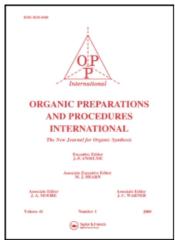
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## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t902189982

# AN UNUSUAL SYNTHESIS OF 2,6-Di-*t*-BUTYL-4-ISOPROPENYLPHENOL

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**To cite this Article** Avar, Lajos(1982) 'AN UNUSUAL SYNTHESIS OF 2,6-Di-*t*-BUTYL-4-ISOPROPENYLPHENOL', Organic Preparations and Procedures International, 14: 3, 197 — 198

To link to this Article: DOI: 10.1080/00304948209354905 URL: http://dx.doi.org/10.1080/00304948209354905

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500 037, India.

- 1. G. Newbery and M. A. Phillips, J. Chem. Soc., 3049 (1928).
- J. R. Geigy A-G., Brit. Patent 729,441 (1955); Chem. Abstr.,
  50, 8731a (1956).
- G. Thuillier, J. Laforest, P. Bessin, J. Bonnet and J. Thuillier, Eur. J. Med. Chem.-Chim. Ther., 10, 37 (1975).
- 4. K. Rufenacht, H. Kristinsson and G. Mattern, Helv. Chim. Acta, 59, 1593 (1976).

AN UNUSUAL SYNTHESIS OF 2,6-Di-t-BUTYL-4-ISOPROPENYLPHENOL

Submitted by Lajos Avar (5/22/81)

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The reaction of 3,5-di- $\underline{t}$ -butyl-4-hydroxy- $\alpha$ -bromo- $\alpha$ -methyl-propiophenone(I)<sup>1,2</sup> with sodium hydroxide leads to 2,6-di- $\underline{t}$ -butyl-4-isopropenylphenol (II), described earlier by Braun and Maier<sup>3</sup> by dehydration of 2(3,5-di- $\underline{t}$ -butyl-4-hydroxyphenyl)-2-propanol with neutral aluminium oxide.

HO CO-C-Br 
$$CH_3$$
  $CH_3$   $CH_3$   $CH_3$ 

#### EXPERIMENTAL

The IR spectra were measured with a Perkin-Elmer 257 Grating Infrared Spectrophotometer. The NMR spectra were determined with A Bruker Spectro Spin WP 60 with TMS as internal standard.

2,6-Di-t-butyl-4-isopropenylphenol (II).- 3,5-Di-t-butyl-4-hydroxy-α-bromo-α-methylpropiophenone (35.5 g, 0.1 mol) was suspended in 150 ml isopropanol at  $58-60^{\circ}$  under  $N_2$  atmosphere in a 350 ml three-necked flask equipped with a thermometer, a stirrer, an addition funnel and a gas inlet tube. Then a solution of sodium hydroxide (4.1 g, 0.125 mol) in 50 ml water was added with stirring to the pale yellow suspension within 15 min. The resulting pale yellow solution was stirred for an additional 45 min. at  $58-60^{\circ}$ , then cooled to  $0-5^{\circ}$  whereupon the product precipitated as pale yellow crystals. The solid was collected, washed with 200 ml water and then recrystallized from 90 ml methanol to yield 17.3 g (70%) almost colorless needles, mp.  $79-80^{\circ}$ , lit.  $377-78^{\circ}$ .

NMR(CDCl<sub>3</sub>):  $\delta$  1.9 (s, 18,  $\underline{t}$ -C<sub>4</sub>H<sub>9</sub>), 2.3 (s, 3, CH<sub>3</sub>), 5.0 (1, OH) 5.24 (d, 2, C=CH<sub>2</sub>), 7.72 (s, 2, ArH).

IR(KBr): 3650 (sterically hindered OH), 1630, 1440, 890 (isopropenyl)  $\,\mathrm{cm}^{-1}$ .

Anal. Calcd. for  $C_{17}^{H}_{26}^{O}$ : C, 82.9; H, 10.6; O, 6.5 Found: C, 82.4; H, 10.4; O, 6.7

### REFERENCES

- L. H. Schwarz and R. V. Flor, J. Org. Chem., <u>34</u>, 1449 (1969).
- A. A. Volod'kin, N. V. Protnykh and V. V. Ershov, Jzv. Akad. Nauk SSSR, Ser. Khim., 1967, 947 (Russ), CA, 68 39279b.
- D. Braun and B. Maier, Makromol. Chemie, <u>167</u>, 119 (1973).