Nomenclature

A', B' = Debye-Hückel constants on molality scale

a = ion-size parameter

m = molality of HCl

k = $2.303\,RT/F$, volt

$sE_m{}^\circ$, $sE_N{}^\circ$, $sE_c{}^\circ$ = standard electrode potentials on molality, mole fraction, and molarity scales in solvent, volt

$wE_m{}^\circ$, $wE_N{}^\circ$, $wE_c{}^\circ$ = standard electrode potentials on molality, mole fraction, and molarity scales in water, volt

M_{xy} = average molecular weight of the solvent (x = wt % diethylene glycol; y = wt % of water)

$s\gamma_\pm$ = mean activity coefficient of HCl in the solvent referred to solvent standard state

$\Delta G_t{}^\circ$, $\Delta H_t{}^\circ$, $\Delta S_t{}^\circ$ = free energies, enthalpies, and entropies of transfer on mole fraction scale, cal

$\Delta G_{t(c)}{}^\circ$ = standard Gibbs free energy of transfer on molarity scale, cal

$\Delta G_{t(el)}{}^\circ$, $\Delta H_{t(el)}{}^\circ$, $\Delta S_{t(el)}{}^\circ$ = electrostatic Gibbs free energy, enthalpy, and entropy of transfer, cal

$\Delta G_{t(nonel)}{}^\circ$, $\Delta H_{t(nonel)}{}^\circ$, $\Delta S_{t(nonel)}{}^\circ$ = nonelectrostatic (chemical) Gibbs free energy, enthalpy, and entropy of transfer, cal

$\log{}_m\gamma_\pm$ = primary medium effect

ϕ_w = volume fraction of water

K_s = $2\,R/F \ln (1000/M_{xy})$

K_w = $2\,R/F \ln (1000/18.016)$

θ = constant characteristic of the medium

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Adsorption Isotherms, Heats of Desorption, and Partial Molal Entropies for Carbon Monoxide on Linde 5A Molecular Sieve

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Adsorption parameters for individual gases on the various zeolites are sparse, despite their great utility as adsorbents (7, 10, 11, 13, 15–17). In particular, the published partial adsorption isotherms for CO on Linde Molecular Sieve 5A ($Ca_{4.5}Na_3[(AlO_2)_{12}(SiO_2)_{12}]\cdot nH_2O$) at 25°, 0°, and −75° and on Linde 4A ($Na_{12}[(AlO_2)_{12}(SiO_2)_{12}]\cdot nH_2O$) at 0° and −75° did not meet our needs in connection with the study of the separation of carbon isotopes by adsorption techniques. We present here adsorption isotherms for CO on Linde Sieve 5A for pressures ranging from 10^{-4} torr to several hundred torr at 77.3, 90.1, 145.0, 194.5, 230.9, and 273.2K. We also present "heat of desorption" data and partial molal entropy of the adsorbed CO derived from the adsorption isotherms.

Experimental Apparatus and Procedure

The experimental apparatus arrangement is indicated schematically in Figure 1. It contained the usual calibrated gas burets, A; a mercury manometer and its associated cathetometer, and a calibrated McLeod gage, B and C, respectively, for pressure measurement; CO supply

storage, D; a cooled adsorbent vessel, E, for final purification of the CO supply; a high vacuum system, F, to provide for evacuation of the adsorption system; and the adsorbent cell, H, and its constant temperature bath, I. The gas burets, the mercury manometer, and the McLeod gage were surrounded by a shield to provide an approximately constant (measured) temperature environment for these elements.

