

## Application of [Hydroxy(tosyloxy)iodo]benzene in the Wittig-Ring Expansion Sequence for the Synthesis of $\beta$ -Benzocycloalkenones from $\alpha$ -Benzocycloalkenones

Michael W. Justik and Gerald F. Koser\*

Department of Chemistry, The University of Akron, Akron, Ohio, 44325. USA. Tel.: (+1) 330 972-6066, Fax: (+1) 330 972-7370.

\* Author to whom correspondence should be addressed; e-mail [koser@uakron.edu](mailto:koser@uakron.edu)

Received: 5 July 2004 / Accepted: 14 July 2004 / Published: 31 January 2005

---

**Abstract:** The conversion of  $\alpha$ -benzocycloalkenones to homologous  $\beta$ -benzocycloalkenones containing six, seven and eight-membered rings is reported. This was accomplished *via* a Wittig olefination-oxidative rearrangement sequence using [hydroxy(tosyloxy)iodo]-benzene (HTIB) is the oxidant, that enables the synthesis of regioisomeric pairs of methyl-substituted  $\beta$ -benzocycloalkenones. The incorporation of carbon-13 at C-1 of the  $\beta$ -tetralone nucleus was also demonstrated. The Wittig-HTIB approach is a useful alternative to analogous sequences in which  $Tl(NO_3)_3 \cdot 3H_2O$  or the Prevost combination ( $AgNO_3/I_2$ ) are employed in the oxidation step.

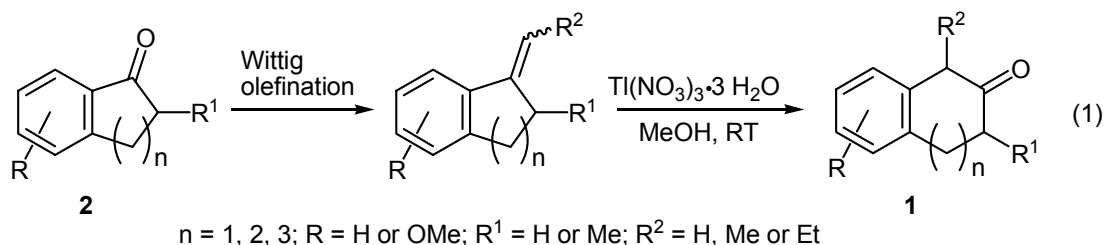
**Keywords:** [Hydroxy(tosyloxy)iodo]benzene, benzocycloalkenones, ring expansion, oxidative rearrangement

---

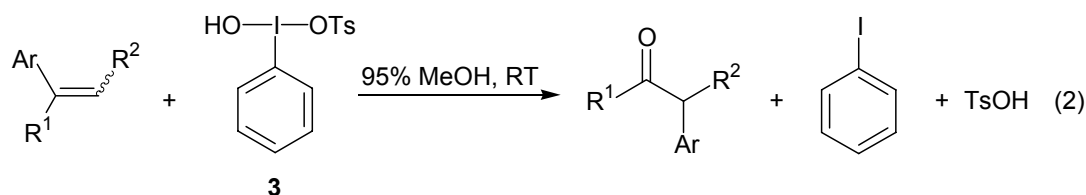
### Introduction

Synthetic access to the  $\beta$ -benzocycloalkenones and their ring-substituted derivatives, **1**, was facilitated by the 1977 publication of a two-step protocol involving Wittig olefination of  $\alpha$ -benzocycloalkenones, **2**, and oxidative ring expansion of the resulting alkenes with thallium(III) nitrate in methanol [1]. This procedure, exemplified by equation 1, enables the regiospecific placement of alkyl groups at the  $\alpha$ -carbon atoms of the  $\beta$ -cycloalkenone ring, and affords dimethyl ketals when trimethyl orthoformate is employed as a co-solvent. It was later demonstrated that thallium(III) nitrate can be replaced with the Prevost combination,  $AgNO_3$  and  $I_2$ , for two-step conversions of  $\alpha$ -tetralones ( $n = 2$ )

to  $\beta$ -benzocycloalkenones [2], although the experimental procedure is a bit less convenient than the thallium nitrate method.



