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of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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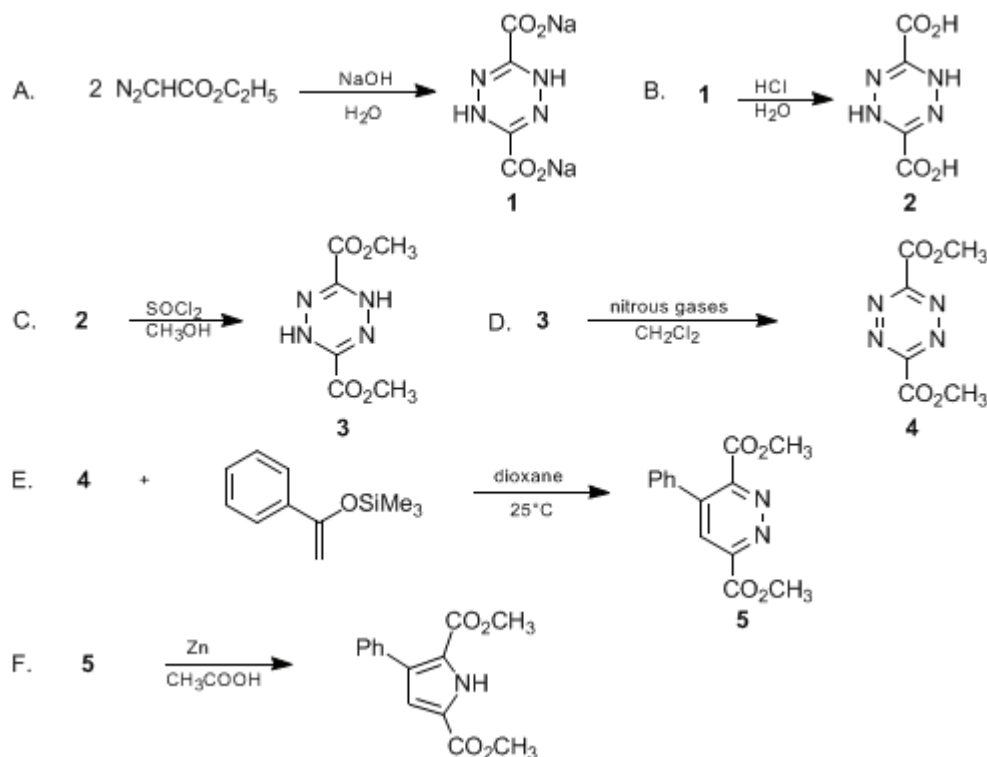
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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**PREPARATION AND DIELS-ALDER REACTION OF A
REACTIVE, ELECTRON-DEFICIENT HETEROCYCLIC
AZADIENE: DIMETHYL 1,2,4,5-TETRAZINE-3,6-
DICARBOXYLATE. 1,2-DIAZINE (DIMETHYL 4-PHENYL-1,2-
DIAZINE-3,6-DICARBOXYLATE) AND PYRROLE (DIMETHYL 3-
PHENYLPYRROLE-2,5-DICARBOXYLATE) INTRODUCTION**

[1,2,4,5-Tetrazine-3,6-dicarboxylic acid, dimethyl ester; 3,6-Pyridazinedicarboxylic acid, 4-phenyl-, dimethyl ester; 1H-Pyrrole-2,5-dicarboxylic acid, 3-phenyl-, dimethyl ester]



Submitted by Dale L. Boger, James S. Panek, and Mona Patel¹.

Checked by Richard Hutchings and Albert I. Meyers.

1. Procedure

2 3 4

A. *Disodium dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate.* A 2-L, three-necked, round-bottomed flask is equipped with an overhead stirrer, thermometer, and a 500-mL addition funnel. Sodium hydroxide (320 g, 8 mol) and 500 mL of water are added. Ethyl diazoacetate (200 g, 1.75 mol, (Note 1)) is placed in the addition funnel and added dropwise to the stirred sodium hydroxide solution so as to maintain the temperature of the reaction mixture between 60°C and 80°C (approximately 1.5 hr, (Note 2)). After the reaction slurry is cooled to room temperature, it is poured into 2 L of 95% ethanol, mixed well, and the liquid is decanted. This washing procedure is repeated five times using 1.5 L of 95% ethanol each time. The precipitate is collected by filtration using a Büchner funnel, the collected solid is washed with 1 L of absolute ethanol and 1 L of ether, and dried (12 hr) in the air to afford 160–184 g (85–97%) of disodium dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate as yellow brown solid.

