



Measurement and Prediction of Single- and Multi-Component Methane, Carbon Dioxide and Nitrogen Isotherms for U.S. Coals

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ABSTRACT

In October, 2000, the U.S. Department of Energy, through contractor Advanced Resources International, launched a multi-year government-industry R&D collaboration called the Coal-Seq project. The Coal-Seq project investigated the feasibility of CO₂ sequestration in deep, unmineable coalseams by performing detailed reservoir studies of two enhanced coalbed methane (ECBM) recovery field projects in the San Juan basin. As part of the overall project, single-component CH₄, CO₂ and N₂ isotherms were measured (or obtained) on coal samples from the Black Warrior, Cherokee, Forest City, Gulf Coast, Illinois, Northern Appalachian, Piceance, Powder River and San Juan basins. In addition, binary- and ternary-component isotherms were measured on the San Juan basin sample to compare actual multi-component sorption behavior to predictions based on single-component data.

Uni-variate analysis of the single-component results indicates a reasonable correlation of CH₄ and N₂ storage capacity (i.e., Langmuir volume) with coal rank (i.e., vitrinite reflectance) exists, but no such (linear) trend appears to exist for CO₂. No uni-variate trends were found to predict Langmuir pressure. The CO₂/CH₄ and N₂/CH₄ ratios both showed reasonable correlation to coal rank. Multiple linear regression of the data yielded acceptable models to predict Langmuir volume and pressure for the three gases based on coal characterization data.

Comparison of actual multi-component isotherm data to predictions based on single-component data showed poor predictive capability for all the theories evaluated. New and more robust multi-component sorption theories are therefore required to predict multi-component sorption behavior from single-component data.

INTRODUCTION

In October, 2000, the U.S. Department of Energy (DOE), through contractor Advanced Resources International (ARI), launched a multi-year government-industry R&D collaboration called the Coal-Seq project¹. The Coal-Seq project investigated the feasibility of CO₂ sequestration in deep, unmineable coalseams by performing detailed reservoir studies of two enhanced coalbed methane (ECBM) recovery field projects in the San Juan basin. As part of the project, single-component CH₄, CO₂ and N₂ isotherms were measured (or obtained) on coal samples from the Black Warrior, Cherokee, Forest City, Gulf Coast, Illinois, Northern Appalachian, Piceance, Powder River and San Juan basins. Maceral composition,

vitrinite reflectance and proximate analyses were also measured on each coal sample to aid in their characterization. One of the objectives of the data collection effort was to establish the beginnings of a public-domain database of such information to serve as a useful reference for companies and/or other organizations considering enhanced coalbed methane and/or carbon sequestration projects.

In addition to the single-component data, binary- and ternary-component isotherms were measured on the San Juan basin sample to compare actual multi-component sorption behavior to predictions based upon various theories using single-component data (e.g., extended Langmuir, loading ratio correlation, equations-of-state, etc.). No such analysis has been published previously, yet modeling of ECBM and carbon sequestration is founded on the assumption that such predictions are reliable. The objective of this work was to test that assumption.

DESCRIPTION OF SAMPLES, MEASUREMENT RESULTS

From 2003 through 2004, a total of 11 coal samples were collected from different basins throughout the United States. Those basins included the Black Warrior, Cherokee, Forest City, Gulf Coast, Illinois, Northern Appalachian, Piceance, Powder River and San Juan basins. All but one sample was in the form of a core from a well drilled from the surface, the exception being a mine sample from the Black Warrior basin. Each sample was identified by location (county, state), and coal seam (name, depth), and was characterized by coal rank (vitrinite reflectance), maceral composition, and chemical composition (proximate analysis). Following characterization, moisture-equilibrated single-component N_2 , CH_4 and CO_2 isotherms were measured at the temperature corresponding to in-situ conditions. The results were reported on an as-received, dry, and dry-ash-free basis.

Coal characterization data for the samples are provided in Table 1. The isotherm results, presented in the form of Langmuir isotherm constants (V_L and P_L), are presented in Table 2. Table 2 also provides the conditions at which the isotherms were measured. Plots of the individual isotherms for N_2 , CH_4 and CO_2 are presented in Figures 1-3. Initial examination of the results suggests that all of the data appear as one would expect - gas storage capacity generally increases with coal rank, and for a given sample the gas storage capacity is greatest for CO_2 , followed by CH_4 , and is the least for N_2 .

SINGLE-COMPONENT ISOTHERM PREDICTIONS FROM COAL CHARACTERIZATION DATA

While the publication of this dataset in and of itself was an important objective of this effort, an additional objective was to identify the existence of any relationships in the data that would enable one to estimate single-component isotherm properties from coal characterization data alone. Such a capability would enable one to reasonably estimate sorptive properties of a coal in the absence of actual isotherm measurements.

The first approach employed was simple uni-variate analysis. Since coal rank is usually an important factor influencing coal sorptive capacity, vitrinite reflectance, a proxy for coal rank, was selected as the independent variable. Plots of Langmuir volume and Langmuir pressure versus vitrinite reflectance for each gas and for each sample were created; the results are presented in Figures 4 and 5.

The results indicate that CH_4 and N_2 sorptive capacity increases with coal rank in a statistically meaningful fashion. However, no such trend exists for CO_2 . If a relationship does exist between coal rank and CO_2 sorptive capacity, it must be influenced by other factors not accounted for in this simple model. Figure 5 suggests there is no meaningful relationship between Langmuir pressure and vitrinite reflectance for any of the gases. Again, other factors may be influencing the result.

The ratio of N_2/CH_4 and CO_2/CH_4 sorptive capacities were also examined as function of vitrinite reflectance. Both Figures 6 and 7 suggest that some relationship does exist in this regard, with the ratio increasing with increasing coal rank for N_2 , and decreasing with increasing coal rank for CO_2 . Figure 8 illustrates both of these ratios and a function of pressure for the Piceance basin sample. Similar trends existed for the other samples.

Principle component analysis together with multiple-linear regression and analysis of covariance were then employed to determine if a more sophisticated mathematical model could be constructed to predict single-component isotherm properties (Langmuir constants) based upon coal characterization data alone. Six individual models were constructed (one for each Langmuir constant for each gas), each with seven input variables. The variables used for each model, their relative impact, and the predictive accuracy of each model are presented in Table 3.

Actual versus predicted Langmuir constants for each model are also provided in Figures 9 & 10. Each model appears to predict the objective Langmuir parameter with excellent accuracy. Note that the corrected R^2 values in Table 3 are different from those in Figures 9 & 10 because the corrected R^2 measures the combined predictive capability of each input parameter in multi-dimensional space, and this is the real indicator of the efficiency of the model². While the predictive accuracy is excellent, the few number of samples limits the widespread applicability and robustness of the model at this time.

In all cases, predictions of CO_2 isotherms were generally more difficult than for N_2 or CH_4 , and predictions of Langmuir pressure more difficult than Langmuir volume. Nevertheless, acceptable models were created for all parameters, which indicates that predictions of single-component isotherm properties are possible from coal characterization data alone, even with a limited dataset as was the case here.

MULTI-COMPONENT ISOTHERM PREDICTIONS FROM SINGLE-COMPONENT DATA

In addition to investigating relationships between coal characterization data and single-component isotherm properties, the validity of multi-component sorption predictions based on single-component data was also examined. Two isotherm models were specifically evaluated for the purposes of predicting multi-component isotherm behavior from single component data. They were:

Extended Langmuir/Loading Ratio Correlation (LRC).

