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Thermodynamic Adsorption of Herbicides on Eight Agricultural Soils

Rounak M. Shariff, Kafia M. Shareef

Abstract— Thermodynamics of adsorption of three pesticides using a kinetic approach was investigated. The objective of this study is to study the effect of temperature on the sorption behavior of three commonly used pesticides in Kurdistan onto eight natural soil samples collected from different agricultural locations. To elucidate the effect of temperature on the sorption process, the experiments were done at 15, 25, 35 ±1ºC. Values of the standard thermodynamic functions: equilibrium constant K_o, free energy change ΔG^o, enthalpy change ΔH^o and entropy change ΔS^o revealed that adsorption of atrazine, picloram and propanil was spontaneous exothermic and physical in nature to some extent. Data obtained revealed that adsorption coefficient decreases with the incresing temperature. Values of $\ln K_o$ were in the range 1.238 to 12.75, 1.674 to 14.032 and 29.83 to 173.1 for atrazine, picloram and propanil respectively. The ΔG^o values were in the range -30.549 to -3.172 $KJmol^{-1}$, -33.614 to -4.2897 $KJmol^{-1}$, and - 414.72 to - 76.412 $KJmol^{-1}$ for atrazine, picloram, and propanil respectively. Values of ΔH^o followed the range -5.010 to -0.738 to $KJmol^{-1}$, -4.769 to -0.848 $KJmol^{-1}$ and -75.779 to - 1.628 $KJmol^{-1}$ for atrazine, picloram, and propanil respectively. ΔS^o followed the range-16.138 to -1.757 $Jmol^{-1} k^{-1}$, -14.711 to -2.502 $Jmol^{-1} k^{-1}$ and -199.01 to -62.457 $Jmol^{-1}k^{-1}$for atrazine, picloram, and propanil respectively.

Index Terms — Adsorption isotherms, Adsorption thermodynamic, atrazine, picloram, propanil, HPLC.

1 Introduction

THERE is considerable public concem about potential adverse impact of pesticide used on ecosystem and human health. To minimize any such detriments, sound understanding of environmental fate and behavior of pesticides is necessary under local soil and environmental conditions [1], [2]. Temperature is an important factor governing the rate of adsorption in soil pore. The existence of a number of solid structures of picloram were suggested and discussed with the application of the values of ΔH^o_{sol} as a correction of solubility- temperature effect on the standard enthalpy of the pesticide adsorption processes [3], [4] . The major factors that determine the extent to which herbicides are adsorbed by soil include: i) physical or chemical characteristic of the adsorbent, ii) physical or chemical properties of the pesticides, and iii) properties of the soil system, such as cleay mineral composition, pH, kinds and amounts of exchangeable cations, and temperature [5], [6]. Since information on the sorption behavior of pesticides in soil is essential in predicting their leaching potential and contamination of ground water and no data are available in literature for sorption kinetics equilibrium parameters of the three commonly used pesticides (atrazine, picloram and propanil) in Kurdistan. Studies were conducted on their sorption and determining the thermodynamic parameters associated their sorption onto natural soil samples.

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2 Methodlogy

2.1 Soils

Fresh soil samples were taken from eight main agricultural locations, representing awide range of physicochemical properties. Subsamples of homogenized soils were analyzed for moisture content, organic matter content, particle size distribution, texture, pH, loss on ignition and exchangeable basic cations the detail were charactrerized in previous article [7].

2.2 Pesticides

Analytical grad substituted atrazine, picloram and propanil were purchased from Riedal-de Haen, Sigma-Aldrich Company. All chemicals used were of analytical grade reagents and used without pre-treatments. Standard stock solutions of the pesticides were prepared in deionised water.

2.3 Adsorption Experiments

The effect of temperature on adsorption of pesticides from aqueous solution was determined at 15, 25, 25±1 C° employing a standard batch equilibrium method [8]. Duplicate air-dried soil samples were equilibrated with different pesticide concentrations (2, 5, 10, and 15 $\mu g ml^{-1}$) at the soil solution ratios: 4:10, 4:8, and 1:10 for atrazine, picloram and propanil respectively. The samples plus blanks (no pesticide) and control (no soil) were thermostated and placed in shaker for 24 h for atrazine and picloram and for 10 h for propanil. The tubes were centrifuged for 20 min. at 3500 rpm. Supernatants were analyzed by PerkinElmer series 200 USA family high performance liquid chromatography (HPLC) for each pesticide concentration. The detailed information about the soil characteristics and their sorption process has been reported in our previous work [8], [9].

