



## Estimation of the dipole moments of the excited state of di(2-methyl-6-chlorophenyl)carbazone and its Co(II), Ni(II) and Zn(II) complexes from the effect of solvent on their ultraviolet absorption spectra

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*Abstract:* Di(2-methyl-6-chlorophenyl)carbazone (2M6CPC) and its Co(II), Ni(II) and Zn(II) complexes were synthesized and characterized by magnetic moment, and infrared and <sup>1</sup>H-NMR spectral measurements. The solvent effect in a series of polar and non-polar solvents of varying dielectric constants and refractive indices was estimated by recording the electronic spectra (S1 band) of the above compounds. The data was used to determine the magnitude and direction of the electric dipole moments in the first electronically excited state. The results indicate that the observed band systems in these compounds may be attributed to a  $\pi \rightarrow \pi^*$  transition.

*Keywords:* carbazole; electric dipole moments; dielectric constants;  $\pi \rightarrow \pi^*$  transition.

### INTRODUCTION

Transition metal complexation has played a vital role in the fields of biochemistry and medicine.<sup>1</sup> After work on the chelation of biologically active ligands,<sup>2</sup> it was thought justifiable to study the dipole moments of some adducts. A survey of the literature revealed that in addition to investigating the problems of electronic structure, bonding stereochemistry and stability constants of metal complexes with diphenyl carbazole (DPC)<sup>3</sup> and its derivatives, an increasing number of studies have been devoted to the dynamic and mechanism of the reaction of metal complexes and their heterocyclic nitrogen base adducts.<sup>4,5</sup> Through spectrophotometry, the synthesis and characterization of Co(II), Ni(II) and Zn(II) complexes with di(2-methyl-6-chlorophenyl)carbazone (2M6CPC) and also the electric dipole moments of these compounds in the first electronically excited

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state are reported here. The dipole moment is an important parameter<sup>6,7</sup> which gives an idea of the electronic structure of the molecule and is of prime importance in the understanding of molecular interactions.<sup>8</sup> The present studies were undertaken keeping these views in mind. From the study of the solvent effect on the ultraviolet absorption spectra of each of the above-cited compounds, it is possible to determine its electric dipole moment in its electronically excited state.<sup>9</sup> This so-determined parameter gives some insight into the electron distribution, reactivity, photochemical, reactions, *etc.*, of the solute molecule in its electronically excited state.<sup>10</sup> Apart from obtaining the permanent dipole moment, it is also possible from these studies to determine the shape parameters, yet another important parameter that gives knowledge about the shape of the cavity in which a solute molecule is supposed to lie.<sup>11</sup>

#### EXPERIMENTAL

Carbon tetrachloride, 2-propanol, dimethylformide, 1-butanol, chloroform, 1,4-dioxane and *n*-propanol used were of Fisher AR grade; the cobalt, nickel and zinc chloride used were also of Fisher AR grade.

Ultraviolet absorptions were recorded on Hitachi 150-20 UV-Vis spectrophotometer. Elemental analyses were realized on a Perkin-Elmer 240.C, H and N analyzer. IR and <sup>1</sup>H-NMR Spectra were recorded on Nicolet-170 FT-IR spectrometer and a VXR 300 S Varian spectrometer, respectively. The magnetic moments of the Co(II) and Ni(II) complexes were determined by the Gouy method. The metal estimation was performed by the EDTA titration method. The dielectric measurements were recorded with the aid of a Forbes Tinsley (FT) 6421 LCR Data Bridge at 10 kHz frequencies. The refractive indices of various dilute solutions (Sodium D line) were determined using an Abbe's refractometer.

The dielectric constants of the dilute solutions were measured in a suitably fabricated cell of usually small capacitance, where the accurate determination of small changes in the capacitance would be possible. This small change in capacitance can be measured with the help of Forbes Tinsley (FT) 6421 LCR Data B ridge at a frequency of 10 kHz. The dielectric sample holder consisted of two concentric brass cylinders kept in position with small strips (to achieve electric isolation) and their leads were coated with gold. This assembly was kept in a glass beaker so the dilute solution could be filled into the cell and the capacitance of the empty cell (air) would be of the order of a pF.

