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Raman study of liquid crystalline *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]methylanilines

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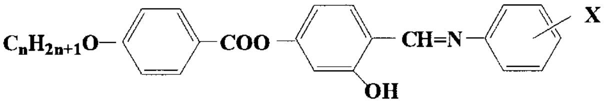
New homologous series of *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]methylanilines [*n*AH*m*M ($n = 1$–8/10; $m = 2$: *ortho*, $m = 3$: *meta*, $m = 4$: *para*)] were synthesized. They exhibited a nematic phase except for 1AH3M. The temperature dependence of their Raman spectra was observed in the spectral range of 900–1700 cm^{-1}. In one group of *n*AH*m*M compounds, the Raman band at about 1360 cm^{-1} abruptly decreased in intensity and wavenumber when the crystalline solid–liquid crystal phase transition was approached. In another group, the corresponding band increased through the phase transition. The bands have been assigned to the coupling mode between the in-plane CCH deformational vibration and the ring–N stretching vibration. Such a behaviour can be explained by the molecular conformation with different twist angles of the aniline ring in relation to the Schiff's base plane of the molecule. Some *n*AH*m*Ms exhibited photochromism.

1. Introduction

Raman spectroscopy is a powerful technique for the study of phase transitions in liquid crystals, with major attention being given to the analysis of either the internal or external vibrations [1]. The internal vibrations relate to the isolated molecule perturbed by the molecular field of neighbouring molecules; the analysis of internal vibrations is expected to provide a good picture of conformational changes [1–5]. The Raman bands ascribed to external modes disappear when the three-dimensional order of the crystal lattice is lost through the crystalline solid–liquid crystal phase transition [6, 7].

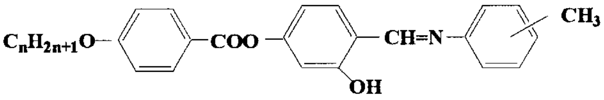
Liquid crystal compounds are mostly composed of two parts, a rigid central group and one or two flexible terminal alkyl or alkoxy chains. So, almost all rod-like liquid crystals reported so far have *para*-substituted terminal groups [8, 9]. Lateral substitution into aromatic rings of the core group increases the molecular breadth, resulting in a destabilization of the liquid crystal phase [10, 11]; in consequence, the liquid crystal phase is hard to detect. We have previously studied the effect of substituents *X* in the *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]-*X*-anilines (**I**), in which *X* is Cl, CN, OH, OCH$_3$ or OC$_2$H$_5$ [12–15].



n (alkoxy chain length); $n = 1$–8
m (substituted position of *X*); $m = 2$ (-*ortho*), 3 (-*meta*), 4 (-*para*).

The all *para*-*X*-substituted compounds exhibited a liquid crystal phase, as did almost all the *ortho*-*X*- and *meta*-*X*-substituted compounds.

The *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]methylanilines, *n*AH*m*Ms (**II**) have now been newly synthesized.



n (alkoxy chain length); $n = 1$–8/10
m (substituted position of CH$_3$); $m = 2$ (-*ortho*), 3 (-*meta*), 4 (-*para*).

They all exhibit a nematic phase except for 1AH3M. These compounds have a core frame of *N*-2-hydroxybenzylideneaniline (HBA), which is also known as

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N-salicylideneaniline (SA); some HBAs show photochromism or thermochromism in the crystalline state. In recent years, the study of solid photochromic and thermochromic materials has grown because of their possible application in the optical processing of information, in addition to general scientific interest [16, 17]. Some *n*AH*m*M compounds exhibit photochromism in the solid phase, as described briefly below.

We have measured the temperature dependence of Raman bands of *n*AH*m*Ms. The Raman band associated with the core part of the molecule shows an interesting temperature dependence through the crystalline solid–nematic liquid crystal phase transition. The temperature dependence of the band can be explained by the change of molecular conformation ascribed to the change of the twist angle of the aniline ring out of the Schiff's base plane of the molecule.