The adsorption cell was fashioned from a 1.75-cm i.d. stainless-steel tube (0.07-cm wall) with a concentric 0.48-cm o.d. inlet tube which extended for 5.4 cm into the 5.75-cm deep cell. The inlet tube was perforated by 20 holes 0.32-cm diam to facilitate CO distribution within the cell. A 20-mesh stainless-steel screen covered the inlet tube to prevent entry of adsorbent. The ~7.4-gram samples of $^1/_{16}$-in. diam Linde Molecular Sieve 5A pellets (as supplied commercially with clay binder) occupied the annular space of the cell. Two samples of sieve were studied. The first, of 7.58-gram desorbed weight, was used for adsorption isotherms at all temperatures. The system was then modified to provide a "trap" of several grams of pure gold foil to protect the adsorbent from mercury vapor diffusing into the sample cell from the ma-

Adsorption isotherms for carbon monoxide on Linde Molecular Sieve 5A were measured at 77.3, 90.1, 145.0, 194.5, 230.9, and 273.2K. The 77.3, 90.1, and 145.0K isotherms were nonreproducible for volumes of adsorbed CO less than characteristic critical amounts; for all other temperatures and for these temperatures when the CO adsorption exceeded these critical amounts, the adsorption isotherms were highly reproducible. Isotherms at and above 194.5K can be approximated by the expression $V = 94.2 \exp(-[RT \ln (f^*/f)/3780]^{2.8} - 0.006 [T - 194.5])$ where V is the volume of adsorbed CO, measured in cc at standard conditions, per gram of desorbed sieve; f, the fugacity of the CO vapor at the point in question; and f^*, the fugacity of saturated CO vapor at the temperature, T (for $T > T_{critical}$, f^* is obtained from an extrapolation of $\ln f^* = A + B/T$). Isosteric heats of desorption were a function of temperature and of the volume of CO adsorbed, ranging from 10,500 cal/mol for 0.1 std cc CO/g at 273K to 1920 cal/mol for 140 std cc CO/g at 77.3K. Partial molal entropies of the adsorbed CO were a function of temperature and the amount of CO adsorbed. For loadings between 20 and 60 std cc CO/g, typical values are 20 ± 2 cal/g mol K, which fall to a minimum of 6 cal/g mol K for 130 std cc CO/g at 77.3K before rising to 17 cal/g mol K for 140 std cc CO/g at 77.3K (compared to an entropy of 18.6 cal/g mol K for saturated liquid at 77.3K).

nometer and McLeod gage. A fresh sample of sieve (7.27-gram desorbed weight) was then used for confirmatory runs at 77.3 and 90.1K.

The CO adsorbate was obtained from cp grade (>99.5%) compressed gas (J. T. Baker Co.). This cp material was contacted at about 5 psig with silica gel at 195K (CO_2-trichloroethylene bath) in the purifier trap, E, before filling bulbs D to ~1 atm.

The mercury manometer was operated with the pressure meniscus at a fiducial mark to present a constant noxious or "dead" volume to the system. This noxious volume and the corresponding noxious volume, when the McLeod gage was connected to the system in lieu of the manometer, were determined from the observed pressure changes produced by displacing mercury in the accurately known volumes of the gas buret A, from the measured temperature of the system and the gas laws. The noxious volume with the mercury manometer in use was 49.7 cm³; that with the McLeod gage in use was 261.4 cm³. The CO contained within the noxious volumes amounted to roughly 7% of that adsorbed on the molecular sieve for pressures below 10 torr (McLeod gage in use) in the worst case (273K) and to ~12% for pressures above 10 torr (manometer in use) in the worst case (1130 torr, 273K). The CO inventories in the noxious volumes were known to ±2% and thus contributed negligible uncertainty to the determination of the isotherm.

Prior to measurement of an adsorption isotherm, the molecular sieve was regenerated by heating it at 350°C for at least 10 hr, and typically for 18 hr, while maintaining the pressure above the absorbent at less than 10-micro torr. Following regeneration, the adsorption cell was allowed to cool to room temperature and was then immersed in the desired constant-temperature bath liquid.