B. *Dihydro-1,2,4,5-tetrazine-3,6-dicarboxylic acid*. A 2-L, three-necked, round-bottomed flask is equipped with an overhead stirrer and a 100-mL addition funnel. Disodium dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate (90.37 g 0.42 mol) in 100 mL of water and 100 g of crushed ice are added. The resulting slurry is cooled with an ice/sodium chloride bath and a solution of concentrated hydrochloric acid (84 mL of 36–38%) is added dropwise with stirring over 45 min. The reaction mixture is washed five times with 200 mL of dry ether and the ether layer is decanted. The product is immediately collected by filtration using a Büchner funnel. Drying the collected solid at room temperature under reduced pressure affords 51.6–53.06 g (72–74%) of dihydro-1,2,4,5-tetrazine-3,6-dicarboxylic acid as a yellow powder: mp 144–148°C (Note 3).

C. *Dimethyl dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate*. Absolute methanol (700 mL, (Note 4)) is placed in a dry, 2-L, round-bottomed flask fitted with an overhead stirrer, thermometer, and a 100-mL addition funnel and is cooled to –30°C. Thionyl chloride (62.12 g, 0.522 mol, 38.1 mL, (Note 5)) is added carefully with stirring and the reaction mixture is allowed to stir at –30°C for 30 min. Dihydro-1,2,4,5-tetrazine-3,6-dicarboxylic acid (45.00 g, 0.261 mol) is added as a solid in portions over 30 min to the cooled, stirred thionyl chloride-methanol solution (Note 6). The reaction mixture is allowed to warm to room temperature (1 hr) and is subsequently warmed to 35–40°C (internal temperature) for 2 hr. The reaction mixture is cooled to –30°C and the precipitate is collected by filtration using a Büchner funnel. The collected solid is washed with ether (115 mL, (Note 7)) and the filtrate is concentrated under reduced pressure to give an orange-brown oil (ca. 15 g). The collected solid is triturated with methylene chloride (2.0 L) and the insoluble inorganic salts are removed by filtration using a Büchner funnel. The orange-brown oil (ca. 15 g) is taken up in water (150 mL) and extracted with methylene chloride (6 × 270 mL). The combined methylene chloride extracts and methylene chloride triturate are dried over anhydrous sodium sulfate and concentrated under reduced pressure to afford 23–31 g (44–51%) of pure dimethyl dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate as an orange-yellow powder: mp 171–172°C (Note 8).

D. *Dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate*. Dimethyl dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate (20 g, 0.1 mol) is slurried in 800 mL of methylene chloride (Note 9) in a 2-L, round-bottomed flask fitted with a magnetic stirring bar and the mixture is cooled with an ice/water bath. A stream of nitrous gases (Note 10) is bubbled into the reaction mixture with stirring for 15 min. The color of the reaction mixture changes from orange to bright red during the addition. Stirring is continued for 1.5 hr as the reaction mixture is allowed to warm to room temperature. The solvent and excess nitrous gases are removed under reduced pressure to afford dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (19.7 g, 99%) as a bright red, crystalline solid: mp 173–175°C (Note 11).

E. *Dimethyl 4-phenyl-1,2-diazine-3,6-dicarboxylate*. A 50-mL, round-bottomed flask equipped with a magnetic stirring bar is charged with dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (1.0 g, 0.005 mol) and 1,4-dioxane (20 mL, (Note 12)). 1-Phenyl-1-(trimethylsiloxy)ethylene (1.07 g, 0.0056 mol, 1.14 mL, (Note 13)) is added and the reaction mixture is stirred under nitrogen at room temperature for 8 hr. The solvent is removed under reduced pressure to give a viscous oil that is triturated with anhydrous ether (2–3 mL). The solid product is collected by vacuum filtration and recrystallized from ethyl acetate/hexane to give 1.23–1.30 g (90–96%) of dimethyl 4-phenyl-1,2-diazine-3,6-dicarboxylate as a light yellow solid, mp 95.5–96°C (Note 14).

