

Synthesis and Delineation of Substituted Hydroxy benzylidene Compounds

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Abstract: Synthesis and delineation of three novel substituted hydroxy benzylidene compounds (E)-1-((2-hydroxy-5-nitrophenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)urea, (E)-1-((2,4-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)thiourea, (E)-1-((2,5-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)thiourea have been synthesized by condensing substituted urea, thiourea compound with substituted aromatic aldehydes and ethanol in one pot. Spectral data have characterized the newly synthesized compounds.

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I. INTRODUCTION

Derivatives of urea and thiourea play a vital role in the field of medicinal chemistry by regulating various pharmacological activities [1]. Thiourea is considered a crucial compound that produces industrial chemical products and is utilized in the synthesis of heterocyclic compounds [2]. Literature survey reveals that urea and thiourea derivatives exhibit a broad spectrum of biological activities, including anti-HIV, antiviral, antibacterial, and analgesic properties [3-6].

Basic features of Salicylaldehyde

Salicylaldehyde, 2-hydroxybenzaldehyde, (C₇H₆O₂), is made by heating sodium phenolate and chloroform with NaOH. It is a clear, colorless, oily liquid. It has a bitter almond like odor and has burning taste. Its boiling point is (196-197) °C, melting point is -7 °C and density is 1.165-1.172 gcm⁻³. It is slightly soluble in water, Soluble in alcohol and ether, gives orange color with sulphuric acid.

Basic features of Salicylaldehyde

Salicylaldehyde is only slightly soluble in water but dissolves easily in alcohol and ether. When mixed with sulfuric acid, it turns orange.

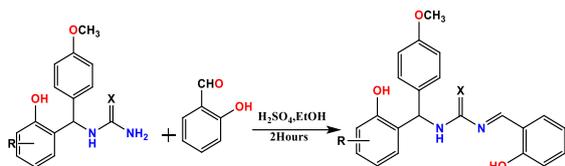
II. MATERIAL AND METHOD

All the reagents and solvents used were laboratory grade and were used without further purification. FT-IR-Alpha Bruker IR spectrometers were used to obtain IR spectra from KBr pellets at 4000 cm⁻¹ to 400 cm⁻¹ for the IR spectra.

Methodology:

A mixture of substituted phenol and substituted benzaldehyde in 10 mL of ethanol was refluxed with a few drops of concentrated H₂SO₄ for 2 hours. The mixture was then cooled and transferred to crushed ice. The solid that formed was filtered under suction and recrystallized from ethanol into a brick-red crystalline solid.

Synthesis pathway of the compound:



Physical data:

Compound code	Chemical formula	Molecular weight	Appearance	Melting point	Yield (%)
I	C ₂₂ H ₁₉ N ₃ O ₆	421.41	Brick-Red	185° C	72%
II	C ₂₂ H ₂₀ N ₂ O ₄ S	408.47	Brick-Red	190° C	78%
III	C ₂₂ H ₂₀ N ₂ O ₄ S	408.47	Brick-Red	210° C	80%

III. RESULT AND DISCUSSION

The synthesis of (E)-1-((2-hydroxy-5-nitrophenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)urea, (E)-1-((2,4-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)thiourea, (E)-1-((2,5-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene)thiourea was carried out in one step, by the condensation substituted phenol, aromatic aldehyde in presence of ethanol and con. H₂SO₄.

Compound code I(E)-1-((2-hydroxy-5-nitrophenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene) urea

IR (cm⁻¹): 3336 (N-H), 3592 (N-H), 3732 (O-H), 3195 (Aromatic C-H), 2751, and 2840 (Aliphatic C-H), 1217, 1258, and 1332 (C-N), 1590 (C=N), 1450-1600 (Aromatic C=C).

