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### New Chelating Resins: 2-Hydroxy-4-methoxyacetophenone-Formaldehyde and 2-Hydroxy-4-methoxyacetophenone Oxime-Formaldehyde

J. S. Parmar<sup>a</sup>; M. R. Patel<sup>a</sup>; M. M. Patel<sup>a</sup>

<sup>a</sup> Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar, India

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## **New Chelating Resins: 2-Hydroxy-4-methoxyacetophenone-Formaldehyde and 2-Hydroxy-4-methoxyacetophenone Oxime-Formaldehyde**

J. S. PARMAR, M. R. PATEL, and M. M. PATEL

Department of Chemistry  
Sardar Patel University  
Vallabh Vidyanagar 388120, India

### **ABSTRACT**

This paper describes the syntheses and characterization of chelating resins, 2-hydroxy-4-methoxyacetophenone-formaldehyde (I) and 2-hydroxy-4-methoxyacetophenone oxime-formaldehyde (II). The number-average molecular weights have been determined by vapor pressure osmometry and nonaqueous conductometric titration methods. Metal chelates of the resins with Co(II), Ni(II) and Cu(II) were prepared. Elemental analysis of the chelates indicates a metal-to-ligand ratio of 1:2. The diffuse reflectance spectra and magnetic moments of Ni(II) and Co(II) chelates show a tetrahedral structure whereas Cu(II) chelate shows a square planar structure. Infrared spectral study show that the metal ions are coordinated through the oxygen of carbonyl group in I or nitrogen of the oximino group in II, and through the oxygen of the phenolic hydroxyl group. The presence of methylene bridges in the resins is also indicated by IR spectral studies.

## INTRODUCTION

2-Hydroxy-4-methoxyacetophenone [2,4-OH(OCH<sub>3</sub>)AP] and 2-hydroxy-4-methoxyacetophenone oxime [2,4-OH(OCH<sub>3</sub>)APO] are well-known complexing agents. The formation of transition metal complexes of 2,4-OH(OCH<sub>3</sub>)APO in aqueous organic mixtures have been studied [1]. The oxime is also used in the determination of uranium by spectrophotometry [2].

Condensation of 2,4-OH(OCH<sub>3</sub>)AP with formaldehyde under acidic condition [3] resulted in the formation of 3,3', 3,5', and 5,5'-bis[2,4-OH(OCH<sub>3</sub>)AP] methylene derivatives, and 5,5' derivative was converted to homobiflavanone. E. I. du Pont de Nemours and Co. [4, 5] has prepared a heat and light stabilizer by condensing 2,4-OH(OCH<sub>3</sub>)AP, phenol, and formaldehyde, and used this compound for the stabilization of polyolefins.

In continuation of our studies on chelating resins and their metal chelates [6, 7], we report here the synthesis and characterization of 2-hydroxy-4-methoxyacetophenone-formaldehyde (I) and 2-hydroxy-4-methoxyacetophenone oxime-formaldehyde (II) resins, and their metal chelates with Co(II), Ni(II) and Cu(II) ions. The characterization was done by elemental analysis, molecular weight determination, magnetic moments, reflectance spectra, and IR spectra.

## EXPERIMENTAL

Chemicals

2-Hydroxy-4-methoxyacetophenone was prepared by monomethylation of 2,4-dihydroxyacetophenone with dimethyl sulfate as the methylating agent [8], mp 52°C [9], and its oxime by oximation with hydroxylamine hydrochloride, mp 129°C [10].

A.R. grade pyridine (BDH) was purified by refluxing it over sodium hydroxide pellets and then distilling with careful exclusion of moisture. A.R. grade methanol and dimethylformamide (DMF) were distilled once and used as solvent.

s-Trioxane (BDH), p-toluene sulfonic acid (p-TSA), and all other reagents were of analytical grade and used without further purification.