2. Experimental

4-(4-*n*-Alkoxybenzoyloxy)-2-hydroxybenzaldehydes were first synthesized by the method described in an earlier paper [13]. The substituted benzaldehydes thus obtained were then coupled with 2-, 3- or 4-methylaniline by heating under reflux in ethanol solution. The crude product was purified by repeated recrystallization from a mixture of ethanol and benzene. The liquid crystalline texture and phase transition temperatures were determined by means of a Nikon polarizing microscope equipped with a Mettler FP-52 microfurnace. The transition temperatures were also measured using a Shimazu differential scanning calorimeter (DSC); each sample was heated and cooled under a flow of nitrogen gas to prevent decomposition. The phase transition temperatures based on texture change were in good accord with those measured by DSC.

Raman spectra were obtained with an SPEX 1403 double monochromator equipped with a photon counting system in a 90° scattering geometry. Radiation of 632.8 nm from a helium–neon laser (NEC GLG 7500) was used to excite the Raman spectra to avoid fluorescence in these compounds. The powder sample was contained in a glass capillary; its temperature was controlled to within 0.5°C during the recording of the spectra.

UV irradiation was performed using a Moritex high pressure mercury lamp (200 W) with a band pass glass filter (transmission maximum around 365 nm); the irradiation time was 5 min. The absorption spectra were obtained with a JASCO Ubest-35 spectrometer at room temperature. For UV irradiation and absorption measurement, the polycrystalline film prepared from the melt was held between a pair of quartz plates separated about 20 μm. The sample film was allowed to stand overnight in a dry box before measurements.

3. Results and discussion

3.1. *Phase transitions*

The phase transition temperatures of the *n*AH*m*Ms are summarized in table 1. In the *n*AH4Ms, each of the homologous series exhibited an enantiotropic nematic phase. In the *n*AH3Ms, 1AH3M did not exhibit a liquid crystal phase and the (2, 3)AH3Ms exhibited only a monotropic nematic phase. The homologues with *n* greater than 4 showed an enantiotropic nematic phase, except for 8AH3M. This characteristic behaviour (that the enantiotropic nematic phase is observed only for members of the homologous series with a relatively long alkoxy chain) has frequently been observed in other homologous series of mesomorphic materials. 8AH3M did not show a nematic phase on heating, as mentioned above, while the (9, 10)AH3Ms showed an enantiotropic nematic phase. The unusual behaviour of 8AH3M may probably be ascribed to its different crystal structure from that of the other homologues. It is difficult at this stage to explain this behaviour exactly.

In the *n*AH2Ms, the first homologue exhibited a monotropic nematic phase. The other homologues of

Table 1. Phase transition temperatures (°C) of *n*AH*m*M compounds. Cr = crystalline solid, N = nematic, I = isotropic; the parentheses show monotropic transitions.

	$m = 4$ (-*para*)					$m = 3$ (-*meta*)					$m = 2$ (-*ortho*)				
n	Cr		N		I	Cr		N		I	Cr		N		I
1		157		284					120			(126)		134	
2		158		286			(116)		153			122		145	
3		125		272			(88)		124			111		124	
4		122		265			95		113			105		125	
5		118		248			87		105			104		117	
6		116		243			83		111			97		121	
7		117		232			103		104			92		115	
8		118		224			(107)		109			87		114	
9							100		103						
10							94		105						

this series exhibited an enantiotropic nematic phase. Moreover, the solid–nematic transition temperature for *n*AH3Ms or *n*AH2Ms is lower than that for the corresponding *n*AH4M homologues. From a comparison of the phase transition behaviour among these homologous series, it is evident that the introduction of a lateral substituent is unfavourable for the appearance of a liquid crystalline phase. It is well known that a lateral substituent results in molecular broading, and reduces the lateral intermolecular force of attraction [10, 11]; mesomorphic thermal stability thus decreases. However, it can be clearly recognized that the *n*AH3Ms and *n*AH2Ms (except for 8AH3M as mentioned above) exhibit an enantiotropic nematic phase as the alkoxy chain is lengthened.

3.2. *Raman spectra*

The Raman spectra for the *n*AH*m*Ms were measured in the 20–1800 cm^{-1} range at various temperatures. The spectra of (1, 2)AH4Ms were not measured because of the high solid–nematic phase transition temperature. The spectra of (3–8)AH4Ms in an isotropic liquid phase were also not measured, because of their high temperatures; the thermal decomposition of the *n*AH*m*Ms begins at about 150°C. The spectra of (2, 3, 8)AH3Ms and 1AH2M, which exhibited a monotropic nematic phase, were not measured. The spectrum of 1AH3M was not measured because it did not exhibit a liquid crystal phase.

