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# Complexes of Si (IV), Ge (IV), Sn (IV), Ti (IV) and Zr (IV) Metals with N, N′—Bis(8-Aceto-7-hydroxy-2-methyl coumarin) Hydrazine Schiff Base

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N,N′-Bis (8-aceto-7-hydroxy-4-methyl coumarin) hydrazine ($AHCH_y$) has been synthesised from 8-acetyl-7-hydroxy-4-methyl eoumarin (AHC) with hydrazine hydrate. Group IV metal complexes with ($AHCH_y$) has been prepared and characterised on the basis of elemental analysis. IR spectroscopy, conductance measurements and molecular weight determination.

In continuation of our earlier work on complexing ability of coumarin and its derivatives[1,2], some complexes of Si(IV), Ge(IV), Sn(IV), Ti(IV) and Zr (IV) with the schiff base ($AHCH_y$) has been taken up for study.

8-Acetyl-7-hydroxy-4-methyl coumarin (AHC) was prepared in the laboratory[3]. $SiCl_4$, $SnCl_4$, $TiCl_4$, $ZrCl_4$ (BDH), $GeCl_4$ (Koch Light) and hydrazine hydrate (fluka) were used. The conductivity measurement was made on an Elico Conductivity bridge type CM-82 T. The molecular weights were determined by viscosity method. The IR spectra (4000–200 $cm^{-1}$) and micro analysis were made as reported earlier[4]. AHC (0.02 mol) taken in 20 ml of acetone was added to hydrazine hydrate (0.01 mol) in methanol (50 ml) and the mixture was refluxed for 2–5 hrs. on water both when a brown compound was obtained. The solid was filtered, washed with ether and dried in *vacuo*.

Metal chloride (0.01 mol) dissolved in 20 ml of $CCl_4$ was added to a solution (20 ml) of the schiff base (0.01 mol) in acetone. A solid was obtained after stirring the mixture for about 4 hrs. which was washed with ether and dried. They had 1:1 ($MCl_4$: schiff base) ratio as confirmed by a quantitative estimation of chlorine.

The analytical results for the schiff base and the complexes are in agreement with proposed composition (Table I). The molar conductance values of the complexes in DMSO are below in the expected range of a uni-unielectrolyte (2.0–7.4 $ohm^{-1}$ $cm^2$ $mol^{-1}$).

The schiff base shows a medium absorption band at 3350 $cm^{-1}$ assigned to phenolic OH which persists even after complexation ruling out the involvement of phenolic OH group. A strong band at 1300 $cm^{-1}$ in the schiff base may be assigned to $\delta$ OH (phenolic). The $\nu$ (C = N) frequency at 1640 $cm^{-1}$ in the ligand is lowered by about 20 $cm^{-1}$ indicating the coordination solely through nitrogen atoms[5,6]. The far IR

TABLE 1

ANALYTICAL AND MELTING POINT OF COMPLEXES OF GROUP (IV) METAL CHLORIDES

| Schiff base/ complex | m.p. (°C) | Found (Calcd.) % | | | | Mol. Wt. |
|---|---|---|---|---|---|---|
| | | C | H | N | Cl | Found (Calcd.) |
| $C_{24}H_{20}O_6N_2$ | 157 | 66.10<br>66.66 | 4.52<br>(4.62) | 6.43<br>(6.48) | — | 425<br>(422) |
| $SiCl_4.L$ | 300$^d$ | 47.20<br>(47.84) | 3.26<br>(3.32) | 4.61<br>(4.65) | 23.42<br>(23.58) | 600<br>(602) |
| $GeCl_4.L$ | 250$^d$ | 44.30<br>(44.53) | 3.08<br>(3.09) | 4.27<br>(4.33) | 21.85<br>(21.96) | 645<br>(646.6) |
| $SnCl_4.L$ | 245$^d$ | 41.00<br>(41.58) | 2.81<br>(2.88) | 4.00<br>(4.04) | 20.45<br>(20.50) | 692<br>(692.6) |
| $TiCl_4.L$ | 290$^d$ | 46.10<br>(46.30) | 3.17<br>(3.21) | 4.43<br>(4.50) | 22.70<br>(22.83) | 620<br>(621.9) |
| $ZrCl_4.L$ | 275$^d$ | 43.04<br>(43.29) | 2.99<br>(3.00) | 4.16<br>(4.20) | 21.00<br>(21.04) | 665<br>(665.2) |

L = $C_{24}H_{20}O_6N_2$ (Schiff Base)

region of the spectra is very significant as it includes the M-N and M-X stretchings. The M-X absorption bands are stronger and appear at lower region (400–310 cm$^{-1}$).

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