#### *Synthesis of the ligand di(2-methyl-6-chlorophenyl)carbazone (2M6CPC)*

The ligand was synthesized by the method described earlier.<sup>12,13</sup> Briefly, di(2-methyl-6-chlorophenyl)carbazone (2M6CPC) was prepared by heating a mixture of 2-methyl-6-chlorophenylhydrazine and urea (2:1) at 155–160 °C for about 3 h. The so-obtained crude carbazole was crystallized from ethanol. About 1 g of the carbazole was dissolved in a mixture of 60 mL glacial acetic acid, 20 mL of 1.0 N sulfuric acid and 2–3 drops of 10 % ferric alum, and oxidized by the addition of 20 ml of aqueous 0.060 M potassium persulfate ( $K_2S_2O_8$ ) added drop wise under vigorous stirring over about 30 min. The resulting carbazole was extracted with diethyl ether, washed several times with water, dried and purified by column chromatography using silica gel (60–120 mesh) column. A mixture of methanol and chloroform (1:4) was used as the eluent. The structure of the ligand is as given in Fig. 1.



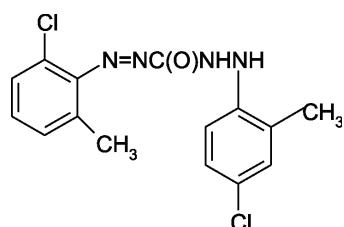
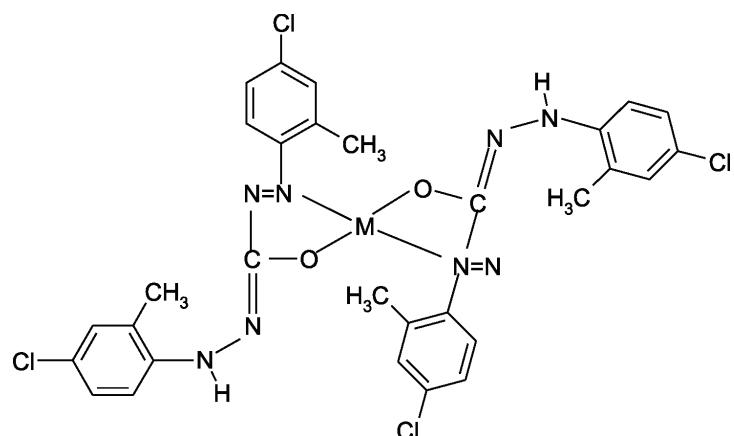


Fig. 1. Structure of the ligand.

#### *Synthesis of the complexes*

Cobalt chloride (1.0 g) was dissolved in acetate buffer (pH 6.2) and added drop wise to a 0.010 M EtOH solution (12 ml) of 2M6CPC at room temperature. The mixture was stirred and the resulting precipitate was collected under suction and washed several times with H<sub>2</sub>O. The complex was dried over P<sub>2</sub>O<sub>5</sub> under vacuum at room temperature and purified by the Soxhlet method,<sup>14</sup> using a 1:1 Et<sub>2</sub>O:petroleum ether mixture as the solvent (3 h). The pure complex was obtained as a shining crystalline powder in the Soxhlet tube. The Ni(II) and Zn(II) complexes were similarly prepared using nickel and zinc chloride, respectively. The structures of the complexes are as given in Fig. 2.



M= Co (II), Ni(II) or Zn(II)

Fig. 2. Proposed structure of the adduct.

#### RESULTS AND DISCUSSION

##### *Physico-chemical methods*

The elemental analyses of the ligand and the complexes together with the magnetic data are reported in Table I. The C, H, N and metal analyses confirm that the stoichiometry of the complexes is 1:2 metal to ligand. The observed magnetic moment for the Co(II) complex was 2.57  $\mu_B$ . The low value of the observed magnetic moment is attributed to the orbital contribution, as it is higher in the case of square planar Co(II) complexes.<sup>15</sup> The subnormal magnetic moment for

the Ni(II) complex requires more explanation, since the spin-only value ( $2.83 \mu_B$ ) is expected for tetrahedral and octahedral geometries.

