served between $MoO_2(dpm)_2$ and *p*-phenylazoanisole, either in the visible or the ultraviolet spectra.

Acknowledgments. The authors are indebted to Mr. David Mattern for mass spectrometric analyses and to Dr. John Gordon for valuable discussions.

Registry No. *t*-BuO_2H, 75-91-2; $MoO_2(dpm)_2$, 34872-98-5; $Mo(CO)_6$, 13939-06-5; CH_3CO_3H, 79-21-0; *m*-$ClC_6H_4CO_3H$, 937-14-4.

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Azabicyclo Chemistry. IV. A New Route to 2-Azabicyclo[3.3.1]nonanes Containing a Functionalized Carbocyclic Ring[1]

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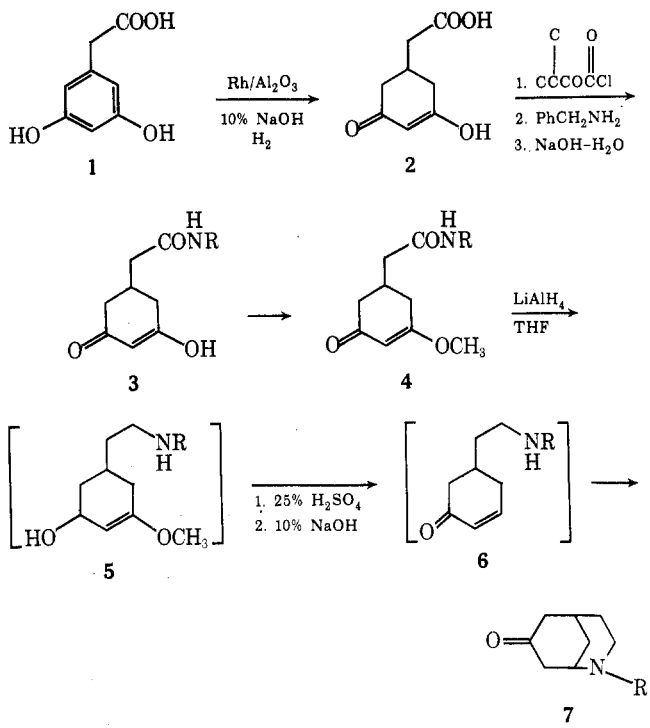
We have been interested in functionalized 2-azabicyclo[3.3.1]nonanes because of their possible elaboration into more complex molecules with potential analgetic activity. An inspection of the literature indicates a paucity of routes to the 2-azabicyclo[3.3.1]nonane skeleton and only a few examples[2] of any derivatives having a functional group in the carbocyclic ring. Our synthetic route was chosen on analogy to that used for another azabicyclo system[3] and was dependent on the success of an intramolecular Michael-type cyclization.

Diethyl 3-oxoglutarate was converted in two steps, by a slight modification of the procedure of Theilacker and Schmid,[4] to crystalline 3,5-dihydroxyphenylacetic acid (**1**), the starting point in the synthesis. The latter authors[4] reported obtaining the dione **2** in a 59% yield by conversion of **1** to its ethyl ester followed by reduction with Pt/H_2. We were able to improve not only the yield, but the time necessary for obtaining **2** by directly reducing **1** in an atmosphere of H_2 (50 psi) with rhodium on alumina in base at elevated temperature, thus affording **2** in a 77% yield. The physical properties were consistent with **2** existing in the expected enolic form, that is, uv max 254 nm (ϵ 14,800) and nmr (DMSO-d_6) δ 5.20 (s, 1, vinyl H).



a, R = $CH_2C_6H_5$
b, R = CH_3

Amide formation directly on **2** is complicated by the fact that primary amines also react with the conjugated enolic system. We were able to overcome this by forming the amide by the mixed carbonic anhydride method used by Anderson, *et al.*,[5] for the synthesis of peptides. When **2** is treated with 2 equiv of isobutyl chloroformate and *N*-methylmorpholine ("inverse addition"[5]), not only is the carboxyl activated for amide formation but the enolic hydroxyl is protected as the carbonate ester. Subsequent treatment with benzylamine or methylamine, followed by hydrolysis of the intermediate carbonate and neutralization, gave the amides **3a** and **3b** in 60 and 64% yields, respectively. A solution of **3a** in refluxing methanol containing *p*-toluenesulfonic acid readily gave the enol ether **4a** in a 89% yield, nmr δ 3.65 (s, 3, OCH_3), uv max 248 nm (ϵ 16,400). The methylamide **3b** was also converted to its methyl enol ether **4b**; however, it apparently was more sensitive to hydrolysis as attempts at crystallization

caused partial formation of the starting enol **3b**. Thus, **4b** was not isolated but used directly in the next step.

