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RECENT PROGRESS IN THE SYNTHESIS AND REACTIONS OF TETRAZINES. A BRIEF REVIEW

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REACTIONS OF TETRAZINES. A BRIEF REVIEW

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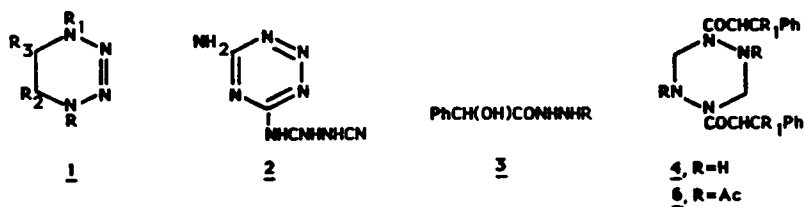
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INTRODUCTION

The tetrazines comprise a family of compounds of outstanding synthetic interest. They present significant challenges to chemists who would study the preparation of their novel and useful analogs, as well as to those intrigued by their unusual structures. This review covers the recent literature of the tetrazines, inclusive of Volume 82 of Chemical Abstracts through the first part of Volume 104, and emphasizes the newer aspects of synthesis. We have attempted to make complete coverage of all the literature references and thus to bring up to date the chemistry of the tetrazines since the time of the last review of encompassing scope.^{1a} Excellent critical interpretations of selected fundamental advances in tetrazine chemistry are also available.^{1b}

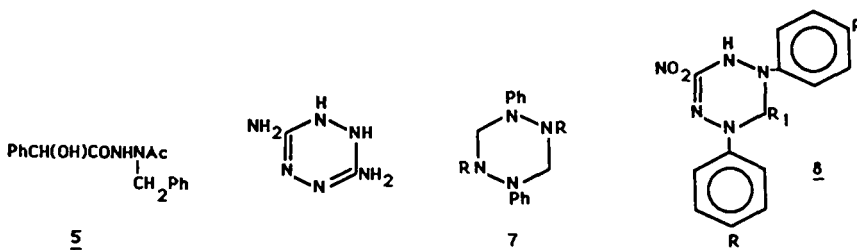
I. SYNTHESIS

Although preparations have been reported of such 1,2,3,4-tetrazines as



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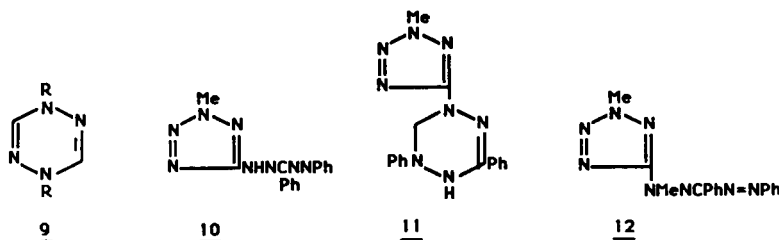
1c,d-3 and such 1,2,3,5-tetrazines as 2⁴⁻⁷ much greater effort has been directed at the synthesis of the 1,2,4,5-compounds,⁸⁻³⁸ spurred on in considerable measure by the observation of their herbicidal and physiological activities.³⁹⁻⁴¹ Acid hydrazides have been especially useful in cyclization approaches.⁴²⁻⁴⁶ Thus Kametani and coworkers discovered that acid-catalyzed cyclization of N'-substituted mandelohydrazides 3 with paraformaldehyde gave hexahydro-1,2,4,5-tetrazines 4, whereas the diacylhydrazine 5 produced 6 when similarly treated. A cyclization-rearrangement



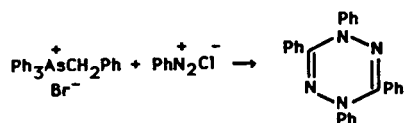
process was proposed.⁴⁷⁻⁴⁸ Alkaline hydrolysis of thiosemicarbazide is noted as leading to a diaminotetrazine, although its oxidation with lead oxide gave the N-aminotriazole.⁴⁹⁻⁵¹