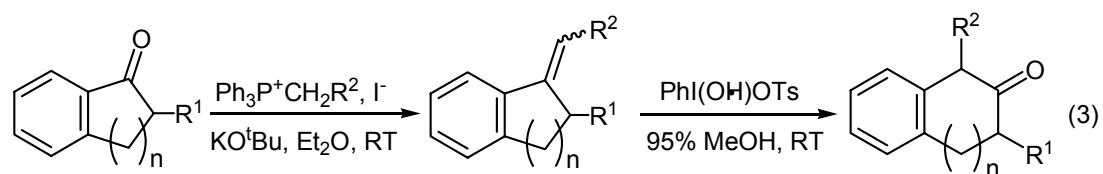
We have recently reported that the treatment of arylalkenes with [hydroxy(tosyloxy)iodo]benzene (**3**, HTIB) in 95% methanol provides a general, regioselective synthesis of  $\alpha$ -aryl ketones (equation 2) [3]. This oxidative rearrangement is fundamentally equivalent to the ring-expansion step depicted in eq. 1, and we now report that HTIB is an excellent alternative to  $Tl(NO_3)_3 \cdot 3H_2O$  or  $AgNO_3/I_2$  for the two-step synthesis of  $\beta$ -benzocycloalkenones from their lower  $\alpha$ -benzocycloalkenone homologs. An obvious advantage of HTIB is its relatively benign character in comparison to  $Tl(NO_3)_3 \cdot 3H_2O$  and  $AgNO_3$  [4].



## Results and Discussion

Syntheses of the exocyclic alkenes and  $\beta$ -benzocycloalkenones shown in Table 1 were accomplished as indicated in equation 3. The olefination procedure was adapted from the Fitjer-Quabeck approach, wherein potassium *tert*-butoxide is utilized as the Wittig base in  $Et_2O$  or benzene [5]. More specifically, the  $\alpha$ -benzocycloalkenones were added to a pre-stirred mixture of potassium *tert*-butoxide and the appropriate alkyltriphenylphosphonium iodide in  $Et_2O$  at room temperature. After approximately 4 h, the reaction mixtures were filtered through Celite® and concentrated. Passage of the residual material through silica gel with hexanes gave the exocyclic alkenes (characterized by  $^1H$ -NMR) which were used without further purification.

The oxidative ring-expansions were effected by the addition of crystalline HTIB (10 mmol) to a small excess of the alkene in 95% MeOH (mildly exothermic) at room temperature. After about 20 minutes and a preliminary aqueous workup, the resulting  $\beta$ -benzocycloalkenones were isolated by column chromatography in yields (from the alkenes) of 80 to 99%.



**Table 1.** Oxidative ring expansions of alkylidenebenzocycloalkenes to  $\beta$ -benzocycloalkenones with HTIB in 95% MeOH

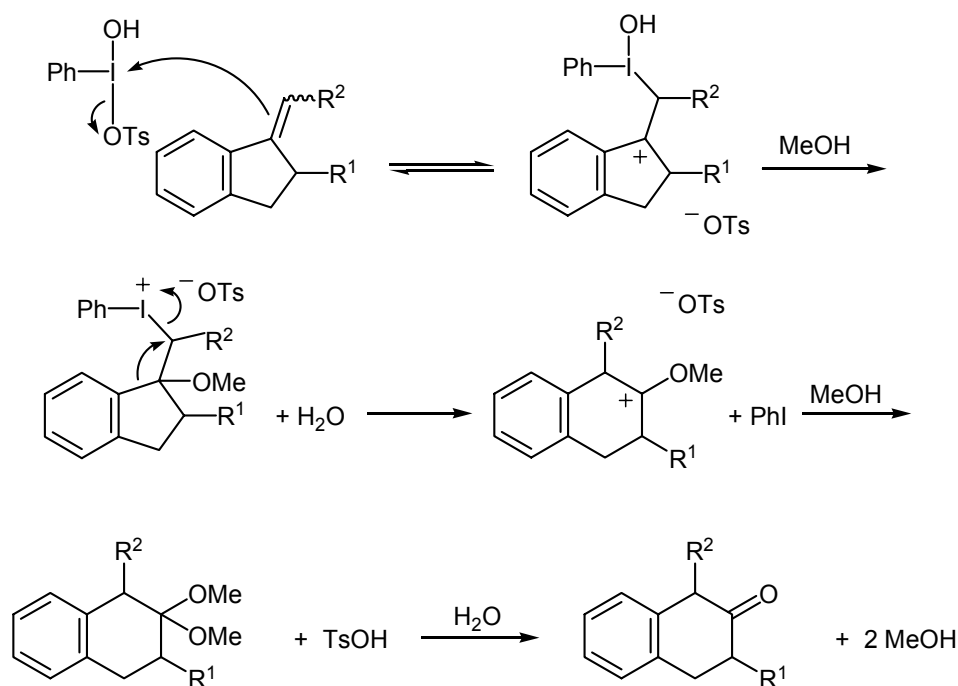
Entry	Alkene	Alkene Yield, %	Product	Isolated Yield, %
1		26		94
2		66		99
3		91		87
4		67		82
5		62		92
6		90		80
7		60		85
8		69		91