Historically, simple models have been used to represent the behavior of pure and mixed gas adsorption on coal. The extended Langmuir model is used almost exclusively in literature studies³, although the Ideal Adsorbed Solution (IAS) model has also been employed^{4,5}. Both of these models work well for essentially ideal adsorbed solutions, but neither is capable of handling nonidealities in the adsorbed phase with any accuracy. The extended Langmuir model is shown below as an illustration of the simple modeling approach used in most previous studies. For mixtures, it takes the form:

$$\theta_i = \frac{\omega_i}{L_i} = \frac{B_i P y_i}{1 + \sum_j B_j P y_j} \quad (1)$$

where ω_i is the amount of component "i" adsorbed (standard cubic feet of "i" adsorbed per ton of coal), L_i and B_i are Langmuir constants for "i", P is pressure, and y_i is the mole fraction of "i" in the gas phase. This relation allows mixture adsorption to be predicted from pure-component data, since values of L_i and B_i may be determined from the pure-component form of Equation (1). For pure-fluid adsorption, Equation (1) reduces to the Langmuir model:

$$\theta = \frac{\omega}{L} = \frac{BP}{1 + BP} \quad (2)$$

The combined Langmuir-Freundlich adsorption isotherm, expressed in terms of ω_i , yields the loading ratio correlation (LRC) for mixtures:

$$\theta_i = \frac{\omega_i}{L_i} = \frac{(B_i P y_i)^{\eta_i}}{1 + \sum_j (B_j P y_j)^{\eta_j}} \quad (3)$$

The additional parameter (η_i) in the LRC lends the Langmuir model more flexibility. Although the simplicity of Langmuir models is attractive, data show that they are inadequate to represent the behavior of mixtures of the gases CO₂, methane, and nitrogen. In fact, errors greater than 100% were found when the extended Langmuir model was applied to data on the adsorption of nitrogen from nitrogen and CO₂ mixtures⁶.

Zhou-Gasem-Robinson Equation-of-State (ZGR-EOS).

Simulations of coalbed gas recovery and CO₂ sequestering require reliable, yet simple, analytic models beyond Langmuir-type correlations. Equation-of-state (EOS) frameworks offer an attractive potential for such requirements. A general two-dimensional EOS can be written as follows⁷:

$$\left[A\pi + \frac{\alpha\omega^2}{1 + U\beta\omega + W(\beta\omega)^2} \right] [1 - (\beta\omega)^m] = \omega RT \quad (4)$$

where A is the specific surface area, π is the spreading pressure, ω is the specific amount adsorbed, and α , β and m are model parameters. The model coefficients, U, W, and m must be specified to obtain a specific form of the 2-D EOS for application. For example, an analog of the van der Waals (VDW) EOS is obtained by setting $m = 1$ and $U = W = 0$; similarly for the Soave-Redlich-Kwong (SRK) ($m = U = 1$ and $W = 0$); the Peng-Robinson (PR) ($m = 1$, $U = 2$, and $W = -1$); and the Eyring ($m = 1/2$ and $U = W = 0$) EOS.

This general 2-D EOS can be used to investigate EOS behaviors by specifying various combinations of model coefficients. Selection of the model coefficient m is the most important among the EOS model coefficients, because it has a significant effect on the shape of the pure adsorption isotherm. If U and W are equal to zero, then by setting m to values of ∞ , 1, and 1/2, we obtain the 2-D ideal gas law, the VDW EOS, and the Eyring EOS, respectively. Actually, the pure gas isotherms vary considerably in shape and we have found that it is sometimes desirable to select an m value even smaller than 1/2 to describe pure isotherms. We have determined that an equation with $m = 1/3$ and $U = W = 0$ (the ZGR EOS) is promising. The 2-D EOS can be applied to adsorbed phases containing mixtures by utilizing the traditional mixing rules (where x is the mole fraction in the adsorbed phase):

$$\alpha = \sum_i \sum_j x_i x_j \alpha_{ij} \quad (5)$$

$$\beta = \sum_i \sum_j x_i x_j \beta_{ij} \quad (6)$$

along with the non-traditional combination rules,

$$\begin{aligned} \alpha_{ij} &= (1 - C_{ij})(\alpha_i + \alpha_j)/2 \\ \beta_{ij} &= (1 + D_{ij})\sqrt{\beta_i \beta_j} \end{aligned} \quad (7)$$

where C_{ij} and D_{ij} are the EOS binary interaction parameters.

Modeling Results

On the San Juan basin sample, in addition to the three single-component isotherms, three binary isotherms, and one ternary isotherm were measured⁹. The feed-gas compositions of the multi-component isotherms were:

- 50% CH₄, 50% N₂
- 40% CH₄, 60% CO₂
- 20% N₂, 80% CO₂
- 40% CH₄, 10% N₂, 50% CO₂

The three single component isotherms for this sample are presented in Figure 11. The three binary isotherms for the coal sample are presented in Figures 12 -14, and the ternary isotherm in Figure 15. Note that in each plot the data points represent the actual measured data, with error bars indicating the experimental error associated with each data point. The lines represent model predictions of the data based on single-component isotherms.

One can readily observe from Figures 10-13 that the ability of either model (LRC or ZGR-EOS) to predict multi-component isotherm behavior from single-component data is frequently well outside the bounds of experimental error. This is an expected outcome since neither model accurately accounts for the physics of adsorption on wet coal. The modeling results are also presented in Table 4, where the LRC results are shown for the case of using an overall optimum fixed value of $\eta = 0.9$. In this table, the absolute average deviation for the model predictions from the actual isotherm data for each gas in each multi-component system is provided. Note the large number of cases where the error is greater than the experimental error (noted with asterisks). This seems to indicate that two commonly-used models for predicting multi-component sorption behavior based on single-component data are inadequate, which could lead to misleading predictions of ECBM and sequestration project performance. Work is currently underway to understand the causes for the errors and develop improved multi-component isotherm models.

CONCLUSIONS

Based on the results of this work, the following conclusions have been drawn:

- Uni-variate analysis of the single-component results indicates a reasonable trend of CH₄ and N₂ storage capacity (i.e., Langmuir volume) with coal rank (i.e., vitrinite reflectance) exists, but no such (linear) trend appears to exist for CO₂. No uni-variate trends were found to predict Langmuir pressure. The CO₂/CH₄ and N₂/CH₄ sorption capacity ratios also showed identifiable trends with coal rank. Multiple linear regression of the data yielded reasonable models to predict Langmuir volume and pressure for the three gases based on coal characterization data.
- Comparison of actual multi-component isotherm data to predictions based on single-component data showed inadequate predictive capability for all the theories evaluated. New and more robust multi-component sorption theories are therefore required to predict multi-component sorption behavior from single-component data if, in fact, such calculations are feasible.

