

Green Synthesis, Structural Characterization and Thermal Analysis of Divalent Transition Metal Complexes Derived from Halo-Substituted Chalcone and Pyrazoline Derivatives

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Abstract

Chalcones and their pyrazoline derivatives continue to attract interest as ligands in coordination chemistry, largely because the adjacent phenolic and carbonyl (or ring-nitrogen) donor sites let them chelate transition metal ions through simple, mild reactions. In the present work, a set of ortho-hydroxy chalcones and their brominated, iodinated, N-hydro-2-pyrazoline and N-acetyl-2-pyrazoline derivatives were converted into divalent Cu(II), Ni(II) and Zn(II) complexes using a room-temperature, solvent-minimized precipitation route rather than the reflux conditions more commonly reported for this class of compound. Twelve complexes (1:2 metal-to-ligand stoichiometry) were characterized by FT-IR and UV-Vis spectroscopy, magnetic susceptibility, and metal/halogen estimation, all of which point consistently to phenolate-oxygen and carbonyl- (or pyrazoline-nitrogen-) mediated chelation. Copper(II) complexes showed magnetic moments close to the spin-only value expected for a d9 system (1.79-1.84 BM), nickel (II) complexes were consistent with high-spin octahedral geometry (3.15-3.20 BM), and the zinc(II) complexes were diamagnetic, as expected for a d10 ion. Thermogravimetric and differential thermal analysis (TGA/DTA) up to 1000 °C showed the complexes to be thermally robust well above ambient conditions, decomposing through the organic ligand framework in one or more exothermic stages before settling into a stable high-temperature residue. Taken together, the results support a straightforward, energy-efficient route to a structurally diverse family of chalcone- and pyrazoline-based metal chelates, and point to several directions for more detailed residue and stoichiometric analysis as this line of work continues.

Keywords: Green synthesis; Chalcone-metal complexes; Pyrazoline ligands; Thermogravimetric analysis; Divalent transition metals

1. Introduction

Coordination chemistry built around biologically inspired organic ligands has grown into one of the more productive corners of inorganic and medicinal chemistry, in large part because a well-chosen ligand can tune a metal centre's stability, solubility and reactivity in ways the free metal ion cannot achieve on its own. Chalcones - open-chain 1,3-diaryl-2-propen-1-ones bearing an α,β -unsaturated carbonyl bridge - are a particularly convenient starting point for this kind of design work. They are accessible in one step from inexpensive aldehydes and acetophenones, and when the ketone ring carries a hydroxyl group ortho to the carbonyl, the resulting molecule already has the two adjacent oxygen donors a chelate need.

Two further modifications extend what these ligands can do. Halogenation of the enone system, whether by electrophilic iodination or NBS-mediated bromination, adds lipophilicity and an additional site for later chemistry, while cyclization of the chalcone with hydrazine or acetylated hydrazine closes the open chain into a 2-pyrazoline ring, introducing a ring nitrogen that can itself act as a donor atom alongside the retained phenolic oxygen. The result is a family of structurally related but electronically distinct ligands - unmodified chalcones, halo-chalcones, N-hydro-pyrazolines and N-acetyl-pyrazolines - that can all be complexed to the same set of metal ions under comparable conditions, making it possible to ask how each structural change on its own effects the resulting complex.

Most synthetic routes to this class of complex still rely on prolonged reflux in ethanol or methanol, which is effective but sits somewhat at odds with the broader push toward greener, lower-energy synthetic protocols consistent with the environmental themes of this conference. The present study set out to establish whether the same chemistry - chelation of Cu(II), Ni(II) and Zn(II) by these chalcone and pyrazoline ligands - could be carried out just as cleanly at room temperature, using a simple aqueous-ethanolic precipitation method with a mild base to neutralize the acid generated on complexation. The resulting complexes were then characterized structurally by FT-IR and UV-Vis spectroscopy, magnetic susceptibility and

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elemental estimation, and their thermal behaviour was examined by TGA/DTA, with the aim of building a coherent structure-property picture across the series.

