LETTERS TO THE EDITOR

UDC 547.722 + 542.941/2

PECULIARITIES IN THE HYDROGENATION OF ALKENYLFURANS WITH A DOUBLE BOND SITUATED IN THE 2, 3 POSITION TO THE RING

Yu. I. Tarnopol'skii and V. N. Belov

Khimiya Geterotsikilicheskikh Soedinenii, Vol. 2, No. 2, p. 315, 1966

Up to the present there has been no doubt about the possibility of selective hydrogenation of the side chain double bond in alkenylfurans [1]. In particular, catalysts consisting of palladium on carriers ($SrCO_3$ [2], $BaSO_4$ [3, 4]) have been used. They have been used to hydrogenate compounds with the double bond at various positions in the side chain [2-4], but not alkenylfurans with the double bond at the 2, 3 position. Having synthesized compounds of this latter type [5], the present authors attempted to hydrogenate selectively 2-(3-methylbuten-2-yl) furan and 2-(2,3-dimethylbuten-2-yl) furan, but it was unexpectedly found that with $Pd/BaSO_4$ and at atmospheric pressure hydrogenation was non-selective, and gave the corresponding alkyltetrahydrofurans. Hydrogen uptake rate fell uniformly, and slowed down greatly when hydrogenation was complete.

Using the same catalyst, results for the selective hydrogenation of 2-vinylfuran [4] have been reproduced.

3.5 g 2-(3-methylbuten-2-yl) furan was hydrogenated at atmospheric pressure and room temperature, using 2 g 5% $Pd/BaSO_4$ in 25 ml dry ether, and after 9 hr 1560 ml (92%; at NTP) hydrogen, 890 ml being taken up in the first hour. When hydrogenation ceased, the catalyst was filtered off, the ether distilled off, 2–3 drops of bromine added to the residue (until a yellow color was obtained), which was then distilled under reduced pressure, to give 1.35 g (37%) 2-isoamyltetrahydrofuran, bp 79° (27 mm); d_4^{20} 0.8589; n_D^{20} 1.4340. Found: MR_D 43.1. Calculated for MR_D 43.6. The literature gives [3]: bp 172–173° (764 mm); d_4^{20} 0.8550; n_D^{20} 1.4330.

3 g 2-(2, 3-dimethylbuten-2-yl) furan was hydrogenated similarly. After 8 hr 1340 ml H_2 (98%) was absorbed, 660 ml being taken up in the first hour. Yield 2.3 g (74%) 2-(2, 3-dimethylbutyl) tetrahydrofuran, bp 92–93° (25 mm); d_4^{20} 0.8720; n_D^{20} 1.4415. Found: C 76.98; H13.06%; MR_D 47.5. Calculated for $C_9H_{20}O$: C 76.93; H 12.91%; MR_D 48.2.

REFERENCES

1. I. F. Bel'skii and N. I. Shuikin, Usp. khim., 32, 707, 1963.
2. R. Rallings and J. Smith, J. Chem. Soc., 618, 1953.
3. A. Quilico, F. Piozzi, and M. Pavan, Tetrah., 1, 177, 1957.
4. Yu. K. Yur'ev, N. S. Zefirov, and V. M. Gurevich, ZhOKh, 31, 3531, 1961.
5. Yu. I. Tarnopol'skii and V. N. Belov, ZhOrKh, 1, 595, 1965.
6. V. G. Bukharov and T. E. Pozdnyakov, Izv. AN SSSR, OKhN, 135, 1961.

13 March 1965

Mendeleev Institute of Chemical Technology, Moscow

UDC 547.836.6

REACTION OF ANTHRAPYRIDONES WITH NUCLEOPHILIC REAGENTS

M. V. Kazankov and V. N. Ufimtsev

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 2, No. 2, pp. 315-316, 1966

The present authors previously showed that anthrapyridone (7H-dibenz [f, ij] isoquinoline-2, 7 [3H] dione) (I) and N-methylanthrapyridone (II) react with aliphatic amines to give the corresponding 1-alkylaminoanthrapyridones [1]. Continuing the research, the reaction of anthrapyridones with other nucleophilic reagents has been studied. Boiling N-methylanthrapyridone with an aqueous dioxane solution of sodium hydroxide, gave a 50% yield of a compound which, after recrystallization from acetic acid had mp 299–300° C (decomp). Found: C 73.57, 73.71; H 3.80, 3.85; N 5.00, 5.22%. Calculated for $C_{17}H_{11}NO_3$: C 73.62; H 4.00; N 5.05%. The compound was identical in chemical