

# Concerning the miscibility of poly(vinyl phenol) blends – FTi.r. study

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The results of a Fourier transform infrared study of poly(vinyl phenol) (PVPh) blends containing a number of chemically and structurally dissimilar polymers are presented. These polymers include the polyesters poly( $\epsilon$ -caprolactone) and poly( $\beta$ -propiolactone); poly(vinyl alkyl ethers) where the alkyl groups are methyl, ethyl and isobutyl respectively; poly(ethylene oxide) and poly(vinyl pyrrolidone). All of these PVPh blends, with the exception of that containing poly(vinyl isobutyl ether), exhibit infrared spectral features consistent with a significant degree of mixing. Intermolecular hydrogen bonding interactions involving the PVPh hydroxyl group and either the carbonyl or ether oxygen moieties of the other polymers in the blend are identified. The relative strengths of these intermolecular interactions are discussed together with ramifications pertinent to the overall subject of polymer miscibility.

**(Keywords: polymer blends; polymer miscibility; poly(vinyl phenol); polyesters; polyethers; poly(vinyl pyrrolidone))**

## INTRODUCTION

In a recent paper<sup>1</sup> we presented results of a rather extensive FTi.r. study of poly(vinyl phenol) (PVPh) blends containing poly(vinyl acetate) (PVAc) and three copolymers of ethylene and vinyl acetate (EVA). PVPh was shown to be compatible with PVAc and the EVA's containing 45 and 70 wt% vinyl acetate at ambient temperature but incompatible with an EVA having 25 wt% vinyl acetate. Infra-red bands attributed to intermolecular bonding interactions involving the PVPh phenolic hydroxyl and the acetate carbonyl groups of the PVAc and EVA's were identified. Furthermore a quantitative measure of the fraction of hydrogen bonded carbonyl groups in the blends was obtained as a function of temperature. The results were considered in terms of an effective equilibrium constant and an estimation of the strength of the intermolecular interaction was obtained. Ramifications concerning polymer-polymer miscibility and the effect of temperature, solvent and glass transition temperature on the phase behaviour of these blends were considered.

In this paper, we present studies of PVPh blends containing a number of chemically and structurally dissimilar polymers. The results are interesting from a number of viewpoints. Previous infra-red studies of compatible blends, where only a frequency shift and broadening of the carbonyl stretching mode was observed, cannot be considered unambiguous evidence for miscibility. All that can be gained from the infra-red spectral data is evidence for the presence of intermolecular interactions between the two polymeric components in the blend. Independent verification of the phase behaviour, from thermal analysis, dynamic mechanical or

other techniques, is necessary. Accordingly, we can only conclude that the infra-red spectral features seen correlate with miscible polymer systems. The key factor missing is a measure of the number or fraction of intermolecular interactions occurring in the blend, which, in turn, would indicate the extent of mixing. Fortunately, the results presented in this and our previous paper<sup>1</sup> indicate that a measure of the degree of mixing is feasible. The intermolecular interaction between the phenolic hydroxyl group of PVPh and polymers containing accessible carbonyl groups is sufficiently strong to cause a major shift in frequency ( $\sim 25 \text{ cm}^{-1}$ ) of the carbonyl stretching mode. Thus we observe two well resolved infra-red bands attributable to carbonyl groups that are and are not involved in the intermolecular interaction. It follows that we are now in a position to judge the degree of mixing for specific polymer blends of given composition from infra-red spectroscopy.

Finally, if we accept the concept described by Patterson and Robard<sup>2</sup> that the phase behaviour of polymer blends at any given temperature is determined by a balance between a free volume contribution (always unfavourable) and an interactional term (potentially favourable) then we must in turn consider the interactional term to be a balance between the relative contributions from the self-association of the pure components to that of the association of the two different polymers in the blend. Accordingly, we postulated that if the relative strength and/or the number of intermolecular interactions could be increased without significantly increasing the relative contribution from self-association, it should be possible to 'compatibilize' a polymer that previously has shown no propensity for miscibility. Certain polymers that have been previously shown to be incompatible with poly(vinyl chloride) (PVC) and/or the poly(hydroxy ether of bisphenol A) (phenoxy) exhibit a substantial degree of mixing

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with PVPh. For example, PVC is compatible with several polyesters in the amorphous state<sup>3</sup>, including poly( $\epsilon$ -caprolactone) (PCL)<sup>4,5</sup>, but is incompatible with poly( $\beta$ -propiolactone) (PPL)<sup>6</sup>. Similarly, the phenoxy polymer is compatible with poly(vinyl methyl ether) (PVME) but incompatible with both poly(vinyl ethyl ether) (PVEE) and poly(vinyl isobutyl ether) (PVIE)<sup>7,8</sup>. As we will see PVPh is miscible with PCL, PPL, PVME and PVEE.

## EXPERIMENTAL

Table 1 lists the polymers used in this study. With the exception of the poly(vinyl pyrrolidone) (PVPr), all of the polymers have been described previously<sup>1,5,6,8,9</sup>. The PVPr used here has a reported molecular weight (undefined) of 360 000.

