OF SULFAMINIC ACID, NH3.SO3

N. S. Zefirov, N. V. Zyk, A. G. Kutateladze,UDC 542.958.3+542.945.22:E. K. Butina, and S. Z. Vatsadze547.592.2:547.538.141

We have shown that the reactions of sulfenamides with terminal and conformationally labile olefins in the presence of free SO_3 or its complex with pyridine lead to β -aminosulfides [1]. In a continuation of a systematic study of the addition of sulfenamides to olefins, we carried out the reaction of sulfenamides with olefins in the presence of sulfaminic acid, $NH_3 \cdot SO_3$ (I), which is the complex of ammonia with SO_3 , and found that heating of a mixture of N,N-diethylphenylsulfenamide (II), sulfaminic acid (I), and an olefin in nitromethane with subsequent treatment of the reaction mixture with IO% hydrochloric acid gives the hydrochloride salts of the corresponding primary amines, containing a β -phenylthio group

 $(III) \xrightarrow{\text{I. PhS}-\text{NEt}_{5}(II), \text{ NH}_{3} \cdot \text{SO}_{3}(I)} (IV)$

a. A mixture of cyclohexene (III), sulfenamide (II), and acid (I) was heated in nitromethane at 90-100°C to give the hydrochloride salt of trans-2-phenylthiocyclohexylamine (IV) in 48% yield. The PMR spectrum of the free base in CDCl_3 (δ , ppm): 7.15 m (5H_{arom}), 2.6 m (2H, HCN and HCS), 1.95 s (2H, NH₂), 1.8-1.0 m (8H). For the hydrochloride, Found: C, 58.78; H, 7.32; N, 5.51%. Calculated for $C_{12}H_{18}ClNS$: C, 59.10; H, 7.44; N, 5.75%.

b. The analogous reaction with styrene (V) gives the hydrochloride salt of 2-phenyl-thio-1-phenylethylamine (VI) in 44% yield. The PMR spectrum of the free base in $CDCl_3$ (δ , ppm): 7.15 m (10H_{arom}), 4.0 d. d (1H, HCN), 3.4-2.8 m (2H, HCS), 1.8 s (2H, NH₂). For the hydrochloride, Found: C, 62.83: H, 6.02; N, 5.26%. Calculated for $C_{14}H_{15}CINS$: C, 63.19; H, 6.06; N, 5.29%.

Thus, we propose a new method for the one-step aminosulfenylation of olefins. The synthetic scope of this reaction is now under study.

LITERATURE CITED

 N. S. Zefirov, N. V. Zyk, A. G. Kutateladze, et al., Dokl. Akad. Nauk SSSR, <u>301</u>, No. 6, 1385 (1988).

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