Manuscript Preparation

An electronic version of accepted manuscripts should be provided on a computer disk. This disk should include the text in Microsoft Word along with drawings prepared and saved as a ChemDraw file. Publication of manuscripts that are not accompanied by an electronic version in the format specified for both the text and drawings will be delayed.

Drawings, graphs and illustrations must be submitted as carefully prepared drawings printed out on a high quality printer. Structural formulas should be prepared using a standard structure drawing program, preferably ChemDraw. Clear high contrast glossy photographs or laser prints are suitable. Transparencies are unacceptable.

Literature references are to be numbered consecutively in order of appearance of the reference in the text and are to be placed at the end of the manuscript under the center heading: REFERENCES AND NOTES. Reference numbers are to be placed in square brackets on the same line as the text. Authors are encouraged to include all pertinent references. The journal abbreviations must be italicized with the punctuation shown in CASSI. Sample citations for referencing a journal article, a book chapter and a patent appear below: [1] J.-K. Luo and R. N. Castle, *J. Heterocyclic Chem.*, **28**, 205 (1991). [2] Y. S. Sanghyi, S. B. Larson, R. K. Robins and C. R. Revenkar, *J. Chem. Soc., Perkin Trans. I*, 2943 (1990). The following is a sample book reference: [3] G. R. Newkome and W. W. Paudler, Contemporary Heterocyclic Chemistry, John Wiley & Sons, New York, NY, 1982, pp 107-109. A reference to a book written with various contributors should be cited as follows: [4] S. W. Schneller, in Comprehensive Heterocyclic Chemistry, Vol **5**, K. T. Potts, ed, Pergamon Press, Oxford, 1984, pp 847-904. Patent citations must be followed by the *Chem. Abstr*., reference. A sample patent citation is as follows: [5] A. Van Schoor, W. Schumann, S. Lust and H. Flemming, German Patent 1,141,487 (1962); *Chem. Abstr*., **58**, 12470g (1963).

Footnotes are only allowed in tables and figures and follow the same format as those of the references where footnotes are designated by lower case letters enclosed in square brackets on the same line, e.g. [a], [b], [c], etc. The footnotes then appear with the same designation at the bottom of the figure or table.

Nomenclature must conform to the recommendations set forth in the following sources: [1] Nomenclature of Organic Chemistry, (IUPAC Blue Book), J. Rigaudy and S. P. Klesney, 1979 Ed., Pergamon Press. [2] Ring Systems Handbook, 1993 Edition, Chemical Abstracts Service, 2540 Olentangy River Road, P.O. Box 3012, Columbus OH 43210 USA.

Tables should be carefully constructed to fit within a printing area of $6^3/_4$ x 9 inches (17 x 23 cm). All information in tables must be double spaced and one space is required after each comma and before each parenthesis. Semicolons and colons may not be used in tables. Footnotes in tables must be designated by lower case letters in square brackets as described above.

Experimental. The experimental part of the manuscript should provide the reader with a clear and unambiguous description of the work reported. It should not be verbose, but should be sufficiently detailed that it is readily reproducible. The title of each experiment should include the chemical name of the compound and the assigned compound number. Either bold face Arabic or Roman numerals may be used for compound numbers, however, once one is chosen it must be used throughout the manuscript. There must be at least one complete sentence per paragraph. Each previously unreported compound must be supported by adequate spectral data and elemental analyses for at least two elements agreeing with the theoretical value to within 0.4%. All elemental analytical data will be published. Chemical names must be used throughout the manuscript including the experimental in place of chemical formulas. The following abbreviations, acronyms and conventions should be used: g, mg, mL, L, mp, bp, tlc, hplc, gc, uv, ir, nmr, pmr, cmr, ms, hrms, lit, dec, mole, mmole, moles, mmoles, or mol, and mmol. Other abbreviations are to be avoided using in place of an abbreviation, the complete word. All acronyms not listed above must be written out the first time it appears in the text. Extensive use of acronyms should be avoided. It is requested that simple punctuation be used in the experimental.

Examples of Experimental Procedures

3-(3'-Thienyl)-2-cyclohexen-1-one (**15**).

