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**Determination of Acid Ionization Constants for Weak Acids by
Osmometry and the Instrumental Analysis Self-Evaluation Feedback
Approach to Student Preparation of Solutions**

Nyanguila Kakolesha

A Dissertation Presented to the Graduate Faculty of Middle Tennessee State University in
Partial Fulfillment of the Requirements for the Doctor of Arts Degree in Chemistry

August, 2001

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Approach to Student Preparation of Solutions**

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ABSTRACT

Determination of Acid Ionization Constants for Weak Acids by Osmometry and the Instrumental Analysis Self-Evaluation Feedback Approach to Student Preparation of Solutions

By Nyanguila Kakolesha

One focus of this work was to develop of an alternative method to conductivity for determining the acid ionization constants. Computer-controlled osmometry is one of the emerging analytical tools in industrial research and clinical laboratories. It is slowly finding its way into chemistry laboratories. The instrument's microprocessor control ensures shortened data collection time, repeatability, accuracy, and automatic calibration. The equilibrium constants of acetic acid, chloroacetic acid, bromoacetic acid, cyanoacetic acid, and iodoacetic acid have been measured using osmometry and their values compared with the existing literature values obtained, usually, from conductometric measurements. Ionization constant determined by osmometry for the moderately strong weak acids were in reasonably good agreement with literature values. The results showed that two factors, the ionic strength and the osmotic coefficient, exert opposite effects in solutions of such weak acids.

Another focus of the work was analytical chemistry students solution preparation skills. The prevailing teacher-structured experiments leave little room for students'

ingenuity in quantitative volumetric analysis. The purpose of this part of the study was to improve students' skills in making solutions using instrument feedback in a constructivist-learning model. After making some solutions by weighing and dissolving solutes or by serial dilution, students used the spectrophotometer and the osmometer to compare their solutions with standard solutions. Students perceived the instrument feedback as a non-threatening approach to monitoring the development of their skill levels and liked to clarify their understanding through interacting with an instructor-observer. An assessment of the instrument feedback and the constructivist model indicated that students would assume responsibility for their own learning if given the opportunity.

This study involved 167 students enrolled in Quantitative Chemical Analysis from fall 1998 through spring 2001. Surveys eliciting students' reactions to the instrument feedback approach showed an overwhelmingly positive response. The results of this research demonstrated that self-evaluation with instrumental feedback was a useful tool in helping students apply the knowledge they have acquired in lectures to the practice of chemistry. A demographic survey to determine whether part-time or full-time jobs had a negative impact on their experiment grades showed a small but significant correlation between hours worked and grade earned. However, the study showed that grades students earned on this experiment were predictive of overall semester lab grades.

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Part 1

Determination of Ionization Constants for Weak Acids by Osmometry

Chapter 1

Introduction

*“To be conscious that you are ignorant of the facts is a great step to knowledge”
-Benjamin Disraeli (1804-1881)*

Background

Many substances, when dissolved in some liquid solvent, dissociate (or ionize) to some extent into charged particles called ions. The dissociation is an equilibrium whose position depends on the substance. If the dissociation is essentially complete, the substance is called a strong electrolyte. Incomplete dissociation is found for weak electrolytes. When an electrolyte dissociates, the resulting ions interact with the surrounding solvent molecules or ions, a process known as solvation, to form charged clusters known as solvated ions. These solvated ions can move through the solution under the influence of an externally applied electrical field. Such motion of charge is known as ionic conduction and the resulting current is ionic current. The ionic current is determined by the nature of the ions, their concentrations, the solvent, and the electric field applied.

Conductance, whether ionic or electronic is the reciprocal of resistance. The resistance, R is defined as

$$R = \rho(l/A)$$

where l is the length of a sample of the electrolyte and A is the cross sectional area. The symbol ρ is the proportionality constant and is a property of the solution. This property is called resistivity. Conductance as such is rarely of analytical use because the conductance of any conductor, solution, or metal depends upon the size and shape of the conductor. To

remove the dependency upon the size and shape of the conductor requires the use of the reciprocal of resistivity called conductivity, κ .

By definition

$$\kappa = 1/\rho = l/AR.$$

The value of the conductivity of any substance is characteristic of that substance.

The SI unit for conductivity is Siemens per meter, S/m. Conductivity is measured with a cell consisting of two platinum electrodes a fixed distance apart. These electrodes are coated with finely divided, electrodeposited platinum black. The conductivity of a solution of a strong electrolyte decreases as the solution concentration decreases. The molar conductivity, Λ is defined as:

$$\Lambda = \kappa/C$$

Experimentally, the value of Λ is found to be independent of C , the concentration, for any electrolyte whenever the solution is sufficiently dilute. Molar conductivity can be measured for any salt, acid, or base in aqueous or non-aqueous solution. For interpretation of the processes, however, the value of Λ_0 is of more fundamental importance than that of Λ because Λ_0 , known as the molar conductivity extrapolated to zero concentration or at infinite dilution, is characteristic only of the ions and solvent and is independent of any ionic interactions. Equipped with a computer-assisted conductivity meter, this researcher determined the equilibrium ionization constant of acetic acid based on the method developed by MacInnis and Shedlovsky [1].

It is also known that the presence of solutes in water affects the colligative properties of the solutions in a predictable manner, dependent on the total concentration of

the solute particles and independent of the number or types of solutes, their molecular masses, particle sizes, or densities. Colligative properties include the vapor pressure, which decreases with increasing concentration of the solute, the osmotic pressure, the freezing point, which decreases with increasing concentrations, and the boiling point, which increases with increasing concentrations. Any one of these properties can be used to calculate the total concentration (osmolality) of the solute particles in the solution.

This dissertation proposes an alternative method to conductivity for determining the degree of ionization of weak acids in water. The proposed new method uses a computer-controlled osmometer to measure the degree of dissociation and by extension, to calculate the thermodynamic equilibrium constants of the weak acids. This method may be preferred over other methods because of its speed, small sample size and ease of measurement. The osmometer uses the freezing point depression method to determine osmolality according to the expression

$$\text{Osmolality} = \text{molality} \cdot n \cdot \phi$$

where n is the total number of particles in solution (ions and molecules), and ϕ is the osmotic coefficient. The percent ionization, which may be determined by measuring colligative properties if the ionization is sufficiently large, provides a means of calculating the ionization constant. The assumption is made that the amount of the acid that is ionized equals the amount of hydrogen ions formed.

Computer assisted osmometry is one of the emerging analytical tools in industrial research and clinical laboratories. The applications of osmometry across a number of medical disciplines assure that the technique will be heavily used in clinical situations to support a variety of patient needs. Because it requires sample sizes as small as 250- μL ,

osmometers can be used in industrial and educational applications where sample sizes may be limited in order to reduce cost and hazardous chemical disposal problems. The instrument's microprocessor control ensures shortened data collection time, repeatability, accuracy, and automatic calibration. The degree of ionization and the thermodynamic equilibrium constants determined by the osmometric method can be used for a variety of purposes, among these the calculation of hydrogen ion concentration or pH. The results of this research will be evaluated against the existing literature values obtained from spectrophotometric, potentiometric, and conductometric measurements.

Review of Literature

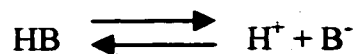
The study of the chemistry of aqueous solutions and their properties occupied much of the latter part of the 19th century and the early part of the 20th century. Claude Louis Berthollet (1748-1822) introduced the idea of the "effect of mass" in 1801 when it was realized that a chemical reaction does not necessarily go to completion; he used the concept of chemical equilibrium. It was Cato Maximillia Gueldberg and Peter Waage who derived the law of mass action in 1864 [2].

It was the need to evaluate the degree of ionization that led to the development of the instruments for its direct measurement. In an attempt to validate Ohm's law, Kohlrausch [3] developed the concepts of equivalent conductance and ionic conductance while measuring specific conductance of solutions of several acids. Since that time, several theories had been advanced in an attempt to explain phenomena occurring in solutions of electrolytes. The modern view of the properties of solutions of electrolytes is the Debye-Hückel theory mentioned in various texts [4, 5]. It takes account of the fact that

the ions are not fully independent, but must attract and repel each other in accordance with Coulomb's law. As a result, the presence of these electrostatic forces causes any selected ion, a positive one for example, to have more negative ions near it than if the distribution were purely random. This phenomenon is referred to as the ionic atmosphere of the selected ion. The effect of the presence of an ionic atmosphere is to reduce the activity coefficient of the ions leading to a tendency of decreasing the ion mobility with increasing ion concentration.

However, the theory of electrolytic conductivity in its present form is to a large extent the work of Onsager [6]. Later the experimental work of MacInnis and Shedlovsky [1] on acetic acid and by Saxton and Lange [7] on chloroacetic acid demonstrated that the interionic attraction theory, although in agreement with measurements of aqueous solutions of strong electrolytes, is equally useful in dealing with weak electrolytes. The results of their experiments led to the investigation and the determination of ionization constants of weak acids by the conductance method.

A quantitative prediction can be made on the effect of ionization of a weak acid on the equilibrium by taking into account the activities of the species in the equilibrium. If the ionization of any weak acid in water is written as



the thermodynamic equilibrium constant, K is

$$K = \frac{a_{\text{H}^+} \cdot a_{\text{B}^-}}{a_{\text{HB}}} = \frac{[\text{H}^+] \gamma_{\text{H}^+} \cdot [\text{B}^-] \gamma_{\text{B}^-}}{[\text{HB}] \gamma_{\text{HB}}}$$

where a 's are activities and γ 's are activity coefficient and square brackets indicate the molar concentration of each species at equilibrium. Since the apparent (concentration) equilibrium constant is

$$K_c = \frac{[H^+][B^-]}{[HB]}$$

then

$$K = K_c \cdot \frac{\gamma_{H^+} \cdot \gamma_{B^-}}{\gamma_{HB}}$$

In very dilute solutions, the activity of HB is nearly the same as its concentration, however, the activity of ionic species depends on the ionic strength of the solution. Hence, at zero ionic strength, $K = K_c$. The degree of ionization of weak acid, α is found from the concentration of hydrogen ion at equilibrium and the initial concentration of the acid according to the relation

$$\alpha = \frac{[H^+]}{[HB]}$$

where α is the fraction of the acid that is ionized. From the degree of ionization of the acid, the ionization constant is determined based on the following expression

$$K_c = \frac{[H^+][B^-]}{[HB]} = \frac{\alpha^2 C}{1 - \alpha}$$

It was the need to evaluate the degree of ionization that led to the development and application of conductometry, potentiometry, and spectrophotometry for its direct measurement. Although the conductance method provides nearly accurate values of these thermodynamic ionization constants of weak acids at very low concentrations, it is

however time consuming and labor intensive. It requires freshly prepared solutions, tedious mathematical manipulations, and suffers from temperature sensitivity.

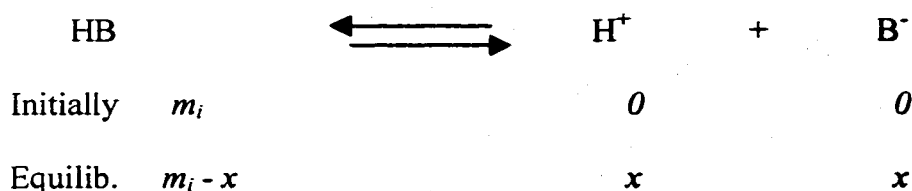
The potentiometric method has also been applied to the evaluation of the ionization constants of weak acids [8, 9]. It consists of titrating a weak acid with a strong base and requires determining the ionization constants from the resulting titration curves. However, the reported tendency of the resulting ions to hydrolyze in aqueous solution makes the method inappropriate [10] in some instances. Other methods [11] suitable for the determination of ionization are spectrophotometry and phase equilibrium studies.

For this research, a method was developed to determine the degree of ionization by measuring the freezing point depression of weak acid solutions using an osmometer. For colligative determinations of this type, concentrations must be expressed in units of osmolality (moles of solute particles per kilogram of water) rather than molarity. The freezing point depression is defined as

$$\Delta T_f = K_f \cdot m$$

where ΔT_f is the freezing point depression, K_f is molal freezing point depression constant (1.858 °C/Osmol/Kg), and m is concentration in Osmol per Kg of water.

Consider the aqueous ionization of any weak acid HB:



where m_i is the initial concentration of weak acid in moles per kilogram of water

and x is the concentration of ionized weak acid in moles per kilogram of water at equilibrium. The total number of moles of all particles present per kilogram of water at equilibrium is

$$(m_i - x) + x + x = m_i + x$$

This sum equals the total particle concentration of the solution in osmole per kilogram of water. The solution's osmolality then is abbreviated Osm ($Osm = Osmol/Kg$ of water)

$$m_i + x = Osm$$

$$x = Osm - m_i$$

From the equilibrium equation,

$$\alpha = \frac{x}{m_i}$$

Substituting for x ,

$$\alpha = \frac{Osm - m_i}{m_i}$$

Knowing the value of α will provide a means of calculating the ionization constant, K_a

$$K_a = \frac{(Osm - m_i)^2}{2m_i - Osm}$$

Chapter 2

Materials and Methods

*"If we knew what it was we were doing, it would not be called research, would it?"
-Albert Einstein (1879-1955)*

Chemicals

Bromoacetic acid, cyanoacetic acid, and iodoacetic acid (Aldrich, Milwaukee, WI), chloroacetic acid (Fisher, Fair Lawn, NJ), and acetic acid (J.T. Baker, Phillipsburg, NJ) were used without further purification. The solvent was a high grade (>17 Mohm resistance) water, filtered through Modulab™ Polisher I HPLC, a laboratory reagent grade water system supplied by Continental Water System Corporation (USFilter Wallace & Tiernan Products, 1901 West Garden Road, Vineland, New Jersey 08360).

Instrumentation

Conductivity measurements were obtained using an YSI Model 3100 Conductivity Meter (YSI, Inc. 1725 Brannum Lane, Yellow Springs, OH). It was equipped with cell Model 3403 having a cell constant, $K = 1.0/\text{cm}$. The YSI Model 3100 is a microprocessor-equipped instrument designed to perform measurement of conductivity, salinity, and temperature. The microprocessor allows the instrument to be easily calibrated; it also performs a self-diagnostic routine each time the instrument is turned on providing useful information about the cell constant, etc. The instrument simultaneously displays temperature (in °C) along with conductivity (in $\mu\text{S}/\text{cm}$ or mS/cm) and others useful parameters of choice.

All osmometric measurements were carried out using an Osmette XL, Model 5007 Autocal osmometer (Precision System, Inc., Sudburg, MA). This unit is a freezing point depression instrument equipped with a microprocessor control module that ensures data accuracy and reproducibility. It has two sample capacities (250 μ L or 2 mL) with a resolution of 1 mOsm/kg and a reproducibility of 2 mOsm/kg between 100 and 500 mOsm/kg, and of 0.5% between 500 and 3000 mOsm/kg. The calibration of the unit was achieved with commercially available standard solutions of sodium chloride (100 mOsm/kg; lot No. 901081, 500 mOsm/kg, lot no. 812262, Precision System, Inc).

Sample preparation

Conductivity measurements

A 0.1098 N stock solution of acetic acid was prepared from glacial acetic acid (Baker Analyzed Reagent, Lot No 38667) and standardized by titration with 0.09806 N NaOH, made from 50% w/w NaOH solution (Fisher Scientific, Lot No 982713-24). The solution of NaOH was in turn standardized with standard potassium hydrogen phthalate (Fisher Scientific, Lot No 976510). In both titration instances, phenolphthalein solution was used as indicator. The serial dilutions were made from the stock solution, N_0 (0.1098 N), by taking a 25-mL aliquot of N_0 and diluting it to 50 mL in volumetric flask to make N_1 . Repeating the process by diluting N_1 to N_2 , ----- N_i , all solutions were diluted in a cascade fashion by a factor of 2 in the following manner:

Dilute	To make	Resulting concentration
N_0	N_0	0.1098 N
$N_0/2$	N_1	0.05498 N
$N_1/2$	N_2	0.02745 N
$N_2/2$	N_3	0.01372 N
$N_3/2$	N_4	0.006862 N
$N_4/2$	N_5	0.003431 N
$N_5/2$	N_6	0.001716 N
$N_6/2$	N_7	0.0008578 N
$N_7/2$	N_8	0.0004289 N

Conductance measurements were made at a solution temperature of $25\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$.

The calibration of the conductivity meter was made with a standard solution of KCl (1500 μS , Lot No 7-0925, Oakton®, Cole-Parmer, 625 East Bunker Court, Vernon Hills, Illinois 60061-1844), and the cell constant, K was set to 1.0/cm. The cell was rinsed several times with reagent grade water and with the solution of interest before use. The probe was immersed in a test tube containing about 20 to 25 mL of the solution to be measured, until all of the platinum rings and the vent hole of the cell were covered. The reading was taken as quickly as possible to minimize the dissolution of carbon dioxide into the solution. All measurements were made on the day in which the solutions were prepared.

Osmometric measurements

Stock solutions of acetic acid (lot No. 944818), bromoacetic acid (lot No. 04330 PU), chloroacetic acid (lot No. 984686), cyanoacetic acid (lot no. 00331JR) and iodoacetic acid (lot No. 08514 JS) were made gravimetrically in high purity water. They were serially diluted by accurately weighing aliquots of stock solutions and diluting them with a weighed amount of water. The resulting concentrations were therefore in moles of solute per kilogram of water.

The Osmette's temperature equilibration required a 10 to 15 minutes warm-up period. A 250 μ L sample of the weak acid solution was pipetted into a sample tube and placed into the cooling chamber maintained at a temperature below the freezing point of the solution; then the probe fitted with stirring needle was lowered into the sample. As the sample was supercooled, the crystallization was initiated by mechanical vibration of the needle. The instrument automatically sensed the freezing point of the solute, and then computed and displayed the osmotic concentration in milliosmoles of particles per kilogram of water (mOsm). Readings were taken at least in triplicate, but usually five readings per solution were acquired.

Special care was taken in handling sample tubes. Prior to use, sample tubes were washed several times with tap water, then rinsed with de-ionized water, followed by soaking overnight in high purity water. A wash bottle was used to pass a high-pressure stream of rinse water into the bulb-like bottom of the tubes. The tubes were dried in the oven at 110° C for at least two hours, and then placed in a watch glass-covered beaker to cool before use. Tubes to be used after an extended period of time were stored in a dust free environment.

