Electronic Supplementary Material (ESI) for RSC Advances

Supporting information

DBU-diheteroaryl halide adduct as fastest N-diheteroarylating agent.

Sanjeev K. Verma, B.N. Acharya, Ramarao Ghorpade, Ajay Pratap and M. P. Kaushik*

Process Technology Development Division, Defence R & D Establishment, Jhansi Road, Gwalior-474002 (MP) India
Fax: +91(751)2340042, E-mail: mpkaushik@rediffmail.com

Contents

Synthesis of Benzothiazol-2-yl-morpholine (General Procedure 1).

In a test tube 0.01 mol of 2-chlorobenzothiazole and 0.01 mol of DBU was added. A dirty green coloured solution was formed. To this solution add 0.01 mol of Morpholine. White coloured precipitate was formed within 5 minutes at room temperature. The prepitate was washed with water to remove DBU.HCl salt formed in the reaction.

Synthesis of Benzothiazol-2-yl-morpholine (General Procedure 2).

In a test tube 0.01 mol of 2-chlorobenzothiazole and 0.001 mol of DBU and 0.009 mol of $NaHCO_3$ or Na_2CO_3 were added. A dirty green coloured mass was formed. To this solution 0.01 mol of Morpholine was added. The reaction mixture was mixed with spatula. Water was added in the reaction mixture. White coloured precipitates were filtered off.

Note: All the compounds reported in table 1 are known compounds. Spectra related to these compounds is available in Verma, S.K.; Acharya, B.N.; Kaushik, M.P.; *Org.Biomol.Chem.* **2011**, *9*, 1324.

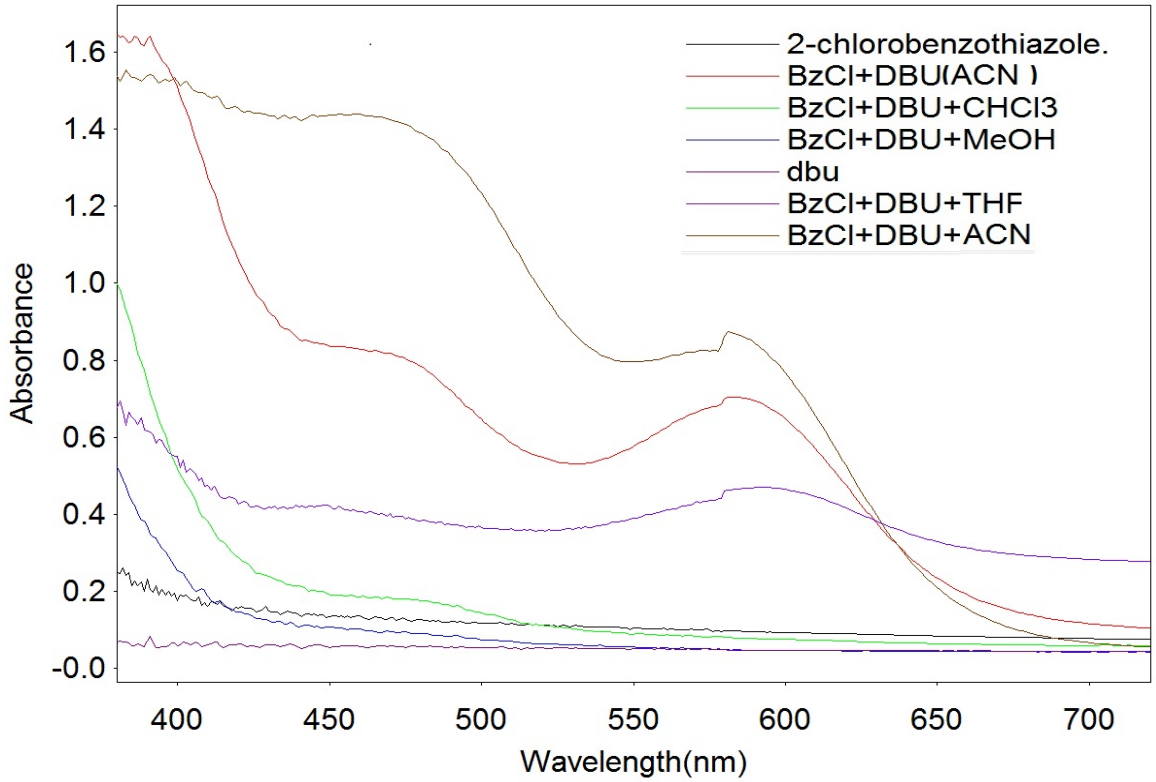

Figure 1: Effect of Solvent on the stability of DBU-ArCl Adduct.