The solid phase is characterized by three-dimensional order; the molecules move cooperatively. In liquid crystal phases, the molecules are loosely coupled to each other and randomly arranged in the direction along the long axis; they move incoherently. Finally, in the isotropic liquid phase, complete disorder prevails, and the molecules undergo a random motion in all dimensions. Furthermore, rotational isomers of the end groups of the liquid crystal molecule appear in the higher temperature phases [4, 5]. The Raman spectroscopic technique has been largely utilized to investigate the lattice mode region (below 150 cm^{-1}) and the conformationally sensitive alkyl chain mode region (200–900 cm^{-1}) [1]. Raman bands due to the lattice mode disappear as the three-dimensional order of the crystal lattice is lost by transition to higher temperature phases [6, 7]. The alkyl chains of the liquid crystal molecules melt in the liquid crystal phases, and are similar to those in the isotropic phase [1–5]. The decrease in intensity in the alkyl chain mode region can be explained by an increase of the degree of randomness in liquid crystal phases. The temperature dependence of Raman bands in these two regions is not reported here, because their bands showed a similar behaviour to those reported previously.

We now report the temperature dependence of Raman bands of the *n*AH*m*Ms in the 900–1700 cm^{-1} region (mainly associated with the core part of the molecule). It is interesting to correlate the change in the Raman bands associated with the core part of the molecule as a function of temperature with intra/inter-molecular interactions and the resulting dynamics of the core. Figure 1 typically shows the Raman spectra of 6AH3M in crystalline solid (Cr) (room temperature), nematic (N) (90°C) and isotropic liquid (I) (127°C) phases. Some of the bands show an abrupt change in intensity through the Cr–N liquid crystal phase transition.

The 1365 cm^{-1} band at room temperature decreases in intensity in the N phase. Figure 2 shows the intensity change and the wavenumber shift of the weak band at 1365 cm^{-1}. The band decreases drastically in intensity and wavenumber when the phase transition Cr–N is approached, but remains unchanged within the N and I phases. The corresponding band appeared to be very strong in the Raman spectrum and weak in the infrared (IR) spectrum for *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]-2/3-hydroxyanilines [14]. The corresponding band was weak in the Raman and IR spectra for *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]-4-hydroxyanilines [14]. Hence this band is reasonably considered to relate to the vibrational mode of the aromatic core and has been ascribed to the coupling mode between the in-plane CCH deformational vibration (ν_9) and the ring–N stretching vibration [14, 18]. The corresponding band at about 1360 cm^{-1} in (5–8)AH4Ms, (4, 6, 7, 9, 10)AH3Ms and (3, 4, 6–8)AH2Ms exhibited a temperature dependence similar to that for 6AH3M.

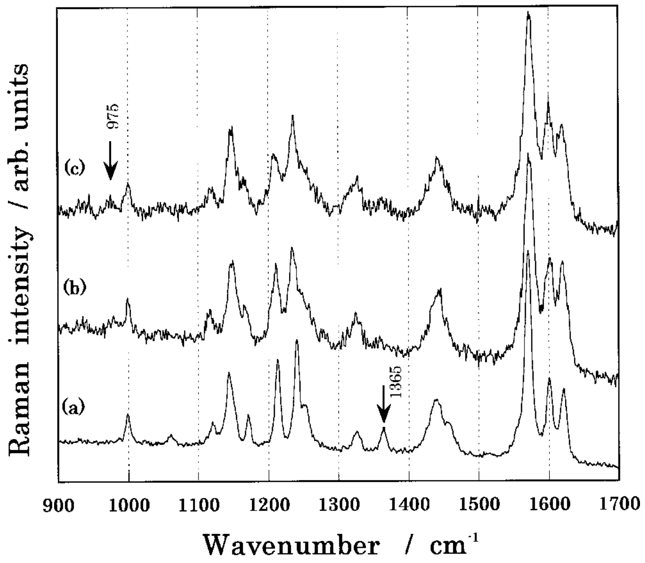

Figure 1. Raman spectra of 6AH3M: in the (*a*) solid (room temperature: 25°C), (*b*) nematic (90°C) and (*c*) isotropic (127°C) phases.