TABLE I. Analytical and magnetic data of the compounds

Compound	Molecular formula	Elemental analysis (found (calcd.), %)				$\mu_{\text{eff}}$	$\mu_B$
		C	H	N	M		
[D2M 6CPC]	$\text{C}_{15}\text{H}_{11}\text{N}_4\text{OC}_{12}$	53.34 (53.43)	4.10 (4.18)	16.41 (16.62)	—	—	—
[Co(D2M6CPC) <sub>2</sub> ]	$\text{Co}(\text{C}_{30}\text{H}_{26}\text{N}_8\text{O}_2\text{C}_{14})$	49.14 (49.27)	3.43 (3.57)	15.22 (15.32)	8.00 (8.06)	2.57	
[Ni(D2M6CPC) <sub>2</sub> ]	$\text{Ni}(\text{C}_{30}\text{H}_{28}\text{N}_8\text{O}_2\text{C}_{14})$	49.24 (49.28)	3.50 (3.59)	15.40 (15.30)	8.06 (8.03)	2.31	
[Zn(D2M6CPC) <sub>2</sub> ]	$\text{Zn}(\text{C}_{30}\text{H}_{25}\text{N}_8\text{O}_2\text{C}_{14})$	48.80 (48.84)	3.50 (3.55)	15.16 (15.19)	8.80 (8.86)	dia	

#### Infrared spectra

The IR spectra of the ligand and its complexes were recorded in the 4000–400  $\text{cm}^{-1}$  range. The ligand showed bands at 3301 and 3150  $\text{cm}^{-1}$  attributed to intra and intermolecular bonded  $\nu_{\text{N}-\text{H}}$  vibrations. The band at 1684  $\text{cm}^{-1}$  is assigned to  $\nu_{\text{C}=\text{O}}$  stretching. The disappearance of this  $\nu_{\text{C}=\text{O}}$  stretching band in the spectra of the complexes indicated that an oxygen atom of the ligand was involved in the coordination with the metal through the enolic form. This was further confirmed by the appearance of a band in the region 1572–1609  $\text{cm}^{-1}$  due to  $\nu_{\text{C}=\text{N}}$  stretching in the spectra of the complexes.<sup>16</sup>

#### <sup>1</sup>H-NMR spectra

The <sup>1</sup>H-NMR spectrum of the ligand was recorded in chloroform using TMS as the internal reference. Singlets were observed at  $\delta$  2.35 and 2.76 ppm due to –CH<sub>3</sub> protons. The broad peaks observed at  $\delta$  6.13 and 8.20 are due to aromatic –NH and amide –NH groups, respectively. The multiplets observed in the region of  $\delta$  6.75–7.70 may be attributed to aromatic hydrogen atoms. For the diamagnetic Zn(II) complex, it was observed that the broad peak of the amide –NH had disappeared, indicating that the azomethine atom is involved in the coordination to the central metal atom via deprotonation. Based on the analytical and spectral data given in Table II, the structures of the ligand and the complexes are assigned as shown in Figs. 1 and 2, respectively.

#### Methodology

For the evaluation of the ground state and excited state dipole moment, the UV absorption spectra (for the S1 band) of a single particular weight fraction (concentration) of each of the pure samples, 2M6CPC and its Co(II), Ni(II) and Zn(II) complexes, were recorded on a Hitachi 150-20 UV-Vis spectrophotometer

with a cell path length of 1 cm in various polar and non-polar solvents (concentration 0.010–0.030 g ml<sup>-1</sup>). The data is presented in Table III. The permanent dipole moments in the excited states ( $\mu_e$ ) and the radius of the cavity in which solute molecules are supposed to lie were obtained using a method reported in the literature and is given below.

TABLE II. IR and <sup>1</sup>H-NMR spectral data for the compound (IR frequencies in cm<sup>-1</sup>)