Adsorption isotherms were obtained by connecting the adsorption cell to the gas burets (and noxious volume) which contained an initial inventory of CO known from

$p \Sigma V/RT$ for the various volume elements. The adsorbent was allowed to equilibrate with the gas buret (and noxious) volumes. Equilibrium was usually attained within 30–60 min; the pressures were monitored typically for as long as 4 hr to insure that observations were equilibrium data. The amount of CO adsorbed was inferred from the change in $p \Sigma V/RT$ for all volume elements of the system. Adsorption isotherms were measured by adsorbing successive increments of CO until the equilibrium at the highest desired pressure was attained.

Isotherms were measured at 77.3, 90.1, 145.0, 194.5, 230.9, and 273.2K, the boiling temperatures of N_2, O_2, and CF_4, the sublimation temperature of CO_2 (in a C_2HCl_3 slurry), the boiling temperature of propane, and the ice point, respectively. The constant-temperature bath was closed by a loosely fitting cover, and a very small stream of gas of the constituent bath liquid was bubbled into the bottom of the liquid baths to prevent superheating or subcooling. The temperatures were obtained from the observed barometric pressure and the known vapor pressure-temperature relationships for the various liquids and varied from run to run by less than ±0.05K.

Upon completion of an isotherm, the cell was connected to the vacuum system, and the CO pumped off and discarded while the sieve was being heated to regeneration temperature. After regeneration of the sieve was complete, the remainder of the system was completely evacuated, the silica gel in the purifier trap was regenerated, and a fresh supply of CO charged into storage bulbs D. The isotherm was then repeated, or a new isotherm with a different bath liquid was then taken.

Measured pressures were corrected for the effects of thermal transpiration where necessary, using the correlation of Liang as modified by Bennett and Tompkins (2).

Adsorption Isotherms

The reduced experimental results (9) are shown in Figure 2 for loading greater than 1 std cc of CO/g desorbed sieve for all isotherms and in Figure 3 for loadings of less than 1.5 std cc CO/g sieve for the 194.5, 230.9, and 273.2K isotherms. At least two runs were made at each isotherm to check reproducibility. The isotherms at 194.5, 230.9, and 273.2K were reproducible over the full adsorption range to within the precision of pressure and adsorbate measurement; namely, ±30% for $p < 10^{-3}$ torr; ±5% for $10^{-3} < p < 1$ torr; and ±1% or better for $p > 3$ torr. For the 145.0K isotherm, the data are reproducible to ±1% for loadings greater than ~70 std cc/g; for loadings less than this, the isotherms fell within the broken lines indicated on Figure 2.

The 90.1K isotherms are reproducible to better than ±1% for CO loadings in excess of ~110 std cc/g, but
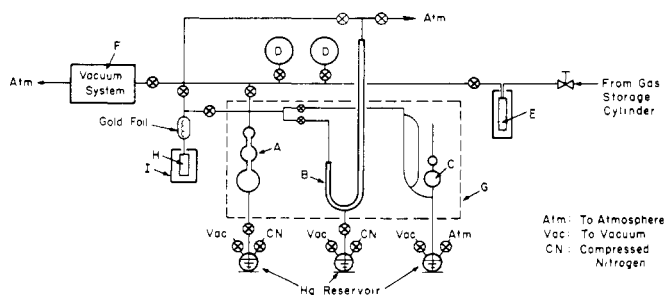

Figure 1. Schematic diagram of experimental apparatus

below this loading, scatter to the extent that the equilibrium pressure for a loading of 30 std cc/g varied by almost sevenfold. At 77.3K, isotherms are reproducible to better than ±1% for CO loadings in excess of 120 std cc/g, but below that scatter so that at a CO loading of 30 std cc/g, equilibrium pressures varied from run to run by an order of magnitude (extreme). The region and extent of the scatter in the nonreproducible cases are indicated by the hatched areas for the 77.3 and 90.1K isotherms on Figure 2.

