

## THE HYDROXYL CONTENT OF SILICA GEL

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Thermogravimetric analysis of silica gel has shown that the loss in weight between 30° and 910°C can be quantitatively explained on the basis of water being lost from three distinct and different populations of sites on the silica gel surface. The results indicate that the site energies of the three different populations are randomly distributed and, consequently, the resulting weight loss steps from each population can be described by the integral of a simple normal distribution with temperature. The calculated weight loss obtained by assuming three different site-groups having randomly distributed adsorption energies is, within experimental error, coincident with the experimental data. It is also shown that the water evolved from the second population of sites originates from strongly bound water and may also contain water generated by the condensation of (geminal) silanol groups contained in the overlapping and neighbouring population.

**Keywords:** silica gel, TG

### Introduction

Silica gel has been the subject of a considerable amount of research over the past two decades. One of the points of interest has been the nature of the surface. It is clear that the surface must consist of silanol groups, but these could be of three types: mono, geminal and (unusually) three hydroxyl groups joined to a surface silicon atom. The existence of the first two is well established [1]: no evidence is available for the existence of the third. In addition to these surface silanol groups, silica gel can adsorb water [2, 3], and it is in the nature of this adsorbed water where controversy exists [4, 5]. It is well known that silica gel will adsorb water and this is readily lost on heating to ca. 120°C [2, 3]. Further heating gives a weight loss which has been ascribed to loss of water by the condensation of single or geminal silanol groups. Recent work has demonstrated that silica gel can have water hydrogen-bonded to the surface which is lost progressively on heating to 180°–200°C [6, 7]. Further evidence exists to suggest that even on heat-

ing to higher temperatures [8–10], water can still be lost which is not due to the condensation of surface silanol groups [11–14].

This paper describes how thermogravimetry has been used to elucidate the nature of the losses obtained on heating silica gel over the temperature range 30°–910°C.

## Theory

During thermogravimetric analysis of silica gel it has been shown that there is a continual loss of water over the temperature range 30° to 1050°C [10]. Water can be held on the surface of the silica gel by different interaction forces: 'weak polar forces' (e.g. physically bound water). 'strong polar forces' (e.g. hydrogen-bonded water), both of which may be removed by heating, or, on stronger heating water can be lost by condensation of silanol groups to siloxane bonds. As a result of these interactions between the adsorbed water and the silica surface, each molecule has a potential energy of adsorption. Furthermore, the surface silanol groups must acquire sufficient kinetic energy before they can condense to siloxane bonds and generate water. As the temperature is raised, so the kinetic energy of the adsorbed water and silanol groups increases, and when the energy is equivalent to, or greater than, the potential energy of adsorption or chemical combination, water will leave the surface in vapour form.

Thus there may be different sources of water associated with the silica gel surface which might be considered as groups of adsorbed or combined water, within which the potential energies of adsorption for each water molecule will be similar but not necessarily identical to one another. Conversely, between groups, the mean potential energy of the adsorbed or combined water may be widely different as the temperature range over which all the water is evolved is large. The distribution of adsorption energy within a group could take one of many forms; however, due to the irregular nature of the silica gel surface, it is likely that the distribution of energy about a mean would be random in nature and thus could be appropriately described by a normal distribution. This can, for convenience, be put in a slightly modified form of the traditional equation, *viz.*,

$$n_t = A e^{-\frac{(T-T_x)^2}{2T_s^2}} \quad (1)$$

where  $n_t$  is the number of adsorbed or combined water molecules having energy,  $e_t$  at temperature  $T$  (where,  $e_t = jT$ , and  $j$  is a constant),

$T_x$  is proportional to the mean energy,  $e_m$  (thus,  $e_m = \psi T_x$ ),

$T_s$  is the standard deviation of the energy distribution,

$\psi$  is a constant.

Finally, the mass of molecules having energy,  $e_t$ , at temperature,  $T$ , will be  $\alpha n_t$ , where  $\alpha$  is the weight of one molecule.

Thus the total weight lost from a given group of similarly adsorbed molecules,  $m_1$  will be given by:

$$m_1 = \int \alpha_1 n_t dn_t = \int A \alpha_1 e^{-\frac{(T-T_x)^2}{2T_s^2}} dT \quad (2)$$

For  $r$  different groups, the total mass lost,  $M$ , during TG will be:

$$M = m_1 + m_2 + m_3 + \dots m_r = \sum_1^r \alpha_r \int A_r e^{-\frac{(T-T_{x_r})^2}{2T_{s_r}^2}} dT \quad (3)$$

Furthermore, assuming that the temperature is linearly programmed, the mass lost up to any arbitrary temperature  $T$ ,  $M_T$ , will be given by:

$$M_T = m_{1T} + m_{2T} + m_{3T} + \dots m_{rT} = \sum_1^r \alpha_r \int_{T_0}^T A_r e^{-\frac{(T-T_{x_r})^2}{2T_{s_r}^2}} dT \quad (4)$$

The above discussion assumes the following possibilities:

- i) There may be more than one type of process that holds water on the surface of silica gel.
- ii) The distribution of site energy for any one type of retaining process may be random and can be described by a simple normal distribution.
- iii) Temperature may be used as a parameter that is proportional to the kinetic energy of adsorbed water molecules.

