was treated with 7 (0.23 g, 1.04 mmol), and the resultant mixture was stirred at 0 °C, for 3 h and then at +5 °C for 2 h. The mixture was poured into a well-stirred, cold solution of ammonium chloride and ammonia (5% each, 100 mL) and extracted with ether. Chromatography on silica gel (EtOAc–hexanes, 3:97) afforded **11** (100 mg, 41%), which was eluted first, and **10** (123 mg, 50%) as colorless oils.

Keto ester 10: ^{1}H NMR (300 MHz) δ 4.12 (2 H, q, J = 7 Hz, Et), 2.98 (1 H, m, H5α), 2.44 (1 H, 4 d, J_{1-2} = 14 and 2 Hz, J_{1-8a} = 6 Hz, H1), 2.33 (1 H, d, J_{1-8a} = 6 Hz, H8a), 2.3–1.4 (11 H, m), 1.27 (3 H, J = 7 Hz, Et), 1.06 (3 H, s, Me); an ^{1}H NOE enhancement at δ 2.33 (H8a) was observed upon irradiation at δ 1.06 (Me); ^{13}C NMR δ 209.2, 173.8, 60.0, 59.0, 42.7, 41.2, 40.2, 38.0, 37.2, 28.8, 20.8, 20.7, 18.5, 14.1; IR (neat) 1730 cm^{-1}; HRMS calcd for $C_{14}H_{22}O_3$ m/e 238.1570, found m/e 238.1573.

Ethyl (1α,4aα,8aα)-4a-Methyl-8-oxodecahydronaphthalene-1-carboxylate (11). In addition to the preparation described above, compound **11** was obtained in the two following isomerization reactions.

A. A solution of **10** (100 mg) and p-toluenesulfonic acid (10 mg) in benzene (5 mL) was heated at 55 °C for 15 min, then washed with aqueous $NaHCO_3$ (5%, 3 × 10 mL), and evaporated to give **11** (100 mg, 100%).

B. Crude products **10** and **11** of the addition reaction of Me_2CuLi to **7** were treated with p-toluenesulfonic acid as described above. Chromatography on silica gel (EtOAc–hexanes, 5:95) afforded **11** (91%): ^{1}H NMR (300 MHz) δ 4.07 (2 H, q, J = 7 Hz, Et), 2.90 (1 H, m, H5α), 2.58 (1 H, m, J_{1-2} = 14 and 7.2 Hz, J_{1-8a} = 11.7 Hz, H1), 2.25 (1 H, d, J_{1-8a} = 11.7 Hz, H8a), 2.22–1.15 (14 H, m including t at δ 1.20 with J = 7 Hz for Et), 0.94 (3 H, s, Me); an ^{1}H NOE enhancement was observed at δ 2.25 (H8a) upon irradiation at δ 0.94 (Me); ^{13}C NMR δ 212.6, 174.2, 60.7, 60.6, 42.2, 37.9, 37.8, 30.9, 27.6, 27.1, 22.1, 19.9, 14.1; IR (neat) 1720 cm^{-1}. Anal. Calcd for $C_{14}H_{22}O_3$: C, 70.55; H, 9.31. Found: C, 70.32; H, 9.36.

(1α,4aα,8aα)-1-Isopropenyl-4a-methyl-8-methylenedecahydronaphthalene (14,8,9-Epi-β-gorgonene). Sodium hydride (96 mg, 4 mmol) was reacted with dry DMSO (3 mL) at 70 °C until evolution of hydrogen ceased (1 h). The solution was cooled to 15 °C and treated with a solution of methyltriphenylphosphonium bromide (1.428 g, 4 mmol) in DMSO (3 mL). The mixture was stirred at 35 °C for 15 min, and the resultant red solution of the Wittig reagent was treated with **11** (300 mg, 1.26 mmol) in DMSO (1 mL), and then stirred at 40 °C for 10 h. Quenching with water (10 mL) at 10 °C was followed by extraction with pentane (4 × 25 mL) and then chromatography on silica gel (AcOEt–pentane, 2:98) to afford methylene ester **12** (158 mg, 53%): ^{1}H NMR (60 MHz) δ 4.68 (2 H, m), 4.10 (2 H, q, J = 7 Hz), 2.75 (1 H, m), 2.07 (4 H, m), 1.58 (9 H, m), 1.08 (3 H, t, J = 7 Hz), 0.92 (3 H, s); HRMS calcd for $C_{15}H_{24}O_2$ m/e 236.1777, found m/e 236.1780.

