Graphical Abstract

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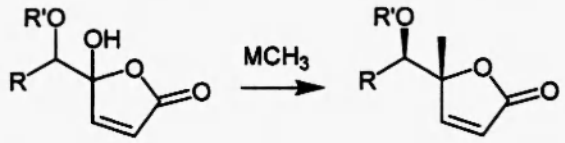
Diastereoselective addition of organotitanium reagents to chiral γ-hydroxybutenolides

William H. Miles,* Daniela G. Duca, Jaryd T. Freedman, Elliot O. Goodzeit, Kristin B. Hamman, Chiquita A. Palha De Sousa and Brandon R. Selfridge, Department of Chemistry, Lafayette College, Easton, PA 18042

The addition of $(i\text{-PrO})_3TiCH_3$ to γ-hydroxybutenolides having a chiral center with an alkoxy group adjacent to the ketone functionality (of the open chain form) gave γ-lactones in good yields and high diastereoselectivity.



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X-ray structures and molecular orbital calculations of 6-phosphapentacyclo-[6.3.1.$0^{2,4}$.$0^{3,7}$.$0^{5,10}$]dodecane 6-oxides

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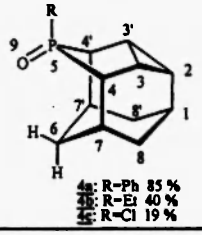
The X-ray analyses of a novel type of the P-caged compounds, 6-ethyl and 6-chloro-6-phosphapentacyclo-[6.3.1.$0^{2,4}$.$0^{3,7}$.$0^{5,10}$]dodecane 6-oxide 4b, 4c established the same *cis*-stereochemistry around the phosphorus atom as that of the previously reported 4a. The results of molecular orbital calculations of 4 and their *trans*-isomers at semiempirical, *ab initio*, and DFT (DGauss) levels were in agreement with the more thermodynamically stable 4 than the non-obseved *trans*-isomer.



4a: R=Ph 85 %
4b: R=Et 40 %
4c: R=Cl 19 %

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Cephem and pyrrolo[2,1-b][1,3]thiazine-4,6-dione ring systems construction from 6-benzoyl-2,3-dihydro-4h-1,3-thiazine-4-ones prepared via rearrangement of 5-benzoyl-3(2H)-isothiazol-3-ones

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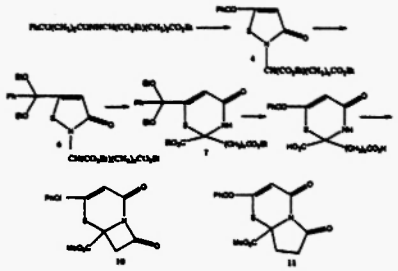
Cephem 10 and Pyrrolo[2,1-b][1,3]thiazine-4,6 dione 11 ring systems' construction from 6-benzoyl-2,3-dihydro-1,3-thiazine-4-ones 9 prepared from properly designed 5-benzoyl-3(2H)-isothiazol-3-ones 4 through experimental simple reactions, is described here.



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Synthesis of some novel 1, 2, 4-triazolo [4, 3-a] 2h-pyrano [3, 2-e] pyridine derivatives

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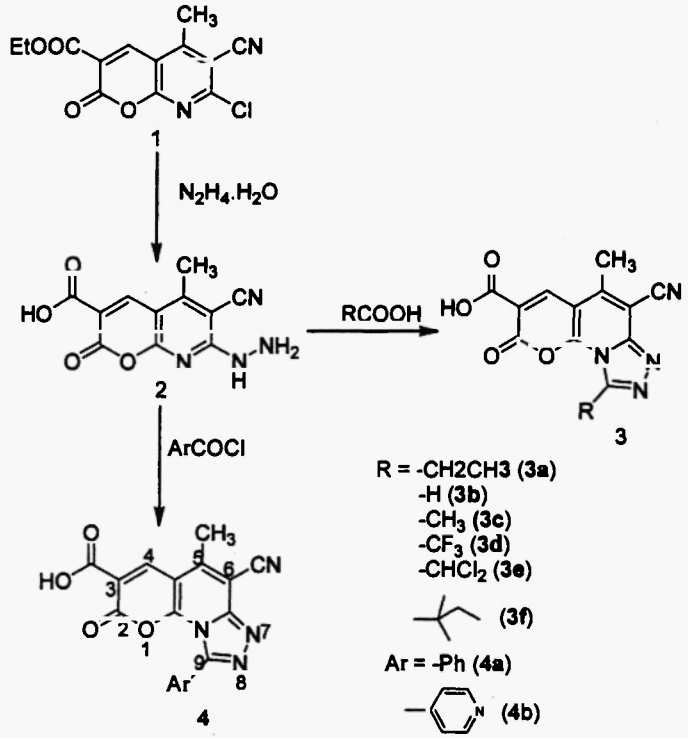
New 1, 2, 4 triazoles containing 2H-pyrano [2, 3-b] pyridine moiety 3a-f and 4a-b have been synthesized. Ethyl-5-methyl-6-cyano-7-chloro-2-oxo-2H-pyrano [2, 3-b] pyridine-3-carboxylate was converted into 5-methyl-6-cyano-7-hydrazino-2-oxo-2H-pyrano [2, 3-b] pyridine-3-carboxylic acid by reacting with hydrazine hydrate, followed by cyclization with aliphatic acids and aromatic acid chlorides to afford 1, 2, 4 triazoles. The structures of all compounds synthesized were assigned on the basis of elemental analysis, IR and ^{1}H NMR spectral data.