2. Literature Review

The chelating behaviour of ortho-hydroxy carbonyl systems is well established in classical coordination chemistry: once the phenolic proton is removed, the resulting phenolate oxygen and an adjacent carbonyl or imine donor readily close a five- or six-membered ring around a divalent transition metal ion, in much the same way as the β -diketones and Schiff-base systems that form the backbone of much of twentieth-century ligand-field theory (Hyheey, 1983; Kauffman, 1966). Chalcones fit naturally into this picture, since the α,β -unsaturated ketone bridge leaves the carbonyl oxygen free to coordinate while extending conjugation across the whole molecule, and this conjugation is itself sensitive to whatever sits on the two aryl rings.

Two structural modifications recur across the literature on this ligand class. Halogenation of the chalcone - typically by electrophilic iodination or by N-Bromo succinimide-mediated bromination - is a standard way to raise lipophilicity and polarizability without disturbing the core donor set, and complexes of halogenated chalcones are generally reported to retain their C-X stretching vibrations largely unchanged on coordination, confirming the halogen sits outside the primary coordination sphere. Cyclization of the chalcone with hydrazine, giving a 2-pyrazoline ring, is the other common route: this converts an open-chain O,O-donor into a ring system that can present either an N-H or an N-acetyl nitrogen alongside the phenolic oxygen, and the loss of the N-H stretch on complexation is a widely used diagnostic for pyrazoline-nitrogen coordination (Nakamoto, 1997).

The electronic and magnetic fingerprints of Cu(II), Ni(II) and Zn(II) are well enough worked out by now that they serve less as a discovery in their own right and more as a diagnostic yardstick: an odd-electron d9 copper centre, a high-spin d8 nickel centre in an octahedral field, and a filled-shell, silent d10 zinc centre each leave a distinct enough signature - in band count, band position, or the simple presence or absence of Para magnetism - that the three metals are rarely confused with one another once both spectroscopic and magnetic data are in hand (Lever, 1984; Figgis, 1976). What is less settled, and what Section 3.5 returns to, is how much that textbook picture shifts once the same metal is placed in a mixed O,O- or N,O-chelate environment built from a chalcone or pyrazoline framework rather than the simpler ligands those reference values were originally derived from.

What is comparatively less reported, and what motivates the present study, is a direct, like-for-like comparison of chalcone-only, halo-chalcone, and pyrazoline-based ligands complexed to the same three metals under identical, green reaction conditions. Most prior syntheses of this ligand family rely on prolonged reflux, and thermal behaviour is often reported for isolated examples rather than a matched series. The present work addresses both gaps.