A solution of **12** (120 mg, 0.51 mmol) in ether (5 mL) was treated with methyllithium in ether (1.5 M, 1 mL, 1.5 mmol) at 0 °C for 5 min, and the mixture was stirred at 0 °C for an additional 15 min. Quenching with water (5 mL) was followed by extraction with pentane (3 × 25 mL) to give tertiary alcohol **13** (113 mg, 100%): HRMS calcd for $C_{15}H_{24}$ ($M^+ - H_2O$) m/e 204.1879, found 204.1882; HRMS calcd for $C_{14}H_{21}$ ($M^+ - H_2O - CH_3$) m/e 189.1644, found 189.1642.

Freshly distilled thionyl chloride (0.24 mL) was added dropwise to a stirred solution of **13** (100 mg, 0.45 mmol) in dry pyridine (4 mL) at −15 °C. The mixture was stirred at 0 °C for 1 h, then poured (via syringe) onto ice, and extracted with pentane. Removal of pyridine from the extract by washing with hydrochloric acid (2 N) was followed by chromatography on silica gel (pentane) to give **14** (72 mg, 78%) as a colorless oil: ^{1}H NMR (300 MHz) δ 4.67 (1 H, m, C8=CH$_2$), 4.61 (2 H, br s, C1C=CH$_2$), 4.51 (1 H, m, C8=CH$_2$), 2.46 (1 H, 3 d, J_{1-2} = 3.3 and 11.7 Hz, J_{1-8a} = 11.7 Hz, H1), 2.15–1.90 (3 H, m), 1.80 (1 H, d, J_{1-8a} = 11.7 Hz, H8a), 1.70–1.16 (11 H, m including s at δ 1.60 for C1CMe), 1.00–0.90 (1 H, m), 0.90 (3 H, s, C4aMe); an ^{1}H NOE enhancement (10%) was observed at δ 1.80 (H8a) but not at δ 2.46 (H1) upon irradiation at δ 0.90 (C4aMe), irradiation at δ 1.80 gave no NOE signal at δ 2.46; ^{13}C NMR δ 149.0, 148.9, 110.1, 109.4, 55.1, 45.3, 40.8, 34.7, 32.4, 30.4, 30.3, 28.6, 23.5, 21.7, 18.0. Anal. Calcd for $C_{15}H_{24}$: C, 88.16; H, 11.84. Found: C, 88.03; H, 11.88.

Acknowledgment. We thank the Instrument Program, Chemistry Division, National Science Foundation for financial assistance toward the purchase of the 300-MHz spectrometer.

Registry No. (±)-**1a**, 90545-43-0; (±)-**1b**, 112400-22-3; (±)-**2**, 112400-19-8; (±)-**3**, 112400-20-1; (±)-**4**, 112400-21-2; **5a**, 112400-23-4; **5b**, 112400-26-7; **6a**, 112421-60-0; **6b**, 112400-27-8; (±)-**7**, 112400-25-6; (±)-**8**, 112400-24-5; (±)-**9**, 112400-28-9; (±)-**10**, 112400-30-3; (±)-**11**, 112400-29-0; (±)-**12**, 112400-31-4; (±)-**13**, 112400-32-5; (±)-**14**, 51260-30-1; ClCOCH=CH$_2$, 814-68-6; (*E*)-HO$_2$CCH=CHSO$_2$Ph, 711-29-5; (*E*)-ClCOCH=CHSO$_2$Ph, 112421-59-7; EtO$_2$CCH=CH$_2$, 140-88-5; Ph$_3$PMe$^+$Br$^-$, 1779-49-3.