3.1 Equilibrium constant

The equilibrium constant K_o was enabled us to calculate the thermodynamic parameters for physico-chemical equilibrium between pesticides and soils [10].

$$K_o = \frac{a_s}{a_e} = \frac{\gamma_s}{\gamma_e}\frac{C_s}{C_e} \quad (1)$$

Where a_s: activity of the adsorbed solute, a_e: activity of the solute in the equilibrium solution, C_s: μg of solute adsorbed per milliliter of solvent in contact with the adsorbent surface, C_e: μg of solute per milliliter of solvent in the equilibrium solution, γ_s: activity coefficient of the adsorbed solute. γ_e: activity coefficient of the adsorbed solute in the equilibrium solution. As the concentration of the solute in the solution approaches zero, the activity coefficient, γ, approaches unity. "Equation (1)," may then be written as:

$$C_s \xrightarrow{\lim} 0 \quad \frac{C_s}{C_e} = \frac{a_s}{a_e} = K_o \quad (2)$$

Values of $\ln K_o$ were obtained from the plot of ln (C_s/C_e) vs. C_s, $\ln K_o$ was obtained at $C_s = 0$, as described by Biggar and Chenung [11]. The results were summarized in Table 1, 2, and 3 for atrazine, picloram and propanil respectively. Values of $\ln K_o$ were in the range 1.238 to 12.75, 1.674 to 14.032 and 29.83 to 173.1 for atrazine, picloram and propanil respectively. It is well known that K_o is constant at constant temperature and the position of equilibrium depends only on thermodynamic quantities and is independent of any consideration of kinetics or mechanism. Values of K_o obtained can vary among soils due to the quantities and composition of soil components. The K_o values were decreased with rise in temperature, confirming that the pesticides had a high preference for adsorption at low temperature.

3.2 Standard Free Ernergy Change

Adsorptions equilibrium constant K_o can be expressed in terms of the standard Gibbs free energy ΔG^o for adsorption [12], [13].

$$\Delta G^o = -RTLnK_o \quad (3)$$

The values of ΔG^o at 288.15, 298.15 and 309.15K were summarized in Tables 1, 2, and 3 for adsorption of atrazine, picloram and propanil respectively. The values of ΔG^o for adsorption of studied pesticides were negative and decreased with temperature rise indicating that adsorption of pesticides on the soils were spontaneous with a high preference of the soil surface. The data revealed that adsorption of pesticides were in the following order: propanil > atrazine > picloram. The magnitude of ΔG^o also showed that the interactions of pesticides with the soil were thermodynamically spontaneous process and adsorption occurred through a bonding mechanism. The ΔG^o values were in the range -30.549 to -3.172 KJmol^{-1}, -33.614 to - 4.2897 KJmol^{-1}, and - 14.72 to - 76.412 KJmol^{-1} for atrazine, picloram and propanil respectively. The results obtained in the present study are similar to those of Gupta et al [14] who reported an increase in the values of ΔG^o with temperature. Variation of ΔG^o with temperature may be due to the increase in the degree of freedom of adsorbed molecules, which enhances desorption rather than adsorption at higher temperatures [15], [16].

3.3 Standard Enthalpy Change

The standard enthalpy change of adsorption ΔH^o represents the difference in binding energies between the solvent and the soil with the pesticides. Values of ΔH^o were determined graphically from the slope of the plot of $\ln K_o$ vs. 1/ T Fig. 1 a, b, and c using the following "equation" [11].

$$\left[\frac{dLnK_o}{d(\frac{1}{T})}\right] = \frac{-\Delta H^o}{R} \quad (4)$$

Values of ΔH^o were summarized in Table 4, followed the range -5.010 to -0.738, -4.769 to -0.848 and -75.779 to -1.628 KJmol^{-1} for atrazine, picloram and propanil respectively. The negative values of ΔH^o indicated the exothermic behaviors of the reaction. The linear nature of the plot indicates that the mechanism of adsorption is not changed as temperature is changed. The values of R^2 were in the range 0.9999 to 0.902, 0.989 to 0.765, and 0.902 to 0.999 for atrazine, picloram and propanil respectively. The negative enthalpy of adsorption indicates an exothermic binding [17], [18]. Showing that the interaction of pesticides with the soil is an energetically stable exothermic process and the adsorption occurred through a bonding mechanism. The ΔH^o values explain the binding strength of pesticides to the soil; the lower negative value of ΔH^o indicates stronger binding. Thus low values of ΔH^o pointed towered chemisorptions; hence the herbicides adsorption may be due to coordination and /or protonation, hydrogen bonding and dipole association and van der Waal's forces. This indicates that the interactions between the pesticides and the studied soil samples were stronger at lower temperature.