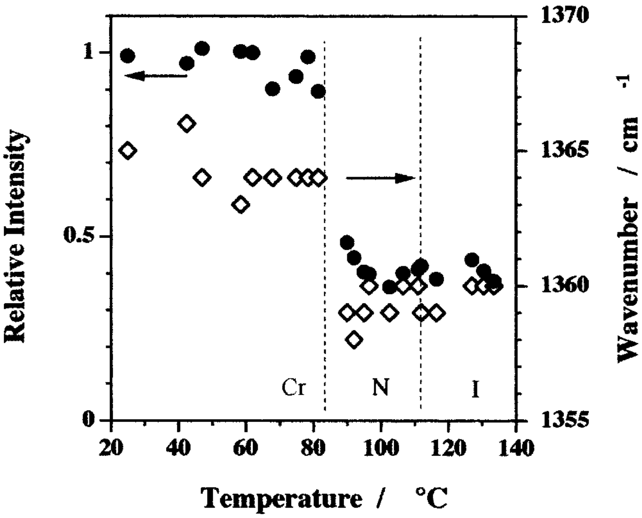

Figure 2. Temperature dependence of the intensity and wavenumber of the 1365 cm^{-1} Raman band of 6AH3M. The Raman band intensity is measured relative to the 1572 cm^{-1} band. Cr, N and I respectively, denote solid, nematic and isotrophic phases: ● intensity, ◇ wavenumber.

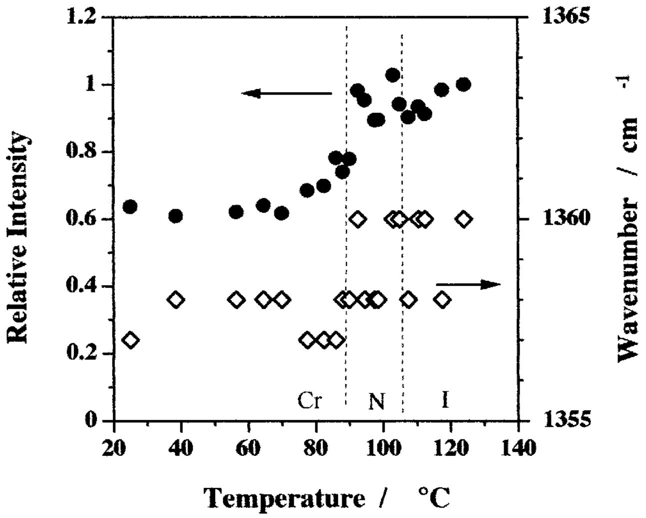

Figure 4. Temperature dependence of the intensity and wavenumber of the 1357 cm^{-1} Raman band of 5AH3M: ● intensity, ◇ wavenumber.

Figure 3 shows the Raman spectra of 5AH3M in the Cr (34°C), N (92°C) and I (112°C) phases. Figure 4 shows the temperature dependence of its 1357 cm^{-1} band in the solid phase. The intensity and wavenumber increase slightly at the Cr–N phase transition, but remain unchanged within the N and I phases. The band can be ascribed to the same vibrational mode as that of 6AH3M. The corresponding band in 3AH4M, 4AH4M and 2AH2M also increased in intensity in higher temperature phases. The phase transition solid (Cr_1)–solid (Cr_2) was also observed for (3, 4)AH4Ms. In 3AH4M, the 1362 cm^{-1} band in the Cr_1 phase intensifies in the Cr_2 phase, as shown in figure 5. It is interesting that the band is sensitive to the solid–solid phase transition. The intensity decreased discontinuously at the Cr_2–N phase transition, yet the intensity in the N phase was stronger than that in Cr_1 phase. Meanwhile, the wavenumber in the nematic phase was lower than that in the Cr_1 phase. The corresponding band in 4AH4M exhibited a similar temperature dependence to that of 3AH4M. Thus, in 3AH4M and 4AH4M, it is considered that this vibrational mode reflects a change of the molecular structure at the solid–solid phase transition. However, this mode is not sensitive to the solid–nematic phase