Convenient Preparation of 11*H*-Benzo[*a*]fluorenone and 11*H*-Benzo[*b*]fluorenone

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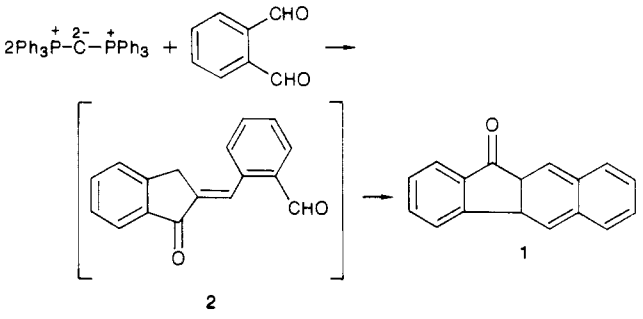
Received March 5, 1987

Benzofluorenones and their corresponding hydrocarbons have achieved recent significance because of their widespread occurrence in particulate matter from combustion of organic materials.[1] The hydrocarbons are useful as indicators in the determination of carbon acidities.[2] Commercial availability of the hydrocarbons has been variable and such samples have sometimes been impure but alternative syntheses have generally been rather lengthy. Thus, we were struck by Bestmann's report that 11*H*-benzo[*b*]fluorenone (**1**) is produced by the reaction of hexaphenylcarbodiphosphorane with phthalaldehyde.[3] On working out a possible mechanism for this reaction during a research group meeting it appeared that **2** is a reasonable intermediate in this reaction. **2** should also



be available even more readily from the chalcone condensation of 1-indanone with phthalaldehyde. In the ev-

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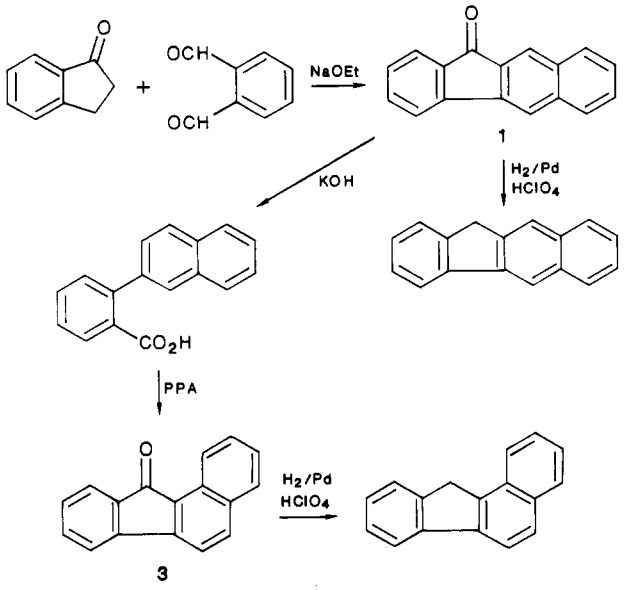
Scheme I



ent, the condensation proceeded readily with ethanolic sodium ethoxide at room temperature. Subsequent heating led to the second ring closure and gave 1 in good yield.

With a simple synthesis of 1 at hand, we considered ways to accomplish a net isomerization to other benzofluorenones. A Haller–Bauer reaction was expected to cleave the naphthyl–carbonyl bond preferentially because the resulting naphthyl anion is more stable than the alternative phenyl anion.[4] Friedel–Crafts ring closure of the resulting carboxylic acid derivative would then lead to 11*H*-benzo[*a*]fluorenone (3). The Haller–Bauer ring opening of 1 was not successful in our hands but the corresponding cleavage reaction with potassium hydroxide[5] worked well to give primarily *o*-2-naphthylbenzoic acid (4). Subsequent reaction with polyphosphoric acid gave 3. Both ketones were readily hydrogenated to the corresponding benzofluorenes (Scheme I).

The reaction sequence is straightforward and should be capable of generalization to other fluorene derivatives, and particularly some of the dibenzofluorenes. The isomerization method, however, is limited to carbanion-stabilizing substituents in order to promote preferential cleavage of the carbonyl bond to the substituted ring; few such substituents are stable to the strongly basic conditions required for the carbonyl cleavage step.