Preparation of 2,4-OH(OCH<sub>3</sub>)AP-F Resin (I)

2-Hydroxy-4-methoxyacetophenone (3.3 g, 0.02 mol), s-trioxane (0.9 g 0.03 mol of CH<sub>2</sub>O), and p-toluene sulfonic acid (0.2 g) were added one after another to the reaction tube. The tube was sealed and placed in an oil bath at 100 ± 1°C for 12 h. The tube was allowed to cool at room temperature, desealed, and the resin lumps were broken into

pieces with a glass rod, filtered, and washed with water. The resin was powdered, placed in a round-bottom flask, and refluxed with methanol (100 mL) for 2 h in order to remove unreacted monomer. The resin was filtered and dried at 60°C. The safron-colored resin was further purified by dissolving in DMF (30 mL) and adding the filtered solution dropwise into a large excess of aqueous sodium chloride solution (1 L, 2%) with constant stirring. The separated resin was filtered, washed several times with distilled water until free from chloride ions, and dried at 60°C. Yield: 3.45 g (98%).

Preparation of 2,4-OH(OCH<sub>3</sub>)APO-F Resin (IIa)  
by Condensation of 2,4-OH(OCH<sub>3</sub>)APO with  
s-Trioxane

2-Hydroxy-4-methoxyacetophenone oxime (3.6 g, 0.02 mol), s-trioxane (0.9 g, 0.03 mol of CH<sub>2</sub>O), and p-TSA (0.2 g) were added to the reaction tube. The tube was sealed and placed in an oil bath at 100 ± 1°C for 24 h. The tube was allowed to cool at room temperature, desealed, and the dark gummy product was collected in a beaker. The product easily dissolved in methanol. On dilution of this solution with water a turbid aqueous solution was formed and a resin paste settled to the bottom. Precipitates were separated from the turbid solution on cooling in an ice bath, filtered, washed with cold water, and dried at 60°C. The yield of precipitates was 60%, mp 121°C, and of resin 35%, softening point 15°C.

Preparation of 2,4-OH(OCH<sub>3</sub>)APO-F Resin (II) by  
Oximation of 2,4-OH(OCH<sub>3</sub>)AP-F Resin

2-Hydroxy-4-methoxyacetophenone-formaldehyde resin (3.56 g, 0.02 mol of repeat unit) was dissolved in ethanolic sodium hydroxide solution (2 N, 12 mL) and placed in a round-bottom flask. To this solution, hydroxylamine hydrochloride (3.6 g, 0.05 mol) in water (5 mL) was added and the contents refluxed on an isomental bath for 20 h. The flask was allowed to cool and the contents were poured on crushed ice, neutralized, and the separated resin filtered, washed with water, and dried at 60°C. The resin was purified by dissolving in DMF (25 mL), and the resulting solution, after filtration, was added dropwise to a large excess of aqueous sodium chloride solution (1 L, 2%) with constant stirring. The light brown resin that separated out was filtered, washed several times with distilled water until free of chloride ions and dried at 60°C. Yield: 3.7 g (98%).

### Preparation of Metal Chelates of 2,4-OH(OCH<sub>3</sub>)AP-F Resin Ni(II) Chelate

2-Hydroxy-4-methoxyacetophenone-formaldehyde resin (3.56 g, 0.02 mol of repeat unit) was dispersed in methanol (50 mL) and methanolic NaOH solution (1%, 5-6 mL) was added to dissolve the resin. To this solution Ni(CH<sub>3</sub>COO)<sub>2</sub>·4H<sub>2</sub>O (4.0 g, 0.016 mol), dissolved in water (25 mL), was added dropwise with constant stirring. The mixture was digested at 50°C for about 3 h in a water bath. The separated greenish yellow colored Ni(II) chelate was filtered, washed with distilled water, and dried at 50°C.

### Co(II) and Cu(II) Chelate

The Co(II) and Cu(II) chelates were prepared by the method described above for the Ni(II) chelate. The Co(II) chelate was brown in color and that of Cu(II) was light green.

