SPECTROPHOTOMETRIC ANALYSIS

No.A393

Analysis of Ethanol and Anhydrous Ethanol in Accordance with the Fifteenth Edition of the Japanese Pharmacopoeia

The Japanese Pharmacopoeia is a compendium of standards for drugs that was established in accordance with the Pharmaceutical Affairs Law. The fifteenth edition came into effect on 1 April 2006. Regarding the differences between this edition and the fourteenth edition, although no changes were made to the "Infrared Spectrophotometry" item that appears under "General Tests, Processes and Apparatus", the infrared spectra for 29 of the items listed under "Official Monographs", including ethanol, anhydrous

ethanol, and acrinol hydrate, were added to "Infrared Reference Spectra". Accordingly, additions or modifications have been made to the identification tests specified for these items, and identification using infrared spectrophotometry is now required. Examples of the analysis of ethanol and anhydrous ethanol, for which the fifteenth edition of the Japanese Pharmacopoeia requires that identification tests are performed using infrared spectrophotometry, are presented here.

Samples

Analysis was performed using Japanese Pharmacopoeia-grade ethanol and anhydrous ethanol sold by Wako Pure Chemical Industries (Fig.1).

■ Demountable Cell

Fig.2 shows the demountable cell that was used for analysis. Usually, a sealed liquid cell is used for the analysis of volatile liquids. In this case, however, as only a short time was required for measurement, a standard demountable cell was used. Because ethanol and anhydrous ethanol were the subjects of analysis, a KRS-5 (thallium bromide-iodide crystals) plate was used. (KBr plates tend to fog up if the analyzed solution contains water.) The analytical conditions specified by the Japanese Pharmacopoeia were used, and measurement was performed in a wavenumber range of 4,000 to 400 cm⁻¹. Details are given in Table 1.



Fig.1 Ethanol and Ethanol Anhydrous



Fig.2 Demountable Cell

Table 1 Analytical Conditions

Resolution : 2 cm⁻¹
Accumulation : 45
Detector : DLATGS

■ Analysis of Ethanol

Fig.3 shows the results of analyzing ethanol using a demountable cell (KRS-5 plate). Background measurement was performed with just one plate. One drop of ethanol was dripped onto this plate, another plate was placed on top so that the drop was enclosed between the plates, and measurement was performed.

Note: Perform the above procedure quickly. If the second plate is not placed over the ethanol immediately, the ethanol will start to evaporate and it may not be possible to obtain good spectra.

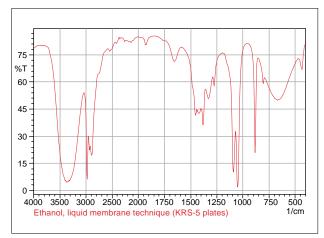


Fig.3 Spectrum of Ethanol

■ Analysis of Anhydrous Ethanol

Anhydrous ethanol was analyzed with a demountable cell using the same method that was used for ethanol. Fig.4 shows the spectrum that was obtained. Fig.5 shows the spectra for ethanol (Fig.3) and anhydrous ethanol (Fig.4) together. On comparing the spectra, it can be seen that absorption is particularly weak for anhydrous ethanol in a neighborhood of 1,660 cm⁻¹ due to the absence of water. (Absorption in a neighborhood of 1,660 cm⁻¹ corresponds to the peak caused by the H-O-H bending vibration of water.)

In this way, identification tests for ethanol and anhydrous ethanol can be performed using a demountable cell with KRS-5 plates.

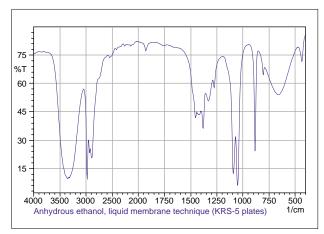


Fig.4 Spectrum of Anhydrous Ethanol

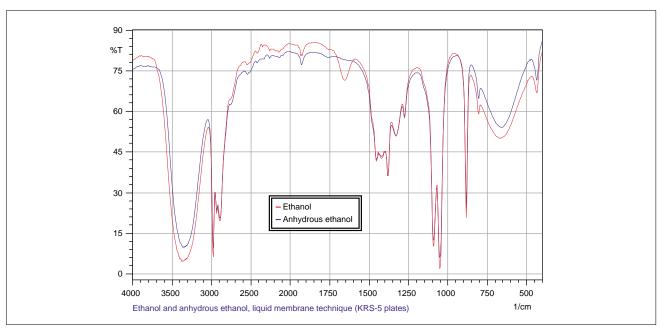


Fig.5 Spectra of Ethanol and Anhydrous Ethanol

NOTES:

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