

Synthesis and antimicrobial studies of Quinoline-8-thioglycolic acid, Quinaldine-8-thioglycolic acid and their derivatives

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Abstract

Quinoline-8-thioglycolic acid, quinaldine-8-thioglycolic acid and their derivatives have been synthesized. All the newly prepared compounds were evaluated for their antibacterial activity against five bacterial strains viz. *Staphylococcus aureus*, *Staphylococcus albus*, *Ps. pyocyanea*, *Proteus* and *Escherichia coli*. Some transition metal chelates of Quinoline-8-thioglycolic acid and Quinaldine-8-thioglycolic acid were found to be active against pathogenic bacteria.

Keywords: Complexes, IR spectra, antibacterial activity.

Introduction

Quinolines are heterocyclic aromatic compounds containing nitrogen with the molecular formula C_9H_7N . Quinoline or benzo-pyridine or 1-aza-naphthalene is a weak tertiary base that tends to form salt with acid and undergoes electrophilic substitution reactions and other chemical reactions resembling benzene and pyridine. Compounds possessing quinoline pharmacophore are of biological importance^{2,5,7,11} and have marked antiviral, antibacterial and antiparasitic properties^{12,14,20,35,36}. Many quinoline derivatives exhibit a wide spectrum of antitumor, antiplatelet, anticancer, antineoplastic, antiplasmodial, analgesic, hypnotic, antihistamine, antimalarial, antiprotozoal, analgesics, anti-cholesterol and antihypertensive cytotoxic properties^{4,6,8,10,13,16-19,22-30,32}.

The process of cell division and transcription is the synthetic purpose of DNA in which two important enzymes viz. DNA-gyrase and Topo-isomerase-IV are involved which re-fashion the super-coiling of chromosome by reforming the DNA topography with the aim to protect its genomic integrity. The mechanism of quinolines involves the interruption by targeting the reticence of the pursuit of the enzymes DNA-gyrase and topo-isomerase IV by bending in a reverse way leading to enhancement of the cleavage process of complexes at the enzyme-DNA involving cleavage-ligation site³¹.

Kouznetsov et al¹⁵ had prepared new ligands by treatment of some aminoquinolines with anti-chloroglyoxime. These ligands were characterized by elemental analysis, ¹H-NMR and FTIR techniques. They were also evaluated for their antibacterial properties against *Salmonella enteridis*,

Klebsiella pneumonia, *Escherichia coli*, *Enterococcus faecalis* and *Pseudomonas aeruginosa* by disc diffusion technique and were found to be effective.

Several transition metal chelates of Zn(II), Mn(II), Cu(II), Co(II), Ni(II) and Fe(III) have been prepared with ligand containing 8-Hydroxyquinolines³⁴. The prepared metal chelates were characterized by elemental analysis, IR, UV-Vis spectral studies and magnetic measurements. Tetrahedral geometry around Zn(II), octahedral geometry around Ni(II), Co(II), Mn(II) and Fe(III) and distorted octahedral geometry around Cu(II) have been proposed. The antimicrobial activities for the prepared ligand and its metal chelates were conducted against various bacteria and were found to be effective.

Thioglycolic acids and their derivatives have found applications^{1,3,9,24,33,37} in various fields like complexing agent, polymer formation, wool protein extraction, plasticizers, hair waving lotion, insecticides, drugs, dyes etc. No thioglycolic acid of quinoline moiety has been reported till now. Quinoline derivatives are found to possess broad-spectrum antimicrobial activities. In view of this, we have decided to synthesize quinoline-8-thioglycolic acid, quinaldine-8-thioglycolic acid, their ester, amide and hydrazide derivatives.

The chelating abilities of quinoline-8-thioglycolic acid and quinaldine-8-thioglycolic acid have also been studied by preparing their metal chelates. All the newly prepared thioglycolic acids and their derivatives were subjected to biological screening using paper disc diffusion technique²¹.

Material and Methods

For the synthesis of compounds, the chemicals and reagents used were of A.R. grade. The melting points of the compounds was determined in capillary tubes with the help of T-1150 m.p. apparatus. CHN analyzer B6450 model was used for carbon, nitrogen and hydrogen elemental analyses. The IR spectra were recorded with the aid of Shimadzu FTIR 8201 spectrophotometer in the region 4000-400 cm^{-1} .

