The quantitative analysis of polyvinylpyrrolidone by infrared spectrophotometry

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The quantitative assay of the binding agent polyvinylpyrrolidone (PVP) in granules of magnesium carbonate has been shown to be possible. The method is to determine the infrared absorption spectrum of a chloroform extract. Quantities of PVP down to 0·1 mg can be successfully assayed to ±1% in solution as dilute as 0·1% w/v.

Polyvinylpyrrolidone is a substance commonly included in granule formulations for its binding properties. However, because of its high molecular weight, its subsequent analysis particularly in combination with other ingredients has proved to be difficult. Despite the fact that its structural formula seems to include chromophoric groups (I), it exhibits no useful ultraviolet absorption peaks. Simonelli, Mehta & Higuchi (1969) have recently analysed PVP by measuring the total differential refractive indices of PVP solutions in a differential refractometer. This method, however, fails for dilute solutions, and almost any other soluble compound interferes.

PVP does, however, show two useful peaks in the middle infrared region. At 1460 cm⁻¹ an absorption peak due to the scissor bending of –CH₂ groups occurs, but of particular interest is the high absorption peak at 1680 cm⁻¹ due to the $>C=O$ groups. This peak has been used to assay PVP and the method has proved successful even in exceedingly dilute solutions.
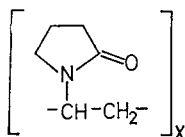
I. Structural formula for polyvinylpyrrolidone.
X = 180–6300.

The analytical method reported here was developed to enable the migration of PVP in the course of granule drying to be followed quantitatively. To do this, successive layers of powder were removed from the dried granules by controlled attrition. The powder removed was assayed for PVP content (Ridgway & Rubinstein, 1971).

METHODS

Spectra were obtained from a Unicam SP 100 infrared spectrophotometer working in the double-beam mode. The sample cell had potassium bromide windows and a fixed path length of 0·2 mm. The reference cell, also with potassium bromide windows, had a variable path length. Interference fringes were recorded for the empty sample cell, and the variable path length cell was adjusted to the same path length by observation of fringes. Chloroform (BDH Ltd., spectroscopic grade)

was injected into both cells, and spectra were run over the range 650–2150 cm^{-1}, checking that no absorption peaks were visible. A range of solutions of PVP (BDH Ltd., mol. wt. 44 000) were made up in the chloroform, and, using pure chloroform in the reference cell, spectra were obtained for each concentration.

Granules of magnesium carbonate were produced using known quantities of PVP as a binder. The method of production has been described elsewhere (Ridgway & Rubinstein, 1971). The granules were dried to constant weight in an oven, ground, weighed and shaken for 10 min with 5 ml of chloroform in a 10 ml stoppered flask. A sample was drawn into a syringe through a sweenex filter (Millipore Corp) fitted to the needle. The clear solution was then injected into the sample cell, and a spectrum obtained for each granule.

RESULTS AND DISCUSSION

The spectrum of PVP is shown in Fig. 1. The 'base line density' technique was used to evaluate the amount of PVP (Heigl, Bell & White, 1947). In this method a base line is drawn between two points selected, before and after the 1680 cm^{-1} peak, to give a line parallel as possible to the zero absorbance line.

The base line absorbance Ab is obtained from the equation

$$Ab = \log_{10} \frac{P_b}{P_o}$$

where P_b is the distance from the zero line to the base line, and P_o is the distance from the zero line to the top of the absorption peak.
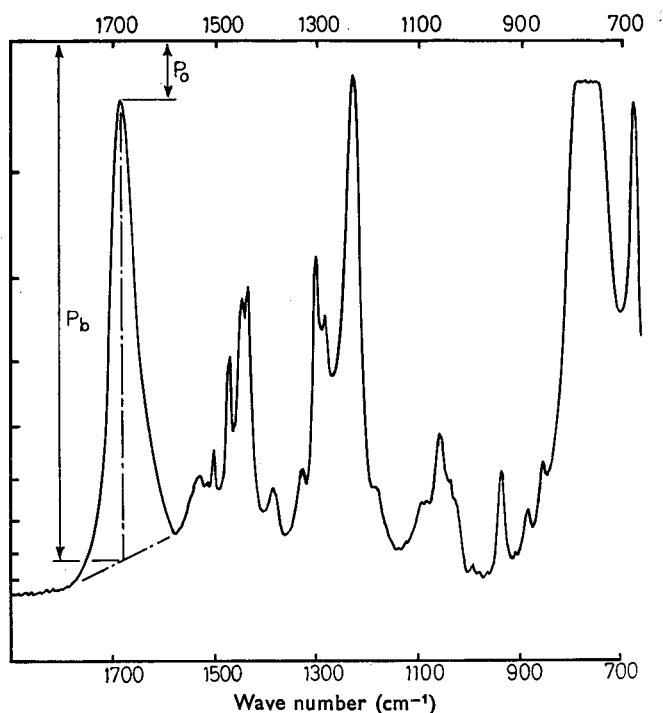

FIG. 1. The spectrum of polyvinylpyrrolidone (5% w/v in chloroform, path length 0·2 mm) in the infrared between 1800 and 650 cm^{-1}. The peak at 1680 cm^{-1} has been measured by the technique of Heigl, Bell & White (1947).

Both P_b and P_o were measured for the $1680 \, cm^{-1}$ line. A graph of absorbance (Ab) against PVP concentration was drawn for the prepared solutions of PVP whose concentration was known. A straight line resulted, showing that Beer's law applies over the concentration range 0·1–5% w/v. The gradient of the Beer's law line was 0·929 so that $\% \, PVP, \% \, w/v = \dfrac{\text{absorbance}}{0·929}$. For the PVP solutions made by chloroform extraction of the granules, the absorbance was calculated and the corresponding concentration of PVP evaluated. From the solution volume and the weight of the granule the percentage of PVP in the granule could be calculated. This value could then be compared with the amount of PVP incorporated during their manufacture. Such a comparison is given below.

PVP (15 g) was used to granulate 290 g of magnesium carbonate, so that the PVP content was 4·91% by weight. Table 1 gives the results for six replicate analyses.

Table 1. *Results of six replicate analyses for ground and extracted $MgCO_3$ granules containing 4·91 wt % PVP.*

Wt of granular material in 5 ml chloroform	Absorbance of solution	% PVP w/v in solution	% PVP w/w in granule
1·23178	1·105	1·189	4·83
1·25473	1·147	1·235	4·92
1·26238	1·161	1·250	4·95
1·24923	1·101	1·185	4·82
1·26748	1·170	1·260	4·97
1·23942	1·119	1·205	4·86

Mean 4·89. Standard deviation = 0·06.

The mean value is close to the expected result, and the standard deviation is about 1%. The analytical method thus seems accurate; if the minimum quantity of PVP needed to make a solution of absorbance $\geqslant 0·1$ were assayed, 0·1mg would be determinable to $\pm 1\%$.

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