Experimental Section

11*H*-Benzo[*b*]fluorenone (1). A solution of sodium ethoxide prepared from 0.64 g (0.028 mol) of sodium metal and 40 mL of absolute ethanol was added to a solution of 18.69 g (0.139 mol) of phthalaldehyde (Aldrich) in 400 mL of absolute ethanol. A solution of 17.32 g (0.131 mol) of 1-indanone (Aldrich) in 300 mL of absolute ethanol was then added dropwise, and the mixture was stirred overnight. The deep red solution was heated at reflux for an additional 24 h to complete the second condensation. Slow cooling produced copious amounts of a yellow solid, which was filtered. Removal of solvent from the filtrate by rotary evaporation left a viscous dark brown residue, which was shaken with ether and water. Insoluble material was separated, and the organic layer was washed several times with water and aqueous sodium chloride and dried over magnesium sulfate, filtered, and evaporated to give fluffy yellow solids. The two crops were recrystallized separately from ethanol to give a total of 15.1 g (50.2% yield) of finely powdered deep yellow crystals, mp 152 °C (lit.[6] mp 153–154.5 °C); ^{1}H NMR (200 MHz) δ 7.0–8.25 (m). Anal. $C_{17}H_{20}O$; C, H.

***o*-2-Naphthylbenzoic Acid (4).** Following the method of Kenner, Robinson, Tylor, and Webster,[5] a mixture of 7.46 g (0.032 mol) of 1, 21 g of finely powdered KOH, and nine clean glass beads in 80 mL of toluene was heated to reflux with vigorous stirring for 4 h. The opaque brown solution was cooled and poured into water. Insoluble yellow-brown material and some residual KOH adhered to the flask and was dissolved with a little water and additional base and added to the workup mixture. The organic layer was washed several times with 10% aqueous KOH. A TLC of this washed toluene solution showed only traces of unreacted 2,3-benzofluorenone. The cloudy orange aqueous solution was acidified with 6 N HCl, and the white solid produced was taken up in ether. The washed and dried solution was filtered and evaporated. The solid product was recrystallized from carbon tetrachloride to yield 3.56 g (45%) of 4 as a finely divided pale yellow solid, mp 185 °C (lit.[8] mp 185–186 °C), and 2.32 g of 3-phenyl-2-naphthoic acid, mp 159–163 °C (lit.[9] mp 165–166 °C) as a separate crop after the crystalline 4 was filtered and the mother liquor was concentrated (total recovery 74%): ^{1}H NMR (200 MHz) δ 7.25–7.98 (m, 11 H), 8.53 (s, 1 H). Anal. $C_{17}H_{12}O_2$: C, H.

11*H*-Benzo[*a*]fluorenone (3). A mixture of 46 g of commercial polyphosphoric acid and 2.02 g of *o*-2-naphthylbenzoic acid was stirred together thoroughly and heated slowly to 75 °C. During this time some of the organic acid dissolved and the suspension became a deep brown-black. The mixture was maintained at this temperature for 4 h with occasional stirring. At the end of this time, all of the organic acid had dissolved, and the solution was a uniform black. The cooled reaction mixture was stirred with ice and water, and the bright yellow suspension of solids was taken up in ether. The washed and dried extract was filtered and evaporated to give an orange solid. The basic washes were combined and acidified to give unreacted starting material which was taken up in ether, washed, dried, and evaporated to give 0.75 g of 4. The product 3 was recrystallized from methanol to yield 1.16 g (62% yield, 98% conversion) of bright orange needles, mp 132 °C (lit.[10] mp 132 °C). Anal. $C_{17}H_{10}O$: C, H.

This appears to be a slow but clean reaction. A reaction run at 100 °C gave no unreacted starting material but a greater amount of polymer and a yield of only 71%.

Hydrogenolysis of Benzofluorenones. The benzofluorenones were hydrogenolyzed by a two-step procedure. Initial hydrogenation was accomplished by stirring an ethanol or methanol solution overnight with 5% palladium on carbon and atmospheric hydrogen. The reaction was monitored by using TLC, which showed complete and clean conversion of the ketone to the corresponding alcohol. The yellow color of the ketone had faded completely. Ten drops of perchloric acid were then added, and hydrogenation was continued overnight. TLC showed that the reaction was complete, with no appearance of base line materials. The mixture was filtered through a frit protected with a layer of Celite to remove the catalyst, the solution was concentrated, and the product was allowed to crystallize directly.