Chapter 3

Results and Analysis

"If an experiment requires statistical analysis to establish a result, then one should do a better experiment."

-Ernest Rutherford (1871-1937)

Conductivity

The MacInnis-Shedlovsky method [1].

Table 4 displays the data collected for the measurement of the equilibrium constant for acetic acid.

Table 1. Conductance measurements and Ka calculation

Conc. (C) Equiv./L	Ls of Soln	Corrected Ls	Λ_c	Λ_∞	α	K'	\sqrt{C}	Log K'
1.10×10^{-1}	5.23×10^{-4}	5.23×10^{-4}	4.75	358	1.33×10^{-2}	1.96×10^{-5}	0.332	-4.71
5.49×10^{-2}	3.69×10^{-4}	3.69×10^{-4}	6.72	364	1.84×10^{-2}	1.90×10^{-5}	0.234	-4.72
2.75×10^{-2}	2.59×10^{-4}	2.59×10^{-4}	9.41	370	2.54×10^{-2}	1.82×10^{-5}	0.166	-4.74
1.37×10^{-2}	1.84×10^{-4}	1.84×10^{-4}	13.4	375	3.58×10^{-2}	1.82×10^{-5}	0.117	-4.74
6.86×10^{-3}	1.29×10^{-4}	1.29×10^{-4}	18.8	379	4.95×10^{-2}	1.77×10^{-5}	0.0828	-4.75
3.43×10^{-3}	8.90×10^{-5}	8.87×10^{-4}	25.9	382	6.76×10^{-2}	1.68×10^{-5}	0.0586	-4.77
1.72×10^{-3}	6.10×10^{-5}	6.13×10^{-4}	35.6	385	9.26×10^{-2}	1.63×10^{-5}	0.0415	-4.79
8.58×10^{-4}	4.47×10^{-5}	4.46×10^{-4}	52.0	386	1.35×10^{-1}	1.79×10^{-5}	0.0293	-4.75
4.29×10^{-4}	2.90×10^{-5}	2.93×10^{-4}	68.3	388	1.76×10^{-1}	1.62×10^{-5}	0.0207	-4.79

In this method, the calculated value of the equilibrium constant is derived by the iterative method. As expressed in the following modified Debye-Hückel equation:

$$\Lambda_\infty (\text{Acetic acid}) = 390.59 - 148.61\sqrt{C_i} + 165.5 C_i (1 - 0.2274\sqrt{C_i})$$

where Λ_∞ is the conductance of completely dissociated acetic acid and C_i is the concentration of i th ion. The theoretical value of conductance at infinite dilution, Λ_0 , of

acetic acid was used as a limiting factor in the first approximation calculation. Assuming a completely dissociated acetic acid at high dilutions, the value of the conductance at infinite dilutions, Λ_∞ is determined from the ion concentration, C_i , defined as

$$C_i = \frac{1000L_s}{\Lambda_\infty}$$

where L_s is the specific conductance of the solution at the given concentration. The degree of dissociation can then be obtained by the use of the expression

$$C_i = \alpha C$$

The values of K_a from each value of C_i were obtained following the equation:

$$K_a = \frac{\alpha^2 C}{1 - \alpha} = \frac{C_i^2}{(C - C_i)}$$

The plot of the log K versus the square root of C_i (Figure 1) produced the following graph with a slope of 0.2491 and the intercept of -4.781 . The inverse log of the intercept, which is the K_a , has a value of 1.654×10^{-5} .

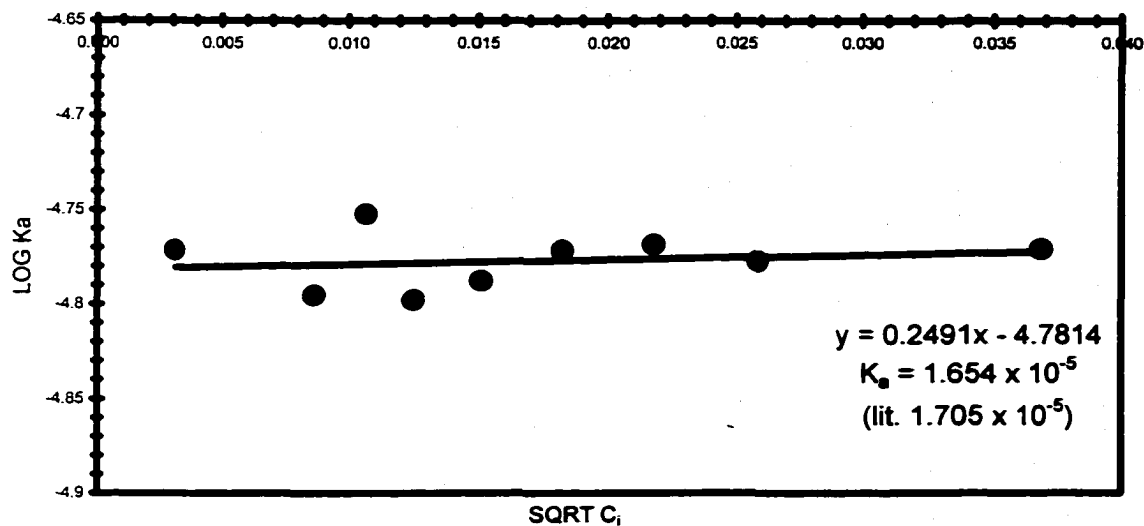


Figure 1. Plot of log K versus $\sqrt{C_i}$ based on MacInnis & Shedlovsky method

Instead of using the successive approximation computation, the degree of dissociation, α , could be equally calculated from the Arrhenius equation

$$\alpha = \Lambda_c / \Lambda_\epsilon$$

in which Λ_c is the equivalent conductance at concentration C. The value of Λ_ϵ is calculated using the modified Debye-Hückel equation as indicated above.

The plot of log K', the "concentration constant" not corrected for activity coefficients, versus square root of C (Figure 2) yielded a straight line with the intercept value of -4.780 and the slope value of 0.2381 . The inverse log of the intercept was found to be 1.661×10^{-5} , which is the value of the K_a .

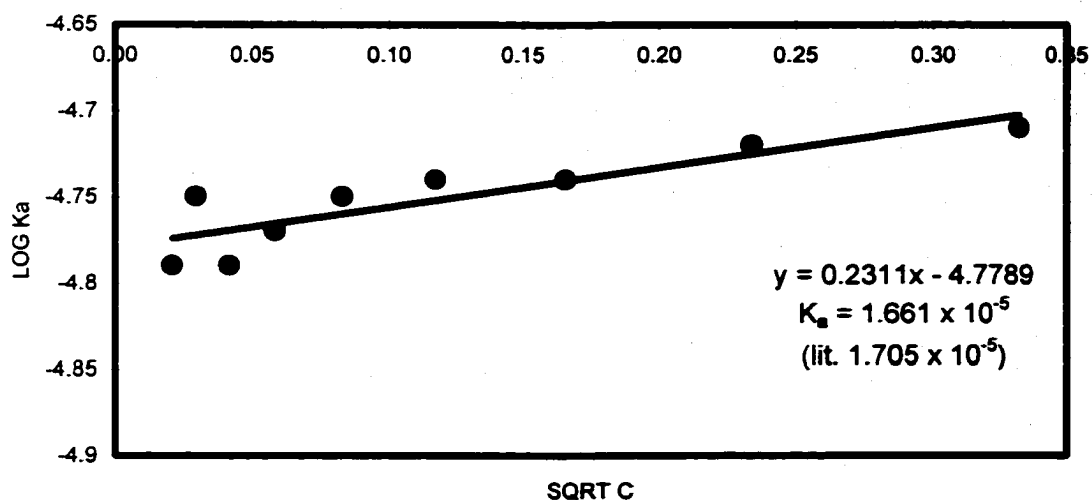


Figure 2. Plot of log K versus \sqrt{C} based on the modified MacInnis & Shedlovsky method

Osmometry

Tables 2 through 6 display solution preparations, average osmolality readings, and computation for ϕ . The fraction of the acid in the stock solution (weight factor) was calculated by dividing the mass of the acid dissolved in the stock solution by the total mass of the solution (the mass of acid plus mass of water). This fraction can also be referred to as percent acid if it is multiplied by 100%. The amount of acid in each dilution (column 4) was computed by multiplying the appropriate mass of stock solution (column 2) by the weight factor. The total mass of water (column 5) is the sum of the amount of water of dilution (column 3) and the difference between column 2 and column 4. The number of moles of solute in each dilution (column 6) was found by dividing the mass of the solute (column 4) by the gram molecular weight (GMW) of the solute. Molalities (column 7) were computed by first converting grams of solvent (column 5) into kilograms (i.e. dividing column 5 by 1000) and dividing the result into column 6.

Table 2

Dilution	g of stk soln	g water added	g HOAc in stk soln	Total g H ₂ O in soln	mol HOAc in soln	molality	Avg mOsm	phi
1	36.4144	50.13	3.904	82.64	0.06501	0.7867	759.67	0.9657
2	33.6203	50.00	3.604	80.02	0.06002	0.7501	723.67	0.9647
3	30.8070	50.03	3.303	77.53	0.05400	0.7094	684.67	0.9652
4	27.9913	50.01	3.001	75.00	0.04997	0.6663	643.67	0.9660
5	21.0113	50.00	2.253	68.76	0.03751	0.5456	528.33	0.9684
6	13.8834	50.10	1.488	62.50	0.02479	0.3966	384.33	0.9691
7	6.9854	50.02	0.7489	56.26	0.01247	0.2217	213.00	0.9608
8	5.6080	50.23	0.6012	55.24	0.01001	0.1813	174.67	0.9637
9	4.2091	50.00	0.4513	53.76	0.007514	0.1398	133.67	0.9562
10	2.1235	49.98	0.2277	51.88	0.003791	0.07308	72.00	0.9852
11	1.4039	50.01	0.1505	51.26	0.002506	0.04889	49.33	1.009
12	0.6981	50.00	0.07484	50.62	0.001246	0.02462	26.00	1.056
13	0.5568	50.00	0.05969	50.50	0.0009940	0.01968	21.33	1.084

Acetic acid dilution data

GMW = 60.05 g/mole

Stock solution: 24.0183g acid in 200.01g water

Molality of the stock solution = 2.000m

Wt factor = 0.1072

Table 3

Dilution	g of stk soln	g water added	g BrHAc in stk soln	Total g H ₂ O in soln	mol BrHAc in soln	molality	Avg mOsm	phi
1	31.9625	50.04	6.964	75.04	0.05012	0.6679	634.67	0.9502
2	23.9630	49.99	5.221	68.73	0.03758	0.5467	529.33	0.9683
3	15.9740	49.99	3.480	62.48	0.02505	0.4009	394.67	0.9845
4	7.9844	50.03	1.740	56.28	0.01252	0.2225	223.67	1.005
5	6.3931	50.03	1.393	55.03	0.01002	0.1822	186.00	1.021
6	4.7988	50.06	1.046	53.81	0.007525	0.1398	143.67	1.027
7	3.2340	50.02	0.7046	52.55	0.005071	0.09650	102.67	1.064
8	2.3892	50.01	0.5206	51.88	0.003746	0.07221	79.00	1.094
9	1.6093	50.03	0.3506	51.29	0.002523	0.04920	55.67	1.131
10	0.8368	50.01	0.1823	50.66	0.001312	0.02510	31.33	1.210
11	0.6486	50.09	0.1413	50.60	0.001017	0.02010	25.67	1.277
12	0.4804	50.05	0.1047	50.43	0.0007535	0.01494	19.67	1.317

Bromoacetic acid dilution data

GMW = 138.95 g/mole

Stock solution: 27.8629g of acid in 100.02g water

Molality of the stock solution = 2.005 m

WT factor = 0.2179

Table 4

Dilution	g of stk soln	g H ₂ O added	g ClHAc in stk soln	Total g H ₂ O in soln	mol CHAc in soln	molality	Avg mOsm	phi
1	29.7348	100.04	4.725	125.05	0.05000	0.3999	401.00	1.003
2	17.8463	100.11	2.836	115.12	0.03001	0.2607	265.80	1.020
3	14.8674	100.02	2.362	112.53	0.02500	0.2222	227.40	1.023
4	11.9020	100.01	1.891	110.02	0.02002	0.1819	189.60	1.042
5	8.9275	100.00	1.419	107.51	0.01501	0.1397	149.00	1.067
6	7.4556	100.03	1.185	106.30	0.01254	0.1179	126.60	1.073
7	5.9550	100.02	0.9462	105.03	0.01001	0.09535	103.60	1.087
8	4.4606	100.06	0.7088	103.81	0.007501	0.07226	81.20	1.124
9	2.9757	100.01	0.4728	102.51	0.005004	0.04882	57.60	1.180

Monochloroacetic acid dilution data

GMW = 94.50 g/mole

Stock solution: 18.8971 g acid in 100.03 g water

Molality of the Stock Solution = 1.999 m

Wt fraction = 0.1589

Table 5

Dilution	G of stk soln	g H ₂ O added	g CNHAc in stk soln	Total g H ₂ O in soln	mol CNHAc in soln	Molality	Avg. mOsm	phi
1	14.5921	50.07	2.126	62.54	0.02499	0.3997	400.00	1.001
2	11.6866	50.01	1.703	59.99	0.02002	0.3337	339.00	1.016
3	8.7676	50.04	1.277	57.53	0.01502	0.2610	271.33	1.039
4	5.9725	50.06	0.8702	55.16	0.01023	0.1855	198.67	1.071
5	4.3790	49.99	0.6380	53.73	0.007501	0.1396	153.00	1.096
6	3.6639	49.95	0.5338	53.08	0.006276	0.1182	131.67	1.114
7	2.9202	49.95	0.4255	52.45	0.005002	0.09538	109.00	1.143
8	2.1955	49.96	0.3199	51.84	0.003761	0.07255	85.33	1.176
9	1.4745	49.90	0.2148	51.16	0.002526	0.04937	61.67	1.249
10	0.7484	50.98	0.1090	51.62	0.001282	0.02483	34.33	1.383

Cyanoacetic acid dilution data

GMW= 85.06 g/mole
 Stock solution: 17.0553g of acid in 100.00 g water
 Molality of the stock solution = 2.005 m
 Wt factor=0.1457

Table 6

Dilution	g of stk soln	g water added	g IHAc in stk soln	Total g H ₂ O in soln	mol IHAc	molality	Avg mOsm	phi
1	29.6567	50.03	4.650	75.04	0.02501	0.3332	319.60	0.9591
2	23.7230	50.02	3.719	70.02	0.02000	0.2857	277.00	0.9697
3	17.7969	50.20	2.790	65.21	0.015001	0.2301	227.20	0.9873
4	11.8665	50.00	1.860	60.01	0.010001	0.1667	166.40	0.9980
5	5.9240	50.01	0.9288	55.01	0.004995	0.09081	94.00	1.035
6	4.4635	50.02	0.6998	53.78	0.003764	0.06998	74.60	1.066
7	2.9789	50.03	0.4670	52.54	0.002512	0.04780	53.00	1.108
8	1.4888	50.00	0.2334	51.26	0.001255	0.02449	30.80	1.258
9	1.1961	50.01	0.1875	51.02	0.001009	0.01977	25.80	1.305
10	0.8932	50.00	0.1400	50.75	0.0007529	0.01484	20.60	1.388

Iodoacetic acid dilution data

GMW = 185.95 g/mole
 Stock solution: 18.5949g of acid in 100.01 g water
 Molality of Stock solution = 0.9999 m
 Wt factor = 0.1568

Graphs of K_a vs. molality for the monosubstituted haloacetic acids, cyanoacetic acid, and acetic acid are shown in Figures 3 through 7. They were generated using Solver, a component of the Microsoft Excel spreadsheet. The data smoothing operations—the fitting of a non-linear equation—was utilized by the application of known equations that the data could fit. A multi-polynomial equation used as a correction for ϕ was derived.

This equation is

$$\phi_{\text{corrected}} = 0.997 + (-0.245 + 0.1086\text{p}K_a - 0.128\text{p}K_a^2) (\sqrt{m}) + (0.07 + 0.057\text{p}K_a - 0.013\text{p}K_a^2) (m)$$

It is generally represented as

$$\phi_{\text{corrected}} = a + b\sqrt{m} + c(m)$$

where m is the concentration in molality and a , b , and c are constants. Constants b and c are functions of $\text{p}K_a$.