This nonreproducibility of equilibrium pressure at "low" loadings was investigated at some length without satisfactory resolution. It appeared that the temperature of regeneration was an important variable. The equilibrium pressures at 77.3K for loadings below 100 std cc/g appeared to be minimized for regeneration temperatures lying somewhere between the ice point and 440°C. However, the effect of regeneration temperature upon the adsorption isotherm at low loading could not be reproduced with convincing precision. Egerton and Stone (8) have recently published data for adsorption of CO at 273K on Zn^{2+} substituted zeolite Y where the loadings for adsorption pressures in the range of 1–10 torr exhibit maxima for regeneration temperatures in the vicinity of 600°C and fall significantly for higher and lower regeneration temperatures.

The scatter in the 77.3, 90.1, and 145.0K isotherms at low loadings is not due to errors in "accounting" for the adsorbate; completely discordant low coverage results converged to better than 1% agreement as the loading was increased without any adjustment or correction of the original loading figures.
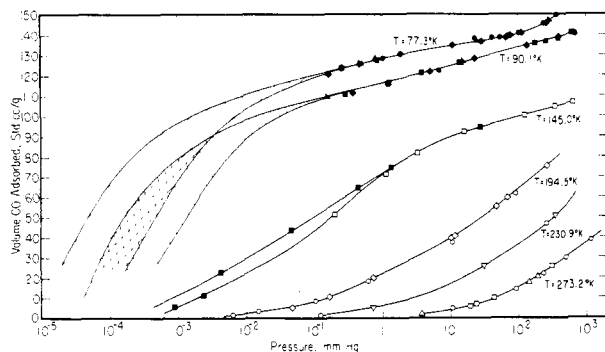

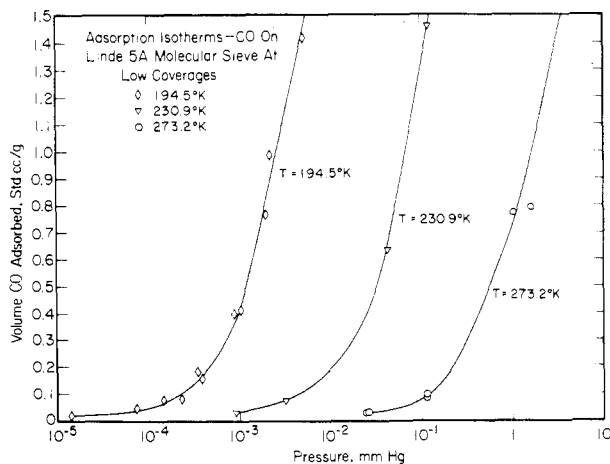**Figure 2.** Adsorption isotherms for CO on Linde 5A Molecular Sieve

Figure 3. Adsorption isotherms for CO on Linde 5A Molecular Sieve for loadings of less than 1.5 std cc CO/g desorbed sieve

"Heat of Desorption"

Slopes from plots of the logarithm of the equilibrium pressures for constant loadings vs. $1/T$ were used in the Clausius-Clayperon equation

$$\frac{\partial \ln P}{\partial (1/T)} \simeq -\frac{(H_{vap} - \bar{H}_{ads})}{R} \qquad (1)$$

to estimate the difference between the enthalpy of the CO vapor and the partial molal enthalpy of the adsorbate, $(H_{vap} - \bar{H}_{ads})_{CO}$, or the isosteric heat of desorption which we have called the "heat of desorption" in the absence of any knowledge of concurrent heat effects associated with the lattice. These results are plotted as a function of sieve loading for the various isotherms in Figure 4.

This determination of heats of desorption obviously becomes highly uncertain and arbitrary in the nonreproducible region of the low-temperature isotherms. Slopes along the high- and low-pressure boundaries of the nonreproducible regions of the isotherms were used to calculate "maximum" and "minimum" values for the heats of desorption in these regions. The spread of these values is indicated by the pairs of symbols connected by vertical dashed tie lines for the respective isotherms on Figure 4. The reasonableness of this procedure is supported by a single experiment in which the sample of sieve with a loading of ~30 std cc/g was equilibrated at 77.3K ($p = 6.9 \times 10^{-5}$ torr); the sample temperature was then raised to 90.1K and came to equilibrium at $p = 3.1 \times 10^{-4}$ torr, yielding a heat of desorption of 1630 cal/mol (solid hexagonal symbol, Figure 4) compared to an apparent average from the high and low limits of the 77.3 and 90.1K isotherms of 1200 cal/mol for this loading.

