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Chapter 1 : Progress In Heterocyclic Chemistry, Volume 10 Download

This volume of Progress in Heterocyclic Chemistry (PHC) is the fourteenth annual review of the literature, covering the work published on important heterocyclic ring systems during In this volume there are two specialized reviews.

CHMO f rom Acinetobacter sp. Although this review is limited in coverage, the potential of biocatalytic chiral heterocycle synthesis is clear. Furthermore, the development of directed evolution technologies allows rapid optimization of inefficient wild type enzymes, providing superior catalysts that are highly active, selective and robust, and that are commercially attractive alternatives to traditional chiral chemical technologies. Given the increasing access to such evolved catalysts, and the growing acceptance of biocatalysis in the synthetic community, the stage is set for continued growth in this field, with many new and exciting applications waiting to be discovered. Chem , 54, Crosby, Tetrahedron , 47, Crosby, Tetrahedron, , 47, Witholt, Chimia , 50, Holt, Chimica Oggi , 14, Swift, Macromolecules , 29, Silvani, Tetrahedron Asymmetry , 7, Pasta, Tetrahedron Asymmetry , 7, Today , 2, Furtoss, Tetrahedron , 53, Asymmtery , 8, Kiener, Tetrahedron Asymmetry , 8, Brevia, Tetrahedron Asymmetry , 8, Acta , 81, Enzym , 5, Faber, Synthesis , Fujisawa, Tetrahedron , 54, Zwanenburg, Tetrahedron Asymmetry , 9, Enzymatic , 7, 99JOC M. Shimizu, Synlett , 99T T. Hirama, Tetrahedron , 55, Wallis, Tetrahedron Asymmetry , 10, Dassar, Tetrahedron Asymmetry , 10, Patel, Tetrahedron Asymmetry , 10, Huet, Tetrahedron Asymmetry , 10, Acta , 73, Furstoss, Tetrahedron Asymmetry , 11, Liu, Tetrahedron Asymmetry , 11, Tabusa, Heterocycles , 54, Vogel, Synthesis , 6, Furstoss, Tetrahedron , 57, Faber, Tetrahedron Asymmetry , Faber, Tetrahedron Asymmetry , 13, Suemune, Tetrahedron Asymmetry , 12, Liu, C-H Wong, Angew. Tabusa, Heterocycles , 58, Stanetty, Synlett , Furstoss, Tetrahedron Asymmetry , 13, Guisan, Tetrahedron Asymmetry , 13, Fernandez-Lafuente, , Tetrahedron Asymmetry , 13, Faber, Tetrahedron Asymmetry, , 12,

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Chapter 2 : Progress in Heterocyclic Chemistry: Volume 29 : Gordon W. Gribble :

This volume of Progress in Heterocyclic Chemistry (PHC) is the fourteenth annual review of the literature, covering the work published on important heterocyclic ring systems during

