

THE LUMINESCENT PROPERTIES OF THE COMPLEX COMPOUNDS OF MANGANESE (II) CHLORIDE WITH 4-BENZYLPIRIDINE HYDRO- CHLORIDE

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The photoluminescent properties were investigated of two complex compounds of manganese (II) chloride with 4-benzylpyridine hydrochloride, viz.: $C_{12}H_{12}N MnCl_3$ (BPMC) exhibiting a red luminescence, and $(C_{12}H_{12}N)_2MnCl_4$ (DBPMC) displaying an orange luminescence. The results obtained theoretically using the model of parabolic configurational curves were checked on DBPMC, and their parameters were calculated. For BPMC, where probably an energy transfer from an excited manganese ion to its neighbours occurs, the experimental results are not in agreement with theoretical ones predicted by the model of configurational curves.

1. Introduction

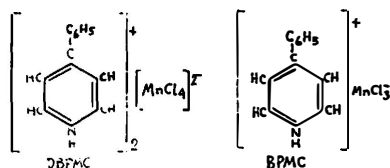
Manganese (II) chloride forms with 4-benzylpyridine hydrochloride two complex compounds with different photoluminescent properties depending on the molecular ratio of pyridine base to manganese (II) chloride. The compound in which the ratio of 4-benzylpyridine to manganese (II) chloride is 2 : 1, $(C_{12}H_{12}N)_2MnCl_4$ (DBPMC), exhibits an orange luminescence, while the compound in which this ratio is 1 : 1, $C_{12}H_{12}N MnCl_3$ (BPMC), displays a red luminescence. The photoluminescence of these compounds exists only in their solid state, because the complex decomposes when it is dissolved in water and ethanol.

We continue our systematic study of the luminescence of the manganese amine complexes¹⁻⁷). We have chosen here a very big amine, 4-benzylpyridine $C_{12}H_{11}N$, in order to compare the results with those obtained with smaller amines: pyridine^{1,5,6}), benzyltrimethylamine and 2-chloropyridine³).

2. Experimental

The manganese complex compounds DBPMC and BPMC were synthesized by taking equivalent quantities of 4-benzylpyridine and manganese (II) chloride dissolved in ethanol in the presence of an excess of hydrochloric acid. On a slow evaporation of this solution on a water bath crystals are formed. Compounds in very pure state are obtained on a repeated recrystallization from ethanolic solution. Both compounds are very stable, and no changes were observed.

On the basis of the results of an analysis it is assumed that the compounds investigated may be assigned the following structure:



The excitation and luminescence spectra were recorded on a spectrophotometer SAFAS 1700.

3. Results and discussion

Excitation of luminescence

The compounds investigated are photoluminescent. The shape of the luminescence spectrum is independent of the wavelength of the excitation radiation, but the intensity of luminescence depends on it. The excitation radiation is supplied by a xenon lamp followed by grating. The light emitted by thus irradiated sample is analyzed by means of a second grating and measured with a photomultiplier. The sample is in a liquid nitrogen cryostat at a temperature of 84 K. We fix the grating analyzing the light emitted at the wavelength of the peak of the luminescence spectrum (595 nm for DBPMC, and 658 nm for BPMC). We make the excitation grating revolve. The maxima of luminescence intensity are obtained with the excitation radiations whose wavelengths are given in Table 1 (with an uncertainty of 2 nm).

TABLE 1

DBPMC	300	365	380 (s)	430 (s)	470	500 (s)	
BPMC	295	330 (s)	360 (s)	370	425 band	470	550

Excitation wavelengths in nm. $T = 84$ K — (s) for shoulder

These peaks displace slightly with temperature. They probably correspond, like for other complexes of manganese amine, to absorption wavelengths. Since we do not know the crystalline structure of these complexes, we do not give any level assignments. BPMC has levels close to those of the compound of manganese (II) chloride with benzyltrimethylammonium chloride (the molecular ratio 1 : 1), BTMA MnCl_3 , and the compound of manganese (II) chloride with 2-chloropyridine hydrochloride (the molecular ratio 1 : 1), $\text{C}_5\text{H}_4\text{ClNH MnCl}_3$ ³⁾.