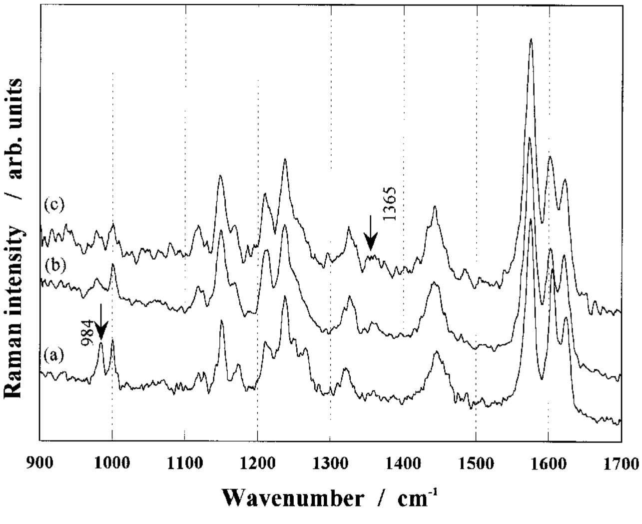

Figure 3. Raman spectra of 5AH3M: in the (*a*) solid (34°C), (*b*) nematic (92°C) and (*c*) isotropic (112°C) phases.

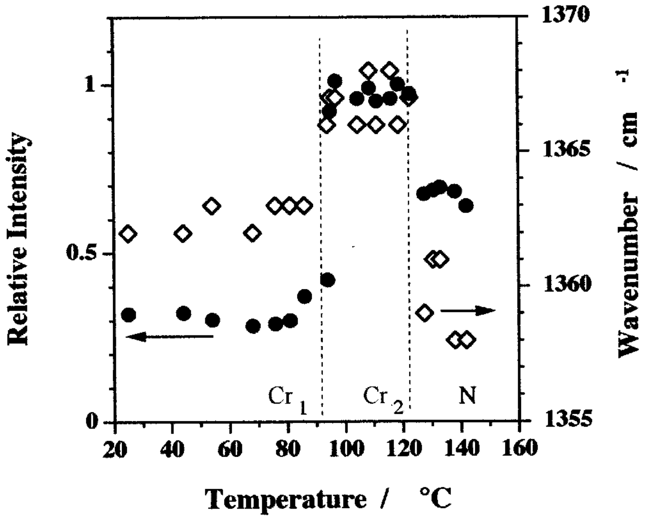

Figure 5. Temperature dependence on the intensity and wavenumber of the 1362 cm^{-1} Raman band of 3AH4M. Cr_1 and Cr_2, respectively, denote solid 1 and solid 2 phases: ● intensity, ◇ wavenumber.

transition and is distinct from the vibrational mode of the terminal group, which is sensitive to this transition [1–5].

In 5AH2M, the temperature dependence of the noted band at about 1360 cm^{-1} was difficult to measure because of the weakness in intensity. Therefore, we are unable to discuss the band for this compound.

It is interesting that the different temperature dependence of the band ascribed to the same vibrational mode has been observed between two goups of *n*AH*m*Ms. The temperature dependence of the band at about 1360 cm^{-1} can be explained by the change of the twist angle of the aniline ring out of the Schiff's base plane. A further explanation will be given below.

In 6AH3M, the 975 cm^{-1} band appeared in the nematic phase, as shown in figure 1. The intensity and wavenumber of the band remained unchanged in the N and I phases. Figure 6 shows the intensity change and the wavenumber shift of the band as a function of temperature. In (5–8)AH4Ms, (4, 6, 7, 9, 10)AH3Ms and (3, 4, 6–8)AH2Ms, the intensity of the corresponding band increased or appeared in the higher temperature phase, similar to the behaviour of 6AH3M described above.

In 5AH3M, the 984 cm^{-1} band due to that mode decreased in intensity in the higher temperature phases, as shown in figure 3. Figure 7 shows the intensity change and wavenumber shift of this band as a function of temperature. The band decreased abruptly at the Cr–N phase transition, and remained unchanged within the N and I phases. The corresponding band in 2AH2M exhibited a temperature dependence similar to that of 5AH3M. In 3AH4M and 4AH4M, the band decreased abruptly at the Cr_1–Cr_2 phase transition, and remained unchanged in consecutive phases. The vibrational mode is also sensitive to the phase solid–solid transition in these two compounds; thus, the band at about 980 cm^{-1} decreased in intensity and wavenumber at higher temperature phases.