Benzo[*b*]fluorene: sparkly white plates, mp 208 °C (lit.[7] mp 208.5–209.5 °C); ^{1}H NMR (200 MHz) δ 4.05 (s, 2 H), 7.2–8.2 (m, 10 H). Anal. $C_{17}H_{12}$: C, H.

Benzo[*a*]fluorene: off-white crystals, mp 177 °C. Sublimation gave a fine white powder, mp 177 °C (lit. mp 185.4–186 °C,[11] 191.5–192 °C[7]); ^{1}H NMR (200 MHz) δ 4.17 (s, 2 H), 7.24–8.03 (m, 10 H). Anal. $C_{17}H_{12}$: C, H.

The NMRs are identical with those of authentic samples used as carbon acidity indicators in our laboratory. The visible spectra of the cesium salts in THF also coincide.[12]

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Acknowledgment. This research was supported in part by NSF Grant CHE85-02137. Preliminary experiments were carried out by Michael Dahlberg.

Registry No. 1, 3074-03-1; 3, 479-79-8; 4, 5693-33-4; 1-indanone, 83-33-0; phthalaldehyde, 643-79-8; 11*H*-benzo[*b*]fluorene, 243-17-4; 11*H*-benzo[*a*]fluorene, 238-84-6.

Preparation of β-Keto Sulfones from [(Phenylsulfonyl)methylene]dilithium and Acid Chlorides

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Received August 4, 1987

As a class of compounds β-keto sulfones provide a range of synthetic versatility.[1-5] The β-keto sulfone moiety may be prepared from a variety of precursors; e.g., from cyano sulfones via the Thorpe reaction,[6] by oxidation of sulfides or sulfoxides,[7] or from esters through Claisen condensations.[8] We have prepared several new β-keto sulfones by a significantly more convenient method. We describe this method herein and report for the first time reactions that yield β-keto sulfones from acid chlorides and [(phenylsulfonyl)methylene]dilithium (Scheme I).

Scheme I

$$C_6H_5SO_2CHLi_2 \xrightarrow{RCOCl} \xrightarrow{NH_4Cl} C_6H_5SO_2CH_2COR$$

R = cyclopropyl, isopropyl, ethyl, methyl, cyclohexyl, phenyl

The dianion of methyl phenyl sulfone is prepared by treating the sulfone with 2.2 equiv of *n*-butyllithium in dry tetrahydrofuran (THF) at −30 °C.[9] The resulting yellow suspension is stirred for 30 min, after which the acid chloride is added slowly to the dianion. The resulting enolate is subsequently quenched with saturated ammonium chloride solution and then extracted with chloroform. The organic extract is dried, filtered, and evaporated. The crude β-keto sulfone is recrystallized from a suitable solvent. The yields and melting points for the products of a series of representative reactions are given in the Experimental Section.

The reactions of acid chlorides with the monoanion of the sulfone were not efficient. Once the β-keto sulfone is formed under these conditions it presumably is quickly deprotonated by any unreacted [(phenylsulfonyl)methyl]lithium; therefore, the maximum yield of β-keto sulfone would be 50%. Since the reaction of the sulfone dianion with acid chloride directly gives the enolate, this undesirable internal quenching is avoided.

Although geminal dilithio derivatives of alkyl sulfones do react with esters to produce β-keto sulfones,[10-12] the analogous reactions performed with acid chlorides are simpler and cleaner. When esters are used in this type of reaction, the use of either tetramethylethylenediamine (TMEDA) or hexamethylphosphoramide (HMPA) is often required. The use of these cosolvents is not necessary when acid chlorides are employed. Additional advantages of our method over others include higher yields, greater ease of product isolation, and minimal synthetic steps. For example, the average yield of β-keto sulfone from Claisen condensations is only 27%,[8] while the present method affords yields from 36% to 74%.

Experimental Section

General Procedure. Infrared (IR) spectra were recorded as KBr pellets or mineral oil mulls on either a Perkin-Elmer 983 or a Perkin-Elmer 1310 spectrophotometer. Nuclear magnetic resonance (NMR) experiments were performed by using a JEOL FX-90Q for ^{13}C at 22.45 MHz and ^1H at 89.55 MHz by using $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the reference. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN. Melting points are uncorrected.