### Preparation of Metal Chelates of 2,4-OH(OCH<sub>3</sub>)APO-F Resin Ni(II) Chelate

2-Hydroxy-4-methoxyacetophenone oxime-formaldehyde resin (1.93 g, 0.01 mol of repeat unit) was dissolved in methanol (60 mL) with warming. Ni(CH<sub>3</sub>COO)<sub>2</sub>·4H<sub>2</sub>O (1.5 g, 0.006 mol), dissolved in water (10 mL), was added dropwise to this hot solution at pH 6 to 6.5 with constant stirring. The solution was allowed to stand at room temperature for 12 h. The separated greenish yellow chelate was filtered, washed with distilled water, and dried at 50°C.

### Co(II) Chelate

2-Hydroxy-4-methoxyacetophenone oxime-formaldehyde resin (1.93 g, 0.01 mol of repeat unit) was dissolved in methanol (60 mL) on warming. Co(CH<sub>3</sub>COO)<sub>2</sub>·H<sub>2</sub>O (1.2 g, 0.006 mol), dissolved in water (10 mL), was added dropwise to the hot resin solution with constant stirring and then allowed to stand at room temperature for 12 h. The separated light brown colored Co(II) chelate was filtered, washed with distilled water, and dried at 50°C.

### Cu(II) Chelate

The Cu(II) chelate of 2,4-OH(OCH<sub>3</sub>)APO-F resin was prepared by the method described above for the Co(II) chelate, and a light green colored chelate was obtained.

Analysis and Physical Measurements

Elemental analysis, diffuse reflectance spectra, infrared spectra, and magnetic moments of the polymeric chelates and determination of the molecular weight of the resins were carried out following the procedures described in an earlier communication [6].

## RESULTS AND DISCUSSION

The resins prepared from 2,4-OH(OCH<sub>3</sub>)AP and 2,4-OH(OCH<sub>3</sub>)APO were of very low molecular weight and contained large amounts of unreacted monomer. Therefore, 2,4-OH(OCH<sub>3</sub>)AP-F resin has been prepared by using a nonaqueous system in which s-trioxane is used as a source of formaldehyde and p-TSA as a catalyst [11] (Fig. 1).

2,4-OH(OCH<sub>3</sub>)APO-F, Resin IIa was found to be dark and gummy as well as of very low molecular weight, and so Resin II was prepared by direct oximation of the parent resin (Fig. 2). Resin I had a softening point of 150°C and Resin II of 152°C.

Resins I and II are soluble in dimethylformamide, tetrahydrofuran, pyridine, and aqueous alkali. Resin II is soluble in hot methanol and ethanol.

Figure 3 represents the conductometric titration curves of Resins I and II. From the formula weight of the repeat unit (I, 178; II, 193), 100 g of the resin should contain 562 meq in I and 517 meq in II of phenolic OH groups. The smallest interval between successive breaks

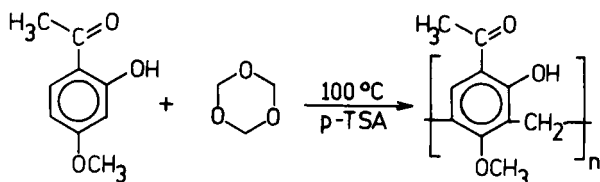


FIGURE 1.

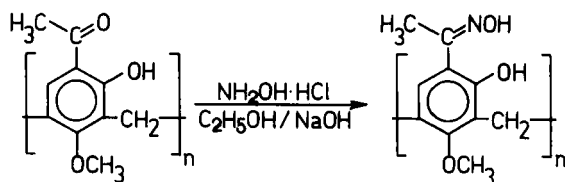


FIGURE 2.