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Intramolecular Hydrogen Bonding in the Nitrobenzopyridines

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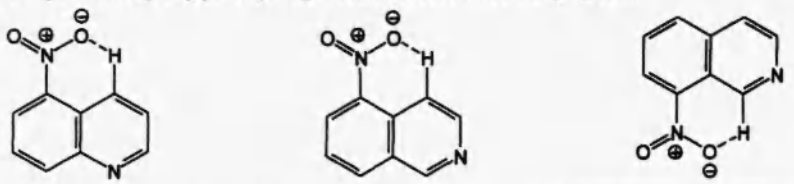
The available data on the spectroscopy of the nitrobenzopyridines are insufficient, out-dated, and modern theory is missing. A careful study of the ^{1}H-NMR spectra of these compounds revealed the existence of C–H---O–N=O hydrogen bonds, the hydrogen being at *peri* position to the nitro group. The existence of these bonds explains the down-field shifts observed in the signals of the involved protons. Spectral data from the naphthalene series and from ^{17}O-NMR measurements, both involving the nitro group/*peri*-hydrogen relation, confirmed our proposal.



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Synthesis of novel isoxazole derivatives from 1,3-diketone derivatives

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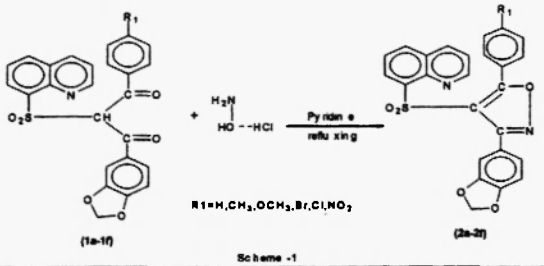
Condensation of β-diketone derivative with hydroxylamine hydrochloride ($NH_2OH.HCl$) in pyridine results in the synthesis of isoxazole derivative. By washing with 15% glacial acetic acid and then recrystallization with 95% C_2H_5OH led to crystal formation. Purity of the newly synthesized isoxazole derivative was checked by TLC.The structure of newly synthesized isoxazole derivative were established on the basis of IR, ^1H NMR, ^{13}C NMR and elemental analysis.



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Synthesis and characterization of irbesartan impurities

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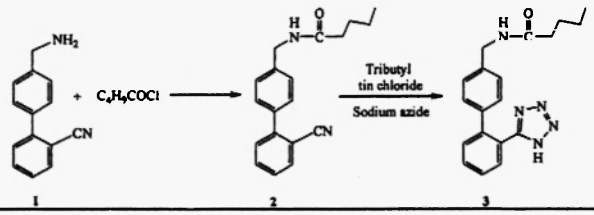
In the course of synthesis of an antihypertensive drug, Irbesartan, five process related potential impurities were detected by using high performance liquid chromatography. They were synthesized and characterized by NMR, Mass, and IR. Synthesis and characterization of these Irbesartan impurities is reported.



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One or two step acid mediated cyclocondensation process for the preparation of 5-carbethoxy-2-thiouracils from diethyl ethoxymethylenemalonate and thioureas

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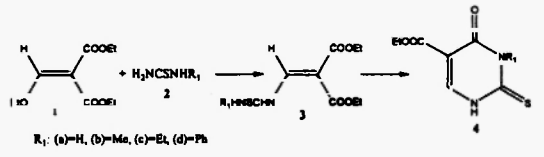
An acid mediated synthesis of 5-carbethoxy-2-thiouracils via an one step cyclocondensation of diethyl ethoxymethylenemalonate and thioureas or the under milder reaction conditions, also obtained, thioureidomethylenemalonates, is described here.



