

Anal. Calcd for  $C_{20}H_{34}N_2O_8$ : C, 55.82; H, 7.96; N, 6.51. Found: C, 55.71; H, 8.03; N, 6.40.

**Note Added in Proof.** A paper which is complementary to ours and describes the preparation of  $\beta$ -keto carboxamides from substituted 1,3-dioxin-4-ones was published while this manuscript was in press. See: Sato, M.; Ogasawara, H.; Komatsu, S.; Kato, T. *Chem. Pharm. Bull.* 1984, 32, 3848-3856.

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**Registry No.** 1, 5394-63-8; 3, 42598-96-9; 3 (alcohol), 111-70-6; 4, 96453-22-4; 4 (alcohol), 589-98-0; 5, 91328-33-5; 5 (alcohol),

590-67-0; 6, 96481-25-3; 6 (alcohol), 27779-29-9; 7, 1473-23-0; 8, 96453-23-5; 8 (alcohol), 96553-53-6; 9, 66888-11-7; 9 (alcohol), 582-52-5; 10, 5459-04-1; 10 (alcohol), 107-21-1; 11, 14276-67-6; 11 (alcohol), 126-30-7; *cis*-12, 96453-24-6; *cis*-12 (alcohol), 3236-47-3; *trans*-12, 96453-30-4; *trans*-12 (alcohol), 3236-48-4; 13, 96453-25-7; 13 (alcohol), 2694-23-7; 14, 96481-26-4; 15, 6864-62-6; 15 (alcohol), 108-95-2; 16, 38432-60-9; 16 (alcohol), 150-76-5; 17, 29816-97-5; 17 (alcohol), 100-02-7; 18, 40053-29-0; 18 (thiol), 108-98-5; 19, 96453-26-8; 19 (thiol), 112-55-0; 20, 96453-27-9; 20 (thiol), 2365-48-2; 21, 51494-47-4; 21 (amine), 111-68-2; 22, 58102-37-7; 22 (amine), 768-94-5; 23, 96453-28-0; 23 (amine), 23978-55-4; 24, 728-49-4; 24 (amine), 26682-99-5; 25, 4835-39-6; 25 (amine), 100-01-6; 26, 96453-29-1; 26 (amine), 1477-42-5; cholesterol, 57-88-5; glucose, 50-99-7.

**Supplementary Material Available:** Spectral (IR,  $^1H$  NMR, and MS) and analytical (combustion analysis) data for all remaining compounds listed in Tables I and II (6 pages). Ordering information is given on any current masthead page.

## Chromic Acid Oxidation of Indans and Tetralins to 1-Indanones and 1-Tetralones Using Jones and Other Cr(VI) Reagents

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The Jones chromic acid oxidation, ordinarily used for converting secondary alcohols to ketones, has been successfully extended to the oxidation of indans and tetralins to 1-indanones and 1-tetralones, respectively. A sixfold excess of the reagent was commonly used to ensure complete oxidation of starting material. Addition of anhydrous magnesium sulfate or oven-dried silica gel provided a yield increase of 15-20%. 2,2'-Bipyridinium chlorochromate was also used and found to be effective in all cases, but this reagent requires a longer reaction time and a 16-fold excess. A comparison between these two reagents and  $CrO_3$  in acetic acid was made. The Jones reagent, being least selective, gives the highest yield of sterically hindered monoketone from 1,2,3,4,5,6,7,8-octahydrophenanthrene.

Chromic acid oxidation of hydrocarbons at a benzylic position to produce ketones generally is carried out in acetic acid.<sup>1a-c</sup> To improve yields and the selectivity of products, we undertook a study of the application of the Jones chromic acid oxidizing reagent<sup>2</sup> and other Cr(VI) oxidations of indans and tetralins to 1-indanones and 1-tetralones.

The Jones reagent,<sup>2a,b</sup> chromium trioxide dissolved in aqueous sulfuric acid, most frequently is used in acetone solvent to oxidize secondary alcohols to ketones.<sup>2c,d</sup> We have found that the Jones method, as previously described,<sup>2a,b</sup> can also be applied to the oxidation of hydrocarbons at a benzylic position with little change in procedure. In adapting the Jones reagent to this new use, we systematically examined several parameters to improve the oxidation. This study includes changes in the structure of the hydrocarbon starting materials, as shown in Table

I, a search for substitutes for sulfuric acid<sup>3</sup> and acetone,<sup>4</sup> trial of other Cr(VI) reagents,<sup>5a-c</sup> and the effect that removal of water produced by the oxidation has on the outcome of the reaction. As expected, since  $Cr_2(SO_4)_3$  is deposited, effective stirring was essential.<sup>6a</sup> The best yield of 1-tetralone was achieved with a sixfold excess of Jones

(3) (a) Methanesulfonic acid and trifluoroacetic acid were substituted (2 mol for 1 mol) for sulfuric acid. Otherwise, the oxidation procedure remained the same. (b) Phosphoric acid (85%) was substituted (2 mol for 3 mol) for sulfuric acid. The procedure remained the same except that water was omitted during preparation of the reagent.