A common solvent was used to make 1% solutions (by weight) of the polymer blends. Thin films of the PVPh blends prepared for FTi.r. studies were cast from the solutions onto potassium bromide windows at room temperature. After a majority of the solvent had evaporated the films were transferred to a vacuum dessicator to completely remove residual solvent. The samples were stored under vacuum to minimize water adsorption.

Infra-red spectra were recorded on a Digilab FTS-15E FTi.r. spectrometer. Two hundred scans at a resolution of  $2\text{ cm}^{-1}$  were signal averaged and stored on a magnetic disc system. The frequency scale is internally calibrated with a reference Helium-Neon laser to an accuracy of  $0.2\text{ cm}^{-1}$ . A SPECAC high temperature cell mounted in the spectrometer was employed to obtain elevated temperature spectra to an accuracy of  $\pm 2^\circ\text{C}$ . All of the films used in this study were sufficiently thin to be within a range where the Beer-Lambert law is obeyed.

## RESULTS AND DISCUSSION

### PVPh blends with poly( $\epsilon$ -caprolactone) and poly( $\beta$ -propiolactone)

It is certainly no surprise that blends of PVPh and PCL

are miscible in the amorphous state at ambient temperatures. PCL is an usual polymer in that it is miscible with a wide variety of chemically and structurally dissimilar polymers including PVC<sup>4,5</sup>, phenoxy<sup>9</sup>, vinylidene chloride copolymers<sup>10-12</sup>, styrene-acrylonitrile copolymers<sup>13,14</sup>, poly(bisphenol A carbonate)<sup>15-18</sup>, chlorinated polyethylene and polypropylene<sup>19,20</sup> and polyepichlorohydrin<sup>21</sup>.

Figure 1 shows infra-red spectra in the carbonyl stretching region ( $1800\text{--}1650\text{ cm}^{-1}$ ) of amorphous PCL-PVPh blends of varying composition cast from THF and recorded at  $75^\circ\text{C}$  (above the melting point of PCL). In common with the spectra shown in our previous publication concerning PVPh blends with PVAc and EVA<sup>1</sup>, two obvious bands are observed; the first attributable to amorphous PCL (self-associated) at  $1734\text{ cm}^{-1}$  and the second at  $1708\text{ cm}^{-1}$  which is assigned to the PCL carbonyl group hydrogen bonded to the PVPh phenolic hydroxyl group. The relative intensity of the  $1708\text{ cm}^{-1}$  band increases at the expense of the  $1734\text{ cm}^{-1}$  band as the concentration of PVPh is increased in the blend.

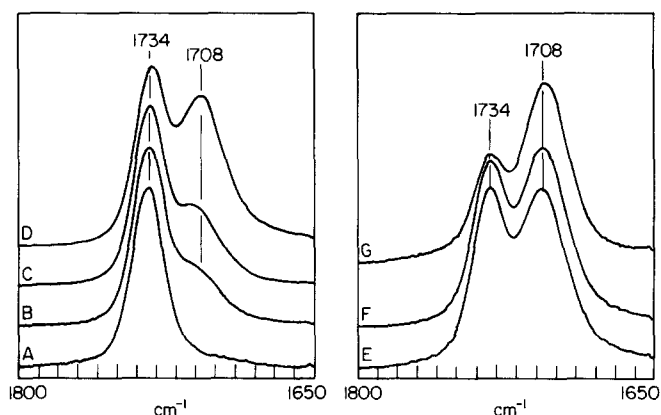


Figure 1 FTi.r. spectra recorded at  $75^\circ\text{C}$  in the  $1800\text{--}1650\text{ cm}^{-1}$  region for PVPh-PCL blends containing: (A) 100, (B) 80, (C) 70, (D) 50, (E) 30, (F) 20 and (G) 10 wt% PCL

Table 1 Polymers used in this study

Name	Acronym	Structure	Source
Poly( <i>p</i> -vinylphenol)	PVPh	$\text{—CH}_2\text{—CH—}$	Polysciences, Inc.
Poly( $\epsilon$ -caprolactone)	PCL	$\text{—CH}_2\text{—COO—}$	Union Carbide Corp.
Poly( $\beta$ -propiolactone)	PPL	$\text{—CH}_2\text{—COO—}$	Dr. K. Holland-Moritz Univ. Koln, W. Germany
Poly(vinyl pyrrolidone)	PVPr	$\text{—CH}_2\text{—CH—}$	Polysciences, Inc.
Poly(ethylene oxide)	PEO	$\text{—CH}_2\text{—CH}_2\text{—O—}$	Polysciences, Inc.
Poly(vinyl methylether)	PVME	$\text{—CH}_2\text{—CH(OCH}_3\text{)—}$	Aldrich Chemical Co.
Poly(vinyl ethylether)	PVEE	$\text{—CH}_2\text{—CH(OC}_2\text{H}_5\text{)—}$	Aldrich Chemical Co.
Poly(vinyl isobutylether)	PVIE	$\text{—CH}_2\text{—CH(OC}_4\text{H}_9\text{)—}$	Aldrich Chemical Co.

