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SYNTHESIS AND CHARACTERIZATION OF 2-[1H- BENZIMIDAZOLE-2YL-SULFANYL]-N-[(E)-(4-METHOXY PHENYL) METHYLIDENE] ACETO HYDRAZIDE

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Abstract

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. 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 novel 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, ^{1}H NMR spectral analysis. Benzimidazole is a lead nucleus for future developments to get effective compounds.

INTRODUCTION

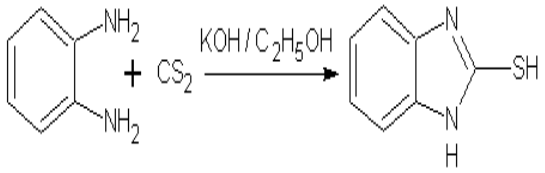
Substituted benzimidazole have received considerable attention during last few decades as they are endowed with variety of biological activities and have wide range of therapeutic properties. A literature survey indicates that benzimidazole derivatives possess different pharmacological and biological activities, of which the most potent is, anti-microbial activity, anti-ulcer. We thought to synthesize some novel benzimidazole moiety incorporating with different aromatic and hetero cyclic aldehyde moiety. The conventional methodology was adopted to synthesize the titled compounds.[1, 2].

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 [3, 4].

MATERIALS AND METHODS

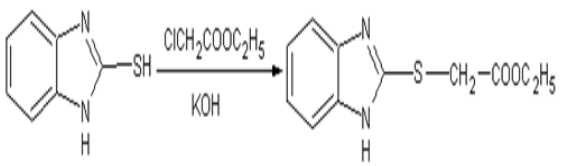
1. Synthesis of 2-mercapto benzimidazole

A mixture of 10.8gm (0.1mol) of o-phenylenediamine, 5.65 gm (0.1mol) 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[5, 6].



2. Synthesis of ethyl (1H-benzimidazol-2-yl-sulfanyl) acetate

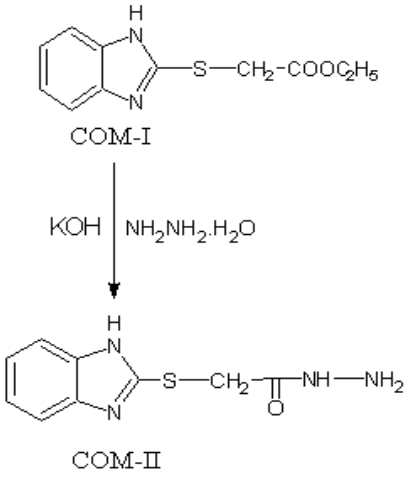
A stirred mixture containing 4.5gm of (0.03mol) of 2-mercaptobenzimidazole, 60ml of ethanol and 1.68gm of (0.03mol) potassium hydroxide was added and heated at 78-80^{o}C for 10-minutes. Then ethyl chloro acetate (3.66ml, 0.03mol) was added in one portion, an exothermic reaction set in causing a temperature 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 [6,7].



2-mercaptobenzimidazole COM-I

3. Synthesis of 2-(1H-benzimidazol-2-ylsulfanyl) acetohydrazide

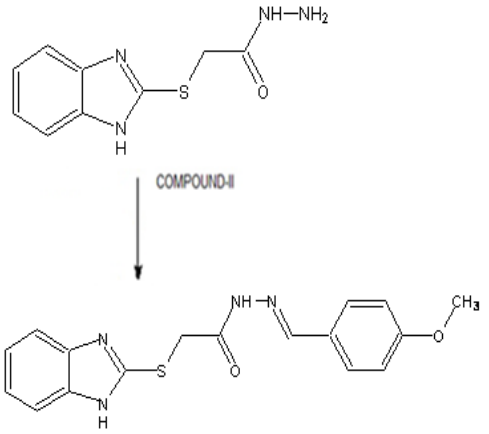
The mixture of 2-carboxy ethyl thio 1H-benzimidazole 4gm (0.004mole) and hydrazine hydrate 6ml (0.01mole) are mixed well in a RBF and heated on water bath for 10 min. then dissolved in 60 ml ethanol, the reaction mixture is heated with reflux the reaction mixture is heated with reflux condenser for six 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 recrystallized from water. Melting point is 180-185^{0}C; the yield is 60-70%[7, 8].