The new value of ϕ (called ϕ_{new}) is

$$\phi_{\text{new}} = \phi_{\text{correct}} - \phi_{\text{experimental}}$$

Knowing that

$$K_a = \frac{(\alpha^* m)_{H^+} \cdot (\alpha^* m)_{B^-}}{\alpha(1-m)_{HB}} = \frac{\alpha^2 m}{1-\alpha}$$

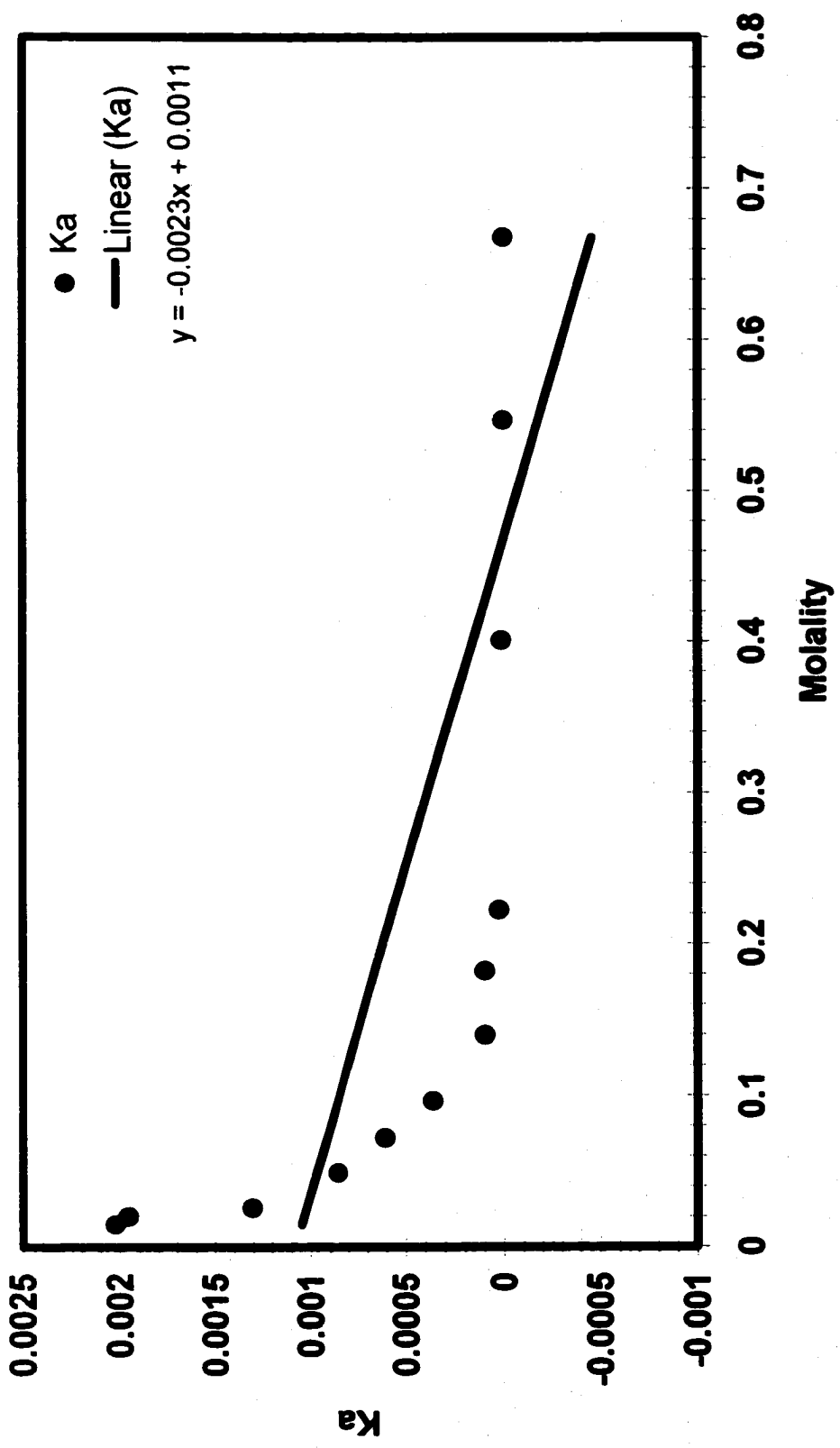


Figure 3. Bromoacetic acid: Value of Ka extrapolated to zero concentration

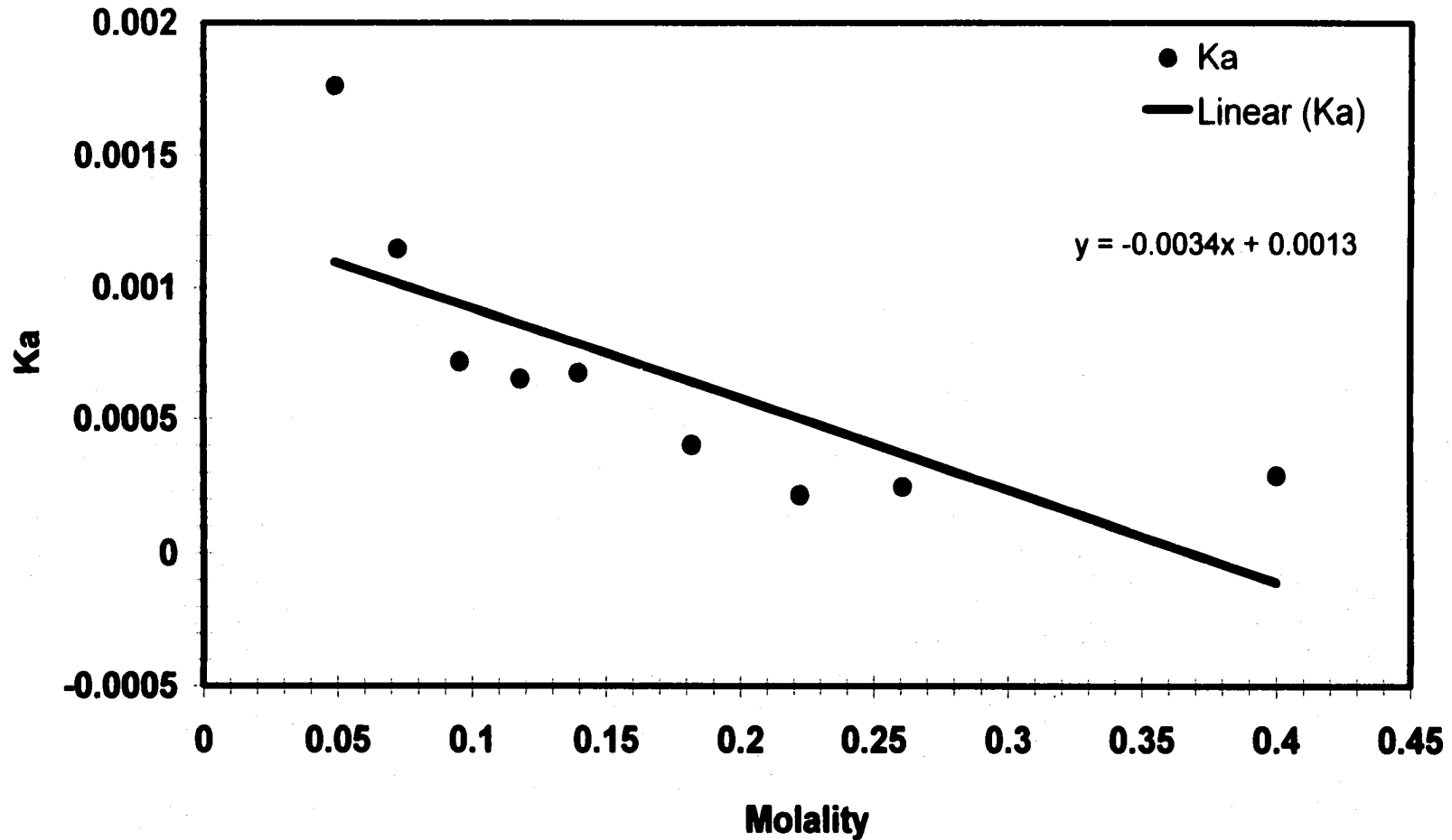


Figure 4. Chloroacetic acid: Value of Ka extrapolated to zero concentration

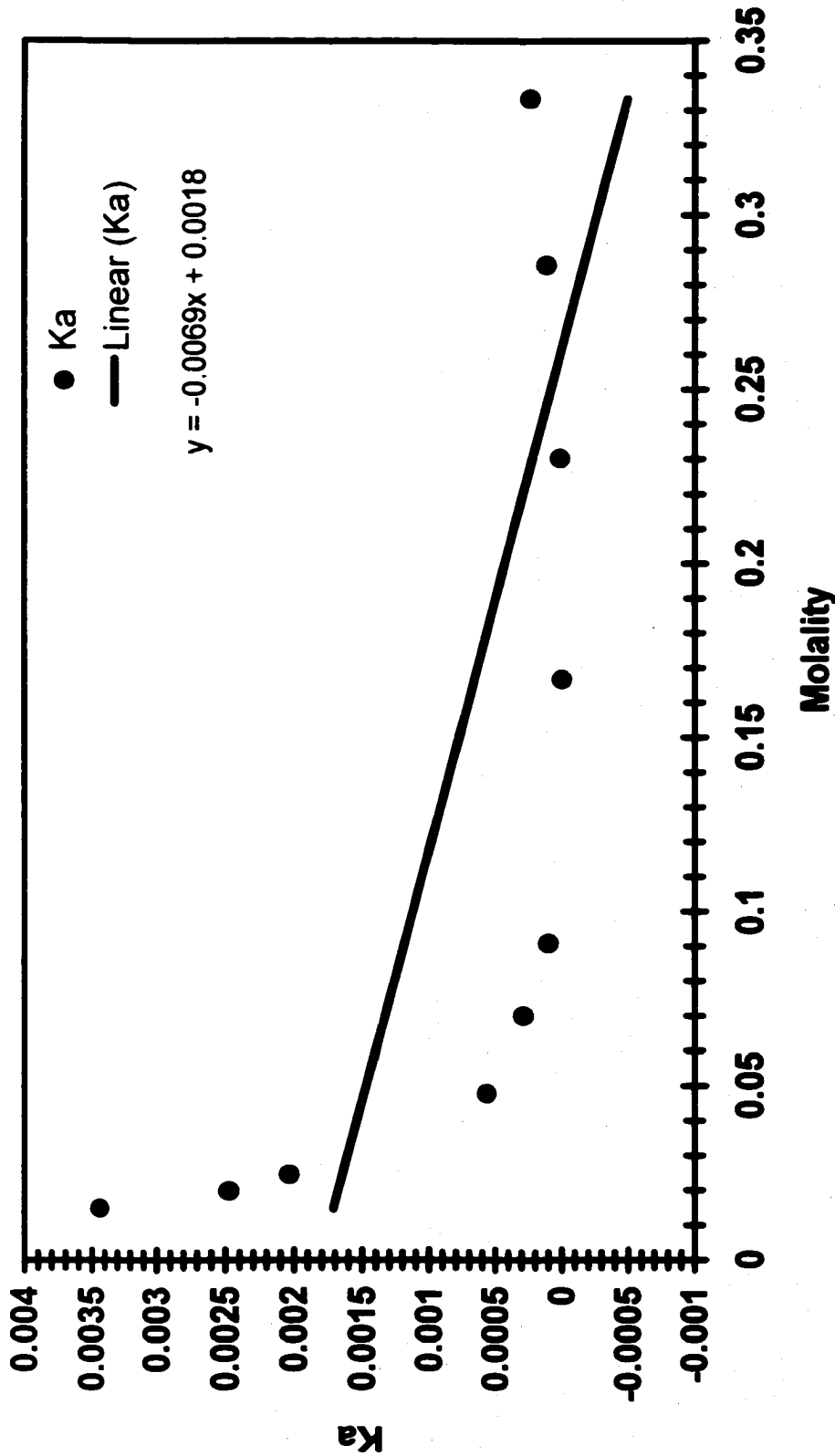


Figure 5. Iodoacetic acid: Value of Ka extrapolated to zero concentration

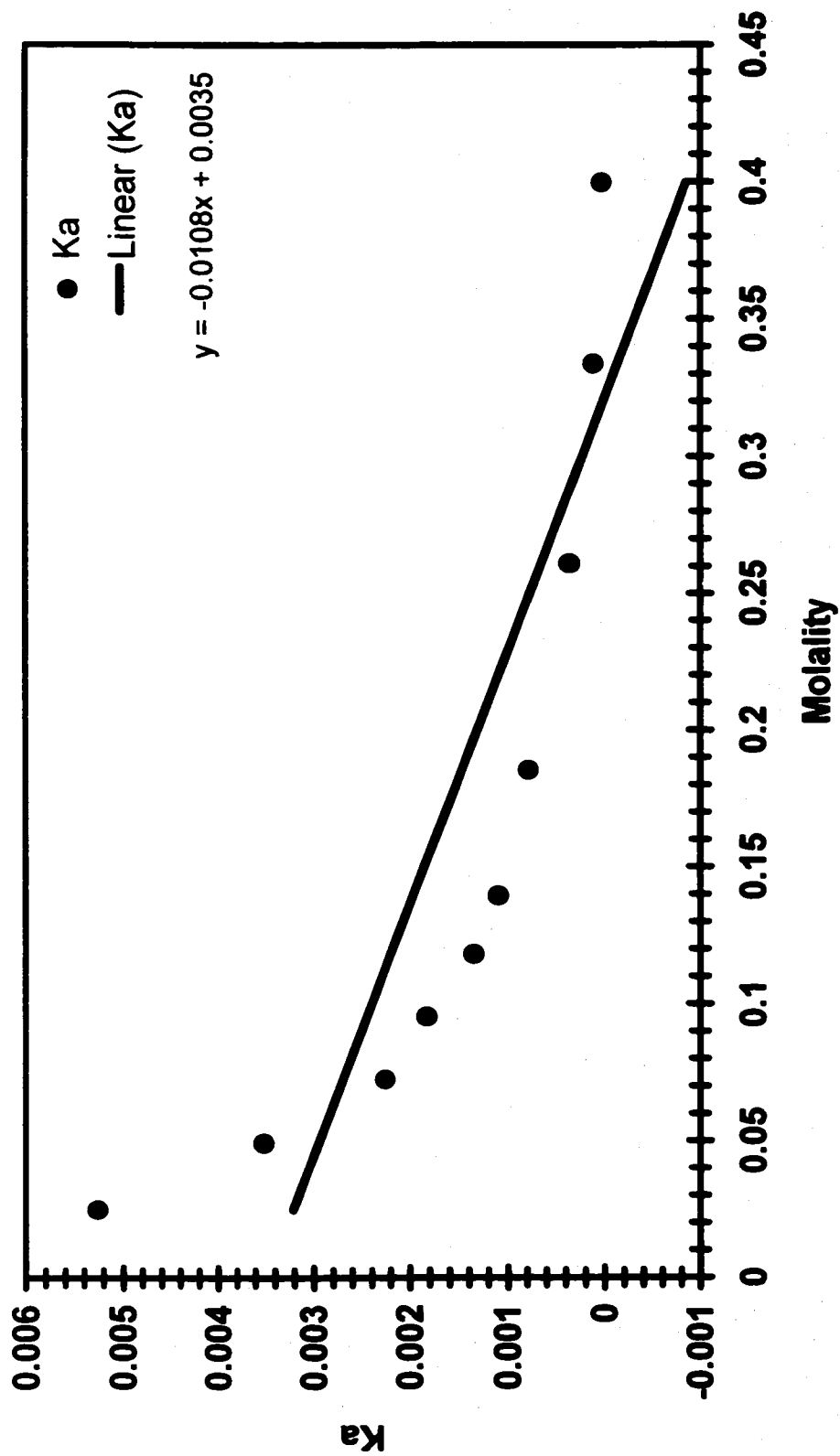


Figure 6. Cyanoacetic acid: Value of K_a extrapolated to zero concentration

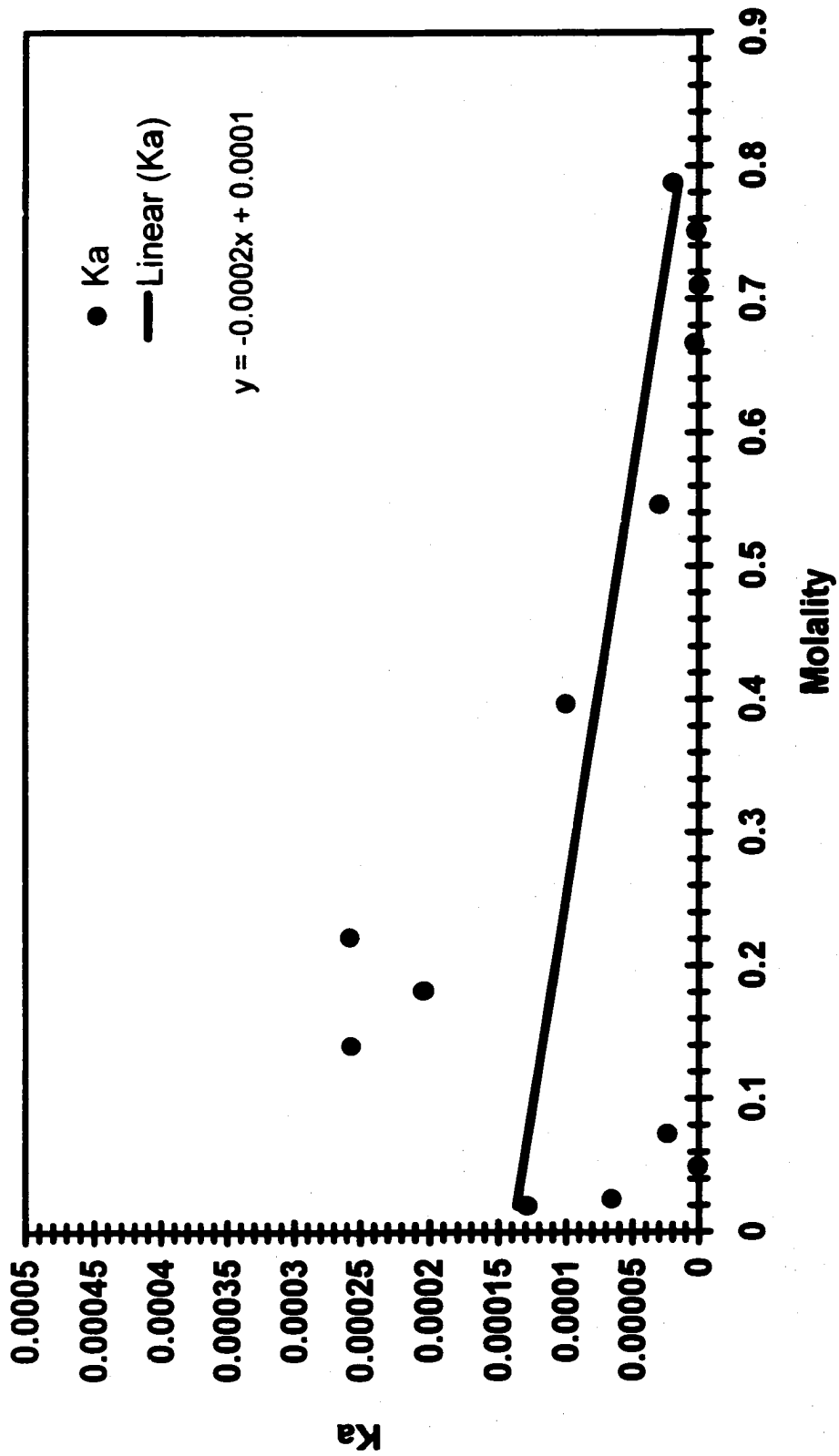


Figure 7. Acetic acid: Value of K_a extrapolated to zero concentration

α is found by taking the positive root of the following quadratic equation :

$$m\alpha^2 + K_a\alpha - K_a = 0$$

the positive root is

$$\alpha = \frac{\sqrt{K_a^2 + 4K_a m}}{2m} - \frac{K_a}{2m}$$

Since $\alpha + 1 = \phi_{\text{new}}$.

then

$$\phi_{\text{new}} = \frac{\sqrt{K_a^2 + 4K_a m}}{2m} - \frac{K_a}{2m} + 1$$

The last equation solved for ϕ_{new} is used iteratively to estimate the value of the pK_a used to compute the constants b and c for the ϕ_{correct} equation. The refined values of ϕ_{new} are then used to calculate α and K_a at each concentration value. Lastly, these values of K_a are plotted against concentration and subsequently extrapolated to zero concentration to determine K_a . These operations were made using a spreadsheet named *Wizard of Os.xls* [12].