ACKNOWLEDGEMENTS

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Table 1: Coal Characterization Data

Basin	County & State	Seam Name	Sample Depth (ft)	Coal Rank		Proximate Analysis (as received)					Petrographic Analysis (w/o minerals)		
				(% Vro)	Description	Moisture (%)	Ash (%)	Volatile Matter (%)	Fixed Carbon (%)	Heating Value (BTU/lb)	Vitrinite (%)	Liptinite (%)	Inertinite (%)
Powder River	Campbell, WY	Big George	1225	0.36	Sub	22.6%	2.6%	35.2%	39.6%	9,659	90.2%	5.8%	0.4%
Gulf Coast	Catahoula, LA	Wilcox	4898	0.41	Sub	10.8%	9.0%	38.7%	41.6%	10,948	57.9%	8.4%	33.7%
Forest City Basin	Franklin, KS	Mineral	780	0.54	HVB	2.1%	15.2%	42.5%	40.2%	12,564	89.8%	4.8%	5.4%
Illinois	Clark, IL	Herrin (No. 6)	604	0.59	HVB	13.1%	11.0%	29.6%	46.3%	11,064	83.8%	7.2%	8.9%
N. Appalachian	Marshall, WV	Pittsburgh	714	0.62	HVB	1.8%	8.0%	38.7%	51.5%	13,567	85.1%	2.7%	12.2%
Cherokee Basin	Montgomery, KS	Mineral	772	0.69	HVB	1.7%	13.0%	36.7%	48.6%	13,128	86.4%	6.9%	6.7%
N. Appalachian	Marshall, WV	Lower Freeport	1361	0.76	HVA	1.3%	11.6%	36.2%	50.9%	13,167	70.4%	12.1%	17.5%
Piceance	Rio Blanco, CO	Cameo	6700	0.80	HVA	1.3%	5.1%	35.6%	58.0%	14,020	92.1%	1.5%	6.3%
Warrior	Walker, AL	Mary Lee	0	0.90	HVA	2.6%	13.7%	30.1%	53.6%	12,345	87.4%	4.6%	8.0%
San Juan	La Plata, CO	Fruitland	3100	1.33	MV	n/a	n/a	30.0%	70.0%	n/a	96.0%	0.8%	3.2%
Warrior	Tuscaloosa, AL	Newcastle	1948	1.40	MV	1.6%	11.5%	21.3%	65.6%	13,600	90.6%	1.0%	8.4%

Table 2: Isotherm Results

Basin	County & State	Seam Name	Isotherm Information (moisture-equilibrated, daf/dmmf)								
			Measurement	Equilibrium	Ash	Nitrogen		Methane		Carbon Dioxide	
			Temperature (deg F)	Moisture (%)	Content (%)	VL (scf/ton)	PL (psi)	VL (scf/ton)	PL (psi)	VL (scf/ton)	PL (psi)
Powder River	Campbell, WY	Big George	75	23.9%	2.9%	81	1344	217	820	2331	1172
Gulf Coast	Catahoula, LA	Wilcox	140	12.0%	9.2%	154	2315	379	1010	1109	885
Forest City Basin	Franklin, KS	Mineral	70	2.6%	14.0%	218	744	465	406	881	185
Illinois	Clark, IL	Herrin (No. 6)	65	13.2%	11.3%	208	1125	432	458	1248	303
N. Appalachian	Marshall, WV	Pittsburgh	64	2.3%	7.7%	427	2919	569	537	987	240
Cherokee Basin	Montgomery, KS	Mineral	75	2.1%	13.1%	249	819	477	357	853	171
N. Appalachian	Marshall, WV	Lower Freeport	69	1.6%	11.2%	393	2478	547	560	898	281
Piceance	Rio Blanco, CO	Cameo	155	1.8%	4.7%	411	2101	616	871	911	340
Warrior	Walker, AL	Mary Lee	86	4.7%	14.0%	256	1023	612	845	1061	292
San Juan	La Plata, CO	Fruitland	130	n/a	n/a	521	1587	848	663	1153	223
Warrior	Tuscaloosa, AL	Newcastle	86	2.3%	18.6%	770	1245	1081	559	1308	218

Table 3: Results of Multiple-Linear Regression Models

Objective Function	Parameter Ranking												Goodness of Fit	
	Vro	Moist.	Ash	VM	FC	HV	Vitr.	Lipt.	Inert.	Temp.	E.Moist.	Ash Cont.	% AAD*	Corr. R ²
VL(N ₂)	4	3	n/a	n/a	7	1	2	5	n/a	6	n/a	n/a	4.81%	0.954
PL (N ₂)	n/a	3	1	n/a	n/a	5	7	6	4	2	n/a	n/a	7.65%	0.884
VL(CH ₄)	1	7	n/a	n/a	5	4	2	3	n/a	6	n/a	n/a	2.80%	0.977
PL (CH ₄)	n/a	n/a	2	n/a	3	1	n/a	6	5	7	4	n/a	1.99%	0.982
VL(CO ₂)	2	n/a	n/a	3	5	7	n/a	6	n/a	4	1	n/a	1.59%	0.991
PL (CO ₂)	6	n/a	n/a	1	3	4	5	n/a	n/a	n/a	2	7	7.91%	0.973

* AAD = average absolute deviation

Table 4: Accuracy of Multi-Component Isotherm Predictions from Single-Component Data

Mixture, (Feed Mole %)	Langmuir % AAD	LRC (n=0.9) % AAD	ZGR-EOS % AAD	Estimated Experimental Error % AAD
CH₄ - N₂:				
CH ₄ (50%)	15.8*	12.0*	11.9*	7.0
N ₂ (50%)	6.2	9.3	10.0	17.0
Total	12.2*	8.2*	11.5*	7.0
CH₄ - CO₂:				
CH ₄ (40%)	25.9*	21.0*	27.0*	7.0
CO ₂ (60%)	9.0*	10.5*	10.4*	6.0
Total	1.2	2.2	1.4	4.0
N₂ - CO₂:				
N ₂ (20%)	44.9*	37.3*	48.7*	29.0
CO ₂ (80%)	5.2	5.7	4.9	6.0
Total	3.5	3.8	3.5	5.0
N₂ - CH₄ - CO₂:				
N ₂ (10%)	47.8*	44.5*	55.9*	14.0
CH ₄ (40%)	20.7	5.2	21.6	27.0
CO ₂ (50%)	13.2*	15.8*	17.6*	5.0
Total	2.9	5.4*	4.3	5.0

