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Arkivoc **2024** (4) 20231498

Professor R. Alan Aitken

A Tribute

This commemorative issue of Arkivoc is dedicated to Dr R. Alan Aitken to acknowledge his contribution to organic chemistry

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Robert Alan Aitken was born on 27th March 1958 to a farming family in the Dumfries and Galloway area of Southwest Scotland. Despite his rural upbringing, he was not the first scientist in the family since his great grandfather's younger brother, Edward Hamilton Aitken ("EHA", 1851–1909), was a noted author and naturalist in India in the days of the Raj and a founder member and first Secretary (1883–1886) of the Bombay Natural History Society. He gave his name to several biological species including five species of Indian ant and a mosquito *Anopheles aitkenii*. When it came time for university, Alan followed the example of the only previous family member to go to a UK university, his paternal grandmother Rona Macrae (MA, 1925), and went to the University of Edinburgh. There he completed a BSc (1979) with a final year research project on stabilised phosphonium ylides and this was followed by a PhD (1982) on flash vacuum pyrolysis of cyclic sulfones, both projects jointly supervised by Dr Ian Gosney and Professor J. I. G. (later Sir John) Cadogan. He then obtained a Fulbright Scholarship for postdoctoral studies with Professor Albert I. Meyers at Colorado State University, Fort Collins (1982-84) in the area of asymmetric synthesis using nitrogen heterocycles. In 1984 he was awarded a Royal Society Warren Research Fellowship and joined the staff at the University of St Andrews, the first new staff appointment there since 1972. He has remained there ever since and is currently Reader in Organic Chemistry. He has been a Fellow of the Royal Society of Chemistry since 1997.

Alan has maintained a small but productive research group and has so far supervised the research of 10 postdoctoral researchers, 29 PhD students, 9 MSc and MPhil students and 145 undergraduate project students. These include coworkers from over 20 countries: Belgium, Brazil, Canada, China, France, Germany, India, Ireland, Israel, Malaysia, Mali, Morocco, Nigeria, Pakistan, Romania, Russia, South Africa, Taiwan, USA, Zaire and Zimbabwe.

He has also managed to combine his love of travel with presenting his research and has given invited lectures and conference presentations in 35 countries: Australia, Austria, Belgium, Canada, China, Denmark, Egypt, Finland, France, Germany, Hong Kong, India, Ireland, Israel, Italy, Jamaica, Japan, Jordan, Kuwait, Latvia, Mexico, New Zealand, Poland, Portugal, Russia, Slovenia, South Africa, South Korea, Spain, Sweden, Taiwan, Trinidad & Tobago, Turkey, USA, Yugoslavia. In 1994 he spent a sabbatical period at the University of Texas, Austin (A. H. Cowley FRS) and has also been Teaching Fellow at the University of Hong Kong (Jan-Feb 1999) and Visiting Professor for short periods at Kuwait University (2002) and the University of the West Indies, St Augustine, Trinidad & Tobago (2012).

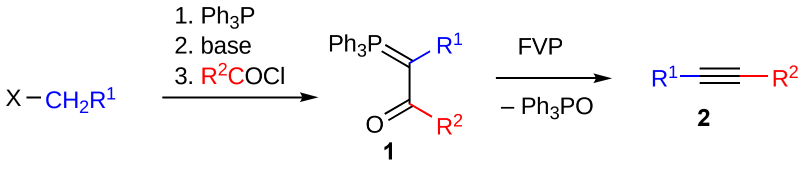
As well as the original research areas described in more detail below, he has made a very large number of contributions (over 90!) to major reference works and annual review series including the following (number of chapters in brackets): Organic Reaction Mechanisms (7), Progress in Heterocyclic Chemistry (40), Comprehensive Organic Functional Group Transformations (1), Comprehensive Heterocyclic Chemistry II (2), Chemistry of the Functional Groups (Patai, 2), Science of Synthesis (19), Advances in Heterocyclic Chemistry (5), Comprehensive Organic Functional Group Transformations II (3), Comprehensive Heterocyclic Chemistry III (4), Comprehensive Heterocyclic Chemistry IV (8).

Alan has also acted as a volume editor for Comprehensive Heterocyclic Chemistry III (volume 7), Comprehensive Heterocyclic Chemistry IV (volume 4), Science of Synthesis Updates (volume 13) and Progress in Heterocyclic Chemistry (since volume 34). He has also been Publicity Chair for the International Society of Heterocyclic Chemistry since 2018 and maintains their web site. Earlier in his career he was involved in writing two undergraduate textbooks. With St Andrews colleagues Ray Mackie and David Smith he co-authored the 2nd (1990) and 3rd (1999) editions of 'Guidebook to Organic Synthesis' (Longman, 387pp). Together with Nick Kilenyi (then at Sanofi) he also edited and wrote most of 'Asymmetric Synthesis' published by Chapman & Hall (hardback 1992, paperback 1994, 233pp) which was the first introductory text aimed at advanced undergraduates on what was then a fast developing area of chemistry.