These possibilities can be easily and elegantly tested using the data obtained from the normal TG curve and Eq. (4). To help in the mathematical iteration process, the differential form of the TG curve can provide useful reference temperatures. Employing a simple iterative curve-fitting computer programme, values for the constants  $\alpha_r$ ,  $A_r$ ,  $T_{x_r}$  and  $T_{s_r}$  can be identified for each desorption process that give the minimum error between the calculated and measured values given in the TG curve.

Optimum values for  $T_{x_r}$  and  $T_{s_r}$  are obtained by searching over a range of values that embrace those suggested by the minima shown in the DTG curves (Figs 1 and 2). The number of minima also helps in deciding the number of desorption processes to consider. However, if the distribution of group adsorption site energies overlap closely, there may be more groups than those suggested by the DTG curve.

There is little value in identifying approximate starting values for  $A'_r$  (where  $A'_r = \alpha_r A_r$ ), so in the initial iteration steps a wide range of values for these variables is examined. If the minima in the DTG curve are well separated, then it is possible to carry out the iterative curve-fitting process separately for each adsorption group over the appropriate temperature range (such is the case in Fig. 1). In

the example given, it was found possible to curve-fit the data for the first desorption step (30°C to 180°C) and calculate the mass lost from the first desorption. The data can then be subtracted from the total TG data and the residual data (for the other desorption groups only) curve fitted to the next desorption processes and the procedure repeated.