Luminescence spectra

We choose for these two compounds the excitation at 4500 nm which gives the maximum of the intensity of the emitted light. We make the network analyzing the emitted light revolve, and record the luminescence spectra: I_λ — luminescence intensity per unit interval of wavelength as a function of the wavelength λ . The results obtained at about 80 K and at about 290 K for the two compounds are given in Fig. 1. I_λ is in arbitrary units and is different for DBPMC and BPMC. The

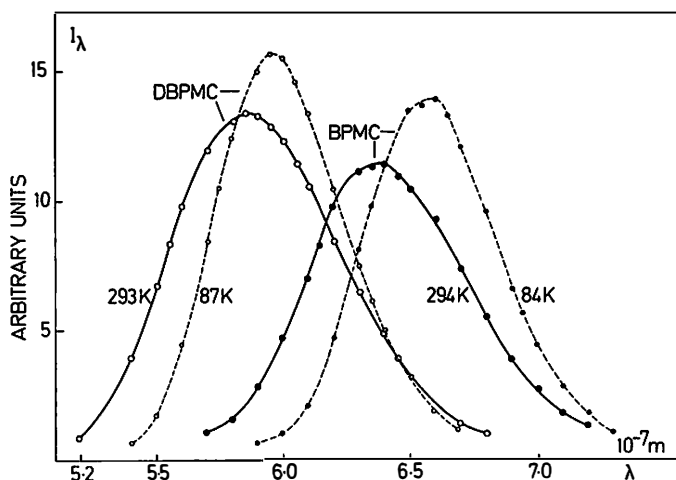


Fig. 1. Luminescence spectra of the compounds investigated.

total intensity of luminescence depends slightly on temperature. As the temperature increases the curve becomes enlarged and the wavelength of its peak λ'_M diminishes: in BPMC λ'_M decreases from 658 nm at 84 K to 637.5 nm at 294 K, and in DBPMC λ'_M decreases from 596 nm at 87 K to 587 nm at 293 K.

Comparison of the experimental spectra of DPBMC with those obtained theoretically by means of the model of parabolic configurational curves with one configurational coordinate $r^{8,1,5}$.

The luminescence center is the Mn^{++} ion. The emission occurs when an electron falls from the lowest excited level, ${}^4T_1 ({}^4G)$, to the ground level, 6A_1 .

The ground level has the potential energy: $U_0 = \frac{1}{2} k_g r^2$

The excited level has the potential energy:

$$U_e = \frac{1}{2} k_e (r - R_0)^2 + U_0.$$

R_0 is the value of the configurational coordinate r corresponding to the minimum of U . The total energy is:

$$E_n^e = \left(n + \frac{1}{2}\right) h \nu_e + U_0 \qquad \nu_e = \frac{1}{2\pi} \sqrt{\frac{k_e}{M}},$$

where M is the mass of vibrating ions, and the corresponding wave function is $\Psi_n^e(r - R_0)$. The theory gives a curve $G\left(\frac{1}{\lambda}\right)$ proportional to $I_\lambda \lambda^6$ having the following properties:

- The diameter of the curve $\left(\frac{1}{\lambda}\right)$ is a straight line with slope m .
- The curve $\ln G\left(\frac{1}{\lambda_1}\right) = \ln G\left(\frac{1}{\lambda_2}\right)$ as a function of $\left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2}\right)^2$ is a straight line with slope a ($\frac{1}{\lambda_1}$ and $\frac{1}{\lambda_2}$ are two wavenumbers corresponding to the same value of G).
- $G\left(\frac{1}{\lambda}\right) = G_M \exp\left\{\frac{m^2}{16a} \left[\sqrt{1 + \frac{16a}{m} \left(\frac{1}{\lambda} - \frac{1}{\lambda_M}\right)} - 1\right]^2\right\}$, (1) [Ref. 3,6]

where G_M is the maximum value of G obtained for the wavelength λ_M .

- The half-width L of G varies as a function of temperature T :

$$L = L_0 \sqrt{\coth \frac{h \nu_e}{2 k T}} \qquad (2) \text{ [Ref. 8]}$$

$$\frac{1}{L^2} = A \text{th} \frac{h \nu_e}{2 k T} + B, \qquad (3) \text{ [Ref. 5]}$$

where L , A and B are constants.