No part of this publication may be reproduced, stored in a retrieval system or transmitted in any form or by any means: Sikorski, Monsanto Company, St. Three-Membered Ring Systems s. Shaun Murphree, Bayer Inc. Four-Membered Ring Systems J. Five-Membered Ring Systems Part 1. Press, Galencia Pharmaceuticals, Inc. Pyrroles and Benzo Derivatives Daniel M. Bradley and David J. Andrews, UK Part 7: Six-Membered Ring Systems Part 1: Pyridine and Benzo Derivatives Daniel L. Diazines and Benzo Derivatives Michael P. Seven-Membered Ring s David J. The first two chapters are review articles. Chapter 1 by C. Sikorski provides a detailed account of the heterocyclic chemistry surrounding the remarkable herbicide glyphosate "Roundup". This latter chapter illustrates the role that heterocyclic chemistry plays in other areas of modern chemistry, since glyphosate is a far cry from being heterocyclic! The remaining chapters deal with recent advances in the field of heterocyclic chemistry arranged by increasing ring size. We thank all authors for providing camera-ready scripts and disks, and most especially for adopting our new uniform format. In this regard, we welcome comments from readers about the style, presentation, and coverage. We are much indebted to David Claridge of Elsevier Science for his invaluable help with the presentation of Chapters and with his input on the new format. Finally, we wish to acknowledge retiring editor Hans Suschitzky not only for his outstanding contributions in all previous volumes of this series as co-editor, but, jointly with Eric Scriven, for launching the series. Heterocyclic chemists owe Hans and Eric a debt of gratitude. Once again, we hope that our readers will find PHC-9 to be a useful and efficient guide to the field of modern heterocyclic chemistry. Access can be gained from the following locations: This work, summarised in the classic treatise in , is the foundation of modern oxazole chemistry. More recently the oxazole ring system has been found in an ever increasing range of natural products, many of them "peptide alkaloids" in which the heterocyclic ring is most likely formed by a modification of a serine or threonine containing peptide. The interesting biological activity associated with these natural products has not surprisingly prompted renewed interest in the synthesis of oxazoles. Other aspects of diazocarbonyl chemistry have been widely reviewed. He and co-workers found that the ketocarbene derived from diazoacetophenone la by thermolysis at C underwent formal cycloaddition with benzonitrile giving a 0. The presence of electron withdrawing groups at the 2-position on the aromatic ring resulted in the formation of the oxazoles 2b and 2c in higher yield. The yield of oxazole 2a was higher when the reaction was carried out in the presence of Cu acac 2. One is the 4-sulfonyloxazole 7 whereas the other product 8 results from rearrangement and reaction with the alcohol. The ratio of products varies with the nature of the sulfone substituent with the benzyl group giving highest yields of oxazole Scheme 5. Although the reaction does not involve addition to a nitrile, it is an interesting application of a diazo compound since the proposed zwitterionic intermediate 10 is a resonance form of a diazo imine, so formally the reaction may be thought of as a thermal decomposition of a diazo imine Scheme 6. N2 N 9 10 11 Scheme 6 The photochemical decomposition of ethyl diazoacetate, methyl diazoacetate and diazoacetophenones 1 in benzonitrile has been studied by Huisgen and Komendantov. The photochemical decomposition of diazoacetophenone la gave the oxazole 2a in extremely low yield. The reaction has been exploited as a general approach for the preparation of 2-perfluoroalkylalanines The oxazole ring is formed from the photolysis of the appropriate perfluoroacyl diazo esters in acetonitrile, and is then degraded under acid hydrogenolysis conditions to give the N-acetyl esters, which are then hydrolysed to the racemic 2-perfluoroalkylalanines 16 Scheme 8. In the thermal and photochemical decomposition of methyl diazoacetate in benzonitfile, the 2H- azirine 5 was formed along with the oxazole 4. However, when the photolysis was conducted in a It was assumed that the formation of the 2H-azirine 5 and oxazole 4 was due to the reaction of methoxycarbonylcarbene in either its singlet or triplet state. The workers assumed that decomposition of the excited a2-singlet state led to the formation of the

