

Applications of the DuraSAMPLIR employing a KRS-5 focusing element

The effectiveness of the DuraSAMPLIR as a single reflection ATR system has been described a number of times. In the systems described so far, the focusing element beneath the diamond prism was made of ZnSe. However, on this occasion, the DuraDisk - which uses a focusing element made of KRS-5 instead of ZnSe - makes its appearance. The infrared light transmission wavenumber region is much wider when the KRS-5 focusing element is used. Whereas the wavenumber range was limited to about 4000cm^{-1} to 600cm^{-1} with the former, the latter enables

measurement from 4000cm^{-1} down to 400cm^{-1} . As a result, by harnessing the usual performance of the DuraSAMPLIR, the measurement range can be further extended to 400cm^{-1} by employing the KRS-5 DuraDisk.

In practical terms, it is effective in confirmatory tests complying with the Japanese Pharmacopoeia where the wavenumber range is set from 4000cm^{-1} to 400cm^{-1} , and for identifying substances in which absorption can be confirmed between 600cm^{-1} and 400cm^{-1} . Examples of these measurements are introduced below.

■ Outline of the DuraDisk with a KRS-5 focusing element

Fig.1 shows the optical system of the DuraSAMPLIR. In the system introduced here the focusing element beneath the diamond prism is made of KRS-5.

Fig.2 shows the throughput for the ZnSe DuraDisk and the KRS-5 DuraDisk. Despite its high overall throughput, the transmittance for the ZnSe(a) below 500cm^{-1} is 0, while the transmission properties of KRS-5(b) are evidently flat down to 400cm^{-1} . Each of these disks can be exchanged for use in a manner suited to objectives.

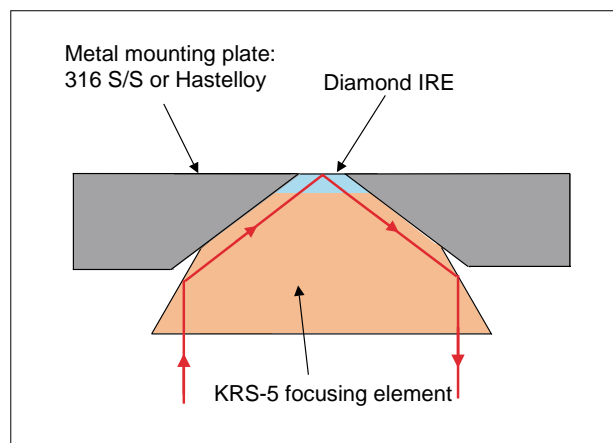


Fig.1 Optical diagram of DuraDisk

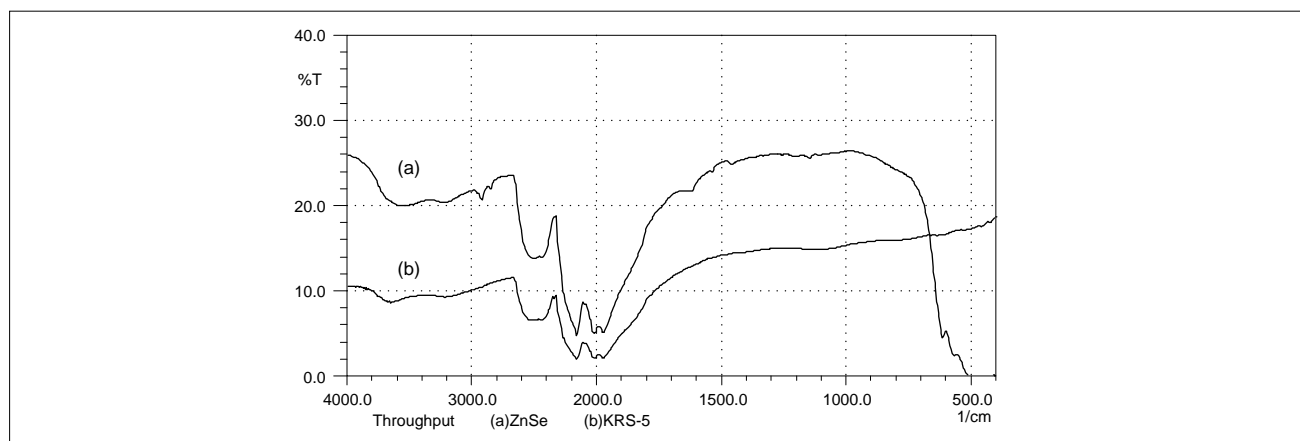


Fig.2 Throughput of each DuraDisk

■ Measurement of Diphenhydramine Hydrochloride

The potassium chloride (KCl) disc method is prescribed as the confirmatory test method for diphenhydramine hydrochloride in the 14th Revision of the Japanese Pharmacopoeia. This takes into consideration the fact that, with the traditional potassium bromide (KBr) disc method, an ion exchange reaction occurs while the tablet is being created, which results in a change in the infrared spectrum. Fig.3 is the infrared spectrum of diphenhydramine hydrochloride measured using a KRS-5 DuraDisk. It is evident that a good spectrum has been obtained.