Shape of spectra

Fig 2 shows the curves $\ln G\left(\frac{1}{\lambda}\right)$ at 293 K and at 87 K, their diameter and the

corresponding curves f . G is in arbitrary units. We observe that the diameter of the curve $\ln G \left(\frac{1}{\lambda} \right)$ is a straight line of the equation:

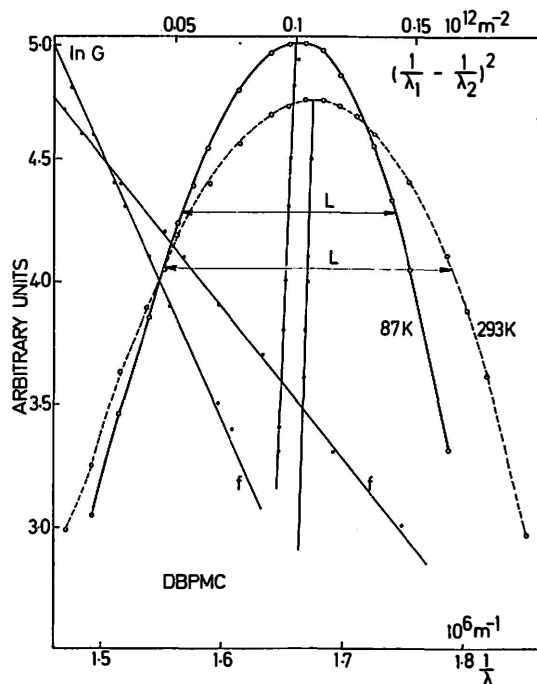


Fig. 2. curves for the DBPMC complex. Experimental dots of $\ln G \left(\frac{1}{\lambda} \right)$ at 293 K and at 87 K. Curve f represents $\ln G = f \left(\frac{1}{\lambda_2} - \frac{1}{\lambda_1} \right)^2$.

$$\ln G = \ln G_M + m \left\{ \frac{1}{2} \left(\frac{1}{\lambda_1} + \frac{1}{\lambda_2} \right) - \frac{1}{\lambda_M} \right\}.$$

The curve f is a straight line of the equation

$$\ln G = \ln G_M + a \left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2} \right)^2.$$

Hence the shape of the curve is that of Eq. (1).

At $T = 293 \text{ K} : \frac{1}{\lambda_M} = 1.674 \cdot 10^6 \text{ m}^{-1}$; $m = 165 \cdot 10^{-6} \text{ m}$;

$$a = -12.3 \cdot 10^{-12} \text{ m}^2 \frac{16a}{\text{m}} = -1.193 \cdot 10^{-6} \text{ m};$$

$$\frac{\text{m}^2}{16a} = -138.3; L = 0.239 \cdot 10^6 \text{ m}^{-1}.$$

At $T = 87 \text{ K} : \frac{1}{\lambda_M} = 1 \cdot 66 \cdot 10^6 \text{ m}^{-1}; m = 117 \cdot 10^{-6} \text{ m};$

$$a = -22 \cdot 10^{-12} \text{ m}^2 \frac{16a}{\text{m}} = -3 \cdot 009 \cdot 10^{-6} \text{ m};$$

$$\frac{\text{m}^2}{16a} = -3.009; L = 0.176 \cdot 10^6 \text{ m}.$$

A study of the transformations of $G \left(\frac{1}{\lambda} \right)$ as function of temperature T

Fig. 3 gives the values of the wavenumber $1/\lambda_M$ of the maximum of G , and those of the width L at different temperatures. The uncertainties are:

$$\Delta \left(\frac{1}{\lambda_M} \right) = 0.002 \mu\text{m}^{-1}; \Delta L = 0.005 \mu\text{m}^{-1}; \Delta T = 1 \text{ K}.$$

λ_M depends slightly on temperature. λ_M is a slowly decreasing function of T :

$$(\lambda_M = 602.4 \text{ nm at } 87 \text{ K}; \lambda_M = 598.1 \text{ nm at } 293 \text{ K}).$$

This decrease is less pronounced than that of λ'_M for the maximum of I_λ .

The curve widens. We have compared the experimental widths with theoretical formulae:

Equation 2 gives rather good results below 300 K with

$$L = 0.174 \cdot 10^6 \sqrt{\coth \frac{h\nu_e}{2kT}} \quad L = 0.174 \cdot 10^6 \sqrt{\coth \frac{170}{T}} \Rightarrow \frac{h\nu_e}{2k} = 170.$$

Equation 3 gives better results with

$$\frac{1}{L^2} = 30 \cdot 10^{-12} \text{ th} \frac{140}{T} + 4.3 \Rightarrow \frac{h\nu_e}{2k} = 140$$

(which corresponds to 0 K at $L = 0.171 \cdot 10^6 \text{ m}^{-1}$).