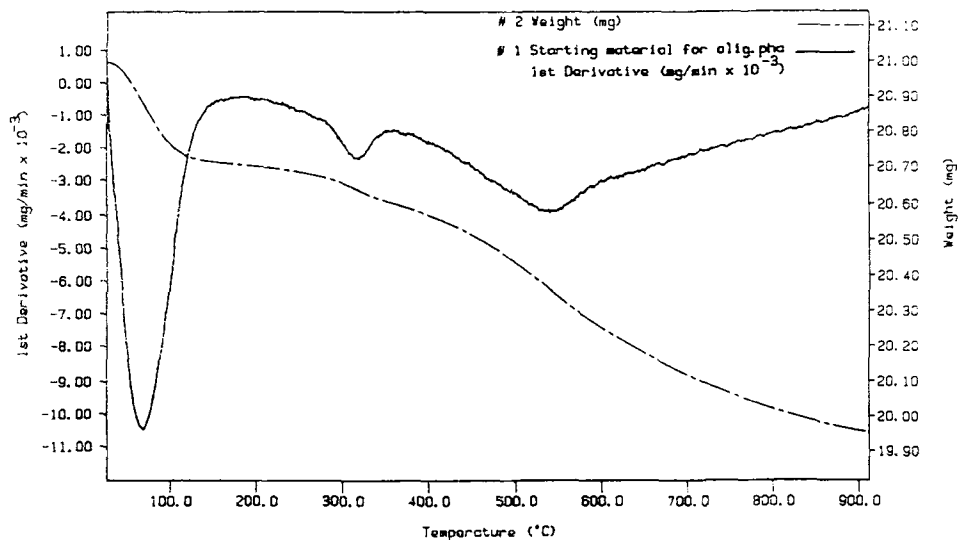


Fig. 1 TG and DTG curves for the first desorption of silica gel

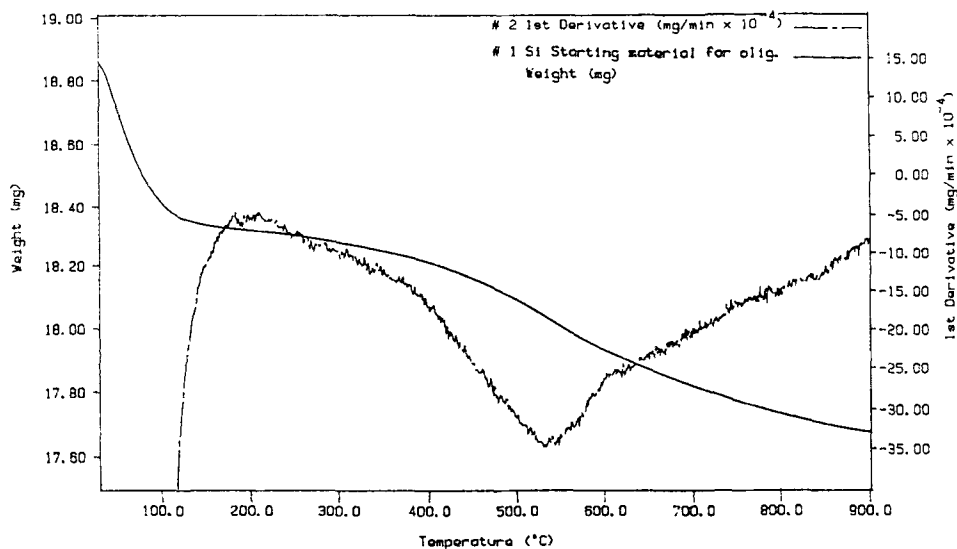


Fig. 2 TG and DTG curves for the second desorption of silica gel

## Experimental

### *Thermal analysis*

The silica gel used was Matrex 20  $\mu\text{m}$  LC silica gel (pore volume  $0.95\text{ cm}^3$ , surface area,  $S_{\text{BET}}$ ,  $250\text{ m}^2\text{ g}^{-1}$ ). Some samples were taken directly and not pretreated in any way, others were equilibrated over water. Thus the initial quantity of 'physisorbed water' on the silica gel would vary from sample to sample. The apparatus used was a Perkin Elmer, TGA 7 Thermogravimetric Analyser. Sample sizes varied between 15 and 25 mg and the heating rate was  $2\text{ deg}\cdot\text{min}^{-1}$  from an initial temperature of  $30^\circ\text{C}$  to a final temperature of  $910^\circ\text{C}$  (the maximum working temperature of the TGA7 under our conditions). A nitrogen pure flow of  $30\text{ cm}^3$  per minute was employed and seven replicate samples of silica gel were examined. Two were selected for further examinations. The first was the TG curve that gave the best resolution between different populations of desorbable water (Fig. 1) and the second the TG curve that gave the worst resolution between the different populations of desorbable water (Fig. 2) i.e. one that had been equilibrated over water. A digital output was obtained for each analysis which was subsequently used in the curve-fitting computer program.

### *Mass spectrometric measurements*

In order to ascertain the nature of the surface after heating at  $180^\circ\text{C}$  a 1 gm sample of the silica gel contained in a glass tube was heated for 2 hours at  $180^\circ\text{C}$  in a current of dry nitrogen.  $5\text{ cm}^3$  of 5% v/v of dimethyloctylchlorosilane in dry xylene was then added to the tube and the tube sealed. A blank tube containing only the silylating solution was treated in similar manner. The tubes were then heated for two hours at  $120^\circ\text{C}$  to complete the reaction. The sample tube and the blank were opened, after cooling, under anhydrous conditions and excess phenol added as a 5% g/v solution in dry xylene to remove excess silylating reagent. The resulting mixture was then filtered and the sample and blank filtrates sealed in glass tubes. The sample and blank filtrates were analysed by mass spectrometry using fast atom bombardment (FAB) as the ionizing process. Spectra taken over the relevant mass range for sample and blank solutions are shown in Figs 3 and 4 respectively.

## Results and calculations

The DTG curve (Fig. 1) shows three minima, this suggesting the existence of three distinctly different sources of desorbable water. Although obtained from the same material but of slightly different sample size, the DTG curve for the second sample (Fig. 2) shows only two distinct minima and a shoulder that might be interpreted as a third minimum. The first set of data was examined using an iterative curve-fitting procedure. Digital data from the TG curve taken every  $5^\circ\text{C}$  were

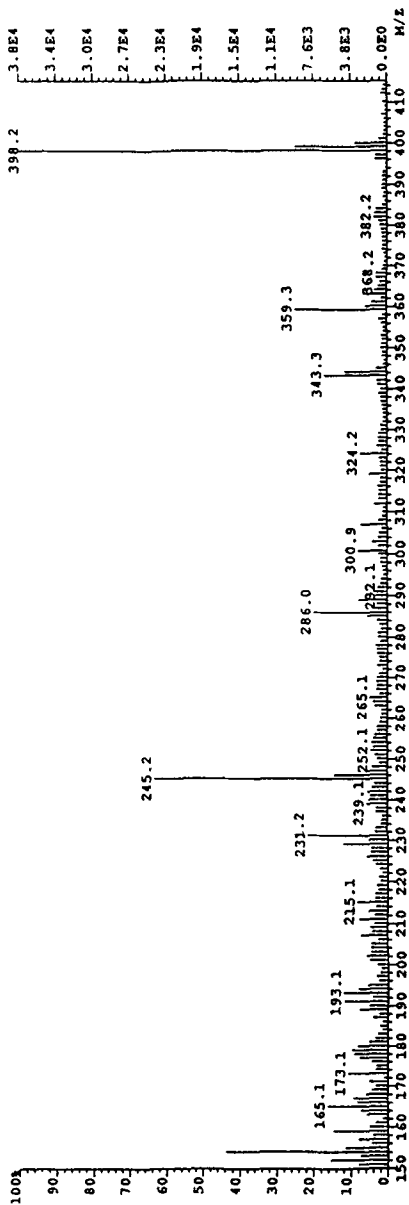


Fig. 3 Mass spectrum of evolved gas following chlorosilane treatment of silica gel after heating to 180°C