A solution of 3.13 g (0.019 mole) of 3-bromothiophene (**12**) dissolved in 50 ml of dry ether was cooled to -78°. The 3-bromothiophene (**12**) was lithiated by slowly adding 12.4 ml of a 1.55 *M* solution of *n*-butyllithium in hexane. After the addition was complete, the solution was stirred at -78° for 30 minutes. A solution of 3.0 g (0.019 mole) of 1,4-dioxaspiro[4.5]decan-7-one in 25 ml of dry ether was added over a period of 20 minutes. The solution was allowed to warm to room temperature overnight. The solution was then neutralized with 10% aqueous ammonium chloride solution. The ether layer was separated, dried and the solvent was evaporated. The residue was then refluxed in aqueous 1.4 *M* hydrochloric acid for 1 hour. The organic product was isolated by extraction with ether. The ether was dried (sodium sulfate) and evaporated to yield an oil which was crystallized from benzene-hexane as yellow needles, 1.4 g (41%), mp 100-102°; ^{1}H nmr (deuteriochloroform): δ 2.0-3.0 (m, 6H), 6.3 (s, 1H), 7.3 (m, 2H), 7.5 (m, 1H); ms: m/z 180 (3.0), 179 (6.4), 178 (54), 150 (95), 122 (100), 121 (84), 39 (86).

Anal. Calcd. for $C_{10}H_{10}OS$: C, 67.38; H, 5.65; S, 17.99. Found: C, 67.06; H, 5.75; S, 17.69.

General Procedure for the Reaction of Tropone Hydrazones 1-3 with Phenylketene (**8**).

All of these reactions were carried out under a nitrogen atmosphere. To a stirred and cooled (0°) solution of tropone benzoylhydrazone (**11**) (1.12 g, 5 mmoles) and triethylamine (0.61 g, 6 mmoles) in dry THF (15 ml), a solution (5 ml) of phenacetyl chloride (0.77 g, 6 mmoles) was added drop by drop for ten minutes and the reaction mixture was allowed to stand at that temperature for an additional thirty minutes. After being warmed to room temperature gradually, the mixture was stirred overnight. The resultant triethylamine hydrochloride was filtered and the filtrate was evaporated *in vacuo* to give an oily residue. The residue was separated by column chromatography (silica gel-chloroform) to give **9a** and **10a** in 25 and 9% yield, respectively.

1-Benzamido-1,2,3,3a-tetrahydro-2-oxo-3-phenyl-1-azaazulene (**9a**).

This compound was obtained as colorless needles (isopropyl alcohol), mp 179-180°; ir: NH 3200, CO 1725, 1675 cm^{-1}; ^{1}H nmr: δ 3.12 (br, 1H, 3a-H), 3.86 (br d, 1H, 3-H, J = 5.7 Hz), 5.24 (dd, 1H, 4-H, J = 3.0, 9.0 Hz), 5.44 (d, 1H, 8-H, J = 6.3 Hz), 6.1-6.5 (m, 3H, 5-, 6-, and 7-H), 7.2-7.9 (m, 10H, phenyl protons), 9.3 ppm (br s, 1H, NH); ms: m/z 342 (M$^+$), 224 (M$^+$-PhCHCO), 119 (PhNCO$^+$), 105, 77.

Anal. Calcd. for $C_{22}H_{18}N_2O_2$: C, 77.17; H, 5.30; N, 8.18. Found: C, 77.16; H, 5.28; N, 8.35.

1,2,3,3a-Tetrahydro-2-oxo-1-phenylacetylbenzamido-3-phenyl-1-azaazulene (**10a**).

This compound was obtained as colorless prisms (isopropyl alcohol), mp 158-159°; ir: CO 1755, 1730, 1705 cm^{-1}; ^{1}H nmr: δ 2.86 (br, 1H, 3a-H), 3.82 (br d, 1H, 3-H, J = 5.4 Hz), 4.15, 4.37 (2d, each, 1H, -CH$_2$-, J = 15.3 Hz), 5.25 (dd, 1H, 4-H, J = 3.2, 9.6 Hz), 5.44 (d, 1H, 8-H, J = 6.3 Hz), 6.0-6.7 (in, 3H, 5-, 6-, and 7-H), 7.1-7.7 ppm (m, 15H, phenyl protons); ms: m/z 460 (M$^+$), 341 (M$^+$-PhCH$_2$CO), 224 (hydrazone 1$^+$), 119 (PhNCO$^+$), 105, 77.

Anal. Calcd. for $C_{30}H_{24}N_2O_3$: C, 78.24; H, 5.25; N, 5.97. Found: C, 78.23; H, 5.25; N, 6.38.

Methyl 5-Cyanomethyl-1-(2-deoxy-β-D-erythro-pentofuranosyl) pyrazole-4-carboxylate (**14**).