* Greater than experimental error

MEASUREMENT AND PREDICTION OF SINGLE- AND MULTI-COMPONENT METHANE, CARBON DIOXIDE AND NITROGEN ISOOTHERMS FOR U.S. COALS

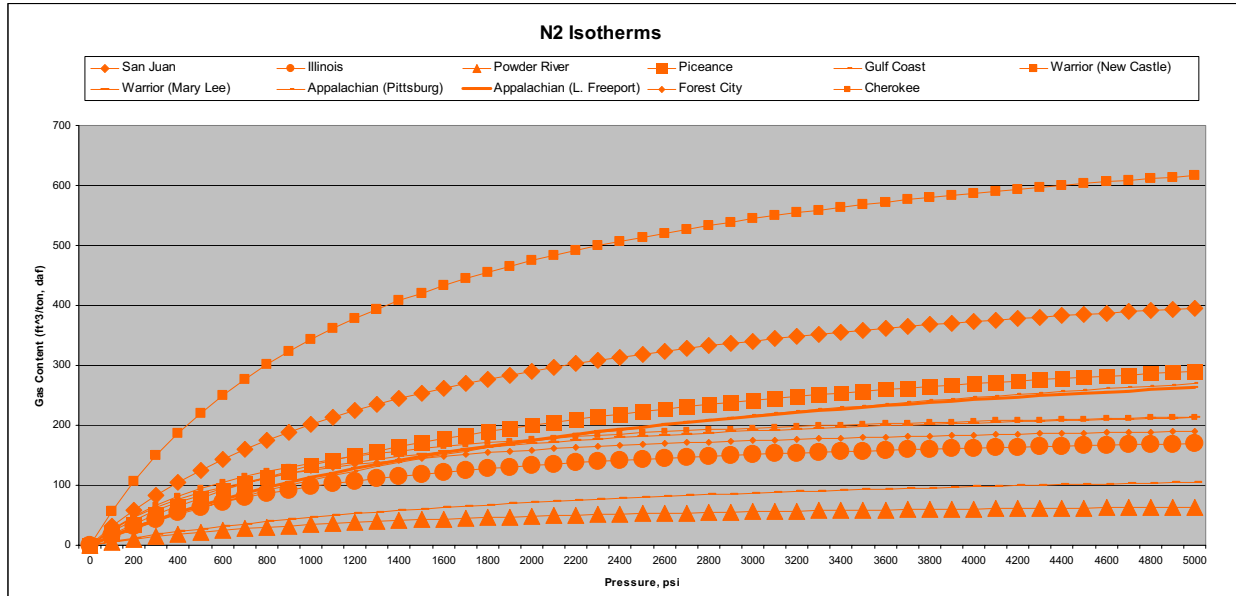


Figure 1: Langmuir Nitrogen Isotherms

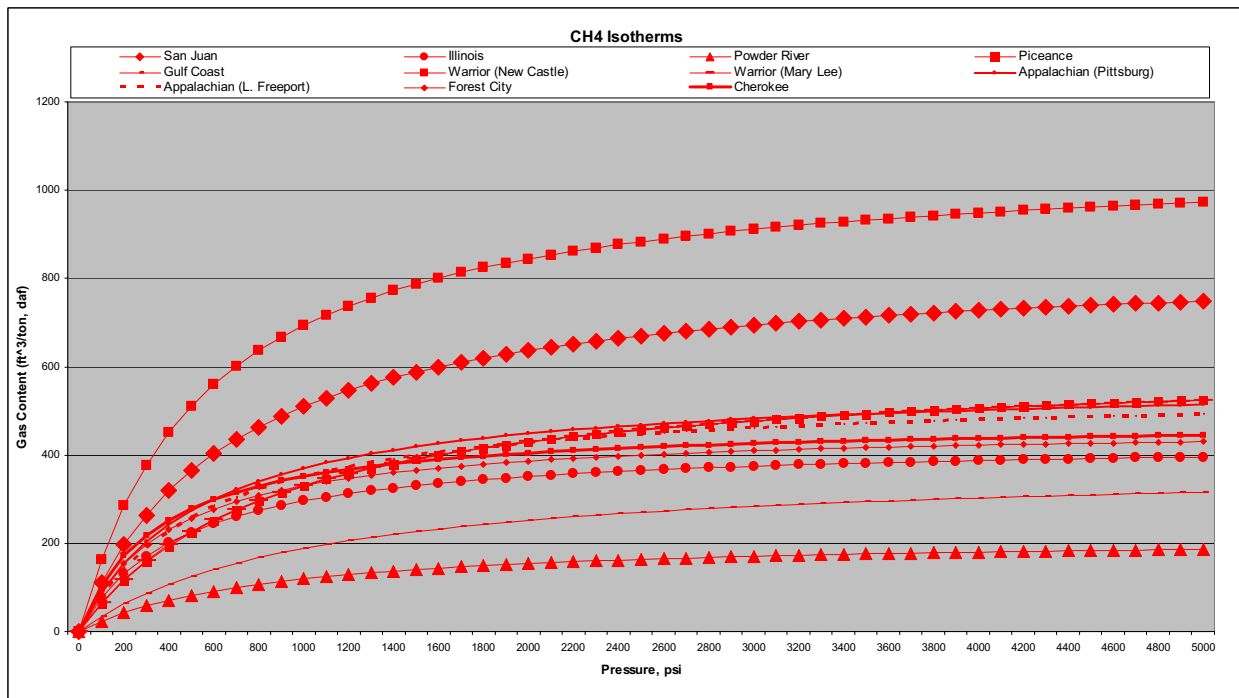