(4) (a) Acetonitrile, *N,N*-dimethylformamide, and *N*-methylpyrrolidinone were exposed to Jones reagent at ice-bath temperature. Since a color change to green appeared within a few minutes, these were considered to be unsuitable as substitutes for acetone. (b) At room temperature *N,N*-dimethylacetamide was attacked by the Jones reagent, since the color changed to blue-green, but salts were not deposited. (c) Brown, H. C.; Garg, C. P.; Liu, K. T. *J. Org. Chem.* 1971, 36, 387. (d) Oxidation of tetralin in acetone gives 1-tetralone in 68% yield. When benzene is substituted as solvent, the yield drops to 10-15%, with most of the tetralin being recovered.


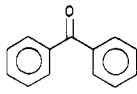
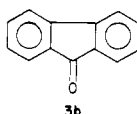
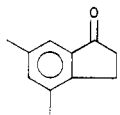
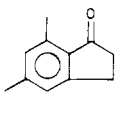
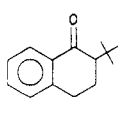
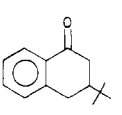
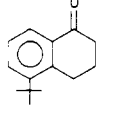
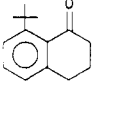
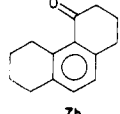
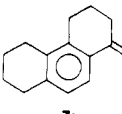
(5) (a) Guziec, F. S.; Luzzio, F. A. *Synthesis* 1980, 691. (b) Corey, E. J.; Suggs, J. W. *Tetrahedron Lett.* 1975, 31, 2647. (c) Lalancette, J. M.; Rollin, G.; Dumas, P. *Can. J. Chem.* 1972, 50, 3058. (d) The original procedures<sup>5a-c</sup> were followed to determine the effectiveness of these Cr(VI) reagents.

(6) (a) A high-speed mechanical stirrer with a multiblade paddle was found to be adequate. Stirring with a Teflon-covered magnetic stirring bar gave approximately the same yields. However, it was necessary to use a powerful magnetic stirrer (we have previously described<sup>10</sup> a stirrer of local construction that has been further modified with a more powerful magnet). Should a magnetic stirring bar go out of control with this powerful magnetic stirrer, the wall of a conventional flask will likely be broken and hence a heavy-wall flask is recommended. (b) For earlier use of silica gel in chromic acid oxidation of primary and secondary alcohols, cf.: Santaniello, E.; Ponti, F.; Manzocchi, A. *Synthesis* 1978, 534.

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(2) (a) Bowden, K.; Heilbron, I. M.; Jones, E. R. H.; Weedon, B. C. L. *J. Chem. Soc.* 1946, 39. (b) Eisenbraun, E. J. "Organic Syntheses"; Wiley: New York, 1973; Collect. Vol. V, p 310. (c) Braude, E. A.; Jones, E. R. H.; Sondheimer, F.; Toogood, J. B. *J. Chem. Soc.* 1949, 607. (d) Curtis, R. G.; Heilbron, I. M.; Jones, E. R. H.; Woods, G. F. *J. Chem. Soc.* 1953, 457.

Table I. Comparison of Cr(VI) Oxidation Procedures

hydrocarbon <sup>a</sup>	product ketone(s)	ratio of ketones and combined yield <sup>b,c</sup>					
		CrO <sub>3</sub> /HOAc <sup>d</sup>		Jones reagent <sup>e</sup>		BiPCC/acetone <sup>f</sup>	
		b:c	yield, %	b:c	yield, %	b:c	yield, %
1a			55		68		63
2a			60		62		50
3a			71		68		70
4a	 	1.0:4.4	75	1.0:5.0	75	1.0:4.8	72
5a	 	1.0:5.8	70	1.0:3.0	75	1.0:6.1	70
6a	 	2.9:1.0	62	1.0:1.0	65	4.0:1.0	60
7a	 	1.0:5.0	70	1.0:1.0	74	1.0:4.5	58