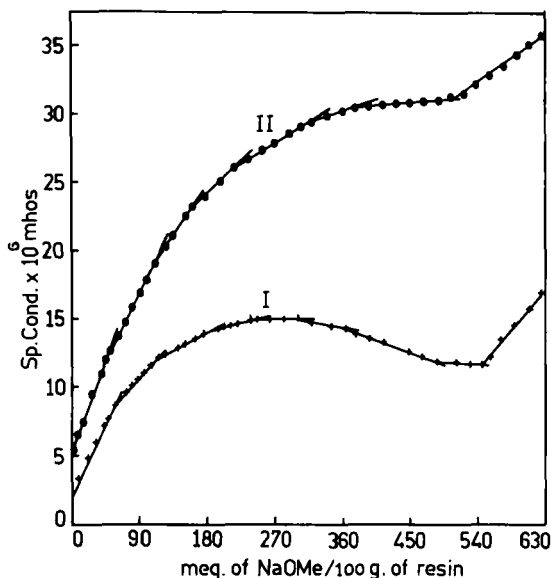


FIG. 3. Conductometric titration curves of 2,4-OH(OCH<sub>3</sub>)AP-F (I) and 2,4-OH(OCH<sub>3</sub>)APO-F (II) resins in pyridine solvent with sodium methoxide in pyridine.

in Curve I is 60 and in Curve II, 54. The average degree of polymerization ( $\overline{DP}$ ) of the resins will thus be  $562/60 = 9.35$  for I and  $517/54 = 9.57$  for II [12, 13]. The number-average molecular mass ( $\overline{M}_n$ )

could thus be obtained by multiplying  $\overline{DP}$  by the respective formula weights. Thus  $\overline{M}_n$  for I is 1664 g/mol and II is 1852 g/mol.  $\overline{M}_n$  of

these two resins was also determined in DMF solvent at 70°C by vapor pressure osmometry. The values are for I,  $1503 \pm 10\%$  g/mol, and for II,  $1596 \pm 10\%$  g/mol. The values from both methods are in good agreement. The increase in molecular weight of Resin II is found to be as expected and thus suggests that oximation of  $>C=O$  group is almost complete. This was also confirmed by a nitrogen estimation of Resin II (Table 1). The values of  $\overline{DP}$  for both resins are almost equal, which indicates that no breaking of resin chains occurs during oximation.

Table 2 and Figs. 4 and 5 represent the important infrared absorption bands and spectra of Resins I and II. The bands appearing at  $3330 \pm 30 \text{ cm}^{-1}$  are due to  $-OH$  stretching vibrations [14]. The strong bands of  $\nu C=O$  and  $\nu C=N$  at lower frequency,  $1620 \pm 10 \text{ cm}^{-1}$ , and the weak band of  $\nu OH$  at  $2750 \text{ cm}^{-1}$  indicate intramolecular hydrogen bonding [15]. The vibration absorption bands at  $2880 \pm 10$ , 1425, and  $715 \text{ cm}^{-1}$  are observed in the spectra of the resins and not in the spectra of the

TABLE 1. Elemental Analysis and Magnetic Moment Data of Resin I, Resin II, and Their Polymeric Chelates

Main unit	Color	C (%)	H (%)	N (%)	Me (%)	$\mu_{\text{eff}}$ (BM)
Resin I	White	66.64	6.18	-	-	-
$\text{C}_{10}\text{H}_{10}\text{O}_3$		(67.42) <sup>a</sup>	(5.62)			
$\text{Cu}(\text{C}_{10}\text{H}_9\text{O}_3)_2$	Light green	55.8 (57.5)	5.10 (4.31)	-	14.24 (15.23)	1.64 (1.73)
$\text{Ni}(\text{C}_{10}\text{H}_9\text{O}_3)_2$	Greenish yellow	57.2 (58.1)	4.8 (4.35)	-	13.90 (14.24)	2.1 (2.83)
$\text{Co}(\text{C}_{10}\text{H}_9\text{O}_3)_2$	Brown	56.6 (58.1)	4.73 (4.35)	-	14.06 (14.28)	3.14 (3.88)
Resin II	Brown	62.10	5.75	7.10	-	-
$\text{C}_{10}\text{H}_{11}\text{NO}_3$		(62.17)	(5.70)	(7.25)		
$\text{Cu}(\text{C}_{10}\text{H}_{10}\text{NO}_3)_2$	Light green	54.4 (53.6)	5.6 (4.9)	6.15 (6.25)	13.80 (14.20)	1.62 (1.73)
$\text{Ni}(\text{C}_{10}\text{H}_{10}\text{NO}_3)_2$	Greenish yellow	54.8 (54.2)	4.9 (4.5)	5.98 (6.32)	13.20 (13.36)	2.91 (2.83)
$\text{Co}(\text{C}_{10}\text{H}_{10}\text{NO}_3)_2$	Light brown	54.8 (54.2)	5.2 (4.5)	5.85 (6.32)	13.10 (13.36)	3.96 (3.88)