Compound	$\nu_{\text{N-H}}$	$\nu_{\text{C=O}}$	$\nu_{\text{C=N}}$	$\nu_{\text{C=C}}$ aromatic	$\nu_{\text{C-O}}$	$\nu_{\text{N-H}}$ bend	<sup>1</sup> H-NMR
D2M6CPC	3301 3150	1684	—	1486	—	763	2.35, 2.76(S-CH <sub>3</sub> ); 6.13 (br-NHAr); 8.20 (br-NH, amide); 6.75–7.70 (m, Ar-H)
Co(D2M6CPC) <sub>2</sub>	3380	—	1609	1498	1158	854	2.63 (S-CH <sub>3</sub> ); 6.42 (br-NH-Ar), 7.24–7.64 (m, Ar-H)
Ni(D2M6CPC) <sub>2</sub>	3005	—	1573	1505	1011	832	2.64 (S-CH <sub>3</sub> ), 6.38 (br-NH-Ar), 7.10–7.80 (m, Ar-H)
Zn(D2M6CPC) <sub>2</sub>	3320	—	1572	1492	1164	828	2.72 (S-CH <sub>3</sub> ), 6.60 (br-NH-Ar), 7.20–7.84 (m, Ar-H)

Following Suppan and Tsiamis,<sup>14</sup> the change in the permanent dipole moment for a series of solvents of different static dielectric constants ( $\epsilon$ ) but similar refractive indices ( $n$ ) are related to the observed energy shift  $\Delta V_{a-b}$  between solvents “a” and “b” by:

$$-\Delta V_{a-b} = \mu_g \Delta \mu_g - e/hc a_g^3 (\Delta(f(\epsilon)) - f(n^2))_{a-b} + \mu^2 e - \mu_g^2 \Delta(n^2)_{a-b} - e/hc a_g^3 \quad (1)$$

where  $\mu_g$  is the permanent dipole moment in the ground state,  $h$  is the Planck's constant,  $c$  is the velocity of light,  $a_g$  is the radius of the cavity in which the solute molecule is supposed to lie and  $f(\epsilon)$  and  $f(n^2)$  are the polarity and polarizability functions, respectively, defined by:

$$f(\epsilon) = 2(\epsilon - 1) / (2\epsilon - 1) \text{ and } f(n^2) = 2(n^2 - 1) / (2n^2 - 1)$$

Recently, a method was proposed by Ayachit *et al.*<sup>17</sup> to determine  $M_e$  by expressing  $Ef(i)$  in the form:

$$x/C1 + y/C2 = 1 \quad (2)$$

which is an equation of a straight line with intercepts on either axes.

By plotting a graph:

$$x = (\Delta(f(\epsilon)) - f(n^2))_{a-b} / -\Delta V_{a-b} \text{ vs. } y = \Delta f(n^2)_{a-b} / -\Delta V_{a-b}$$

the intercepts:

$$C1 = hca_g^3 / (\mu_g \Delta \mu_g - e) \text{ and } C2 = hca_g^3 / (\mu^2 e - \mu_g^2)$$

on the  $x$  and  $y$  axes, respectively, are determined and the magnitude and direction of them are obtained. The required data were obtained by measuring the static per-



TABLE III. Dielectric constants and refractive indices of the compounds in benzene at 25 °C ( $\rho_1 = 0.874 \text{ g cm}^{-3}$ ;  $\varepsilon_1 = 2.278$ ;  $n_1 = 1.5015$ )

Compound	Molecular weight $M_{1,2}$	Weight fraction $w_2 \times 10^3$	Dielectric constant $\varepsilon_{12}$	$\varepsilon_{12}-\varepsilon_1/w_2$	Refractive index $n_{12}$	Intercepts			Calculated $\mu\text{g / D}$	
						$n_{12}-n_1/w_2$	$\Delta'$	$\Delta''$	Eq. (3)	Eq. (4)
D2M6CPC	337.09	0.5991	2.4033	209.147	1.5040	12.5417	196.0	12.2	183.80	23.96
		1.1560	2.4650	161.76	1.5050	9.1027				24.37
		1.7100	2.5205	141.8128	1.5055	7.0339				
		2.3302	2.5630	122.3070	1.5060	5.8084				
		2.8943	2.5900	108.4890	1.5063	4.9812				
		0.5564	2.3926	205.9264	1.5050	18.9122	152.00	14.40	137.60	30.53
Co[D2M6CPC] <sub>2</sub>	731.350	0.5964	2.42037	125.2815	1.5055	10.5843				31.60
		1.1364	2.4765	115.7704	1.5060	7.8932				
		2.2991	2.4927	93.3843	1.5065	6.5416				
		2.8966	2.5159	82.1307	1.5070	5.7124				
		0.5957	2.3510	122.5440	1.5035	10.0889	139.00	9.00	130.00	29.67
		1.2715	2.4069	101.3763	1.5040	5.9093				30.21
Ni(D2M6CPC) <sub>2</sub>	731.11	1.7561	2.4329	88.2068	1.5045	5.1352				
		2.3302	2.4452	71.7534	1.5050	4.5158				
		2.8966	2.4576	62.0037	1.5055	4.1524				
		0.5968	2.4165	232.0710	1.5045	15.1105	188.00	10.80	177.20	34.79
		1.1791	2.4365	134.1701	1.5050	8.9243				
		1.7446	2.4460	96.2971	1.5060	7.7575				
Zn[D2M6CPC] <sub>2</sub>	737.75	2.329	2.4633	79.5620	1.5070	7.1046				35.30
		2.8966	2.4788	69.3226	1.5080	6.7533				