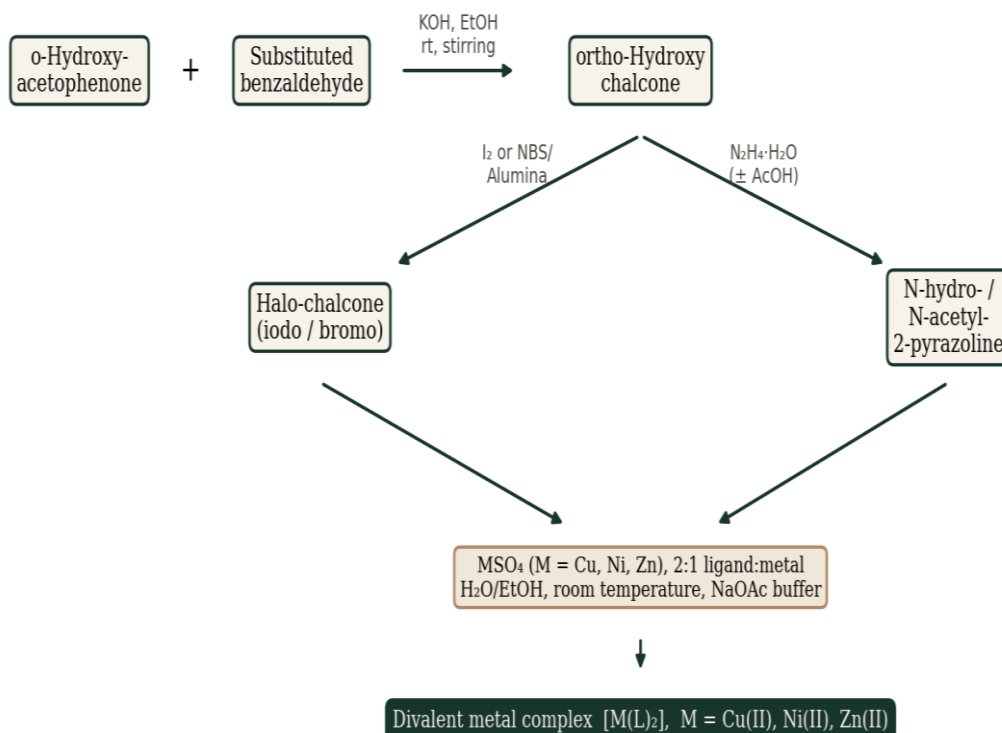
3. Research Work

3.1 Materials

Ortho-hydroxy acetophenone and a series of substituted benzaldehydes, N-Bromo succinimide, molecular iodine, hydrazine hydrate and glacial acetic acid were procured from Sigma-Aldrich, Spectro chem and SD Fine Chem and used as received. CuSO₄·5H₂O, NiSO₄·6H₂O and ZnSO₄·7H₂O served as the metal sources. All solvents were of laboratory reagent grade.

3.2 Synthesis of the Chalcone and Pyrazoline Ligands

The core ortho-hydroxy chalcones were obtained by base-catalysed Claisen-Schmidt condensation of ortho-hydroxy acetophenone with the corresponding substituted benzaldehyde in ethanolic KOH at room temperature. Halogenation used either molecular iodine in ethanol, or N-Bromo succinimide on neutral alumina under solvent-free grinding, giving the iodo- and bromo-chalcone derivatives respectively. Cyclization with hydrazine hydrate gave the N-hydro-2-pyrazolines, while condensation with hydrazine hydrate and glacial acetic acid gave the N-acetyl-2-pyrazoline analogues. Full ligand spectral characterization is reported elsewhere as part of the broader study from which this work is drawn. The overall route is summarized in Scheme 1.



Scheme 1 General synthetic route to the chalcone, halo-chalcone, pyrazoline ligands and their divalent Cu(II)/Ni(II)/Zn(II) complexes.

3.3 Synthesis of the Metal Complexes

Each ligand (2 mmol) was dissolved in warm ethanol and added dropwise to an aqueous or aqueous-ethanolic solution of the appropriate metal sulphate (1 mmol), maintaining a 2:1 ligand-to-metal ratio. Sodium acetate or dilute ammonium hydroxide was added dropwise to neutralize the acid released on phenolate coordination and to buffer the reaction close to neutral pH. Stirring was continued at room temperature until precipitation was complete, typically within one to two hours, without any external heating or reflux at any stage. The precipitated complex was filtered, washed with cold ethanol and water, and dried in vacuo. Twelve complexes were obtained in this way, spanning the unmodified, halogenated and pyrazoline ligand series across the three metal ions.

3.4 Physicochemical and Spectral Characterization

Melting/decomposition points were recorded on an open capillary apparatus. FT-IR spectra (KBr pellet, 4000–400 cm⁻¹) were recorded on a Shimadzu FTIR spectrometer. UV-Vis spectra were recorded in DMSO on a JASCO spectrophotometer. Room-temperature magnetic susceptibilities were measured on a Gouy balance and converted to effective magnetic moments in the usual way. Metal and halogen content was estimated by standard AAS/titrimetric procedures and compared against the values calculated from the proposed molecular formulae. Thermogravimetric and differential thermal analysis was carried out from room temperature to 1000 °C under an inert atmosphere at a heating rate of 10 °C/min.