<sup>a</sup>Values calculated on the basis of main unit weight.



TABLE 2. IR Spectral Data ( $\text{cm}^{-1}$ )

	Resin I	Ni(II) chelate	Co(II) chelate	Cu(II) chelate	Probable assignments
2,4-OH(OCH <sub>3</sub> )AP					
3300 (sh) <sup>a</sup>	3360 (sb)	3360 (sb)	3360 (sb)	3365 (sb)	-OH stretching and
3180 (vw)	3180 (m)	3180 (sh)	3180 (sh)	3180 (sh)	$\nu$ OH intramolecular H bonded.
1635 (vs)	1625 (vs)	1625 (s)	1620 (vs)	1610 (vs)	$\nu$ C=O stretching
-	2890 (mb)	2890 (mb)	2890 (mb)	2890 (mb)	$\nu$ as -CH <sub>2</sub> - stretching
-	1425 (m)	1425 (m)	1425 (m)	1425 (m)	-CH <sub>2</sub> - scissoring
1260 (s)	1265 (s)	1265 (s)	1260 (s)	1265 (s)	$\delta$ -OH deformation or in-plane bending or $\nu$ as =C-O stretching
830 (vs)	830 (s)	840 (s)	850 (s)	840 (s)	2 adjacent H on phenyl ring or
-	800 (s)	800 (s)	800 (s)	800 (s)	1,2,4-trisubstituted phenyl ring
-	865 (m)	865 (m)	870 (m)	860 (m)	1,2,4,5-tetrasubstituted and
-	910 (w)	910 (w)	910 (w)	910 (w)	pentasubstituted phenyl ring

2,4-OH(OCH <sub>3</sub> ) <sub>2</sub> APO	Resin II	Ni(II) chelate	Co(II) chelate	Cu(II) chelate	Probable assignments
3280 (sb)	3350 (mb)	3380 (mb)	3380 (sb)	3350 (sb)	-OH stretching and
2780 (m)	2750 (m)	-	-	-	$\nu$ OH intramolecular H bonded
1610 (s)	1610 (s)	1610 (s)	1610 (s)	1610 (s)	$\nu$ C=N stretching
-	2900 (m)	2870 (s)	2870 (s)	2870 (s)	$\nu$ as -CH <sub>2</sub> - stretching
-	1440 (s)	1440 (s)	1440 (s)	1440 (s)	-CH <sub>2</sub> - scissoring
-	715 (s)	710 (w)	705 (w)	705 (w)	-CH <sub>2</sub> - rocking
1275 (s)	1270 (s)	1275 (s)	1275 (s)	1270 (s)	$\delta$ -OH in-plane bending
970 (s)	980 (s)	1010 (sh)	1010 (sh)	1010 (sh)	$\nu$ N-O stretching
815 (s)	800 (s)	800 (m)	800 (m)	790 (m)	2 adjacent H on phenyl ring
860 (s)	860 (s)	860 (s)	870 (s)	860 (s)	1,2,4,5-tetrasubstituted phenyl ring and isolated H.
	910 (w)	910 (w)	910 (w)	910 (w)	

<sup>a</sup> $\nu$  denotes very, s strong, w weak, m medium, and b broad.