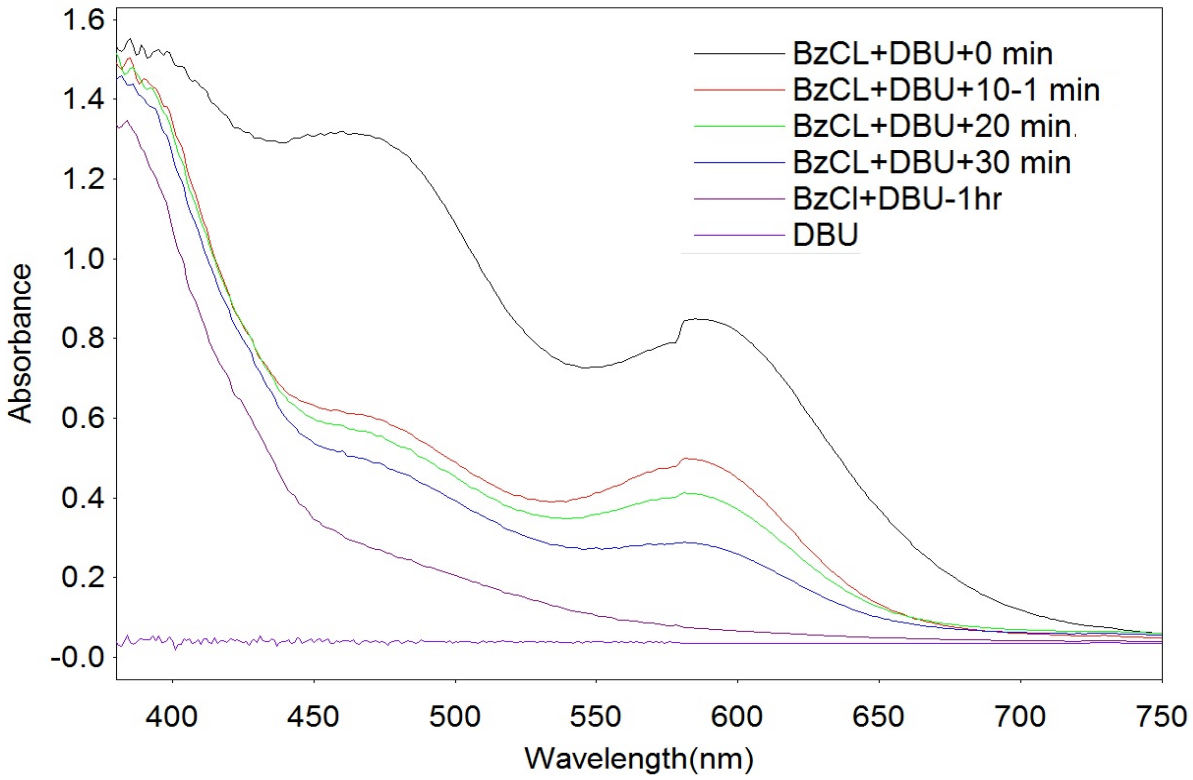

Figure 2: Effect of Time on the stability of DBU-ArCl Adduct.

Note:

BzCl----------2-chlorobenzothiazole

BzCl +DBU + Solvent------2-chlorobenzothiazole mixed with equimolar ratio of DBU and then diluted with respective solvent.

BzCl+DBU(ACN)---------- 2-chlorobenzothiazole and DBU were dissolved in acetonitrile separately and then mixed.

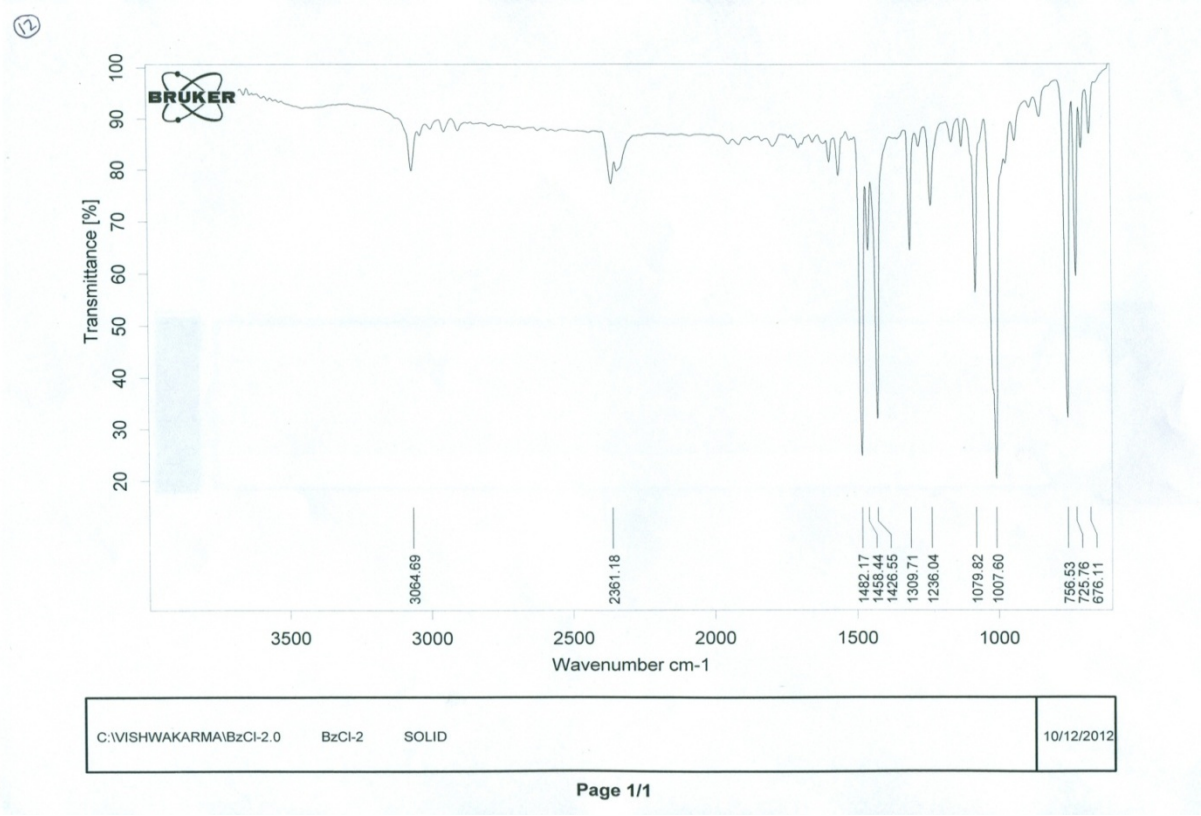

Figure 3: FTIR specta of 2-chlorobenzothiazole.

