First International Electronic Conference on Synthetic Organic Chemistry (ECSOC-1), www.mdpi.org/ecsoc/, September 1-30, 1997

[A0017]

Arenechromiumtricarbonyl Derivatives as Chiral Auxiliaries: Synthesis of Enantiomerically Pure b-Lactones

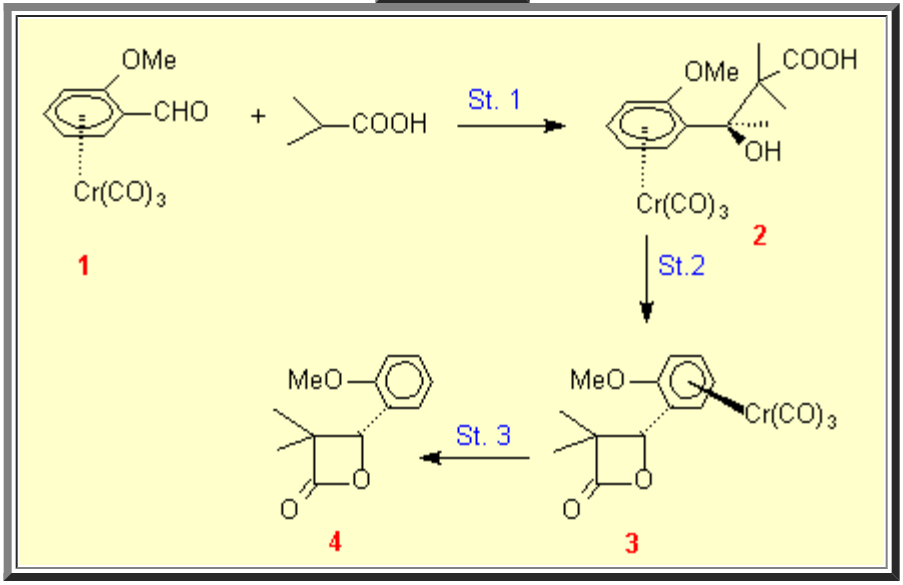
Del Buttero Paola* and Baldoli Clara

Dipartimento di Chimica Organica e Industriale, Via Venezian, 21 20133-Milano Italy
Fax ++39-2-2364369, E-mail: delbuttero@ICIL64.CILEA.IT

With biographical summary

Received: 23 July 1997 / Uploaded: 23 July 1997

Scheme



Background:

- The 2-oxetanone (beta.lactone) ring is present in several natural biologically active compounds.
- The use of chiral (eta6) tricarbonylchromium derivatives for the stereoselective synthesis of small ring heterocycles is already well documented.[1-4]
- Among the different methods for preparing b-lactons, we envisage the lactonization "via" b-hydroxy carboxylic acids as promising approch to this class of compounds starting from optically pure substituted chromium tricarbonyl benzaldehydes.
- Following the procedure reported in the scheme, a series of enantiomerically pure b-lactones have been obtained.
- One example is reported in this Poster.

Experimental:

- **Step 1):** To 6.6 mmol of LDA (in situ generated) in 15 ml of dry THF at -50 °C under nitrogen, 3.3 mmol of *iso*-butyric acid are added. After 1h at 30 °C, the solution is again cooled at 0 °C and then 1.1 mmol of aldehyde **1** in 2 ml of THF is added. Usual work-up (pH=4) affords the complexed hydroxy acid **2** in 95% yield. (d.e.>98%).
- **Step 2):** To a solution of **2** (0.55 mmol) in 0.6 ml of dry Py, 1.1 mmol of benzensulfonyl chloride is added dropwise. After 30 min. the reaction is quenched with ice, extracted with ether and washed with $NaHCO_3$. The pure **3** is isolated in 70% yield. (d.e.>98%)
- **Step 3):** A solution of **3** in CH_2Cl_2 is exposed to sunlight for about 3h. After removal of the solvent, the residue is treated with ether, filtered and the solvent evaporated. **4** is recovered in 80% yield.

	(e.e.>98%)

Analytical, spectroscopic data and References

Product 1: $[a]_D$ = -1001.3° (c=0.2 $CHCl_3$)

Product 2: m.p. 131-132 °C (petroleum ether)

H NMR ($CDCl_3$+**DMSO**) ppm 1.18 (s, 3H); 1.22 (s, 3H); 3.75 (s, 3H); 4.93 (t, 1H, J=6.1 Hz); 5.01 (d, 1H, J=6.5 Hz); 5.19 (s, 1H); 5.57 (t, 1H, J=6.5 Hz); 5.84 (d, 1H, J=6.1 Hz).
$[a]_D$ = +36.5° (c=0.2 $CHCl_3$)
Yield = 95%

Product 3: m.p. 117-118 °C (ether)

H NMR ($CDCl_3$) ppm 1.05 (s, 3H); 1.5 (s, 3H); 3.77 (s, 3H); 4.95 (t, 1H, J=6.4 Hz); 5.1 (d, 1H, J=6.6 Hz); 5.4 (s, 1H); 5.5 (t, 1H, J= 6.6 Hz); 5.8 (d, 1H, J=6.4 Hz).
IR (nujol) 1/cm 1834, 1866, 1822, 1963.
d.e.>98% (H NMR)
$[a]_D$ = +42.8° (c=0.2 $CHCl_3$)
Yield = 70%

Product 4: oil

H NMR ($CDCl_3$) ppm 0.88 (s, 3H); 1.6 (s, 3H); 3.8 (s, 3H); 5.45 (s, 1H); 6.8-7.4 (m, 4H).
IR (film) 1/cm 1829
e.e.> 98% (by H NMR)
$[a]_D$ = +94.7° (c=1 $CHCl_3$)
Yield = 80%

References

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Paola Del Buttero

Born in Milano some years ago. I am associate professor since 1986 in Organic Chemistry and I am responsible for two courses, experimental laboratory for organic chemistry and " chemistry of organometallic compounds" in Milano University. I am working on the following research topics:
New methodologies for the synthesis of organic compounds using tricarbonyl chromium complexes
Enzymes catalyzed stereoselective transformation of organometallics
Chiral organometallic auxiliaries for stereoselective synthesis of heterocycles also with biological activity.
I have one husband, the same for the last 27 years, two daughters, Patrizia (23 years old and future biologist), Francesca (18, future, I hope, chemist), one son Massimo (22, future engineer) , one cat (

whose name is obvious, CO_2). At present we are living in a cosy green village on the outskirts of Milano, where bicycle is a must, and I appreciate classical music and walk.

Comments

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