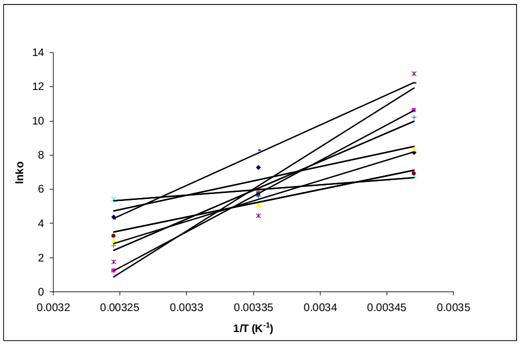

Fig 1 **a Variation of ln K_o with 1/T for adsorption of atrazine on the eight soil samples (♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ●S_6, +S_7, -S_8)**.

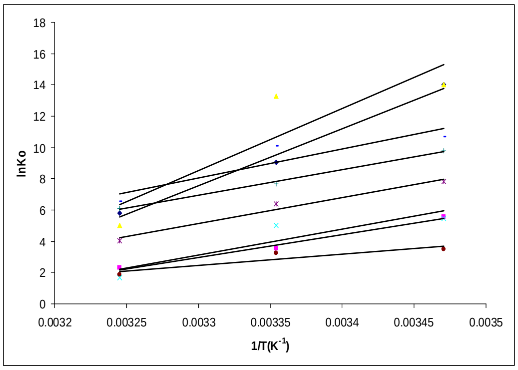

Fig.1. b **Variation of ln K_o with 1/T for adsorption of picloram on the eight soil samples (♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ●S_6, +S_7, -S_8)**.

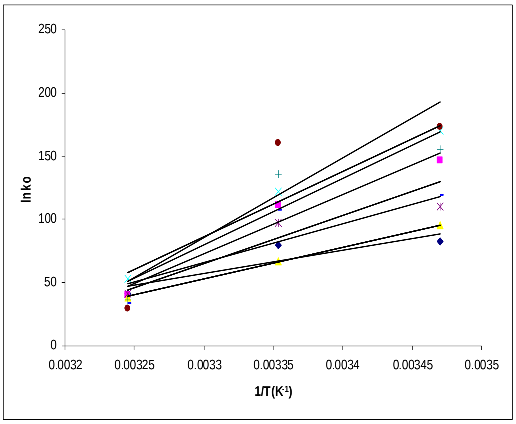

Fig. 1 c **Variation of ln K_o with 1/T for adsorption of propanil, on the eight soil samples (♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ●S_6, +S_7, -S_8)**.

3.4 Isosteric Enthalpy Of Adsorption

The isosteric enthalpy of adsorption ΔH is the standard enthalpy of adsorption at a fixed surface coverage. Values of ΔH were calculated by the expression [17].

$$\Delta H = R[\frac{dLnC_e}{d(\frac{1}{T})}]x \quad (5)$$

Where x is amount of pesticide adsorbed, and the average was calculated for each concentration. The values of ΔH Table 5 were in the following range -0.05 to -0.0084 $kJmol^{-1}$, -0.0057 to - 0.014 $kJmol^{-1}$, and -0.0141 to -0.0034 $kJmol^{-1}$ for atrazine, picloram and propanil respectively. The values of ΔH of adsorption as a function of amount of pesticide adsorbed was almost the same for all the three pesticides on eight soils that support our inference regarding the mechanism of adsorption. These values were relatively small and were of the order which was consistent with a physical type of adsorption [18].