Outside chemistry Alan has been a keen mountaineer. Inspired by his father taking him to the summit of Mt Toubkal (Morocco) at age 15, he managed during the two postdoc years in Colorado to climb half of that state's 54 14,000 foot mountains as well as the highest peaks in Arizona and New Mexico, and Mitchell Peak and the East Temple in Wyoming's Wind River Range. During his time in St Andrews, further more ambitious adventures have included the Annapurna Circuit and Everest base camp (Nepal), Kilimanjaro (Tanzania), K2 base camp and Concordia (Pakistan), Mt Teide (Tenerife), the Inca Trail (Peru), Kinabalu (Borneo), Roraima (Venezuela) and Mt Fuji (Japan).

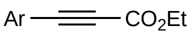
Research Interests

In his first independent publication,[1] the application of the flash vacuum pyrolysis (FVP) technique from his PhD work to b-oxophosphonium ylides **1** from his honours research project provided a versatile synthesis of aliphatic alkynes **2**.[2] Given the ready access to the ylides from alkyl halides and acid chlorides, this represents an unusual and general approach to construction of the C≡C triple bond (Scheme 1) and was subsequently developed and extended in a series of over 40 publications.



Scheme 1. General synthesis of alkynes using FVP of stabilised ylides.

Using this method products prepared in good yield have included acetylenic esters **3** and terminal alkynes **4**,[3] diacetylenic esters **5** and terminal diynes **6**,[4] diacyl alkynes **7**,[5] conjugated enynes **8**,[6] unsymmetrical 1,3-diynes **9**,[7] trioxo alkynes **10**,[8] trisubstituted naphthalenes such as **11**,[9] extended dialkynes such as **12** and **13**,[10] alkynyl ketones **14** and alkoxycarbonyl dienes **15**,[11] and oxygen heterocycle-containing alkynes such as **16–18**.[12]



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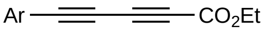

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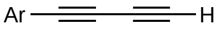

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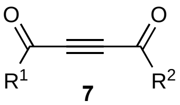

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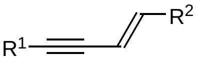

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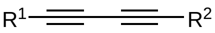

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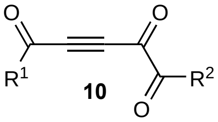

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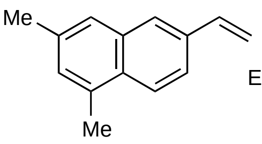

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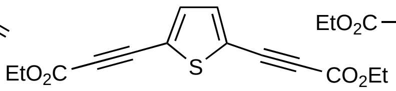

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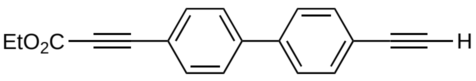

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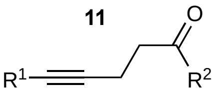

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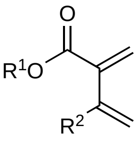

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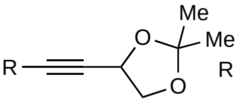

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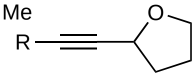

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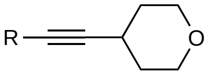

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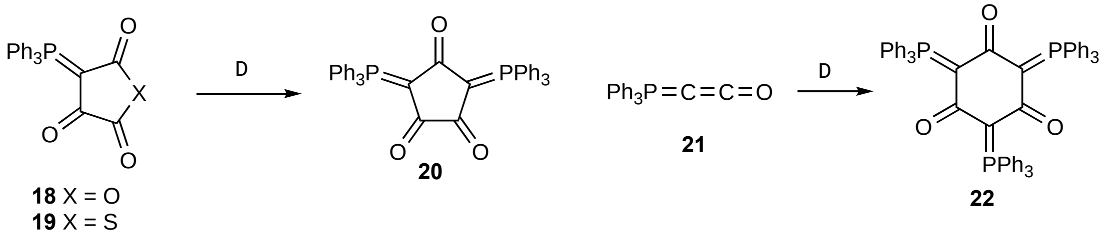
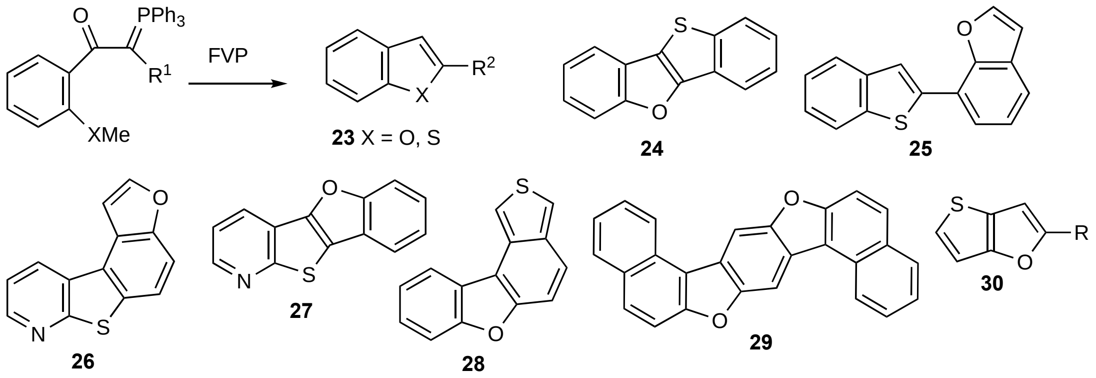
In a few cases, the ylide pyrolysis led to discovery of unexpected transformations such as thermal conversion of **18** or **19** into **20** and **21** into **22**.[13]