F. *Dimethyl 3-phenylpyrrole-2,5-dicarboxylate*. A 250-mL, round-bottomed flask equipped with a magnetic stirring bar is charged with dimethyl 4-phenyl-1,2-diazine-3,6-dicarboxylate (1.36 g, 0.005 mol) and glacial acetic acid (55 mL, (Note 15)). Zinc dust (3.25 g, 0.05 mol, (Note 16)) is added and the reaction mixture is stirred at room temperature for 6 hr. A second portion of zinc dust (3.25 g, 0.05 mol) is added and the reaction mixture is stirred for an additional 18 hr. The zinc dust is removed by filtration through a pad of Celite and the residue is washed with ether (100 mL). The filtrate and washes are combined, made basic (pH 10) with the addition of saturated sodium bicarbonate, and extracted with ether (2 × 100 mL). The combined ether extracts are dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Purification of the product is effected by flash chromatography on a 4.5 × 9-cm column of silica gel (Aldrich 951, CH₂Cl₂ eluant), collecting 20-mL fractions. The fractions are analyzed by thin-layer chromatography (Kieselgel 60, CH₂Cl₂ eluant) and those containing product are combined and concentrated in vacuo to give 0.676 g (52%) of dimethyl 3-phenylpyrrole-

2,5-dicarboxylate as a white solid: mp 122–123°C (ethyl acetate-hexane, (Note 17)).