As indicated in Table 1 (entries 1-3), conversions of unsubstituted  $\alpha$ -benzocycloalkenones to homologous  $\beta$ -benzocycloalkenones containing six, seven and eight-membered rings, *via* the Wittig-HTIB sequence, were demonstrated. Furthermore, as with the Tl(III)-induced rearrangements, the proper selection of substrates enables syntheses of regioisomeric pairs of methyl-substituted  $\beta$ -benzocycloalkenones (cf. entries 4-7). For example, the treatment of 2-methyl-1-methylideneindan with HTIB gave 3-methyl-2-tetralone (entry 4), while similar treatment of 1-ethylideneindan gave 1-methyl-2-tetralone (entry 5).

The Wittig-HTIB sequence was also employed for incorporation of a  $^{13}\text{C}$ -label into the  $\beta$ -tetralone nucleus (entry 8). To this end,  $^{13}\text{C}$ -methyltriphenylphosphonium iodide was prepared from  $^{13}\text{C}$ -labeled iodomethane and utilized for the olefination of 1-indanone. Exposure of 1-( $^{13}\text{C}$ -methylidene)indan (5.0 mmol) to HTIB (4.54 mmol) in 95% MeOH (25 mL) gave 1- $^{13}\text{C}$ -2-tetralone in 91% isolated yield. The location of the label at C-1 in the  $\beta$ -tetralone ring was clearly revealed by NMR analysis. Thus, the singlet at  $\delta$  3.58 in the  $^1\text{H}$ -spectrum of unlabeled  $\beta$ -tetralone appears as a doublet at  $\delta$  3.62 ( $J_{\text{CH}} = 128.6$  Hz) in the  $^1\text{H}$  spectrum of the  $^{13}\text{C}$ -isotopomer. A  $^{13}\text{C}$ -NMR spectrum of the labeled compound, recorded after only 16 scans, exhibits a markedly enhanced singlet at  $\delta$  45.03, while the resonances of the remaining carbons are either very weak in comparison or not perceptible.

A plausible mechanism for the HTIB-induced ring-expansions reported herein is presented in Scheme 1. It is analogous to that proposed for the oxidative rearrangement of arylalkenes to  $\alpha$ -aryl ketones [3], and accounts for the regiochemistry of  $\beta$ -benzocycloalkenone formation. The similarity between iodine(III) and thallium(III) reagents in the context of oxidative rearrangements has been reviewed by Prakash [6] and almost certainly originates from their electrophilic character and capacity for reduction to the respective iodine(I) and thallium(I) oxidation states.

Scheme 1.



## Conclusions

In summary, the Wittig-HTIB sequence is a useful method for regiospecific syntheses of  $\beta$ -benzocycloalkenones from  $\alpha$ -benzocycloalkenones, and is environmentally preferable to similar protocols based on Tl(III) and Ag(I) reagents.

## Experimental

### General

NMR spectra were recorded on a Varian model Gemini-300 spectrometer at resonance frequencies of 300 ( $^1\text{H}$ -) and 75 ( $^{13}\text{C}$ -) MHz. The NMR solvent in all cases was  $\text{CDCl}_3$ ; chemical shifts are expressed relative to residual  $\text{CHCl}_3$  ( $^1\text{H}$  spectra) or to  $\text{CDCl}_3$  ( $^{13}\text{C}$ - spectra). Multiplets in  $^1\text{H}$ -NMR spectra are sometimes specified with a range of chemical shifts corresponding to the highest and lowest lines in the multiplet. IR spectra were recorded on a Bomem MB-100 FT-IR spectrophotometer. IR samples were neat films or a Nujol® mull (Entry 4). The elemental analysis was performed by Midwest Microlab, LTD (Indianapolis, IN). Melting points were recorded on a Thomas-Hoover Unimelt melting point apparatus and are uncorrected. 2-Methyl-1-indanone and 2-methyl-1-tetralone (used for preparation of the alkenes in entries 4 and 6) were prepared by adaptation of literature methods [7, 8]. All other solvents and chemical reagents were obtained from commercial sources and used as received. Flash column chromatography was performed on a 42 mm internal diameter column packed with Kieselgel (230-400 mesh) silica gel purchased from the Aldrich Chemical Company. Thin layer chromatography (TLC) was done with glass backed 250 micron silica gel plates containing a fluorescent indicator and purchased from Alltech. The 95% methanol used as the solvent for the treatment of the substrate alkenes with [hydroxy(tosyloxy)iodo]benzene (HTIB), refers to a 95:5 (v/v) mixture of methanol and water. Consumption of the oxidant was verified prior to work-up by adding a drop of the reaction mixture to 10% KI (aq.) solution. Yields are based on the material used for the spectroscopic data presented in this paper.