Synthesis of Quinoline-8-thioglycolic acid: Quinoline-8-sulphochloride was prepared and further it was converted into tin salt of 8-mercaptoquinoline by the usual method. 5 g of the tin salt of 8-mercaptoquinoline was decomposed with 5 g sodium hydroxide and 9 g sodium tartarate. The aqueous solution was filtered to obtain sodium salt of 8-

mercaptoquinoline which was then treated with 1.5 g monochloroacetic acid solution previously neutralized. The reaction mixture was warmed and stirred for three hours. The reaction mixture was filtered and the filtrate was concentrated to one-fourth of its initial volume. It was then acidified with conc. HCl but quinoline-8-thioglycolic acid could not be isolated. This may be due to the formation of zwitter ion.

To overcome this difficulty, the above reaction mixture after acidification was treated with saturated solution of CuSO_4 till the reaction is complete and the blue colour of CuSO_4 stops changing. The reaction mixture was left overnight. Green precipitate of copper chelate was obtained which was filtered, washed and dried (yield: 70%).

5 g of the copper chelate was dissolved in 100 mL (2N) HCl. The green colour solution was warmed and H_2S was passed through it till complete precipitation of copper sulphide as black precipitate is achieved. The clear yellowish filtrate was then evaporated to dryness in a basin on water bath with continuous scratching with a glass rod. Yellow crystals of quinoline-8-thioglycolic acid hydrochloride separated out which were recrystallized from methanol.

Molecular formula: $\text{C}_{11}\text{H}_{10}\text{O}_2\text{NSCl}$; Yield: 40%; m.p.: 180 °C; Found (%): C, 51.50; H, 3.34; N, 5.30; Calculated (%): C, 51.66; H, 3.91; N, 5.47; IR (KBr, cm^{-1}) ν_{max} : 1690 cm^{-1} (C=O), 1600 cm^{-1} (C=N), 2700-2250 cm^{-1} (—OH associated).

Synthesis of Quinoline-8-thioglycolic ethyl ester: 1 g of quinoline-8-thioglycolic acid hydrochloride was dissolved in 10 mL dry ethanol and refluxed on a water bath for 12 hours. The excess of alcohol was distilled out and rest of the content was cooled and poured into crushed ice and made alkaline with 10% aq. NaHCO_3 . Light yellow coloured precipitate of quinoline-8-thioglycolic ethyl ester separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $\text{C}_{13}\text{H}_{13}\text{O}_2\text{NS}$; Yield: 60%; m.p.: 63 °C; Found (%): C, 62.30; H, 5.30; N, 5.57; Calculated (%): C, 63.15; H, 5.26; N, 5.66; IR (KBr, cm^{-1}) ν_{max} : 1715 cm^{-1} (C=O).

Synthesis of Quinoline-8-thioglycolic acid amide: 5 g of quinoline-8-thioglycolic acid hydrochloride was refluxed with 30 mL dry ethanol for 10 hours and then 2 mL liq. ammonia was added slowly with constant stirring. The reaction mixture was refluxed for 4 hours. On cooling, white precipitate of quinoline-8-thioglycolic acid amide separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $\text{C}_{11}\text{H}_{10}\text{ON}_2\text{S}$; Yield: 70%; m.p.: 220 °C; Found (%): C, 60.00; H, 4.50; N, 12.40; Calculated (%): C, 60.55; H, 4.58; N, 12.84; IR (KBr, cm^{-1}) ν_{max} : 3350 cm^{-1} and 3150 cm^{-1} (—NH); 1680 cm^{-1} (C=O).

Synthesis of Quinoline-8-thioglycolic acid hydrazide: 5 g of quinoline-8-thioglycolic acid hydrochloride in 30 mL absolute alcohol was refluxed for 12 hours and then the volume of the reaction mixture was reduced to two-third of its initial volume by distilling off alcohol. 0.25 mL hydrazine hydrate in alcoholic medium was added drop wise with stirring and the reaction mixture was refluxed for 3 hours.