It is necessary for methods that are not regulated by the pharmacopoeia to be sufficiently validated if they

are to be used. It is clear that this method, which requires no pretreatment whatsoever, can also be applied as a method that is outside of the regulations.

References

14th Revision of the Japanese Pharmacopoeia (2001), edited by the Society of Japanese Pharmacopoeia

Table 1 Analytical Conditions

Resolution	: 4cm ⁻¹
Accumulation	: 40
Detector	: DLATGS

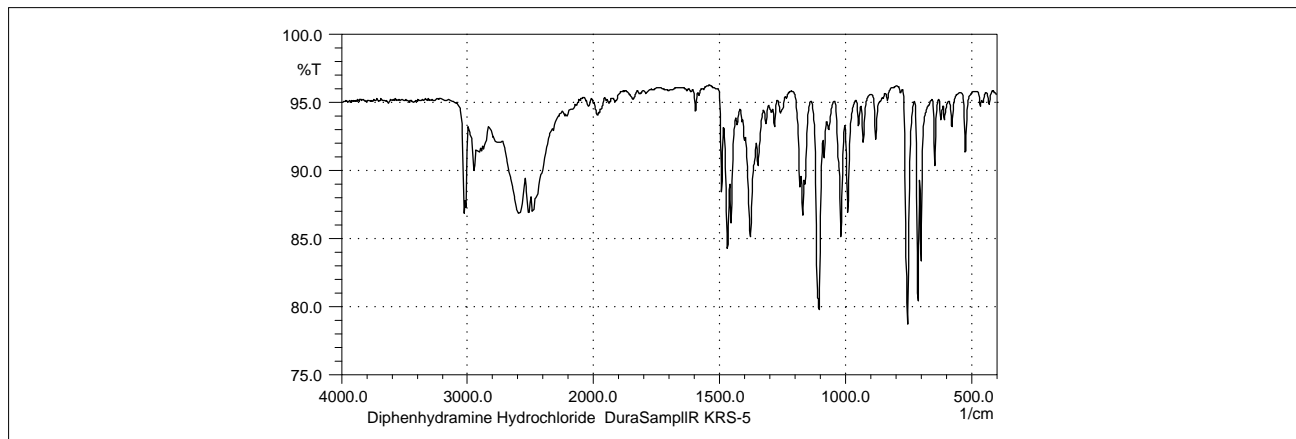


Fig.3 ATR spectrum of Diphenhydramine Hydrochloride

■ Measurement of Polymers

As a sample where absorption can be confirmed between 600 and 400cm⁻¹, 2 types of polymers were measured under the analytical conditions described above. Fig.4 is the spectrum of FEP (Tetrafluoroethylene Hexafluoropropylene-copolymer), and Fig.5 is the spectrum of PVDC (Polyvinylidene Chloride). Characteristic absorption in the low

wavenumber regions can be identified for both of them. In this way, the DuraSamplIR employing the KRS-5 element is effective for the purposes of qualitatively determining compounds with absorption in the low wavenumber regions without pretreatment.

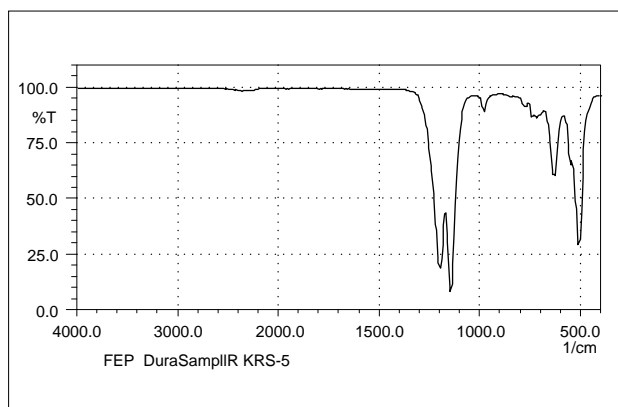


Fig.4 ATR spectrum of FEP

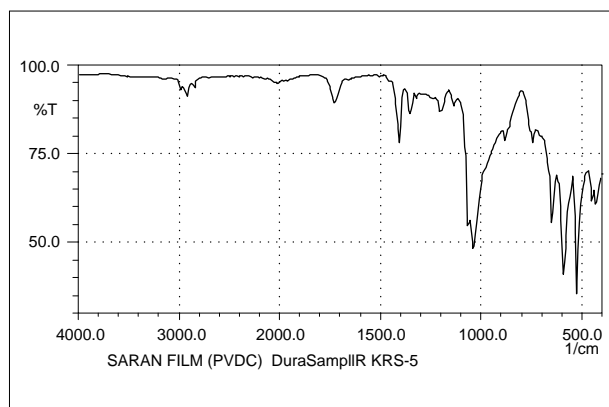


Fig.5 ATR spectrum of PVDC



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