Using the technique above, the experimental values of the ionization constants for weak acids as determined by osmometry are shown in Table 7.

Table 7. K_a values of weak acids as determined by osmometry

Acids	K_a determined by osmometry	Literature Values [4]
Acetic acid	1.4×10^{-4}	1.75×10^{-5}
Bromoacetic acid	1.1×10^{-3}	1.25×10^{-3}
Chloroacetic acid	1.3×10^{-3}	1.36×10^{-3}
Cyanoacetic acid	3.5×10^{-3}	3.37×10^{-3}
Iodoacetic acid	1.8×10^{-3}	6.68×10^{-4}

K_a values for iodoacetic acid and acetic acid are in poor agreement with the literature values for the reasons explained in the next chapter.

Chapter 4

Discussion and Conclusions

"A first rate laboratory is one in which mediocre scientists can produce outstanding work".

-Patrick Maynard Stuart Blackett (1897-1974)

Overview

Although the conductivity method was applied to confirm the K_a value of acetic acid, the main focus of this dissertation was the development of an alternative method for determining the acid ionization constants. Even though ionization constants, K_a , of some of the weak acids studied were in an approximate agreement with the literature values, others deviated lamentably from the reported literature values. Such is the case of acetic and iodoacetic acids. The reasons for this deviation may be the nature and the strength of these acids. Ideally, the plot of osmolality vs. molality of a soluble, non-electrolyte solute should give a straight diagonal line. For weak acids, however, this line would fall slightly above the ideal line, with greater deviation at the lower concentration (see Figure 8). For the benefit of interpretation, a new term ϕ must be defined. It is the quotient of osmolality (instrument readings in osmole per kilogram of solvent) divided by the molality of the solution. For an ideal solution of a non-electrolyte solute in which molality equals osmolality, the plot of ϕ versus molality would give a horizontal line at ϕ equals 1.

Examination of a graph of a weak acid such as bromoacetic acid (Figure 9) shows that the line is not horizontal. This is to be expected because the data are real, not ideal.

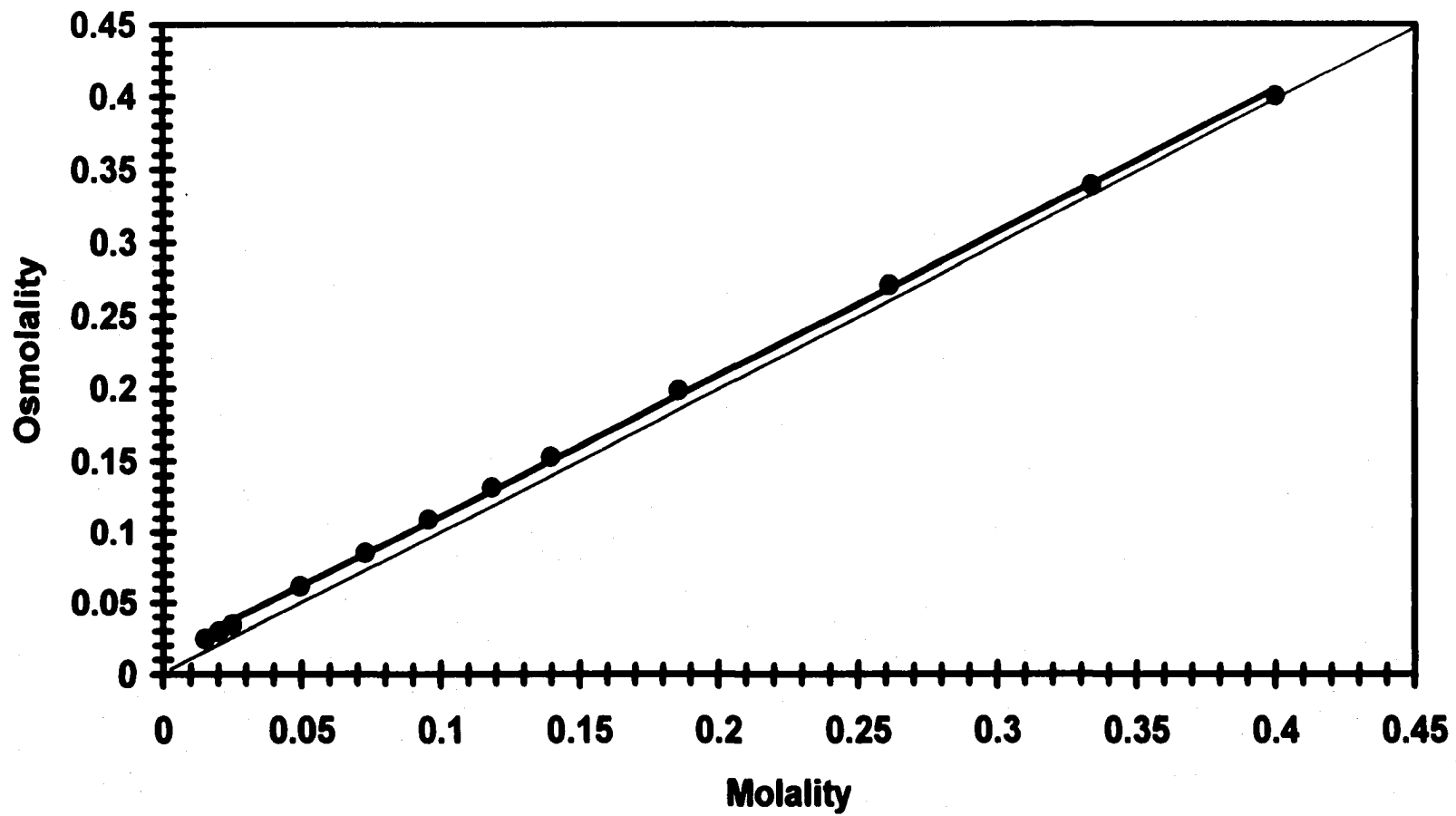


Figure 8. Cyanoacetic acid: Osmette readings vs. molality; data line slightly above theoretical diagonal line.

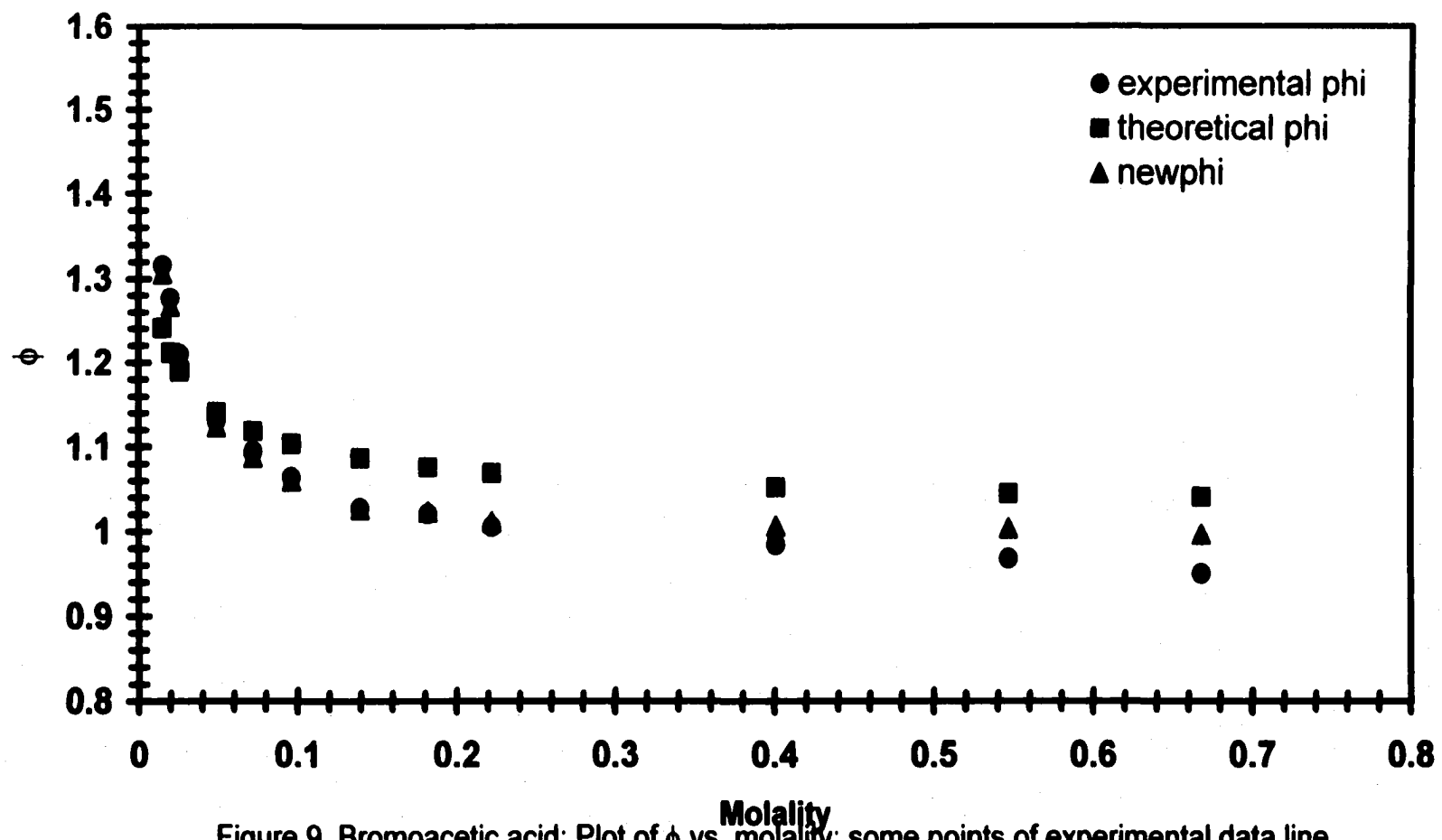
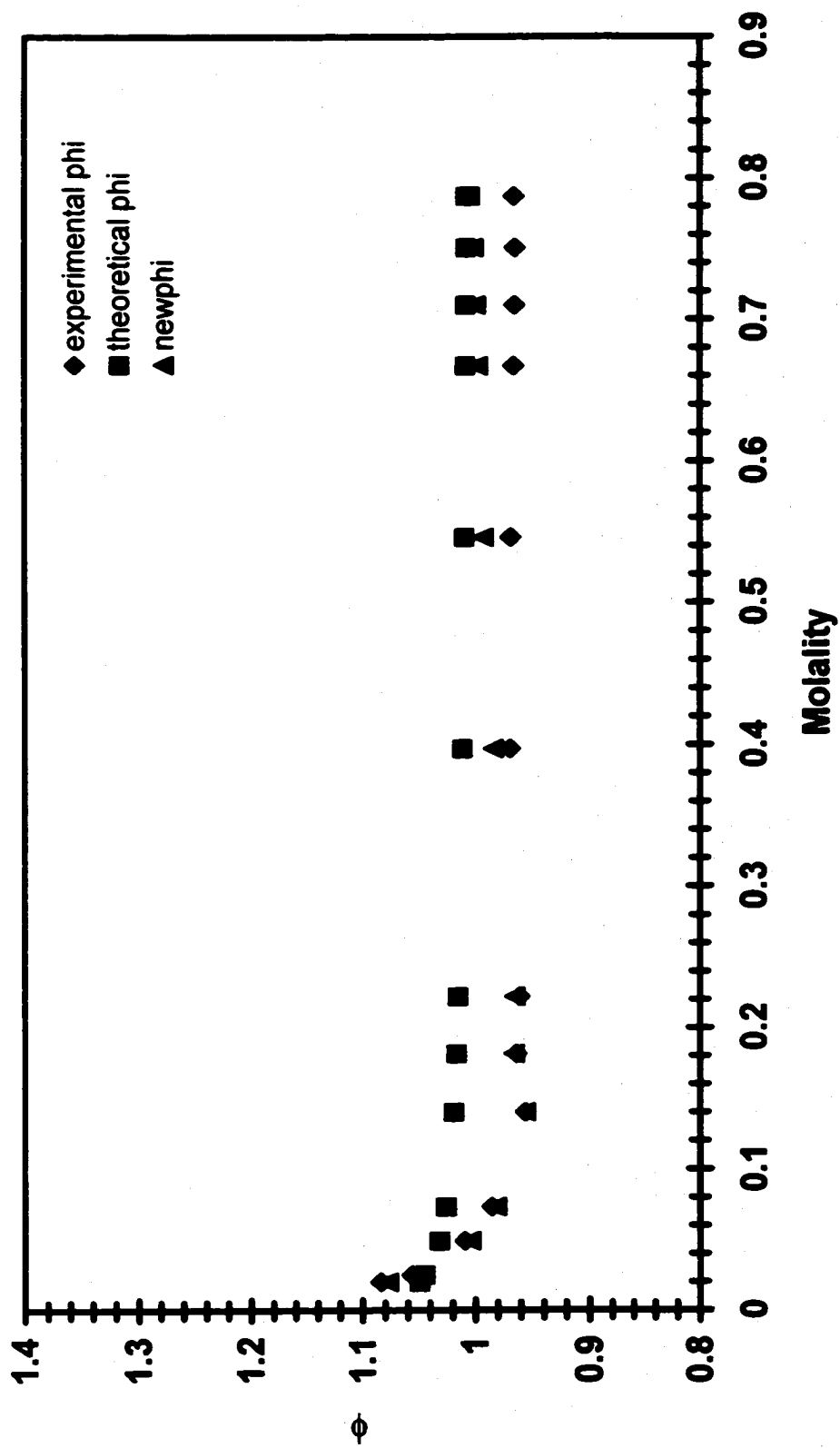


Figure 9. Bromoacetic acid: Plot of ϕ vs. molality; some points of experimental data line fall below $\phi = 1$

The problem is that this line goes below the line where $\phi = 1$. Ideally, this cannot happen. The plot should show the fraction of the acid that is ionized (α) as a region above the line of $\phi = 1$. Given that $\phi - 1 = \alpha$, calculations give positive α values up to about a concentration of $m = 0.2$. Above this concentration, they give negative α values. If plugged in the K_a expression, the negative α values squared become positive, implying more ionization as the concentration increases, which cannot be the case. Few acids studied had this problem of negative α values. Two reasons account for this behavior: (1) the osmotic coefficient of these weak acid solutions is less than 1, and (2) the activity coefficient is also less than 1.

Since the ionic strength is low, something to be expected in weak acid solutions, the activity coefficient becomes less important, making the osmotic coefficient more and more predominant. Furthermore, the ionic strength factor works in the opposite direction from the osmotic coefficient; that is, the osmotic coefficient tends to pull the readings down to a value lower than ideal while the ionic strength factor pulls them up. Weaker acids such as the acetic acid and iodoacetic acid used in this study display this behavior (Figures 10 and 11); the osmotic coefficient exerts a very strong pull downward sending the data line too low from the theoretical line for the correction factor to bring it up. Moderately strong acids in this class exhibit mid-range characteristics (Figures 12 and 13). Their data line is closer to the theoretical line because there are not many points below $\phi = 1$. In stronger acids however, the ionic strength takes precedence over the osmotic coefficient. As a result, the data points are far above the theoretical line. The strength of acid (its pK_a value) determines the direction and the magnitude of the pull