<sup>a</sup> Corresponding to ketones of this table. <sup>b</sup> Ratio determined by GLC. A 5% Carbowax 20 M coating on 80-100-mesh Gas Pak contained in a 0.25 in. × 10 ft copper tube was used. Two peak areas for calculating ratios were obtained by triangulation of GLC peaks. <sup>c</sup> Authentic samples of 4b, 4c, 5b, 5c, 6b, 6c, 7b, and 7c were available from earlier work. <sup>d</sup> Reference 1 reports 2-4 h of stirring. <sup>e</sup> CrO<sub>3</sub>, H<sub>2</sub>O, H<sub>2</sub>SO<sub>4</sub>/acetone for 8-10 h. <sup>f</sup> Stirring for 24-28 h.

reagent (1 mol of tetralin/8 mol of CrO<sub>3</sub>).

The data in Table I show that the Jones oxidation at the benzylic position did not give an overall improved yield over other Cr(VI) reagents. However, the ratio of oxidation products was significantly different from the ratios obtained through use of CrO<sub>3</sub>/HOAc or 2,2'-bipyridinium chlorochromate (BiPCC)/acetone when multiple benzylic oxidation sites were available and one of these was sterically hindered. Thus, in most cases, oxidation with the Jones reagent markedly increased the yield of a sterically hindered ketone product at the expense of the other isomer. We attribute this ratio difference to the more powerful oxidizing nature of the Jones reagent, which thus shows decreased selectivity.

These results prompted a study of the effect of excess sulfuric acid on the performance of the Jones reagent. Examination of the CrO<sub>3</sub>:H<sub>2</sub>SO<sub>4</sub> ratios used in earlier publications shows that there has been close adherence to use of the stoichiometric amount of sulfuric acid. In an attempt to minimize the need for excess Jones reagent, we increased the sulfuric acid concentration and decreased the equivalents of Jones reagent from 6× to 4×, 3×, and 2×. In none of these runs did the yield of 1-tetralone from tetralin equal the yields in Table I. Thus, we believe that use of an excess of sulfuric acid will not help to increase

the yield. Possibly aldol condensation of acetone took place and Jones reagent was consumed in the oxidation of these condensation products or 1-tetralone.

Sulfuric acid remains the most effective acid<sup>3</sup> in driving the reaction. Methanesulfonic acid,<sup>3a</sup> 85% phosphoric acid,<sup>3b</sup> and trifluoroacetic acid<sup>3a</sup> proved unsuitable substitutes. Although color changes were observed, 1-tetralone was not obtained and most of the starting material was recovered in each case.

Of the solvents examined as substitutes, acetone remains the best.<sup>4</sup> Acetonitrile, *N,N*-dimethylformamide, and *N*-methylpyrrolidinone were tried as substitutes for acetone in the Jones oxidation. These were not suitable because of their rapid oxidation.<sup>4a</sup> In contrast, *N,N*-dimethylacetamide (DMA) was stable for several hours on exposure to the Jones reagent at 5-10 °C,<sup>4b</sup> and in this solvent the reagent has the potential to oxidize alcohols to ketones, since benzhydrol was rapidly oxidized to benzophenone. However, tetralin and diphenylmethane were not oxidized by the reagent in this solvent, and hence the utility of DMA for hydrocarbon oxidation is questionable. Ethyl ether, used by Brown in chromic acid oxidations,<sup>4c</sup> was not a suitable solvent in the Jones oxidation of tetralin. When undiluted Jones reagent was added to cold diethyl ether, there was an immediate and vigorous reaction. The

**Table II. Determination of Chromium Trioxide Consumed and Acetic Acid Formed during Jones Oxidation of Acetone<sup>a</sup>**

stirring time, h	CrO <sub>3</sub> consumed, <sup>b</sup> %	HOAc formed, <sup>c</sup> %
4	1.9	0.2
8	3.6	0.3
12	5.0	0.6

<sup>a</sup> Average of duplicate runs. <sup>b</sup> [(Mol of CrO<sub>3</sub> remaining)/(mol of CrO<sub>3</sub> introduced)](100). <sup>c</sup> [(Mol of acetic acid produced)/(mol of acetone introduced)](100).