Figure 2 shows the corresponding hydroxyl stretching region from  $3700$  to  $3000\text{ cm}^{-1}$  for PCL–PVPh blends recorded at  $75^\circ\text{C}$ . The bottom spectrum of pure PVPh shows an infra-red band at  $3525\text{ cm}^{-1}$  attributed to 'free' or non-hydrogen bonded hydroxyls and a broad band centred at  $3370\text{ cm}^{-1}$  which is assigned to a wide distribution of hydrogen bonded hydroxyl groups (self-association)<sup>1</sup>. As the concentration of PCL is increased in the blend the spectrum in the hydroxyl region may be considered to consist of three major components. The first component is associated with free hydroxyls ( $3525\text{ cm}^{-1}$ ). The second is attributed to the self-association of the PVPh ( $3370\text{ cm}^{-1}$ ); that is, hydroxyl–hydroxyl interactions. Both of these bands decrease in relative intensity with increasing PCL concentration. Finally the third component at about  $3420\text{ cm}^{-1}$  arises from hydroxyl groups hydrogen bonded to the PCL carbonyl groups. Naturally, this component increases as the concentration of PCL increases. The frequency difference between the free hydroxyl absorbance and those of the hydrogen bonded species ( $\Delta\nu$ ) is a measure of the average strength of the intermolecular interactions<sup>22</sup>. The above results imply that the average strength of the hydrogen bond between the PCL carbonyl group and the PVPh hydroxyl group ( $\Delta\nu = 105\text{ cm}^{-1}$ ) is less than that occurring between hydroxyl groups in pure PVPh ( $\Delta\nu = 165\text{ cm}^{-1}$ ). This is entirely consistent with the results obtained in the phenoxy–PCL system<sup>9</sup>.

Spectra of PCL–PVPh blends cast from THF and recorded at room temperature exhibit additional features attributed to crystalline PCL. At temperatures below the crystalline melting point, a portion of the PCL in the

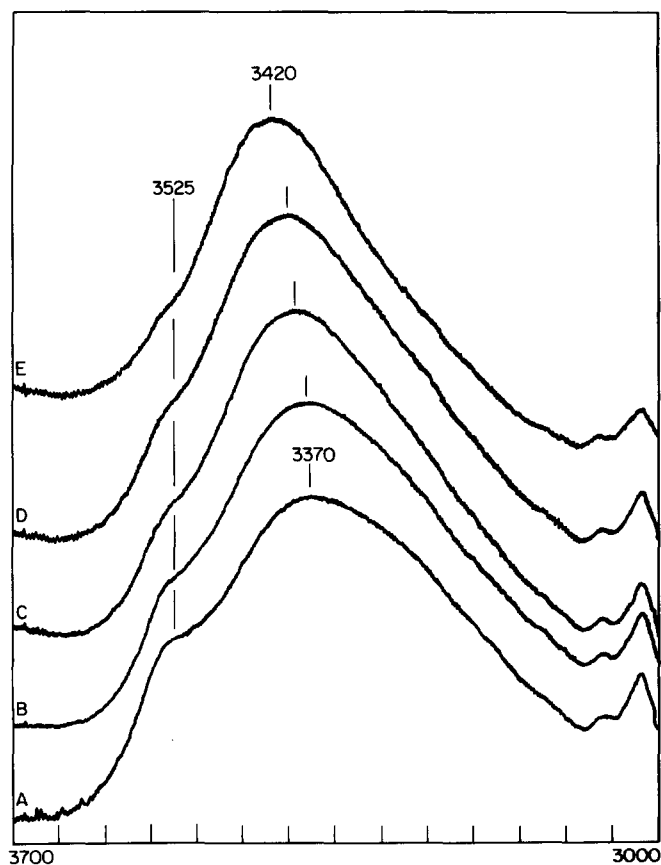


Figure 2 FTIR spectra recorded at  $75^\circ\text{C}$  in the  $3700$ – $3000\text{ cm}^{-1}$  region for PVPh blends containing: (A) 0, (B) 10, (C) 20, (D) 30 and (E) 50 wt% PCL