Figure 10. Acetic acid: Plot of ϕ vs. molality

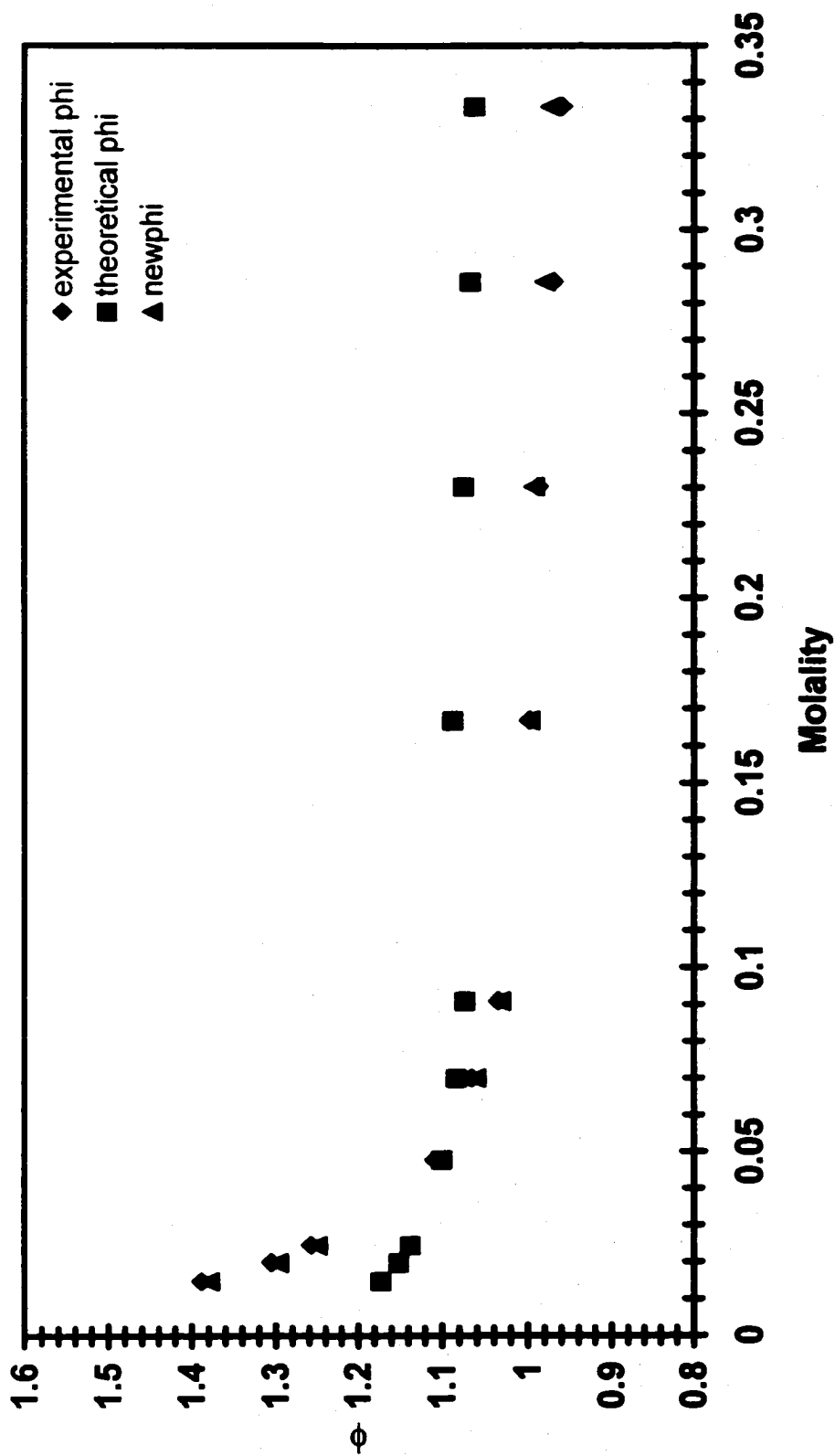
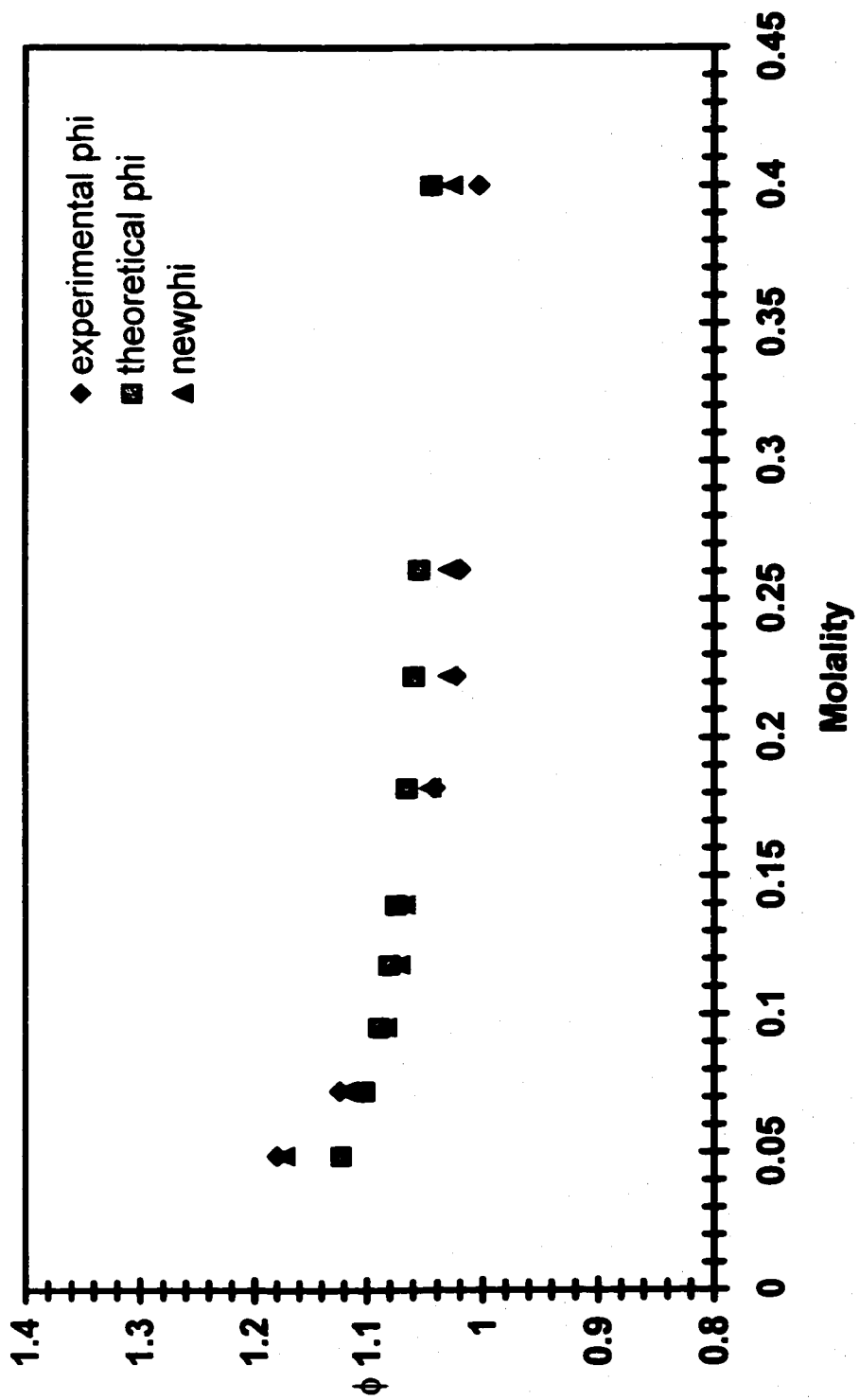


Figure 11. Iodoacetic acid: Plot of ϕ vs. molality

Figure 12. Chloroacetic acid: Plot of ϕ vs. molality

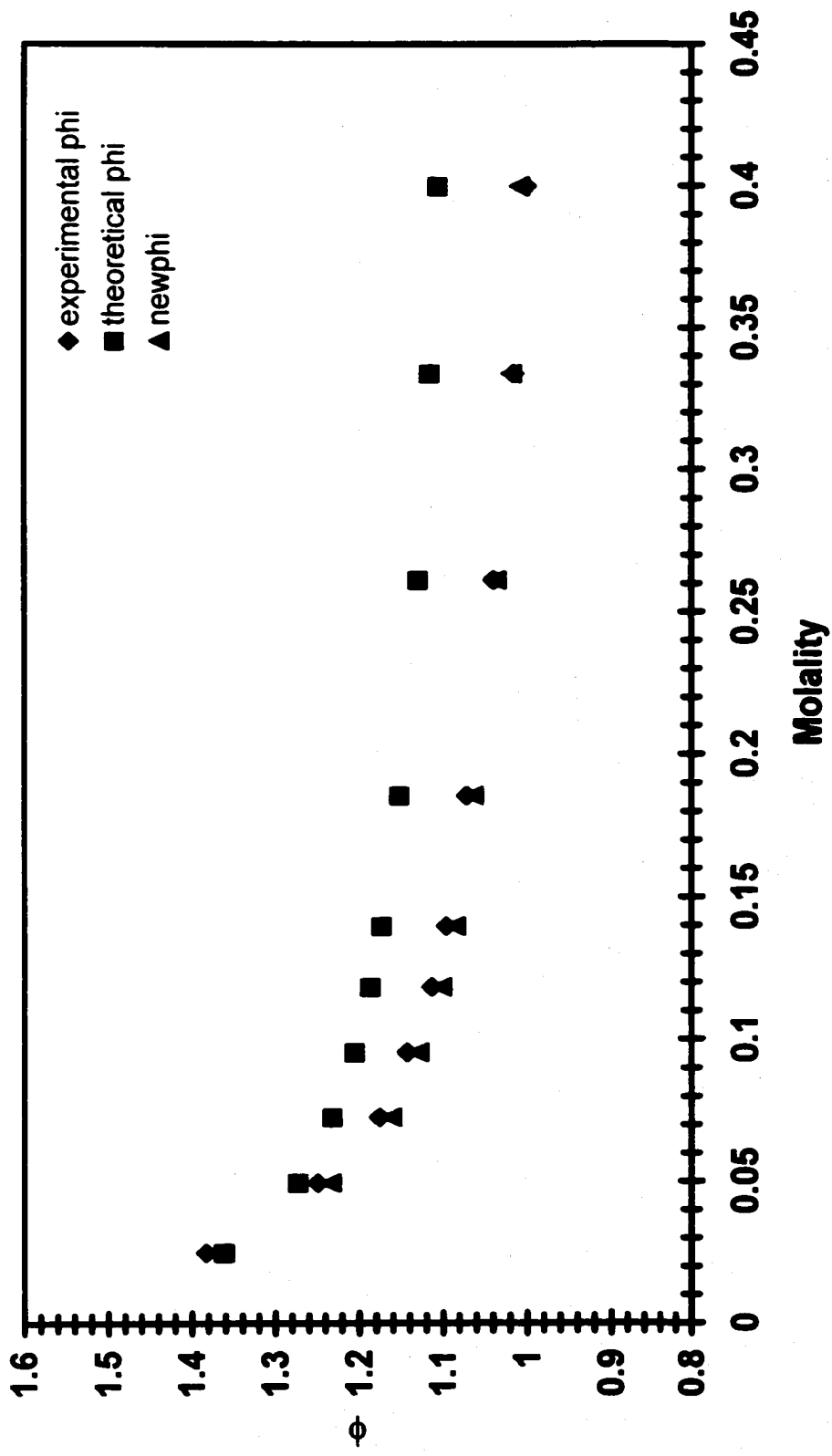


Figure 13. Cyanoacetic acid: Plot of ϕ vs. molality

phenomenon. The stronger the acid, the more predominant is the ionic strength factor, making the data line fall above the $\phi = 1$ line. It was to compensate for these deviations that an empirical, multi-polynomial equation to correct ϕ was developed and used as explained in chapter 3 of this work.

Conclusions

The equilibrium constants for five weak acids have been measured using osmometric methodology. It has been shown that for such weak acid measurements, two factors, the ionic strength and the osmotic coefficient, exert opposite effects in solutions. This study also demonstrated the important role that osmometry can play in chemistry by introducing a rapid method in the determination of equilibrium constants. The measurements and interpretation of K_a is an interesting and important topic in analytical and physical chemistry. Although the conductance method provides nearly accurate values of these thermodynamic ionization constants of weak acids at very low concentrations, it is however time consuming and labor intensive. It requires freshly prepared solutions, tedious mathematical manipulations, and suffers from temperature sensitivity. Though still in its infancy as an analytical tool, the computer-assisted osmometric method is rapid and shows promise for use in the determination of equilibrium constants and other physical properties of compounds.

Limitation of this study and future considerations

This project was designed in part as an education tool to be used in quantitative analysis and higher physical chemistry laboratory courses. The weak acids used for this study were chosen for their availability, safety in handling with relatively inexperienced students in mind, low volatility, and solubility in water. Care was taken not to have acids whose ions might undergo hydrolysis, fueling more complications in the measurements and in computations.

The method could be extended to the determination of solubility product constants of some slightly soluble salts and to the determination of molecular weights of water-soluble polymers. With the improvement in spreadsheet technology, better curve-fitting equations can be derived to assist students with data analysis.

PART 2

Instrumental Self-Evaluation and Feedback Approach to Student

Preparation of Solutions

Chapter 1

Introduction

*“You cannot teach a man anything; you can only help him find it within himself.”
Galileo Galilei (1564-1642)*

Background

Throughout the history of higher education, science teaching has stressed laboratory instruction. Attempts have been made to bring about improvements not only in lecture halls but also in the laboratory. Recently, there has been an explosion of education research with important implications for the teaching and learning processes. Certain methodologies used successfully by some instructors in the past are now being validated by scientific studies of how people best learn [13]. There is recognition for the need to empower students to be responsible and to be at the center stage in the learning process (one of the principal tenets of constructivism theory), encouraging in them the internal locus of control that is essential to self-motivation. There is also a shift from emphasis on content to greater attention to process [14]. Attention is now focussed on teaching methods that prepare students for self-directed, life-long learning [15].

One of the objectives of teaching improvement is for students to acquire cognitive and investigative skills. The former applies to the essence of becoming an educated scientist and the latter can be developed through laboratory experimentation. In quantitative volumetric analysis, preparing solutions well is critical to the success of an experiment; incorrectly prepared solutions will produce erroneous results. Laboratory exercises test the ability to learn and to retain as well as to apply the new knowledge. In

order to facilitate students' learning, to monitor their attitude toward the course, and to reinforce students' investigative skills, a self-evaluation and an instrument feedback method, as well as a constructivist environment are utilized in this research.

Purpose and Significance of this Study

The prevailing approach to teaching chemistry in the laboratory involves having students perform teacher-structured laboratory experiments. Each step of a procedure is carefully described and students are expected to follow these steps exactly. With this approach, little room is left to the students' ingenuity. Memorization may enhance a student's lecture grades, but it cannot replace hands-on practice. Understanding the manner of development of scientific constructs is as important as memorizing the facts of science. Despite the advances and advantages of modern teaching tools and methods, such as computer-assisted instruments and constructivism theory, many instructors are reluctant to use these techniques. Moreover, most are concerned that it will take too much extra time from their busy schedules. As a theory of learning, constructivism assumes that knowledge is constructed in the mind of the learner with the help and the guidance of the teacher [16]. For a meaningful learning to take place according to this theory, students actively construct new information on what exists already in his or her memory. It must be understood however, that activities designed to expand the critical thinking potential of students also challenge teachers to adapt to new ways of instruction. It also requires students to be active participants in the learning process rather than passive recipients. This process, however, offers promise of moving students beyond rote acquisition to the analysis, synthesis, and evaluation of new information.

The research design of the study is both quantitative and qualitative. The quantitative purpose of this study was to develop an experiment that helps students improve their skills in making solutions by using a non-threatening instrument feedback approach based on the constructivist model within which students prepared solutions of the required concentration and then prepared serial dilutions from them. Serial dilution in the preparation of solutions is used in many areas of science including chemical, environmental, and clinical laboratories. By definition, to dilute means to add more solvent without the addition of more solute. It is a process of carefully removing a known amount of stock standard solution to another container, which is brought to the desired volume with water to make a dilute solution. The resulting solution must be thoroughly mixed by inversion of the volumetric flask several times to ensure that the dissolving substance is distributed homogeneously in the solution. The standard dilution equation is

$$M_1 V_1 = M_2 V_2$$

where M_1 and M_2 , are concentrations in moles per liter of the aliquot taken before and after dilution respectively, and V_1 and V_2 are volumes of concentrated aliquot and dilute solutions respectively. The product of molarity times volume ($M_1 V_1$) refers to moles of solute in the aliquot before dilution, which is equal to $M_2 V_2$, which refers to moles of the same solute after dilution. This equation teaches students the fact that the amount of solute remains unchanged.

After preparing solutions of known concentrations and serially diluting them, students use a spectrophotometer and an osmometer to find the absorbance of colored solutions and the osmolality of colorless solutions, respectively. The instruments allow students a self-evaluation of their ability to prepare dilutions by comparing their results

with those from the standard solutions prepared and tested by an experienced instructor. Students are normally briefed and instructed as to why care must be taken in proper handling and cleaning of the equipment in order to reduce contamination. They may or may not follow these instructions. The data recorded by the students from these instruments help them to immediately assess their triumph in solution making or their disappointment and decision to try one more time. It was hoped that the incorporation of the self-evaluation method using instrument feedback together with a constructivist approach in which students assess their own performance would be a more effective teaching tool for solution mixing than traditional methods such as detailed instructions that students follow like a recipe.

In addition to quantitative evaluation of the experiment, qualitative tools such as informal interviews and observation of students were utilized in order to assess students' previous skills in making careful measurements of any type and to monitor their attitudes towards the dilution solution experiment.

A secondary purpose of this experiment but not less important was to reinforce in the students the concept of colligative properties of solutions that they have encountered in freshman chemistry courses and to introduce to them the phenomenon of the interaction of light energy with matter. In the former, the freezing point lowering of a solvent is directly proportional to the concentration of the dissolved particles in the solvent for strong electrolytes according to the equation:

$$\Delta T_f = K_f \cdot m \cdot i$$

where ΔT_f is the change in freezing point, K_f is the molal freezing point depression constant, m is molality, and i is the Van't Hoff factor which equals the stoichiometric

number of charged particles per formula unit of solute. The computer-controlled osmometer automatically computes all the parameters involved in the above expression and displays the concentration in milliosmoles per kilogram of solvent water (mOsm/kg).

The use of spectrophotometry introduces students to the concept of the interaction of matter with electromagnetic radiation. When an amount of light of intensity, I_o , passes through a colored substance, some of the radiation is absorbed. This absorption diminishes the intensity of light from I_o to I . At a chosen wavelength, the reduction in intensity is directly proportional to the concentration of dissolved substance in solution and to the path length through the solution. The relationship between these variables, universally referred to as Beer's law, is given in the equation:

$$A = \log \frac{I_o}{I} = \epsilon bc$$

where the quantity A is called absorbance, the proportionality constant ϵ is the molar absorptivity, the path length b is measured in centimeters, and the concentration c is in moles per liter.

Research Questions

This study explored ways to teach a quantitative chemical analysis laboratory in an environment that is conducive to developing students' solution making skills. To determine if this experiment promotes student learning, the researcher investigated the following questions: (1) Does self-evaluation using instrument feedback and a constructivist-style environment help students perform at a more skilled level in mixing solutions?; (2) What are students' perspectives on the effectiveness of using the instruments in developing the skills of mixing solutions?; (3) Do holding a paying job and

the number of hours that a student works affect the grade obtained in quantitative analysis laboratory?; (4) Could the grade on the solution dilution experiment be predictive of overall grades in quantitative analysis laboratory?

Chapter 2

Literature Review

"The most extraordinary thing about a really good teacher is that he or she transcends accepted educational methods."

-Margaret Mead (1901-1978)

Background

The search for innovative teaching methods has been in the forefront of education for many years. The essence of teaching science in general and chemistry in particular is to prepare students to acquire cognitive thinking and critical reasoning skills. These investigative skills can be developed through laboratory experimentation. It is the purpose of this research to find a suitable method to enhance students' ability to prepare solutions. The constructivist-style environment and the self-evaluation methodology will be implemented to reach this purpose.