Jones reagent in benzene,<sup>4b</sup> used with prolonged stirring (24 h, three times the usual period), oxidizes tetralin. However, this was no advantage since the benzene modification was less convenient and the yield was less.<sup>4d</sup>

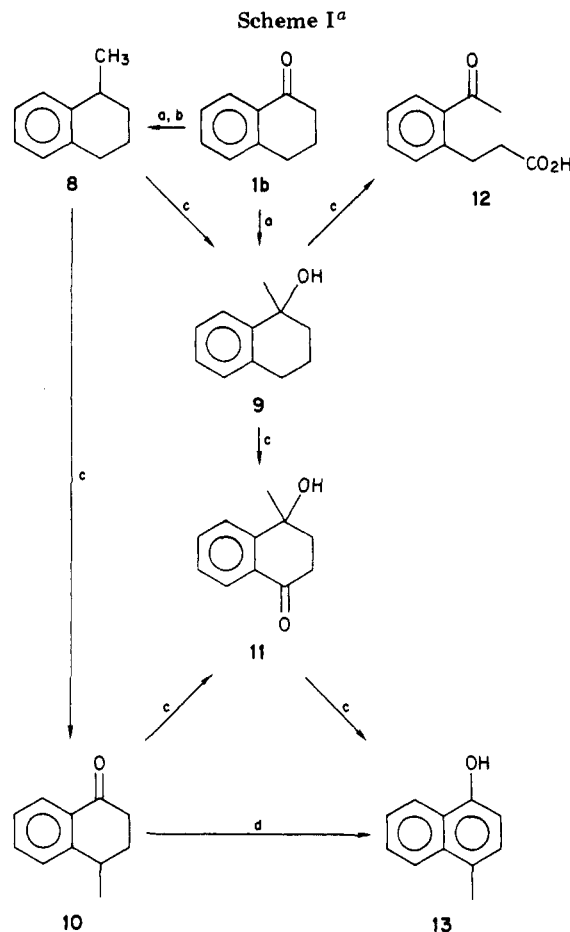
2,2'-Bipyridinium chlorochromate in dichloromethane or, better, in acetone, caused benzylic oxidation of hydrocarbons. The usual molar ratio of 4:1 (BiPCC to substrate), found effective for secondary alcohols,<sup>5a</sup> was increased to 16:1, whereupon good yields, as shown in Table I, were realized.<sup>5a</sup> Pyridinium chlorochromate in dichloromethane<sup>5b</sup> and chromium(VI) oxide in graphite<sup>5c</sup> (Seloxcette, 200 mesh) in toluene were tried as hydrocarbon oxidizing reagents and found to be unreactive.<sup>5d</sup>

Water, though minimized from earlier procedures,<sup>2a-d</sup> is necessary to dissolve chromium trioxide in the preparation of the Jones reagent. As the reaction proceeds more water is produced (3 mol/CH<sub>2</sub> group), and consequently, as the Cr(III) salts appear, the reaction mixture becomes sticky. These salts cling to the sides of the flask and foul the stirrer paddle, at times enough to cause a stirrer imbalance and thus interfere with mixing. These salts also occlude starting material and product and thus cause a 15–20% yield decrease and can prevent completion of the reaction. To minimize this problem we added anhydrous magnesium sulfate to the reaction mixture. Presumably, the formation of magnesium sulfate hydrates would decrease the water content and thus keep the salts in suspension. Oven-dried silica gel also reduced stickiness of the reaction mixture.<sup>6b</sup> These drying agents were about equally effective in improving the ease of isolation of product.

We sought information about the stability of 1-tetralone toward an excess of the Jones reagent. 1-Tetralone (0.02 mol) was subjected to the same oxidation conditions as used in its preparation and 85% of 1-tetralone was found to survive the oxidation.

It is to be expected that some acetone and any aldol dimer formed<sup>7</sup> would be converted to acetic acid by prolonged exposure to Jones reagent. Accordingly, blank studies were carried out in which the acetic acid formed during these experiments was steam-distilled and estimated by titration with standardized alkali as is done in a Kuhn–Roth determination.<sup>8</sup> The percent of Cr(VI) consumed in blank runs also was determined.<sup>8</sup> The data from these studies, presented in Table II, show that the consumption of Cr(VI) was not excessive but did appear approximately to parallel the production of acetic acid.

1-Methyltetralin (8, Scheme I) was selected as a test compound to determine whether hydrocarbons of this type would be readily oxidized to ketones in reasonable yield and whether attack upon methine hydrogen at a benzylic position would be preferred over that at a methylene



<sup>a</sup> (a) CH<sub>3</sub>MgBr, anhydrous ether; (b) H<sub>2</sub>, Pd/C, acetic acid; (c) CrO<sub>3</sub>, H<sub>2</sub>O, sulfuric acid, acetone; (d) Pd/C, 250 °C, Δ, 4 h.

position. Wiberg<sup>1b</sup> has shown that the rates of chromic acid oxidation of aliphatic CH bonds are in the ratio 7000:1100:1 for tertiary, secondary, and primary hydrogen, respectively.