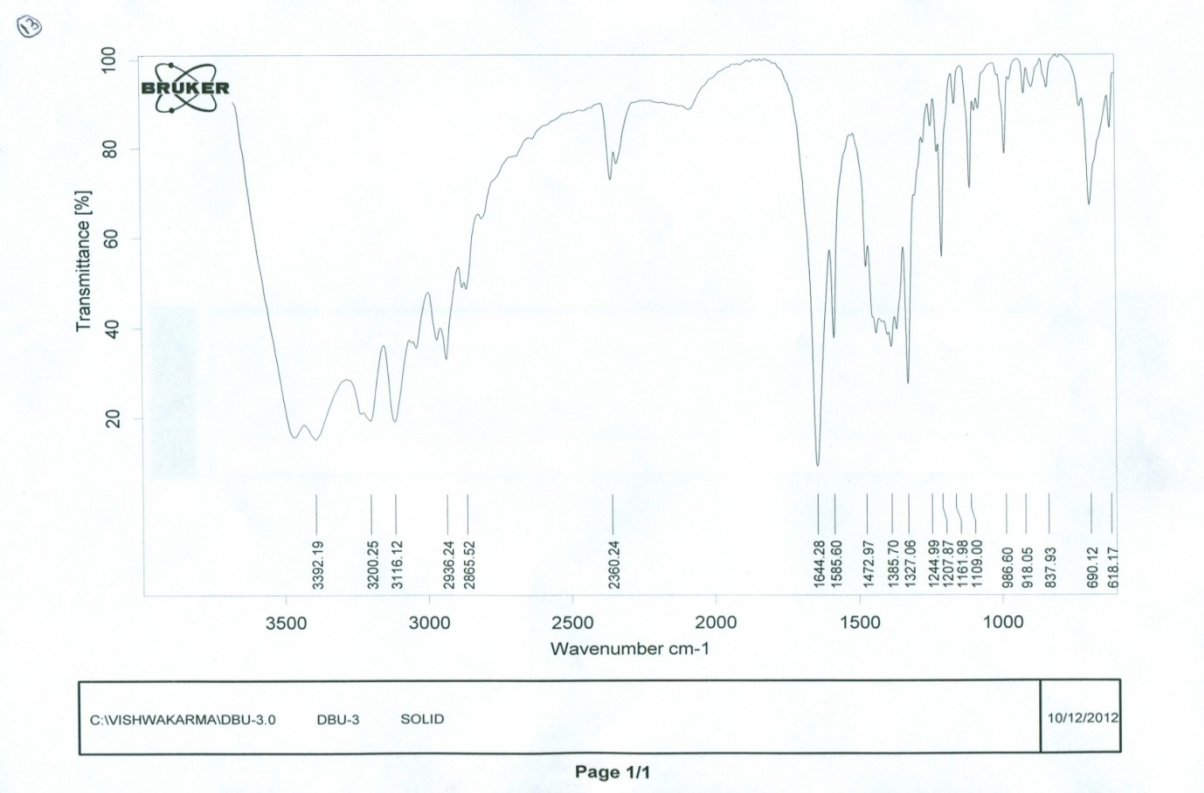

Figure 4: FTIR specta of DBU.

