A NOVEL SYNTHESIS OF N’-SUBSTITUTED-N”-(2-AMINO)-BENZOTHIAZOLELOTHIOUREAS

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Abstract

Recently in this laboratory we carried out by interactions of m-chloro-2-amino-benzothiazole with various thioureas in isopropanol medium to synthesized a novel series of N’-substituted-N”-(2-amino)benzothiazolothiourea. Structure determination and justification of the synthesized compounds were done on the basis of elemental analysis, chemical characteristics and spectral studies.

Keywords:-Various thiourea, m-chloro-2-aminobenzothiazole and isopropanol.

Introduction

In a field of chemical sciences the main stream is synthetic chemistry, which involves a development and synthesis of newer types of molecules. Since from preceding years pharmaceutical chemistry had been massive growth not only in terms of development of novel methodologies for construction of carbon-carbon and carbon-hetero atom bonds but also in terms for development of new strategies, reagents, catalysts, transformations and technologies. A broad spectrum of biological activities is reported associated with a number of heterocyclic compounds. It is found that compounds containing benzothiazolo and triazolo nucleus in a molecule possess to acquire and exhibit a wide range of biological and medicinal activities.
The benzothiazolo nucleus containing molecules showed biological and anti-cancer\(^1\), anti-convulsant\(^2\), anti-allergic\(^3\) anti-tumor\(^4\) anti-malarial\(^5\) antimicrobial\(^6-8\) analgesic\(^9\) anti-inflammatory\(^10\), anti-cancer\(^11-13\) anti-helmintic\(^14\) anti-diabetic\(^15\) activities. Benzothiazol based molecules have application in industry as anti-oxidant agent and vulcanisation accelerators. By considering all these facts we synthesize a novel series N’-substituted-N”-(2-amino)benzothiazolothiourea (IIIa-d) by the interactions of m-chloro-2-aminobenzothiazole (Ia) various thiourea (IIa-d) in isopropanol medium. The newly synthesized compounds may possess more practical utility and also the new thiocarbamido substituent may enhance the potency of the molecules and can also show important biological activities. The tentative reaction is depicted below (Scheme-I)

\[ \text{m-chloro-2-aminobenzothiazole} + \text{substituted thiourea} \rightarrow \text{N'-substituted-N"-(2-amino)benzothiazolothiourea} \]

Where, R= -phenyl, -thiourea, -ethyl, -allyl

(Scheme-I)

**Experimental**

The melting points of synthesized compounds were recorded using hot paraffin bath. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Perkin Elmer spectrometer in the range 4000-400 cm\(^{-1}\) in KBr pellets. PMR spectra were recorded on BRUKER AVANCE II 400 NMR spectrometer with TMS as an internal standard using CDCl\(_3\) and DMSO-d\(_6\) as a solvent. The purity of the compounds were checked on silica gel G plates by TLC with layer thickness of 3mm. All chemicals used were of Merck’s Millipore (Indian made).

**RESULTS AND DISCUSSION**

**Synthesis of N’-phenyl-N”-(2-amino)benzothiazolothiourea (IIIa)**

N’-Phenyl-N”-(2-amino)benzothiazolothiourea (IIIa) was synthesized by refluxing m-chloro-2-aminobenzothiazole (Ia) and phenylthiourea (IIa) in isopropanol medium for 4 hours on water bath, lemon coloured crystals were separated out at room conditions, filtered and dried. Recrystallized from aqueous ethanol.
Properties of N’-phenyl-N”-(2-amino)benzothiazolothiourea (IIIa): Lemon crystalline solid, M.F. C_{14}H_{12}N_{4}S_{2}, Yield 94%, M.P. 186^0\text{C}; \text{^1}H \text{ NMR (400 MHz,DMSO-d}_{6}): 9.9632 (1 H S), 9.8012 (1 H, S J=14.8Hz), 7.7424 (5 H, S), 6.5127 (2 H s), 2.5193 (2 H s), \text{^{13}C}: 180.95, 149.83, 140.83 138.77, 132.81, 129.77, 128.41, 124.61, 123.20, IR: 3419.79 str., 3186.40 Str., 1751.36 Str., 1521.84 Str., 1425.40 Str., 1294.24 Str, 1060.34 Str.; LC-MS (m/z) Mol. Wt.:300.02., 300.10(M+), 224, 150, 92.2.

Properties of N’-thiocarbamido-N”-(2-amino)benzothiazolothiourea (IIIb)
Shining Yellow solid, C_{8}H_{8}N_{4}S_{2}, Yield-89%, M.P. 192^0\text{C}, \text{^1}H \text{ NMR (400 MHz,DMSO-d}_{6}): 9.1054 (1 H S), 8.8801 (1 H, S J=14.8Hz), 8.5621 (1 H, S J=14.8Hz), 6.5127 (1 H s), 2.5193 (2 H s), \text{^{13}C}: 190.20, 152.43, 144.83 142.77, 136.81, 135.77, 130.41, 128.61, IR: 3456.21 str. 3142.45 str., 1636.45 Str., 1747.50 Str, 1450.51 Str., 1154.20 Str, 1059.34 Str.; LC-MS (m/z) Mol. Wt.224.30., 224.10(M+), 165.21, 135.18.

Properties of N’-ethyl-N”-(2-amino)benzothiazolothiourea (IIId)
Dark Yellow solid, C_{10}H_{12}N_{4}S_{2}, Yield-90%, M.P. 182^0\text{C}, \text{^1}H \text{ NMR (400 MHz,DMSO-d}_{6}): 7.9805 (1 H S), 7.6223 (1 H, S J=13Hz), 7.3891 (1 H, S J=13Hz), 6.6223 (1 H, S), 3.5657 (2 H s), 2.5193 (2 H s), 1.0913 (3 H s), \text{^{13}C}: 183.69, 140.46, 140.83 132.95, 129.75, 123.89, 122.33, 120.91, 78.61, 39.43 IR: 3325.21 str., 3095.45 Str., 1654.45 Str, 1545.0 Str., 1256.54 Str., 1062.34 S; LC-MS (m/z) Mol. Wt:252.35., 252.32(M+), 165.20, 92.2.

Properties of N’-allyl-N”-(2-amino)benzothiazolothiourea (IIIId)
Yellow solid, C_{10}H_{11}N_{4}S_{2}, Yield-83%, M.P. 187^0\text{C}, \text{^1}H \text{ NMR (400 MHz,DMSO-d}_{6}) : 9.3512 (1 H S), 9.2041 (1 H, S J=15Hz), 9.2041 (1 H, S J=15Hz), 5.7424 (1 H, S), 4.5127 (2 H s), 2.5193 (2 H q), 1.2039 (3 H t), \text{^{13}C}: 182.95, 151.83, 142.83 140.03, 134.60, 131.77, 129.41, 127, 35.1,38.6 IR: 3345.32 str., 3145.45 Str., 1684.45 Str, 1562.50 Str., 1241.51 S, 1059.34 Str.; LC-MS (m/z) Mol. Wt.: Wt.:252.35., 252.32(M+), 165.20, 150.

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