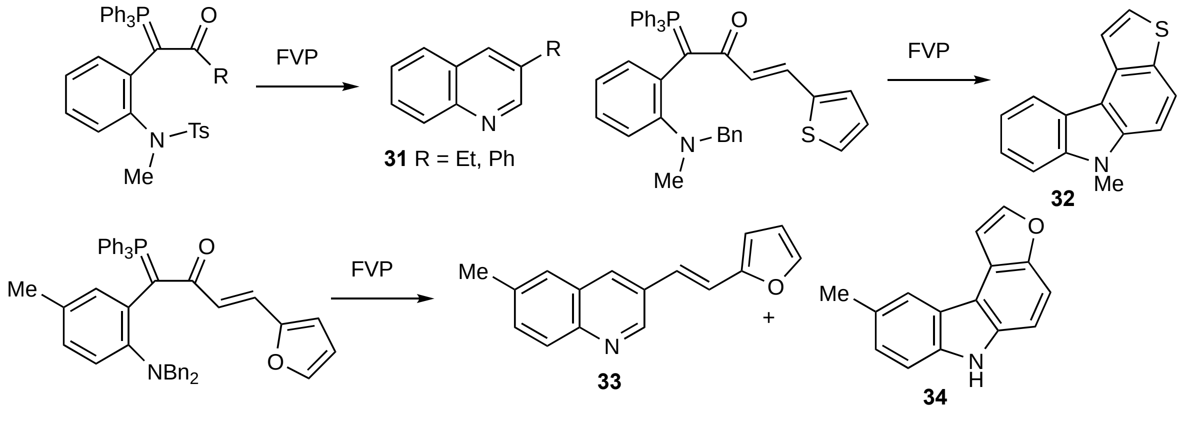
18 X = O
19 X = S
20
21
22

One of the most productive developments arose from the early observation that alkyne formation could be combined with thermal generation of an *ortho*-phenoxy or phenylthio radical leading to formation of benzofuran and benzothiophene products **23**.[14,15] This was developed into a series of "domino" cyclisation processes leading to convenient synthesis of fused ring heterocyclic products such as **24**,[16] **25**,[17] **26** and **27**,[18] **28** and **29**,[19] and **30**.[20]



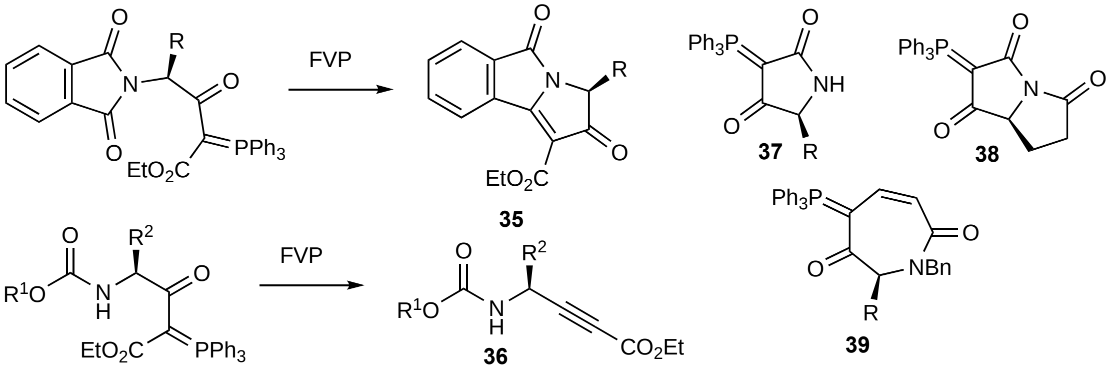
23 X = O, S
24
25
26
27
28
29
30

Moving to nitrogen as the cyclising atom brought extra complications. The *ortho* methylamino ylides unexpectedly gave 3-substituted quinolines **31** for simple R groups due to transposition of the reactive site from N to C, but for more complex R groups the originally planned cyclisation to give ring-fused carbazoles such as **32** was successful.[21] This complication was also observed when the method was applied to synthesis of the natural product eustifoline D, with a quinoline product **33** being obtained in addition to the target **34**,[22] and further examples also gave mixed quinoline and carbazole products.[23]