Lithium aluminum hydride reduction of the enol ethers of 1,3-dicarbonyl compounds is a well-documented[6] route to α,β-unsaturated ketones, and when this is coupled with the presence of an amine function, intramolecular Michael-type cyclizations are possible.[3] Reduction of the enol ethers **4a** and **4b** with $LiAlH_4$ in tetrahydrofuran presumably gave the unisolated amino alcohols **5a** and **5b**, respectively. Hydrolysis of **5a** and **5b** in aqueous acid followed by treatment with sodium hydroxide effected cyclization, through the conjugated ketones **6a** and **6b**, to the desired 2-azabicyclo[3.3.1]nonan-7-ones **7a** and **7b**, respectively. The mass spectral data (see Experimental Section) substantiated the structural assignments for **7a** and **7b**.

Experimental Section

Infrared spectra were recorded on a Beckman IR-8 spectrophotometer. Ultraviolet spectra were determined in 95% ethanol on a Perkin-Elmer 202 ultraviolet-visible recording spectrophotometer. Nmr spectra were recorded on a Varian A-60 spectrometer in $CDCl_3$ with tetramethylsilane as internal standard. Melting points were taken in open capillary tubes on a Thomas-Hoover melting point apparatus and are uncorrected. Thin layer chromatograms were carried out with silica gel GF (Analtech, Inc.) and the spots were located with uv light or Dragendorff's reagent. All concentrations were done under reduced pressure. Prior to concentration, all organic layers were dried over anhydrous $MgSO_4$ powder. Mass spectra were determined on an LKB Model 9000 spectrometer at 70 eV. Tetrahydrofuran (THF) was purified and dried by distillation from $LiAlH_4$ and stored over beads of 3A molecular seive. Microanalyses were performed by Spang Microanalytical Laboratory, Ann Arbor, Mich., and Galbraith Laboratories, Inc., Knoxville, Tenn.

1,3-Cyclohexanedione-5-acetic Acid (2). Crystalline 3,5-dihydroxyphenylacetic acid[4] (mp 125–127° from CH_2Cl_2–ether, 21.5 g, 0.128 mol) and 5% rhodium on alumina catalyst (2.15 g) were mixed with a 10% NaOH solution (153 ml, 0.384 mol) and hydrogenated in a Parr apparatus at 55° and 55 psi H_2 for 24 hr. After this time the catalyst was removed by filtration, a fresh quantity (2.15 g) of catalyst was added to the filtrate, and the mixture was again hydrogenated for 48 hr. The catalyst was removed by filtration, and the filtrate was acidified with concentrated HCl and allowed to crystallize, thus affording, in two crops, 16.7 g (76.5%) of the dione **2**: mp 125–127° (lit.[4] mp 127–128°); ir (Nujol) 5.85 (carboxylic acid), 6.21 and 6.35 μ (1,3-diketone); mass spectrum m/e 170 (molecular ion). The observed melting point was obtained after drying in a vacuum desiccator over P_2O_5 at 0.05 mm for 48 hr.

***N*-Benzyl-3,5-cyclohexanedione-1-acetamide (3a).** The following procedure was carried out at a temperature of −10 to −15°. To a stirred solution of 4-methylmorpholine (4.85 g, 0.048 mol) in 100 ml of THF was added a solution of isobutyl chloroformate (6.51 g, 0.048 mol) in 10 ml of THF and the solution was allowed to react for 15 min. After this time, a solution of the dione **2** (3.36 g, 0.020 mol) in 35 ml of THF was added dropwise, and then the solution was allowed to stir for another 20 min. A solution of benzylamine (2.56 g, 0.024 mol) in 15 ml of THF was then added to the mixture and the mixture was allowed to react for 15 min. The mixture was filtered and the filtrate was concentrated. The residue was dissolved in CH_2Cl_2 and washed once with 15 ml of 5% H_2SO_4 and the organic layer was concentrated without drying. The remaining oil was suspended in 50 ml of a 2.5% NaOH solution containing 5 ml of CH_3OH and stirred for 35–40 min at 50–60°. The resulting solution was cooled in ice, washed twice with CH_2Cl_2, and made acidic with concentrated H_2SO_4. After cooling overnight, the crystalline amide **3a** was collected and dried (3.1 g, 60%), mp 168–170°. Two recrystallizations from water gave the analytical sample: mp 169–171°; ir (Nujol) 6.11 (amide carbonyl), 6.49 μ (1,3-diketone); uv max 254 nm (ϵ 15,600).