### Preparation of Hypervalent Iodine Reagents

#### Typical Synthesis of (Diacetoxyiodo)benzene

A solution of 32% (w/v) peracetic acid (100 mL, 421 mmol) in acetic acid was added dropwise with mechanical stirring to a cooled flask (15 °C) containing iodobenzene (65.28 g, 320.0 mmol), over a period of 1 hour. The rate of addition was adjusted to keep the temperature of the reaction mixture between 25-30 °C. Mechanical stirring was continued for 4 hours during which time a white precipitate separated. Water (100 mL) was added to facilitate precipitation and to dilute any remaining oxidant. The solid was isolated by vacuum filtration, washed with water (2 x 100 mL) and ether (150 mL), dried over  $\text{P}_2\text{O}_5$  in a vacuum desiccator overnight and identified as (diacetoxyiodo)benzene; yield 94.86 g (92%); mp 160-161 °C (lit. [9] mp 159-161 °C).

*Typical Synthesis of [Hydroxy(tosyloxy)iodo]benzene (HTIB, 3)*

A solution of (diacetoxyiodo)benzene (40.26 g 125.0 mmol) in boiling acetonitrile (120 mL) was added at once to a solution of *p*-toluenesulfonic acid monohydrate (24.72 g, 130.0 mmol) in boiling acetonitrile (80 mL), with magnetic stirring, to give a yellow-green solution. The solution was allowed to cool to room temperature, and the crystalline [hydroxy(tosyloxy)iodo]benzene that separated was isolated by vacuum filtration and washed with acetonitrile (100 mL) and ether (100 mL); yield, 45.39 g (93%); mp 136-7 °C, dec. (lit.[10] mp 136-138.5 °C).

*Representative Procedure - Reaction of HTIB with 1-Methylideneindan (4) (Entry 1).*

Crystalline HTIB (3.92 g, 10.0 mmol) was added to a stirred solution of 1-methylideneindan (1.43 g, 11.0 mmol) in 95% methanol (40 mL). The solid dissolved rapidly (~15 sec) with the evolution of heat (41 °C) to give a colorless solution. The solution was stirred at room temperature for 20 minutes and the solvent was removed *in vacuo* to give an oily mixture. This mixture was partitioned between CH<sub>2</sub>Cl<sub>2</sub> (40 mL) and H<sub>2</sub>O (25 mL) and transferred to a separatory funnel. The organic layer was separated, washed with H<sub>2</sub>O (2 x 25 mL) and brine (1 x 30 mL), dried over MgSO<sub>4</sub>, and concentrated *in vacuo* to a bright yellow oil (1.51 g), which was subjected to flash column chromatography on silica gel (hexanes; 5 % ethyl acetate/hexanes) to give  $\beta$ -tetralone (**5**) as a light yellow oil ( $R_f = 0.29$ , 5 % ethyl acetate/hexanes); yield, 1.38 g (94 %); <sup>1</sup>H-NMR  $\delta$  2.54 (t,  $J = 6.6$ , 2H), 3.06 (t,  $J = 6.6$ , 2H), 3.58 (s, 2H), 7.11-7.15 (m, 1H), 7.20-7.24 (m, 3H); <sup>13</sup>C-NMR  $\delta$  28.13, 37.95, 44.91, 126.80, 126.87, 127.58, 128.19, 133.28, 136.71, 210.71; IR (C=O) 1719 cm<sup>-1</sup>.