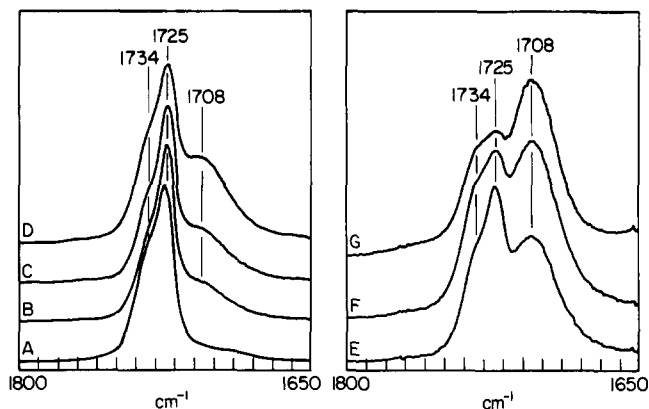


Figure 3 FTIR spectra recorded at room temperature in the  $1800$ – $1650\text{ cm}^{-1}$  region for PVPh–PCL blends containing: (A) 100, (B) 80, (C) 70, (D) 50, (E) 30, (F) 20 and (G) 10 wt% PCL

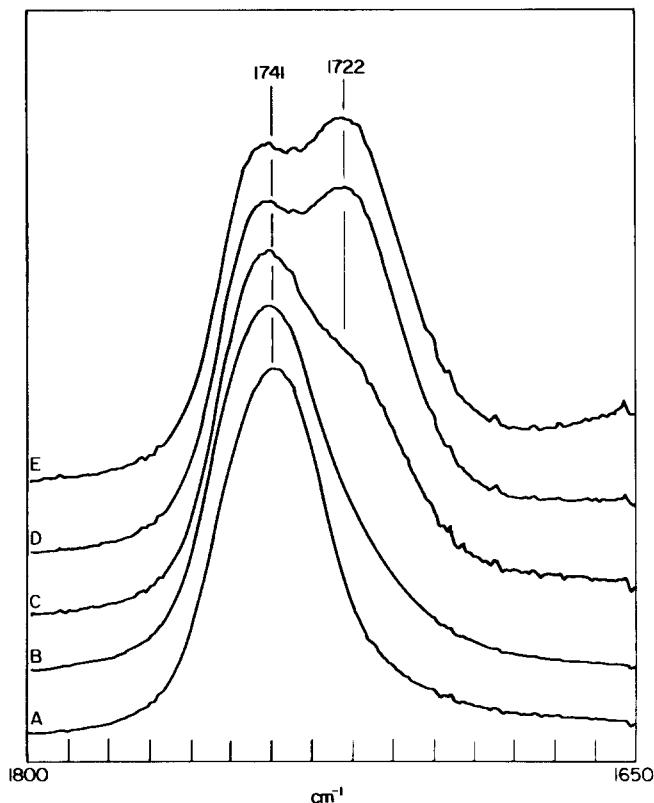
blend may crystallize. The composition of the amorphous phase is now richer in PVPh as a fraction of the PCL is now in crystalline domains. These blends have been termed crystalline/compatible blends<sup>23</sup>.

Figure 3 shows the carbonyl stretching region of PCL–PVPh blends of varying composition cast from THF and recorded at room temperature. The spectrum of pure PCL exhibits two bands. A relatively sharp one at  $1724\text{ cm}^{-1}$  attributed to PCL in its preferred ('crystalline') conformation and another seen as a shoulder at  $1735\text{ cm}^{-1}$  which is associated with amorphous PCL<sup>5,23</sup>. As the concentration of PVPh is increased in the blend a third band at  $1708\text{ cm}^{-1}$  is observed which increases in relative intensity with PVPh content. In the spectrum of the 10:90 PCL–PVPh blend there is still evidence of the band at  $1725\text{ cm}^{-1}$  which infers that there is still some crystalline PCL present. It must be emphasized that the phase behaviour of these blends depends upon the method of sample preparation, effective glass transition temperature and thermal history<sup>15</sup>.

In the hydroxyl stretching region of the spectra obtained for the PCL–PVPh blends recorded at room temperature the results are very similar to those seen at  $75^\circ\text{C}$  (Figure 2). There are some subtle differences reflecting the lower temperature and the compositional variations in the amorphous state arising from the crystallization of a portion of the PCL but the essential features are the same as those described above.

To reiterate, we anticipated the compatibility of PCL–PVPh blends. In contrast PPL was known to be incompatible with PVC<sup>6</sup>. Potentially the same type of intermolecular interaction is feasible and the question arises can we 'compatibilize' PPL by increasing the strength of this interaction?

Figure 4 shows the carbonyl stretching region for blends of PPL–PVPh cast from THF and recorded at  $80^\circ\text{C}$  (above the  $T_m$  of PPL). Pure PPL in the amorphous state at  $80^\circ\text{C}$  is characterized by a rather broad infra-red band centred at  $1741\text{ cm}^{-1}$  attributed to the carbonyl stretching vibration. Upon increasing the PVPh content in the blend a new band at  $1722\text{ cm}^{-1}$  becomes increasingly obvious. This band may readily be assigned to PPL carbonyl groups hydrogen bonded to the PVPh hydroxyls. It should be noted that no such analogous band was observed in the PPL–PVC system<sup>6</sup>. The spectral features observed in the hydroxyl stretching region of the



**Figure 4** FTi.r. spectra recorded at 80°C in the 1800–1650  $\text{cm}^{-1}$  region for PVPPh–PPL blends containing: (A) 100, (B) 80, (C) 50, (D) 20 and (E) 10 wt% PPL

PPL–PVPh blends are similar to those obtained for the PCL–PVPh blends (Figure 2). With increasing PPL content in the blend the contributions from free hydroxyls ( $3525 \text{ cm}^{-1}$ ) and self-associated hydroxyls ( $3370 \text{ cm}^{-1}$ ) decrease while a new band at about  $3440 \text{ cm}^{-1}$  increases. The latter is again ascribed to PVPh hydroxyls hydrogen bonded to the carbonyl groups of PPL.