All acid halides were distilled prior to use. THF was distilled from sodium benzophenone ketyl under nitrogen immediately before use. Methyl phenyl sulfone (Parish Chemical) was used without further purification. Butyllithium (1.6 M in hexane) was obtained from Aldrich Chemical Company.

Typical Procedure. 1-Cyclopropyl-2-(phenylsulfonyl)-ethanone. Butyllithium (18 mL of 1.6M solution, 28 mmol) was added slowly with efficient stirring under dry nitrogen to a solution of methyl phenyl sulfone (2.0 g, 13 mmol) in 40 mL of anhydrous THF at −30 °C. After 30 min, cyclopropanecarboxylic chloride (1.6 g, 15 mmol) was slowly added by syringe so as to minimize the temperature increase that results from the exothermic reaction. Subsequently, the reaction mixture was poured into 150 mL of saturated NH_4Cl solution and stirred. The β-keto sulfone was extracted with chloroform and the organic phase was washed with saturated NaCl solution, dried with anhydrous magnesium sulfate, filtered, and evaporated. The 1-cyclopropyl-2-(phenylsulfonyl)ethanone was recrystallized from CCl_4: recrystallized yield 74%; mp 58 °C (lit.[13] mp 56 °C). [Alternatively, this β-keto sulfone may be recrystallized from water.] ^1H NMR ($CDCl_3$), ppm: 0.6–1.0 (m, 4 H), 1.8–2.2 (m, 1 H), 4.0 (s, 2 H), 7.0–8.0 (m, 5 H). ^{13}C NMR ($CDCl_3$), ppm: 13.2 (t), 22.0 (d), 68.0 (t), 128.2 (d), 129.2 (d), 134.1 (d), 138.7 (s), 198.2 (s). IR (cm^{-1}) 3012, 1695, 1585, 1477, 1445, 1384, 1323, 1155, 1087, 1060, 1024, 898. Anal. Calcd for $C_{11}H_{12}SO_3$: C, 58.91; H, 5.39; S, 14.30. Found: C, 58.67; H, 5.43; S, 14.61.

3-Methyl-1-(phenylsulfonyl)-2-butanone: recrystallized yield from CCl_4 43%; mp 66.5–67 °C. ^1H NMR ($CDCl_3$), ppm: 1.1 (d, 6 H), 2.9 (septet, 1 H), 4.3 (s, 2 H), 7.5–8.0 (m, 5 H). ^{13}C NMR ($CDCl_3$), ppm: 17.2 (q), 41.7 (d), 64.2 (t), 128.0 (d), 129.0 (d), 133.9 (d), 138.8 (s), 201.7 (s). IR (cm^{-1}) 3040, 2935, 1680, 1450, 1375, 1290, 1140, 740, 680. Anal. Calcd for $C_{11}H_{14}SO_3$: C, 58.38; H, 6.24; S, 14.17. Found: C, 58.46; H, 6.25; S, 14.58.

1-(Phenylsulfonyl)-2-butanone: recrystallized yield from CCl_4 60%; mp 44–45 °C. ^1H NMR ($CDCl_3$), ppm: 1.1 (t, 3 H), 2.7 (q, 2 H), 4.2 (s, 2 H), 7.7–7.9 (m, 5 H). ^{13}C NMR ($CDCl_3$), ppm: 6.7 (q), 37.1 (t), 65.7 (t), 127.6 (d), 128.8 (d), 133.7 (d), 138.4 (s), 198.4 (s). IR (cm^{-1}) 3045, 3000, 2960, 2920, 1705, 1575, 1445, 1300, 1140, 740, 680. Anal. Calcd for $C_{10}H_{12}SO_3$: C, 56.59; H, 5.70; S, 15.10. Found: C, 56.79; H, 5.75; S, 15.32.

1-(Phenylsulfonyl)-2-propanone: recrystallized yield from CCl_4 36%; mp 52–54 °C. ^1H NMR ($CDCl_3$), ppm: 2.4 (s, 3 H), 4.2 (s, 2 H), 7.7–7.9 (m, 5 H). ^{13}C NMR ($CDCl_3$), ppm: 31.6 (q),

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