For this compound $\frac{\alpha}{m}$ is not constant.

Calculation of the parameters of configurational curves

We shall not make use of the measurement of m which is very unprecise, but of:

$$\frac{h \nu_e}{2k} \text{ measured by band widths}$$

$$\frac{hc}{\lambda_{abs}} - \frac{hc}{\lambda_M} = \frac{1}{2} (k_e + k_g) R_0^2,$$

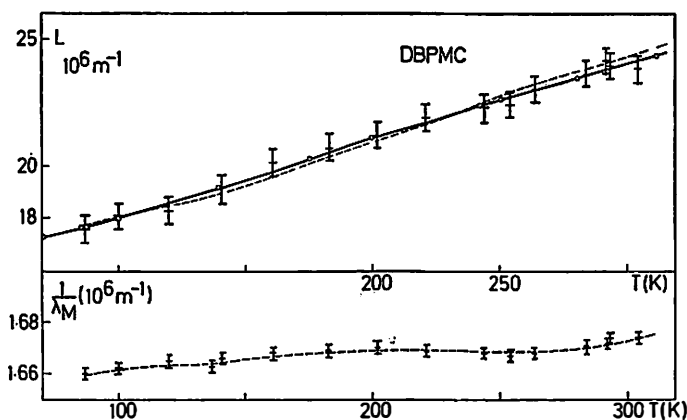


Fig. 3. $\frac{1}{\lambda_M}$ — the wave number of the maximum of G as a function of temperature. The half-width L of G ($\frac{1}{\lambda}$) for the DBPMC compound. Experimental dots. Dotted line:

$$L = 0.174 \cdot 10^6 \sqrt{\coth \frac{170}{T}}. \text{ Solid line: } L = \frac{10^6}{\sqrt{30 \operatorname{th} \frac{140}{T} + 4.3}}$$

where λ_{abs} is the wavelength of the absorption of the lowest energy (500 nm)

with either Eq. (2)⁶⁾: $\frac{1}{L_0^2} = \frac{hc^2 \nu_e}{16\pi^2 MR_0^2 \nu_g^4 \ln 2}$

or with Eq. (3)⁵⁾: $A = \frac{hc^2}{4 k_g^2 R_0^2 \ln 2} \frac{k_e}{h \nu_e} = \frac{hc^2 \nu_e}{16\pi^2 MR_0^2 \nu_g^4 \ln 2}$.

1) Calculations with the formula of the width of Eq. (2):

$$\frac{h \nu_e}{2k} = 170 \Rightarrow \boxed{\nu_e = 7.02 \cdot 10^{12} \text{ Hz or } \frac{\nu_e}{c} = 23\,600 \text{ m}^{-1}}$$

$$\left(\frac{1}{\lambda_{abs}} - \frac{1}{\lambda_m} \right) \frac{1}{L_0^2} = \frac{(\nu_e^2 + \nu_g^2) c \nu_e}{8 \nu_g^4 \ln 2}.$$

With $\frac{1}{\lambda_M} = 1.66 \mu^{-1}$ (low temperature), we find numerically in S. I.:

$$11.23 \cdot 10^{-6} = \frac{1 + \left(\frac{\nu_g}{\nu_e}\right)^2}{\left(\frac{\nu_g}{\nu_e}\right)^4} \frac{1}{8 \ln 2 \frac{\nu_e}{c}}$$

$\frac{\nu_g}{\nu_e} = 1.11$	\Rightarrow	$\nu_e = 7.86 \cdot 10^{12} \text{ Hz or } \frac{\nu_g}{c} = 26\,200 \text{ m}^{-1}.$
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This result is that of the frequency of vibration $\nu_1 (a_1)$ of a MnCl_4^- ion^{4,9)}:

$$\tilde{\nu}_1 (a_1) = 25\,900 \text{ m}^{-1}$$

$$\tilde{\nu}_1 (a_1) = 25\,800 \text{ m}^{-1}.$$

Hence we shall make further calculations by choosing for M the mass of 4 Cl:
 $M = 2.359 \cdot 10^{-25} \text{ kg}$

$$\frac{1}{\lambda_{\text{obs}}} - \frac{1}{\lambda_M} = \frac{2\pi^2 MR_0^2}{hc} (\nu_e^2 + \nu_g^2)$$

$$MR_0^2 = 3.059 \cdot 10^{-47}$$

$R_0 = 1.14 \cdot 10^{-11} \text{ m}$

$$\left. \begin{aligned} k_e &= 4\pi^2 M \nu_e^2 = 467 \text{ Jm}^{-2} \\ k_g &= 4\pi^2 M \nu_g^2 = 575 \text{ Jm}^{-2} \end{aligned} \right\} \left\| \begin{aligned} U_0 &= \frac{hc}{\lambda_M} + \frac{1}{2} k_g R_0^2 \\ U_0 &= 3.673 \cdot 10^{-19} \text{ J.} \end{aligned} \right.$$