Lamberton and Nelson noted that a mixture of phenylhydrazine, morpholine and formaldehyde produced the tetrazine 7;⁵² although a somewhat more conventional approach to cyclocondensation was taken by Dychenko and Pel'kis, in which isolated phenylhydrazones of nitroformaldehyde were heated with aldehydes in ethanol to allow formation of the compounds 8.⁵³ The reaction with hydrazine of several benzonitriles in which the aromatic ring was substituted with electron-donating groups, as well as of the parent benzonitrile, led directly to 3,6-bis-substituted-1,2,4,5-tetrazines.⁵⁴ The reagent N,N-dimethylamide-phosphoryl chloride reacts with substituted hydrazines in benzene to produce dihydrotetrazines 9, but the method is somewhat limited by low yields and the formation of mixtures.⁵⁵

Cyclization of tetrazolylformazan 10 with formaldehyde gave 11, subsequently oxidized in a procedure which is typical with lead oxide, to the

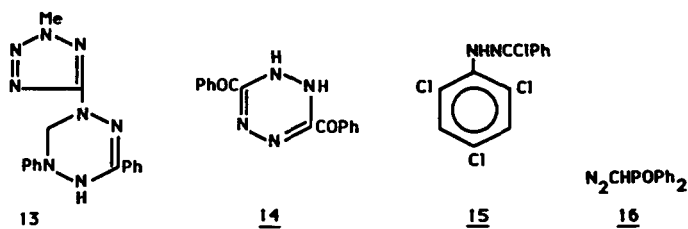


corresponding verdazyl.⁵⁶ Phenacyl- and benzyltriphenylarsonium bromides react with aromatic diazonium salts to produce nitrilimines, which



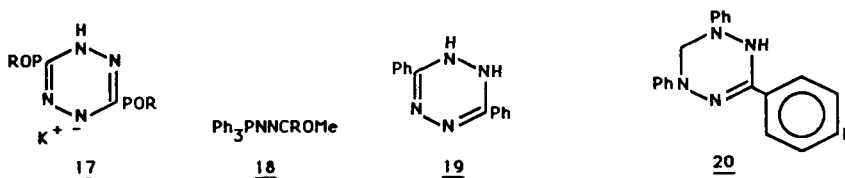
cyclize spontaneously under the reaction conditions, to give the corresponding 1,2-dihydro-1,2,4,5-tetrazines.⁵⁷ Several other variations on the basic parameters of the cyclocondensation procedure have been attempted.⁵⁸⁻⁷⁷

A number of novel rearrangements produce tetrazines. Jensen and Larson observed the rapid rearrangement at room temperature of thioacetylhydrazide to 1,4-dihydro-3,6-dimethyl-1,2,4,5-tetrazine via elimination of hydrogen sulfide.⁷⁸ Interestingly, dye laser excitation methods³⁵⁶ have allowed the production of useful quantities of pure sym-tetrazine-¹³C and sym-tetrazine-¹⁵N.⁷⁹ Thermal rearrangement at 100° converted the tetra-



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zole 12 to the biheterocyclyl 13.⁸⁰ The reaction of α -diazacetophenone with methanolic sodium methoxide produced the dihydrotetrazine 14, among several products.⁸¹ Tetrazines have also been isolated from the treatment of 2-aminophthalazinium chloride with sodium hydroxide,⁸² from the solid



phase photolysis of 15,⁸³ from the dimerization of 16 in base to 17⁸⁴ and from the hydrolysis rearrangement of 18 to 19.⁸⁵ Recent mechanistic studies have now helped to clarify certain reaction pathways generally useful in tetrazine synthesis.⁸⁶⁻⁸⁸ An elegant total synthesis of streptonigrin employs an important tetrazinecarboxylate intermediate.⁸⁹

II. OXIDATION AND REDUCTION

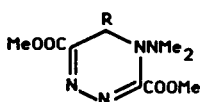
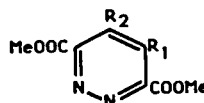
Not much work has been done in a systematic way to investigate the oxidation and reduction reactions of the tetrazines.³⁶⁶ sym-1,2,3,4-Tetrahydrotetrazines were prepared by the hydrogenation of the corresponding dihydro compounds with hydrazine.⁹⁰ Substituted dihydrotetrazines can be electrochemically oxidized to give stable radical cations.⁹¹ Although several other oxidative procedures have been recorded,⁹²⁻⁹⁶ it is fair to say that the majority of articles in this vein have had the specific goal of generating verdazyls, as opposed to the conversion of one stable tetrazine compound at a given level of saturation to another stable tetrazine.