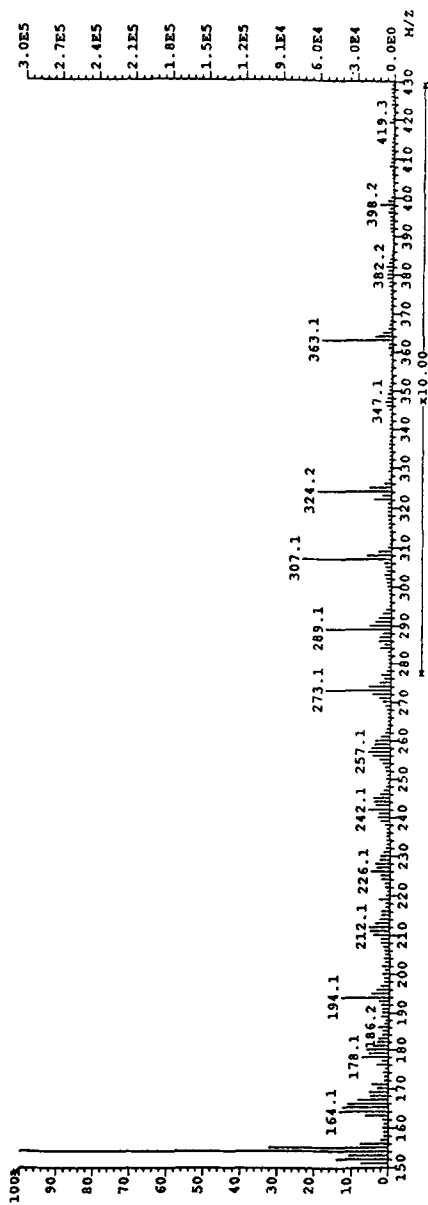


Fig. 4 Mass spectrum of blank

read into the program and all values subtracted from the first value, providing the weight lost every 5°C. Equation (2) was then fitted to the data between 30° and 180°C (thus excluding the range over which the second and third desorption took place) and the values of  $T_x$ ,  $T_s$  and  $A'$  identified that would provide the minimum sum of variances between calculated values and experimental data (Table 1). The iterative curve-fitting procedure was initiated by searching for the optimum value of  $T_x$  around 80°C and for  $T_s$  around 25°C which were deduced from the DTG curve given in Fig. 1. The optimum values of  $A'$ ,  $T_x$  and  $T_s$  obtained in this way for the first stage of water evolution are given in Table 1. The graph of site distribution as given by Eq. (2) is shown as the first error function curve in Fig. 5.

Calculated values of weight loss due to the first evolution of water were then subtracted from the experimental data, giving the TG data for the weight loss due to the second and third sources of desorbed water. In a similar manner, Eq. (3) was then fitted to the data for the second desorption between 180° and 350°C and the values of  $T_x$ ,  $T_s$  and  $A'$  identified that would provide the minimum sum of variances between calculated values and experimental data for the second desorption process. The iterative curve-fitting procedure was initiated by searching for the optimum value of  $T_x$  around 300°C and for  $T_s$  around 50°C, which were again deduced from the DTG curve given in Fig. 1. The optimum values of  $A$ ,  $T_x$  and  $T_s$  obtained in this way for the second stage of water evolution are included in Table 1. The graph of site distribution as given by Eq. (1) for the second desorption process is shown as the second normal distribution curve in Fig. 5.

**Table 1** Results from curve fitting procedure on data from 1st thermogravimetric analysis

Desorption group	Proportionality constant (A)	Temperature equivalent to mean site energy ( $T_1$ )	Standard deviation of site energy distribution ( $T_2$ )
1	0.0220	75.15	26.6
2	0.00382	270	46
3	0.00912	584	146

Total number of measurements taken 176; sum of variances between calculated and experimental results 0.01904.

Again, calculated values of the weight loss due to both the first and second evolution of water were then subtracted from the experimental data giving the TG data for the weight loss due to third source of desorbed water only. The same procedure was adopted, Eq. (2) was then fitted to the data for the third desorption sources between 350° and 910°C (the range over which the third desorption only took place) and the values of  $T_x$ ,  $T_s$  and  $A'$  identified that would provide the minimum sum of variances between the calculated values and the experimental data for the third desorption process. This time the iterative curve-fitting procedure was initiated by searching for the optimum value of  $T_x$  around 600°C and for  $T_s$  around 150°C which were, as before, deduced from the DTG curve given in

Fig. 1. The optimum values of  $A$ ,  $T_x$  and  $T_s$  obtained in this way for the third and last stage of water evolution are also included in Table 1. The graph of site distribution as given by Eq. (2) for the third desorption process is shown as the third and last normal distribution curve given in Fig. 5.