On cooling, white precipitate of quinoline-8-thioglycolic acid hydrazide separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $\text{C}_{11}\text{H}_{11}\text{ON}_3\text{S}$; Yield: 60%; m.p.: 170 °C; Found (%): C, 56.30; H, 4.62; N, 18.00; Calculated (%): C, 56.65; H, 4.72; N, 18.02; IR (KBr, cm^{-1}) ν_{max} : 3280 cm^{-1} (—NH); 1690 cm^{-1} (C=O).

Preparation of transition metal chelates of Quinoline-8-thioglycolic acid: The equimolar ratio of quinoline-8-thioglycolic acid hydrochloride and the metal acetate was mixed in absolute alcohol and refluxed for 4 to 8 hours. The precipitate separated out which was filtered, washed with absolute alcohol and dried. It was further recrystallized from absolute alcohol.

Synthesis of Quinaldine-8-thioglycolic acid: The tin salt of 8-mercaptoquinaldine was synthesized by the usual method. 5 g tin salt of 8-mercaptoquinaldine was mixed with 70 mL of 10% caustic soda solution. The change in colour and evolution of heat indicated decomposition. After complete decomposition, the reaction mixture was cooled and filtered.

It was treated with saturated solution of 1.25 g monochloroacetic acid previously neutralized. The reaction mixture was stirred and warmed. It was left overnight. Further, it was concentrated to one-fourth of its initial volume and acidified with conc. HCl. But, no precipitate was obtained due to zwitter ion formation. Hence, to overcome this difficulty, the concentrated reaction mixture was treated with saturated solution of CuSO_4 till the reaction is complete and the blue colour of CuSO_4 stops changing. Yellow precipitate separated out which was filtered, washed and dried (yield: 50%).

5 g of the copper chelate was dissolved in 50 mL (2N) HCl. The greenish coloured solution was warmed and H_2S was passed through it till the complete precipitation of copper sulphide. The clear filtrate was concentrated in a basin on water bath. On cooling, pale yellow needles of quinaldine-8-thioglycolic acid hydrochloride separated out which were filtered, washed with absolute alcohol and dried. It was further recrystallized from water.

Molecular formula: $\text{C}_{12}\text{H}_{12}\text{O}_2\text{NSCl}$; Yield: 60%; m.p.: 138 °C; Found (%): C, 46.98; H, 5.20; N, 4.30; Calculated (%): C, 47.13; H, 5.23; N, 4.58; IR (KBr, cm^{-1}) ν_{max} : 1712 cm^{-1} (C=O), 1630 cm^{-1} (C=N).

