Synthesis and identification of the novel urazolediamine with phthalamide group

H Mighani*, H Nasr Isfahanib & S Mighanib

aDepartment of Chemistry, Golestan University, Gorgan, P.O.Box 155, Iran
bDepartment of Chemistry, Shahrood University, Shahrood, Tehran Street, Iran
cDepartment of Petroleum Engineering, Amir Kabir University, Tehran, Hafez Street, Iran
E-mail: hmighani@gu.ac.ir

Received 13 September 2010; accepted (revised) 25 November 2011

A diamine, 4-(4-phthalimidophenyl)-1,2,4-triazolidyne-3,5-diamine or urazolediamine (UD) has been synthesized in 8 steps from 4-nitrobenzoic acid. The diamine has been characterized by FTIR, 1H NMR and melting point and can be used as a monomer for the preparation of polyesters, polyamides and polyimides.

Keywords: Urazolediamine, 4-nitrobenzoic acid, monomer, FTIR, 1H NMR

Phthalamide-containing compounds have attracted attention for scientists1-8. Introduction of bulky alkyl or aryl substituents reduces the hydrogen bonding at the amide linkage, resulting in flexible linkage9,10, asymmetric/symmetric bulky units in the aromatic rings, pendant phenyl group into the polymers backbone11,12, etc. The object of this work is to prepare Urazolediamine (UD) which literature search showed has not been prepared before. UD can be a useful monomer for preparation of polyamides, polyesters and polyimides and it can also be used in resin and polymer production.

Experimental Section

Preparation of compounds

4-(4-Phthalimidophenyl)-1,2,4-triazolidyne-3,5-dione13

A 25 mL round bottomed flask was charged with 4-(4-aminophenyl)urazole (0.2 g, 1.04 mmol) and a mixture of solvents, acetic acid/pyridine (3/2) and phthalic anhydride (0.15 g, 1.04 mmol). The mixture was subsequently stirred at RT for 2 days so that the amic acid was produced as a white precipitate. The solution was stirred at reflux temperature for 8 hr and then filtered and the white precipitate washed with ethanol, filtered and dried. The white purified sample (1.38 g) was obtained in 76% yield (0.46 g) with m.p. 363-65°C. IR (KBr): 3200 (w), 3100 (m), 2800 (w), 1760 (w), 1700 (s,br), 1520 (s), 1460 (s), 1380(s), 1290 (m), 1220 (m,sh), 1160 (w), 1120(s), 1080(s), 880 (s), 840 (s), 790 (m,sh), 770 (m), 720 (s), 680 (s), 660(m), 530 (s) cm⁻¹; 1H NMR (DMSO-d6): δ 10.57 (s, 2H, br), 8.00(m, 2H, J=10 Hz), 7.93 (m, 2H, J=10 Hz), 7.63 (d, 2H, J=10 Hz), 7.58 (d, 2H, J=10 Hz).

4-(4-Phthalimidophenyl)-1,2,4-triazolidyne-3,5-diamine

A 10 mL round bottomed flask was charged with 4-(4-Phthalimidophenyl)-1,2,4-triazolidyne-3,5dione (2.5 g, 7.737 mmol), hexamethylenediisocyanate (0.2615 g, 1.555 mmol) and 3 mL dimethylformamide. The mixture was subsequently stirred at RT for 2 days and the precipitate obtained was resuspended in a solution with low concentration of water/acid, filtered and dried. The purified sample, it was confirmed that the prepared compounds, it was confirmed that the

Note
urazolediamine can be prepared in relatively high yields. The obtained compounds were characterized by FTIR, $^1$H NMR and melting points. The introduction of phthalimide pendant groups in the structure of the diamine resulted in preparation of amorphous polyamides, polyesters and polyimides with very good solubility in aprotic solvents.

References