We anticipated that chromic acid oxidation of 8 could provide 9 and 10 (Scheme I) as initial products. These were found in 36% combined yield in the ratio of 1:6. The bicarbonate-soluble fraction yielded the crystalline keto acid 12 in 39% yield. This suggests that 9 was oxidized to 12, as expected from the Fieser and Szmuszkovicz oxidation<sup>9</sup> of tertiary alcohols to keto acids. To confirm this point we prepared alcohol 9 from 1b, as shown in Scheme I, and then subjected 9 to Jones oxidation. This afforded 12 in 60% yield.

We also sought further oxidation products of 9 and 10, i.e., 11 and 13, but were unable to isolate or detect these products. We are not aware of a study comparable to Wiberg's treatment of aliphatic primary, secondary, and tertiary hydrogens as applied to the benzylic position. Nevertheless, the benzylic oxidation of secondary vs. tertiary is feasible with Jones reagent or with BiPCC, which gives comparable results, but the yields can be predicted to be low.

### Experimental Section

**Jones Reagent Preparation.** The Jones reagent was made up in 1.00-mol batches by dissolving 100 g of CrO<sub>3</sub> in a minimum of distilled water. Sulfuric acid (86.5 mL) was added and more water (about 194 mL total) was then added to effect solution of

(7) While we have no evidence that acetone undergoes dimerization, the Jones reagent rapidly attacks 4-methyl-3-penten-2-one to give a green color and to deposit salts.

(8) Eisenbraun, E. J.; McElvain, S. M.; Aycock, B. F. *J. Am. Chem. Soc.* 1954, 76, 607.

(9) Fieser, L. F.; Szmuszkovicz, J. *J. Am. Chem. Soc.* 1948, 70, 3352.  
(10) Hall, H.; Eisenbraun, E. J. *J. Chem. Educ.* 1977, 54, 229.

precipitated  $\text{CrO}_3$ .<sup>2b</sup> The final volume was 336 mL (3.36 M with respect to  $\text{CrO}_3$ ).

#### General Oxidation Procedure Using the Jones Reagent.

To a two-necked, 300-mL Morton flask, fitted with a high-speed mechanical stirrer and immersed in an ice bath, was added 0.02 mol of the hydrocarbon in 50 mL of acetone. The cooled mixture was stirred<sup>6a</sup> vigorously during the addition of 46 mL of Jones reagent over a period of 30 min. The temperature of the reaction mixture was maintained between 5 and 15 °C throughout the reaction. As the green  $\text{Cr}_2(\text{SO}_4)_3$  began appearing, 8.4 g (0.07 mol) of anhydrous  $\text{MgSO}_4$  was added. The maximum yield was observed with this quantity. As stated earlier, this treatment minimized stirrer imbalance resulting from adhering salts. The use of 50 g of oven-dried (120–130 °C) silica (50 mol/mol of hydrocarbon) as a substitute for anhydrous magnesium sulfate proved equally effective.<sup>6b</sup>

Samples were withdrawn at intervals, treated with anhydrous sodium carbonate, filtered, and analyzed by gas chromatography (Table I) to determine the completeness of reaction. In most cases, 6–8 h of reaction time was adequate for complete consumption of starting hydrocarbon. Additional Jones reagent was added to those hydrocarbons that oxidized slowly, and stirring was continued for a total of 10 h.

On disappearance of starting material, excess Jones reagent was destroyed by adding isopropyl alcohol.<sup>2b</sup> The contents of the flask were filtered through Dicalite to remove suspended chromium salts. The filter cake was washed, as needed, with acetone and ether. The filtrate was rotary evaporated, the reaction product was dissolved in ether, and the ether solution was washed with sodium bicarbonate solution to remove any remaining sulfuric acid and acetic acid formed through the oxidation of acetone. The ether extract was dried ( $\text{MgSO}_4$ ), filtered, and concentrated to obtain the ketone of interest.

#### Yield Optimization in Converting Tetralin to 1-Tetralone.

Five experiments were carried out in which all conditions, including the amount of  $\text{MgSO}_4$ , were held constant except that the molar ratio of  $\text{CrO}_3$ :tetralin ranged from 4:1 to 10:1. The

maximum yield of 1-tetralone was obtained with a ratio of 7:1. The theoretical chromium trioxide:tetralin ratio is 4:3.