The dashed curve in Figure 4, drawn to connect the end of the curves for $(H_{vap} - \bar{H}_{ads})$ determined from the reproducible portions of the various isotherms, is speculative and has no basis other than that the 273, 231, and 195K isotherms merge as the coverage is increased and the locus of this common curve and that of the 77.3/90.1K common curve appear to intersect at the 145K isotherm.

Entropies of Adsorbed Phase

Partial molal entropies, as calculated from the known molar entropy in the gas phase at the equilibrium pressure for a given loading and from the heat of desorption for that loading

$$\bar{S}_{ads} = S_{vap} - \frac{(H_{vap} - \bar{H}_{ads})}{T} \qquad (2)$$

are plotted as a function of sieve loading in Figure 5. Entropies for CO vapor were obtained from Clayton and Giauque (5). Corrections for nonideality were inconsequential for the approximate values reported here. The results are compromised, at best, by the modest precision of the heats of desorption; the results become highly uncertain for loadings in the nonreproducible regions of the isotherms because of the enormous uncertainty in the desorption heats (as well as the uncertainty as to whether one has a true equilibrium process in these regions). The apparent locus of the entropies of the adsorbed CO in these regions is indicated by the hatched areas for the 77.3, 90.1, and 145.0K isotherms in Figure 5.

Discussion

Breck et al. (3) report adsorption at 700 torr at −78° and at 750 torr at 0° for (completely?) calcium exchanged type A zeolite. Our loadings at these pressures

and temperatures are about 60–70% of those they report, but we have no knowledge of the effect of total substitutions of Na^+ by Ca^{2+} in their case compared to the partial substitution of Na^+ by Ca^{2+} in ours (Sieve 5A) or of the clay binder in our material.

Data from Linde Data Sheet #192 (16) for the $-75°$ isotherm for CO on Linde 5A agree with our 194.5K isotherm to about 15% at higher pressures, but our loadings are about fivefold greater than reported there at 1-torr pressure. For the $0°$ isotherms, our loadings are some 65–80% of those reported on Linde Data Sheet #192 (16). We are unaware of other "heat of desorption" data for CO on Type A zeolites.

Egerton and Stone (8) report "heats of desorption" for CO on partially substituted Y-type zeolites ($Na_{56}[(AlO_2)_{56}(SiO_2)_{136}] \cdot nH_2O$) where varying amounts of the

Figure 4. Isosteric heats of desorption for CO from Linde 5A Molecular Sieve vs. volume of CO adsorbed

Figure 5. Partial molal entropies of CO adsorbed on Linde 5A Molecular Sieve vs. volume of CO adsorbed

Na^+ had been substituted by Ca^{2+}, Mn^{2+}, or Ce^{3+}. The values reported are derived from isotherms in the temperature range from 0 to $\sim50°C$, for loadings from 0.2 to ~2.5 cm^3/g. As is the case for the 273K isotherm of Figure 4, their values decrease rapidly with increasing loading ranging between ~12 and 9 kcal/mol for CaY, ~11 and 8 kcal/mol for MnY, ~9 and 6 kcal/mol for CeY. The heats also appear to depend upon the extent of Na^- substitution.

Tsitsishvili et al. (18) report "heat of desorption" values determined by gas chromatography (presumably for temperatures near 300K) on various substituted X-type zeolites ($Na_{86}[(AlO_2)_{86}(SiO_2)_{106}] \cdot nH_2O$). The reported values range from 9 kcal/mol on LiX to 5 kcal/mol for K, Rb, and CsX. They report comparable values for CO on NaX in which partial substitution of Na^+ by divalent alkaline earth ions has occurred. It is difficult to know to what loadings to assign these heats.