2H-azirine, whilst the ground triplet state gave the oxazole. They rationalised the observed product ratio as being due to the presence of the inert solvent, hexafluorobenzene, and assumed it caused enhancement of the singlet-triplet transition, leading to more oxazole formation. These investigators found that only singlet ethoxycarbonylcarbene reacts with nitriles to yield oxazoles. Upon benzophen0ne sensitisation of the reaction mixture, no oxazole formation takes place; instead the triplet carbene reacts with benzophenone to give the diradical, which adds to acetonitrile yielding ethyl 5,5-diphenylmethyl-4,5-dihydro-oxazolecarboxylate. Despite the above, there is also considerable evidence to suggest that oxazole formation proceeds via an intermediate nitrile ylide, particularly in the catalysed reactions see below. Nitrile ylides have been detected in laser flash photolysis studies of diazo compounds in the presence of nitriles, and stable nitrile ylides can be isolated in some cases. Although 2-acyl-2H-azirines are known to give oxazoles upon irradiation, the reaction is wavelength dependent, and isoxazoles are formed at some wavelengths, as they are in the thermal rearrangement of 2-acyl-2H-azirines. Since the thermal reaction of diazocarbonyl compounds with nitriles leads to oxazole formation, it would seem that mechanistic path C is unlikely in these reactions. In a more detailed study, a range of Lewis acids was screened for catalytic activity, using diazoacetophenone l a and acetonitrile as the test reaction. Unfortunately, it was found that in the case of boron trifluoride etherate, the nitrile had to be used in a ten-fold excess, however the use of antimony V fluoride allowed the use of the nitrile in only a three fold excess Table 1. They found that not only diazoketones, as reported by Doyle, but also diazoketoesters could be decomposed in the presence of nitriles to give oxazoles Table 2. They also studied the range of nitriles that could be employed, finding that substituted thiocyanates and cyanamides, along with chloroacetonitrile also participate in the reaction Table 2. Holt and co-workers found that diazoacetophenone l a in the presence of trifluoromethanesulfonic acid and acetonitrile gave 2-methylphenyl oxazole It was assumed that protonation of the diazo compound occurred to give a diazonium ion which underwent nucleophilic attack by acetonitrile to give a nitrilium ion which subsequently cyclised. On the other hand, two mechanisms for the Lewis acid mediated process have. Ibata favours initial attack by the Lewis acid on the diazocarbonyl oxygen to give a diazonium betaine which suffers nucleophilic attack by the nitrile to give, with loss of nitrogen, a nitrilium betaine which subsequently cyclises Scheme Synthesis of Oxazoles from Diazocarbonyl Compounds Doyle however favours a mechanism involving the initial formation of a Lewis acid-nitrile adduct which suffers nucleophilic attack by the diazocarbonyl oxygen to give a 2-imidatoalkenediazonium salt, which cyclises, with extrusion of nitrogen gas, to the oxazole Scheme Doyle has successfully employed the reaction in the synthesis of annuloline 20, a disubstituted oxazole isolated from the roots of the annual rye grass. Et20 u - "CH2Br 2. Their studies pointed towards the copper undergoing two key changes in the catalytic process, the first being a reduction under the reaction conditions of copper II to copper l , and the second being a change of ligands around the copper I. The reactants, the nitrile and diazo compound, were found to play an important role in the formation of the most effective catalytic species. The reaction with acetonitrile was studied with a range of other metal chlorides, but all proved less satisfactory than WC Subsequently Teyssi6 et al. The authors claim that these observations fit with an oxazole formation mechanism involving the decomposition of the diazoester via co-ordination with the palladium. They assumed the nitrile formed an active complex with the catalyst, which decomposed the diazo compound. Much of the early work into the rhodium II -catalysed formation of oxazoles from diazocarbonyl compounds was pioneered by the group of Helquist. They first reported, in , the rhodium II acetate catalysed reaction of dimethyl diazomalonate with nitriles. A range of nitriles was screened, including aromatic, alkyl and vinyl derivatives; with unsaturated nitriles, cyclopropanation was found to be a competing reaction Table 3. Synthesis of Oxazoles from Diazocarbonyl Compounds N2. The oxazole 28 obtained from bromoacetonitrile was found to give a heteroaromatic benzylic organozinc derivative which underwent reaction with aldehydes and ketones leading to a range of alcohols 29 in good yield Scheme Utilising this reaction has led Helquist et al. The ester was then reduced to the alcohol, with simultaneous removal of the 5-methoxy group, which was then transformed to the nitrile 34 via the aldehyde and oxime. Shi and Xu have shown that ethyl 3,3,3-trifluoro

diazopropionate 37 will undergo oxazole formation in the presence of rhodium II acetate and a range of nitriles Scheme Likewise the rhodium II perfluorobutyrate catalysed reaction of the silyl diazo compound 38 with methyl cyanoformate in the presence of benzaldehyde gave the corresponding oxazole; no products resulting from carbonyl ylide formation with the aldehyde were isolated. In the rhodium II acetate catalysed decomposition of 2-diazo-l,3-cyclohexanedione in the presence of dihydrofuran and acetonitrile, two products were formed; the bis-tetrahydrofuran 39 and the oxazole 40 Scheme Initially due to its Lewis acidity it reversibly forms a complex with the nitrile; nitriles are known to complex to the free axial coordination sites in rhodium II carboxylates as evidenced by the change of colour upon addition of a nitrile to a solution of rhodium II acetate, and by X-ray crystallography. Secondly the metal catalyses the decomposition of the diazocarbonyl compound to give a transient metallocarbene which reacts with the nitrile to give a nitrile ylide intermediate. Whether the nitrile ylide is metal bound or not is unclear. Whatever the exact mechanism, the rhodium II catalysed reaction of diazocarbonyl compounds with nitriles is a useful route to oxazoles. A further example from our own laboratory illustrates the use of the reaction in the synthesis of the oxazolylindole alkaloids pimprinine 43a, pimprinethine 43b, and WSA 43c. Diazoacetylindole 42 reacted with simple nitriles in the presence of rhodium II trifluoroacetamide to give the corresponding oxazoles, deprotection of which gave the natural products 43 Scheme

Chapter 3 : Progress in Heterocyclic Chemistry: Volume 17 : Gordon W. Gribble :

Progress in Heterocyclic Chemistry (PHC), Volume 30, the latest in this annual review series commissioned by the International Society of Heterocyclic Chemistry (ISHC), contains both highlights of the previous year's literature on heterocyclic chemistry and articles on new and developing topics of interest.

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Chapter 7 : progress_in_heterocyclic_chemistry_volume_14

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Chapter 8 : T.L. Gilchrist | Open Library

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This volume of Progress in Heterocyclic Chemistry (PHC) is the eleventh annual review of the literature, covering the work published on most of the important heterocyclic ring systems during , with inclusions of earlier materials as appropriate.