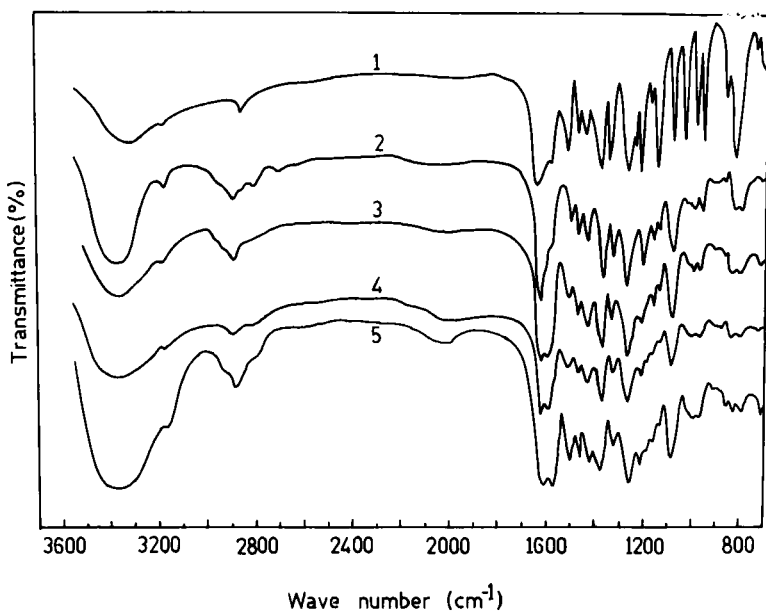


FIG. 4. IR spectra of 2,4-OH(OCH<sub>3</sub>)AP (1), Resin I (2), (Resin I)<sub>2</sub>Ni (3), (Resin I)<sub>2</sub>Co (4), and (Resin I)<sub>2</sub>Cu (5).

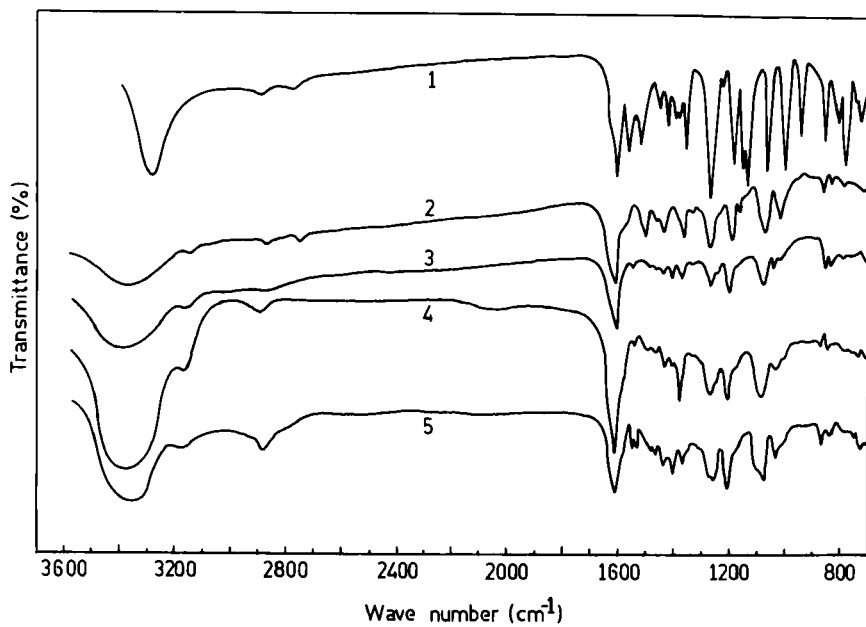
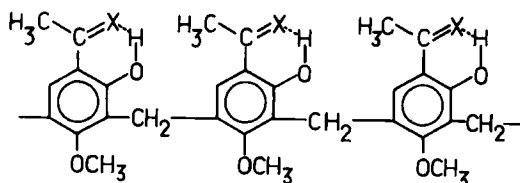


FIG. 5. IR spectra of 2,4-OH(OCH<sub>3</sub>)APO (1), Resin II (2), (Resin II)<sub>2</sub>Ni (3), (Resin II)<sub>2</sub>Co (4), and (Resin II)<sub>2</sub>Cu (5).

respective monomers, which suggest that phenolic moieties are linked through methylene bridges in the resins [15, 16]. The strong band at  $980\text{ cm}^{-1}$  in Resin II is attributed to N-O stretching [15].