Let us calculate the coordinates of the intersection point of the configurational curves:

$$\begin{aligned} \frac{1}{2} k_g R_1^2 &= \frac{1}{2} k_e (R_1 - R_0)^2 + U_0 \\ R_1 &= 3.68 \cdot 10^{-11} \text{ m} \end{aligned}$$

$$\mathcal{W} = \frac{1}{2} k (R_{1e} - R_0)^2 = 1.505 \cdot 10^{-13} \text{ J.}$$

Since \mathcal{W} is very much higher than kT , there is no thermal extinction.

2) Calculation with the width formula given by H. Payen de la Garandrie (Eq. (3)):

$$\frac{h \nu_e}{2k} = 140 \Rightarrow \boxed{\nu_e = 5.83 \cdot 10^{12} \text{ Hz or } \frac{\nu_e}{c} = 19\,440 \text{ m}^{-1}}$$

$$\left(\frac{1}{\lambda_{abs}} - \frac{1}{\lambda_M} \right) A = \frac{1 + \left(\frac{\nu_g}{\nu_e} \right)^2}{\left(\frac{\nu_g}{\nu_e} \right)^4} \frac{1}{8 \ln 2 \frac{\nu_e}{c}}$$

$$A = 30 \cdot 10^{-12} \text{ m}^2 \quad \boxed{\frac{\nu_g}{\nu_e} = 1.229} \Rightarrow \boxed{\begin{aligned} \nu_g &= 7.17 \cdot 10^{12} \text{ Hz or} \\ \frac{\nu_g}{c} &= 23\,900 \text{ m}^{-1}. \end{aligned}}$$

Here the results are different from the frequencies of vibration of MnCl_4^{--} . However, we continue the calculations, assuming M to be the mass of 4 Cl.

$$MR_0^2 = 4.01 \cdot 10^{-47}$$

$$U_0 = 37 \cdot 10^{-19} \text{ J}$$

$$\boxed{R_0 = 1.3 \cdot 10^{-11} \text{ m}}$$

$$R_1 = 491 \cdot 10^{-11} \text{ m}$$

$$k_e = 317 \text{ Jm}^{-2}$$

$$W = 206 \cdot 10^{-19} \text{ J}$$

$$k_g = 478.5 \text{ Jm}^{-2}.$$

We want to point out that it is difficult to obtain precisely the coordinates of configurational curves. Since semi-classical theories take only a normal mode of vibration into consideration, they do not comprehend all the phenomena.

Comparison with neighbouring products

Table 2 also shows data on the compound of manganese (II) chloride with pyridine hydrochloride in which the ratio of pyridine base to manganese (II) is 2 : 1, $(\text{C}_5\text{H}_5\text{NH})_2\text{MnCl}_4$, and on the compound of manganese (II) chloride with benzyltrimethylammonium chloride in which the ratio of this quaternary base to manganese (II) is 2 : 1, $(\text{BTMA})_2\text{MnCl}_4$.

The DBPMC complex absorbs and emits the light of larger wavelength than the neighbouring products. Hence the crystal field is here stronger.

A study of the spectra of BPMC

Shape of the spectra

Fig. 4 shows the curves $\ln G \left(\frac{1}{\lambda} \right)$ at 294 K and at 84 K, their diameter and the corresponding curves f .