mitivities of the solvents and various dilute solutions (in benzene only for this purpose) at 10 kHz with the help of FT 6421, LCR Data Bridge. The estimated values of  $\mu_g$  using the modified Guggenheim equation<sup>18</sup> were found to be accurate up to the second decimal place. The equations used are as follows:

$$\mu_g = 0.0128(3/(E_1 + 2)2M_{r,2} / \rho_1 T \Delta)^{0.5} \quad (3)$$

where

$$\begin{aligned} \Delta &= \Delta' - \Delta'' = ((E_{12} - E_1) / w_2) w_2^0 - (n_{12}^2 - n_1^2/w_2) w_2 \\ \mu_g &= 0.0128(3/(e_1 + 2)^2 M_{r,2} / \rho_1 T 0.97((E_{12} - E_1) / w_2)) \end{aligned} \quad (4)$$

The quantities  $n$ ,  $\rho$ ,  $M_r$ ,  $w$  and  $T$  involved in Eqs. (3) and (4) are the refractive index, density, molecular weight, weight fraction and absolute temperature, respectively. The subscripts 1, 2, and 12 refer to the solvent, solute, and solution, respectively. The data of dielectric constants, the refractive indices and hence the ground state dipole moments for the compounds are presented in Table IV. The ultraviolet spectral data of the compounds in different solvents are summarized in Table V. The values of the  $x$  and  $y$  intercepts are presented in Table VI.

TABLE IV. The electronic spectral data of the compounds in different solvents

Solvent	D2M6CPC		Co(D2M6CPC) <sub>2</sub>		Ni(D2M6CPC) <sub>2</sub>		Zn[D2M6CPC] <sub>2</sub>	
	$\nu_{\max}$ cm <sup>-1</sup>	$\nu$ cm <sup>-1</sup>						
Cyclohexane	44247	Ref	44404	Ref	44563	Ref	44722	Ref
Carbon tetrachloride	33738	10509	34340	10064	34819	9744	34530	10192
Benzene	34722	9525	36023	8381	34843	9720	34867	9855
2-Propanol	46296	2049	35460	8944	45955	1392	36040	1318
Dimethyl formamide	34698	9549	35511	8893	36927	7636	35511	9211
1-Butanol	43706	541	43029	1375	42589	1974	40916	3806
Chloroform	40916	3331	40716	3688	40983	3580	40849	3873
1,4-dioxane	32051	12196	31766	12638	31806	12757	31928	12799
1-propanol	47348	3101	47258	2854	46992	2429	47080	2358

The magnitude and directions of the dipole moments for the first excited state together with the corresponding ground state dipole moment values are given in Table VI. It may be observed that the excited state dipole moment values are rather higher compared to the values obtained using the Guggenheim Equation for ground state ones C, as entered in the third column of Table VI. Such large values are reported in the literature for some polymers both in polar and non-polar solvents. The method of calculation (vector addition of group moments) has its own limitation of not accounting for the possible inductive/mesmeric/hydrogen bonding effects in these systems. Under these circumstances, if it is assumed that these values would not improve much when bonding effects are also



taken into account; the presently observed dipole moment values remain higher. In the light of these considerations, the observed values of the dipole moments may be considered as inductive and hence the present ligand and complexes may be associated with rather large values of dipole moments, which in turn may be taken as suggestive of their structure.<sup>19</sup>

TABLE V. Values of the  $x$  and  $y$  intercepts and the molecular radius ( $a_g$ ) of the compounds

