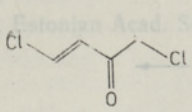
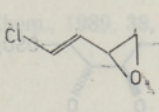
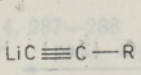


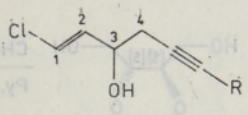
(1)



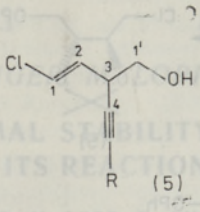
(2)



(3)



(4)



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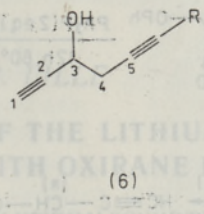
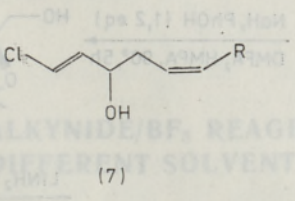


(6)



(7)

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REFERENCES

1. *Лопп М., Паю А., Кангер Т., Вялимяэ Т., Лилле Ю.* Алкилирование этиленового кеталя 1-хлоро-4-бромо-1Е-бутен-3-она. Синтез иненовых и диеновых фрагментов лейкотриенов и феромонов // Ж. орг. хим., 1989, **25**, вып. 4, 869—870.
2. *Mäeorg, U.* Zink/copper as a reducing agent. 1. Reduction of some enyols and alkynols // Acta Comm. Univ. Tartuensis, 1982, N 616, 50—54.

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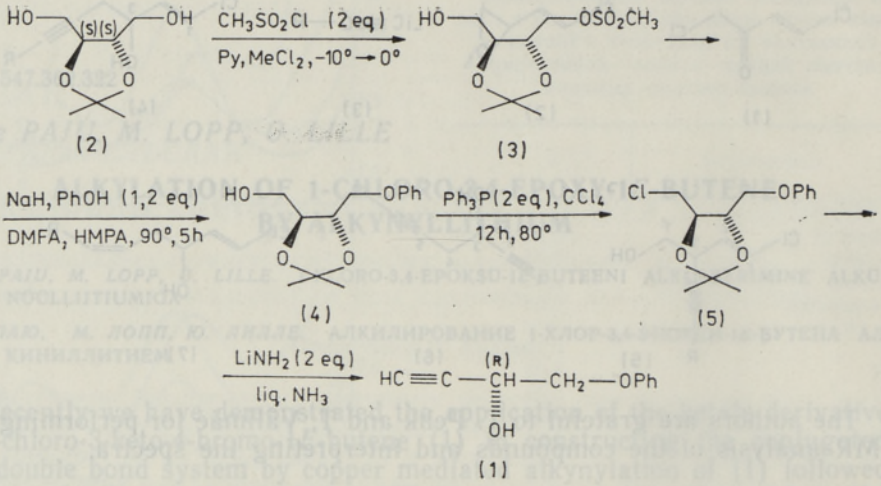
SYNTHESIS OF (R)-(-)-4-PHENOXY-3-HYDROXY-1-BUTYNE FROM TARTARIC ACID DERIVATIVES

Piret NIIDAS, T. KANGER, M. LOPP, Ü. LILLE. (R)-(-)-4-FENOKSÜ-3-HÜDROKSÜ-1-BUTÜÜNI SÜNTEES VIINHAPPE DERIVAATIDEST

Пиpет НИЙДАС, Т. КАНГЕР, М. ЛОПП, Ю. ЛИЛЛЕ. СИНТЕЗ (R)-(-)-4-ФЕНОКСИ-3-ГИДРОКСИ-1-БУТИНА ИЗ ПРОИЗВОДНЫХ ВИННОЙ КИСЛОТЫ

Tartaric acid is a readily available chiral natural product which can be used as a source of chiral building blocks for synthesis [1]. We have synthesized (R)-(-)-4-phenoxy-3-hydroxy-1-butyne (1), a ω-chain precursor in prostaglandin synthesis [2], starting from 2,3-O-isopropylidene-1.2(*S*), 3(*S*), 4- butane-tetraol (2) [3]. Monomesylate (3) was alkylated with sodium phenylate in DMFA to give phenoxy substituted butanol (4) (65%; $[\alpha]_D^{25} = -11.3°$, c 8.23 $CHCl_3$). After the chlorination by $CCl_4 - Ph_3P$ according to [4] (83%) and the elimination according to [5] (85%),



(R)-(-)-4-phenoxy-3-hydroxy-1-butyne (1) $[\alpha]_D^{25} = -27°$, c 7.02 $CHCl_3$ was obtained. HPLC, IR and UV spectra of the compound (1) were identical to those of the racemic compound (1) (synthesized according to [2]). The optical purity of alkynol (1) determined by HPLC using the Mosher [6] and O-methoxymandelate [7] ester was found to exceed 99%.

REFERENCES

1. *Seebach, D., Hungerbühler, E.* Synthesis of enantiomerically pure compounds. Tartaric acid, an ideal source of chiral building blocks for synthesis // Modern Synthetic Methods, 1980, **2**, 91—171.
2. *Толстиков Г. А., Мифтахов М. С., Данилова Н. А., Галин Ф. З.* Простаноиды V. ω-Цепь для 16-арилокси-17, 18, 19, 20-тетранорпростагландинов // Ж. орг. хим., 1983, **19**, вып. 9, 1857—1866.
3. *Feit, P. W.* 1,4-Bismethanesulfonates of the stereoisomeric butaneteraols and related compounds // J. Med. Chem., 1964, **7**, 14—17.
4. *Gruber, L., Tömösközi, I., Radics, L.* Convenient preparation of β-halovinyl-ketones under non-acidic conditions // Synthesis, 1975, N 11, 708.
5. *Yadav, J. S., Chander, M. C., Joshi, B. V.* An expeditious approach for the synthesis of optically active acetylenic alcohols // Tetrahedron Lett., 1988, **29**, N 22, 2737—2740.
6. *Dale, J. A., Dull, D. L., Mosher, H. S.* α-Methoxy-α-trifluoromethylphenylacetic acid, a versatile reagent for the determination of enantiomeric composition of alcohols and amines // J. Org. Chem., 1969, **34**, 2543—2549.
7. *Trost, B. M., Belletire, J. L., Godelski, S., McDougal, P. G., Balkovec, J. M.* On the use of the O-methylmandelate ester for establishment of absolute configuration of secondary alcohols // J. Org. Chem., 1986, **51**, 2370—2374.

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Institute of Chemistry

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