TABLE 2

	$\frac{1}{\lambda_{abs}} \cdot 4T_1 (4G)$ at about 80 K	$\frac{1}{\lambda_M'} (I\lambda)$ at about 80 K	$\frac{1}{\lambda_M} \left(G \left(\frac{1}{\lambda} \right) \right)$ at about 80 K	$\frac{\nu_e}{c}$	References
$(C_5H_5NH)_2 MnCl_4$	$2.13 \cdot 10^6$	$1.912 \cdot 10^6$	$1.887 \cdot 10^6$	18000 15400	5 6
$(BTMA)_2 MnCl_4$	$2.145 \cdot 10^6$	$1.78 \cdot 10^6$	$1.766 \cdot 10^6$	13900	3
DBPMC	2.10^6	$1.678 \cdot 10^6$	$1.674 \cdot 10^6$	23600 19400	

Characteristic wave number of neighbouring products in m^{-1}

The curve f is a straight line of the equation:

$$\ln G = \ln G_M + \alpha \left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2} \right)^2.$$

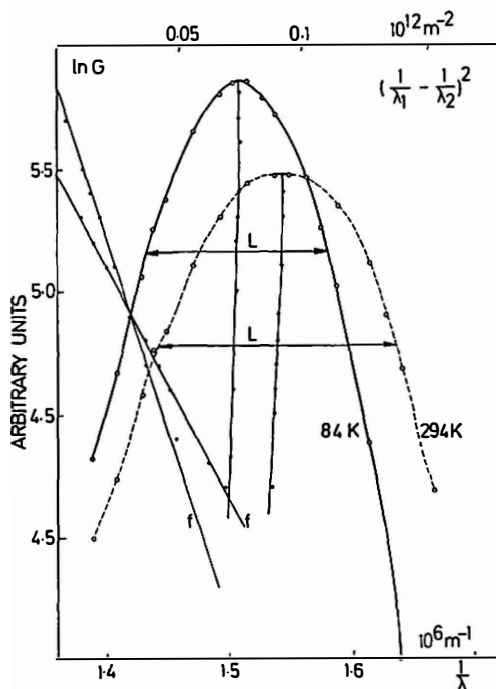


Fig. 4 Curves for the BPMC complex. Experimental dots of $\ln G \left(\frac{1}{\lambda} \right)$ at 294 K and experimental dots at 84 K. Curve f represents $\ln G = \left(\frac{1}{\lambda_2} - \frac{1}{\lambda_1} \right)^2$

The diameter of the curve $\ln G\left(\frac{1}{\lambda}\right)$ is not a straight line. It is vertical toward the apex $\left(\frac{G}{G_M}\right) > 0.7$, and for $\frac{G}{G_M} < 0.6$ it is a straight line of a positive slope m .

The shape of the curve is not that of Eq. (1).

$$\text{At } T = 294 \text{ K} : \frac{1}{\lambda_M} = 1.544 \cdot 10^6 \text{ m}^{-1}; \quad a = -18.8 \cdot 10^{-12} \text{ m}^2;$$

$$L = 0.192 \cdot 10^3 \text{ m}^{-1}$$

$$\text{At } T = 84 \text{ K} : \frac{1}{\lambda_M} = 1.508 \cdot 10^6 \text{ m}^{-1}; \quad a = -31.4 \cdot 10^{-12} \text{ m}^2;$$

$$L = 0.146 \cdot 10^6 \text{ m}^{-1}$$

Shift of the maximum of $G\left(\frac{1}{\lambda}\right)$ and variation of the width L

Fig. 5 gives the values of the wavenumber $1/\lambda_M$ of the maximum of G and those of the width L at different temperatures. The uncertainties are $\Delta\left(\frac{1}{\lambda_M}\right) = 0.003 \mu^{-1}$; $\Delta L = 0.006 \mu^{-1}$.

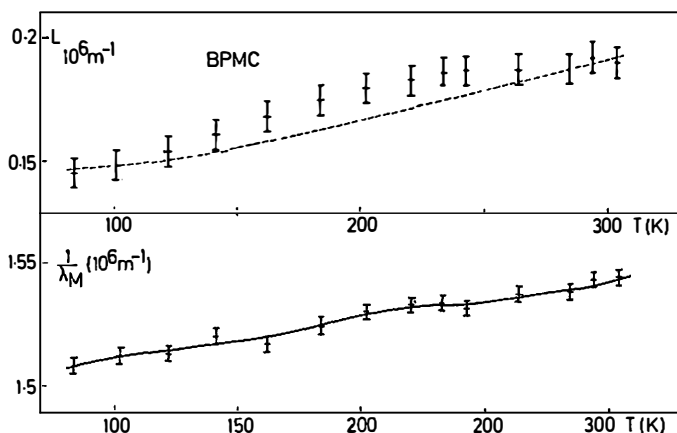


Fig. 5. $\frac{1}{\lambda_M}$ — the wave number of the maximum of G . L — the halfwidth of $G\left(\frac{1}{\lambda}\right)$ as a function of temperature. Experimental dots. Dotted line: $L = 0.146 \sqrt{\coth \frac{200}{T}}$

