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Research Article

Synthesis and Characterization of2-[1H- benzimi - dazole-2yl-sulfanyl]-N-[(E) - (3-methylphenyl) methylidene] acetohydrazide

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Abstract: Many important biochemical compounds and drugs of natural origin contain heterocyclic ring structures. Among these e.g. Carbohydrates, essential amino acids, vitamins, alkaloids, glycosides etc. the presence of heterocyclic structures in such diverse types of compounds is strongly indicative of the diverse types of the pharmacological activity. Diversity of biological response profile has attracted considerable interest of several researchers across the globe to explore this skeleton for its assorted therapeutic significance. By using different synthetic methods new benzimidazole derivatives were synthesized and further Melting points were determined by using Precision melting point apparatus in open capillaries and are uncorrected. The purity of the compounds was checked by TLC on silica gel G plates using n-Hexane, ethyl acetate (1:3) and methanol: chloroform (1:9) solvent system. The synthesized benzimidazole derivatives were characterized by IR spectral analysis. Benzimidazole is a lead nucleus for future developments to get effective compounds.

Keywords: Benzimidazole, Imidazole, n-Hexane, Ethyl acetate, Chloroform, Methanol

INTRODUCTION

Pharmaceutical chemistry is a science that makes use of general laws of chemistry to study drugs, i.e. their preparation, chemical nature, composition, structure, influence on an organism and studies of the physical and chemical properties of drugs, the methods of quality control and the conditions of their

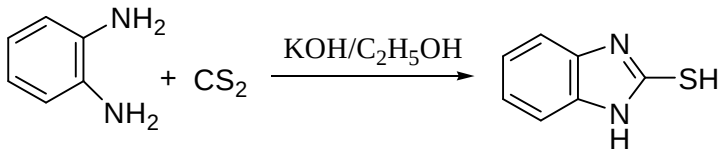
storage. Heterocyclic chemistry is the branch of chemistry dealing with the synthesis, properties, and applications of heterocyclics.

Heterocyclic chemistry comprises at least half of all organic chemistry research worldwide. In particular, heterocyclic structures form the basis of many pharmaceutical, agrochemical and veterinary products. The benzimidazole contains a phenyl ring fused to an imidazole ring, as indicated in the structure of benzimidazole.The important group of substances has found practical application in a number of fields.

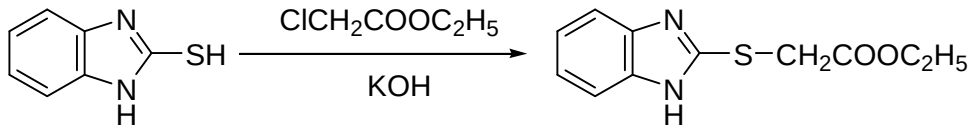
Recently in benzimidazole chemistry has been revived somewhat by the discovery that the 5, 6-dimethyl benzimidazole moiety is a part of the chemical structure of vitamin B12.

EXPERIMENTAL

Synthesis of 2-mercapto benzimidazole: A mixture of 10.8gm (0.1mole) of o-phenylenediamine, 5.65 gm (0.1mole) of potassium hydroxide and 7.67 gm (0.1mole, 6.19ml) of carbon disulfide, 100ml of 95% ethanol and 15 ml of water was taken in a 500ml round bottom flask heated under reflux for three hours. Then 1-1.5 gm of charcoal was added cautiously and the mixture is further heated at the reflux for 10 minutes, the charcoal is removed by filtration. The filtrate is heated to 60-70^{o}C, 100ml of warm water is added, and acidified with dilute acetic acid with good stirring. The product separated as glistening white crystals, and the mixture is placed in a refrigerator for three hours to complete the crystallization. The product is collected on a Buckner funnel and dried over night at 40^{o}c. The dried product is recrystallised by ethanol the yield is 8.5gm (73%) melting point is 300-305^{o}C.

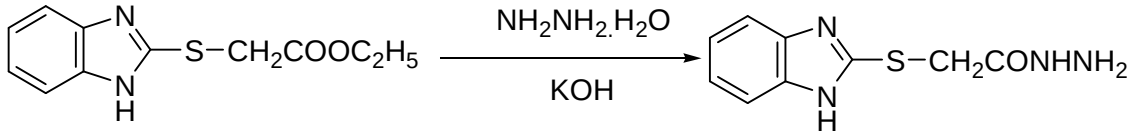


Synthesis of ethyl (1H-benzimidazol-2-yl-sulfanyl) acetate: A stirred mixture containing 4.5gm of (0.03mole) of 2-mercaptobenzimidazole, 60ml of ethanol and 1.68gm of (0.03mole) potassium hydroxide was added and heated at 78-80^{o}C for 10-minutes. Then ethyl chloro acetate (3.66ml, 0.03mol) was added in one portion, an exothermic reaction set in causing a temperature rise from 30-40^{o}C. After stirring at 25-30^{0}c for 18-hours, the reaction mixture was added to 100gm of ice-water and stirred for 30-minutes at 0-10^{0}C. The precipitate was collected by filtration washed with water until free of chloride and air dried at 50^{0}c and recrystallised by water the yield is 6 gm (62.25%). melting point is 105^{0}C.