*Summary of Purification, Yield and Spectral Data for  $\beta$ -benzocycloalkenones (Entries 2-7)**Conversion of Alkene 6 to Ketone 7 (Entry 2)*

Flash column chromatography of the oil on silica gel (petroleum ether; 10% ethyl acetate/petroleum ether) gave 2-benzosuberone (**7**) as a colorless oil; yield, 1.58 g (99%); <sup>1</sup>H-NMR  $\delta$  1.99 (m, 2H), 2.57 (dd,  $J = 6.9$ , 6.9, 2H), 2.95 (m, 2H), 3.73 (s, 2H), 7.14-7.22 (m, 4H); <sup>13</sup>C-NMR  $\delta$  26.11, 32.85, 43.56, 50.04, 127.14, 127.59, 129.24, 129.60, 133.61, 140.51, 208.90; IR (C=O) 1710 cm<sup>-1</sup>; 2,4-dinitrophenylhydrazone, mp 174-176 °C (lit. [11] mp 169-170 °C).

*Conversion of Alkene 8 to Ketone 9 (Entry 3)*

Flash column chromatography of the oil on silica gel (hexanes; 5% ethyl acetate/hexanes) gave 7,8,9,10-tetrahydro-5H-benzocyclooctan-6-one (**9**) as a colorless oil ( $R_f = 0.32$ , 5% ethyl acetate/hexanes); yield, 1.51 g (87%); <sup>1</sup>H-NMR  $\delta$  1.76 (m, 2H), 1.87 (m, 2H), 2.35 (m, 2H), 2.84 (m, 2H), 3.82 (s, 2H), 7.14-7.30 (m, 3H) [12]; <sup>13</sup>C-NMR  $\delta$  24.63, 31.14, 32.98, 40.87, 48.58, 126.73, 127.99, 129.98, 130.27, 133.66, 141.13, 212.05; IR (C=O) 1700 cm<sup>-1</sup>; oxime, mp 128-129 °C (lit.[13] mp 130-131 °C).

*Conversion of Alkene 10 to Ketone 11 (Entry 4)*

Flash column chromatography on silica gel (5% ethyl acetate/hexanes; 10% ethyl acetate/hexanes) gave a yellow oil ( $R_f = 0.78$ , 10% ethyl acetate in hexanes). Kugelrohr distillation of this oil gave *3-methyl-2-tetralone* (**11**) as white rosettes; yield, 1.31 g (82%); mp 38-40 °C (lit. [14] mp 37-40 °C);  $^1\text{H-NMR}$   $\delta$  1.20 (d,  $J = 6.9$ , 3H), 2.54 (m, 1H), 2.84 (dd,  $J = 15.4$ , 11.0, 1H), 3.08 (dd,  $J = 15.4$ , 5.8, 1H), 3.61 (s, 2H), 7.12-7.26 (m, 4H);  $^{13}\text{C-NMR}$   $\delta$  14.89,  $\delta$  36.73,  $\delta$  42.31,  $\delta$  44.07,  $\delta$  126.76,  $\delta$  126.87,  $\delta$  127.80,  $\delta$  128.13,  $\delta$  133.51,  $\delta$  136.24,  $\delta$  212.28; IR (C=O) 1722  $\text{cm}^{-1}$ .

*Conversion of Alkene 12 to Ketone 13 (Entry 5)*

Flash column chromatography of the oil on silica gel (hexanes; 5% ethyl acetate/hexanes) gave *1-methyl-2-tetralone* (**13**) as a colorless oil ( $R_f = 0.24$ , 5% ethyl acetate/hexanes); yield, 1.48 g (92%);  $^1\text{H-NMR}$   $\delta$  1.48 (d,  $J = 6.9$ , 3H), 2.43-2.69 (overlapping m's, 2H), 3.08 (m, 2H), 3.54 (quartet,  $J = 6.9$ , 1H), 7.21-7.29 (m, 4H);  $^{13}\text{C-NMR}$   $\delta$  13.74, 27.70, 36.82, 47.12, 125.72, 126.36, 126.73, 127.18, 136.56, 137.63, 211.83; IR (C=O) 1714  $\text{cm}^{-1}$ ; Anal. Calcd. for  $\text{C}_{11}\text{H}_{12}\text{O}$ : C: 82.46%, H: 7.55 %, Found: C 82.35 %, H 7.60%.