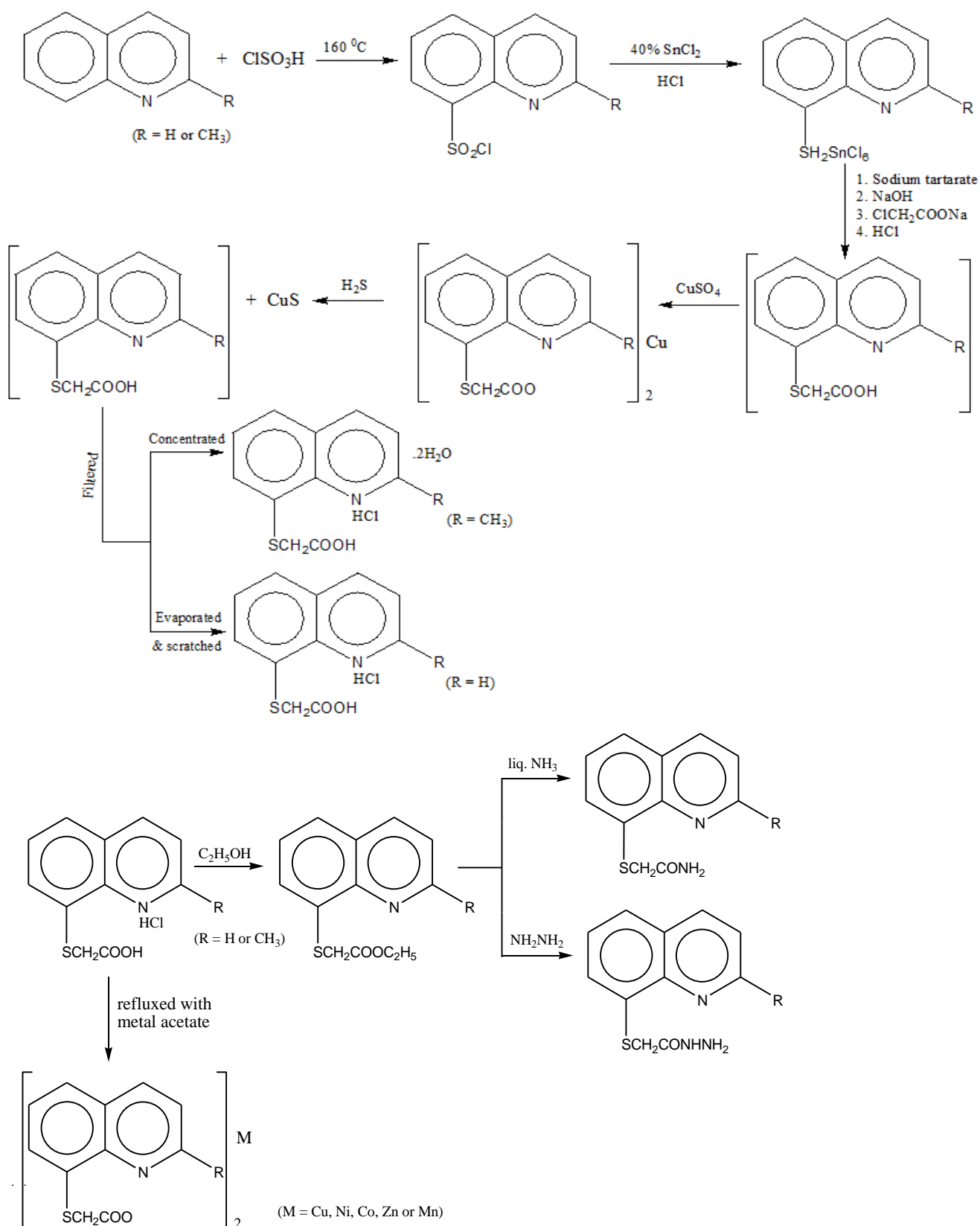


Figure 1: Scheme for synthesis of Quinoline-8-thioglycolic acid, Quinaldine-8-thioglycolic acid and their derivatives

Synthesis of Quinaldine-8-thioglycolic acid ethyl ester: 1 g of quinaldine-8-thioglycolic acid was dissolved in 10 mL absolute ethanol and refluxed on a water bath for 12 hours. The excess of alcohol was distilled out and rest of the content was cooled and poured into crushed ice and made alkaline

with 10% aq. NaHCO_3 . Light yellow coloured precipitate of quinaldine-8-thioglycolic acid ethyl ester separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $C_{14}H_{15}O_2NS$; Yield: 60%; m.p.: 43 °C; Found (%): C, 64.20; H, 5.42; N, 5.30; Calculated (%): C, 64.36; H, 5.74; N, 5.36; IR (KBr, cm^{-1}) ν_{max} : 1720 cm^{-1} (C=O).

Synthesis of Quinaldine-8-thioglycolic acid amide: 5 g of quinoline-8-thioglycolic acid was refluxed in 20 mL absolute ethanol for 12 hours and then 2 mL liq. ammonia was added dropwise with constant stirring. The reaction mixture was refluxed for 4 hours. On cooling, white precipitate of quinaldine-8-thioglycolic acid amide separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $C_{12}H_{12}ON_2S$; Yield: 50%; m.p.: 215 °C; Found (%): C, 61.90; H, 5.10; N, 11.90; Calculated (%): C, 62.06; H, 5.17; N, 12.06; IR (KBr, cm^{-1}) ν_{max} : 3360 cm^{-1} and 3180 cm^{-1} (–NH); 1640 cm^{-1} (C=O).

Synthesis of quinaldine-8-thioglycolic acid hydrazide: 2 g of quinaldine-8-thioglycolic acid was refluxed in 10 mL absolute alcohol for 12 hours and then 0.25 mL hydrazine hydrate was added dropwise and the reaction mixture was again refluxed for 4 hours. The excess of alcohol was distilled out. On cooling the reaction mixture, white precipitate of quinaldine-8-thioglycolic acid hydrazide separated out which was filtered, washed with ethanol and dried. It was further recrystallized from ethanol.