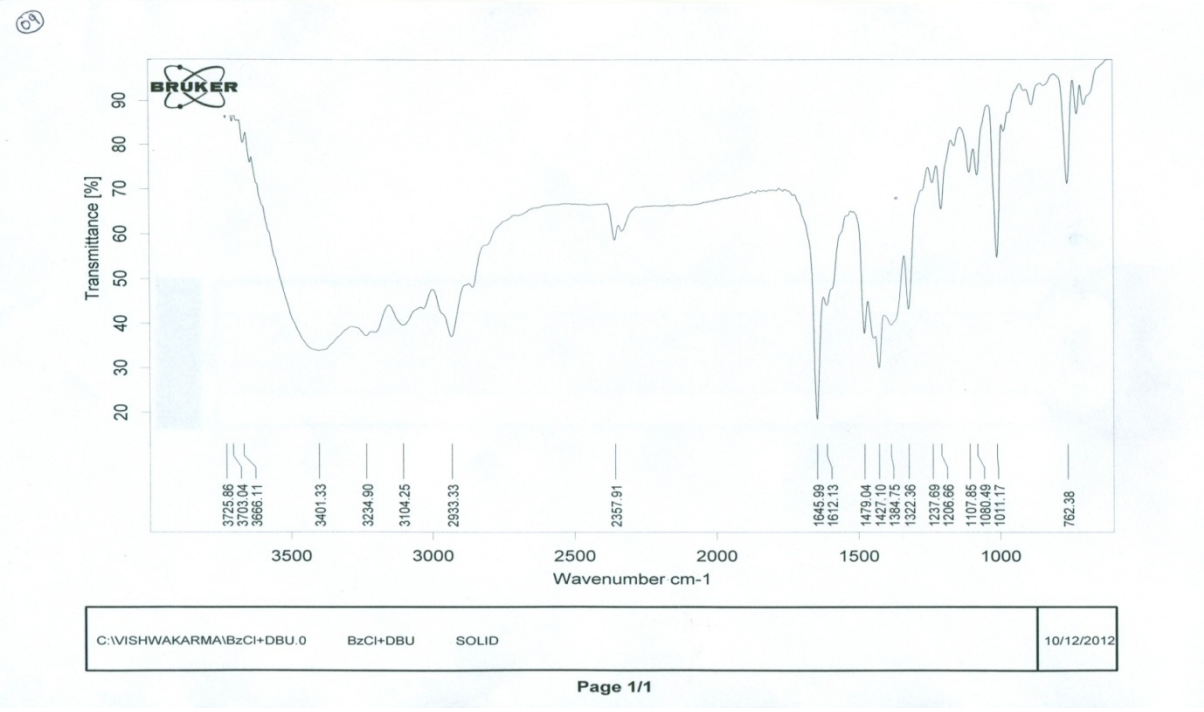

Figure 5: FTIR specta of DBU-2-chlorobenzothiazole adduct

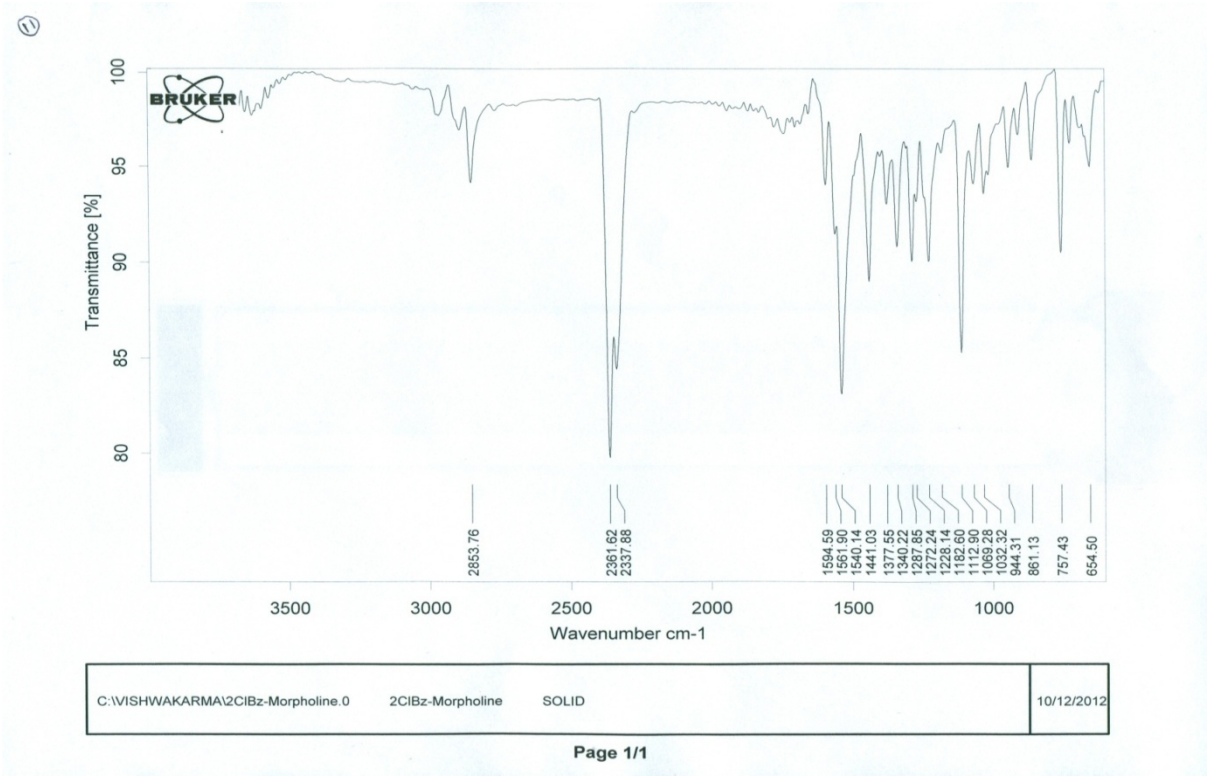

Figure 6: FTIR specta of 4-(benzothiazol-2-yl) morpholine

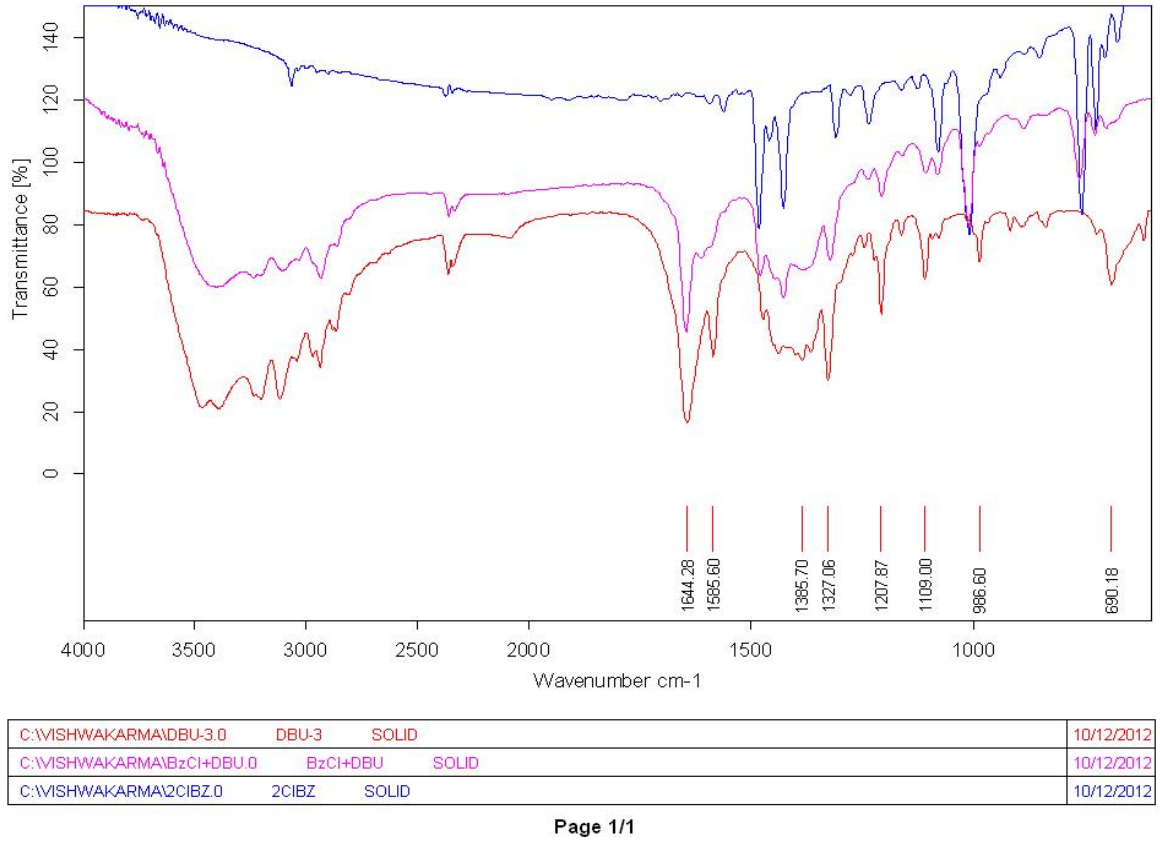

Figure 7: FTIR specta of 2-chlorobenzothiazole, DBU-2-chlorobenzothiazole adduct and DBU.

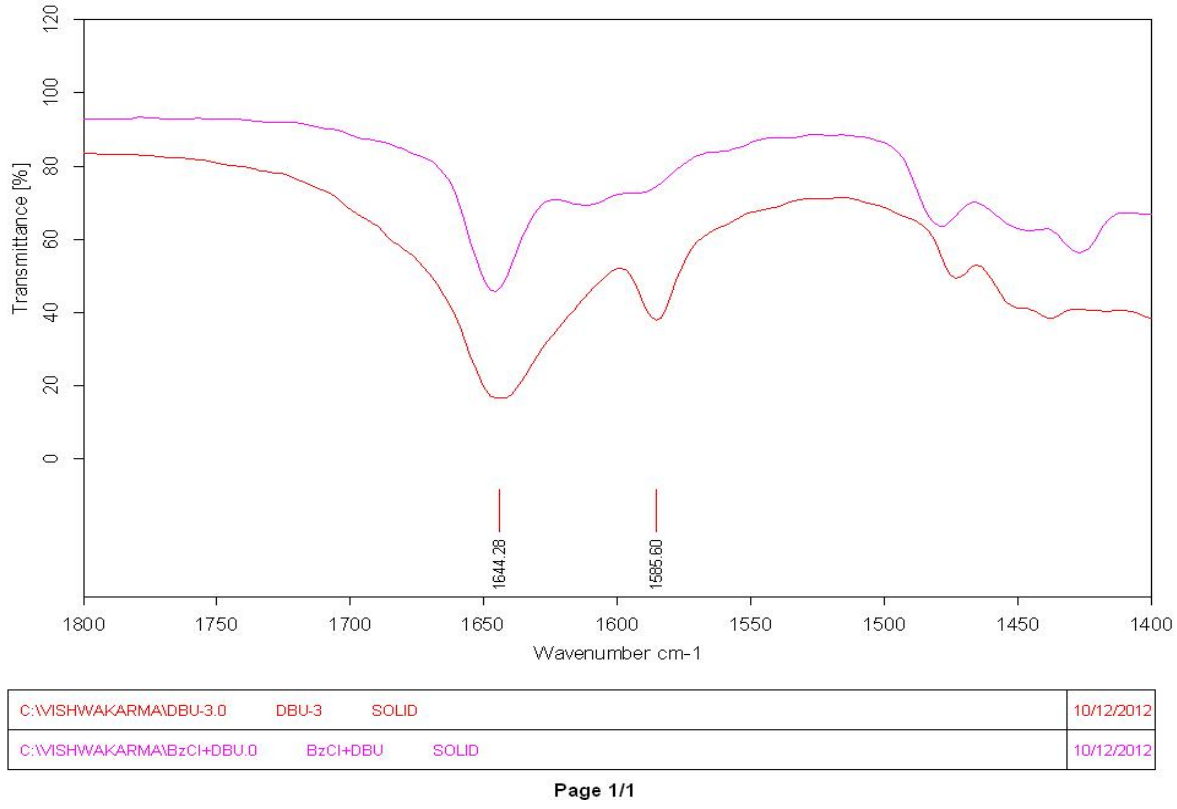

Figure 8: FTIR specta showing shift in the value of DBU at 1585 to 1611 with decrease in intensity.

