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Synthesis and Biological Activity of Some New β-Diketone Complexes of Cr(III)

M. B. HOGALE* AND B. R. KHOT

Department of Chemistry, Shivaji University,
Kolhapur—416 004, India

Various substituted β-diketone complexes of Cr(III) have been synthesised and characterised on the basis of analytical, infrared, nuclear magnetic resonance, electronic spectral and magnetic susceptibility data. The antimicrobial study of the complex was made against *S. aureus* and *E. coli*. Most of them have exhibited moderate antibacterial activity. A probable correlation of activity and the structure has been established.

INTRODUCTION

The wide profile of biological activities such as bactericidal[1], fungicidal[2], germicidal[3,4] and anticancer[5] have been shown by chalcones. Some methoxy and hydroxy β-diketones are of pharmaceutical[6] important compounds. Some naphthyl and phenanthryl β-diketones exhibit potent bactericidal and carcinogenic activity[7]. The *o*-hydroxy β-diketones are good chelating agents used for determination of metal ions[8-11]. In the present paper the co-ordination behaviour of some new various substituted β-diketone Cr(III) complexes is reported and structure activity relationship has been established.

EXPERIMENTAL

All the chemicals used were of reagent grade quality. The ligands diaroyl methanes were synthesised by Baker Venkataraman transformation[12,13] of *o*-hydroxy acetophenones. The substituted acrylophenones were prepared by condensation of various substituted aromatic aldehyde in the presence of base[14].

Preparation of Cr(III) Metal Complexes

Aqueous solution of chromium chloride and ethanolic solution of substituted acrylophenones were mixed in the 1 : 3 molar ratio and dilute solution of ammonium hydroxide was added till yellowish brown coloured complexes precipitate out (pH$>$ 4.5). The complexes were filtered, washed with distilled water, ethanol and dried. The microanalytical data of Cr(III) complexes have been presented in Table 1.

RESULTS AND DISCUSSION

The elemental analyses indicate that the metal to ligand ratio of 1 : 3 for chromium(III) complexes. All the complexes are non-hygroscopic,

TABLE 1
PHYSICAL, ANALYTICAL AND MAGNETIC MOMENT DATA OF CHROMIUM(III) COMPLEXES

Compound	Groups							M.pt. Decomp. temp. °C	Analysis % Found (Calcd)			μ_{eff} B.M.
	R_3	R_5	R_6	X_4	Y_4	Y_5	Y_6		N	Cl	Cr	
$[C_{69}H_{30}O_{24}N_6Cr]Cl_3$	NO_2	CH_3	H	NO_2	H	OH	H	270	5.53 (5.65)	7.04 (7.16)	3.04 (3.50)	3.74
$[C_{69}H_{48}O_{15}N_3Cr]Cl_3$	H	CH_3	H	NO_2	H	H	H	304	3.08 (3.19)	7.94 (8.07)	3.83 (3.94)	3.70
$[C_{69}H_{48}O_{18}N_3Cr]Cl_3$	H	H	CH_3	NO_2	OH	H	H	300	2.96 (3.07)	7.70 (7.79)	3.70 (3.81)	3.75
$[C_{69}H_{42}O_{21}N_6Cr]Cl_3$	NO_2	CH_3	H	NO_2	H	H	Cl	260	5.69 (5.79)	7.23 (7.34)	3.48 (3.59)	3.73
$[C_{69}H_{45}O_{15}N_3Cr]Cl_3$	H	CH_3	H	NO_2	H	Cl	H	300	3.10 (3.19)	8.00 (8.09)	3.85 (3.95)	3.78
$[C_{69}H_{51}O_9Cr]Cl_3$	H	CH_3	H	H	H	H	H	310	— —	8.90 (9.00)	4.29 (4.40)	3.80
$[C_{69}H_{51}O_{12}Cr]Cl_3$	H	CH_3	H	H	H	OH	H	165	— —	8.54 (8.65)	4.12 (4.22)	3.75
$[C_{72}H_{51}O_{24}N_6Cr]Cl_3$	NO_2	CH_3	H	NO_2	H	H	OCH_3	225	5.33 (5.44)	6.78 (6.90)	3.27 (3.37)	3.81
$[C_{72}H_{54}O_{18}N_3Cr]Cl_3$	H	CH_3	H	NO_2	H	H	OCH_3	300	2.79 (2.98)	7.45 (7.56)	3.60 (3.69)	3.85
$[C_{72}H_{57}O_{15}Cr]Cl_3$	H	CH	H	H	H	OCH_3	OH	295	— —	7.95 (8.06)	3.85 (3.94)	3.72
$[C_{75}H_{57}O_9Cr]Cl_3$	H	CH_3	H	H	H	H	C_2H_3	135	— —	8.33 (8.44)	4.00 (4.12)	3.83
$[C_{75}H_{57}O_{27}N_6Cr]Cl_3$	NO_2	CH_3	H	NO_2	H	OCH_3	OCH_3	250	5.02 (5.14)	6.40 (6.51)	3.09 (3.18)	3.80
$[C_{75}H_{63}O_{15}N_6Cr]Cl_3$	H	H	CH_3	NO_2	H	H	$N(CH_3)_2$	300	5.69 (5.81)	7.23 (7.35)	3.48 (3.59)	3.77
$[C_{75}H_{60}O_{21}N_3Cr]Cl_3$	H	CH_3	H	NO_2	OCH_3	H	OCH_3	285	2.69 (2.80)	6.97 (7.10)	3.36 (3.47)	3.79

insoluble in water and many of the common organic solvents. Magnetic moment of the Cr(III) complexes have been found to lie in the range 3.70–3.85 B.M. (Table 1) indicating the presence of three unpaired electron supporting octahedral geometry of complexes.

Due to intramolecular hydrogen bonding in *o*-hydroxy aromatic aldehydes and ketones νC—OH generally observed at 3410 cm^{-1}.[15] This band was found to be absent in IR spectra of Cr(III) complexes. The co-ordination of the oxygen of the hydroxyl group is confirmed by the lowering of νC—O from 1100–1120 to 1040–1060 cm^{-1}. Two sharp and strong bands assigned for νC—O at 1680, 1600 cm^{-1} in the free ligand, the band at lower frequency region is lowered by 50–60 cm^{-1} on complexation thereby indicating the co-ordination from carbonyl oxygen. All the complexes showed a band at 450 cm^{-1} to ν(Cr—O). The ν(C—O) (phenolic) is observed in the free ligand at 1280 cm^{-1} and on complexation with transition metals this band shift to higher energy by about 40–55 cm^{-1} indicating phenolic oxygen co-ordination of the acrylophenone.

The bands have been observed for the Cr(III) complexes, at 20833 and 26315 cm^{-1}. These may be assigned to the $A_{2g} \rightarrow T_{2g}$ and $A_{2g} \rightarrow T_{1g}$ transitions respectively.

In the ^1H nmr spectra, the signals due to aromatic —CH_3 observed as singlet at δ, 2.3 while the aromatic protons are encountered as the multiplet between δ, 6.8–7.5 ppm. The nmr signal due to —OH (phenolic) group was found to be disappeared because of the complexation with the metal atom.

All the complexes were screened for their antimicrobial activity by agar diffusion technique[16] using dimethyl formamide solvent against *Staphylococcus aureus* (gram +ve) and *Escherichia coli* (gram −ve) bacteria at 25 to 50 μg/ml concentration. The tetracycline was used as standard compound for comparison. These complexes were found to be moderately active against gram + ve and gram − ve bacteria. The presence of halogen as well as nitro group in the ligand moiety of the complexes enhances the bacteristatic activity.

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