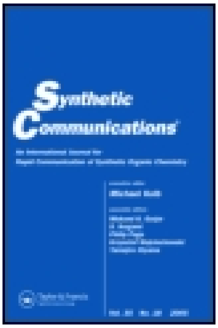

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Study on the Reactions of Telluronium Ylide VIII. A Facile Synthesis of Substituted Cyclopropanes

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STUDY ON THE REACTIONS OF TELLURONIUM YLIDE VIII. A FACILE SYNTHESIS OF SUBSTITUTED CYCLOPROPANES

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Abstract: Some telluronium salts react easily with substituted chalcones to afford derivatives of cyclopropane in 52-95% yield and with high stereoselectivity.

In modern organic synthesis, the applications of telluronium ylides have afforded new methods for the synthesis of olefinic derivatives and epoxides.[1-4] To our knowledge, the application of telluronium ylide to the synthesis of substituted cyclopropanes has not been reported in the literature. In our investigation, we found that some telluronium ylides could react with substituted chalcones to afford the derivatives of cyclopropane. In this communication, we wish to report the facile method for the synthesis of substituted cyclopropanes by the reaction of benzyldibutyltelluro-

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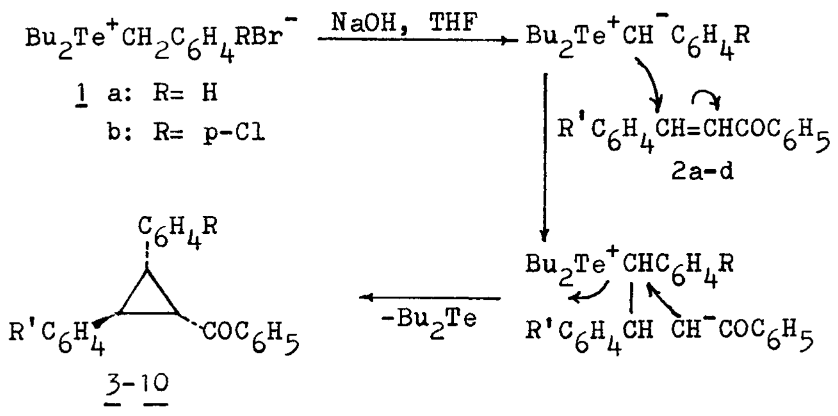
Prod.	R	R'	Reaction[a] condition(°C/h)	Yield[b] (%)
3	H	H	reflux/10	87
4	H	p-Br	r.t./ 8	89
5	H	m-NO_2	r.t./ 2	93
6	H	p-NMe_2	reflux/30	52
7	p-Cl	H	reflux/ 8	90
8	p-Cl	p-Br	r.t./ 8	92
9	p-Cl	m-NO_2	r.t./ 2	95
10	p-Cl	p-NMe_2	reflux/30	69

a: Solvent: THF

b: Isolated yields after column chromatography.

nium bromide(1a) and p-chlorobenzyldibutyltelluronium bromide(1b) with chalcones(2a-d) in the presence of bases. We studied the effect of solvent, base and temperature on the reaction and selected the best reaction condition(see Table). The results show that base has a remarkable effect on the reaction. For example, in the presence of solid sodium hydroxide, 1b reacted with 2a under reflux for 8 hrs, to afford 1-phenyl-2-(p-chlorophenyl)-3-benzoylcyclopropane (7,yield: 90%). In the presence of solid sodium carbonate, however, the

Scheme



2	a	b	c	d
R'	H	p-Br	m-NO_2	p-NMe_2

reaction did not occur. The reaction is likely to proceed via telluronium ylide(see Scheme,) and with high stereoselectivity.

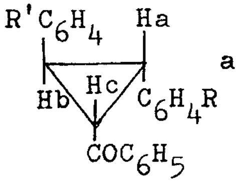
Experimental

Melting points are uncorrected and were taken on a Büch 535 apparatus. Elemental analysis were carried out on Carlo Erba 1106 apparatus. IR spectra measured with a Nicolet 740 FT-IR spectrophotometer. ^1H NMR spectra were recorded on a JNM-FX 90Q spectrometer in $CDCl_3$ using TMS as internal standard.

The synthesis of telluronium salts(1a,b) see literature 4.

General procedure for the synthesis of 3-10.

1(0.55 mmol), 2(0.5 mmol) and sodium hydroxide(1 mmol) were mixed in THF(5 mL) and stirred. The reaction was monitored by HPLC. After the reaction was completed, water was added. The resulting mixture was extracted with dichloromethane and the dichloromethane extract was dried over anhydrous sodium sulphate. Usual work-up followed by column chromatography on silica gel gave products 3-10. All products have the following configuration:



as confirmed by their ^{1}H NMR spectra.