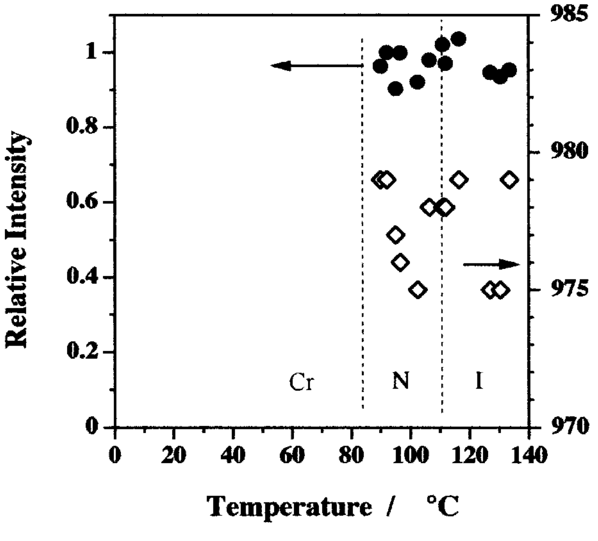

Figure 6. Temperature dependence of the intensity and wavenumber of the 975 cm^{-1} Raman band of 6AH3M: ● intensity, ◇ wavenumber.

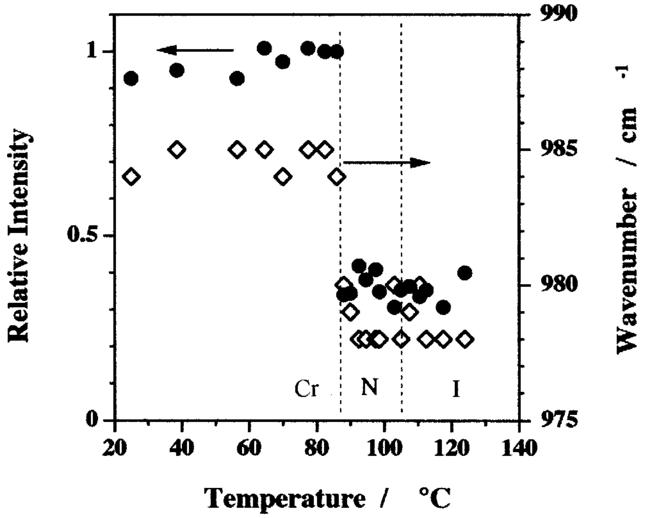

Figure 7. Temperature dependence of the intensity and wavenumber of the 984 cm^{-1} Raman band of 5AH3M: ● intensity, ◇ wavenumber.

A difference in temperature dependence of the band ascribed to the same vibrational mode has been observed also between two groups of the *N*-[4-(4-*n*-alkoxybenzoyloxy)-2-hydroxybenzylidene]chloroaniline (*n*AH*m*C) homologous series [15]. The band around 980 cm^{-1} can be assigned to the CH in-plane deformation vibrational mode of the aniline rings [19–21]. It has been reported that the twist angle of the aniline ring out of the C=N−C plane of MBBA (4-methoxybenzylidene-4-*n*-butylaniline) changes at phase transition [2]. That is, the change of the molecular conformation causes a change in the steric interaction between the hydrogen of the Schiff's base and the hydrogen atoms of the aniline ring. Consequently, the CH in-plane deformation vibrational modes may be affected by the change in the force constant of the steric interaction. In the *n*AH*m*Cs, two groups have different conformation of the twist angle between the aniline ring and the Schiff's base in the crystalline solid phase. In one group, the aniline ring is considered to adopt a nonplanar conformation with respect to the other part of the molecule, in which conformation the twist angle is large. In the other group, the molecule adopts a planar confirmation in which the twist angle is small.