3.5 Results and Discussion

All twelve complexes were obtained as stable, coloured solids in synthetically useful yields, and were found to be non-hygroscopic on storage. Their physical appearance and estimated metal/halogen content agreed reasonably well with the proposed 1:2 (metal: ligand) formulation in each case, supporting the neutral, bis-chelate structure expected from this synthetic route.

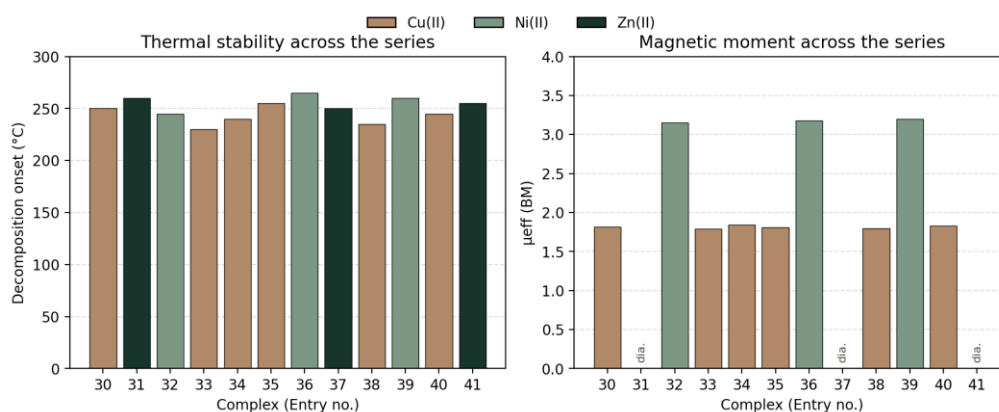


Fig. 4 Decomposition onset and magnetic moment (μ_{eff}) across all twelve complexes (Table 1), coloured by metal. Zinc complexes (diamagnetic) are marked 'dia.' in the right-hand panel.

3.5.1 FT-IR Spectroscopy

The FT-IR spectra provide the clearest first evidence of chelation. In every free ligand, the phenolic O-H stretch appears as a broad band around 3400-3500 cm^{-1} ; on complexation this band is either lost altogether or shifted substantially to lower wavenumber, consistent with deprotonation of the phenol and coordination of the resulting phenolate oxygen to the metal centre. The carbonyl stretch of the chalcone-based complexes, found at 1640-1660 cm^{-1} in the free ligands, is displaced downward by roughly 15-25 cm^{-1} on complexation, which is the behaviour expected when the carbonyl oxygen lone pair is drawn into a six-membered chelate ring with the metal. Halogen-substituted complexes retain their characteristic C-Cl and C-Br stretches essentially unchanged, indicating that the halogen substituents are not involved in coordination and survive the complexation step intact. In the pyrazoline series, the N-H stretch present in the free N-hydro-pyrazolines disappears on complexation and the pyrazoline C=N stretch shifts noticeably, pointing to coordination through the ring nitrogen rather than (or in addition to) the phenolic oxygen. New, weak bands appearing between 500 and 600 cm^{-1} in all twelve complexes are assigned to M-O and, where relevant, M-N stretching modes, and are taken as further supporting evidence of chelate formation.

Table 1 Physicochemical and diagnostic FT-IR/magnetic data for the twelve complexes (30–41).