Garden and Kington (10) and Garden et al. (11) measured ($H_{vap} - \bar{H}_{ads}$) for argon and oxygen in natural chabazite ($Na_{2x}Ca_{(4-x)}[(AlO_2)_8(SiO_2)_{16}] \cdot nH_2O$) and argon in calcium chabazite ($Ca_4[(AlO_2)_8(SiO_2)_{16}] \cdot nH_2O$) at 90.2K. Their values for argon approximate 3800 cal/mol, exhibit a maximum (3820 cal/mol) at about 75% of the saturation loading (~125 cm^3/g), a shallow minimum (3675 cal/mol) at about 30% of saturation loading, and begin to rise steeply as the loading decreases (4300 cal/mol at $\sim1\%$ loading). Their data for ($H_{vap} - \bar{H}_{ads}$) for O_2 exhibit a maximum (4280 cal/mol) at $\sim75\%$ of saturation loading, a minimum (4000 cal/mol) at $\sim25\%$ saturation, and rise steeply for loadings below 5% of saturation loading (4500 cal/mol at $\sim1\%$ loading). Our curves at 90K for CO are similar to theirs for Ar and O_2 except for the relative height of the maximum and our inability to get any data at very low loading.

Barrer and Coughlan (1) report isosteric heats, ($H_{vap} - \bar{H}_{ads}$), for CO_2 in various zeolites above room temperature as a function of zeolite loading. Kiselev and Lopatkin (14) report isosteric heats for miscellaneous substances in NaX as a function of loading. Several systems display a maximum in ($H_{vap} - \bar{H}_{ads}$) as saturation loading is approached.

Tsitsishvili et al. (18) report partial molal entropies for CO adsorbed in various substituted type X zeolites at 298K ranging from 37 cal/mol K in 54% RbX to 27 cal/mol K in 91% LiX. Again, the loadings to which these data apply are not specified.

Garden and Kington's data (10) (their "differential entropy") for O_2 in natural chabazite at 90.2K do not show the minimum followed by rising values as saturation loading is approached that the 90K curve for Figure 5 shows. Their values rise from 6 cal/mol K at 90% of saturation loading to 23 cal/mol K at 7% of saturation loading. Our inability to get data at low loadings for 145, 90, and 77K precludes direct comparison with their work; subjectively, the curves for $\bar{S}_{ads}$ vs. loading appear similar except for the aforementioned rise in ours at high loadings.