Molecular formula: $C_{12}H_{13}ON_3S$; Yield: 80%; m.p.: 185 °C; Found (%): C, 58.00; H, 5.10; N, 16.50; Calculated (%): C, 58.28; H, 5.25; N, 17.00; IR (KBr, cm^{-1}) ν_{max} : 3350 cm^{-1} and 3150 cm^{-1} (–NH); 1640 cm^{-1} (C=O).

Preparation of transition metal chelates of Quinaldine-8-thioglycolic acid: The equimolar ratio of quinaldine-8-thioglycolic acid and the metal acetate was mixed in absolute alcohol and refluxed for 4 to 8 hours. The coloured precipitate separated out which was filtered, washed with absolute alcohol and dried. It was further recrystallized from absolute alcohol.

Antimicrobial Studies: The antibacterial activity of the metal complexes of quinoline-8-thioglycolic acid and quinaldine-8-thioglycolic acid were tested against different test organism viz. *Staphylococcus aureus*, *Staphylococcus albus*, *Ps. pyocyanea*, *Proteus* and *Escherichia coli* by the simple paper disk diffusion method²¹. Sterilized culture medium plates were used and different concentrations of the complexes viz. 0.25 mg mL⁻¹, 0.5 mg mL⁻¹ and 1 mg mL⁻¹ in DMSO were prepared and tested. The plates were incubated for a period of 24 h at 37 °C. After the incubation period, the plates were observed for zones of inhibition and measured.

Results and Discussion

The synthetic scheme for the novel Quinoline-8-thioglycolic acid (Qn8TGA), quinaldine-8-thioglycolic acid (Qd8TGA) and their derivatives is discussed (fig. 1). For the preparation of quinoline-8-thioglycolic acid, tin salt of 8-mercaptoquinoline was decomposed with sodium hydroxide and sodium tartarate. It was further reacted with monochloroacetic acid and dil. HCl. Finally, it was treated with saturated copper sulphate solution. Yellow crystals of quinoline-8-thioglycolic acid were obtained in good yield. Further, different transition metal chelates of quinoline-8-thioglycolic acid were prepared.

Similar steps were adopted for the preparation of quinaldine-8-thioglycolic acid for which tin salt of 8-mercaptoquinoline was decomposed and further treated with 10% caustic soda, monochloroacetic acid and saturated copper sulphate solution to obtain pale yellow needles of quinaldine-8-thioglycolic acid. Different transition metal chelates of quinaldine-8-thioglycolic acid were prepared. The physical parameters of the novel metal chelates are shown in table 1 and table 2.

The study of antibacterial activity of thioglycolic acids, their ester amide and hydrazide derivatives showed that all of them were inactive against above mentioned test organisms. The synthesized metal chelates were also tested for their antibacterial activity by utilizing a wide range of bacteria viz. *Staphylococcus aureus*, *Staphylococcus albus*, *Ps. pyocyanea*, *Proteus* and *Escherichia coli*.

Table 1
Physical and Elemental data of Quinoline-8-thioglycolic acid

| Compound | Colour | Melting Point (°C) | Analysis (%) | | | | % Yield |
|---------------------------|--------------|--------------------|--------------------|----------------|----------------|------------------|---------|
| | | | Found (Calculated) | | | | |
| | | | C | H | N | M | |
| (Qn.8TGA) ₂ Cu | Green | 173 md | 52.51 (52.84) | 3.33 (3.02) | 5.71 (5.60) | 12.50 (12.71) | 72 |
| (Qn.8TGA) ₂ Co | Purple | 300 md | 53.72 (53.34) | 3.41 (3.23) | 5.81 (5.65) | 11.62 (11.90) | 78 |
| (Qn.8TGA) ₂ Zn | Light yellow | 274 md | 53.10 (52.65) | 3.01 (3.19) | 5.38 (5.58) | 12.86 (13.03) | 66 |
| (Qn.8TGA) ₂ Mn | Pale yellow | 310 md | 53.21 (53.77) | 3.31 (3.25) | 5.77 (5.70) | 10.92 (11.18) | 70 |