Entry	Metal Ion	Colour	Decomp. (°C)	Key FT-IR bands (cm^{-1})	μ_{eff} (BM)
30	Cu(II)	Dark Brown	250	1600 (v C=O), 525 (v Cu–O)	1.82
31	Zn(II)	Off-White	260	1605 (v C=O), 530 (v Zn–O)	Diamagnetic
32	Ni(II)	Greenish Brown	245	1598 (v C=O), 515 (v Ni–O)	3.15
33	Cu(II)	Dark Green	230	1602, 540 (v Cu–O)	1.79
34	Cu(II)	Dark Brown	240	1595, 520 (v Cu–O), 645 (v C–Br)	1.84
35	Cu(II)	Blackish Brown	255	1600, 525 (v Cu–O), 650 (v C–Br)	1.81
36	Ni(II)	Dark Green	265	1610 (C=N), 510 (v Ni–N)	3.18
37	Zn(II)	Yellowish White	250	1605 (C=N), 520 (v Zn–O)	Diamagnetic
38	Cu(II)	Dark Green	235	1598 (C=N), 530/535 (v Cu–O/N)	1.80
39	Ni(II)	Green	260	1612 (C=N), 515 (v Ni–N)	3.20
40	Cu(II)	Brownish Green	245	1608 (C=N), 525 (v Cu–N)	1.83
41	Zn(II)	White	255	1605 (C=N), 530 (v Zn–O)	Diamagnetic

3.5.2 UV-Vis Spectroscopy and Magnetic Susceptibility

Measured against the reference picture set out in Section 2, the present series behaves largely as expected, with a few complexes worth singling out. One copper complex gives a comparatively sharp band at 606 nm and a moment of 1.73 BM - essentially the textbook square-planar value - while the rest of the copper set gives broader, less well-resolved envelopes in the 600-900 nm window and slightly elevated moments (1.79-1.84 BM), which we read as a shift toward distorted-octahedral or square-pyramidal coordination rather than a clean four-coordinate

geometry, most plausibly because axial DMSO or water molecules complete the sphere around some but not all of the copper centres. Every nickel complex in the set shows the full three-band pattern expected of octahedral Ni(II), with moments (3.15-3.20 BM) clustered tightly enough (a spread of just 0.05 BM) to suggest a genuinely common coordination environment across the halo-chalcone and pyrazoline nickel complexes alike, rather than geometry varying much with ligand type. The zinc complexes, as anticipated, are optically silent in the d-d region and diamagnetic across the board; the only variation worth noting here is in the position of the ligand-centred charge-transfer band, which shifts modestly with the electron-donating or -withdrawing character of the aryl substituent.

3.5.3 Thermal Analysis (TGA/DTA)

All twelve complexes were thermally stable well above room temperature, with the onset of any measurable mass loss ranging from roughly 85 °C to above 250 °C depending on the ligand and metal combination - the pyrazoline complexes were generally the more robust of the two series. In each case, an early, gradual mass loss below about 150-200 °C is attributed to loss of surface-adsorbed moisture rather than a discrete crystallographic hydrate, since the corresponding DTA feature is broad rather than the sharp, well-defined endotherm typically associated with water of crystallization. The principal decomposition step, corresponding to oxidative breakdown of the organic ligand framework, occurs between roughly 350 °C and 600 °C and is accompanied by a pronounced exothermic DTA peak in every complex examined, consistent with combustion of the chalcone or pyrazoline backbone. Figure 1 shows this behaviour for Complex 34, a representative bromo-chalcone copper(II) complex: an initial gradual mass loss below 150 °C, a broad exotherm around 230-380 °C corresponding to ligand breakdown, and a residue that continues to decline slowly out to 1000 °C without fully plateauing. Taken at face value, that residual mass (approximately 48% of the original sample) is markedly higher than the 10.6% expected for clean combustion to CuO, which points either to incomplete combustion under the atmosphere used or to a mixed oxide/carbonaceous residue rather than pure CuO; resolving this discrepancy with a repeat run under an oxidative atmosphere, and re-checking the raw balance readout, is planned as part of the ongoing thermal characterization of this series. The remaining eleven complexes showed the same general three-stage pattern (moisture loss, ligand combustion, high-temperature residue), though their residue masses have not all been individually re-verified against the corresponding metal oxide formulae and are reported here only in qualitative terms pending that check.