2. Notes

1. The submitters employed, without purification, ethyl diazoacetate obtained from Aldrich Chemical Company, Inc.
2. A time lag (10–15 min) is observed before the exothermic reaction begins. Addition of ethyl diazoacetate is then maintained at such a rate that the reaction temperature does not rise above 80°C. The checkers had to heat the mixture to 60°C.
3. Drying of the free acid should be rapid with a large surface area since traces of hydrochloric acid promote hydrolysis of the product to hydrazine salts. Slight warming ($\leq 60^\circ\text{C}$) during drying accelerates the drying process. The IR spectrum is as follows: IR (KBr) ν_{max} cm^{-1} : 3700–3100, 3320, 3000–1850, 1710, 1630. The checkers found that this step does not work as well on a smaller scale (0.14 mol).
4. Methanol is distilled from magnesium turnings immediately before use.
5. The submitters employed, without purification, thionyl chloride obtained from Fisher Scientific Company. The procedure should be performed in a well-ventilated hood since thionyl chloride is a lachrymator. The yield of dimethyl ester was found to be lower in instances when the thionyl chloride-methanol solution was not allowed to stir (30 min, -30°C) prior to the addition of dihydro-1,2,4,5-tetrazine-3,6-dicarboxylic acid.
6. The temperature is maintained at -30°C during the additions.
7. The submitters employed ether distilled from sodium benzophenone ketyl.
8. The spectral properties of the product are as follows: ^1H NMR (CDCl_3) δ : 3.92 (s, 6 H, CO_2CH_3), 7.42 (br s, 2 H, NH); IR (KBr) ν_{max} cm^{-1} : 3160, 3050, 1740, 1720.
9. The submitters employed methylene chloride from Fisher Scientific Company, which was distilled before use.
10. Nitrous gases are generated in a separate vessel by the disproportionation of nitrous acid (HONO): 200 mL of 6 N NaNO_2 (1.2 mol) is added dropwise to 125 mL of concentrated hydrochloric acid (1.5 mol) in a 500-mL, three-necked, round-bottomed flask fitted with a nitrogen inlet, a 500-mL addition funnel, and an outlet tube leading to the reaction flask. The brown gases evolved are bubbled directly into the reaction mixture through a 5-mm (inside diameter) glass tube (smaller inlet tubes occasionally became plugged) using a nitrogen stream. *CAUTION: all operations involving nitrous gases should be conducted in a well-ventilated hood because of the toxicity of these gases.*
11. The checkers observed some starting material in the product which depressed the mp. It could be removed by crystallization from ethyl acetate to give pure **3**, mp 176–177°C, but with significant loss of product. The spectral properties of the product are as follows: ^1H NMR (CDCl_3) δ : 4.22 (s, 6 H, CO_2CH_3); IR (KBr) ν_{max} cm^{-1} : 2970, 1752, 1445, 1385, 1219, 1175, 1082, 960, 912; UV (dioxane) λ_{max} (log ϵ) 520 nm (2.754).
12. The submitters employed 1,4-dioxane obtained from Fisher Scientific Company and distilled before use.
13. The submitters employed, without purification, 1-phenyl-1-(trimethylsiloxy)ethylene obtained from Aldrich Chemical Company, Inc.
14. The elemental analysis and the spectral analysis of the product are as follows: Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_4$: C, 61.76; H, 4.44; N, 10.29. Found: C, 62.01; H, 4.50; N, 10.19; ^1H NMR (CDCl_3) δ : 3.89 (s, 3 H, CO_2CH_3), 4.12 (s, 3 H, CO_2CH_3), 7.40–7.60 (m, 5 H, Ph), 8.27 (s, 1 H, C5-H); IR (KBr) ν_{max} cm^{-1} : 2955, 1742, 1584, 1447, 1399, 1287, 1244, 1142, 766; EI-MS (70 eV): m/e (relative intensity) 272 (M^+ , 9), 242 (7), 241 (6), 214 (34), 182 (10), 155 (base), lit³ mp 94–95.5°C.
15. The submitters employed, without purification, glacial acetic acid obtained from Fisher Scientific Company.
16. Zinc dust obtained from Fisher Scientific Company was activated prior to use following an established procedure.⁵
17. The elemental analysis and the spectral analysis of the product are as follows: Anal. Calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_4$: C, 64.86; H, 5.05; N, 5.40. Found: C, 65.10; H, 4.99; N, 5.48; ^1H NMR (CDCl_3) δ : 3.82 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 6.94 (d, 1 H, $J = 3$, C4-H), 7.30–7.60 (m, 5 H, Ph), 9.80 (br, s, 1 H, NH); IR (KBr) ν_{max} cm^{-1} : 3314, 2958, 1726, 1564, 1464, 1436, 1270, 1096, 1008, 940, 846, 762, 696. The additional formation of methyl 3-phenyl-5-carboxamidopyrrole-2-carboxylate in 32% yield is observed. The spectral properties of the product are as follows: ^1H NMR (CDCl_3) δ : 1.75 (br s, 2 H), 3.92 (s, 3 H), 7.14 (s, 1 H), 7.40–7.60 (m, 5 H); IR (KBr) ν_{max} cm^{-1} : 3442, 3346, 2950, 1708, 1642, 1580,

1528, 1476, 1366, 1280, 1132, 1022, 936, 808, 728, 618; CI-MS (70 eV): m/e (relative intensity) 245 (M⁺ + H, base).

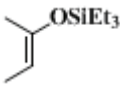
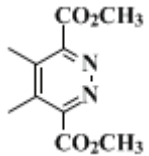

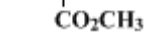

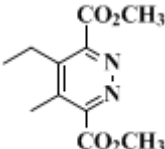

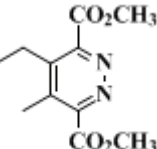
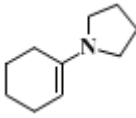
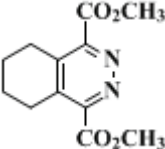
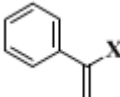
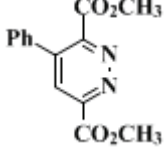
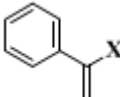
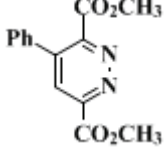
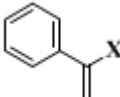
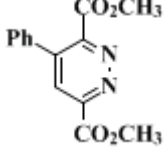
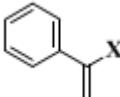
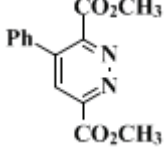
Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