Table 2
Physical and Elemental data of Quinaldine-8-thioglycolic acid

| Physical and Elemental data of Quinidine 6-ethyleneglycol acid | | | | | | | |
|--|--------------|--------------------|---------------------------------------|----------------|----------------|------------------|---------|
| Compound | Colour | Melting Point (°C) | Analysis (%) Found (Calculated) | | | | % Yield |
| | | | C | H | N | M | |
| (Qd.8TGA) ₂ Cu | Green | 154 md | 47.42 (48.03) | 4.58 (4.67) | 4.53 (4.67) | 10.43 (10.59) | 76 |
| (Qd.8TGA) ₂ Ni | Bluish green | 305 d | 48.11 (48.42) | 4.67 (4.70) | 4.53 (4.70) | 9.60 (9.87) | 72 |
| (Qd.8TGA) ₂ Co | Sky blue | 250 d | 48.10 (48.40) | 4.57 (4.70) | 4.44 (4.56) | 9.72 (9.90) | 68 |
| (Qd.8TGA) ₂ Zn | Light yellow | 205 md | 47.59 (47.89) | 4.40 (4.65) | 4.47 (4.65) | 10.56 (10.87) | 78 |
| (Qd.8TGA) ₂ Mn | Light yellow | 305d | 48.50 (48.73) | 4.62 (4.73) | 4.59 (4.78) | 9.10 (9.29) | 74 |

Table 3

Antimicrobial studies of transition metal chelates of Quinoline-8-thioglycolic acid and Quinaldine-8-thioglycolic acid

| Compounds | <i>S. aureus</i> | <i>S. albus</i> | <i>Proteus</i> | <i>E. coli</i> | <i>Ps. pyocyanea</i> |
|---------------------------|------------------|-----------------|----------------|----------------|----------------------|
| (Qn.8TGA) ₂ Co | – | – | + | ++ | ++ |
| (Qn.8TGA) ₂ Zn | – | – | + | – | ++ |
| (Qn.8TGA) ₂ Mn | – | – | – | – | – |
| (Qn.8TGA) ₂ Cu | + | – | – | – | ++++ |
| (Qd.8TGA) ₂ Ni | – | – | – | – | ++ |
| (Qd.8TGA) ₂ Co | – | – | – | ++ | + |
| (Qd.8TGA) ₂ Zn | – | – | + | – | + |
| (Qd.8TGA) ₂ Mn | – | – | – | – | ++ |
| (Qd.8TGA) ₂ Cu | + | + | – | – | – |

– = no inhibition; + = zone size 6-8 mm; ++ = zone size 8-10 mm; +++ = zone size 10-12 mm; ++++ = zone size more than 12 mm

The results are shown in table 3. It is inferred that the compound (Qn8TGA)₂Cu exhibits the most potent activity against *Ps. pyocyanea*. Compounds (Qn8TGA)₂Co and (Qd8TGA)₂Co exhibit good activity against *E. coli* whereas compounds (Qn8TGA)₂Co, (Qn8TGA)₂Zn, (Qd8TGA)₂Ni and (Qd8TGA)₂Mn are fairly active against *Ps. pyocyanea*. Compounds (Qn8TGA)₂Co, (Qn8TGA)₂Zn and (Qd8TGA)₂Zn are active against *Proteus*. Only compound (Qd8TGA)₂Cu is active against *Staphylococcus albus* whereas compounds (Qn8TGA)₂Cu and (Qd8TGA)₂Cu are active against *Staphylococcus aureus*.

Conclusion

Quinoline-8-thioglycolic acid (Qn8TGA), quinaldine-8-thioglycolic acid (Qd8TGA) and their derivatives were synthesized. Further, transition metal chelates of quinoline-8-thioglycolic acid and quinaldine-8-thioglycolic acid were prepared and tested for their antibacterial activity by paper disc diffusion method²¹. Many of the prepared compounds were found efficient in their antibacterial activity among which compounds (Qn8TGA)₂Co, (Qn8TGA)₂Zn, (Qd8TGA)₂Ni, (Qd8TGA)₂Co and (Qd8TGA)₂Mn were active against different test organism.

Compound (Qn8TGA)₂Cu is found to be most potent against *Ps. pyocyanea*. Therefore, it may be inferred that transition metal chelates of quinoline-8-thioglycolic acid, quinaldine-8-thioglycolic acid their derivatives possess important pharmaceutical properties and applications.

Acknowledgement

The authors gratefully acknowledge BHU, Varanasi for spectral measurements.

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- (Received 17th September 2024, accepted 19th November 2024)