3: m.p. 148-9^{o}C; Anal. calcd. for $C_{22}H_{18}O$; C 88.56 H 6.08; Found. C 88.74, H 5.89; IR(KBr): 1024, 969($\overset{CH-}{-CH-CH-}$), 1683($>C=O$)cm^{-1}; ^{1}H NMR(δppm): 3.20(1Ha, q,Jab =4.5, Jac=9.0 Hz), 3.35(1Hb, t, Jba=4.5, Jbc=6.3 Hz), 3.62(1Hc, q, Jca=9.0, Jcb=6.3 Hz), 7.15-7.99(15H, m).

4: m.p. 122-3^{o}C; Anal. calcd. for $C_{22}H_{17}BrO$; C 70.64, H 4.54; Found. C 70.29, H 4.67; IR(KBr): 1009, 972 ($\overset{CH-}{-CH-CH-}$), 1681($>C=O$)cm^{-1}; ^{1}H NMR(δppm): 3.25(1Ha, q, Jab=4.5, Jac=9.0 Hz), 3.37(1Hb, q, Jba=4.5, Jbc=3.6 Hz), 3.63(1Hc, q, Jca=9.0, Jcb=3.6 Hz), 7.09-7.99(14H, m).

5: m.p. 129-30°C; Anal. calcd. for $C_{22}H_{17}NO_3$: C 76.95, H 4.99, N 4.08; Found. C 77.01, H 4.73, N 3.77; IR(KBr): 1022, 984(-$\overset{\text{CH-}}{\text{CH-CH}}$-), 1662(>C=O)$cm^{-1}$; ^1H NMR (δppm): 3.36(1Ha, q, Jab=4.5, Jac=9.0 Hz), 3.44(1Hb, q, Jba=4.5, Jbc=5.4 Hz), 3.71(1Hc, q, Jca=9.0, Jcb=5.4 Hz), 7.15-8.17(14H, m).

6: m.p. 52-53°C; Anal. calcd. for $C_{24}H_{23}NO$: C 84.42, H 6.79, N 4.10; Found. C 84.17, H 6.59, N 3.94; IR (KBr): 1019, 981(-$\overset{\text{CH-}}{\text{CH-CH}}$-), 1654(>C=O)$cm^{-1}$; ^1H NMR(δppm) : 2.91(6H, s), 3.19(1Hb, q, Jba=3.6, Jbc=4.5 Hz), 3.26 (1Ha, q,Jab=3.6, Jac=9.0 Hz), 3.58(1Hc, q, Jca=9.0, Jcb =4.5 Hz), 7.09-8.06(14H, m).

7: m.p. 112-3°C; Anal. calcd. for $C_{22}H_{17}ClO$: C 79.39, H 5.15; Found. C 79.14, H 5.43; IR(KBr): 1015, 976 (-$\overset{\text{CH-}}{\text{CH-CH}}$-), 1669(>C=O)$cm^{-1}$; ^1H NMR(δppm): 3.25(1Hb, q, Jba=3.6, Jbc=4.5 Hz), 3.35(1Ha, q, Jab=3.6, Jac=6.3 Hz) 3.63(1Hc, q, Jca=6.3, Jcb=4.5 Hz), 7.11-7.99(14H, m).

8: m.p. 128-9°C; Anal. calcd..for $C_{22}H_{16}BrClO$: C 64.18, H 3.92; Found. C 64.37, H 3.75; IR(KBr): 1009, 973(-$\overset{\text{CH-}}{\text{CH-CH}}$-), 1668(>C=O)$cm^{-1}$; ^1H NMR(δppm): 3.21(1Ha,q Jab=3.6, Jac=6.3 Hz), 3.31(1Hb, q, Jba=3.6, Jbc=3.6 Hz) 3.52(1Hc, q, Jca=6.3, Jcb=3.6 Hz), 7.07-8.09(13H, m).

9: m.p. 133-4°C; Anal. calcd. for $C_{22}H_{16}ClNO_3$: C 69.93, H 4.27, N 3.71; Found. C 69.74, H 4.10, N 3.58; IR(KBr): 1018, 978(-$\overset{\text{CH-}}{\text{CH-CH}}$-), 1661(>C=O)$cm^{-1}$; ^1H NMR (δppm): 3.21(1Ha, q, Jab=4.5, Jac=6.3 Hz), 3.43(1Hb, q, Hba=4.5, Jbc=4.5 Hz), 3.66(1Hc, q, Jca=6.3, Jcb=4.5 Hz) 7.23-8.13(13H, m).

10: Brown mucilage, Anal. calcd. for $C_{24}H_{22}ClNO$: C 76.68, H 5.90, N 3.73; Found. C 76.14, H 5.47, N 3.32; IR(KBr): 1018, 981($-\overset{\mathrm{CH-}}{\mathrm{CH-CH}}-$), 1652(>C=O)$cm^{-1}$; ^1H NMR (δppm): 2.92(6H,s), 3.20(1Hb, q, Jba=3.6, Jbc=4.5 Hz), 3.26(1Ha,q, Jab=3.6, Jac=9.0 Hz), 3.58(1Hc, q, Jca=9.0, Jcb=4.5 Hz), 7.13-8.04(13H, m).

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