The diminution in the adsorptive capacities of the type A zeolites with decreasing temperature for molecules of certain critical sizes is well known (4). We speculate that the dimensions of CO begin to become critical (for at least some of the cavities) in Linde 5A as the temperature falls below $\sim150K$; that as the loading of CO is increased, force constants between the lattice and whatever ions define the critical apertures in the lattice are modified so that vibration amplitude is increased and CO again "freely" enters the restricted cavities; that as the temperature is further lowered, an increasing amount of adsorbed CO is required to "relax" the lattice and so obtain the reproducible portion of our isotherms.
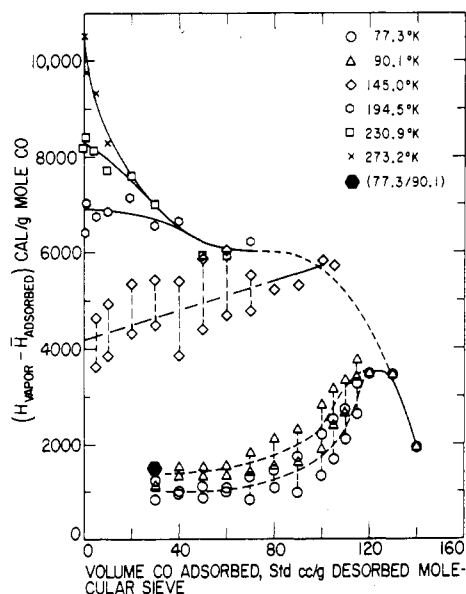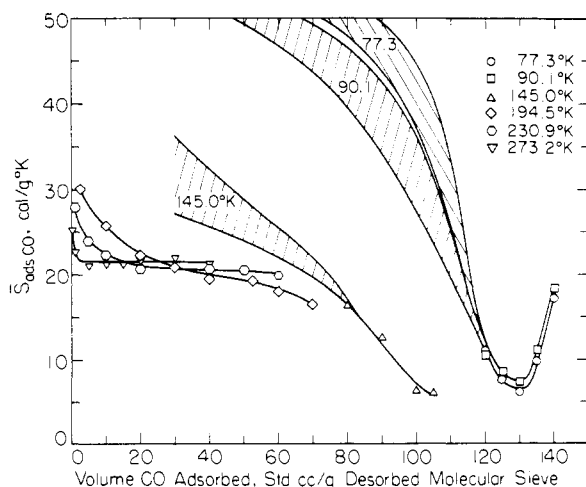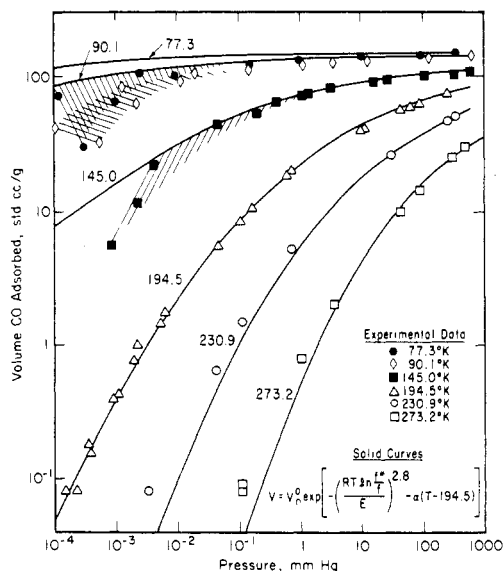
Figure 6. Comparison of experimental data with Dubinin's adsorption equation. $V_0° = 94.2$ cc/g; $E = 3780$ cal/g mol: $\alpha = 0.006$ K^{-1} for $T > 194.5°$K; $\alpha = 0.0038$ K^{-1} for $T < 194.5$K

Representation of Isotherms

If one uses Dubinin's (6) development of Polanyi's characteristic absorption curve,

$$V = V_0° \exp\left[-\left(\frac{RT \ln\left(\frac{f^*}{f}\right)}{E}\right)^n - \alpha\,(T - T°)\right] \quad (3)$$

we find the adjustable parameters to be $V_0° = 94.2$ std cc/g; $E = 3780$ cal/g mol; $n = 2.8$; $T° = 194.5$K; $\alpha = 0.006$ ($T > 194.5$K) and 0.0038 ($T < 194.5$K); and $f^* =$ fugacity of saturated CO [extrapolated above $T_{critical}$ by fitting the data of Hust and Stewart (12) to $\ln f^* = 8.18 - 666/T$ for f^* in atmospheres].

The expression represents the volume adsorbed at a designated pressure at 194.5K to ±10% over more than six decades of pressure. It does not represent the 90.1 and 77.3K isotherms, even though fit to the high-pressure datum in determining α. The expression is moderately

successful for the 145, 230.9, and 273.15K isotherms except at very low pressures, as seen from inspection of Figure 6 where the solid lines represent Equation 3 and the symbols are representative experimental data.

It is unreasonable to expect the free energy of adsorption of the polar CO molecule at the various sites within the zeolite to be characterized by a single value of E. One could undoubtedly improve the fit by more elaborate expressions. However, this commercial technical adsorbent is a heterogeneous material ($\sim$20% "clay binder"), and we are wary of attempts to read too much into the data. We offer Equation 3 and the parameters given as a means of estimating adsorbed volumes at other temperatures within the experimental range.

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