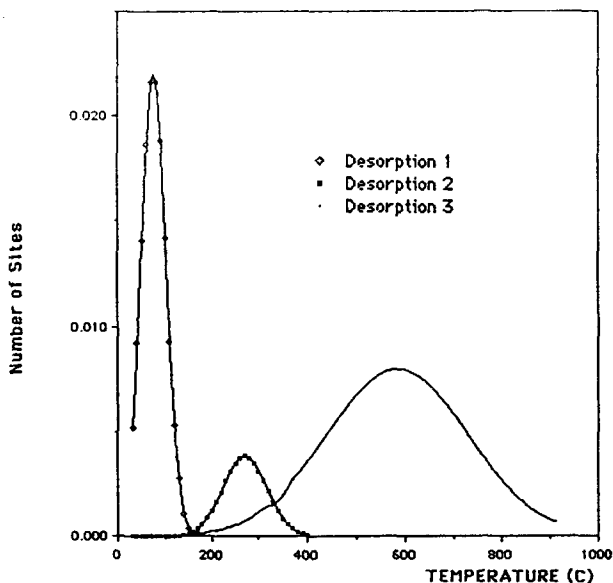


Fig. 5 Site energy dispersion for each desorption population temperature. Run 1

Using Eq. (3), the integral curves for the three desorption processes were calculated and are shown in Fig. 6. The height of the steps of these curves gives the weight loss by each process. Summing the three curves given in Fig. 6 provides the theoretical thermal analysis curve. This calculated curve is shown in Fig. 7 and the experimental points are included on the same graph. In Fig. 8, calculated values for the mass lost from the silica gel are plotted against the actual weight loss. It can be seen that the expected straight line has a slope very close to unity (0.9978), a nearly zero intercept (0.021) is obtained, and that the correlation between calculated and actual weight loss is excellent. The sum of the squares of the difference between experimental and calculated values was also determined (Table 1).

The second set of data was examined, again using an iterative curve-fitting procedure but in a slightly different way. The digital data from the TG curve taken every 5°C were again read into the program and all values subtracted from the first value providing the weight lost every 5°C. Due to the poor resolution obtained from TG, as shown by the weight loss curve in Fig. 2, a different curve-fitting procedure was adopted. The values for  $T_x$  and  $T_s$ , for all three populations