The procedure describes the preparation and use of a reactive, electron-deficient heterocyclic azadiene suitable for Diels-Alder reactions with electron-rich, unactivated, and electron-deficient dienophiles.^{6 7 8 9 10 11} Dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate, because of its electron-deficient character, is ideally suited for use in inverse electron demand (LUMO_{diene}-controlled)¹² Diels-Alder reactions. Table I and Table II detail representative examples of the reaction of dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate with electron-rich carbon dienophiles¹³ and heterodienophiles,^{6,7,8} respectively. Complete surveys of the reported Diels-Alder reactions of dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate have been compiled.^{6,7,8,9,10,11} Reductive ring contraction of the substituted dimethyl 1,2-diazine-3,6-dicarboxylate [4 + 2] cycloadducts effected by zinc in acetic acid provides the corresponding substituted dimethyl pyrrole-2,5-dicarboxylates.^{13,14} Table III details representative examples of this general reductive ring contraction reaction.^{14,15}

TABLE I
DIELS-ALDER REACTIONS OF DIMETHYL 1,2,4,5-TETRAZINE-3,6-DICARBOXYLATE: 1,2-DIAZINE INTRODUCTION¹³

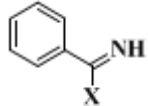
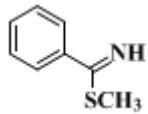
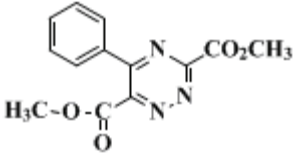
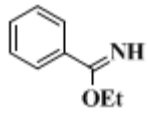
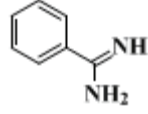
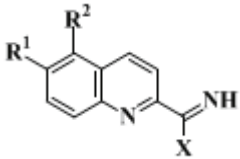
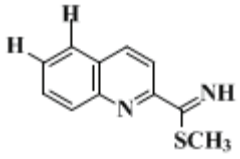
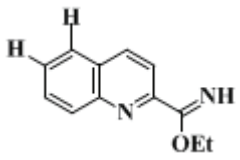
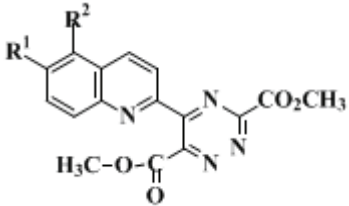
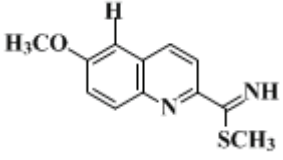
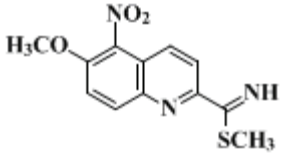
Entry	Dienophile	Conditions ^a Equiv. Temp °C (time hr)	1,2-Diazine	%Yield
1		1.5,25(12)		1 87
2		2-6,25(12)		1 trace
3	X = morpholine 	2,25(48)		2 70
4	= pyrrolidine 	2,25(48)		trace
5		1.5,25(12)		3 85
6	X = OSi(CH ₃) ₃ 	1,25(5)		4 92
7	= morpholine 	1.2,25(1.5)		87
8	= pyrrolidine 	1.5,25(12)		trace
9		1.5,25(0.5)		5 65

10	R = Si(Me) ₂ -t-Bu		1.5,5-25(0.5)		6	33
11			2-3,25(6)		6	82
12			2.5,101(3)		7	71
13			1,25(5)		8	69

(a) All Diels-Alder reactions were carried out in dioxane.