Figure 5 shows the corresponding spectra of PPL–PVPh blends cast from THF and recorded at room temperature. In common with PCL, PPL is also a crystallizable polymer. Pure PPL at room temperature exhibits a rather broad band centred at  $1732 \text{ cm}^{-1}$  which is asymmetrically skewed to the high frequency side. This is a composite band consisting of two major components. PPL in its preferred ‘crystalline’ conformation absorbs at  $1732 \text{ cm}^{-1}$  while amorphous PPL absorbs at  $1741 \text{ cm}^{-1}$ . Upon blending with PVPh the now familiar hydrogen bonded carbonyl band is observed at  $1720 \text{ cm}^{-1}$  and this band increases in relative intensity with increasing PVPh content. Thus the PPL–PVPh blend spectra at room temperature consist of three major components attributed to hydrogen bonded carbonyls and non-hydrogen bonded carbonyls in amorphous and preferred conformations. The hydroxyl stretching region is very similar to that discussed above for the elevated temperature spectra.

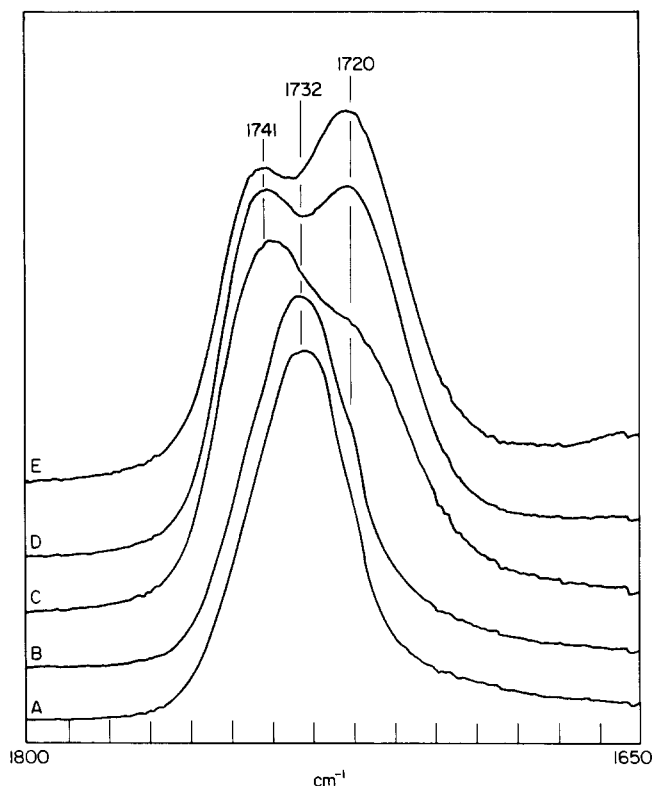
In summary, the results obtained from the elevated temperature FTi.r. studies of the PVPh blends with both PCL and PPL in the amorphous state are entirely consistent with compatible blend systems. At room temperature these blends may be considered to fall under the category of crystalline/compatible blends.

#### PVPh blends with poly(vinyl pyrrolidone)

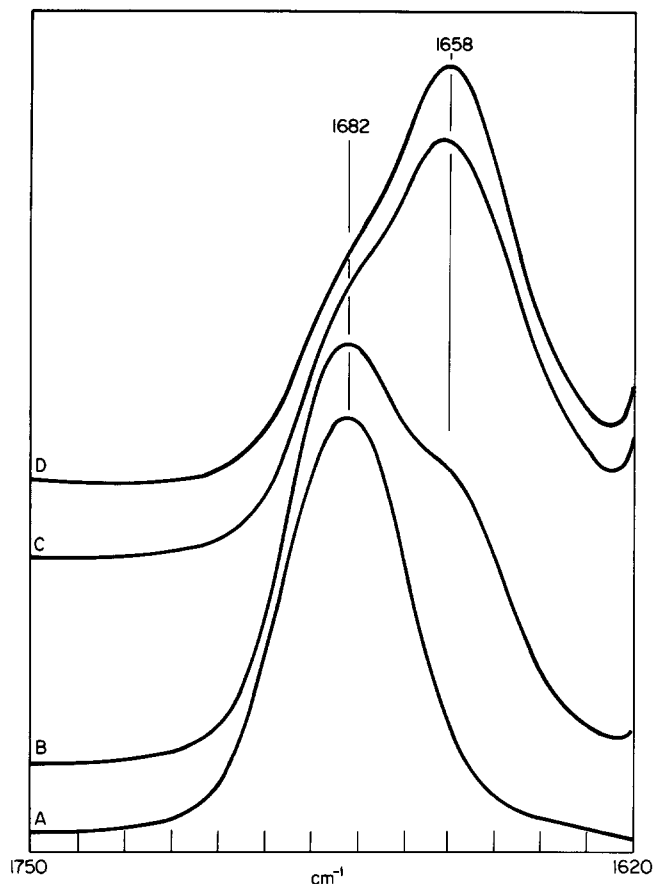
Poly(vinyl pyrrolidone) (PVPPr) is an atactic amorphous