Figure 2: Langmuir Methane Isotherms

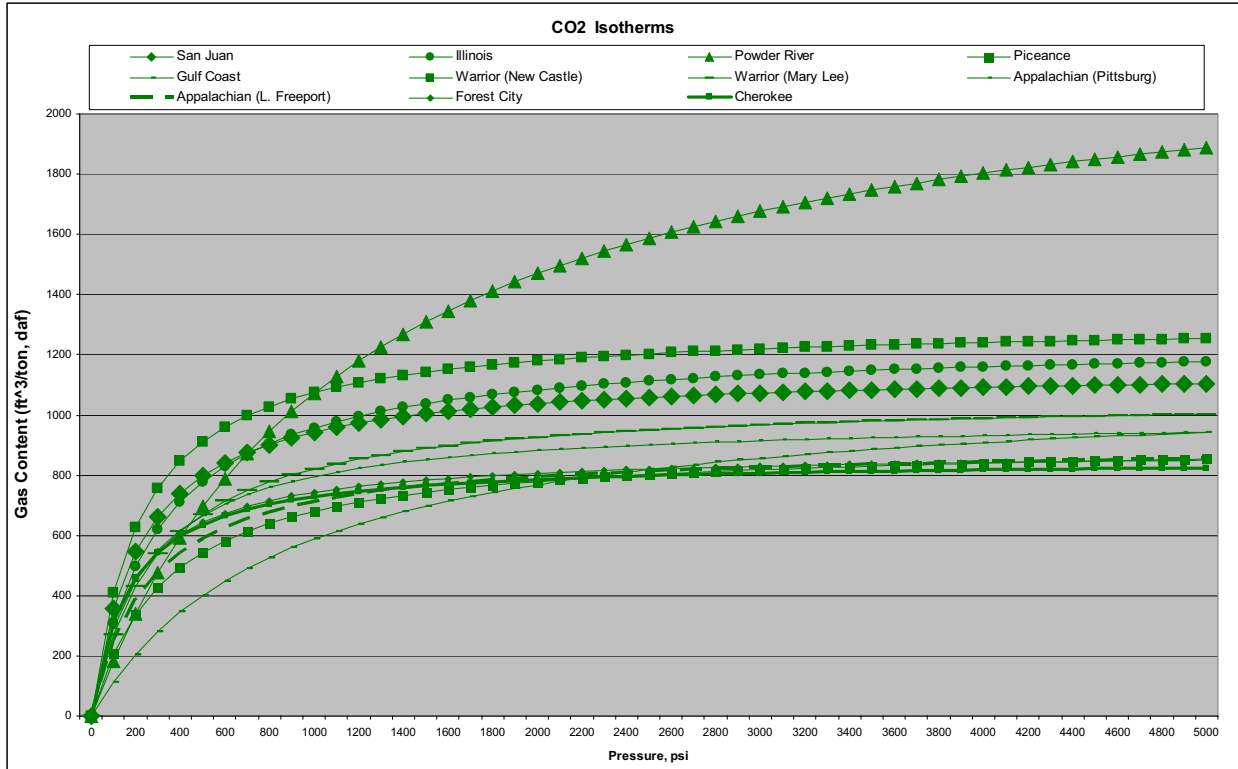


Figure 3: Langmuir Carbon Dioxide Isotherms

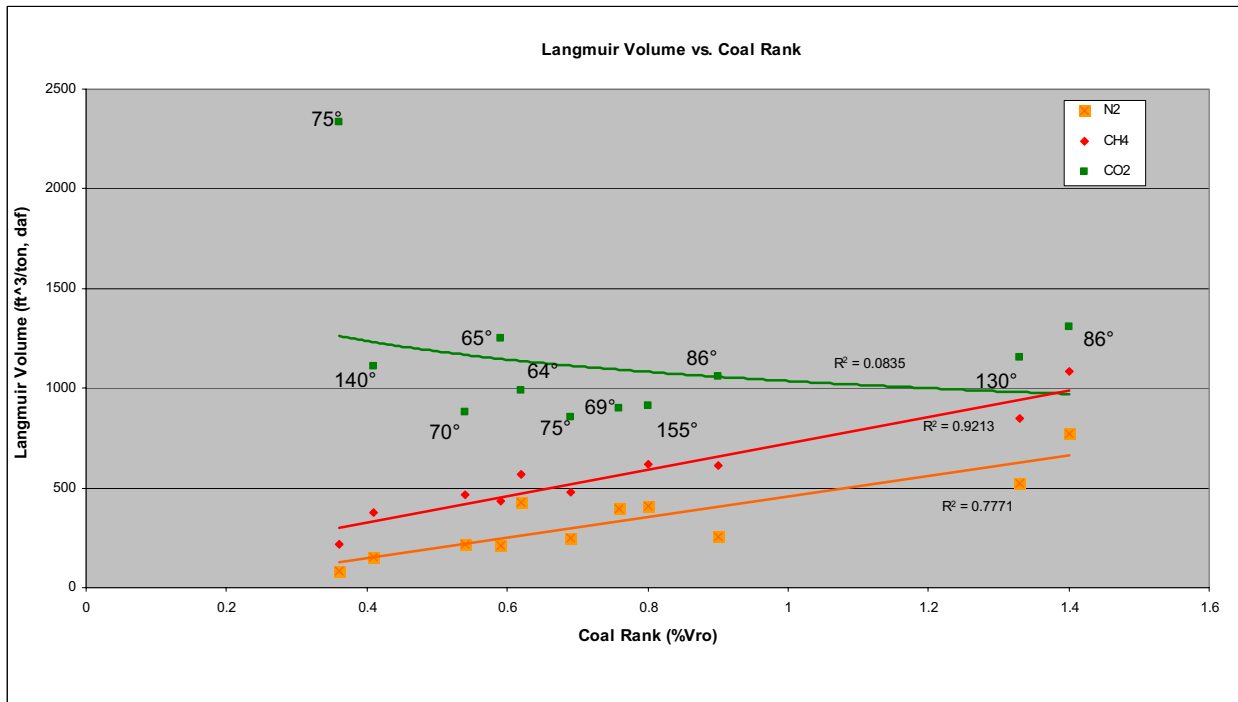


Figure 4: Langmuir Volume vs. Vitrinite Reflectance

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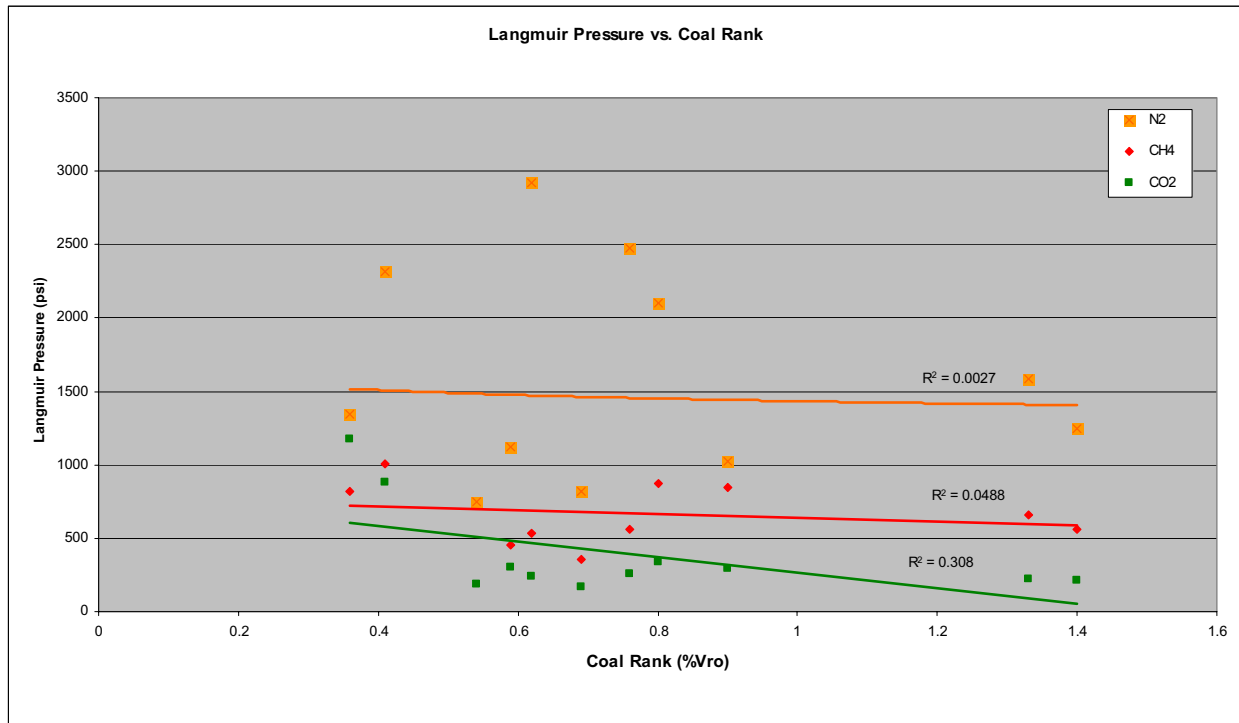