There are many teaching strategies that may be classified as constructivist; however, the approaches have a number of common aspects. Constructivism is not a new concept. It has been recognized and is well established as a modern theory of learning and has gained acceptance at the elementary and high school levels as the best tool for teaching of science. Constructivism rests on the premise that knowledge is constructed in the mind of the learner with the help and guidance of the teacher. Formal definitions of constructivism as a learning approach can be found elsewhere in the literature [17-22]. However, as philosophical theory, constructivism is generally accredited to Jean-Piaget [23, 24]. It made its way into the educational context in the late 1970s [25-27] and into science classrooms as an educational tool in the early 1990s [21, 28, 29]. Assessing

constructivist models as an effective learning approach in science, Leonard [30] points out that “students best learn sciences in a laboratory setting where they are given the opportunity to collect, process and internalize chemical concepts through interactive discussion.” Domin [31] finds the traditional “cook-book” approaches ineffective because “students are not afforded the time necessary for the deep processing of information.” He concludes that “it is through deep processing that students are able to integrate new experiences with prior knowledge, establish a context for the purpose of the laboratory activity, and determine its relevance to themselves all of which are characteristics of meaningful learning.”

There are as many facets to a constructivist-style approach in science teaching as there are users. Each of these facets is treated as a different part of the same whole dependent on the description of the method used. The most cited methods that have been incorporated into laboratories from freshman level to organic and biochemistry levels are active, cooperative, and collaborative learning methods [32-38]. Cooperative and collaborative methods are techniques by which students assist each other in the learning process acting as partners with the teacher and with each other. Other techniques frequently used are the learning-cycle approach [39, 40]; the problem-solving approach [41]; and the guided inquiry, a methodology that engages students in finding solutions to important and meaningful questions through investigations and collaboration with others [42, 43].

In the area of quantitative volumetric analysis, research is being conducted to address the problem of students' lack of the ability to properly mix solutions.

Quigley [44] expresses concern about students' techniques by pointing out that the majority of institutions "make the lives of their students easier by having most, or even all of the stock standard solutions prepared in advance of a class." Risley [45] comments that "when upper division undergraduate students and graduate students first begin research, few have acquired the skill of preparing solutions...." There are a few additional published reports in the literature that address the concern of solution preparation [46]. Recently, Wang [47] reported the effectiveness on students' solution preparation skills of utilizing visual aids such as the classic chemical oscillator and iodine clock reactions as feedback tools to challenge students to prepare solutions that "work." The only published report that was at all similar to this research was the work of Janusa, et al. [48]. They designed an experiment that reportedly improved students' solutions preparation in a general chemistry course. The authors claimed that the experiment fills a much needed "gap between cook-book experiments,...and discovery laboratory" and that it is "a way to enhance students' thinking and problem-solving skills". In this experiment, students prepared two solutions of each acid and base by direct weighing of solutes or by dilution of a stock solution, then measured the hydrogen ion concentration (pH). Students then compared their measured pH to the pH values they had calculated from theory. Though it was not the focus of the experiment, students were instructed to reprepare their solutions if the two solutions differed by more than 0.1 pH units. The authors, however, did not analyze students' results to establish whether instrument feedback helped students develop laboratory skills.

Because of the cited concerns about students' lack of ability to prepare solutions and the reported benefits of the constructivist model of learning that include students'

improved understanding of chemical concepts, laboratory skill enhancement, and the ability to think critically, this researcher sought to implement the self-evaluation method using instrument feedback coupled with the constructivist model approach to serial dilutions of solutions.

Chapter 3

Methodology

“One must learn by doing the thing; for though you think you know it, you have no certainty until you try.”

-Sophocles (c. 496-406 B.C.)

Background

The research objective was to improve laboratory instruction by providing students in quantitative chemical analysis classes a more effective environment for learning solution preparation techniques. One two-part laboratory exercise was developed. It was designed to contain instrumental analysis with immediate feedback to students for self-evaluation of their results in preparing solutions. To comply with federal regulations for the use of humans as objects of research, permission was obtained from the Middle Tennessee State University Institutional Review Board. The letter granting permission can be found in Appendix F.

The experiment was scheduled for the fifth and eighth weeks of the semester after students had been introduced to some specific techniques, theoretical principles, and sample handling procedures during lectures and in other laboratory exercises.

Research Design

The experiment was designed to be simple, routine, and reliable, using chemicals and equipment familiar to the students. The goals were to assign a practical task (preparing solutions accurately) in an environment that facilitates and enhances learning, to allow student self-evaluation of their results, and to evaluate students' attitudes toward

the use of instruments as a feedback tool. Two compounds, nickel nitrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ and potassium chloride (KCl) were chosen for this study. Aqueous solutions of nickel nitrate have a green color and are suitable for spectrophotometer measurements. Solutions of potassium chloride are colorless; students determined the concentrations of KCl solutions by osmometry. Students diluted 1.000 M stock solutions of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ or made some stock solutions of KCl and serially diluted these stock solutions to solutions of lesser concentrations. They evaluated their skills in solution making based on the data and the feedback they received from instrument readings. The laboratory was staffed with advanced student assistants in order to monitor and proctor the progress of the experiment and to answer any questions that students might have about instrument usage. Precautions were taken not to direct experimental procedures, but to encourage students to proceed with their investigation on their own. At the end of the experiment, students were asked to evaluate the relative effectiveness of the instructional approach using a written questionnaire.

Research Sample

This study involved 167 students enrolled in the Quantitative Chemical Analysis (CHEM 222) course and was conducted over the period of fall semester, 1998 to spring semester, 2001. The quantitative analysis course is required for the bachelor's degrees in chemistry and in environmental science and technology. It is also required for several pre-health science professional programs (pre-medicine, pre-pharmacy, pre-veterinary medicine, etc). According to the Middle Tennessee State University (MTSU) undergraduate catalog, CHEM 222 is a five-credit hour course (three hours lecture and

two three-hour laboratories) covering gravimetric and volumetric determinations, introduction to optical and electrical methods of analysis, and to the stoichiometry of analytical chemistry. CHEM 222 is open to students who have completed requirements for general chemistry (CHEM 122 or CHEM 122H) and its laboratory components. The Department of Chemistry offers both undergraduate and graduate (Master of Science and Doctor of Arts) programs, and its professional chemistry undergraduate program is certified by the American Chemical Society (ACS). It has twenty-five full time faculty members holding doctoral degrees from well-recognized universities in the United States and Canada and one adjunct faculty member. In addition to the ACS certified courses, the department offers chemistry courses for students enrolled or planning to pursue careers in health and earth sciences. The department is an integral part of the College of Basic and Applied Sciences which, along with five other undergraduate colleges and a College of Graduate Studies, make up MTSU.

MTSU provides quality educational programs in a supportive campus environment conducive to learning and personal development. Founded in 1911, MTSU is an emerging major institution of higher education in the state, region, and nation. It is a member of the State University and Community College System of Tennessee. It is located in Murfreesboro near Nashville. The campus is located less than a mile from the exact geographic center of the state. The University is a fast growing institution of nearly 19,000 students and a faculty of 745 (Spring 2000 profile).

Instrumentation

The absorbance and the percent transmittance of serially diluted solutions of nickel nitrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ were measured on the Spectronic 20D Spectrophotometer (Spec

20D, Milton Roy, Analytical Products Division, 820 Linden Ave., Rochester, N.Y. 14625) set at the maximum wavelength (λ_{max}) for $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ of 395 nanometers. The Spec 20D is a conventional, digital spectrophotometer (analog models also exist) that consists of a tungsten source lamp, a dispersive element, an exit slit, and a solid-state silicon detector. It simultaneously displays wavelength and either percent transmittance or absorbance. Its useful range is from 340 to 950 nm with a spectral bandwidth of 20 nm. Its ease of use makes it the most widely applied instrument in undergraduate chemistry laboratories.

Concentrations, in units of milliosmoles of solute per kilogram of water (solvent), of both nickel nitrate and potassium chloride solutions were measured on a computer controlled osmometer, Osmette XL (Autocal Osmometer, model 5007, Precision System, Inc., 16 Tech Circle, Natick, MA 01760-1029) described in Part 1 of this work. It was calibrated with osmometry standard solutions of NaCl (100 mOsm/kg H_2O , Lot No. 901081 and 500 mOsm/kg H_2O , Lot No. 812262, Precision Systems, Inc).

The researcher designed two questionnaires to assess student attitudes about the experiment. The first questionnaire (see Appendix C) contained questions related to demographics and scholastic background. The second, an exit questionnaire (see Appendix D), contained questions related to students' attitude with respect to the use of instruments, to the perceived change in their learning experience, and to the overall evaluation of the experiment. It also contained two open-ended questions to provide students the opportunity to comment on what they liked or disliked about the experiment. This questionnaire was administered in the week following the second part of the serial dilution experiment. A graduate student who had no knowledge of the nature of the

investigation and had no teaching responsibility within this course administered it for the investigator. Students' responses to the questionnaire were voluntary and were treated as confidential.

Experimental Procedure

A written description of the experiment is included in Appendix A. It was given to students followed by a procedure briefing on safety and the use of the instruments. Since this was "their experiment," students were involved in planning, calculations, mixing of solutions, measurements, and analysis. The researcher and laboratory assistants served as observers and guides for use of instruments.

Solutions of Nickel Nitrate

For safety reasons, a one molar stock solution (1.000 M) of nickel nitrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ Fisher Scientific, Lot No. 984710) was provided for student use. Because dusts of solid nickel nitrate are suspected cancer causing agents, the solid form of the compound must be handled by experienced workers. From the 1.000 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution, students were told to prepare 50 mL of 0.100 M solution and label it as solution 1. From this solution 1, they were instructed to prepare 50.00 mL of 0.0500 M solution and label it as solution 2. Both solutions 1 and 2 were then turned in to the researcher or a lab assistant.

Students were instructed to again use the already prepared stock solution (1.000 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and to make 50.00 mL of 0.200 M solution and label it as solution 3. From this solution 3 they were asked prepare 25 mL of 0.0800 M solution and to label it

as solution 4. It is this serially diluted solution 4 that they used for instrument feedback. Using the Spec 20D, they compared the absorbance against the absorbance of a 0.0800 M standard solution. If the absorbances were in poor agreement, the student could refer back to his/her lecture notes or reflect back to the solution preparation and cuvette rinsing technique to look for poor techniques where the mistake was incorporated. The experiment could be repeated if the student was unhappy with the initial instrument results.

From measured absorbances, students could calculate the actual concentration of their solution using the equations:

$$\frac{A_{std}}{C_{std}} = \frac{A_{unk}}{C_{unk}}$$

$$C_{unk} = \frac{A_{unk} \cdot C_{std}}{A_{std}}$$

where A_{std} is the absorbance of the standard solution, A_{unk} is the absorbance of student's solution, and C_{unk} and C_{std} are the concentrations of students' solutions and standard solution, respectively.

After the nickel nitrate portion of the experiment, two weeks passed while students were conducting non-instrument, non-self-evaluation-oriented, regularly scheduled experiments. The layoff period allowed students to internalize the serial dilution

technique. The potassium chloride portion of the experiment was scheduled after this period.

Solutions of Potassium Chloride

The dilution procedure was somewhat like that of the nickel nitrate solution preparation. However, the students were expected to prepare 100 mL of 1.500 M KCl standard solution from dry, weighed, solid potassium chloride. This standard KCl solution was labeled solution 1. From solution 1, they prepare 25.00 mL of 0.600 M KCl solution and labeled it solution 2. They then serially diluted solution 2 to make 50.00 mL of 0.120 M KCl labeled solution 3. Solutions 2 and 3 were turned over to the researcher or to a lab assistant. From solution 1 (standard solution), students then prepared 50.00 mL of 0.150 M KCl solution and labeled it solution 4. Similarly, from solution 4, students made 25.00 mL of 0.0600 M KCl solution and labeled it solution 5. Once again, the students used the instrument to “grade” their solution. They used readings from the freezing point depression apparatus to test the ionic concentration of their solutions with a target concentration of 0.0600 M KCl. A 0.0600 M KCl standard solution was provided as a control solution. Students used the osmometer to measure the concentrations of both the control solution and their own preparation. Then, they had the option of remaking solutions 4 and 5 based on the comparative data feedback from the osmometer. Most students were able to complete each dilution process within one two-hour lab period.

Chapter 4

Data Analysis

*"It is not enough to have a good mind. The main thing is to use it well."
-René Descartes (1596-1650)*

Demographic Profile of the Sample

Table 8 shows a summary of data about students who completed the quantitative analysis course from fall semester, 1998 to spring semester, 2001. Of the 167 students who participated in this study, 5.4 % were freshmen, 15.7 % were sophomores, 42.5 % were juniors, 35.3 % were seniors, and 1.2 % were graduate level students. Even though CHEM 222 is a sophomore level course, for various reasons many students delay taking it until their junior or senior year. There were 11.4 % of participants majoring in biology, 31.1 % in chemistry, 22.8 % in environmental science, and 26.9 % in pre-professional fields such as medicine, pharmacy and other health sciences. Other majors (7.8%) included non-scientific disciplines such as English and music. The sample was 46.1 % female and 53.9 % male. The racial profile of participants was representative of the university population: 81.4 % Caucasians, 9.0 % African-Americans, 9.0 % Asians and "others", and 0.6 % Native American -Indians. On the demographic survey, 20.4 % of participants reported not holding a job on or off campus, 27.5 % reported working an average of 2 to 8 hours weekly, and 52.1 % reported spending more than 18 hours per week at on-campus or off-campus work.

Table 8
Profile of Students who Completed Quantitative Analysis Course
From Fall 1998 to Spring 2001
Number of Students: 167

Category	Number of Students	Percentage
Class Standing		
Freshman	9	5.4
Sophomore	26	15.7
Junior	71	42.5
Senior	59	35.3
Graduate	2	1.2
Majors		
Biology	19	11.4
Chemistry	52	31.1
Environment	38	22.8
Sciences (Pre-Professional)	45	26.9
Other	13	7.8
Gender		
Female	77	46.1
Male	90	53.9
Race		
Caucasian	136	81.4
African-American	15	9.0
Native American (Indian)	1	0.6
Other	15	9.0
Hours worked on and off campus		
2 to 18	46	27.5
More than 18	87	52.1
Not Working	34	20.4

Report of Students' Instrument Readings

Figure 14 shows the plot of the students' measured absorbance of a 0.0800 M nickel nitrate solution versus the student identifying number for the fall 2000 class. The absorbance of the standard 0.0800 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was 0.418 (38.6 %T). The graph includes the highest and the lowest student readings, and the standard solution measurements against which all readings are compared. Absorbance readings from solutions number 130 and 164 are from two standard solutions prepared by the researcher mentor and by the researcher, respectively. The graph shows that most students performed well at their first trial. Others made several attempts before they were satisfied with their results. For example, the graph shows that student number 131 had four attempts before he/she finally closed the gap. Because of the closeness of some instrument readings, this y-axis scale does not show all of the readings from multiple attempts of solution preparation. It is difficult to see that student number 144 had made two trials, or that student number 157 attempted three preparations. However, an expansion of the absorbance axis (Figure 14a) makes this obvious with absorbance readings of 0.378 and 0.384 for student number 144's two trials, and readings of 0.400, 0.414, and 0.416 for the three preparations of student number 157.

The same trend occurred with the osmometer measurements. Figure 15 is the plot of the actual concentrations of the 0.0600 M KCl solutions versus the student number for the fall 2000 semester students. Once again, readings number 130 and 164 are the instructors' two standard 0.0600 M KCl solutions, both with the osmotic concentration of 112 mOsm/kg. In this graph, the results of several attempts can be clearly seen. Student number 135 had at least four attempts. Because of the closeness of some measured data,