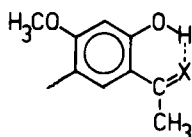
On the basis of the reactivity of the positions in the monomers, the resins should have a general structure of the type shown in Structure I.



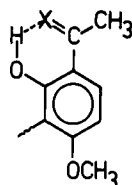
Where  $X = \text{O}$  or  $\text{NOH}$

Structure I

According to Structure I, each of the phenyl rings (except the end-groups) of the chain would be penta-substituted and contain only one isolated aromatic hydrogen atom. The endgroup could be either Structure II or Structure III.



Structure II



Structure III

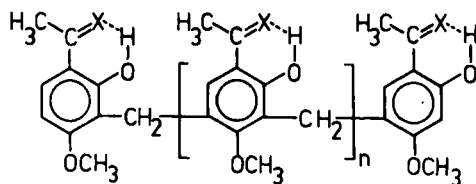
Where  $X = \text{O}$  or  $\text{NOH}$

Thus in a single polymer chain the inner phenyl rings as well as the endgroup phenyl ring (Structure II) contain an isolated H atom. The strong out-of-plane C-H bending band around  $900\text{ cm}^{-1}$  is due to penta-substituted and 1,2,4,5-tetrasubstituted phenyl rings [17]. The other possible type of endgroup (Structure III) contains two adjacent H atoms which are observed around  $830$  and  $800\text{ cm}^{-1}$  in the spectra [17].

From the infrared spectral study and the analytical results (Table 1), the structure of these two resins could be as shown in Structure IV.

On treatment with aqueous solutions of  $\text{Co(II)}$ ,  $\text{Ni(II)}$  and  $\text{Cu(II)}$  acetates, methanolic solutions of the resins containing a small amount of alkali precipitated the polymeric chelates. These chelates are almost insoluble in any solvent.

Elemental analyses data of the polymeric chelates (Table 1) suggest a metal-to-ligand ratio of 1:2. The important IR absorption bands

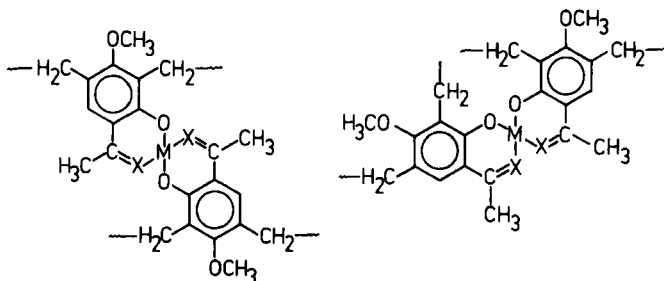


Where X = O or NOH

Structure IV

of polymeric chelates are shown in Table 2 and the IR spectra in Figs. 4 and 5. The strong bands at  $3350\text{--}3380\text{ cm}^{-1}$  are due to  $\text{--OH}$  stretching vibrations. The band at  $2750\text{ cm}^{-1}$  is absent in chelates, suggesting replacement of the hydrogen of the phenolic group by metal ions. The bands appearing at  $1570\text{--}1600\text{ cm}^{-1}$  are attributed to  $\nu\text{C=O}$  or  $\nu\text{C=N}$  stretching vibrations. These bands are observed at lower frequencies compared to the usual position, indicating coordination of metal ions through the oxygen of the carbonyl group or the nitrogen of the oximino group. The band at  $980\text{ cm}^{-1}$  in the resin of  $\nu\text{N--O}$  is found to be at a higher frequency,  $1010\text{ cm}^{-1}$ , in the chelates. This also indicates the coordination with nitrogen of the oximino group in Resin II [18]. The other bands due to  $\text{--OCH}_3$  and substitution patterns were found to be similar in polymeric chelates and resins.