III. VERDAZYL RADICALS

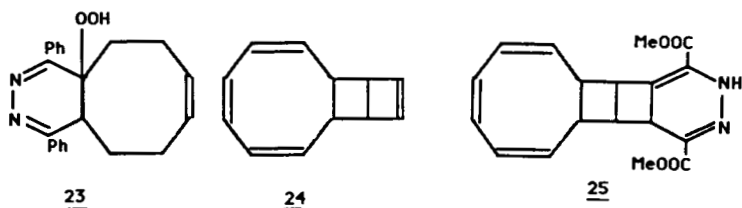
Clear-cut approaches to the preparation and reactions of verdazyls have emerged, typically involving the oxidation of tetrahydrotetrazines by potassium hexacyanoferrate,⁹⁷ lead oxide,⁹⁸ or a combination of oxidizing agents.^{99,111} In turn, the colorful verdazyls are readily reduced under mild conditions back to their colorless precursors. Compounds 20 were prepared in moderate to good yield by reduction of the corresponding radicals with phenylhydrazine in benzene.¹⁰⁰ Other choices of reducing agent have been hydrazine¹⁰¹ and hydrazobenzene.¹⁰² The verdazyls undergo bimolecular homolytic substitution with Grignard reagents to produce the N-alkylated tetrahydrotetrazines.¹⁰³⁻¹⁰⁵ Strengths for the N-H bond have been determined in certain of these systems¹¹² and promise to shed further light on the requirements for the homolysis reaction itself. A number of interesting new perfluorophenyl verdazyls have now been prepared.¹¹³ Several other applications of verdazyls to problems in synthesis have been made,¹⁰⁶⁻¹⁰⁹ and a review of recent advances in the chemistry of verdazyl radicals has appeared.¹¹⁰

IV. REARRANGEMENTS

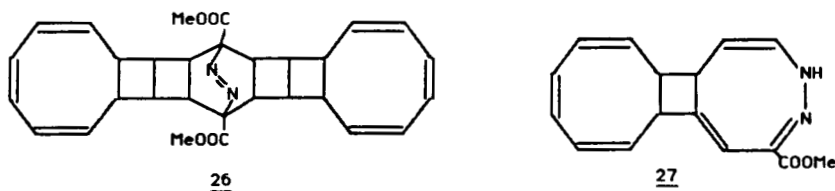
The rearrangement reactions of tetrazines have been particularly useful in heterocyclic synthesis. A recent report includes the details for the thermal conversion of s-tetrazines to 1,2,4-triazoles.¹²⁹ A number of workers have made a thorough experimental investigation of the Diels-Alder inverse electron demand cycloaddition reactions of tetrazines.¹¹⁴⁻¹¹⁹

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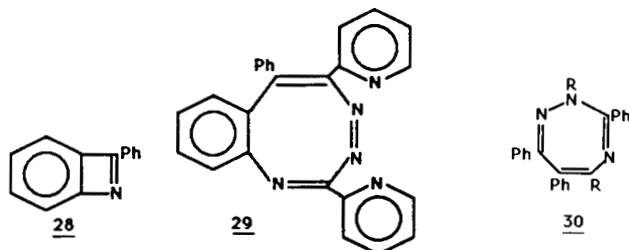
Thus 3,6-dimethoxycarbonyl-1,2,4,5-tetrazine reacted with aldehyde dimethylhydrazones to produce triazines 21, whereas the related ketone hydrazones yielded the pyridazines 22, quite possibly because they reacted in the enehydrazine form;¹²⁰ and the cyclization of nontautomerizing anti- α -aminohydrazones into N-aminoimidazolidines has been carried out.¹³⁰ Haddadin, Firsan and Nader noted that a variety of enolate ions condensed with 3,6-diphenyl-1,2,4,5-tetrazine to give the 3,6-diphenyl-



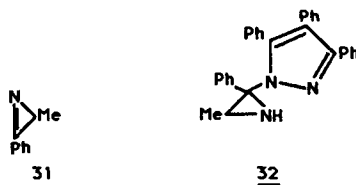
4,5-disubstituted pyridazines.¹²¹ In the reaction of 3,6-diphenyl-sym-tetrazine with *cis,cis*-1,5-cyclooctadiene, the distribution of products was found to be highly dependent on reaction conditions,¹²² with the chief



product being the cyclooctapyridazine 23 under mild treatment. Tetrazines react readily with strained small ring compounds to provide a rich complement of novel heterocyclics.¹⁶⁰ The strained double bond of 24, for example, reacted with 3,6-dimethoxycarbonyl-sym-tetrazine to bring forth