To a solution of **9** (0.80 g, 1.54 mmoles) in dry methanol (25 ml) was added 1 *M* sodium methoxide in methanol (1.25 ml) and the mixture was stirred at room temperature for 1 hour. The reaction mixture was neutralized with Dowex-50 H+ resin and filtered. The filtrate was evaporated to dryness. The residual semisolid was purified on a silica gel column (2.5 x 45 cm), prepacked in chloroform. Elution of the column with chloroform:methanol (19:1, v/v) gave a homogeneous residue, which was crystallized from aqueous ethanol to yield 0.17 g (37%), mp 118-120°; ir (potassium bromide): 1700 (C=O), 2260 (C≡N), 3400 (OH) cm^{-1}; uv: (*p*H 1): λ max 218 nm (ε 11,000); (*p*H 7): λ max 218 nm (ε 11,100); (*p*h 11): λ max 224 nm (ε 10,200); ^{1}H nmr (DMSO-d$_6$): δ 3.82 (s, 3, CO$_2$C*H*$_3$), 4.40 (s, 2, CH$_2$), 6.16 (t, 1, peak width 14.5 Hz, C1'H), 8.18 (s, 1, C$_3$*H*), and other sugar protons.

Anal. Calcd. for $C_{12}H_{15}N_3O_5$ (281.17): C, 51.24; H, 5.33; N, 14.94. Found: C, 50.99; H, 5.43; N, 14.80.

5-Amino-1-[4-methylsulfonyl)phenyl]-1*H*-pyrazole-4-carbonitrile (**5b**).

A solution of 50.0 g (0.268 mole) of 4-methylsulfonyi)phenylhydrazine (**3b**) and 34.3 g (0.281 mole) of ethoxymethylenemaononitrile (**4**) in 250 ml of ethanol was heated at reflux. A prcipitate was observed after 15 minutes. After 15 hours, the mixture was cooled and the yellow solid was collected and air-dried to yield 51.4 g (73%) of **5b**, mp 236-238°; ir (Nujol): 3450, 3315 and 3170 (NH), 2225 (CN), 1635 cm^{-1}; ^{1}H nmr (dimethyl sulfoxide-d$_6$): δ 8.11 (d, J = 8 Hz, 2H, phenyl), 7.89 (s, 1H, C3-H), 7.84 (d, J = 8 Hz, 2H, phenyl), 6.94 (broad s, 2H, NH, deuterium oxide-exchangeable), 3.25 (s, 3H, CH$_3$); ms: (70 eV, electron impact) m/z 262 (molecular ion).

Anal. Calcd. for $C_{11}H_{10}N_4O_2S$: C, 50.37; H, 3.84; N, 21.36. Found: C, 50.40; H, 3.87; N, 21.57.

3-Methoxy-4*H*-pyran-4-one-2-carboxaldehyde (**1**).

2-Hydroxymethyl-3-methoxy-4*H*-pyran-4-one (700 mg) was dissolved in 50 ml of dichloromethane. Barium manganate (7.5g) was ground to a fine powder and added immediately to the dichloromethane solution. The mixture was stirred with the aid of a magnetic stirrer for two hours at room temperature. Inorganic by-products were removed by filtration of the reaction mixture through Celite. The Celite was washed with dichloromethane; the latter solution was added to the dichloromethane filtrate previously obtained. Evaporation of dichloromethane *in vacuo* gave a white residue, which was recrystallized from cyclohexane to give 635 mg (92%) of 3-methoxy-4*H*-pyran-4-one-2-carboxaldehyde (**1**), mp 85-86°; ^{1}H nmr (deuteriochloroform): δ 4.16 (s, 3H, OCH$_3$), 6.43 (d, 1H, H5), 7.73 (d, 1H, H6), 10.14 (s, 1H, CHO); ir (potassium bromide): 3100 (CH), 1695 (aldehyde CO), 1655 (pyrone CO), 1575, 1460, 1430, 1395, 1275, 1225, 1200, 1175, 1050, 950, 850, 830, 750, 640 cm^{-1}.

Anal. Calcd. for $C_7H_6O_4$: C, 54.55; H, 3.92. Found: C, 54.58; H, 3.91.

The above experimental procedures illustrate the following:

1. The requirement of the chemical name and assigned compound number in the heading.
2. The requirement of a sufficiently detailed experimental description to enable an experienced chemist to repeat the experiment successfully.
3. The manner in which physical, spectral and analytical data **must** be reported.
4. In the case of a general procedure for the synthesis of two or more compounds, this must be followed by a heading composed of the chemical name and the assigned compound number of each compound prepared by the general procedure.
5. At least one complete sentence must be included in the paragraph followed by the physical, spectral and elemental analytical data as illustrated for **9a** and **10a** above.
6. Percentage yields may be reported to the nearest whole percent. The reporting of fractional yields is valid only for yields of less than 10%.

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