Figure 5: Langmuir Pressure vs. Vitrinite Reflectance

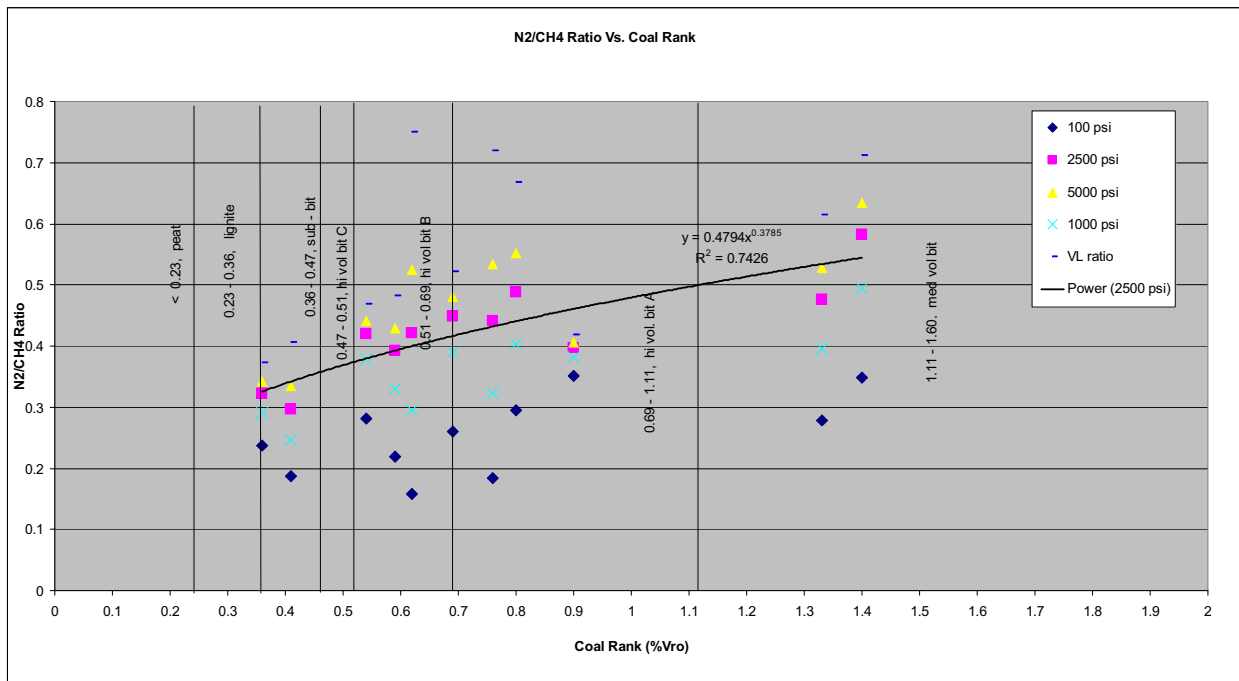


Figure 6: N₂/CH₄ Sorption Capacity vs. Vitrinite Reflectance

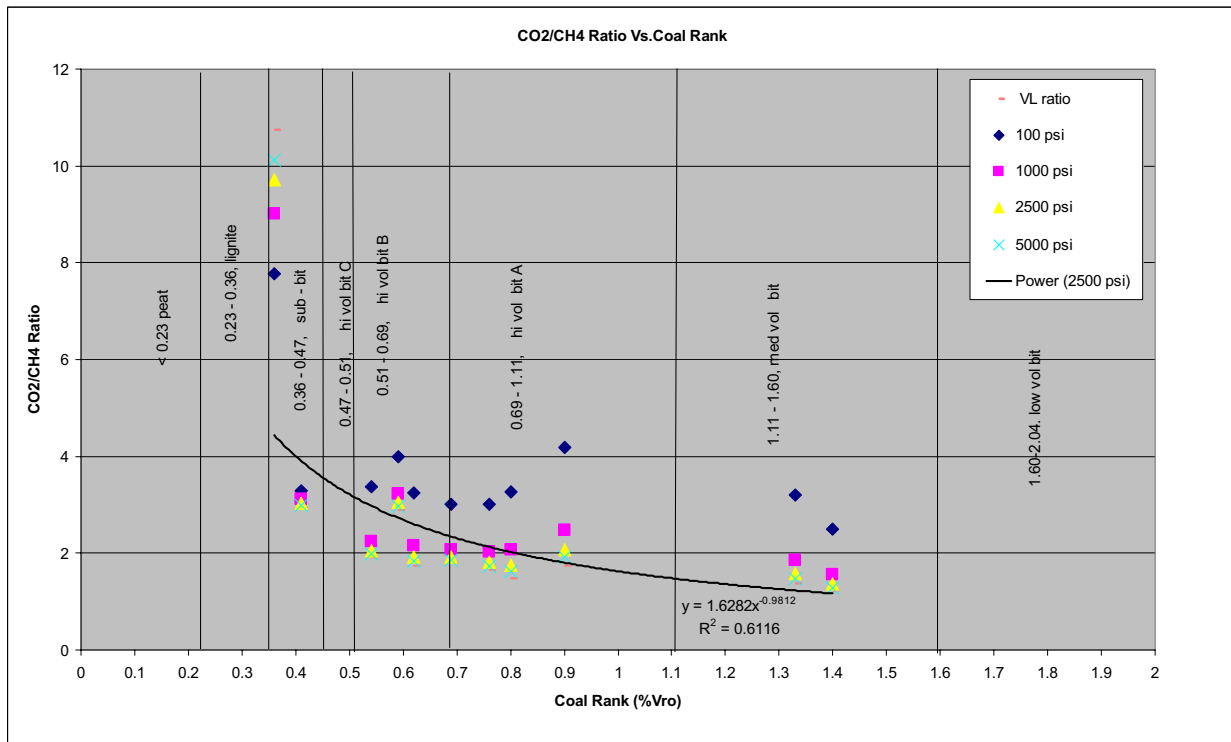


Figure 7: CO₂/CH₄ Sorptive Capacity vs. Vitrinite Reflectance

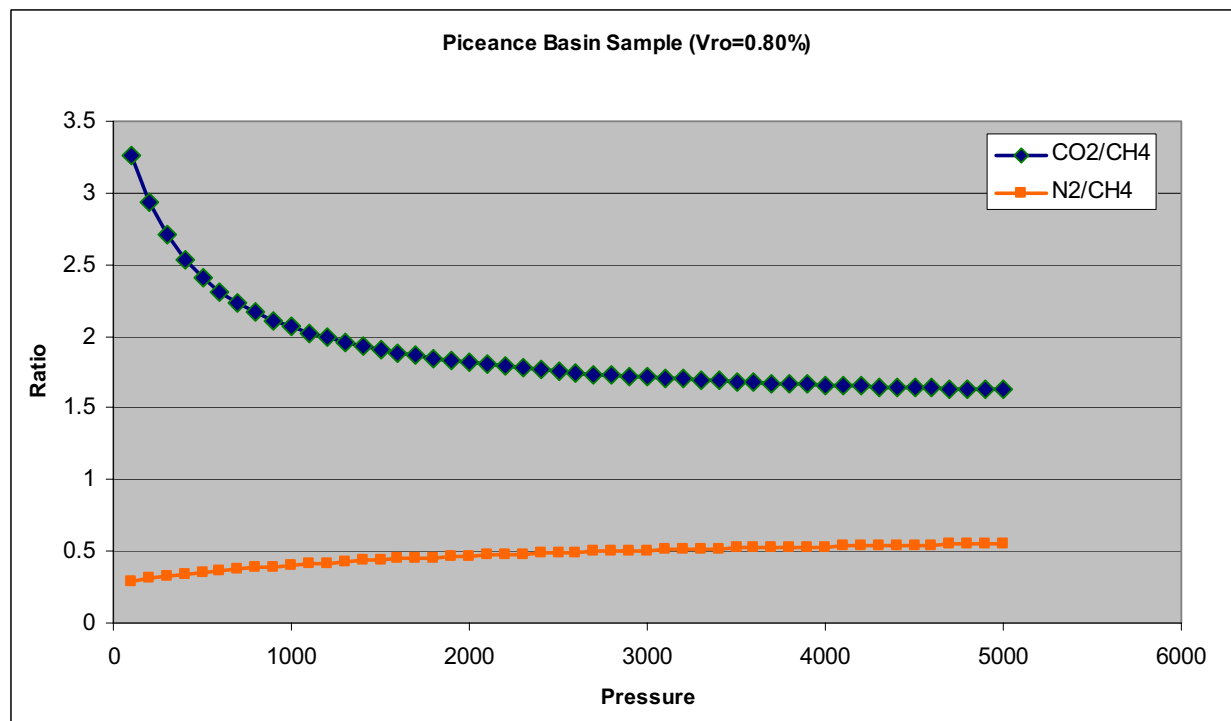


Figure 8: N₂/CH₄ and CO₂/CH₄ Sorptive Capacity Ratios, Piceance Basin