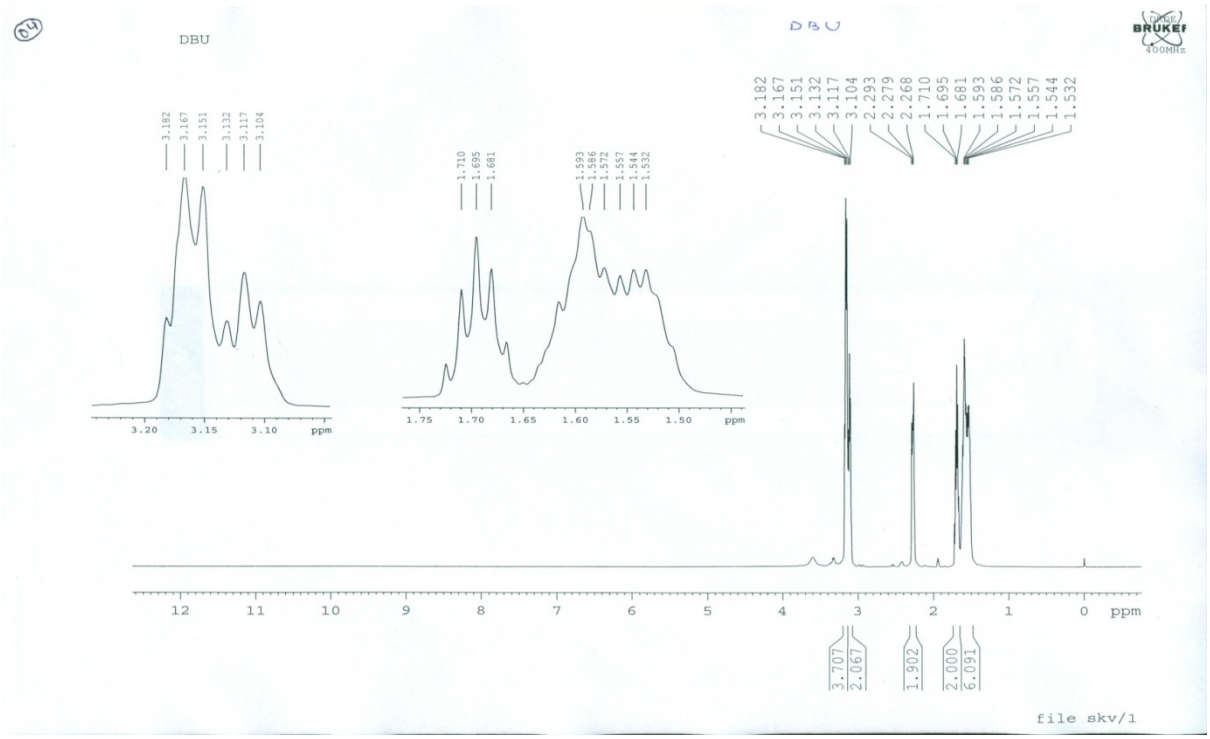

Figure 9: ^{1}H-NMR of DBU

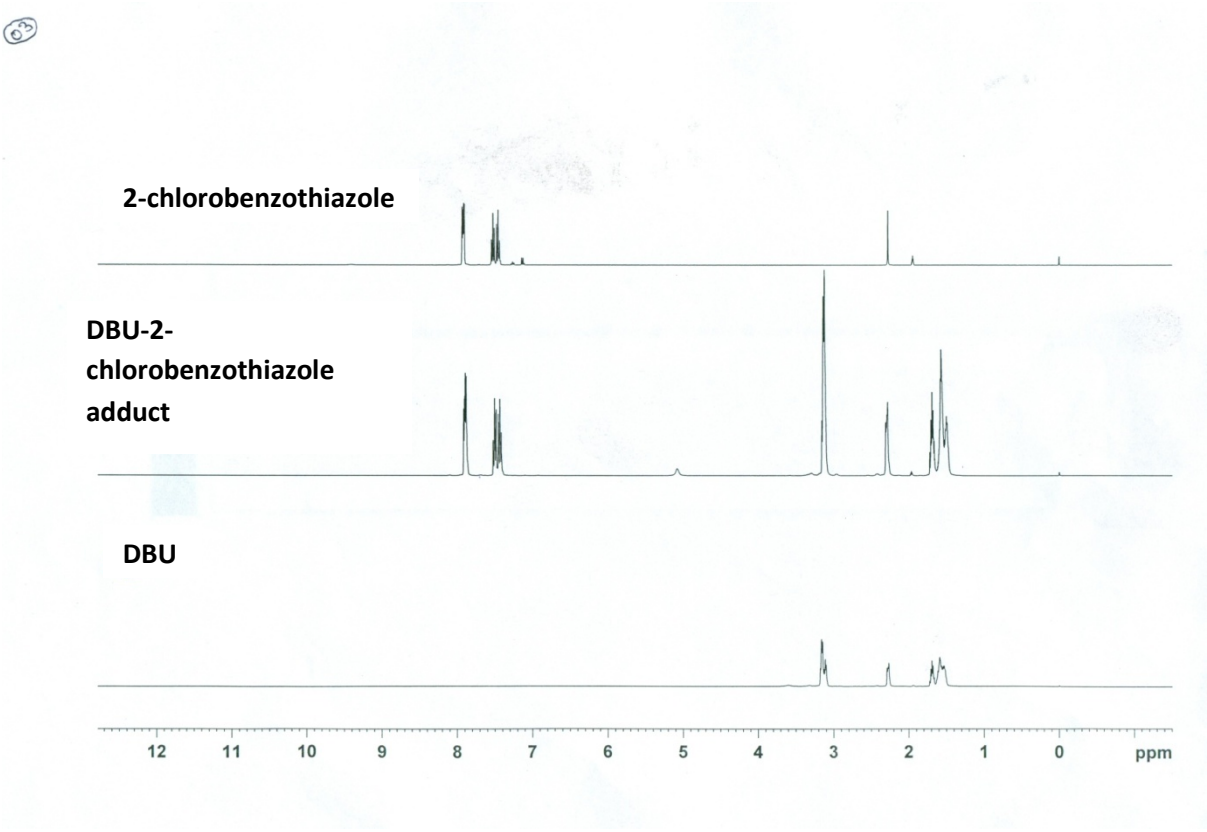

Figure 10: ^{1}H-NMR of 2-chlorobenzothiazole, 2-chlorobenzothiazole-DBU adduct and DBU