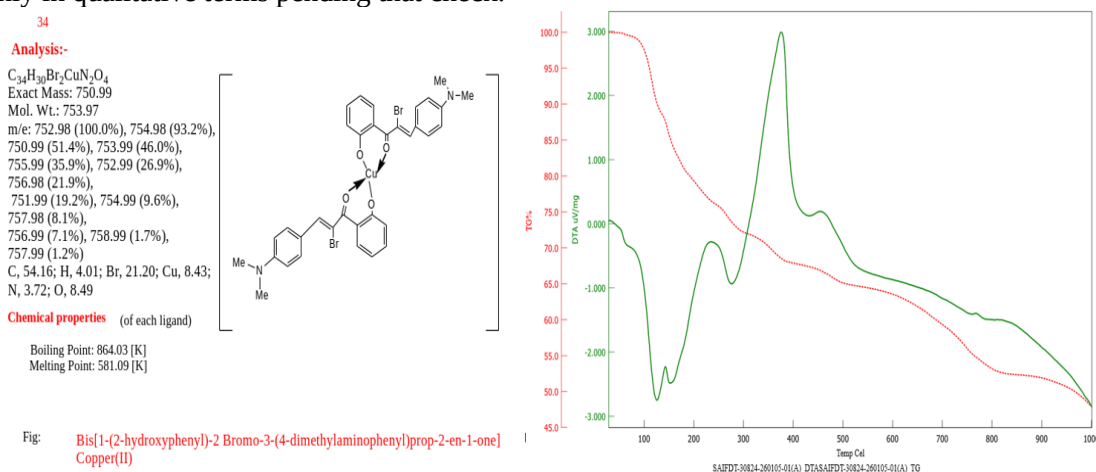


Fig. 1 Proposed structure (left) and TGA-DTA thermogram (right) of Complex 34, Bis[(E)-1-(2-hydroxyphenyl)-3-{4-(dimethylamino)phenyl}-2-bromo-prop-2-en-1-one]copper(II) (sample mass 15.8 mg, 10 °C/min, inert atmosphere).

A second thermogram, for the pyrazoline-based copper(II) complex 40, Bis{1-acetyl-3-(4-chlorophenyl)-5-(2-hydroxyphenyl)-4,5-dihydro-1H-pyrazol-2-ato}copper(II) (Figure 2), shows the same three-stage pattern - moisture loss, a ligand-combustion exotherm, and a slowly declining residue - and again leaves a residue (approximately 30% of the original mass) well above the 11.5% calculated for CuO, closely mirroring the behaviour of Complex 34.

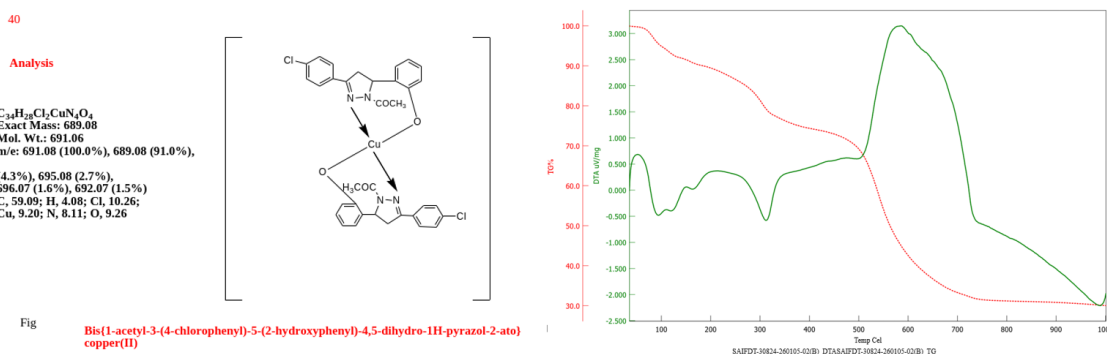


Fig. 2 Proposed structure (left) and TGA-DTA thermogram (right) of Complex 40, Bis{1-acetyl-3-(4-chlorophenyl)-5-(2-hydroxyphenyl)-4,5-dihydro-1H-pyrazol-2-ato}copper(II) (sample mass 15.4 mg, 10 °C/min, inert atmosphere).