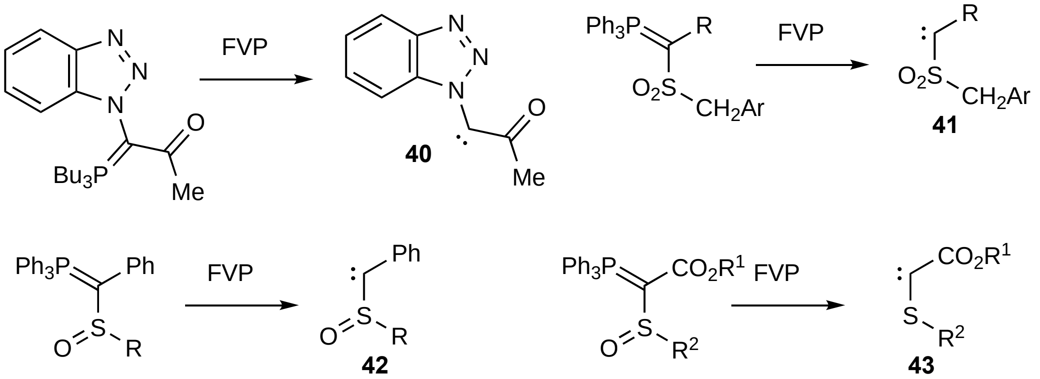


31 R = Et, Ph
32
33
34

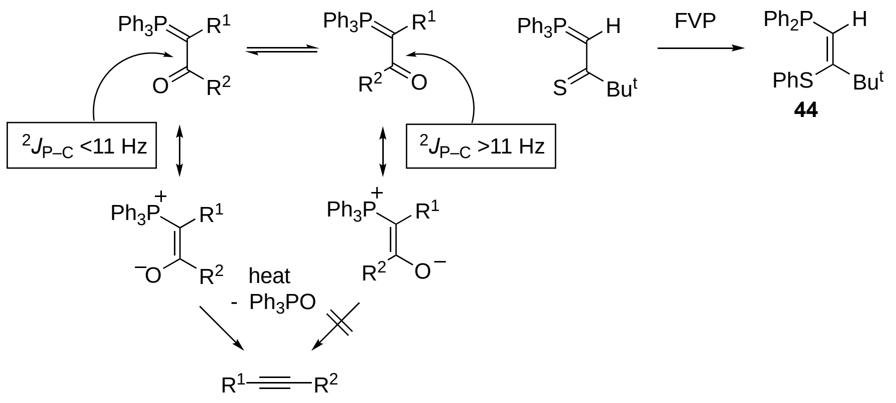
Extension to amino acid-derived ylides also gave useful results. While pyrolysis of phthalimidoacyl ylides led to elimination of Ph_3PO in an unexpected way to give pyrroloisoindolediones **35**,[24] a change to simple alkoxycarbonyl protection led to the desired alkyne products **36** and following hydrogenation to GABA analogues.[25] Further chemistry of such amino acid-derived ylides led to novel structures such as **37**–**39** .[26]



More fundamental changes to the stabilised ylide system allowed the generation of reactive intermediates such as carbenes **40**,[27] **41**,[28] **42**,[29] and **43**.[30] and pioneering studies of their reactivity.