TABLE II
IELS-ALDER REACTIONS OF DIMETHYL 1,2,4,5-TETRAZINE-3,6-DICARBOXYLATE WITH C=N HETERODIENOPHILES^{16 17 18}

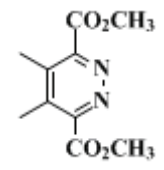
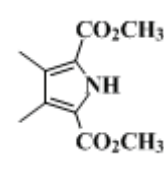
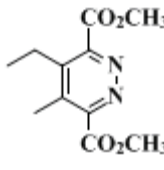
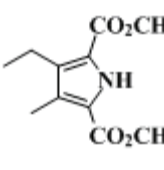
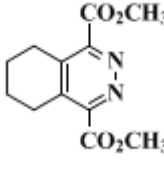
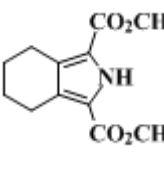
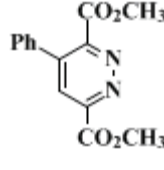
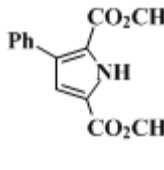
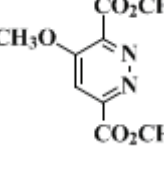
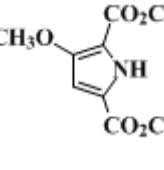
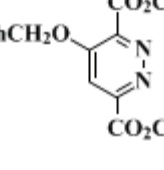
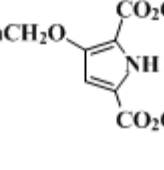
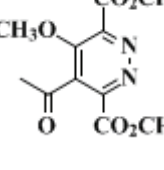
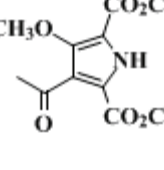
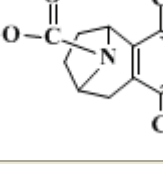
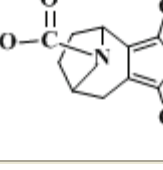
Dienophile	Conditions Temp °C (time hr) ^a	Product	% Yield
	80(20)		68
	80(8-12)		37
	25(5)		-
	25-50(25)		-

			
	80(24)		65
	60(10)		27
	25(5)		-
			
	80(4)		70
	80(20)		33
	80(4)		78
	80(20-24)		82

(a) All Diels-Alder reactions were carried out in dioxane.

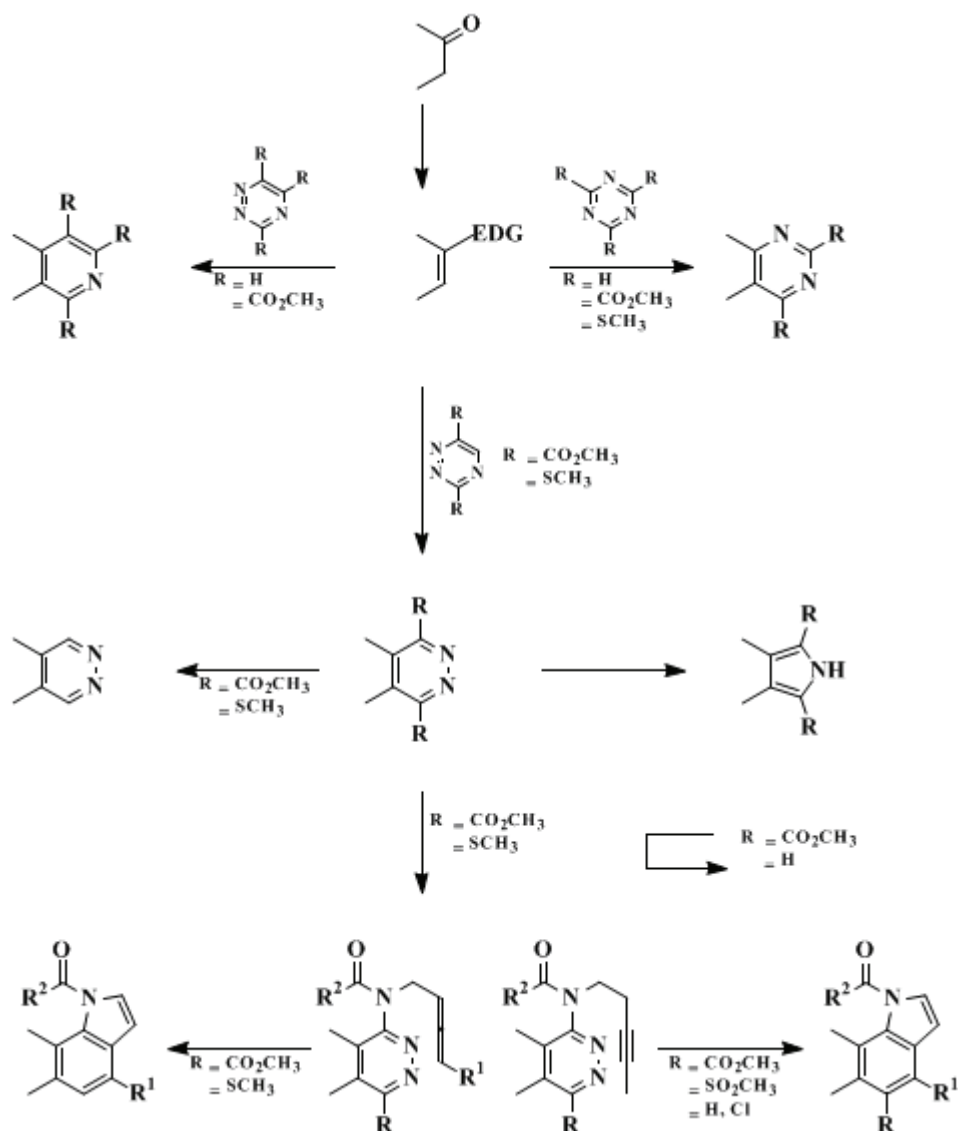
TABLE III
REDUCTIVE RING CONTRACTION OF SUBSTITUTED DIMETHYL 1,2-DIAZINE-3,6-DICARBOXYLATES: PYRROLE INTRODUCTION¹³

