). He detailed the use of non-conventional leaving groups for Ni-catalyzed coupling of Grignard reagents with aryl ethers, using tetrazoles or pheno- derivatives [28].

Jerome Lacour (University of Geneva) demonstrated the power of helicoidal TRISPHAT anions (Fig. 5) as chiral resolving agents (for chiral cationic complexes, such as [Fe(4,4'-Me₂bppy)₂]F⁺, or as NMR shift reagents. A new modification, the chiral binaphthol-derived BINF PHAT (Fig. 5) was also described, allowing the induction of asymmetry in configurationally labile monometinium dyes [29].

Sandra Jonsson (University of Stockholm, Sweden) continued with a fascinating lecture on the dihydroxylation of olefins using hydrogen peroxide as a stoichiometric oxidant. She detailed the complex intertwined catalytic cycles involving the hydrogen peroxide oxidizing a flavin, itself oxidizing NMM into NMO, which then oxidized osmium to dihydroxylate the olefin (Scheme 4). Even more impressively, an asymmetric reactions using chiral N-acylated oxaziridines. His systems were able to transfer ureas with very high diastereoselectivities [25].
version was also developed. The addition of the Sharpless ligand (DHQD$_2$PHAL), led to ees up to 95% [30].

Nico Margiotta (University of Bari, Italy) completely changed the field in the final lecture, and showed a new approach for cancer chemotherapy. It is indeed known that some cancers have a viral origin or are virus-related. Hence, it is legitimate to try targeting both the tumor and viruses by a single drug. This strategy was adopted by Margiotta, who showed hybrid compounds, derived from cisplatin (currently used as antitumor) and acyclovir/penciclovir (antiviral). Interestingly, these compounds exhibited good activity against HIV, but also good cytotoxicity. This is promising for a simultaneously treating viral diseases and tumors [31].

A few words by the conference chairman, Prof. Philippe Renaud, concluded this exciting workshop. In four days and 34 lectures, the participants could get a clear overview of the high-quality organic chemistry that is currently being carried out in Europe. The format chosen by the organizer was particularly well adapted to fuel the enthusiasm of hearing about such a broad range of topics. The fact that most of the participants were also active lecturers also contributed to keep the interest during the question sessions, meals and late-night discussions. There are no doubts that the future editions will be as successful, and let us hope that the next one will occur very soon.

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General References

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