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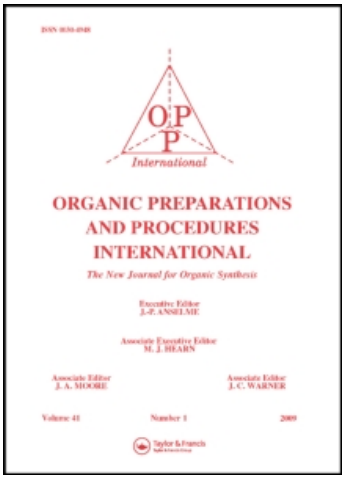

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SYNTHESIS OF 3-(2-METHYLPHENOXY)-AND 3-(2,6-DIMETHYLPHENOXY)PROPYNE

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REFERENCES

1. Part 6 of the series of "Studies on Friedel-Crafts Chemistry." Part 4: M. Tashiro and T. Yamato, Submitted to J. Org. Chem.
2. M. Tashiro, G. Fukata and T. Yamato, Org. Prep. Proced. Int., 8, 263 (1976).
3. C. Ruchart and S. Eichler, Chem. Ber., 95, 1921 (1962).
4. L. Harvey, G. J. Gleicher and W. D. Totherour, Tetrahedron, 25, 5019 (1969).
5. M. M. Tchitchibabin, S. Elgasine and V. Lengold, Bull. Soc. Chim. France, 43, 238 (1928).

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SYNTHESIS OF 3-(2-METHYLPHENOXY)- AND 3-(2,6-DIMETHYLPHENOXY)PROPYNE

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New compounds IIa and IIb have been prepared from the reaction of 3-bromopropyne with Ia and Ib respectively in refluxing acetone and anhydrous potassium carbonate

$$\underset{\text{I}}{\text{ArOH}} + \text{BrCH}_2\text{C}{\equiv}\text{CH} \xrightarrow[\Delta,\ \text{Acetone}]{\text{K}_2\text{CO}_3} \underset{\text{II}}{\text{ArOCH}_2\text{C}{\equiv}\text{CH}}$$

a) Ar = $2\text{-CH}_3\text{C}_6\text{H}_4$ b) Ar = $2,6\text{-(CH}_3)_2\text{C}_6\text{H}_3$

EXPERIMENTAL

An F & M gas chromatograph, Model 810, equipped with a flame ionization detector and a 4 ft. by 0.25 in. 10% Carbowax 20 M, was operated at 200° for all vpc analyses. IR spectra were obtained using a Beckman Model 10 grading IR spectrophotometer with potassium bromide cells. Nmr spectra

were recorded in carbon tetrachloride with a Varian EM-390 spectrometer employing tetramethylsilane as an internal reference. The elemental analyses were performed by M-H-W Laboratories, Garden City, Michigan 48135. Indices of refraction were measured with a Bausch and Lomb Abbe-3L refractometer. Boiling points are corrected.

Preparation of 3-(2-methylphenoxy)propyne(IIa).- Into a 1-l. two-necked flask equipped with a condenser and a mechanical stirrer were placed 130 g (1.2 moles) of 2-methylphenol, 250 ml of acetone and 182 g (1.3 moles) of anhydrous potassium carbonate. To this stirred refluxing solution, 127 g (1.17 moles) of 3-bromopropyne was added dropwise. After being heated to reflux for 15 hrs, the reaction mixture was cooled, poured into 500 ml of water and extracted with 100 ml of ether. The aqueous layer was separated and extracted with three 75 ml portions of ether. The ethereal extracts were combined, washed with 5% aqueous sodium hydroxide and then with water until the aqueous layer was neutral and then dried over magnesium sulfate. The ether was evaporated and the residue distilled in vacuo to give 76.3 g (46%) of 3-(2-methylphenoxy)propyne, bp. 83°/1.5 mm, n_D^{25} 1.5301, which analyzed, by vpc, to be greater than 99% pure.

nmr spectrum: δ 2.23 (s, 3 protons), 2.42 (t, 1 proton, J = 2.40Hz), 4.60 (d, 2 protons, J = 2.40Hz) and 6.98 (m, 4 protons).

Anal. Calcd. for $C_{10}H_{10}O$: C, 82.07; H, 6.90.

Found: C, 82.16; H, 6.71.

In a similar fashion, 3-(2,6-dimethylphenoxy)propyne(IIb) was obtained in 82% yield (60°, 72 hrs), bp. 81°/1.5 mm, n_D^{25} 1.5188.

Anal. Calcd. for $C_{11}H_{12}O$: C, 82.46; H, 7.56.

Found: C. 82.86; H, 7.55.

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