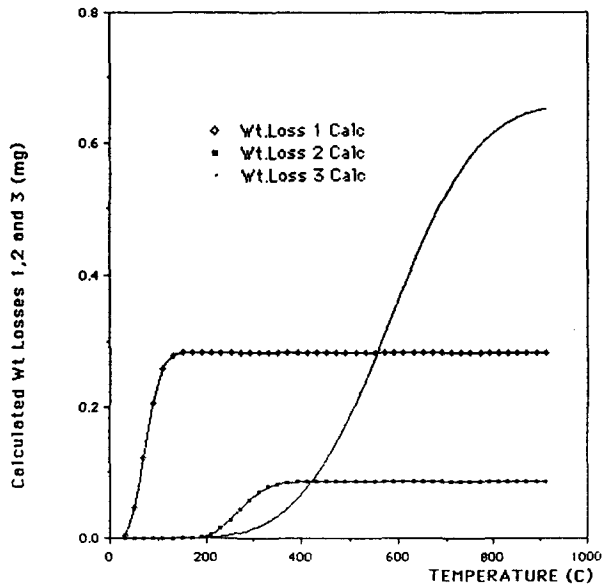


Fig. 6 Weight loss vs. temperature for the three desorption processes. Run 1

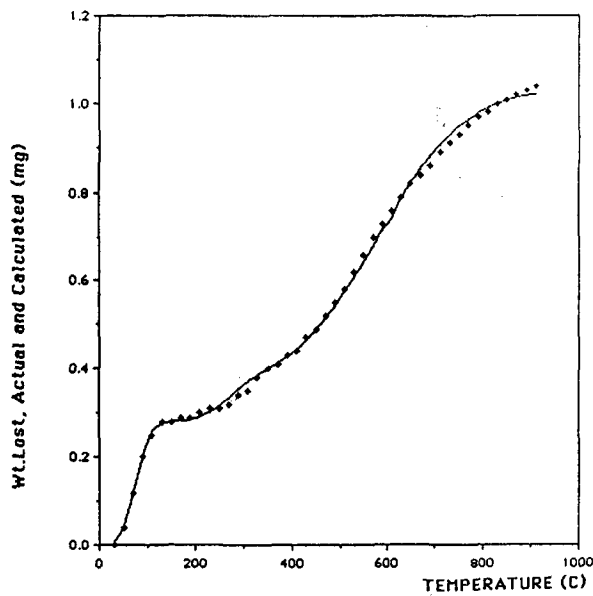


Fig. 7 Comparison of actual and calculated weight loss vs. temperature for run 1

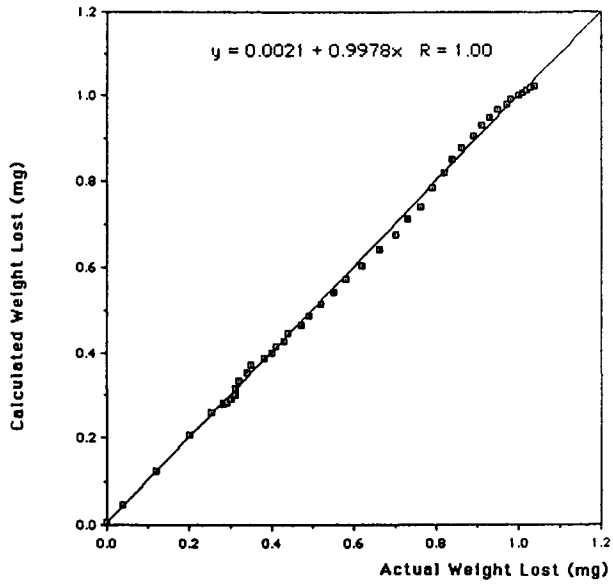


Fig. 8 Comparison of actual vs. calculated weight loss for run 1

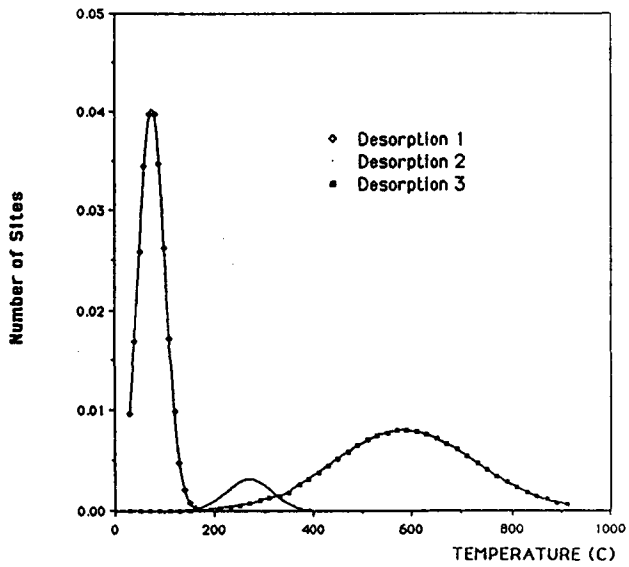


Fig. 9 Site energy dispersion for each desorption population temperature. Run 2

were taken from the first curve-fitting procedure and the three values for  $A'$  found by fitting Eq. (4) to the data over the entire temperature range ( $30^\circ$  to  $910^\circ\text{C}$ ). The three values for  $A'$  were identified that would provide the minimum sum of variances between the calculated values and the experimental data. The graph of site distribution against temperature, as given by Eq. (2), for all three desorption processes are shown in Fig. 9. This figure shows that the first population (that involving physically adsorbed water) is proportionally much greater than in sample 1, which accounts for the poor resolution between the first two site groups.

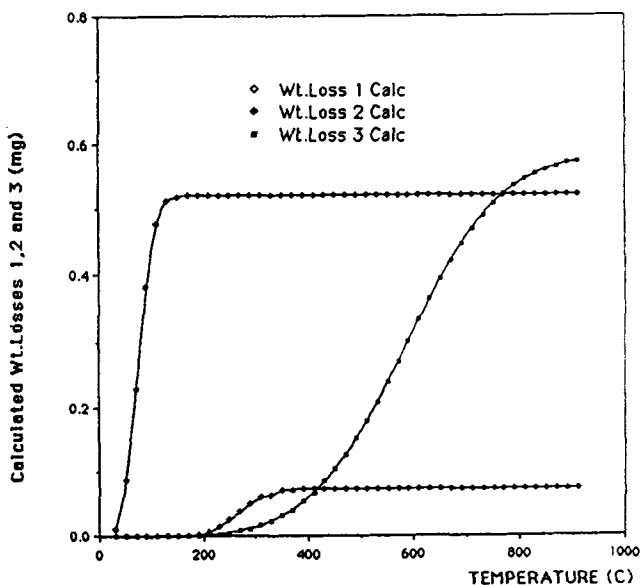


Fig. 10 Weight loss vs. temperature for the three desorption processes. Run 2

Using Eq. (3), the integral curves for the three desorption processes were calculated (Fig. 10). The height of the steps of these curves again gives the weight loss by each process. Summing the three curves given in Fig. 9 provides the theoretical thermal analysis curve and the calculated and experimental curves are shown on the same graph in Fig. 11. In Fig. 12, calculated values for the mass lost from the silica gel are plotted against the actual weight loss. It is seen that again the expected straight line is obtained having a slope very close to unity (1.0167) and nearly zero intercept ( $-0.017$ ). However, there is a significant deviation for the first three points of the graph, probably due to the silica being overloaded with water and, consequently, a finite time being required for the temperature program to reach a steady state. Nevertheless, it is seen that the correlation between the calculated and actual weight loss is excellent. The sum of the squares