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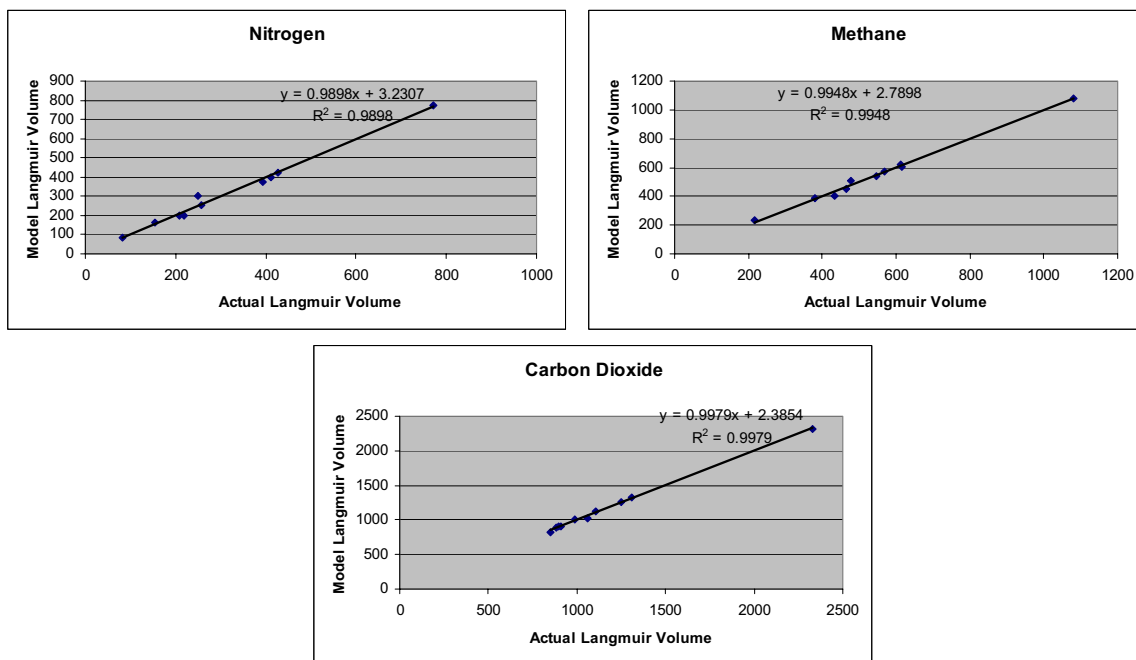


Figure 9: Actual vs. Predicted Langmuir Volumes, Multiple Linear Regression Models

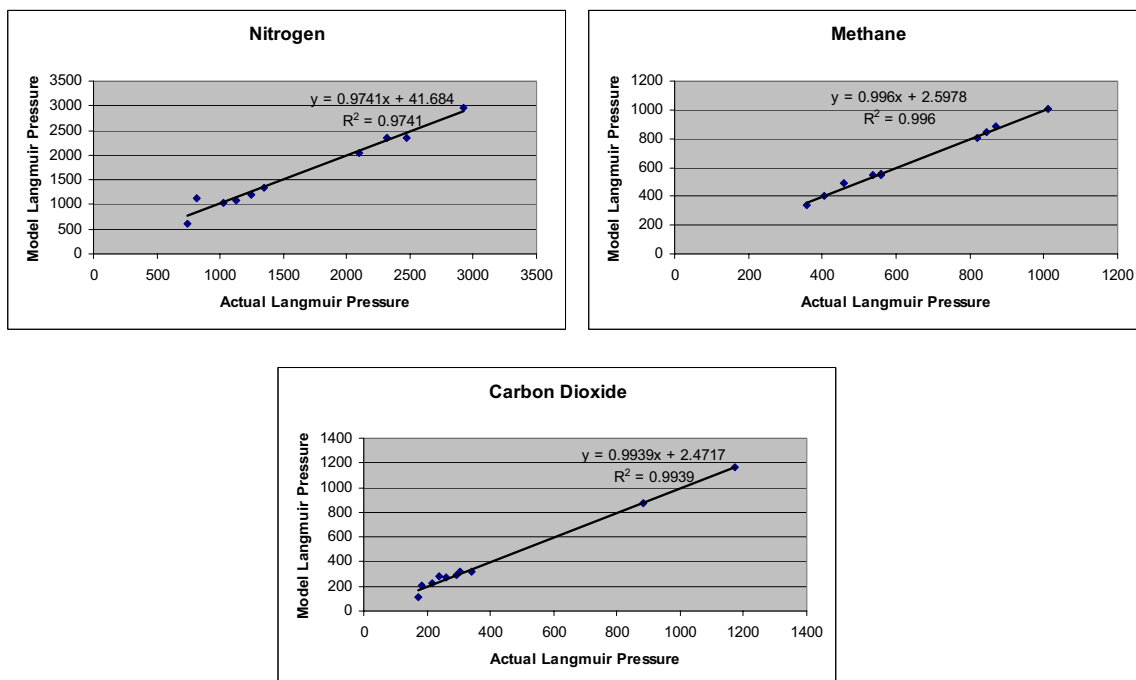


Figure 10: Actual vs. Predicted Langmuir Pressures, Multiple Linear Regression Models

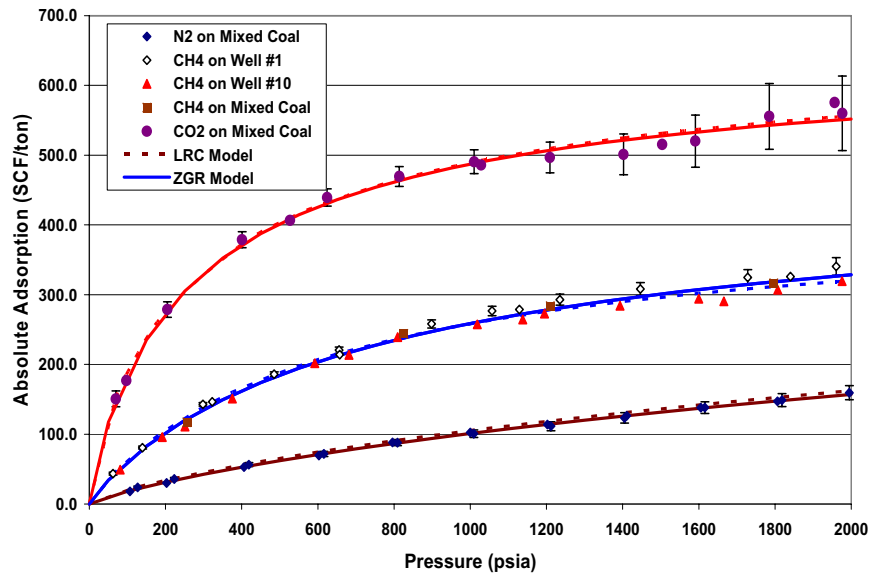


Figure 11: Single-Component Nitrogen, Methane and Carbon Dioxide Isotherms for San Juan Basin Coal at 130°F