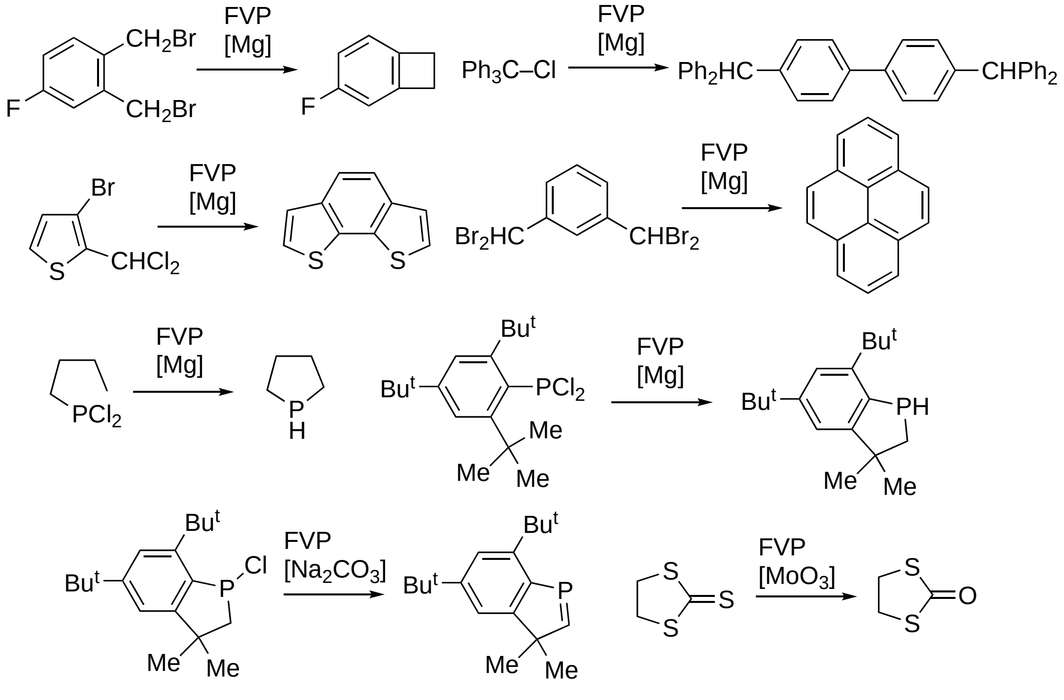
With such a huge range of stabilised phosphonium ylides available for the pyrolysis studies, their structure has been studied in detail, with a particular focus on the relative alignment of C=P and C=O as determined by X-ray diffraction,[31,32] and the electron distribution as reflected in the values of P–C NMR coupling constants.[5,32] As a result, the value of $^{2}J_{\text{P–CO}}$ was found to be a reliable indicator of the likely outcome of thermal Ph_3PO elimination as summarised in Scheme 2.[32] The kinetics of the elimination process was also examined in a number of cases in collaboration with Prof Nouria Al-Awadi (Kuwait University).[33–35] While the corresponding C=S stabilised ylides generally behave in the same way, those with R^1 = H instead rearrange to the useful bidentate phosphine–sulfide ligands such as **44** upon FVP.[36]



Scheme 2. A diagnostic indicator for thermal reactivity and an unexpected rearrangement.

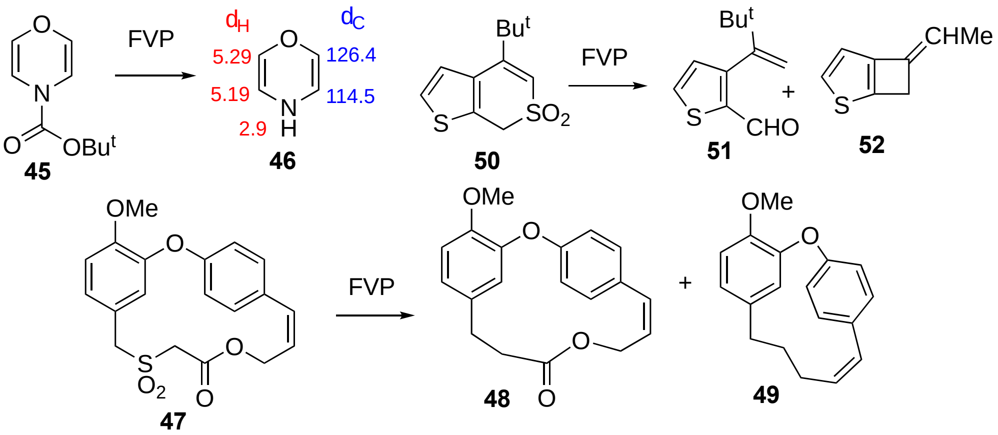
While the majority of FVP studies have been conducted through an empty furnace tube, pyrolysis through a bed of solid reagent has been useful in some cases. The initial discovery of the dehalogenating effect of resublimed magnesium on organic halides,[37–40] has been followed by application of both magnesium[41] and sodium carbonate[42] to P–Cl compounds, resulting in the former case in generation of phosphinidenes and in the latter a phosphindole (Scheme 3). More recently, FVP over molybdenum trioxide has been used in conversion of 1,3-dithiolane-2-thione into 1,3-dioxolan-2-one.[43]

The FVP technique has been applied to effectively solve various other problems. It provided the ideal method for generation of the highly reactive and unstable 1,4-oxazine **46** by elimination of CO_2 and isobutene from the Boc precursor **45**, allowing spectroscopic characterisation of this fundamental heterocycle for the first time.[44] It also proved instrumental in an original synthesis of the combrestatins in collaboration with Prof Rainer Schobert (Universität Bayreuth) where extrusion of SO_2 from sulfone **47** to give the desired product **48** was accompanied as a side reaction by additional extrusion of CO_2 to give the cyclophane **49**.[45] A collaboration with Prof Adrian Schwann (University of Guelph, Ontario) involving FVP of thiopyran *S,S*-dioxides turned up some



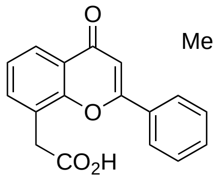
Scheme 3. Some transformations by FVP over solid reagents.