*Conversion of Alkene 14 to Ketone 15 (Entry 6)*

Flash column chromatography of the oil on silica gel (hexanes; 5% ethyl acetate/hexanes) gave *3-methyl-2-benzosuberone* (**15**) as a colorless oil ( $R_f = 0.27$ , 5% ethyl acetate/hexanes); yield, 1.40 g (80%);  $^1\text{H-NMR}$   $\delta$  1.03 (d,  $J = 6.3$ , 3H), 1.56 (m, 1H), 2.13 (m, 1H), 2.80 (m, 1H), 2.92 (m, 2H), 3.72 (AB doublet pair,  $J = 54.9$ , 15.4, 2H), 7.14-7.26 (m, 4H);  $^{13}\text{C-NMR}$   $\delta$  10.80, 27.94, 30.75, 41.69, 45.15, 122.57, 122.98, 124.46, 125.00, 129.39, 136.17, 206.11; IR (C=O) 1708  $\text{cm}^{-1}$ .

*Conversion of Alkene 16 to Ketone 17 (Entry 7)*

Flash column chromatography of the oil on silica gel (hexanes; 5% ethyl acetate/hexanes; 10% ethyl acetate/hexanes) gave *1-methyl-2-benzosuberone* (**17**) as a colorless oil ( $R_f = 0.26$ , 5% ethyl acetate/hexanes); yield, 1.47 g (85%);  $^1\text{H-NMR}$   $\delta$  1.45 (d,  $J = 7.1$ , 3H), 1.92 (m, 1H), 2.08 (m, 1H), 2.47 (m, 1H), 2.66 (m, 1H), 2.79 (m, 1H), 2.95 (m, 1H), 3.88 (quartet, 6.9, 1H), 7.12-7.26 (m, 4H);  $^{13}\text{C-NMR}$   $\delta$  14.50, 27.07, 32.33, 42.21, 50.80, 127.12, 127.16, 127.21, 129.40, 138.61, 139.65, 211.89; IR (C=O) 1709  $\text{cm}^{-1}$ ; 2,4-dinitrophenylhydrazone, mp 144-146 °C (lit. [15] mp 150-151 °C).

*Preparation of  $^{13}\text{C}$ -Methyltriphenylphosphonium Iodide*

A solution of  $^{13}\text{C}$ -labeled iodomethane [16] (5.00 g, 35.0 mmol) in benzene (20 mL) was added dropwise to a cooled (-4 to 0 °C) solution of triphenylphosphine (8.39 g, 32.0 mmol) in benzene (50 mL) over one hour. A white solid began to separate after 40 minutes. This mixture was allowed to warm to room temperature and stirred for a period of 4 hours. The white solid was collected by vacuum filtration and identified by melting point as  $^{13}\text{C}$ -methyltriphenylphosphonium iodide; yield, 12.60 (97%); melting point, 182-184 °C (lit. [17] mp 183-184 °C).

### Preparation of $^{13}\text{C}$ -Methylideneindan (**18**)

Potassium *tert*-butoxide (1.35 g, 12.0 mmol) was added at once under argon to a mechanically stirred mixture of  $^{13}\text{C}$ -methyltriphenylphosphonium iodide (4.86 g, 12.0 mmol) in dry ether (50 mL) to give a canary yellow mixture. This mixture was vigorously stirred for 30 minutes. A solution of 1-indanone (1.32 g, 10.0 mmol) in dry ether (20 mL) was added to the mixture over a period of 5 minutes. The color of the mixture gradually became a vivid blue as it was stirred overnight (14 hours). The resulting mixture was filtered through Celite® (10 g), and the filtrate was concentrated *in vacuo* to give a light yellow oil. The oil was eluted with hexanes through a pad of silica gel (30 g) on a sintered glass funnel under aspirator vacuum. The eluant was concentrated *in vacuo* to give 1-( $^{13}\text{C}$ -methylidene)indan (**18**) as a colorless oil; yield, 0.91 g (69%);  $^1\text{H-NMR}$   $\delta$  2.97 (m, 2H), 3.14 (m, 2H), 5.16 (dt,  $J_{\text{HH}} = 2.1$ ,  $J_{\text{HC}} = 127.20$ , 1H), 5.68 (dt,  $J_{\text{HH}} = 2.1$ ,  $J_{\text{HC}} = 125.4$ , 1H), 7.34-7.44 (m, 3H), 7.66 (d,  $J = 6.9$ , 1H);  $^{13}\text{C-NMR}$   $\delta$  12.93, 30.01, 31.10, 102.73 (enhanced), 120.50, 120.53, 125.24, 126.31, 128.13, 128.15; IR ( $^{12}\text{C}=\text{C}$ )  $1715\text{ cm}^{-1}$ .