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One-pot synthesis of pentasubstituted pyrroles from tertiary amides and maiononitrile via an indirect condensation reaction on amidic carbonyl group

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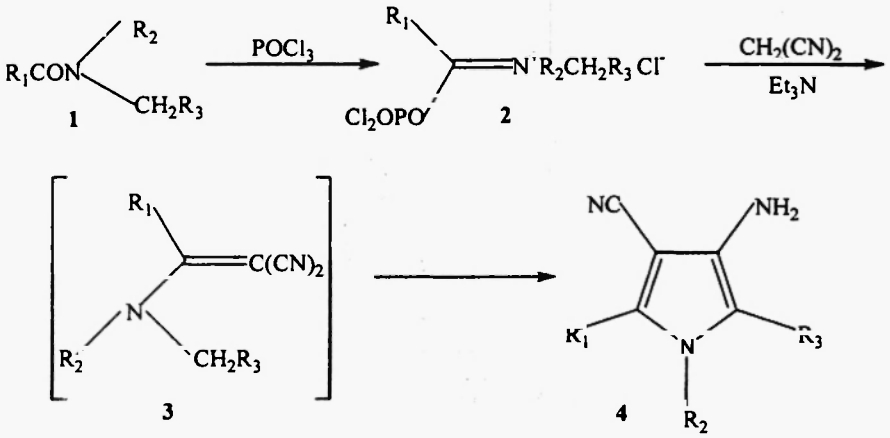
An one-pot procedure for the preparation of pentasubstituted pyrrole derivatives, using tertiary acid amides-phosphorus oxychloride adducts on reaction with malononitrile, in the presence of triethylamine, is described.



R_1, R_2, R_3: (a) Me, Me, CN; (b) Me, Me, CO_2Me; (c) Me, $PhCH_2$, CN; (d) Me, $PhCH_2$, CO_2Me; (e) Me, Ph, CN; (f) Me, Ph, CO_2Me; (g) Ph, Me, CN; (h) Ph, Ph, CN; (i) Ph, $PhCH_2$, CN

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Phase transfer catalysts promoting the one-pot synthesis and biological activities of 1-(5-methylisoxazoyl-4-carbonyl)-4-arylthiosemicarbazides and their related heterocyclic compounds

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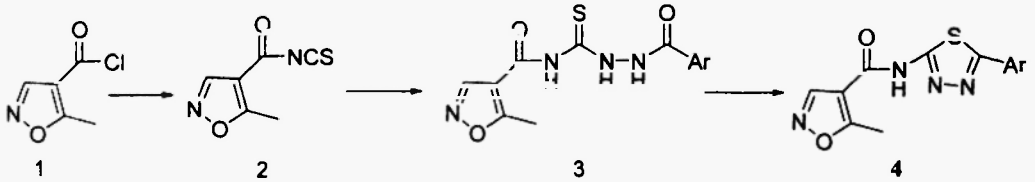
Reaction of aryl acylhydrazine with 5-methylisoxazoyl chloride and ammonium thiocyanate under the condition of solid–liquid phase-transfer catalysis using polyethylene glycol-600 (PEG-600) as the catalyst and ultrasonic irradiation yielded 1-(5-methylisoxazoyl-4-carbonyl)-4-arylthiosemicarbazides **3a-j** in good-to-excellent yield. Furthermore a series of 1,3,4-thiadiazole derivatives **4a-j** were synthesized via reaction of the thiosemicarbazide with acetate acid. The chemical structures of all compounds were established by ^1H NMR, FTIR, MS, and elemental analysis, and some of these compounds were investigated for fungicidal activity. The bioassay results indicated that some of these compound exhibit moderate fungicidal activities.



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Condensation of *n*'-tosylamidrazones with oxalyl dichloride and dichlorophosphorus derivatives

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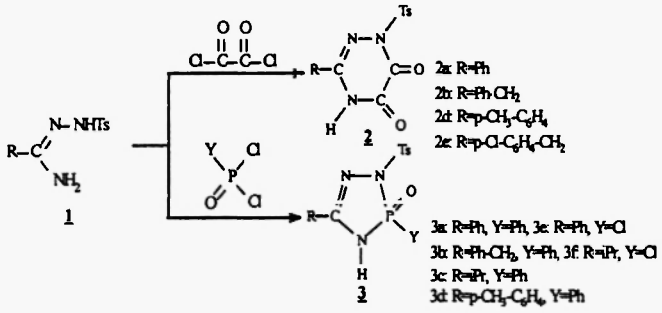
A series of news 1,2,4-triazin-5,6-diones 2 and 1,2,4,3-triazaphospholines-3-oxides derivatives 3 have been synthesized. The products 2 and 3 were characterized by IR, ^{1}H, ^{13}C, and ^{31}P NMR and elemental analysis.



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Microwave-promoted conversion of heterocyclic amines to corresponding amides under solvent-free conditions

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An array of heterocyclic amides was synthesized efficiently by combining corresponding amines and benzoyl chloride in one-pot under microwave irradiation. The reaction times were shorter, yields were higher. What is more, the regioselectivity was excellent, which made the protocol support us an entry to selective protection of diverse amino groups.