unexpected transformations, such as conversion of **50** where loss of SO to give aldehyde **51** was accompanied by rearrangement and further loss of acetone affording the ethylidenecyclobuta[*b*]thiophene **52**.[46]



In the early 1990s, encouraged by his then St Andrews colleague the late Colin Thomson, Alan started a collaborative study with Profs John Double and Mike Bibby at the Clinical Oncology Unit, University of Bradford in which a total of 60 analogues of the known anticancer agent flavone-8-acetic acid **53** were prepared and tested *in vitro* and *in vivo* for antitumour activity. The more active compounds included **54**,[47] **55**,[48] **56**,[49] and **57**[50] although none ultimately reached the clinic. In another collaborative study with Dr Russel Ramsewak, following his visit to the University of the West Indies, the structure of the unusual trichlorodihydroxybibenzyl **58** isolated from a terrestrial plant was confirmed by total synthesis.[51]

Alan's first research council-funded project examined an early example of what would now be termed organocatalysis, where highly functionalised achiral *meso* compounds were subjected to methanolysis in the presence of cinchona alkaloids. As shown in the example of **59** the resulting product containing six contiguous stereocentres is formed in reasonable enantiomeric excess, which for crystalline examples could be enhanced to a useful level by simple recrystallisation.[52,53]



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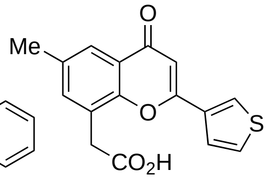

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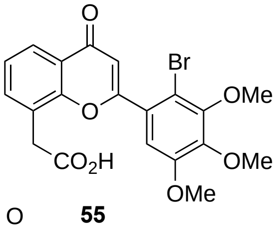

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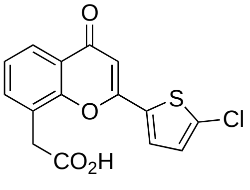

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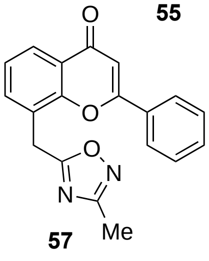

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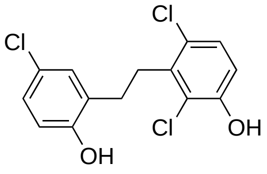

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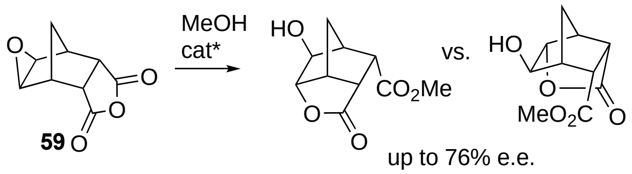
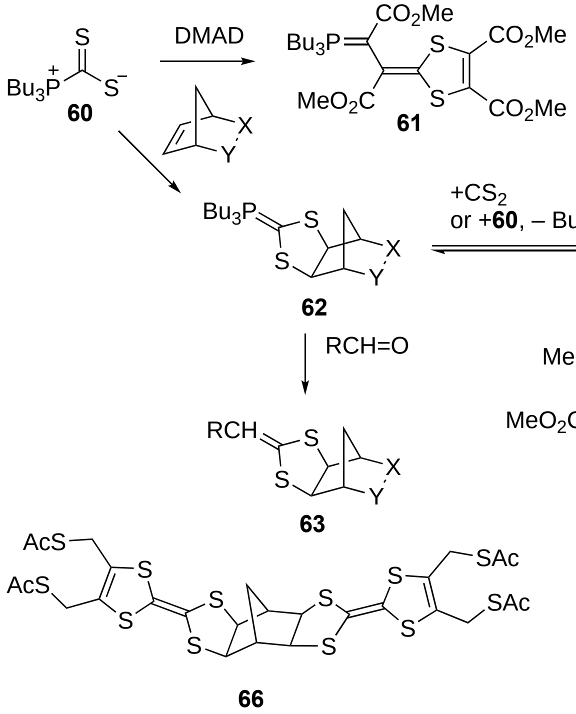


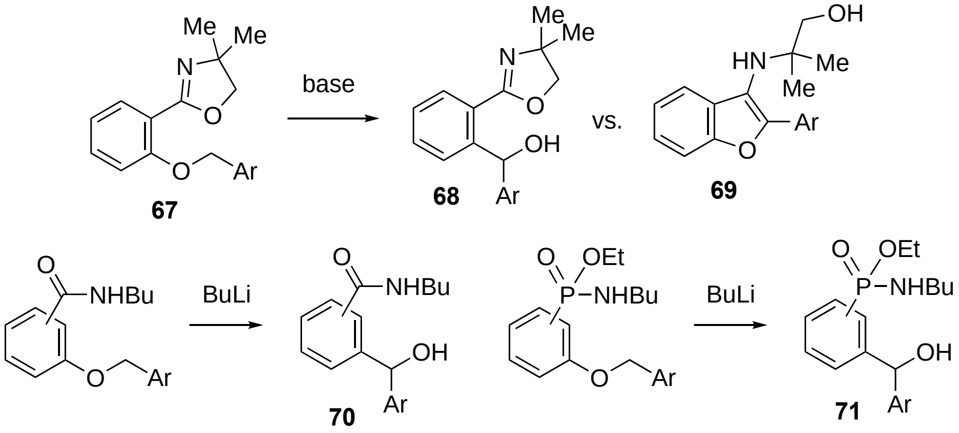
up to 76% e.e.