polymer whose structure is shown in Table 1. Figure 6 shows the carbonyl stretching region of PVPPr–PVPh blends cast from THF and recorded at room temperature. Pure PVPPr is characterized in the carbonyl stretching region by a rather broad band at  $1682 \text{ cm}^{-1}$ . This band is a mixed mode containing contributions from the carbonyl stretching and N–C stretching vibrations which explains the relatively low frequency of this mode compared to acetate and ester carbonyl frequencies considered previously. Upon mixing PVPPr with PVPh a second band is observed at  $1658 \text{ cm}^{-1}$  which is readily assigned to hydrogen bonded PVPPr carbonyl groups. This shift to lower frequency of about  $24 \text{ cm}^{-1}$  is comparable to that observed for the polyester–PVPh blends described above. However, we must be cautious in attempting to equate these shifts in frequency with the absolute strength of the intermolecular interactions. It must be emphasized that the  $1682 \text{ cm}^{-1}$  band is a mixed mode which complicates the interpretation. Furthermore, the shifts in the carbonyl stretching region are measured relative to the frequency of the polymer in the self-associated state and not from the isolated ‘free’ frequency as in the case of the hydroxyl stretching mode.

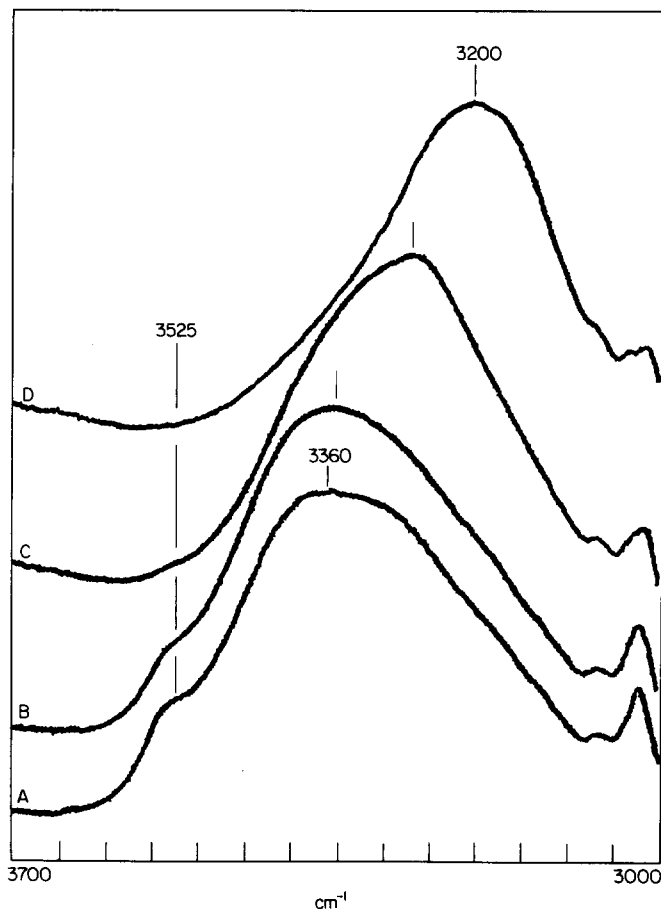
The corresponding hydroxyl stretching region of the PVPPr–PVPh blends is given in Figure 7. In sharp contrast to the spectral features observed in the compatible PVPh blends with PVAc, EVA’s<sup>1</sup>, PCL (Figure 2) and PPL, the frequency of the hydrogen bonded PVPh hydroxyl occurs at a lower frequency ( $3230 \text{ cm}^{-1}$ ) than that ascribed to the self-association of pure PVPh ( $3360 \text{ cm}^{-1}$ ). (We must caution that both PVPh and PVPPr are hydrophilic; PVPPr actually dissolves in water and the blend sample readily absorbs moisture. Although every attempt was made to exclude moisture, we cannot definitively state that there is no contribution from water in this region of