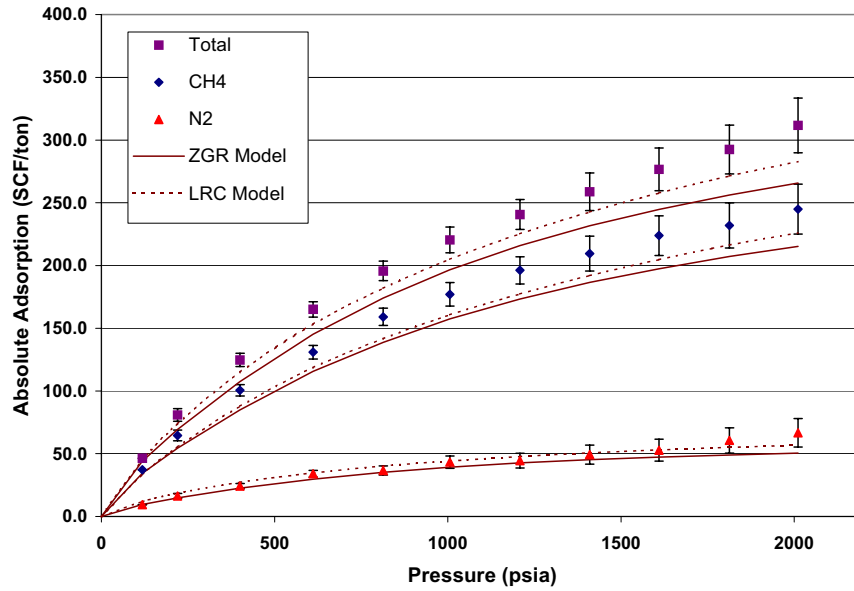


Figure 12: CH₄-N₂ Binary Adsorption Isotherms for San Juan Basin Coal (50% CH₄, 50% N₂) at 130°F

MEASUREMENT AND PREDICTION OF SINGLE- AND MULTI-COMPONENT METHANE, CARBON DIOXIDE AND NITROGEN ISOTHERMS FOR U.S. COALS

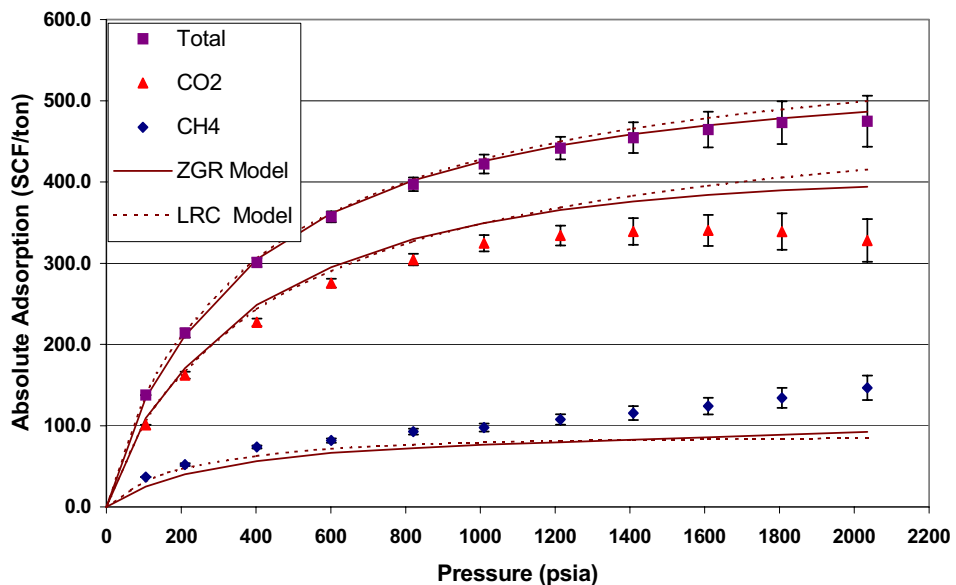


Figure 13: CH₄- CO₂ Binary Adsorption Isotherms for San Juan Basin Coal (40% CH₄, 60% CO₂) at 130°F

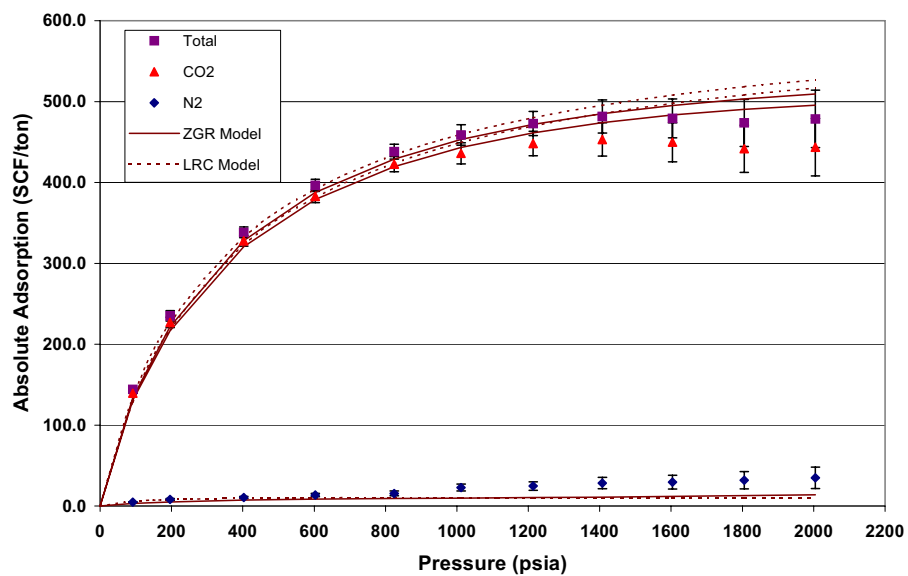


Figure 14: N₂-CO₂ Binary Adsorption Isotherms for San Juan Basin Coal (20% N₂, 80% CO₂) at 130°F

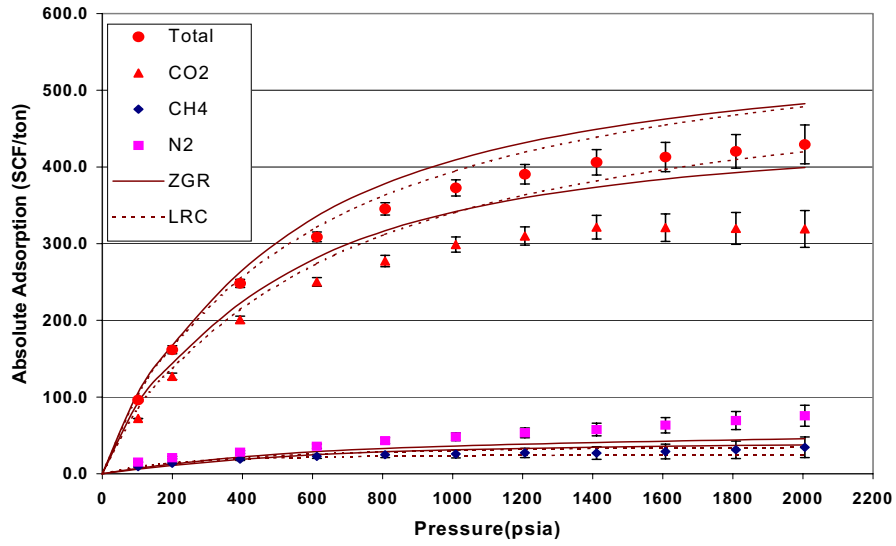


Figure 15: Ternary Adsorption Isotherms for San Juan Basin Coal (40%CH₄, 10% N₂, 50% CO₂) at 130°F