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A Facile Synthesis of Bicyclo[4,1, O]Heptan-2-ones by Telluronium Ylides

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A FACILE SYNTHESIS OF BICYCLO[4, 1, 0]HEPTAN-2-ONES BY TELLURONIUM YLIDES

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Abstract The cyclopropanation reaction of cyclohexenones with telluronium ylides was studied. The products were proved to be the derivatives of bicyclo[4, 1, 0]heptan-2-one.

Organotelluronium compounds in organic synthesis have been widely investigated¹. We had studied the reaction of telluronium ylide in organic synthesis systemtically²⁻³. Considering the importance of the cyclopropyl subnit in natural and synthetic products⁴⁻⁶ and the few work of telluronium ylide in the synthesis of substituted cyclopropane, recently, we started to synthesize the derivatives of cyclopropane and bicyclopropane by telluronium ylides ⁷⁻¹⁰. The synthesis of cyclopropane^{7, 8} were carried out by the reaction of telluronium salts with some chalcones and bicyclopropane derivatives^{9, 10} were prepared by the reaction of bis(dibuty)telluronium)p-xylylene dibromide with α , β -unsaturated carbonyl compounds or telluronium ylides with 1,4-bis(3-substituted phenyl-3-oxo-1-propenyl)benzene and 1, 5-

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scheme

disubstituted phenyl-1,4-pentadien-3-one in the presence of sodium hydroxide at the room temperature in THF. To our knowledge, the application of telluronium ylide to produce ring bridged compounds has not been reported in the literature. So, here, we wish to report the facile method for the synthesis of bicyclo[4, 1, 0]heptan-2-ones by the reaction of cyclohexenones and telluronium ylides (Scheme).

We studied the effect of solvent, base and temperature on the reaction and found that bases do remarkable effect on its yield. The followed table shows the best reaction condition.

Experimental

IR spectra were measured with a Nicolet 740 FT-IR spectrophotometer. ¹HNMR spectra were recorded on a JNM-FX 90Q spectrometer in CDCl₃ using TMS as internal standard. Elemental analysis data were recorded on a Carlo Erba - 1106 apparatus.

2-Cyclohexenone (**1a**) was aldrich product. 3-Methyl-2-cyclohexenone (**1b**) and telluronium salts **2** were synthesized according to literature 11 and 12, respectively.

| Prod. | R | Ar | Reaction Time (hours) | Yield (%) |
|-------|----|-----------------------------------|----------------------------|--------------|
| 3 | н | C ₆ H₅- | 24 | 67 |
| 4 | Н | p-ClC ₆ H₄- | 36 | 42 |
| 5 | Н | C ₆ H ₅ CO- | 20 | 48 |
| 6 | Me | C ₆ H ₅ - | 48 | 51 |
| 7 | Me | C ₆ H ₅ CO- | 40 | 40 |

* The reaction was performed in dry MeCN under room temperature, and all the products were oil.

General procedure for the synthesis of 3 - 7:

1 (1.5mmol), 2 (1.2mmol) and potassium hydroxide (2.5mmol) were mixed in dry MeCN and stirred. After the reaction was completed (by TLC), the mixture was purified by column chromatography on silica gel (petroleum : ethyl acetate = 15 : 1). Removal of the solvent gave pure oil products 3 - 7 confirmed by the ¹HNMR spectra, IR spectra. and elemental analysis data.

3: IR(film): 1712, 1615, 1500, 1030, 998cm⁻¹; ¹HNMR (δppm): 7.45 - 7.37 (5H, m),
3.15 (1H, q, J= 4.7, 6.3Hz), 2.52-2.19 (3H, m), 2.19-1.68 (5H, m); Anal. Calcd C₁₃H₁₄O:
C, 83.87, H, 7.53; Found: C, 84.11, H, 7.76.

4: IR(film): 1710, 1600, 1570, 1490, 1076, 1008; ¹HNMR (δppm): 7.22 (4H, d), 3.50 (1H, q, J = 4.6, 6.1 Hz), 2.51 - 2.15 (3H, m), 2.15 - 1.74 (5H, m); Anal. Calcd C₁₃H₁₃OCl: C, 70.75, H, 5.90; Found: C, 71.04, H, 6.04.

5: IR(film): 1710, 1675, 1591, 1500, 1203, 1013 cm⁻¹; ¹HNMR (δppm): 8.00 - 7.04

(5H, m), 3.12 (1H, q, J = 4.8, 6.1 Hz), 2.63 - 2.14(3H, m), 1.90 - 1.64 (5H, m); Anal. Calcd $C_{14}H_{14}O_2$: C, 78.50, H, 6.54; Found: C, 78.35, H, 6.74.

6: IR(film): 1709, 1610, 1534, 1182, 1013 cm⁻¹; ¹HNMR (δppm): 7.33 (5H, s), 3.29
(1H, d, J = 4.5 Hz), 2.52 - 2.08(3H, m), 2.08 - 1.82 (4H, m), 1.25 (3H, s); Anal. Calcd C₁₄H₁₆O: C, 84.00, H, 8.00; Found: C, 84.23, H, 7.88.

7: IR(film): 1710, 1675, 1591, 1500, 1203, 1013 cm⁻¹; ¹HNMR (δ ppm): 7.96 - 7.19 (5H, m), 3.41 (1H, d, J = 4.7 Hz), 2.65 - 2.14 (3H, m), 2.14 - 1.83 (4H, m), 1.24 (3H, s); Anal. Calcd C₁₅H₁₆O₂: C, 78.95, H, 7.02; Found: C, 79.08, H, 6.84.

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