3.5 Standard Entropy Change

The values of standard entropy change ΔS^o of adsorption were determined by using the "equation bellow" [18].

$$LnK_o = \frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R} \quad (6)$$

The values of ΔS^o were determined from the plot of $-lnK_o$ against 1/ T and the results were summarized in Table 4. The values of ΔS^o followed the range -16.138 to -1.757 $Jmol^{-1}$ k^{-1}, -14.711 to -2.502 $Jmol^{-1}$ k^{-1} and -199.01 to - 62.457 $Jmol^{-1}k^{-1}$ for atrazine, picloram, and propanil respectively. The negative values of ΔS^o indicate that the adsorption of the herbicide formed on all eight soil samples were stabilization, association, fixation or immobilization of the pesticides molecules as a result of adsorption decreased the degree of freedom, causing negative entropy effect[19], [20]. The values of ΔS^o pointing to the formation of the complexity by coordination or association of the herbicides and an exchangeable cation with the resultant of the loss in the degree of freedom of the pesticide.

3.6 Organic Matter Normalized Free Energy Change Of Adsorption

Organic matter was the most important factor that governed the adsorption of pesticides on soils. The organic

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matter normalized free energy changes ΔG^o_{OM} of adsorption of atrazine, picloram and propanil were calculated by using the following equation [21].

$$\Delta G^o{}_{OM} = -RTLnK_{OM} \quad (7)$$

The values of ΔG^o_{OM} in Table 6 were in the range -8.0106 to -10.188, -10.629 to -7.7422, and - 7.7984 to - 2.0458 $KJmol^{-1}$ for atrazine, picloram and propanil respectively. The negative values of ΔG^o_{OM} revealed that the adsorption of the three pesticides on the eight soil samples are spontaneous process and the adsorption has physical nature [22]. This signifies that there is a constant partitioning of the three pesticides. Between soil and solution, and water molecules do not pose strong competition for the adsorption sites, and also more affinity of the studied pesticides towards soil particulate matter than soil solution. Because most of the available sites in these soils are probably present at the surface of SOM and are therefore readily available for adsorption. The values of ΔG^o_{OM} for adsorption under the effect of temperatures were in the order T_1> T_2 >T_3 for the studied pesticides (with some exceptions). This indicates that as the temperature increases the adsorption coefficients decrease for atrazine, picloram and propanil.

4 Temperature Dependency Of Adsoption Coefficient

Variation of adsorption coefficient with temperature Fig. 2 a, b and c indicted that as the temperature increased the values of adsorption coefficient for these herbicides decreased. This can be explained by the types of the interactions between the pesticides and soil through adsorption that may occur as a result of two types of forces: enthalpy-related and entropy-related forces [23]. Hydrophobic bonding is anexample of an entropy-driven process; it is due to a combination of London dispersion forces (instantaneous dipole-induced dipole) associated with large entropy changes resulting from the removal of the sorbate from the solution. For polar chemicals, the enthalpy-related forces are greater, due to the additional contribution of electrostatic interactions. A small temperature effect was detected with the adsorption of studied pesticides by the soil samples; this behavior has been interpreted as due to physical adsorption. Results obtained in the present study are similar to those reported by Biggar et al [11].

4 Conclusion

Adsorption experiments were conducted at 15, 25, and 35°C to study the thermodynamic (equilibrium) parameter, associated the adsorption of the studied pesticides on the selected soil samples. The net effect of pesticide adsorption associated to the temperature. Adsorption efficiency for the studied pesticides was found to depend on the nature of adsorbent and adsorbate and the nature of the interactions between them.

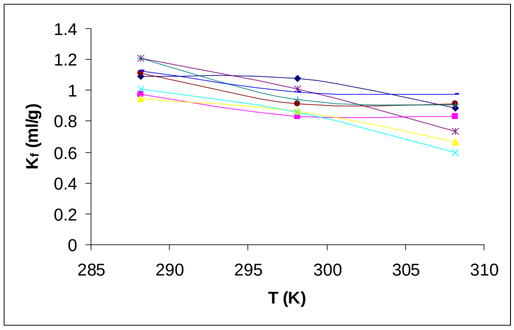

Fig. 2.a **Variation of adsorption coefficient with Temperature for Atrazine on the eight soil samples(♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ● S_6, + S_7 , - S_8)**.

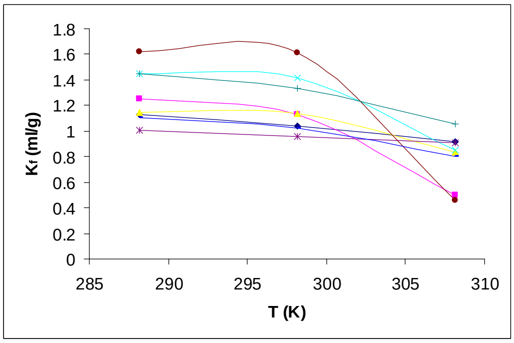

Fig.1. b **Variation of ln K_o with 1/T for adsorption of picloram on the eight soil samples (♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ● S_6, + S_7, - S_8)**.