Anal. Calcd for $C_{15}H_{17}NO_3$: C, 70.02; H, 5.88; N, 5.44. Found: C, 69.96; H, 5.90; N, 5.29.

***N*-Benzyl-3-methoxy-5-oxo-3-cyclohexeneacetamide (4a).** A solution of **3a** (3.10 g, 0.012 mol) in 50 ml of methanol containing 50 mg of *p*-toluenesulfonic acid was refluxed for 24 hr and then concentrated. The residue was dissolved in 50 ml of methanol, refluxed for an additional 24 hr, and again concentrated to dryness. The solid material was dissolved in CH_2Cl_2, washed with 20 ml of saturated $NaHCO_3$ solution, and concentrated to dryness, leaving a white solid, 2.90 g (89%), of **4a** (homogenous on tlc), mp 134–136°. An analytical sample of **4a** was obtained by sublimation at 125° (0.005 mm): mp 134–136°; ir ($CHCl_3$) 6.08 (amide and conjugated carbonyl) and 6.23 μ (C=C); nmr δ 4.38 (d, 2, J = 6 Hz, aromatic CH_2), 5.28 (s, 1, C=CH), 6.67 (m, 1, NH), 7.25 (s, 5, aromatic H).

Anal. Calcd for $C_{16}H_{19}NO_3$: C, 70.31; H, 7.01; N, 5.12. Found: C, 70.39; H, 7.12; N, 5.19.

2-Benzyl-2-azabicyclo[3.3.1]nonan-7-one (7a). To a solution of **4a** (2.73 g, 0.010 mol) in 200 ml of THF was added solid $LiAlH_4$ (1.48 g, 0.040 mol) while stirring and then the solution was refluxed for 12 hr. The suspension was cooled and treated with 10% NaOH until a white precipitate no longer formed. The organic layer was filtered and the salts were washed several times with THF. The combined filtrate and washings were concentrated and the residual oil was stirred with 25 ml of 25% H_2SO_4 at ambient temperature for 2 hr. The reaction mixture was cooled in an ice bath and slowly made strongly basic with 10% NaOH. The alkaline mixture containing an oily layer was extracted five times with 50-ml portions of CH_2Cl_2, and the combined organic layers were washed twice with a saturated NaCl solution, dried, and concentrated. The resulting crude amine (2.71 g) was further purified on a column (2.3 × 20 cm) of Woelm neutral alumina (90 g, activity grade V) packed in benzene. The amine **7a** was eluted with benzene and concentrated to an oil which in ether was converted to its hydrochloride salt. Crystallization from acetonitrile gave 1.1 g (42%) of **7a** HCl: mp 182–184°; ir ($CHCl_3$, free base) 5.88 μ (ketone); nmr (free base) δ 3.52 (s, 2, aromatic CH_2), 7.18 (s, 5, aromatic H); mass spectrum[7] m/e (rel intensity) 229 (29), 186 (19, loss of CH_3CO, metastable ion at 151.1), 172 (88, α cleavage of molecular ion followed by a McLafferty rearrangement, loss of CH_3COCH_2, metastable ion at 129.2), 91 (100, tropylium ion).

Anal. Calcd for $C_{15}H_{20}ClNO$: C, 67.78; H, 7.56; N, 5.27. Found: C, 67.78; H, 7.59; N, 5.45.