The different temperature dependence of the CH in-plane deformation vibrational mode has been explained by the different temperature dependence of the conformation. The different temperature dependence of *n*AH*m*Ms can be explained in a similar manner to that of *n*AH*m*Cs, from their similar molecular structure. Here, it is considered that in the non-planar conformation the band ascribed to the CH in-plane deformational vibration is strong in intensity and high in wavenumber because of a weak steric interaction due to the long distance between the hydrogen of the Schiff's base and the hydrogen of the aniline ring. It is considered that in 5AH3M the group adopts the non-planar conformation, by analogy with the Raman spectra in *n*AH*m*Cs. On the other hand, the band in the planar conformation is weak in intensity and low in wavenumber because of steric hinderance due to the short distance between the hydrogen of the Schiff's base and the hydrogen of the aniline ring. The 6AH3M group is considered to have the planar conformation. In the 5AH3M group with the non-planar conformation in the solid, the twist angle between the aniline ring and the Schiff's base is presumably decreased during the solid–liquid crystal phase transition; hence the intensity and the frequency of the band decrease in the liquid crystal phase. In the other group, the twist angle is presumably increased; and the intensity and the frequency of the band increase in the liquid crystal phase. Thus, in the two groups, the different behaviour of temperature dependence of the band at about 980 cm^{-1} can be explained by the different internal twist in the aniline ring in relation to the other part.

The interesting temperature dependence of the band at about 1360 cm^{-1} can also be explained by the change of the twist angle of the aniline ring in relation to the rest of the molecule, in a similar manner to that of the band ascribed to the CH in-plane deformation vibration. The 1360 cm^{-1} band is assigned to the coupling mode between the in-plane CCH deformational vibration (ν_9) and the ring–N stretching vibration [14, 18], as mentioned above. It is known that the in-plane CCH deformation vibration appears at about 1180 cm^{-1} and is strong in intensity [22]; however, in the *n*AH*m*Ms the band is moderate in intensity and exhibits no significant change at phase transitions, as shown in figures 1 and 3. Therefore, the ring–N stretching can be considered to play an important role in the change of the band during phase transition. 6AH3M adopts the planar conformation in the crystalline solid phase, as mentioned above; the angle increases as the Cr–N transition is approached and remains almost unchanged within subsequent phases. Hence, the band that appeared strong in the planar conformation decreases in intensity and wavenumber, as shown in figure 2. In 5AH3M, the twist angle decreases at higher temperature phases; consequently, the 1357 cm^{-1} band increases in intensity and wavenumber in the higher temperature phases, as shown in figure 4. The different temperature dependences of the band can thus be explained by the change of molecular conformation related to the twist angle of the aniline ring out of the Schiff's base plane.

3.3. *Photochromism*

Some *n*AH*m*Ms exhibit photochromism in the crystalline solid phase. The occurrence of photochromism and the rate constants for each homologue are shown in table 2; the open circles indicate that definite photochromism is observed for the compound. All the homologues studied are yellow or pale yellow when freshly prepared; at room temperature the colour of the homologues exhibiting photochromism deepens to red–orange/orange under UV irradiation. In the case of the *n*AH2Ms, photochromism was observed only for 1AH2M. 1AH3M did not exhibit photochromism but the other *n*AH3Ms did. Kinetic analysis of the thermal fading of the species coloured by irradiation with UV light exhibited a good fit to a double-exponential equation, consisting of a two-stage transient decay (rate constants k_1 and k_2) [23, 24]. The rate constants were not measured for weak coloured specimens; they could not be exactly determined because of the small difference between the absorptions before and after irradiation. The rate constants for 3AH4M, 6AM3M and 8AH3M were close to those observed with photochromic HBA [23]. The rates of the other homologues were faster than those of HBA. A study of the correlation between photochromism and Raman spectra is in progress.

Table 2. Photochromism (indicated by ○) and rate constants k_1 and k_2 (s^{-1}) of *n*AH*m*M compounds.

n	$m = 4$ (-*para*)			$m = 3$ (-*meta*)			$m = 2$ (-*ortho*)		
1							○	1.1×10^{-1},	1.4×10^{-2}
2	○			○					
3	○	6.8×10^{-5}	6.7×10^{-5}	○	1.5×10^{-3},	2.5×10^{-4}			
4				○					
5	○	6.9×10^{-2},	5.6×10^{-2}	○	2.5×10^{-3},	2.4×10^{-4}			
6	○			○	4.9×10^{-4},	9.0×10^{-5}			
7				○					
8				○	2.6×10^{-4},	1.2×10^{-5}			

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