Another long-standing area of interest has been synthesis of dithiole and tetrathiafulvalene products by cycloaddition reactions of the crystalline adduct **60** formed between tributylphosphine and carbon disulfide. In the earliest studies, reaction with dimethyl acetylenedicarboxylate (DMAD) and analogues was found to proceed in a 1:2 ratio to afford the stable ylides **61**.[54,55] This was soon followed by the discovery that **60** also added to strained alkene double bonds such as that in norbornene. Regardless of the reacting ratio of **60** and the alkene, the product was obtained as a pink solid with the zwitterionic structure **64**. However in solution it is clearly in equilibrium with the 1:1 adduct **62** since addition of an aldehyde results in Wittig reaction to give the alkylidenedithiolanes **63**.[56]

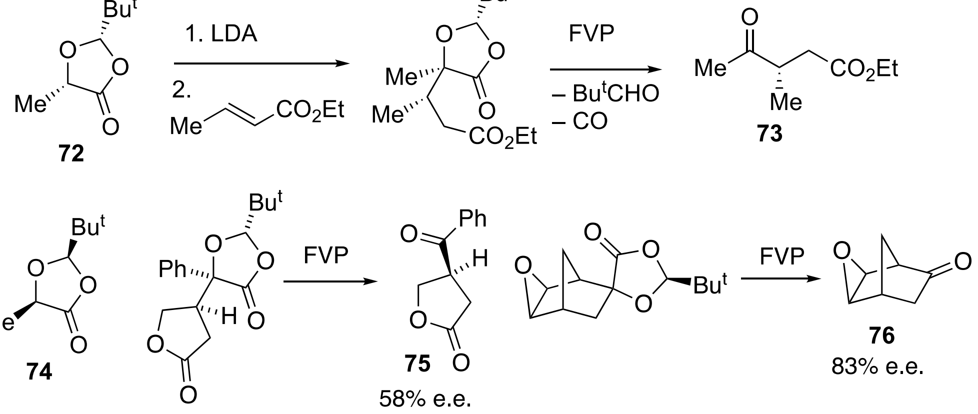


By using di- and trialdehydes this was later applied to formation of extended and polymeric dithiolane derivatives.[57] On the other hand, when the solution of **62**/**64** is treated with a dipolarophile such as DMAD, cycloaddition with **64** occurs with loss of tributylphosphine to afford the dihydrotetrathiafulvalene **65**.[58] By starting with norbornadiene, or better a norbornadiene diester (X–Y = MeO_2C–C=C–CO_2Me) the products **65** were found to undergo a retro Diels-Alder reaction upon FVP to provide a direct synthesis of tetrathiafulvalene diesters.[59] Subsequent development of this chemistry led to a range of dihydro-TTF derivatives such as **66** suitable for absorption on metal surfaces which were studied in collaboration with Prof Neville Richardson (St Andrews).[60]

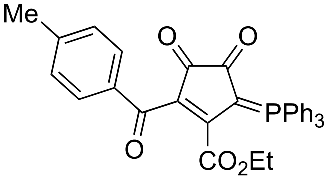
A recent major area of interest has been the development of groups to facilitate the [1,2]-Wittig rearrangement and thus render this reaction more amenable to general use. Following a chance discovery during a natural product synthesis, the well known 2-oxazoline was found to promote the rearrangement in substrates such as **67** to give **68** but there was always competition from direct cyclisation to form **69**.[61] The process could be diverted to exclusive formation of **69** by use of BuLi/KOBu^t,[61] but selectivity for **68** required a different group. In thiophene based systems also there was found to be competition between the two processes with the outcome depending on the spatial proximity of the two *ortho* groups.[62] Finally the *n*-butyl amide group was found to promote the rearrangement in moderate to high yield and was effective in *ortho*-, *meta*-, and *para*-positions giving diarylmethanol products **70**.[63] This method has since been extended to the phosphonamide directing group giving **71**,[64] and further studies on oxazoline-based systems have also been reported.[65]



Arising from a fundamental study on FVP of a-functionalised carboxylic acid derivatives, dioxolanones derived from lactic or mandelic acid were found to be effective chiral acyl anion equivalents. This is exemplified by Michael addition of dioxolanone **72** to ethyl crotonate followed by FVP to give product **73** in 86% e.e.[66] In the course of these studies, a cost-effective route to the enantiomeric dioxolanone **74** from (*R*)-alanine was also developed.[67] More recently further results including FVP of dioxolanones to give **75** and **76** involving dioxolanes acting respectively as chiral benzoyl anion and chiral ketene equivalents have been reported.[68]