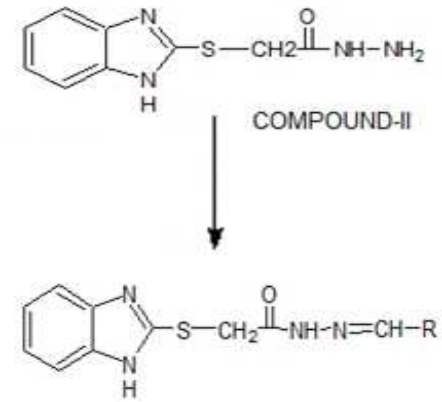


4. General procedure for the preparation of Schiff bases compound

A equimolar solution of carboxyl hydrazide (0.009 mol, 2gm) is dissolved in 10ml of

ethanol and to this solution substituted aldehydes in equimolar 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 cold water. Compound gets separated as solid filtered, dried and recrystalized with chloroform [9, 10].





RESULTS AND DISCUSSION

The present study explains the synthesis and characterization of benzimidazol derivative i.e., 2-[1H- benzimidazole-2yl-sulfanyl] N-[(E)-(4-methoxy phenyl) methylidene] acetohydrazide. At present studies find the structural-activity relationship (SAR) and to optimize the structure. The synthesized benzimidazol derivative characterized by IR, ^{1}H NMR spectral data analysis. The purity of the synthesized benzimidazol derivative was checked by (TLC) thin layer chromatography and R_f value was recorded.

CONCLUSION

By this study concluded that to find the structure-activity relationship (SAR) and to optimize the structure of the synthesized novel benzimidazol derivative i.e., 2-[1H-benzimidazole-2yl-sulfanyl]-N-[(E)-(4-methoxy phenyl) methylidene] acetohydrazide.

The compound was characterized by IR spectral data, the purity of the compound was checked by TLC and it produces good yield. The compound was confirmed by physicochemical and spectral data analysis.

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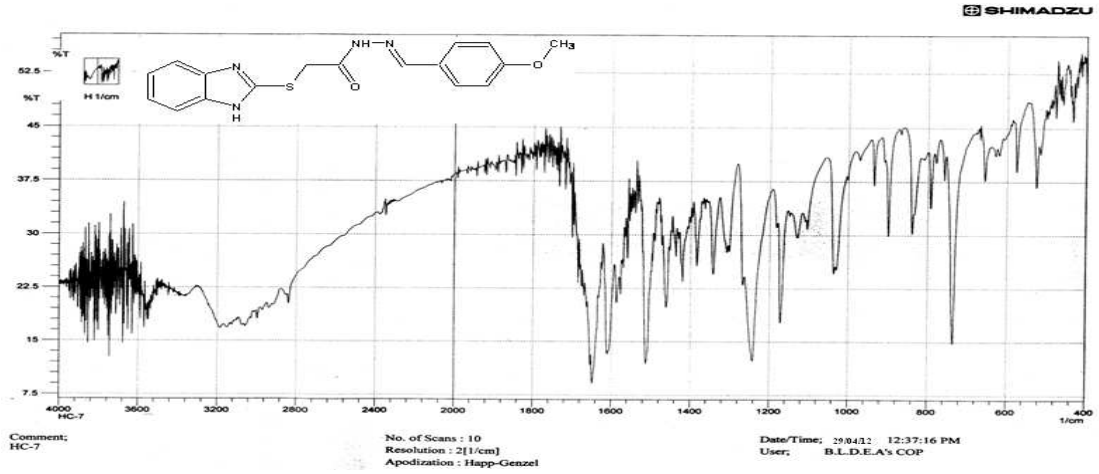

Figure 1 IR Spectra of 2-[1H-benzimidazole-2yl-sulfanyl]-N-[(E)-(4-methoxy phenyl) methylidene] acetohydrazide

Table 1

Physicochemical analysis

Sr. 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

Physicochemical analysis

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

Table 3

Physicochemical analysis

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

Table 4

Physicochemical analysis

Sr. No	2-[1H-benzimidazole-2yl-sulfanyl]-N-[(E)-(4-methoxy phenyl) methylidene] acetohydrazide	
1.	Mol. Formula	$C_{17}H_{16}N_4O_2S$
2.	Melting Point	228-235^{o}C
3.	% Yield	65.5%
4.	Mol. Weight	340

Table 5

IR spectral data

Functional group assigned	Group frequency in Wave number (cm^{-1})
(-NH-)	3334,1328
(>C=O)	1675
(-C=N-)	1617

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