***N*-Methyl-3,5-cyclohexanedione-1-acetamide (3b).** The same procedure described for **3a** was used. Thus, 12.14 g (0.12 mol) of 4-methylmorpholine in 200 ml of THF, 16.4 g (0.12 mol) of isobutyl chloroformate in 20 ml of THF, 8.5 g (0.05 mol) of dione **2** in 50 ml of THF, and 2.17 g (0.07 mol) of monomethylamine in 15 ml of THF were allowed to react as previously described except that the final reaction was continued for 70 min. The work-up was the same except that the hydrolysis of the carbonate ester was carried out with 50 ml of 1 *N* NaOH solution containing 5 ml of CH_3OH for 70 min at room temperature. This solution was washed four times with CH_2Cl_2 and the aqueous phase was cooled in ice and made acidic to litmus paper with concentrated H_2SO_4. Scratching and cooling overnight gave, in two crops, 5.88 g (64%) of white, crystalline **3b**, mp 189–191°. Sublimation at 190° (0.005 mm) gave the analytical sample: mp 189–191°; ir (Nujol) 6.15 (amide carbonyl), 6.25 and 6.33 μ (1,3-diketone); uv max 257 nm (ϵ 10,760).

Anal. Calcd for $C_9H_{13}NO_3$: C, 59.00; H, 7.15; N, 7.65. Found: C, 58.87; H, 7.04; N, 7.46.

2-Methyl-2-azabicyclo[3.3.1]nonan-7-one (7b). The dione **3b** (5.55 g, 0.030 mol) was refluxed for 24 hr in 25 ml of CH_3OH containing 25 mg of *p*-toluenesulfonic acid. The solution was concentrated and the residue was redissolved in 25 ml of CH_3OH, refluxed for an additional 24 hr, and then concentrated. The residue was dissolved in 200 ml of dry THF and cooled, and solid $LiAlH_4$ (4.43 g, 0.12 mol) was added portionwise with stirring. After stirring and refluxing for 18 hr, the mixture was cooled in an ice bath and treated with 10% NaOH until the white precipitate ceased forming. The organic layer was filtered and the salts were washed several times with THF. The combined filtrate and washings were concentrated, leaving a clear oil which was cooled in an ice bath and treated with 30 ml of 25% sulfuric acid. After stirring at room temperature for 90 min, the mixture was made strongly basic with 10% NaOH, forming a white, oily emulsion. After stirring for an additional 10 min at 0–4°, the mixture was extracted with CH_2Cl_2, and the combined organic layers were washed once with a saturated NaCl solution and concentrated, leaving 4.0 g of crude oil. The oil was purified by filtration through a column (2.3 × 17 cm) of Woelm neutral alumina (75 g, activity grade V) packed in 3:1 $CHCl_3$–ether. The amine **7b** was eluted from the column with the same solvent mixture. Evaporation of the solvent afforded 3.80 g of an oil (one spot on tlc and one peak upon vpc) which, in ether, was treated with picric acid, yielding 7.26 g (63%) of **7b** picrate, mp 224–226° dec from CH_3OH. The free amine **7b** gave the following physical data: ir ($CHCl_3$) 5.94 μ (ketone); nmr δ 2.30 (s, 3, NCH_3); mass spec-

trum[7] m/e (rel intensity) 153 (17), 96 (100, α cleavage of molecular ion followed by a McLafferty rearrangement, loss of CH_3COCH_2, metastable at 60.2), 70 (15, α cleavage followed by a retro Diels–Alder[8] reaction). Recrystallization of **7b** picrate from CH_3OH gave the analytical sample, mp 227–229° dec.

Anal. Calcd for $C_{15}H_{18}N_4O_8$: C, 47.11; H, 4.74; N, 14.65. Found: C, 47.37; H, 4.86; N, 14.62.

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Registry No. 1, 4670-09-1; **2,** 43152-25-6; **3a,** 43152-26-7; **3b,** 43152-27-8; **4a,** 43152-28-9; **7a,** 43152-29-0; **7a HCl,** 43152-30-3; **7b,** 43152-31-4; **7b** picrate, 43152-32-5.

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Synthesis and Acid-Catalyzed Decomposition of *o*-Nitrophenylsulfonyldiazomethane

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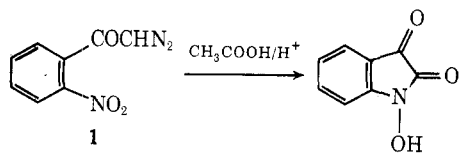
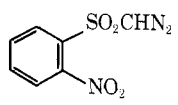
Chemical transformations involving neighboring-group interaction in ortho-substituted nitrobenzene derivatives is a subject of continuing interest.[1] The acid-catalyzed cyclization of *o*-nitrobenzoyldiazomethane (1) to 1-hydroxyisatin (2) is an intriguing example.[2-4] The detailed mechanism of this unusual reaction has not been defined, although ^{14}C labeling has been employed to eliminate some of the possible reaction paths.[5] The initial stages of the reaction likely involve protonation of the diazo ketone function (on carbon) followed by intramolecular nucleophilic attack of the nitro group on the diazonium ion.