Entry	1,2-Diazine	Conditions ^a Temp °C (time hr)	Pyrrrole	% Yield
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1		25(24)		63
2		25(24)		70
3		25(22)		52
4		25(9)		65
5		25(24)		67
6		25(24)		62
7		25(24)		56
8		25(36)		48

(a) All zinc (9-20 molar equiv) reductions were carried out in acetic acid (0.09 M in substrate).

This approach to 1,2-diazine and pyrrole introduction based on the inverse electron demand Diels-Alder reaction of dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate complements the [4 + 2] cycloaddition reactions of a range of electron-deficient heterocyclic azadienes which permits the divergent preparation of a range of heterocyclic agents employing a common dienophile precursor, Scheme I.



References and Notes

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

sodium benzophenone ketyl

1,2-DIAZINE (DIMETHYL 4-PHENYL-1,2-DIAZINE-3,6-DICARBOXYLATE)

PYRROLE (DIMETHYL 3-PHENYLPYRROLE-2,5-DICARBOXYLATE)

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ethyl acetate (141-78-6)

methanol (67-56-1)

ether (60-29-7)

sodium hydroxide (1310-73-2)

thionyl chloride (7719-09-7)

sodium bicarbonate (144-55-8)

magnesium (7439-95-4)

sodium sulfate (7757-82-6)

nitrogen (7727-37-9)

nitrous acid (7782-77-6)

carbon (7782-42-5)

zinc (7440-66-6)

methylene chloride (75-09-2)

Pyrrole (109-97-7)

magnesium sulfate (7487-88-9)

ethyl diazoacetate (623-73-4)

Dihydro-1,2,4,5-tetrazine-3,6-dicarboxylic acid (3787-09-5)

hexane (110-54-3)

1,4-dioxane (123-91-1)

1,2-DIAZINE (289-80-5)

Dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate,
1,2,4,5-Tetrazine-3,6-dicarboxylic acid, dimethyl ester (2166-14-5)

Dimethyl 4-phenyl-1,2-diazine-3,6-dicarboxylate,
3,6-Pyridazinedicarboxylic acid, 4-phenyl-, dimethyl ester (2166-27-0)

Dimethyl 3-phenylpyrrole-2,5-dicarboxylate,
1H-Pyrrole-2,5-dicarboxylic acid, 3-phenyl-, dimethyl ester (92144-12-2)

Disodium dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate (96898-32-7)

Dimethyl dihydro-1,2,4,5-tetrazine-3,6-dicarboxylate (3787-10-8)

1-Phenyl-1-(trimethylsiloxy)ethylene (13735-81-4)

methyl 3-phenyl-5-carboxamidopyrrole-2-carboxylate

dimethyl 1,2-diazine-3,6-dicarboxylate