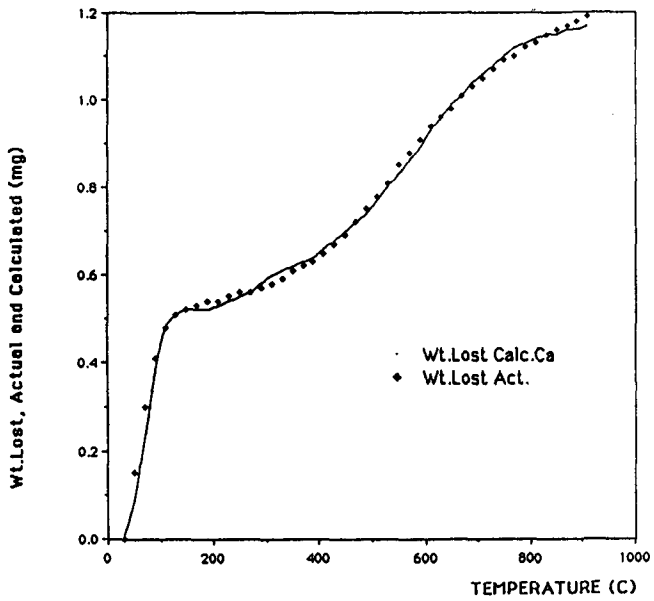


Fig. 11 Comparison of actual vs. calculated weight loss for run 2

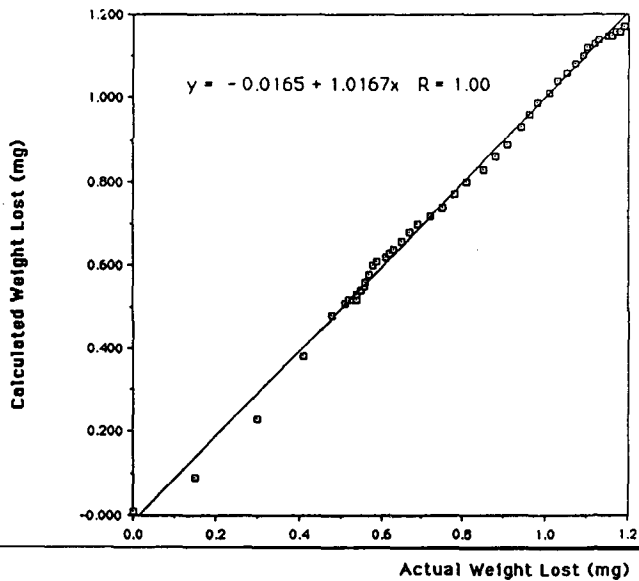


Fig. 12 Comparison of actual and calculated weight loss for run 2

**Table 2** Results from curve fitting procedure on data from 2nd thermogravimetric analysis

Desorption group	Proportionality constant (A)	Temperature equivalent to mean site energy ( $T_1$ )	Standard deviation of site energy distribution ( $T_2$ )
1	0.0405	75.15	26.6
2	0.00320	270	46
3	0.00798	584	146

Total number of measurements taken 176; sum of variances between calculated and experimental results 0.01925

of the difference between experimental and calculated values was also determined for the second TG experiment (Table 2).

## Discussion

### *Nature of the silica surface*

Data from the two thermogravimetric analyses can be explained on the basis that there are three distinct and separate sources of desorbable water on the surface of silica gel. Furthermore, the data fit the concept that the distribution of site adsorption energies are random for each desorbable water source. The mean energy of adsorption and the distribution of the adsorption energies about the mean are the same for the second and third desorption processes respectively in both analyses. However, the second sample (equilibrated over water) contains about twice as much physisorbed water as that on the first sample. Furthermore, the early part of the first desorption process (sample 2) in the TG curve (30°–90°C) differs slightly from the calculated weight loss. This difference could be due to desorption of multilayers of water physisorbed to the surface which, as the energy differences between each adsorbed layer is small, the theory presented treats as a single population.

The second desorption process is attributed to either condensation of geminal silanol groups to siloxyl bonds or to the desorption of strongly bound or hydrogen-bonded water. The mass spectra shown in Figs 3 and 4 show the presence of the bis(dimethyloctyl)siloxane+proton ( $M+1$ ,  $m/e$  359.3) in the sample but not in the blank. The ion at  $m/e$  359.3 is that which would be expected from the silyl ether should it be present. This indicates that the silica contained water when reacted with dimethyloctylchlorosilane. Prior to reaction, the silica had been heated to 180°C for 2 h to ensure the removal of all physisorbed water that was contained in the first group of desorbable water.

The persistence of water in the sample after heating to 180°C for 2 h, confirmed by the mass spectra, strongly indicates that the second desorption process involves the loss of hydrogen-bonded water and not the condensation of silanol groups, single, geminal or otherwise. This is supported by Sindorf and Maciel

[14] who concluded that there is likely to be free water still present on the surface of silica gel after heating in vacuo at 200°C. There is, however, some small loss of silanol groups between 180° and 350°C, and this is confirmed by the overlap of the second and third desorption groups shown in Figs 5 and 6. Loss of water from the second group is further substantiated by the fact that, after the removal of physisorbed water, only two resources of desorbable water appear to exist. As one of these is known to be due to condensation of silanol groups to siloxyl groups, the remaining source must be that of hydrogen-bonded or very strongly adsorbed water.