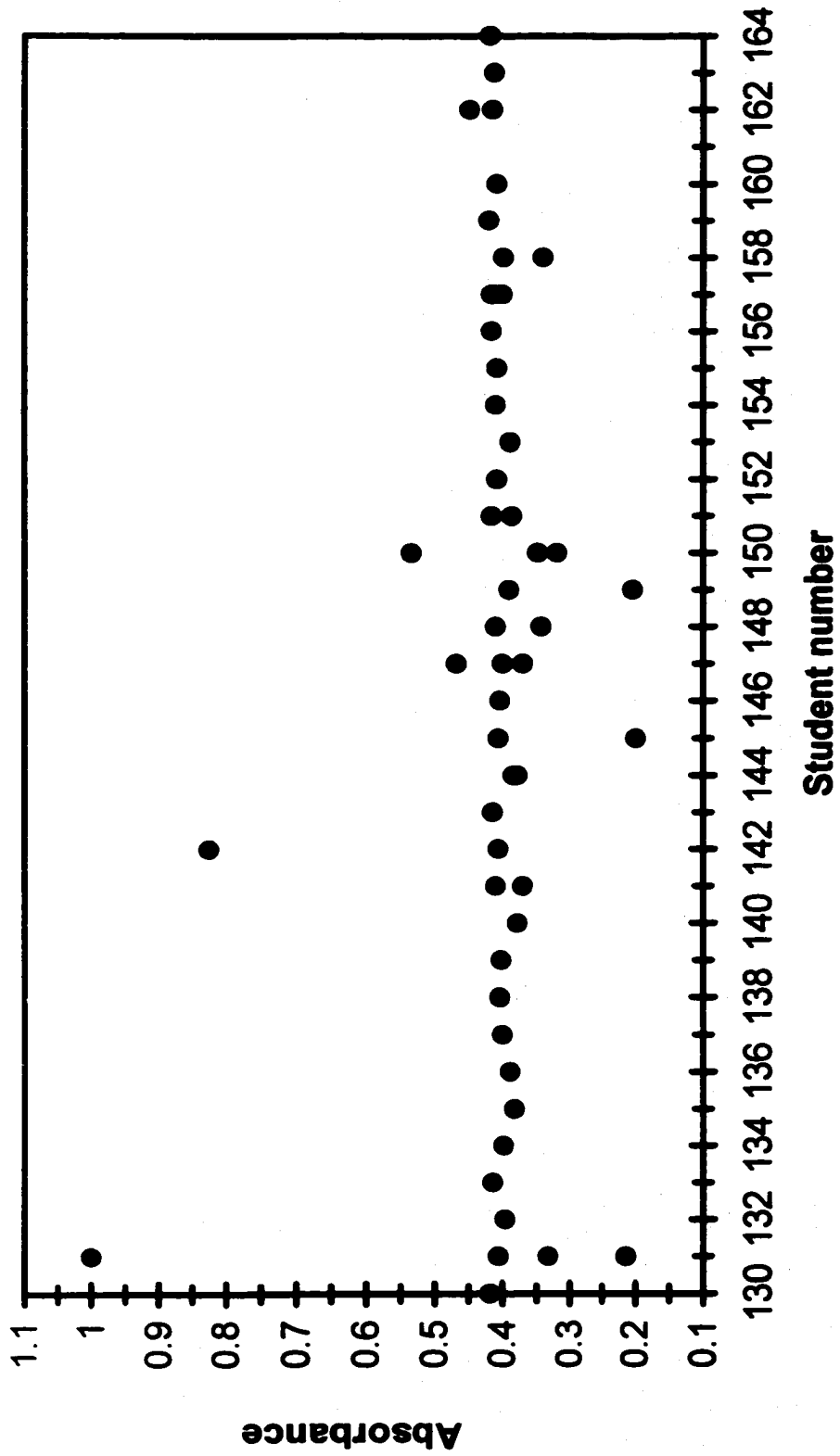


Figure 14. Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions, Fall 2000

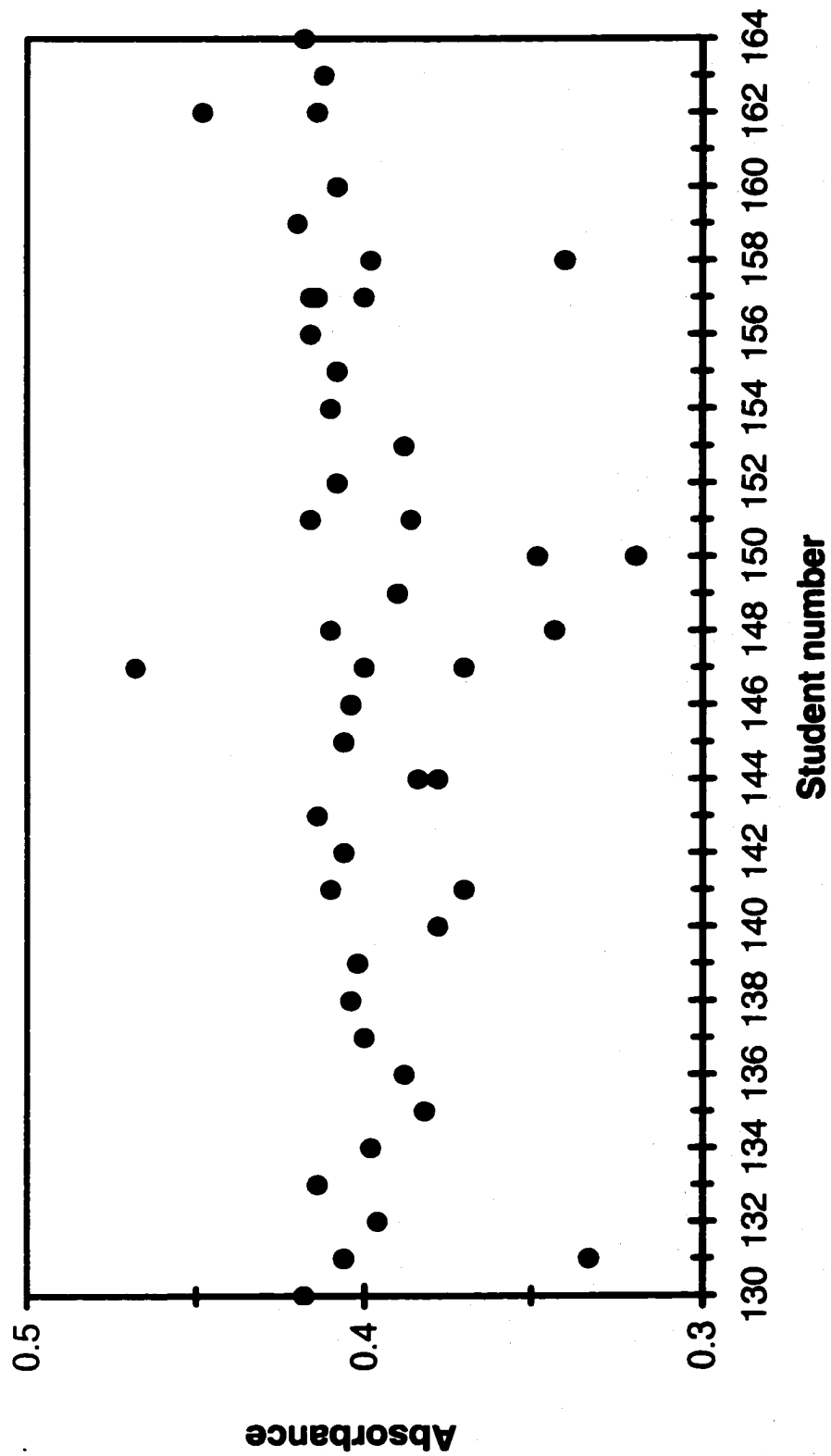


Figure 14a. Expanded Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions, Fall 2000

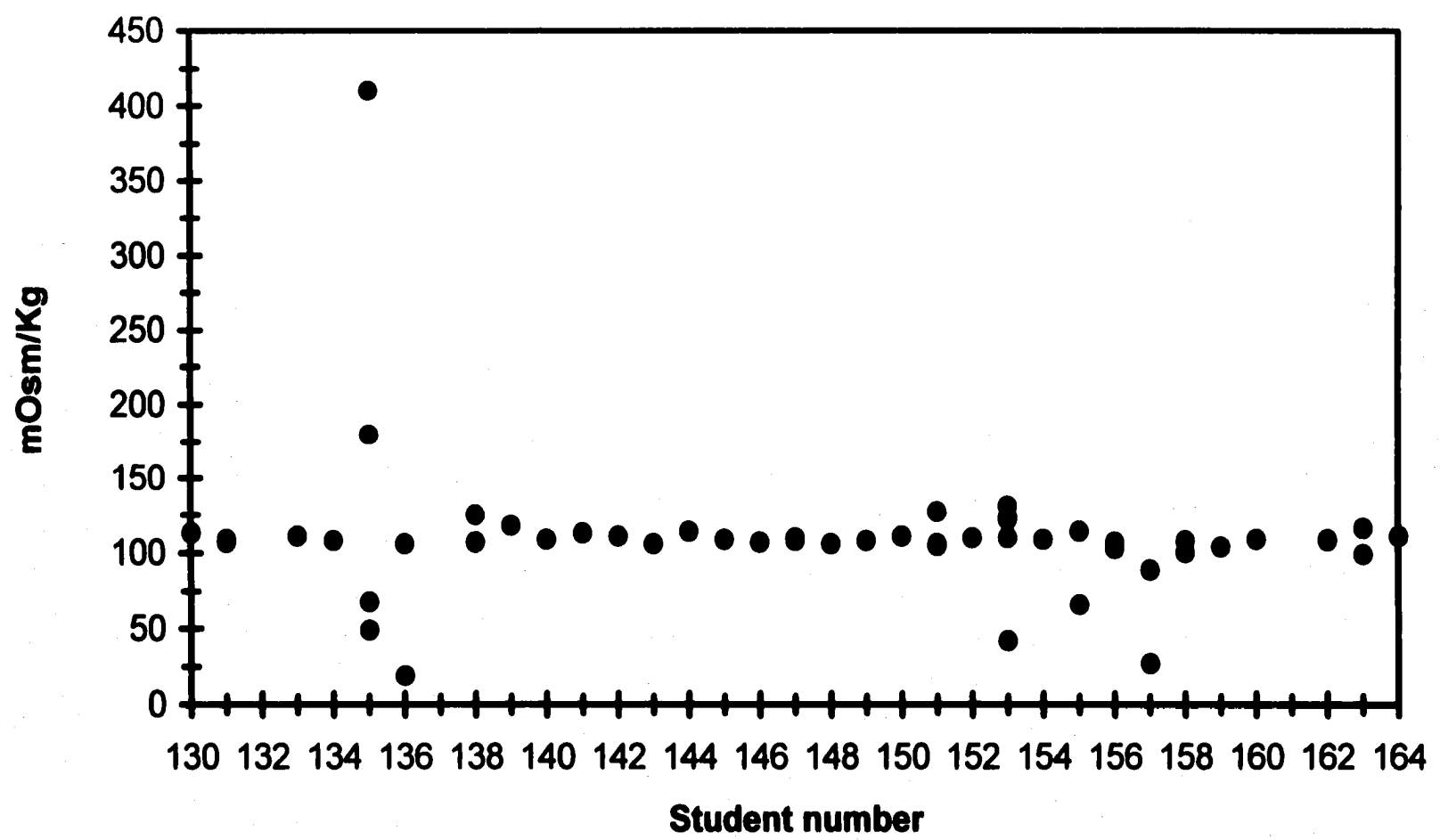


Figure 15. Osmometer Readings for 0.0600 M KCl, Fall 2000, View 2

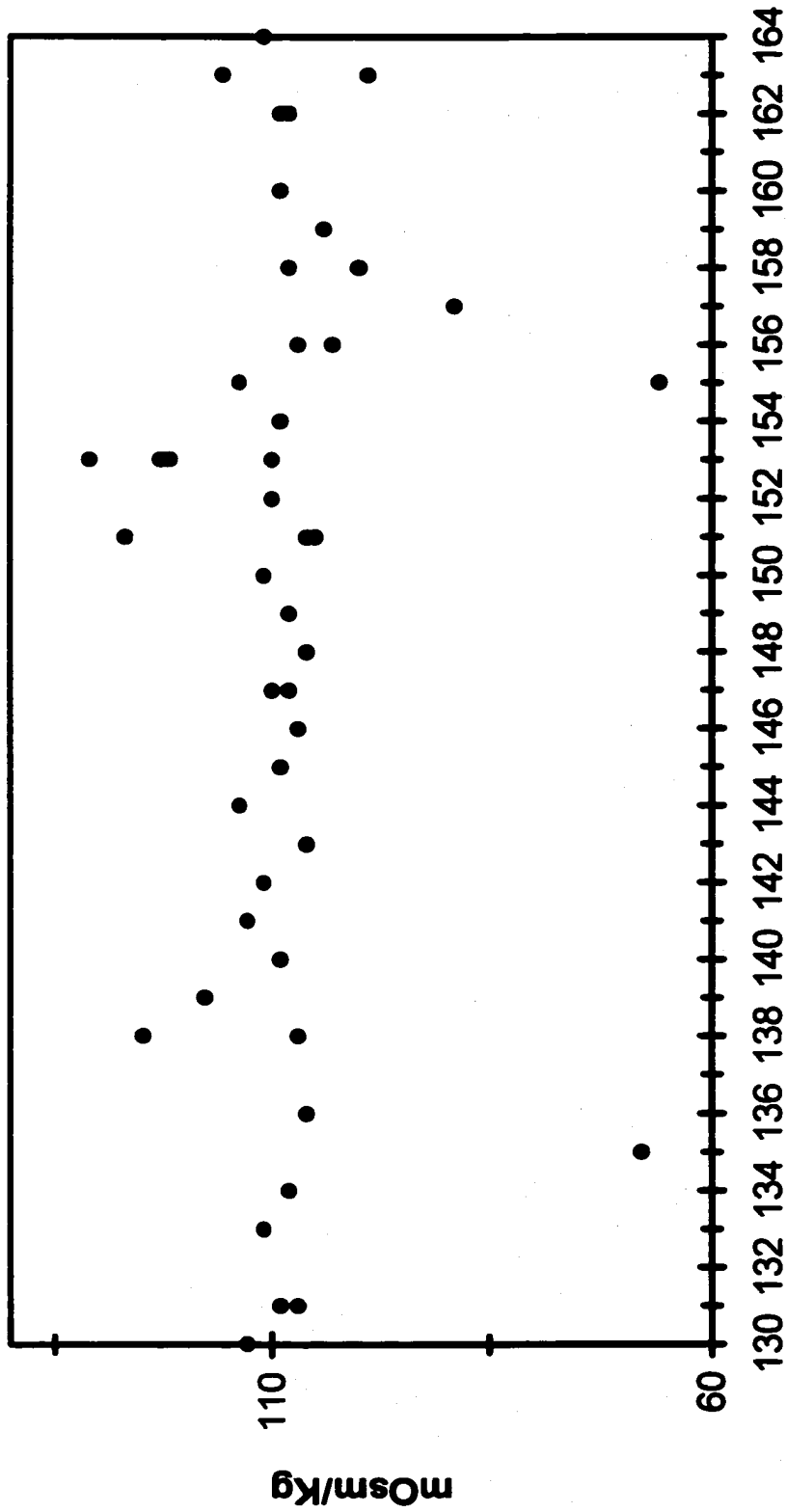


Figure 15a. Expanded graph of Osmometer Readings for 0.0600 M KCl, Fall 2000, View 2

it is difficult to resolve them on this scale. An expanded graph (Figure 15a) shows that student number 131 had two readings, 107 and 109 mOsmol/kg; student number 135 had four (49, 68, 180, 410 mOsmol/kg, including the point off scale); student number 147 had two (108, 110 mOsmol/kg), student number 153 had five attempts (42, 110, 122, 123, 131 mOsmol/kg, including the ones off scale), and student number 162 had two preparations (108, 109 mOsmol/kg). The multiple readings and sometimes the closeness of these data clearly indicate the determination of some students to “match exactly” the instrument readings of the standard solutions. The graphs of instrumental readings for all semesters in the study will be found in Appendices B1 through B6.

Exit Questionnaire Item Responses

Responses of 160 students who participated in the exit questionnaire are given in Table 9. Seven (7) students elected not to complete the exit questionnaire. Students' reactions to questionnaire item 1 were overwhelmingly positive: 97.5 % responded that the experiment was very helpful. Responding to questionnaire item 7, a little more than six seventh (86.3 %) felt their ability had improved and only 22 (13.7 %) felt no improvement. Handouts, use of instruments, and organization were all highly rated also as indicated by questionnaire items 8, 9, 10, and 11. Moreover, 93.8 % of respondents to item 5 reported that plenty of time was allocated to allow them to finish the experiment; 83.8 % agreed that the content of the experiment was appropriate for their level of study (item 4); 70.0 % reported that the experiment was the easiest they have done thus far (item 2), and 80.0 % reported that previous experiments gave them enough practice (item 3) to construct new knowledge to handle dilution of solutions.

Table 9. Student Responses to the Exit Questionnaire

1. "In general, how well did this experiment help you focus on the important points in the course"?

Response	Number	Percentage
Very well	60	37.5
Reasonably well	96	60.0
Poorly	4	2.5

2. "How would you rate the relative difficulty of this experiment compared to other experiments you have completed in this course (or other labs) this academic term"?

Response	Number	Percentage
(Least difficult) 5	61	38.1
4	51	31.9
3	37	23.1
2	10	6.2
(Most difficult) 1	1	0.6

3. "The activities in previous quant experiments gave me sufficient practice to do this experiment".

Response	Number	Percentage
Strongly agree	58	36.2
Agree	70	43.8
Neutral	23	14.4
Disagree	9	5.6
Strongly disagree	0	0.0

4. "The content level of the experiment was appropriate for my level of experience".

Response	Number	Percentage
Strongly agree	56	35.0
Agree	78	48.8
Neutral	21	13.1
Disagree	3	1.9
Strongly disagree	2	1.8

5. "Proper time was allocated to this experiment".

Response	Number	Percentage
Strongly agree	88	55.0
Agree	62	38.8
Neutral	7	4.4
Disagree	2	1.2
Strongly disagree	1	0.6

6. "Which instrument was easier to use? Help us explain your choice".

Response	Number	Percentage
Spectronic 20	74	46.2
Osmometer	70	43.8
Both	10	6.2
Neither	4	2.5
No response	2	1.2

Table 9 (continued)

7. "Has your ability to prepare solutions improved as a result of this experiment"?

Response	Number	Percentage
Yes	108	67.5
Somewhat	30	18.8
Stayed the Same	19	11.9
No	2	1.2
No response	1	0.6

In general, how would you rate this experiment for the following points?

8. "Was it interesting"?

Response	Number	Percentage
(Excellent) 5	18	11.2
4	59	36.9
3	62	38.8
2	15	9.4
(Poor) 1	4	2.5
No response	2	1.2

9. "Clarity of handouts"

Response	Number	Percentage
(Excellent) 5	87	54.4
4	53	33.1
3	16	10.0
2	2	1.2
(Poor) 1	0	0.0
No response	2	1.2

10. "Helpfulness of instruments"

Response	Number	Percentage
(Excellent) 5	69	43.1
4	63	39.4
3	23	14.4
2	3	1.9
(Poor) 1	0	0.0
No response	2	1.2

11. "Organization of experiment"

Response	Number	Percentage
(Excellent) 5	76	47.5
4	65	40.6
3	13	8.1
2	2	1.2
(Poor) 1	2	1.2
No response	2	1.2

Of the two instruments used in this experiment, 46.2 % of students surveyed reported that Spectronic 20D was easier to use, while 43.8 % favored the osmometer. The majority of students in the sample had no prior experience with the osmometer nor had they heard of the instrument before this experiment. However, some students indicated that they have had some experience with the Spectronic 20D. In addition, 82.5 % of respondents reported that the use of these instruments as a feedback tool was helpful (item 10).

A summary of responses to the questionnaire open-ended items 12 and 13 is shown in Table 10.

Table 10. Summary of Student Responses to the Exit Questionnaire Open-ended Questions

<p>12.- What did you like least about this experiment?</p> <p><i>"the pipette bulbs are hard to get off without sucking solution into the bulb"</i> <i>"manual pipetting"</i> <i>"cleaning the pipettes"</i> <i>"the most time was spent just ensuring pipettes were dry".</i> <i>"pipetting"</i></p>
<p>13.- What was the most significant thing you learned from this experiment?</p> <p><i>"Accuracy is very important and a slight mistake could cause a big change"</i> <i>"It showed me how easy it is to make a mistake"</i> <i>"How to use the pipette properly"</i> <i>"To be careful making solutions"</i> <i>" To think on my own"</i></p>

A content analysis of students' responses to question item 12 (see Table 11) revealed that what the students liked the least about the experiment was not only the pipetting (19 reporting), but that they were also worried about the accuracy and precision of their data (17 students). The content analysis also indicated that 13 respondents believed they were not sufficiently prepared to perform the experiment. Three students stated that the experiment was neither exciting nor challenging enough, while 21 students reported that there was "nothing" they did not "like least" about the experiment. Many students (22) expressed displeasure about waiting in line to use the instruments. There were no other predominant complaints. For unknown reasons however, 56 students chose not to respond to this question.