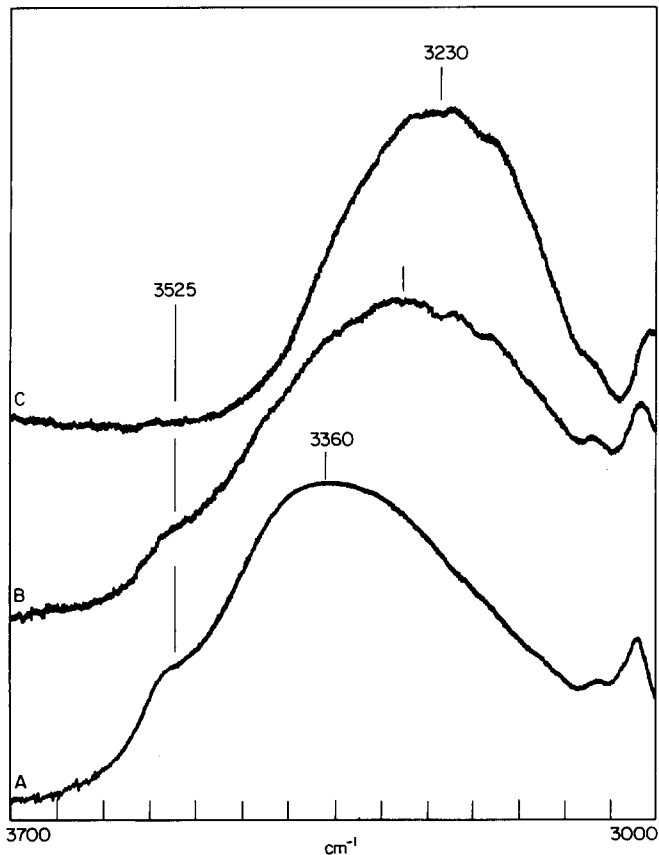
**Figure 5** FTi.r. spectra recorded at room temperature in the 1800–1650  $\text{cm}^{-1}$  region for PVPPh–PPL blends containing: (A) 100, (B) 50, (C) 20 and (E) 10 wt% PPL



**Figure 6** FTIR spectra recorded at room temperature in the 1750–1620  $\text{cm}^{-1}$  region for PVPh–PVPr blends containing: (A) 100, (B) 65, (C) 35 and (D) 20 wt% PVPr



**Figure 8** FTIR spectra recorded at room temperature in the 3700–3000  $\text{cm}^{-1}$  region for PVPh–PEO blends containing: (A) 0, (B) 20, (C) 50 and (D) 80 wt% PEO



**Figure 7** FTIR spectra recorded at room temperature in the 3700–3000  $\text{cm}^{-1}$  region for PVPh–PVPr blends containing: (A) 0, (B) 20 and (C) 65 wt% PVPr

the spectrum.) Nonetheless, if we use the position of the free hydroxyl stretching vibration at 3525  $\text{cm}^{-1}$  as a reference, then the median frequency difference ( $\Delta\nu$ ) for hydroxyl–hydroxyl intermolecular interactions (self-association) in PVPh is about 165  $\text{cm}^{-1}$  while that of the PVPh hydroxyl–PVPr carbonyl interactions is 295  $\text{cm}^{-1}$ . For comparison PVPh hydroxyl interactions with carbonyl groups of PVAc, EVA's, PCL and PPL exhibit frequency differences in the range of 95–105  $\text{cm}^{-1}$ . This would imply that the intermolecular interaction in PVPr–PVPh blends is considerably stronger than either the self-association of hydroxyl groups in PVPh or the interactions in PVPh–polyester and PVPh–acetate containing polymers.

#### PVPh blends with poly(ethylene oxide)

Poly(ethylene oxide) (PEO) is a crystallizable polymer which has been previously shown to be compatible with the phenoxy polymer in the amorphous state<sup>9</sup>. Figure 8 shows the hydroxyl stretching region of PEO–PVPh blends recorded at room temperature. With increasing concentration of PEO there is a concurrent increase in the contribution from a band at approximately 3200  $\text{cm}^{-1}$ , which is assigned to hydrogen bonded PVPh hydroxyl groups to PEO ether oxygens. In the 80:20 PEO–PVPh blend there is no evidence for the presence of free hydroxyls and the concentration of hydroxyl–hydroxyl interactions appears minimal. The frequency difference between the free hydroxyl band and the band attributed to hydroxyl groups hydrogen bonded to ether oxygens ( $\Delta\nu$ ) is about 325  $\text{cm}^{-1}$ . This is somewhat greater than

that observed for PEO-phenoxy system (c.a.  $270\text{ cm}^{-1}$ )<sup>9</sup> and presumably reflects a moderate increase in the relative strength of the PEO-PVPh intermolecular interaction; a reasonable conclusion considering the enhanced affinity for hydrogen bonding of the hydroxyl groups of PVPh compared to the phenoxy polymer. Analogous shifts are also seen in the spectra of phenol-diethyl ether and methanol-diethyl ether mixtures<sup>24,25</sup>.

