

SYNTHESIS OF DIBAZOLE IN THE SOLID PHASE UNDER HIGH PRESSURE AND SHEAR STRESS

N. P. Chistotina, A. A. Zharov, N. E. Agafonov,
and V. S. Bogdanov

UDC 541.12.034.2:542.91:547.785.5

We are the first to report that an equimolar mixture of o-phenylenediamine and phenylacetic acid in the solid phase under high pressure and shear stress (HP + SS) react to give 2-benzylbenzimidazole (dibazole). These experiments were carried out on an anvil apparatus [1]. The rate of rotation of the anvil in the HP + SS experiments was 40°/sec. The samples were analyzed after the experiments by PMR spectroscopy on a Bruker WM-250 spectrometer. For comparison, we present the experimental results for 1 GPa and 373 K (in a cylinder-piston apparatus) and melting the reaction mixture at 0.1 MPa (in glass ampuls). Upon maintenance for 5 h at 1 GPa and 373 K, only trace amounts of dibazole are formed in the reaction mixture. The results of other experiments are given in Tables 1 and 2.

TABLE 1. Yield of Dibazole under HP + SS Conditions at 293 K

P, GPa	Angle of rotation of the anvil, deg	Yield, %
4	360	5.3
8	90	5.6
8	360	11.1

TABLE 2. Yield of Dibazole in Experiments with the Melt at 0.1 MPa

T, K	Time, min	Yield, %
383	10	3.0
383	40	22.0
393	10	8.3

As in the dimerization of cyclopentadiene [2], we compared the rate of formation of dibazole in the liquid phase and in the solid phase under HP + SS conditions. For this purpose, the data in Tables 1 and 2 were extrapolated to the same pressure and temperature. The rate of dibazole formation upon HP + SS conditions is more than 10^6 times greater than the reaction rates in the liquid phase and in the case of solid reagents under high pressure but without shear stress.

LITERATURE CITED

1. A. A. Zharov and N. P. Chistotina, Prib. Tekh. Éksp., No. 2, 229 (1974).
2. A. A. Zharov, V. S. Abramov, and V. M. Zhulin, Izv. Akad. Nauk SSSR, Ser. Khim., No. 8, 1706.

N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 6, p. 1452, June, 1989. Original article submitted February 15, 1989.