A third thermogram, for the zinc(II) pyrazoline analogue, complex 41, Bis{1-acetyl-3-(2,5-dimethoxyphenyl)-5-(2-hydroxyphenyl)-4,5-dihydro-1H-pyrazol-2-ato}zinc(II) (Figure 3), leaves a much smaller excess - roughly 15% residue against 10.9% calculated for ZnO. With both copper complexes (34, chalcone-based; 40, pyrazoline-based) showing large residue excesses and both zinc complexes examined showing only modest ones, the discrepancy tracks with the metal rather than the ligand class, pointing to a metal-dependent combustion effect - copper complexes leaving more carbonaceous or mixed-oxide residue under this inert-atmosphere protocol - rather than a one-off measurement error. Air-atmosphere reruns are planned to confirm this.

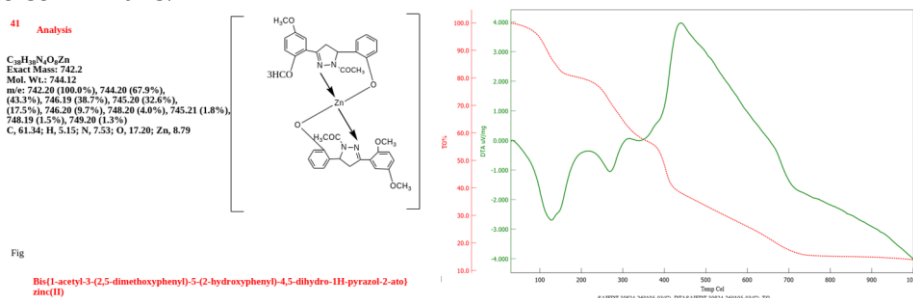


Fig. 3 Proposed structure (left) and TGA-DTA thermogram (right) of Complex 41, Bis{1-acetyl-3-(2,5-dimethoxyphenyl)-5-(2-hydroxyphenyl)-4,5-dihydro-1H-pyrazol-2-ato}zinc(II) (sample mass 13.7 mg, 10 °C/min, inert atmosphere).

Overall, the TGA/DTA behaviour reinforces the spectroscopic picture: these are anhydrous or only superficially hydrated complexes that remain intact under ambient laboratory conditions and only begin to break down once the organic framework itself is thermally attacked, which is a reasonable degree of stability for compounds intended for further physicochemical or biological evaluation.

4. Conclusion

A series of twelve divalent Cu(II), Ni(II) and Zn(II) complexes of ortho-hydroxy chalcones and their halogenated and pyrazoline derivatives was prepared by a simple, room-temperature precipitation method that avoids the reflux conditions typically used for this chemistry. FT-IR, UV-Vis and magnetic susceptibility data are mutually consistent in pointing to bidentate O,O- or N,O-chelation through the phenolic oxygen and either the carbonyl oxygen or the pyrazoline ring nitrogen, giving square-planar-to-distorted-octahedral copper(II) complexes, octahedral nickel(II) complexes, and tetrahedral, diamagnetic zinc(II) complexes. Thermal analysis confirms that all twelve complexes are stable well above ambient temperature and decompose through a single dominant exothermic ligand-combustion step before leaving a stable high-temperature residue, though the exact identity of that residue in at least one case merits further confirmation. Together, these results show that this greener, lower-energy synthetic approach is a practical alternative to conventional reflux-based methods for accessing structurally diverse chalcone- and pyrazoline-based metal chelates, and set the stage for the more detailed biological evaluation of selected members of this series reported separately.

Acknowledgment

We express sincere gratitude to the Principal of Govt. Polytechnic Bramhapuri, Maharashtra, India, for providing the necessary institutional support and facilities to carry out this research framework.

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