¹H NMR (DMSO) δ: 8.91 (s, OH), 6.27 (d, CH), 6.94 (d, CH), 6.24 (d, CH), 9.6 (s, OH), 5.19 (d, CH), 16.67 (d, CH), 7.21 (d, CH), 7.21 (d, CH), 6.89 (d, CH), 6.89 (d, CH), 3.81 (s, CH₃), 8.24 (d, CH), 7.65 (d, CH), 7.16 (multiplet), 7.32 (multiplet, CH), 6.93 (d, CH), 11.11 (s, OH)

¹³C NMR (DMSO) δ: 158.1 (C), 161.1 (C), 164 (C), 141 (C), 118.5 (C), 120.4 (C), 133.6 (C), 144.8 (CH), 126 (CH), 129.2 (CH), 116.1 (CH), 114.8 (CH), 117.8 (CH), 126.6 (CH), 129.2 (CH), 132.1 (CH), 132.4 (CH), 121.4 (CH), 164.8 (C), 55.8 (Aliphatic, CH₃), 169.7 (Aliphatic, CH), 51.9 (Aliphatic, CH)

m/z: 421.13

Elemental Analysis: C, 62.70; H, 4.54; N, 9.97; O, 22.78

Compound code II(E)-1-((2,4-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene) thiourea

IR (cm⁻¹): 3394 (N-H), 1658 (C=O), 2838 (CHO), 1510 (NO₂), 1383 (NO₂), 1450-1600 (C=C Aromatic), 2940 (Aromatic C-H)

¹H NMR (DMSO) δ: 8.91 (s, OH), 6.27 (d, CH), 6.94 (d, CH), 6.24 (s, CH), 9.68 (s, OH), 5.19 (CH), 7.21 (2d, CH), 6.89 (2d, CH), 3.81 (s, CH₃), 16.67 (s, NH), 8.24 (s, CH), 11.11 (s, OH), 6.93 (d, CH), 7.32 (multiplet), 7.16 (multiplet), 7.65 (d, CH)

¹³C NMR (DMSO) δ: 158.1 (C), 161.1 (C), 156.7 (C), 157.4 (C), 118.5 (C), 112.1 (C), 133.6 (C), 104.1 (CH), 114.8 (CH), 129.2 (CH), 114.8 (CH), 109 (CH), 117 (CH), 131 (CH), 129.2 (CH), 132.1 (CH), 132.4 (CH), 121.4 (CH), 189.8 (C), 55.8 (CH₃), 163.7 (CH), 58 (Aliphatic, CH)

m/z: 408.11

Elemental Analysis: C, 64.69; H, 4.94; N, 6.86; O, 15.67; S, 7.85

Compound code III(E)-1-((2,5-dihydroxyphenyl)(4-methoxyphenyl)methyl)-3-(2-hydroxybenzylidene) thiourea

IR (cm⁻¹): 3754 (O-H), 3350 (N-H), 1644 (C=O), 2786 (C-H), 755 (C=S), 3007 (Aromatic C-H), 1450-1600 (Aromatic C=C)

¹H NMR (DMSO) δ: 9.29 (s, OH), 6.91 (d, CH), 6.67 (d, CH), 6.65 (d, CH), 9.68 (s, OH), 5.19 (CH), 16.67 (d, NH), 7.21 (2d, CH), 6.89 (2d, CH), 3.81 (s, CH₃), 8.24 (s, CH), 11.11 (s, OH), 6.93 (d, CH), 7.32 (multiplet, CH), 7.16 (multiplet), 7.65 (d, CH)

^{13}C NMR (DMSO) δ : 150.1 (C-OH), 117.8 (CH), 114.8 (CH), 120.9 (CH), 115.3 (CH), 150.3 (C-OH), 58.3 (C), 113.6 (C), 129.2 (2CH), 114.8 (2CH), 158.1 (C-O), 55.8 (CH₃), 189.8 (C=S), 163.7 (C=N), 118.5 (C), 132.1 (CH), 121.4 (CH), 132.4 (CH), 117.8 (CH), 161.1 (C-OH)

m/z: 408.11

Elemental Analysis: C, 64.69; H, 4.94; N, 6.86; O, 15.67; S, 7.85

IV. CONCLUSION

We synthesized novel substituted hydroxy benzylidene compound derivatives in this study, and these compounds were investigated using FT-IR, NMR, and mass spectrometry, which shows good results.

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