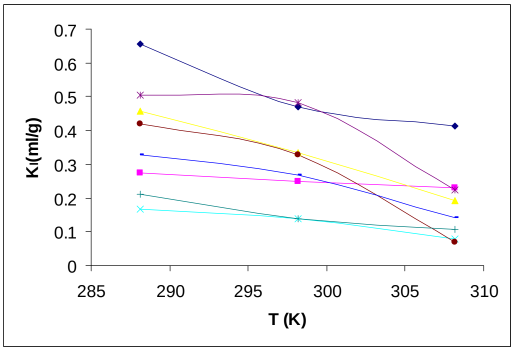

Fig 2 c **Variation of ln K_o with 1/T for adsorption of propanil, on the eight soil samples (♦ S_1, ■ S_2, ▲ S_3, x S_4, * S_5, ●S_6, +S_7, -S_8)**.

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TABLE 1
EQUILIBRIUM CONSTANTS AND STANDARD FREE ENERGY CHANG AT THREE TEMPERATURES FOR ADSORPTION OF ATRAZINE ON THE SELECTED SOIL SAMPLES

Soil	T_1 K		T_2 K		T_3K	
	lnK_0	ΔG^o (KJ/mol)	lnK_0	ΔG^o(KJ/mol)	lnK_0	ΔG^o(KJ/mol)
S_1	8.154	-19.534	7.271	-18.023	4.365	-11.185
S_2	10.64	-25.482	5.783	-14.334	1.238	-3.172
S_3	8.365	-20.039	5.106	-12.657	2.973	-7.618
S_4	6.820	-16.339	5.713	-14.162	5.446	-13.953
S_5	12.75	-30.549	4.467	-11.073	1.754	-4.493
S_6	6.918	-16.573	5.703	-14.136	3.273	-8.386
S_7	10.24	-24.526	5.557	-13.775	2.711	-6.944
S_8	12.20	-29.218	8.26	-20.465	4.24	-10.856

TABLE 2
EQUILIBRIUM CONSTANTS AND STANDARD FREE ENERGY CHANG AT THREE TEMPERATURES FOR ADSORPTION OF PICLORAM ON THE SELECTED SOIL SAMPLES

Soil	T_1K		T_2K		T_3 K	
	lnK_0	ΔG^o (KJ/mol)	lnK_0	ΔG^o(KJ/mol)	lnK_0	ΔG^o(KJ/mol)
S_1	14.03	-33.616	9.029	-22.382	5.817	-14.902
S_2	5.57	-13.349	3.561	-8.8273	2.287	-5.8602
S_3	14.03	-33.614	13.28	-32.919	5.013	-12.842
S_4	5.46	-13.076	5.009	-12.416	1.674	-4.2897
S_5	7.79	-18.678	6.407	-15.881	4.021	-10.300
S_6	3.48	-8.333	3.256	-8.0710	1.876	-4.8065
S_7	9.79	-23.472	7.653	-18.969	6.107	-15.648
S_8	10.7	-25.612	10.09	-25.024	6.520	-16.704

TABLE 3
EQUILIBRIUM CONSTANTS AND STANDARD FREE ENERGY CHANG AT THREE TEMPERATURES FOR ADSORPTION OF PROANIL ON THE SELECTED SOIL SAMPLES

Soil	T_1 K		T_2 K		T_3 K	
	$\ln K_0$	ΔG^o(KJ/mol)	$\ln K_0$	ΔG^o(KJ/mol)	$\ln K_0$	ΔG^o(KJ/mol)
S_1	83.00	-198.85	79.87	-197.99	41.35	-105.93
S_2	146.2	-350.18	111.3	-275.97	40.54	-103.87
S_3	95.06	-227.74	66.88	-165.79	38.87	-99.593
S_4	170.6	-408.79	122.3	-303.13	53.42	-136.87
S_5	110.4	-264.51	97.73	-242.28	41.35	-105.93
S_6	173.1	-414.72	160.6	-397.98	29.83	-76.412
S_7	155.7	-372.98	135.6	-336.15	36.40	-93.257
S_8	119.3	-285.73	107.3	-265.95	33.09	-84.764