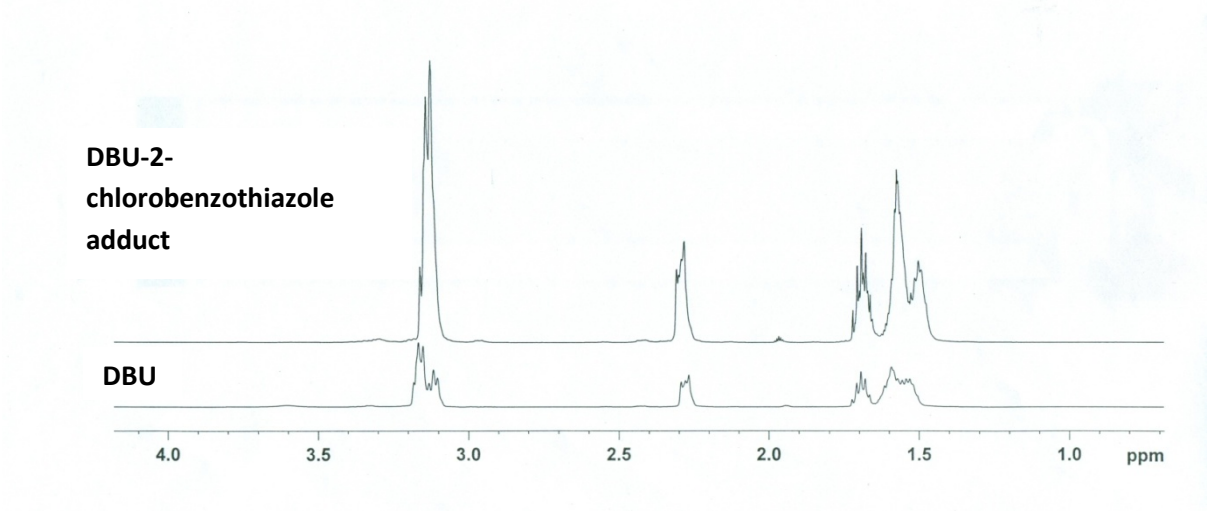

Figure 11: ^{1}H-NMR of DBU-2-chlorobenzothiazole adduct and DBU showing shifting.

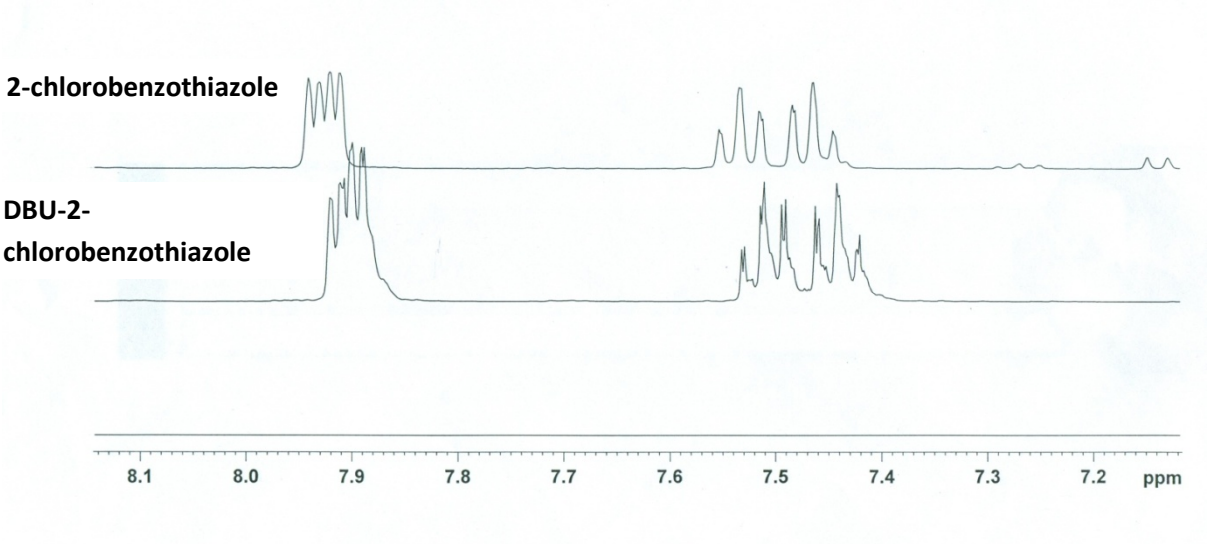

Figure 12: ^{1}H-NMR of 2-chlorobenzothiazole and DBU-2-chlorobenzothiazole adduct showing shifting and splitting in the peaks.