The presence of water on the silica gel surface gives concern as to the accuracy of the currently accepted value for the number of silanol groups per square metre of surface. The hydroxyl group content is obtained by measurement of the methane evolved when methyl lithium is allowed to react with the silica surface. The silica is heated to 120°C for some time before treatment and it is assumed that all free water has been removed in this way. From the present results (and those of Sindorf and Maciel [14]), it would appear that all hydrogen-bonded or strongly held water is not removed completely until the silica gel has been heated to about 400°C. Thus, some of the methane collected from the reaction with methyl lithium would have come from the adsorbed water and not from silanol groups. Consequently, a false, high reading for concentration of silanol groups is obtained and this effect is exacerbated by the fact that one molecule of water will give methane equivalent to two silanol groups. Unfortunately due to overlap of the population of hydrogen-bonded or strongly held water and the population of different types of silanol groups, the water can not be removed by heating without removing some of the silanol groups.

From the data given above, it is possible to calculate the number of surface silanols on silica gel:

Weight of silica taken	21.00 mg
Weight of water lost in the third desorption stage from condensation of silanol groups as determined by the iteration process.	0.65 mg
Weight of water lost in the second desorption stage from removal of hydrogen-bonded water as determined by the iteration process.	0.086 mg
Surface area of the silica gel (BET)	250 m <sup>2</sup> .g <sup>-1</sup>

Now the concentration of silanol groups on the surface,  $C$ , in  $\mu\text{mol}\cdot\text{m}^{-2}$  of surface can be calculated from the following equation:

$$C = m \cdot 10^6 / (mM_{\text{OH}}A) \quad (5)$$

where  $m$  is the mass of water lost during silanol condensation,  $m$  is the mass of the silica gel sample,  $M_{\text{OH}}$  is the molecular weight of a silanol group, and  $A$  is the surface area per gram of the silica gel.

In a similar way, the concentration of silanol groups on the surface,  $c$ , in  $\mu\text{mol}\cdot\text{m}^{-2}$  equivalent to the mass of hydrogen-bonded water lost can be calculated from the following equation:

$$c = 2 w 10^6 / (mM_{\text{H}_2\text{O}}A) \quad (6)$$

where  $w$  is the mass of water lost during the removal of hydrogen-bonded water,  $m$  is the mass of the silica gel sample,  $M_{\text{H}_2\text{O}}$  is the molecular weight of a water molecule, and  $A$  is the surface area per gram of the silica gel.

Employing Eqs (5) and (6) it can be shown that:

Concentration of silanol groups on the surface	7.28 $\mu\text{mol}\cdot\text{m}^{-2}$
Concentration of silanol groups equivalent to hydrogen-bonded water lost	1.93 $\mu\text{mol}\cdot\text{m}^{-2}$

Therefore silanol concentration if determined by the methyl lithium process would be

$$9.21 \text{ mmol}\cdot\text{m}^{-2}$$

The actual value for the silanol concentration as determined by the methyl lithium procedure on silica gel preheated to 200°C has been reported to be  $9 \mu\text{mol}\cdot\text{m}^{-2} \pm 1.0 \mu\text{mol}\cdot\text{m}^{-2}$  [15]. The calculated value of  $9.21 \mu\text{mol}\cdot\text{m}^{-2}$  agrees well with the experimentally determined value.

It must be emphasized that the accepted value for the silanol concentration on the surface of silica gel of  $9.0 \mu\text{mol}\cdot\text{m}^{-2} \pm 1.0 \mu\text{mol}\cdot\text{m}^{-2}$  [15] must be considered to be in error and should be reduced by the amount equivalent to the hydrogen-bonded water. Results obtained in this work indicate the true concentration of silanol groups on the surface to be  $7.3 \mu\text{mol}\cdot\text{m}^{-2} \pm 0.2 \mu\text{mol}\cdot\text{m}^{-2}$ .

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**Zusammenfassung** — Bei der thermogravimetrischen Analyse von Silikagel konnte gezeigt werden, daß der Gewichtsverlust zwischen 30° und 910°C quantitativ auf der Grundlage von Wasser erklärt werden kann, welches aus drei unterschiedlichen Populationen von Orten an der Silikageloberfläche stammt. Die Ergebnisse zeigen, daß die Energie dieser Orte der drei verschiedenen Populationen eine Random-Verteilung aufweisen und daß die resultierenden Gewichtsverlustschritte jeder Population mit einem Temperaturintegral über die Normalverteilung beschrieben werden können. Der berechnete Gewichtsverlust, den man unter der Annahme dreier verschiedener Ort-Gruppen mit randomverteilten Adsorptionsenergien erhält, stimmt innerhalb der Fehlergrenzen mit den experimentellen Angaben überein. Außerdem wird gezeigt, daß das aus der zweiten Population von Orten freigesetzte Wasser aus stark gebundenem Wasser stammt und kann auch Wasser enthalten, welches durch die Kondensation von (geminalen) Silanolgruppen entstanden ist, die in überlappenden und benachbarten Populationen enthalten sind.