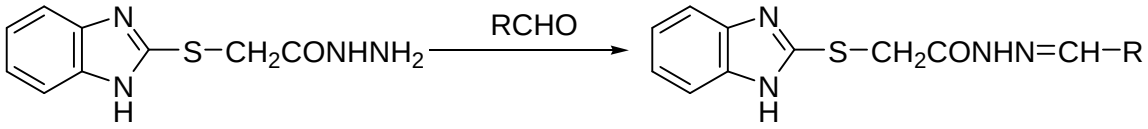
1*H*-benzo[*d*]imidazole-2-thiol **Compound-1**

Synthesis of 2-(1H-benzimidazol-2-ylsulfanyl)acetohydrazide : The mixture of 2-carboxy ethyl thio 1H-benzimidazole 4gm (0.004mole) and Hydrazine hydrate 6ml (0.01mole) are mixed well in a RBF and heated on water bath for 10 min. then dissolved in 60 ml ethanol, the reaction mixture is heated with reflux the reaction mixture is heated with reflux condenser for six hours, cooled to room temperature and the reaction mixture was added to 100gm of ice-water, and kept aside for the crystallization. The colorless crystals are collected by filtration, and recrystallised from water. Melting point is 180-185^{0}C; the yield is 60-70%.



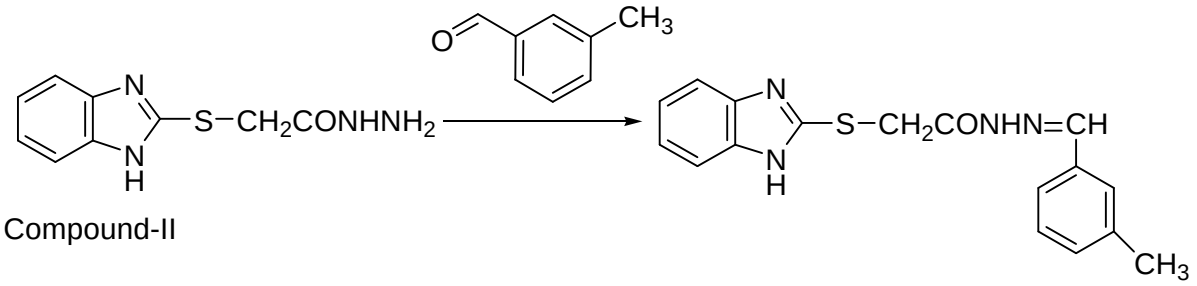
Compound-I Compound-II

General procedure for the preparation of Schiff bases Compound:



Compound-II

A equimolar solution of Carboxy Hydrazide(0.009 mol, 2gm) is dissolved in 10ml of ethanol and to this solution substituted aldehydes in equi molar qty (0.009mol, 0.917) is added with 4-6 drops of glacial acetic acid was added, this reaction mixture is kept under reflux for 8 hours. After cooling to room temperature was added to ice cold water. Compound gets separated as solid filtered, dried and recrystalised with chloroform.



Compound-II

RESULTS AND DISCUSSION

The present study explains the synthesis and characterization of benzimidazol derivative i.e., 2-[1H-benzimidazole-2yl-sulfanyl]-N-[(E)-(3- methyl phenyl) methylidene]aceto hydrazide. At present studies find the structural-activity relationship (SAR) and to optimize the structure. The synthesized benzimidazol derivative characterized by IR spectral studies. The purity of the synthesized benzimidazol derivative was checked by (TLC) thin layer chromatography and R_f value was recorded.

Physico-chemical analysis of synthesized derivatives

Table.1

S.no	2-mercapto benzimidazole	
1.	Mol. Formula	$C_7H_6N_2S$
2.	Melting Point	300-305^oC
3.	% Yield	73%
4.	Solvent system used	hexane: ethyl acetate (1:3)

Table.2

S.no	Ethyl (1H-benzimidazol-2-yl-sulfanyl) acetate	
1.	Mol. Formula	$C_{11}H_{12}O_2N_2S$
2.	Melting Point	105°C
3.	% Yield	62.25 %
4.	Solvent system used	hexane: ethyl acetate (1:3)