#### PVPh blends with poly(vinyl alkyl ethers)

We have recently published a FTi.r. study of phenoxy blends with the poly(vinyl alkyl ethers), PVME, PVEE and PVIE<sup>8</sup>. The infra-red spectral results obtained supported the observations of Robeson *et al.*<sup>7</sup> that phenoxy-PVME blends are compatible whereas phenoxy-PVEE blends are not. In attempting to rationalize this observation we suggested that steric and/or variations in the intermolecular interaction strengths may play a role.

Figure 9 shows FTi.r. spectra in the hydroxyl stretching region of blends of both PVME-PVPh and PVEE-PVPh. The essential features of the spectra of the two systems are similar. As the PVME (or PVEE) content in the blend is increased, the hydrogen bonded hydroxyl band shifts to lower frequency and narrows significantly. In addition, the band at  $3525\text{ cm}^{-1}$  attributed to free hydroxyl groups decreases in relative intensity with increasing poly(vinyl alkyl ether) concentration. This is entirely consistent with the results presented for the PVME-phenoxy blends<sup>8</sup>. There are however some important subtle differences. The shift in frequency of the hydrogen bonded hydroxyl band relative to that of the free hydroxyl is 205 and  $200\text{ cm}^{-1}$  for the PVME-PVPh and PVEE-PVPh blends respectively. In contrast, compatible PVME-phenoxy exhibit an analogous shift in frequency of approximately  $150\text{ cm}^{-1}$ . This implies that the PVPh hydroxyl interaction with the ether oxygen of the poly(vinyl alkyl ethers) is stronger than that of the phenoxy polymer.

Finally, Figure 10 shows analogous spectra for the PVIE-PVPh system. No significant changes in the hydroxyl stretching region of the spectrum is observed as a function of PVIE concentration in the blend. This strongly suggests an incompatible system<sup>6,8</sup>.

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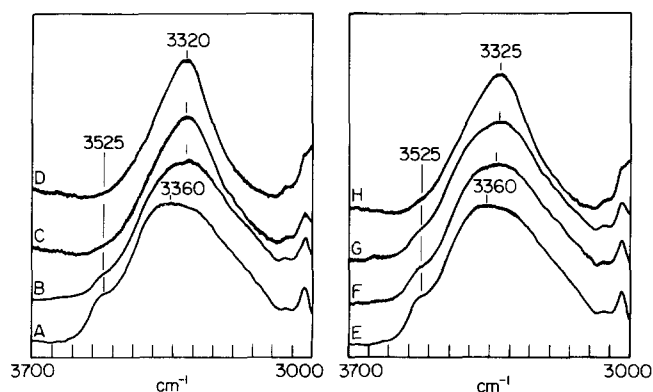


Figure 9 FTi.r. spectra recorded at room temperature in the  $3700\text{--}3000\text{ cm}^{-1}$  region for PVPh-PVME and PVPh-PVEE blends containing: (A) 0, (B) 20, (C) 50 and (D) 80wt% PVME and (E) 0, (F) 20, (G) 50 and (H) 80wt% PVEE

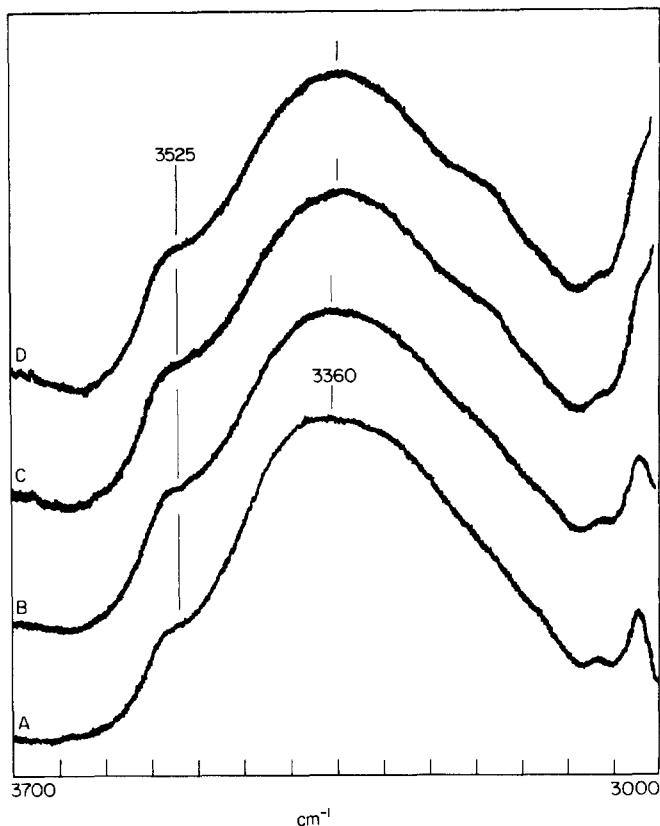


Figure 10 FTi.r. spectra recorded at room temperature in the  $3700\text{--}3000\text{ cm}^{-1}$  region for PVPh-PVIE blends containing: (A) 0, (B) 20, (C) 50 and (D) 80 wt% PVIE

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