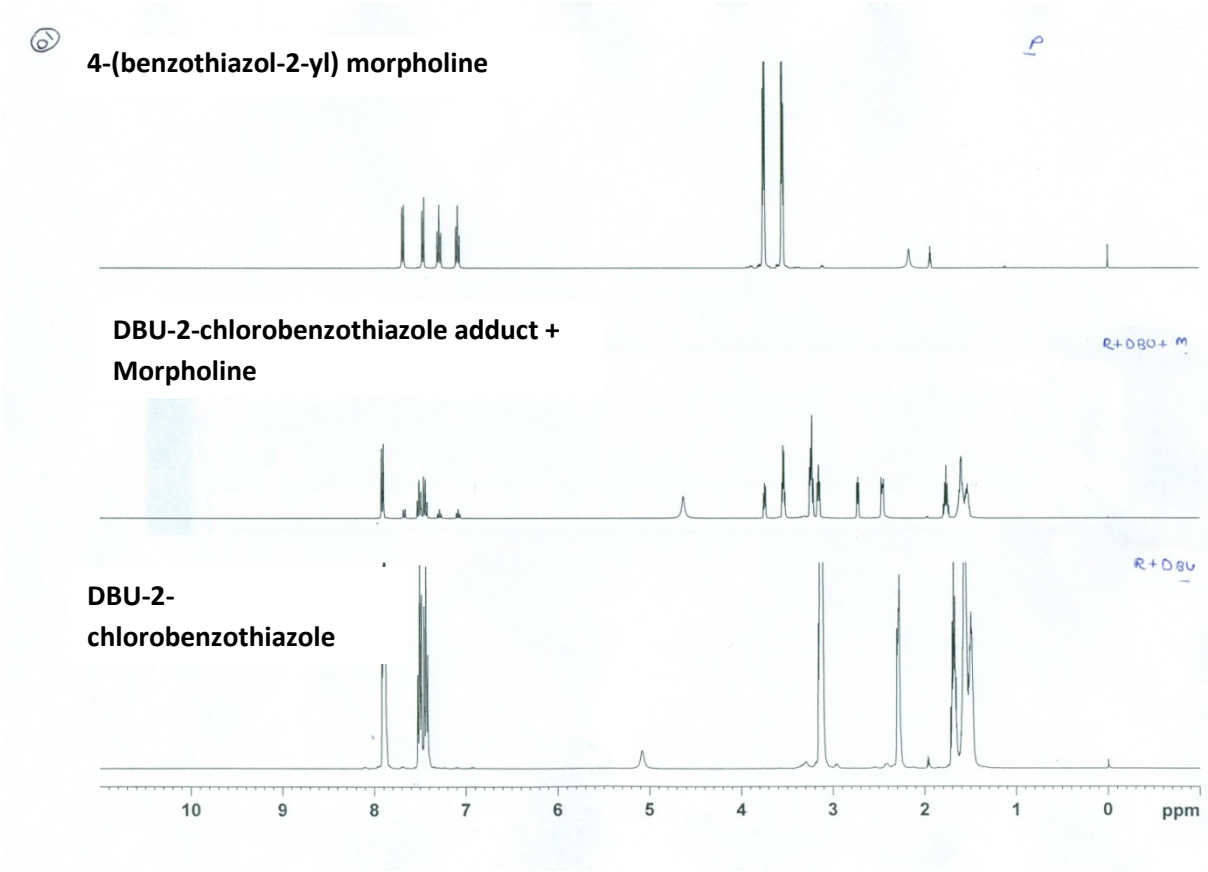

Figure 13: ^{1}H-NMR of 4-(benzothiazol-2-yl) morpholine, DBU-2-chlorobenzothiazole adduct and morpholine in acetonitrile (after 20 minutes) and 2-chlorobenzothiazole-DBU adduct. The spectra showing the formation of 4-(benzothiazol-2-yl) morpholine, however; conversion is not complete.

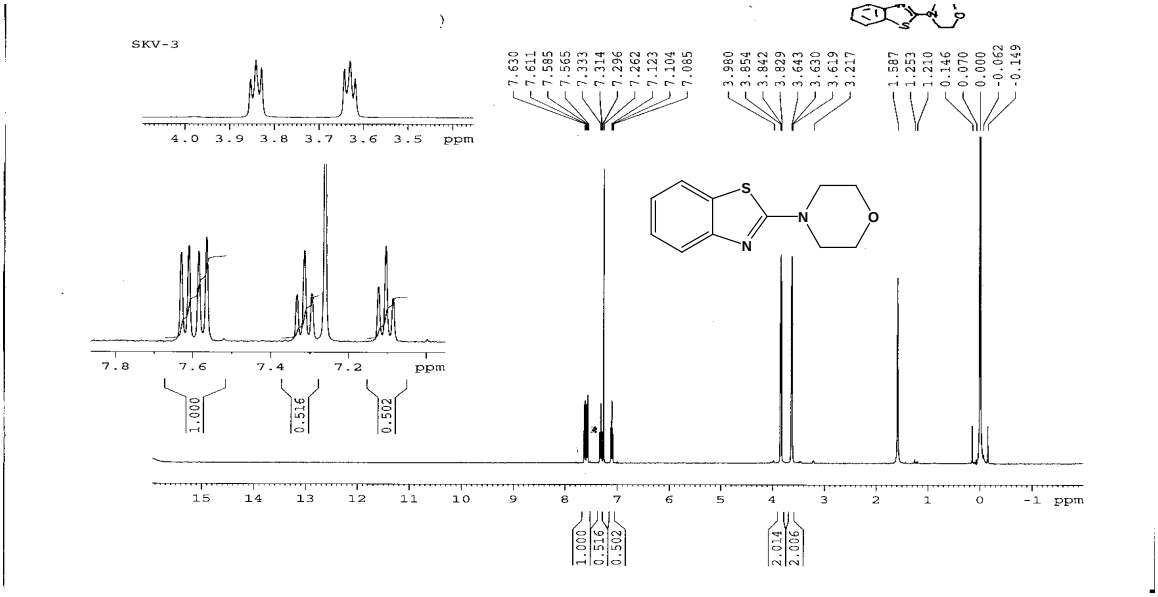

Figure 14: ^{1}H NMR of 2-Morpholin-4-yl-benzothiazole ($CDCl_3$)

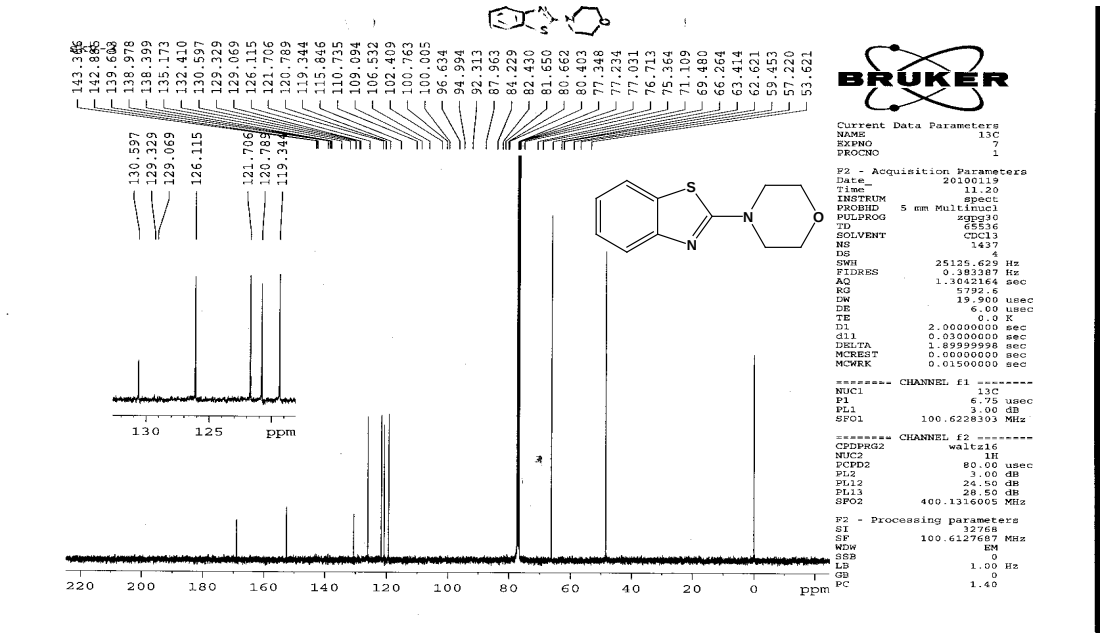

Figure 15: ^{13}CNMR of 2-Morpholin-4-yl-benzothiazole ($CDCl_3$)