Table.3

S.no	2-(1H-benzimidazol-2-yl-sulfanyl)acetohydrazide	
1.	Mol. Formula	$C_8H_{11}ON_4S$
2.	Melting Point	180-185°C
3.	% Yield	60-70%
4.	Solvent system used	hexane: ethyl acetate (1:3)

Table.4

S.no	2-[1H-benzimidazole-2yl-sulfanyl]-N-[(E)-(3- methyl phenyl) methylidene] acetohydrazide	
1.	Mol. Formula	$C_{17}H_{16}N_4OS$
2.	Melting Point	245-250°C
3.	% Yield	60%
4.	Mol. Weight	324

IR spectral data Table. 5

Functional group assigned	Group frequency in Wave number (cm-1)
(-NH-)	3340,1330
(>C=O)	1728
(-C=N-)	1612
C-S-C	730

^{1}HNMR spectral data Table.6

S.no	Value (δ)	Nature of segment	Type
1	11.38	Singlet	1H, NH Ar
2	9.53	Singlet	1H, NH-N
3	8.0	Singlet	1H,N=CH
4	7.01-7.97	Multiplet	8H, Ar-H
5	4.48	Singlet	2H, S-CH2
6	2.38	Singlet	CH3

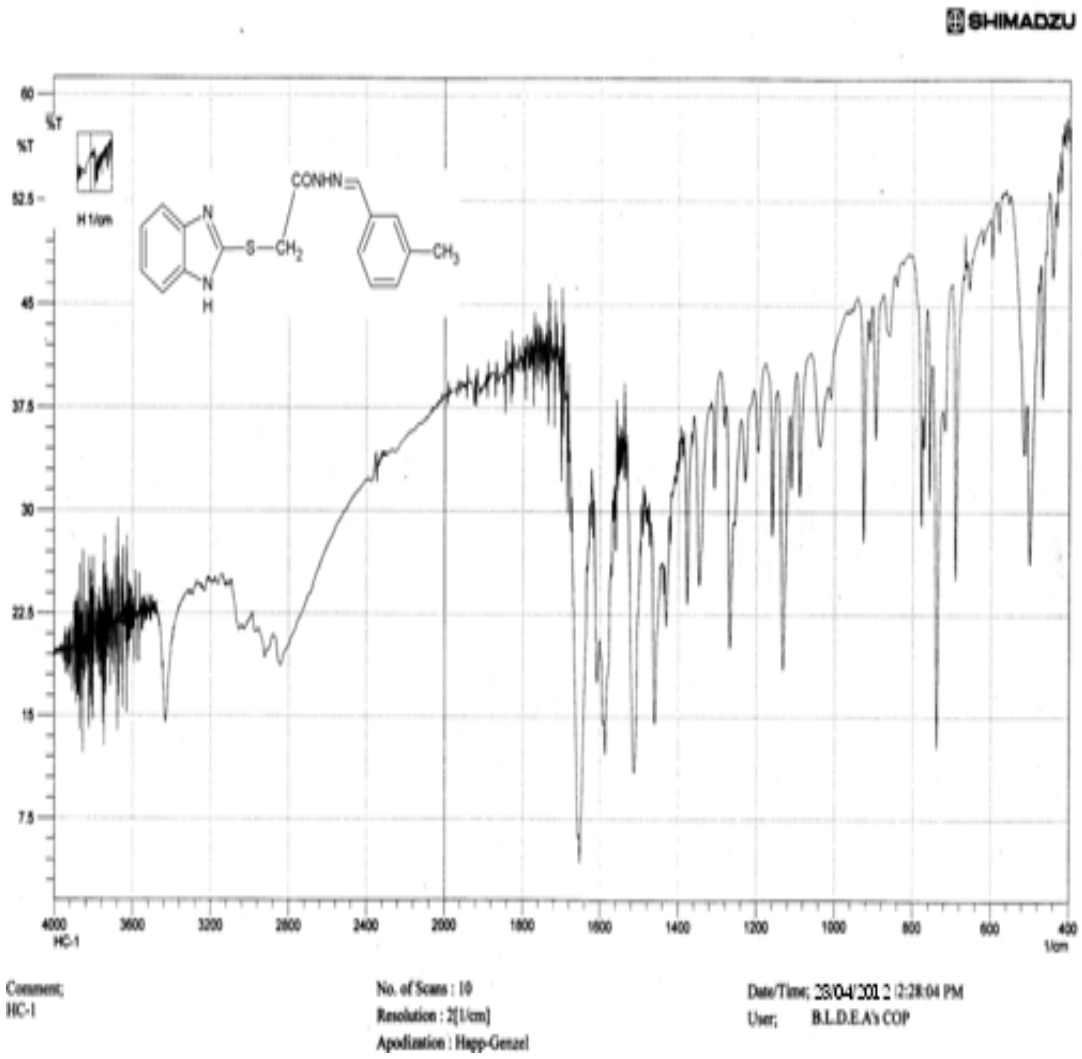


Fig. 1 IR Spectra of 2-[1H- benzimidazole- 2yl-sulfanyl]-N-[(E)-(3- methyl phenyl) methylidene] acetohydrazide