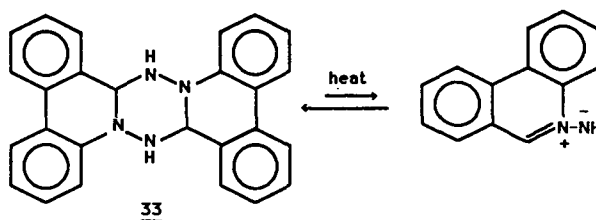
25-27.¹²³ Adducts of 28 with dipyridyltetrazine were found to lose carbon monoxide and nitrogen spontaneously to give the benzotriazocine 29.¹²⁴ A simple and very effective method for the preparation of 1,2-diazocines has



also been advocated.¹⁶⁸ Compound 30 was prepared by cycloaddition of 2-phenyl-1-azirine to 3,6-diphenyl-sym-tetrazine.¹²⁵ Similarly, the latter compound also condensed with 31 to give 32.¹²⁶ Small ring oxacycles have been examined in this context as well.^{127,128}

V. CYCLOADDITIONS

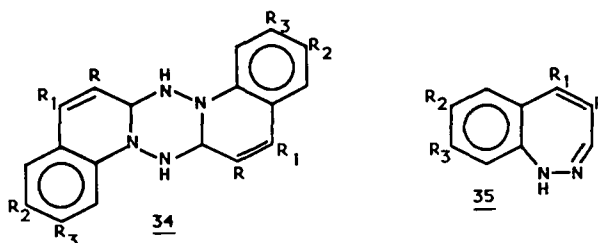
Fused ring derivative 33 in solution behaved as a 1,3-dipole, giving



cycloaddition products with α,β -unsaturated carbonyl compounds.¹³¹ In related examples, the irradiation of substituted dimers 34 in methylene chloride containing acetic acid gave the 1H-1,2-benzodiazepines 35 in moderate yields.¹³²⁻¹³⁵ Some work has been done on substituent effects on the reaction rate of 3,6-diaryltetrazines with styrene.³⁶⁵ Tetrazines have been employed as cycloaddition reactants in probing the behavior of val-

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enes^{136,145,170} and fulvenes^{141,144} and several other highly reactive hydrocarbon derivatives,^{137,138,140,149,150,166,168,171} Indoles,¹³⁹ oxacycles,^{142,158,167} 2-phenylazirines¹⁴³ and several azacycles^{146,147,157,164} have also been investigated, as well as arsabenzene,¹⁴⁸ thioformamides,¹⁵¹ isocyanides,¹⁵² hydrazones,^{154,162} imines,¹⁵⁵ silylacetylenes,¹⁵⁹ diketene,¹⁶¹ N,N-dimethylaniline,¹⁶³ tricarbonyliron complexes,¹⁶⁵ and arylthioimidates.¹⁶⁹ Some sulfur-containing



rings have been examined in this context.^{153,156}

VI. PHYSICAL ORGANIC METHODS AND APPLICATIONS

NMR spectroscopy has found widespread application to the chemistry of the tetrazines.^{143,172-175} The earlier extensive studies of the NMR spectra of verdazyl radicals having aliphatic substituents¹⁷⁶ have been followed up in experiments providing new data bearing on the long range ordering of triphenylverdazyl.¹⁷⁷⁻¹⁷⁹ Intra- and intermolecular exchange rates¹⁸⁰ and conformational equilibria¹⁸¹⁻¹⁸² have been examined for the sym-hexahydrotetrazines as well. The conformations of bi- and tricyclic hexahydrotetrazines have been identified on the basis of their photoelectron spectra,¹⁸³ and the analysis of several other problems has been made possible through this method.¹⁸⁴⁻¹⁸⁹ Some mass spectrometric studies have been made on the tetrazines,^{362,364} although this specific area has not seen a great deal of activity. The single-electron conversion of the