Throughout his career Alan has had an interest in unexpected or surprising products, many of which have required X-ray structure determination to establish their true structure. In this he has been greatly assisted by excellent crystallographic skills of Prof Alex Slawin (St Andrews). A selection of these is shown: compounds **77**,[69] **78**,[59] **79**,[70] **80**,[71] **81**,[72] **82**,[73] **83**,[74] **84**,[75] **85** and **86**,[76] **87**,[77] **88**,[78] and **89**.[79]



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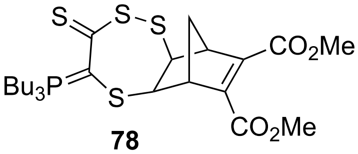

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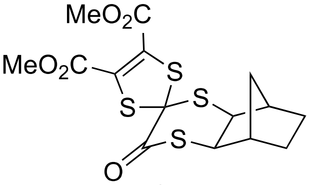

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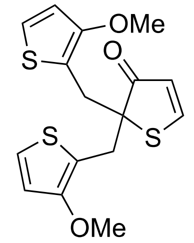

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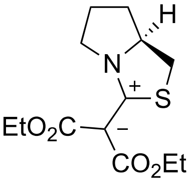

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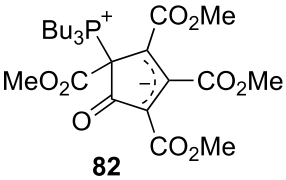
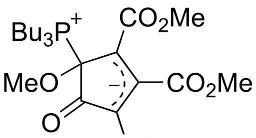
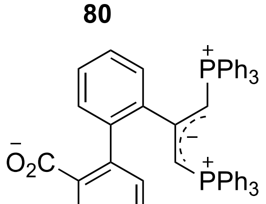


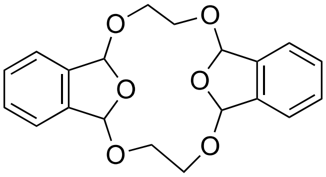
82



83



84



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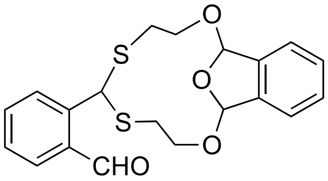
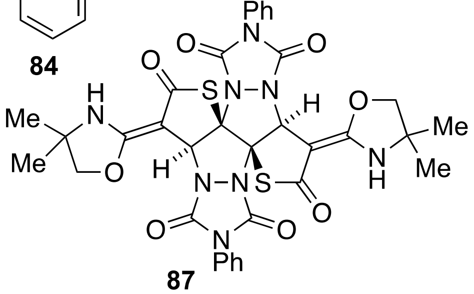
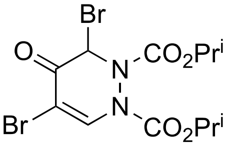


86



87



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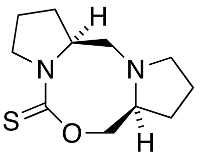

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Some of Alan's earliest publications involved collaboration with Dr Michael Palmer (University of Edinburgh) who recorded photoelectron spectra of compounds prepared during his PhD work and coupled this with a theoretical treatment.[80,81] Later this collaboration was resurrected and led to a series of publications where a variety of simple heterocyclic compounds prepared in St Andrews were subjected to detailed spectroscopic analysis and accompanying computational study: isothiazole,[82,83] 1,2,5-thiadiazole,[84] 1,2,5-selenadiazole,[85] 1,3,4-oxadiazole,[86] 1,3,4-thiadiazole,[87] 1,2,3-thiadiazole,[88] the mono *N*-oxides of pyridazine, pyrimidine and pyrazine,[89] 1,2,3-triazine,[90] and formamidinium formate.[91,92] More recently Alan has provided the synthetic component for a multi-national project on high resolution spectroscopic and theoretical studies of simple organic compounds, including 1- and 2-methyltetrazoles,[93] 1,3,5-cycloheptatriene,[94,95] norbornadiene and quadricyclane,[96,97,98] azulene,[99,100] 6,6-dimethylfulvene,[101] and 4-pyranone.[102]

During his years in St Andrews Alan has taken a keen interest in the history of chemistry there and has unearthed and preserved many historical items. A highlight was the 2014 discovery of a collection of lecture charts dating in some cases to the 1880s. One of these, a periodic table chart published in Vienna in 1885 and delivered to St Andrews in 1888, is thought to be the earliest such item still in existence,[103] and the details of its story, as well as the part that this and other charts had to play in teaching at that time, have formed the basis of two recent articles.[104,105]

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