Solvent	D2M6CPC		Co(D2M6CPC) <sub>2</sub>		Ni(D2M6CPC) <sub>2</sub>		Zn[D2M6CPC] <sub>2</sub>	
	$x \times 10^5$	$y \times 10^5$	$x \times 10^5$	$y \times 10^5$	$x \times 10^5$	$y \times 10^5$	$x \times 10^5$	$y \times 10^5$
Carbon tetrachloride	0.2326	0.0207	0.2429	0.2162	0.2509	0.2230	0.2398	0.2135
Benzene	0.0747	0.0495	0.0849	0.5634	0.0732	0.4850	0.0722	0.4791
1-Propanol	27.14	-0.1655	6.218	-0.3793	39.95	-2.437	42.20	-2.574
Dimethyl formamide	6.180	0.0031	6.636	0.0336	7.729	0.3910	6.4074	0.0324
1-Butanol	98.53	-0.3460	38.77	-1.361	27.01	-0.9480	14.01	-0.4918
Chloroform	8.966	0.0394	8.099	0.3560	0.8343	0.3660	7.712	0.3390
1,4-Dioxane	0.3585	-0.0021	0.3459	-0.0211	0.3430	-0.0209	0.3417	-0.0208
1-Propanol	17.88	-0.0939	19.43	-1.021	22.82	-1.199	23.51	-1.236
$C_1 \times 10^5$		11.5		7.10		12.0		6.60
$C_2 \times 10^5$		0.128		0.570		1.12		0.500
$a_g / \text{\AA}$		3.955		4.978		4.985		5.006

The dipole moment values of the excited states are expected to be greater than their ground state values. In the present investigation, the excited state dipole moment values are certainly greater than the ground state ones. Based on these observations, it may be presumed that the observed transitions belong to  $\pi \rightarrow \pi^*$  transitions.

TABLE VI. Ground state and excited state dipole moments

Compound	Equation	$\mu_g / \text{D}$	$\mu_e / \text{D}$	$\theta / {}^\circ$
D2M6CPC	(3)	23.9566	39.1620	60 08' 29"
	(4)	24.3650	39.4137	59 32' 22"
[Co(D2M6CPC) <sub>2</sub> ]	(3)	30.5318	72.3362	74 35' 22"
	(3)	29.6718	55.4710	65 31' 31"
Zn[(D2M6CPC) <sub>2</sub> ]	(4)	30.2179	55.7651	65 09' 07"
	(3)	34.7989	78.7067	72 10' 00"
	(4)	35.3019	78.9304	71 50' 06"

### CONCLUSIONS

An interesting dynamic and mechanistic way of viewing the reaction involving the metal complexes was traced in the present study by application of spectrophotometry. The electric dipole moments of the synthesized compounds in



their first electronic excited state were determined. An attempt at predicting the structure and geometry of the complexes was made. All these observations put together lead us to propose the structures shown in Fig. 2.

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#### ИЗВОД

#### ПРОРАЧУН ДИПОЛНИХ МОМЕНТА ЕКСЦИТОВАНОГ СТАЊА ДИ(2-МЕТИЛ-6-ДИ-ХЛОРОФЕНИЛ)КАРБАЗОНА И ЊЕГОВИХ Co(II), Ni(II) И Zn(II) КОМПЛЕКСА НА ОСНОВУ УТИЦАЈА РАСТВАРАЧА НА ЊИХОВЕ УЛТРАЉУБИЧАСТЕ АПСОРПЦИОНЕ СПЕКТРЕ

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Добијен је ди(2-метил-6-хлорофенил)карбазон (2M6CPC) и његови Co(II), Ni (II) и Zn(II) комплекси који су окарактерисани магнетним моментом, инфрацрвеним спектрима и  $^1\text{H-NMR}$  спектралним мерењима. Утицај растварача у серији поларних и неполарних растварача променљивих диелектричних константи и индекса рефракције су прорачунати снимањем електронских спектара (S1 траке) поменутих једињења. Подаци су искоришћени за одређивање величине и правца електричног диполног момента у првом електронском ексцитованом стању. Резултати указују да се примећена трака у овим једињењима може приписати  $\pi \rightarrow \pi^*$  прелазу.

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