**Stability of 1-Tetralone during Jones Oxidation.** 1-Tetralone (2.92 g, 0.02 mol) and 46.0 mL of Jones reagent in 50 mL of acetone were stirred for 4 h at ice-bath temperature. The isolation was carried out as described in the general procedure. The product showed a single GLC peak (1-tetralone), and the recovered yield (85%) of 1-tetralone was determined by GLC analysis using added tetralin as an internal standard.

**Chromium Trioxide Consumed and Acetic Acid Formed during Blank Runs.** Samples of acetone (50 mL, 1.17 mol) were stirred with 10 mL of Jones reagent for 4, 8, and 12 h, under conditions typical of those used to oxidize hydrocarbons. At the end, the excess Jones reagent was destroyed by adding isopropyl alcohol, and the contents were steam-distilled. Several 100-mL distillation fractions were collected and titrated with 0.1 N NaOH to the phenolphthalein end point. These data are found in Table II.

The amount of chromium trioxide consumed for the same oxidation was also determined by repeating the reaction for 4, 8, and 12 h under identical experimental conditions and rotary evaporating the remaining acetone. The residue was diluted to 1000 mL, and several 50-mL portions were titrated with standardized sodium thiosulfate solution after the addition of potassium iodide and 1% starch solution for end point determination.<sup>8</sup> Again, the results are shown in Table II.

**Acknowledgment.** We thank Drs. O. C. Dermer and R. A. Bunce for their interest in this work and for reading the manuscript.

**Registry No.** 1a, 119-64-2; 1b, 529-34-0; 2a, 101-81-5; 2b, 119-61-9; 3a, 86-73-7; 3b, 486-25-9; 4a, 1685-82-1; 4b, 1685-81-0; 4c, 6682-69-5; 5a, 42044-22-4; 5b, 42981-75-9; 5c, 42981-74-8; 6a, 42044-24-6; 6b, 51015-35-1; 6c, 51015-36-2; 7a, 5325-97-3; 7b, 13250-73-2; 7c, 75490-06-1; 8, 1559-81-5; 9, 14944-28-6; 10, 19832-98-5; 12, 66405-16-1.

## Synthesis of a Representative Cis/Trans Pair of 4,5-Disubstituted Cyclopentenyllithium Reagents

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A useful procedure for effecting the conversion of 2,3-disubstituted cyclopentanones to cis- and trans-4,5-disubstituted cyclopentenyllithium reagents has been developed. The preferred sequence of reactions (at least for obtaining the cis isomer) involves *uncatalyzed* tosylhydrazone formation and conversion by the Shapiro reaction to epimeric vinylstannanes that are readily separable by spinning band distillation. Of particular relevance is the ease and high efficiency of the subsequent iodination and metalation of these pivotal intermediates. This methodology is amenable to reasonable scale-up and convenient to implement in practice.

Utilization of the anionic oxy-Cope rearrangement in organic synthesis most often relies upon preliminary condensation of a  $\beta,\gamma$ -unsaturated ketone with a vinyl organometallic reagent.<sup>2</sup> While notable achievements have

been made through proper application of vinyl lithium, vinylmagnesium bromide, and related simple nucleophilic reagents, acquisition of complex natural products understandably can become limited by the availability of stereochemically more ornate substrates. In connection with a convergent approach to ikarugamycin (1) being pursued

(1) Postdoctoral Fellow of the Deutscher Akademischer Austauschdienst (NATO), 1983–1984.

(2) For example: (a) Evans, D. A.; Golob, A. M. *J. Am. Chem. Soc.* 1975, 97, 4765. (b) Still, W. C. *Ibid.* 1977, 99, 4186. (c) Jung, M. E.; Hudspeth, J. P. *Ibid.* 1978, 100, 4309. (d) Evans, D. A.; Nelson, J. V. *Ibid.* 1980, 102, 774. (e) Kahn, M. *Tetrahedron Lett.* 1980, 21, 4547. (f) Koreeda, M.; Tanaka, Y.; Schwartz, A. *J. Org. Chem.* 1980, 45, 1172. (g) Schreiber, S. L.; Santini, C. *Tetrahedron Lett.* 1981, 22, 4651. (h) Geetha, P.; Huq, C. A. M. A.; Rajagopalan, K.; Swaminathan, S. *Ibid.* 1982, 23, 569.

