

## Measurement of Water in Silica Gel with DSC

Silica gel is a porous and, therefore, highly hygroscopic substance with a lot of silanol groups on its surface. That is why it is used for hygroscopic agents, as well as packing materials for chromatographic columns.

The water existing at portions containing less water takes a form of combined water or non-freezing water which has strong binding power in silica gel. As it increases, it is retained in the numberless pores in a form of pore water, which has comparatively greater binding power. When it further increases until pores are full, free water with less retention strength increases in the outer portion of silica gel. When silica gel containing absorbed water is heated from a low temperature with DSC, it generates a peak corresponding to the fusion of water. It is expected that the study and research of fusion peaks of water will clarify the three-dimensional structure of silica gel, testifying the present theory that the form of retained water is closely related with its fusion peak.

The following is a comparison measurement of fusion peaks of three different kinds of silica gel with water adsorbed in it.

Fig 1 is DSC curves of silica gel with different water contents. Fusion peak is not observed in the curve A for water contents of 8.9% as molecules of water are firmly bound to the surface of silica gel. There is a peak at  $-5^{\circ}\text{C}$  in the curve B for water content of 35.1%. This peak corresponds to the fusion of water in the pores. In the curve for water contents of 73.3%, two fusion peaks are observed, one of which is a fusion peak (at  $-4.2^{\circ}\text{C}$ ) of water in pores and another fusion peak (at  $0.7^{\circ}\text{C}$ ) of free water in outer portion of silica gel. Water contents and peak areas are plotted in Fig.2. In this, there is no peak in the concentration range of 0 to 0.2 (equivalent to 0 to 16.7%). In the range of 0.2 to 0.9 (equivalent to 16.7 to 47.4%), peak of pore water increases until 0.9 where pores become full, and then, the peak of free water continues increasing thereafter.

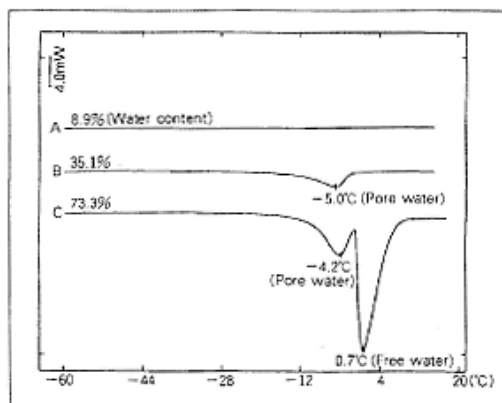


Fig.1 DSC Curve of Water Fusion in Silica Gel

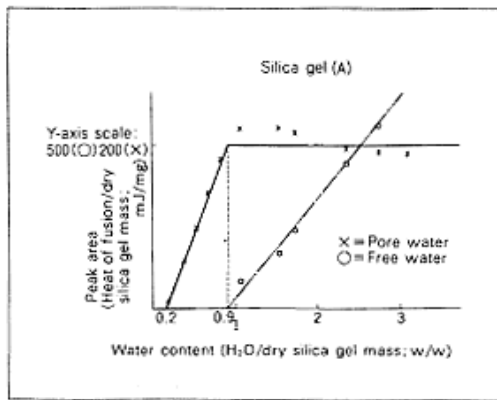


Fig.2 Plotting Curve of Water Content versus Fusion Peak Area

Fig.3 is a sketch of surface structure of silica gel of water content of 0.9 or more.  $\circ$ ,  $\odot$ , and  $\times$  represent bound water, pore water, and free water respectively.