λ_M varies more with temperature T than in the case of DBGMC:

$$\lambda_M = 663.1 \text{ nm at } 84 \text{ K}; \quad \lambda_M = 647.2 \text{ nm at } 304 \text{ K}.$$

We have compared the experimental widths with theoretical formulae. Eq. (2) adjusted to the experimental values at 100 K and 300 K gives:

$$L = 0.146 \sqrt{\coth \frac{200}{T}}.$$

We observe (see Fig. 5) that the experimental widths L do not follow this theoretical formula.

We have made an attempt with the formula of Eq. (3) with the experimental results at 100 K, 150 K and 300 K (obtained by interpolating the experimental curve).

$$\text{Let us set } x = \frac{h\nu_e}{2kT} \quad \text{for } T = 300 \text{ K. } \frac{1}{L^2} = A \text{th } \frac{h\nu_e}{2kT} + B$$

$$T = 100 \text{ K } 45.04 \cdot 10^6 = A \text{th } 3x + B$$

$$T = 150 \text{ K } 35.86 \cdot 10^6 = A \text{th } 2x + B$$

$$T = 300 \text{ K } 27.7 \cdot 10^6 = A \text{th } x + B$$

$$y = \frac{\text{th } 3x - \text{th } x}{\text{th } 3x - \text{th } 2x} = 1 \cdot 89.$$

Now the minimum value of y is 2, obtained for x or $\nu_e = 0$. The spectra of BPMC do not follow the laws predicted by the model of configurational curves: the diameter of $\ln G \left(\frac{1}{\lambda} \right)$ is not rectilinear and the width does not follow the theoretical formulae.

Comparison with neighbouring products

Table 3 gives a survey of the spectral characteristics of the investigated BPMC compound and of the complex compounds of manganese investigated in previous works: among these there is also the compound of manganese (II) chloride with tetramethylammonium chloride (the molecular ratio 1 : 1).

The results are close to those on TMMC which has not a structure with isolated anions but a chain structure where the Mn^{++} ions are interlinked by the Cl^- ion bridges¹⁰⁾. Hence we cannot any more apply the usual theory because the linked Mn^{++} ions interact and an excitation transfer from Mn^{++} to its neighbours occurs¹¹⁾.

TABLE 3

	$\frac{1}{\lambda_{obs}} {}^4T_1(4G)$ at about 80 K	$\frac{1}{\lambda_M} (I\lambda)$ at about 80 K	$\frac{1}{\lambda_M} \left(G \left(\frac{1}{\lambda} \right) \right)$ at about 80 K	Referen- ces
TMMC or $[(CH_3)_4N] MnCl_3$	$1.862 \cdot 10^6$	$1.515 \cdot 10^6$	$1.493 \cdot 10^6$	
$(C_5H_5NCl) MnCl_3$	$1.869 \cdot 10^6$	$1.484 \cdot 10^6$	$1.468 \cdot 10^6$	3
BTMA $MnCl_3$	$1.852 \cdot 10^6$	$1.546 \cdot 10^6$	$1.522 \cdot 10^6$	3
BPMC	$1.818 \cdot 10^6$	$1.52 \cdot 10^6$	$1.508 \cdot 10^6$	

Characteristic wave numbers of neighbouring products in m^{-1}

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LUMINESCENTNE OSOBINE KOMPLEKSNIH JEDINJENJA MANGANO (II) HLORIDA SA 4-BENZILPIRIDIN-HIDROHLORIDOM

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Proučavane su fotoluminescentne osobine kompleksnih jedinjenja manganu (II) hlorida sa 4-benzilpiridin-hidrohloridom i to: $(C_{12}H_{12}N)_2 MnCl_4$ (DBPMC) i $C_{12}H_{12}N MnCl_3$ (BPMC). Za interpretaciju emisionih luminescentnih spektara korišćen je teoriski model paraboličnih konfiguracionih krivi. Kod jedinjenja DBPMC dobijena su dobra slaganja teoriskih i eksperimentalnih rezultata, što nije bio slučaj i kod jedinjenja BPMC. Pretpostavlja se da su neslaganja sa teorijskim modelom konfiguracionih krivi verovatno posledica prenosa energije sa pobuđenog manganu jona na susedne jone.