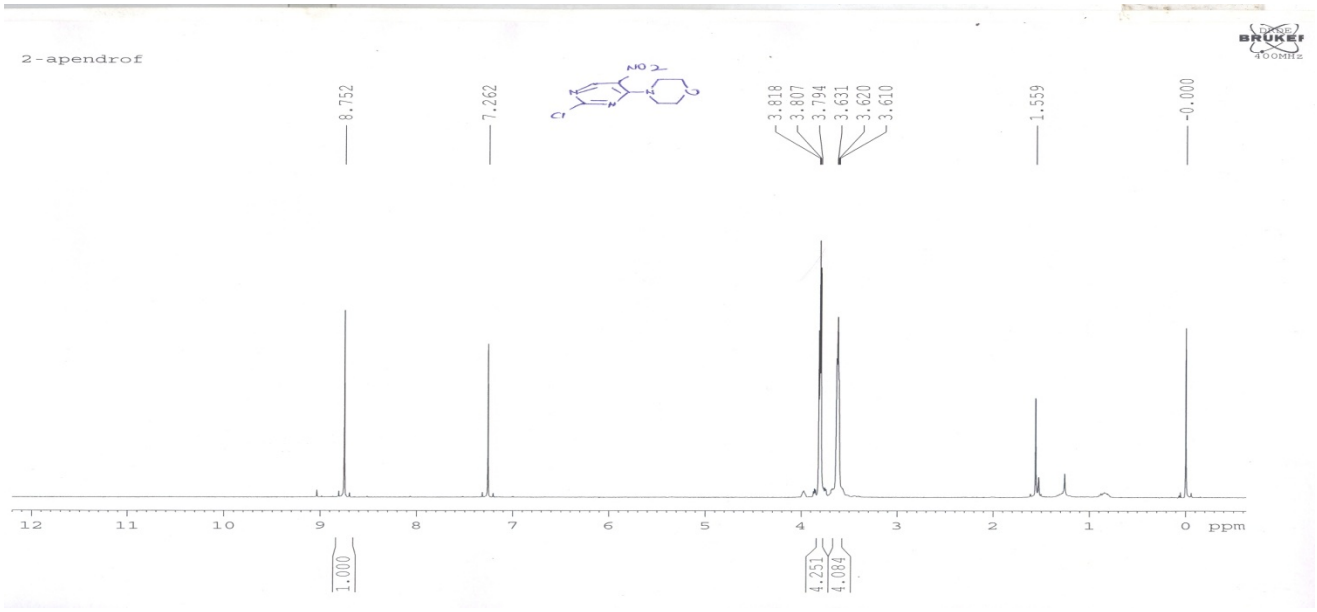

Figure 15: ^{1}H NMR of 4-(2-chloro-5-nitropyrimidin-4-yl)morpholine ($CDCl_3$)

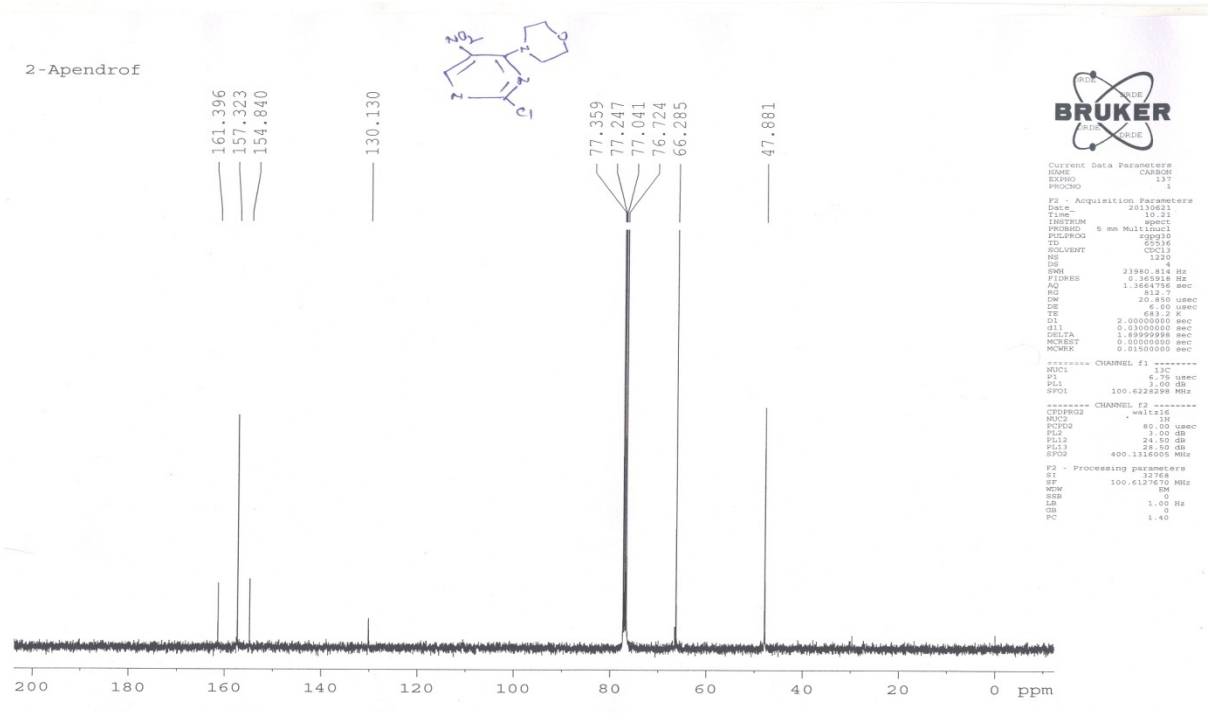

Figure 15: ^{13}CNMR of 4-(2-chloro-5-nitropyrimidin-4-yl)morpholine ($CDCl_3$)