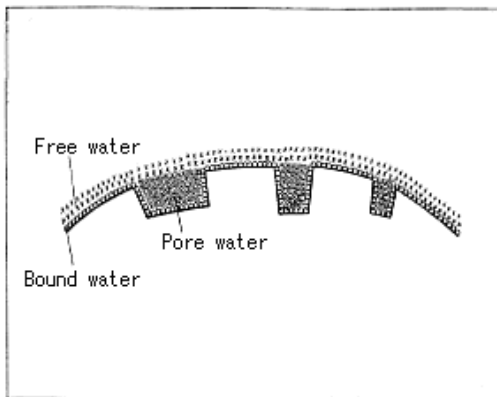


Fig.3 Sectional Sketch of Silica Gel Surface

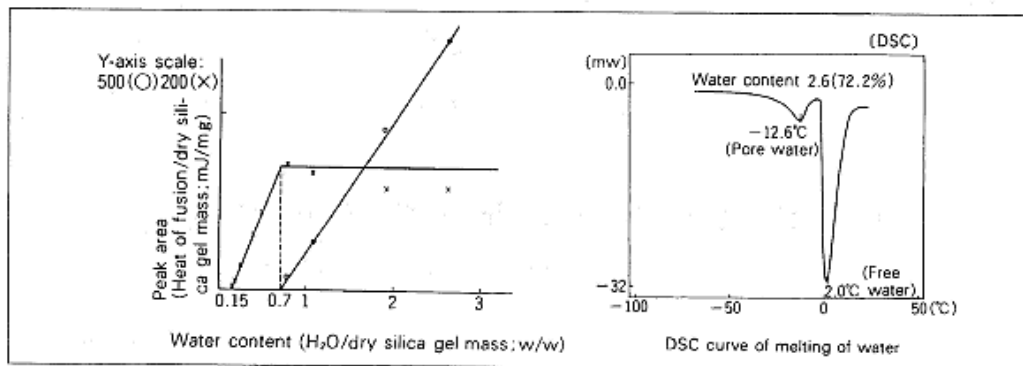


Fig.4 Result of Measurements of Silica Gel (B)

Figs. 4 and 5 show a result of measurement of silica gel (B) and (C) tested with different water contents as (A). A remarkable difference is observed between the values of peak areas for (B) and (C) when they become constant. Seemingly, the difference corresponds to pore cubatures or the mean value of pore diameters. Furthermore, the difference in fusion peak temperatures in DSC curves, which are  $-4.2^{\circ}\text{C}$  in (A),  $-12.6^{\circ}\text{C}$  in (B), and  $-2^{\circ}\text{C}$  in (C), is also considered to be in close relation to the mean value of pore diameters. Table 1 shows test result with DSC and data of pores of the three different specimens measured in gas adsorption method.

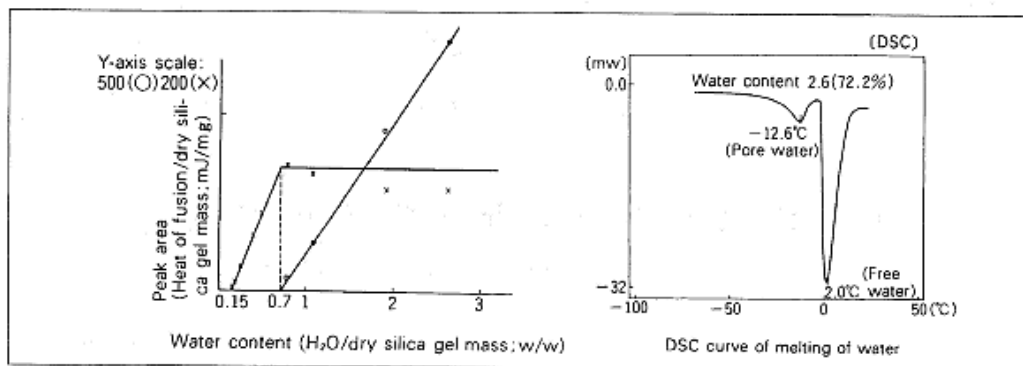


Fig.4 Result of Measurements of Silica Gel (B)

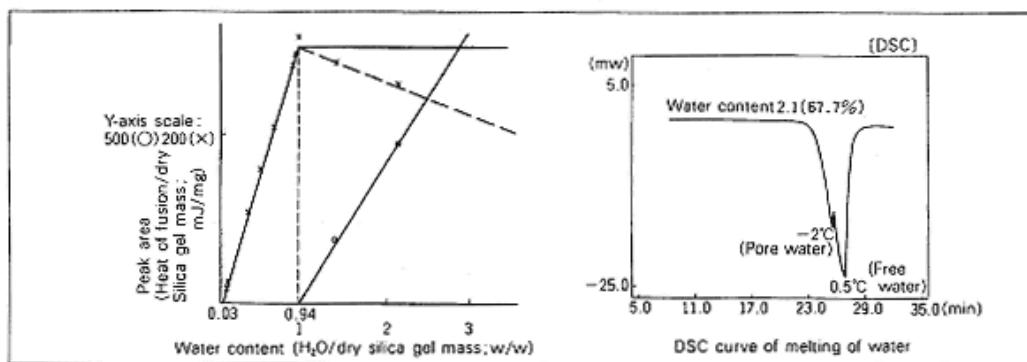


Fig.5 Result of Measurements of Silica Gel (C)

Table 1 Comparison of Measurement Result

Specimens	Fusion of pore water	Fusion of pore water	Pore Cubature (cc/g) in gas adsorption method	Mean Pore Diameter (Å) in gas adsorption method
	Max. peak area	Peak temperature		
	(mJ/mg)-DSC	(°C)-DSC		
B	140	-12.6	0.406	56
A	200	-4.2	0.833	89
C	304	-2.0	1.058	216

\* Please be advised that data obtained before the implementation of the current Weights and Measures Law may be presented in terms of gravimetric unit.

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