The infrared spectral studies, elemental analysis, and solubility data of polymeric chelates suggest that chelation might have occurred between two chelating groups from different polymeric chains and not from the adjacent groups in the same chain, as shown in Structures V and VI.



Structure V

Structure VI

Where X = O or NOH

M = Co, Ni or Cu

The diffuse reflectance spectra of the polymeric chelates were studied in the range  $11,110\text{--}25,000\text{ cm}^{-1}$  and the spectra are shown in Fig. 6. Cu(II) chelate exhibits one broad band centered at  $15,870\text{ cm}^{-1}$ . This band could be assigned to  $d\text{--}d$  transition corresponding to the  ${}^2E_g \text{--} {}^2T_{2g}$  transition. The position of this band is in the range gener-

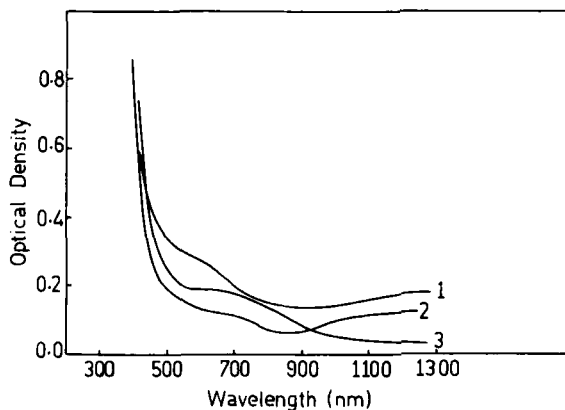


FIG. 6. Diffuse reflectance spectra of  $(\text{Resin I})_2\text{Co}$  (1),  $(\text{Resin I})_2\text{Ni}$  (2), and  $(\text{Resin I})_2\text{Cu}$  (3).

ally observed for planar  $\text{Cu}(\text{II})$  complexes [19].  $\text{Ni}(\text{II})$  chelate also exhibits a broad band around  $16,390\text{ cm}^{-1}$  which is in the range of tetrahedral  $\text{Ni}(\text{II})$  complexes and is assigned to the  ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_1(\text{P})$  transition [20]. The  $\text{Co}(\text{II})$  chelate shows a broad band centered at  $17,540\text{ cm}^{-1}$  which is assigned to the  ${}^4\text{A}_2 \rightarrow {}^4\text{T}_1(\text{P})$  transition, indicating a tetrahedral geometry [20].

Magnetic moments of the polymeric chelates have been studied to examine the type of metal-polymer bond and valence state of the metal in the polymeric chelates. The values of the magnetic moments show that the chelates are paramagnetic. All square planar complexes of  $\text{Ni}(\text{II})$  are diamagnetic, and octahedral complexes of  $\text{Ni}(\text{II})$  have magnetic moments in the range 3.1 to 3.16 BM [21]. The observed values, 2.10-2.91 BM, therefore suggest that  $\text{Ni}(\text{II})$  polymeric chelate may have a tetrahedral configuration [22]. Complexes of  $\text{Co}(\text{II})$  with a square planar geometry are reported to have magnetic moments in the range 2.1-2.9 BM [23] and with tetrahedral geometry in the range 4.1-4.9 BM [24]. Thus, from the values in the range 3.14-3.96 BM observed for  $\text{Co}(\text{II})$  chelates, a tetrahedral configuration may be concluded. In the case of  $\text{Cu}(\text{II})$  polymeric chelates, the observed magnetic moments, 1.62-1.64 BM, are slightly less than expected for a square planar configuration [25].

All the polymeric chelates can be partially decomposed with dilute hydrochloric acid ( $\text{pH} = 1.0$ ) and completely with concentrated hydrochloric acid. The original resin and the metal salts were recovered in quantitative yield.

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