TABLE4
STANDARD ENTHALPY CHANGE AND STANDARD ENTROPY CHANGE (DETERMINED GRAPHICALLY) FOR ADSORPTION OF ATRAZINE, PICLORAM, AND PROPANIL ON THE SELECTED SOIL SAMPLES

Soil	atrazine			picloram			propanil		
	ΔH^o (KJ/mol)	R^2	ΔS^o J/mol.k	ΔH^o (KJ/mol)	R^2	ΔS^o (J/mol.k)	ΔH^o (KJ/mol)	R^2	ΔS^o (J/mol.k)
S_1	-2.010	0.902	-5.953	-4.396	0.989	-13.597	-22.025	0.791	-65.57
S_2	-5.019	0.999	-16.14	-1.757	0.988	-5.442	-56.168	0.955	-176.64
S_3	-2.885	0.989	-9.023	-4.769	0.797	-14.711	-29.995	0.999	-92.628
S_4	-0.738	0.901	-1.757	-2.003	0.823	-6.235	-1.628	0.986	-62.457
S_5	-5.904	0.931	-19.057	-2.009	0.972	-6.015	-36.605	0.869	-112.86
S_6	-1.938	0.957	-5.869	-0.848	0.838	-2.502	-75.779	0.799	-239.79
S_7	-4.029	0.985	-12.781	-1.973	0.995	-5.679	-63.204	0.859	-199.01
S_8	-4.247	0.999	-13.266	-2.208	0.841	-6.318	-45.634	0.838	-142.76

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TABLE 5
ISOSTERIC HEAT CHEAHG OF ADSORPTION OF ATRAZINE, PICLORAM, AND PROPANIL ON THE SELECTED SOIL SAMPLES.

Soil	atrazine X(J/mol)		picloram X (KJ/mol)		propanil X(J/mol)	
	$T_1:T_2$	$T_2:T_3$	$T_1:T_2$	$T_2:T_3$	$T_1:T_2$	$T_2:T_3$
S_1	-0.0183	-0.0135	-0.0105	-0.0087	-0.0141	-0.0093
S_2	-0.0243	-0.0042	-0.0115	-0.0082	-0.0113	-0.0069
S_3	-0.0289	-0.0120	-0.0119	-0.0059	-0.0096	-0.0089
S_4	-0.0291	-0.0202	-0.0140	-0.0057	-0.0112	-0.0030
S_5	-0.0501	-0.0084	-0.0116	-0.0091	-0.0117	-0.0739
S_6	-0.0215	-0.0119	-0.0119	-0.0108	-0.0131	-0.0034
S_7	-0.0327	-0.0100	-0.0129	-0.0085	-0.0126	-0.0050
S_8	-0.0183	-0.0145	-0.01231	-0.0102	-0.0130	-0.0056

TABLE 6
ORGANIC MATTER NORMALIZED FREE ENERGY CHANGE OF ADSORPTIO OF ATRAZIN, PICLORAM, AND PROPANIL ON THE SELECTED SOIL SAMPLES

Soil	atrazine			picloram			propanil		
	ΔG^o_{OM} (KJ/mol)			ΔG^o_{OM} (KJ/mol)			ΔG^o_{OM} (KJ/mol)		
	T_1	T_2	T_3	T_1	T_2	T_3	T_1	T_2	T_3
S_1	-9.001	-8.935	-8.746	-8.757	-8.854	-8.8203	-7.449	-6.890	-6.792
S_2	-9.967	-9.546	-9.861	-10.22	-10.311	-8.5742	-6.566	-6.564	-6.564
S_3	-9.905	-9.662	-9.297	-10.01	-10.325	-9.8902	-7.798	-7.297	-6.116
S_4	-10.19	-9.707	-9.100	-10.63	-10.942	-10.011	-5.459	-5.181	-3.908
S_5	-9.029	-8.545	-8.011	-8.247	-8.427	-8.5593	-6.597	-6.719	-4.978
S_6	-9.720	-9.204	-9.517	-10.26	-10.599	-7.7422	-7.031	-6.669	-8.424
S_7	-9.795	-9.137	-9.354	-9.863	-10.009	-9.7379	-5.257	-4.376	-2.046
S_8	-9.319	-8.969	-9.236	-8.928	-9.051	-8.7286	-5.829	-5.732	-5.759