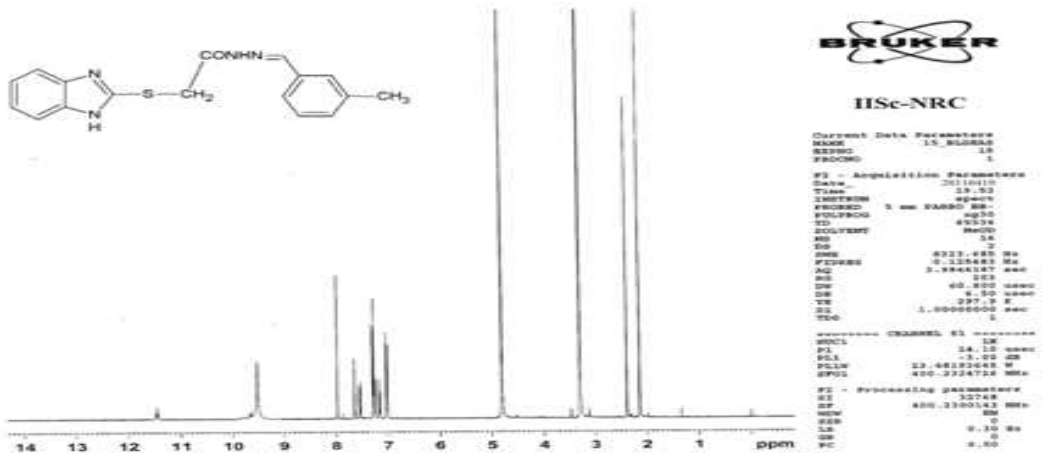


Fig. 2 ^{1}HNMR Spectra of 2-[1H- benzimidazole- 2yl-sulfanyl]-N-[(E)-(3- methyl phenyl)

methylidene] acetohydrazide

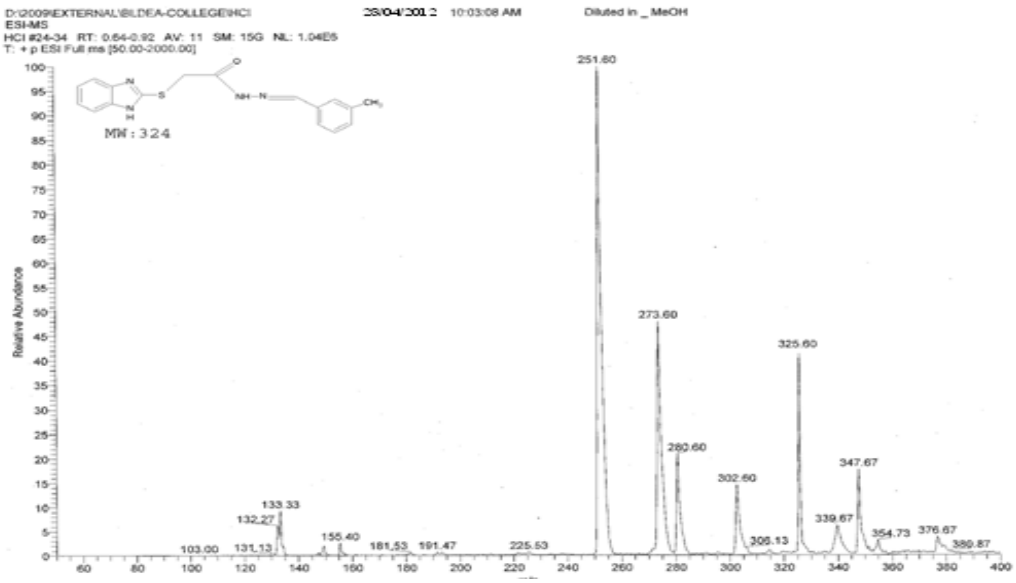


Fig. 3 MASS Spectra of 2-[1H- benzimidazole- 2yl-sulfanyl]-N-[(E)-(3- methyl phenyl) methylidene] acetohydrazide

CONCLUSION

By this study concluded that to find the structure-activity relationship (SAR) and to optimize the structure of the synthesized new benzimidazol derivative i.e., 2-[1H- benzimidazole- 2yl-sulfanyl]-N-[(E)-(3- methyl phenyl) methylidene] aceto hydrazide. The compound was characterized by IR spectral studies, the purity of the compound was checked by TLC and it produces good yield. The compound was confirmed by physicochemical and spectral analysis.

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