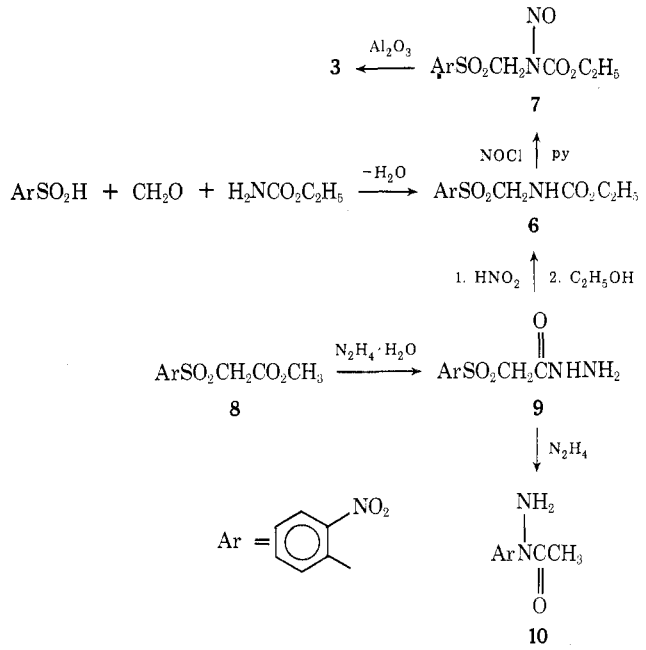


1 2



3

Scheme I



As part of our study of the chemistry of α-diazo sulfones,[6] we wish to describe the synthesis of *o*-nitrophenylsulfonyldiazomethane (3), the sulfonyl analog of **1**, and some reactions of **3** with strong acids.

The two main questions we posed in this study follow: does the nitro group participate in the acid-promoted decomposition of **3** and, if so, are the *o*-nitro substituted compounds[7] **1** and **3** significantly more labile toward acids than their *p*-nitro substituted isomers (p-$NO_2C_6H_4COCHN_2$, **4**; p-$NO_2C_6H_4SO_2CHN_2$, **5**) as a result of this participation?

Results and Discussion

Synthesis. α-Diazo sulfone **3** was prepared by the usual method[8] from the *N*-nitrosocarbamate **7** (Scheme I). Carbamate **6** was obtained by a Mannich-type condensation[9] of *o*-nitrobenzenesulfinic acid with formaldehyde and ethyl carbamate. The low yield (18%) was due to the formation of unidentified by-products and decomposition of the sulfinic acid (at 70°).[10] As an alternative route to **6**, hydrazide **9** was prepared by reaction of ester **8** with hydrazine at −10°. At higher temperatures, hydrazine-catalyzed Smiles rearrangement[11] to **10** is a competing reaction. Diazotization of **9** and Curtius rearrangement gave **6** in 40% yield. Attempts to obtain **6** by oxidation of ethyl *o*-nitrophenylthiomethylcarbamate (**11**) were unsuccessful.

Nitrosation of **6** to **7** gave no problems. The desired **3** was obtained by stirring a solution of **7** with a slurry of active, basic Al_2O_3 in a mixture of dichloromethane and ether. The usual work-up provided **3** in 30–60% yield as yellow crystals: ir ν (N≡N) at 2109 cm^{-1} (CCl_4); nmr ($CDCl_3$) δ (CHN_2) 5.88 ppm;[12] uv, no discrete n → π^* maximum, ϵ 240 at 370 nm in 60% (v/v) dioxane–water. *Unfortunately, the crystalline* **3** *proved to be highly explosive.* Although a number of α-diazo sulfones have been prepared in this laboratory[13] (including *p*-nitrophenylsulfonyldiazomethane), explosive properties have not been encountered before. *Unless appropriate safety precautions are observed, manipulations with solid* **3** *should not be undertaken.* However, dilute solutions of **3** in inert solvents at room temperature or below can be kept for extended periods of time[14] without noticeable decomposition.

Acid-Catalyzed Reactions. Treatment of **3** with HCl in