### Reaction of $^{13}\text{C}$ Labeled Methylideneindan with HTIB (Entry 8)

Crystalline HTIB (1.78 g, 4.54 mmol) was added to a stirred solution of 1-( $^{13}\text{C}$ -methylidene)indan (**18**, 0.66 g, 5.0 mmol) in 95% methanol (25 mL). The solid dissolved rapidly with the evolution of heat to give a colorless solution. The solution was stirred at room temperature for 20 minutes, and the solvent was removed *in vacuo* to give an oily mixture. This mixture was partitioned between  $\text{CH}_2\text{Cl}_2$  (25 mL) and  $\text{H}_2\text{O}$  (25 mL). The organic layer was washed with  $\text{H}_2\text{O}$  (2 x 25 mL) and brine (1 x 20 mL), dried over  $\text{MgSO}_4$ , and concentrated *in vacuo* to give a bright yellow oil (0.79 g). Flash column chromatography of the oil on silica gel (hexanes; 5 % ethyl acetate/ hexanes) gave 1- $^{13}\text{C}$ - $\beta$ -tetralone (**19**) as a light yellow oil ( $R_f = 0.30$ , 5 % ethyl acetate/hexanes); yield, 0.61 g (91 %);  $^1\text{H-NMR}$   $\delta$  2.58 (t,  $J = 6.6$ , 2H),  $\delta$  3.10 (t,  $J = 6.6$ , 2H),  $\delta$  3.62 (d,  $J_{\text{HC}} = 128.6$ , 2H),  $\delta$  7.15-7.19 (m, 4H);  $^{13}\text{C-NMR}$ , (16 transients)  $\delta$  45.03; IR (C=O)  $1718\text{ cm}^{-1}$ .

## References and Notes

1. Taylor, E. C.; Chiang C.-S.; McKillop, A. *Tetrahedron Lett.* **1977**, 1827-1830.
2. El-Hossini, M. S.; McCullough, K. J.; McKay, R.; Proctor, G. R. *Tetrahedron Lett.* **1986**, 27, 3783-3786.
3. Justik, M. W.; Koser, G. F. *Tetrahedron Lett.*, **2004**, 45, 6159-6163.
4. According to current MSDS information (obtained online from Aldrich Chemical Company), thallium(III) nitrate and thallium(I) nitrate are listed as “highly toxic” with a “danger of cumulative effects”. Silver nitrate is described as “corrosive” and “dangerous for the environment”. HTIB is listed as an “irritant”, and iodobenzene as “harmful”.
5. Fitjer, L.; Quabeck, U. *Synth. Commun.* **1985**, 15, 855-864.
6. Prakash, O. *Aldrichimica Acta* **1995**, 28, 63-71.
7. Bhattacharya, A.; Segmuller, B.; Ybarra, A. *Synth. Commun.* **1996**, 26, 1775-84.
8. Kulagowski, J. J.; Moody, C. J.; Rees, C. W. *J. Chem. Soc., Perkin Trans. 1* **1985**, 12, 2733-9.
9. Waddington, T.; Alcock, N. W. *J. Chem. Soc.* **1963**, 4103-9.



10. Koser, G. F.; Wettach, R. H.; Troup, J. M.; Frenz, B. A. *J. Org. Chem.* **1976**, *41*, 3609-11.
11. Page, G. A.; Tarbell, D. S. *J. Am. Chem. Soc.* **1953**, *75*, 2053-5.
12. The low integration is probably due to a pulse delay time of only one second.
13. Wong, H. N. C.; Chan, T. -L.; Sondheimer, F. *Tetrahedron Lett.* **1978**, 667-70.
14. Verbel, J.; Carrie, R. *Bull. Soc. Chim. Fr.* **1982**, *2*, 161-6.
15. Crabb, T. A.; Schofield, K. *J. Chem. Soc.* **1958**, 4276-84.
16. Product of Aldrich Chemical Co., product number 277185, isotopic purity 99 atom %.
17. Devlin, C. J.; Walker, B. J. *J. Chem. Soc., Perkin Trans. 1* **1974**, 453-60.

*Sample Availability:* Not available.

© 2005 by MDPI (<http://www.mdpi.org>). Reproduction is permitted for noncommercial purposes.