1,3,5-triphenylverdazyl radical into the corresponding cation has been studied by infrared spectroscopy.¹⁹⁰⁻¹⁹² The assignments have been made of visible absorptions of sym-tetrazine,^{193,350} and geometrical changes have been correlated with their related absorptions of ultraviolet radiation.¹⁹⁴⁻²⁰² The radiationless decay of several azabenzenes has been studied by means of opto-acoustic spectroscopy.²⁰³ The fluorescence excitation spectrum of sym-tetrazine at low temperature reveals a smaller geometrical change on electronic excitation than recorded from the analysis of room temperature optical spectra.^{204-209,355} Electron-spin resonance methods have allowed determination of nitrogen hyperfine coupling constants,²¹⁰ N-H bond strength in leucoverdazyl,²¹¹ conformational preferences,²¹² characteristics of liquid crystals,²¹³⁻²¹⁵ and several other properties of the verdazyls.²¹⁶⁻²¹⁹ The molecular symmetry of the tetrazines is so low that the magnetic circular dichroism spectra contain only the Faraday parameter β ,²²⁰ dependent to a significant extent on the substituents present. The spectroscopy, photophysics and photochemistry of tetrazines in crystals and mixed crystals at low temperatures have been examined.²²¹⁻²²⁵ Tunable dye laser technology has now made possible the determination of individual molecular isotopes in an unseparated natural mixture.²²⁶⁻²²⁸

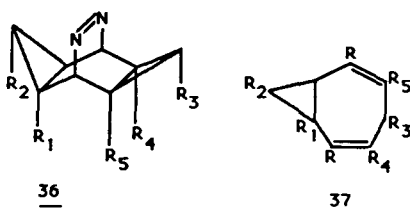
Advances in the area of theory of structure³⁵⁴ and bonding³⁵⁹ in the tetrazines include a process for obtaining the diamagnetic anisotropy relative to that of benzene,²²⁹ calculated bond orders³⁶³ and bond lengths²³⁰ and improvements in calculations involving simple ab initio and semiempirical approaches.²³¹⁻²³⁵

The photodecomposition of tetrazines has been investigated;²³⁶⁻²⁴⁰ although much remains to be worked out in terms of the basic photochemical

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and photophysical effects,^{241-246,353,356,359} it now seems clear that the parent compound decomposes with laser light to yield one nitrogen molecule and two molecules of hydrogen cyanide. In spite of the fact that the Diels-Alder adducts of tetrazines to substituted olefins are modestly stable in a thermal sense,²⁴⁷⁻²⁵² some are photolytically labile. Thus the photolysis of 36 led either to homotropilidenes 37 or to the related tetracyclooctanes, depending on the substituents present.²⁵³

Stable verdazyl radicals have been employed in a variety of roles in several kinds of free radical processes: as hydrogen abstraction agents,²⁵⁴⁻



²⁶⁵ as quenchers of peroxides,²⁶⁶⁻²⁸⁰ as self-regulating antioxidants for organic materials,²⁸¹ and as substrates for oxidation-reduction processes.²⁸²⁻²⁹⁹ Thermochromism of bis-verdazyls has been studied.³⁰⁰ It is interesting to note that crystal structures have been determined not only for various substituted tetrazines³⁰¹⁻³⁰⁴ but for stable verdazyls as well.³⁰⁵⁻³⁰⁷

In agricultural applications, tetrazines have been found useful as bactericidal and fungicidal materials,^{308,309} as well as herbicides,^{310,311,361} and pesticides.³⁵¹ Their toxicology has been discussed.³¹²⁻³¹⁵ In photographic science,^{352,357} tetrazines are viable fogging agents for direct positive color diffusion transfer film.³¹⁶⁻³¹⁸ sym-Tetrazine has been examined as a candidate material for an organic photovoltaic system.³⁵⁸ Part of the impetus for the investigation of the isotopically selective processes induced by laser excitation was a desire for a better under-

standing of tetrazine photochemistry in the solid state.³¹⁹⁻³²⁷ 3,6-bis(4-n-Nonylphenyl)-1,2,4,5-tetrazine has been examined in relation to switching on and switching off time in digital display devices.³²⁸ In the field of synthetic high polymers,³⁶⁷ the stable triphenylverdazyl has been suggested as an inhibitor of ethyl acrylate with styrene,³²⁹ and in other like processes.³³⁰⁻³⁴⁵ Trisazo dyes containing a diphenyltetrazinyl moiety were even found efficacious in dyeing cotton, silk and wool bluish black shades³⁴⁶⁻³⁴⁹ and tetrazines have been used in water purification,³⁶⁹ as modifiers of solid propellant burning rates³⁷⁰ and in pulp processing.³⁶⁸ With so many useful applications, further rapid growth in tetrazine synthesis can be anticipated.

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