Impact of Demographics on Students' Performances in the Experiment

In addition to evaluating students' solution preparation instrumentally and by exit questionnaire, other tools were utilized such as the demographic survey already mentioned elsewhere, informal interviews of students during the progress of the experiment (see Appendix E), and the researcher's own informal observations. For example, educators ask questions among themselves quite often about whether or not students working long hours on or off campus at a paying job will have an impact on their ability to concentrate on academic demands. Usually faculty suspects that students' grades suffer if they work more than 20 hours in addition to their course load. Figure 16 is the plot of students' hours on an outside, paying job versus the grades earned in the experiment.

**Table 11. Content Analysis for Exit Survey, Question 12:
What Did You Like Least About This Experiment?**

Category	Number of Students Responding
Pipetting	19
“Nothing”	21
Inconvenience	
waiting in line to use instruments	22
finding glassware	3
small volumetric flasks	2
lack of help	1
cleaning glassware	1
time consuming	1
work/lab schedule conflict	1
long briefing—not needed	1
Insufficiently Prepared	
took 6 times to do one	5
not understanding the functioning of Spec 20	5
to do it without a partner	1
to determine which equipment was better to use	1
not understanding osmette	1
Worry About Precision	
not knowing how precise is precise	5
that we had to be precise drove me nuts	1
getting different readings on same sample	2
Osmette reading	1
Worry About Accuracy	
accuracy	3
feeling like a loser when numbers didn’t match	2
contamination affecting results	1
only able to test 2 solutions	1
didn’t know if you did it right or wrong (not a lot of instructions)	1
Level of Difficulty	
not challenging	1
not exciting	1
not exciting as titration or making solid	1
No Response to Question 12	56

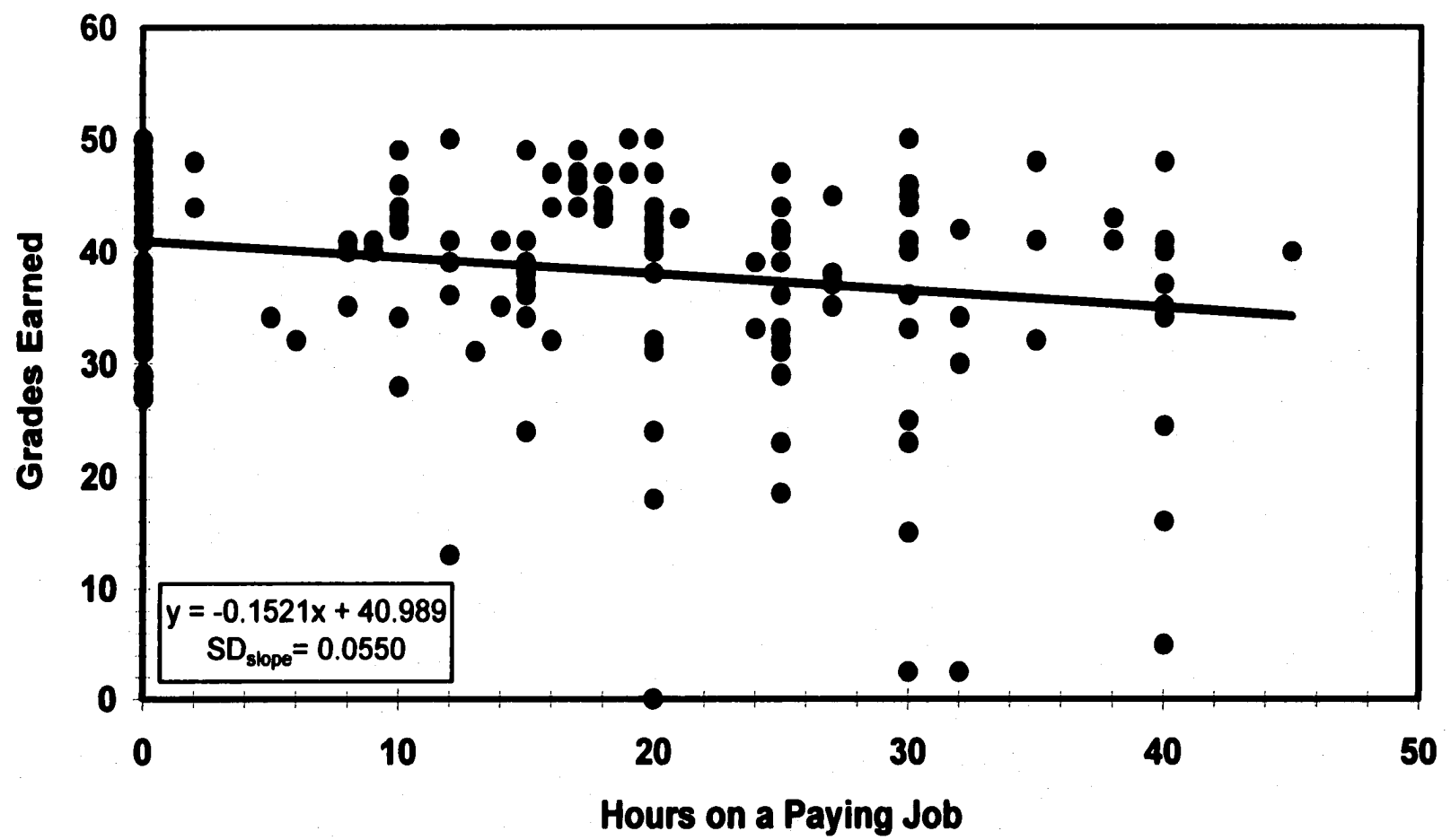


Figure 16. Students' Hours On/Off Campus Work vs. Dilution Experiment Grades

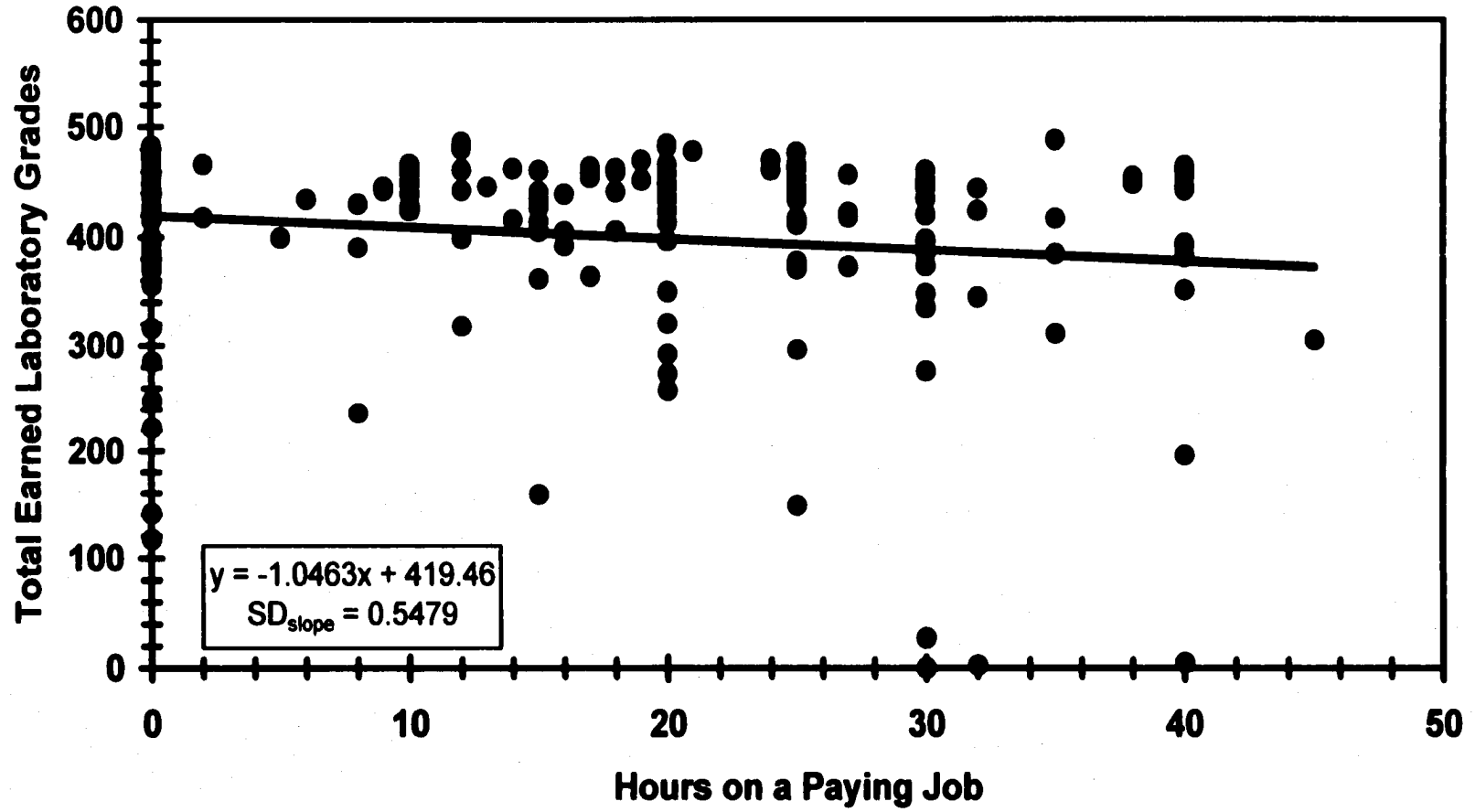


Figure 17. Students' Hours on the Job vs. Laboratory Total Grades

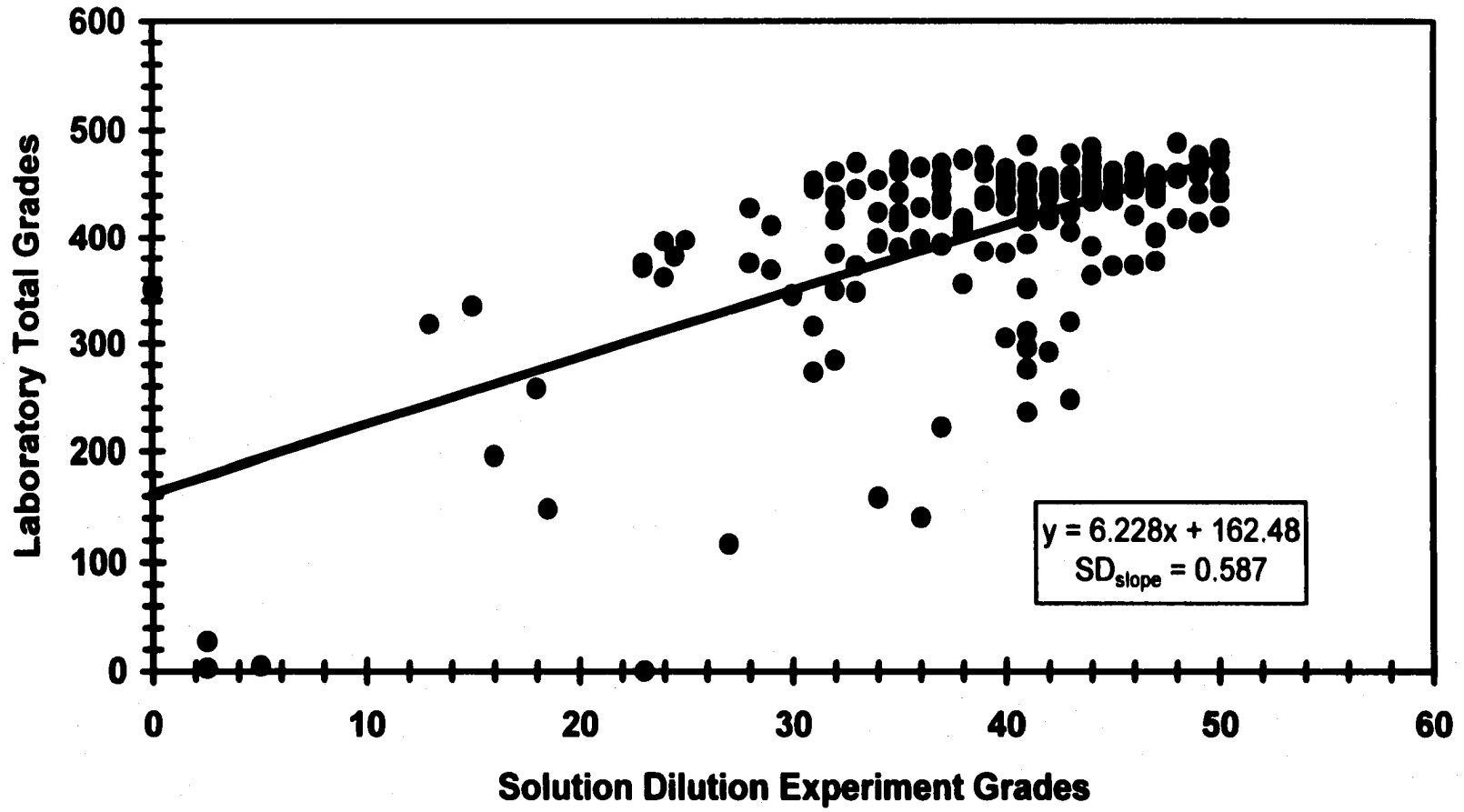


Figure 18. Solution Dilution Experiment Grades vs. Laboratory Total Grades

Surprisingly, the graph shows that there is only a minor correlation between grades earned from the dilution experiment and the time that students spend on extra curricula work (slope = -0.1521 ± 0.0550 , 95% confidence level). Figure 17 indicates that there is also some correlation between hours on the job and the final grades students earned in the laboratory segment of quantitative analysis (slope = -1.0463 ± 0.5479 , 95% confidence level). In other words, students generally lose about one point on their lab grade for every hour worked per week at an outside job. One of the research questions was to find out whether or not the grades students earn from the solution dilution experiment could be predictive of the overall grades in the quantitative analysis laboratory. It was interesting to find that the dilution experiment grades do indeed predict the students' total laboratory grades (slope = $+6.228 \pm 0.587$, 95% confidence level, Figure 18).

Chapter 5

Discussion and Conclusion

“The principal goal of education is to create men who are capable of doing new things, not simply of repeating what other generations have done -- men who are creative, inventive and discoverers.”

-Jean Piaget (1896-1980)

Overview

Rapid instrument feedback provided students the opportunity to engage in self-evaluation of their laboratory work. Students seemed to view the feedback as objective and non-punitive. Their confidence in the instruments was evidenced by their reactions when the instrument readings of the standard solution were either far different or very close to their own preparations. Some were surprised that the instruments could report reproducible results for a given solution in repeat measurements. Others seemed astounded that instruments could measure the difference between carefully prepared and carelessly prepared solutions. If properly calibrated, they believed that instruments are infallible. Instrument feedback methodology proved to be an excellent teaching tool that enables students to accurately determine the concentration of solutions they have prepared by serial dilution. It taught students to ask probing questions that get to the experimental design, while it poised students to re-examine and analyze a laboratory procedure critically. In addition, it helped students in the understanding and interpretation of data (tasks that require more complex thinking skills). The experiment demonstrated clearly to the students that the proper use of pipettes was a needed skill for preparing solutions accurately.

A closer look at the instrumental reading graphs revealed that some of the poorly prepared solutions could have been avoided. In some cases, solutions were so incorrectly prepared that their readings sometimes fell outside the printable range of the graphs. It was interesting to observe that many students who had to try again learned that they could have bypassed this second exercise by simply inverting the volumetric flask to mix the solution more completely before taking the reading. Other factors that affected students' preparation were simply obvious. Among these are:

- a) Choice of wrong glassware: Some students turned in their solutions prepared in Erlenmeyer flasks instead of appropriate volumetric flasks. In some cases, solutions were prepared in the wrong size volumetric flasks.
- b) Choice of wrong measuring apparatus: Judging from the comments the researcher received, some students found pipettes hard to use (or didn't know they should use them), and therefore chose beakers and graduated cylinders instead.
- c) Cuvette rinsing: After calibration of the Spec 20D with a blank (water in this instance), some students continued to rinse the cuvette with water between measurements in lieu of rinsing the cuvette with a small amount of the next solution to be measured. This process made their solutions more dilute than the target concentration.

The student interviews as the experiment progressed revealed that most students had no difficulty calculating the proper amount of samples to be diluted. Most of the dilution errors resulted from the practical factors mentioned above.

Students were encouraged to prepare solutions again if they were not satisfied with their instrument readings. Figure 19 shows that 134 students (78.4%) were either successful at first try, or they simply decided not to repeat the preparation regardless of the outcome. Whereas 29 students (16.9%) made two trials, seven students (4.1%) had three trials, while only one student (0.6%) had five trials. Figure 20 indicates the same trend for the potassium chloride (KCl) solutions with 144 students (84.2%) happy after the first trial, 17 students (9.9%) with two trials, nine students (5.3%) performing three repeats, and only one student (0.6%) with five trials.

Instructors often adjust the course content when they perceive a difference in performance of students from one semester to another. These instructors seem to ignore that they are dealing with different group of students who possess different academic, social, and environmental backgrounds. Also, students bring different learning styles and different approaches to the study of chemistry. The students in this study had more control over the experiment than is usual in a sophomore laboratory. The serial dilution experiment involved in this project covers the period from fall 1998 to spring 2001. A comparison of the experiment data from semester to semester is depicted in Figures 21 and 22. These charts suggest that the classes of spring 1999, spring of 2000, and fall 2000 did not perform as well as those in other semesters based on an examination of the scattering of points. There is no one-factor known to have contributed to this discrepancy. It could have been the class size (fall 1998 is small). Or it could have been an external factor such as an effort to reach out to students in order to increase the enrollment in the course: the instructor contacted prospective students by letter after fall 1998, thereby

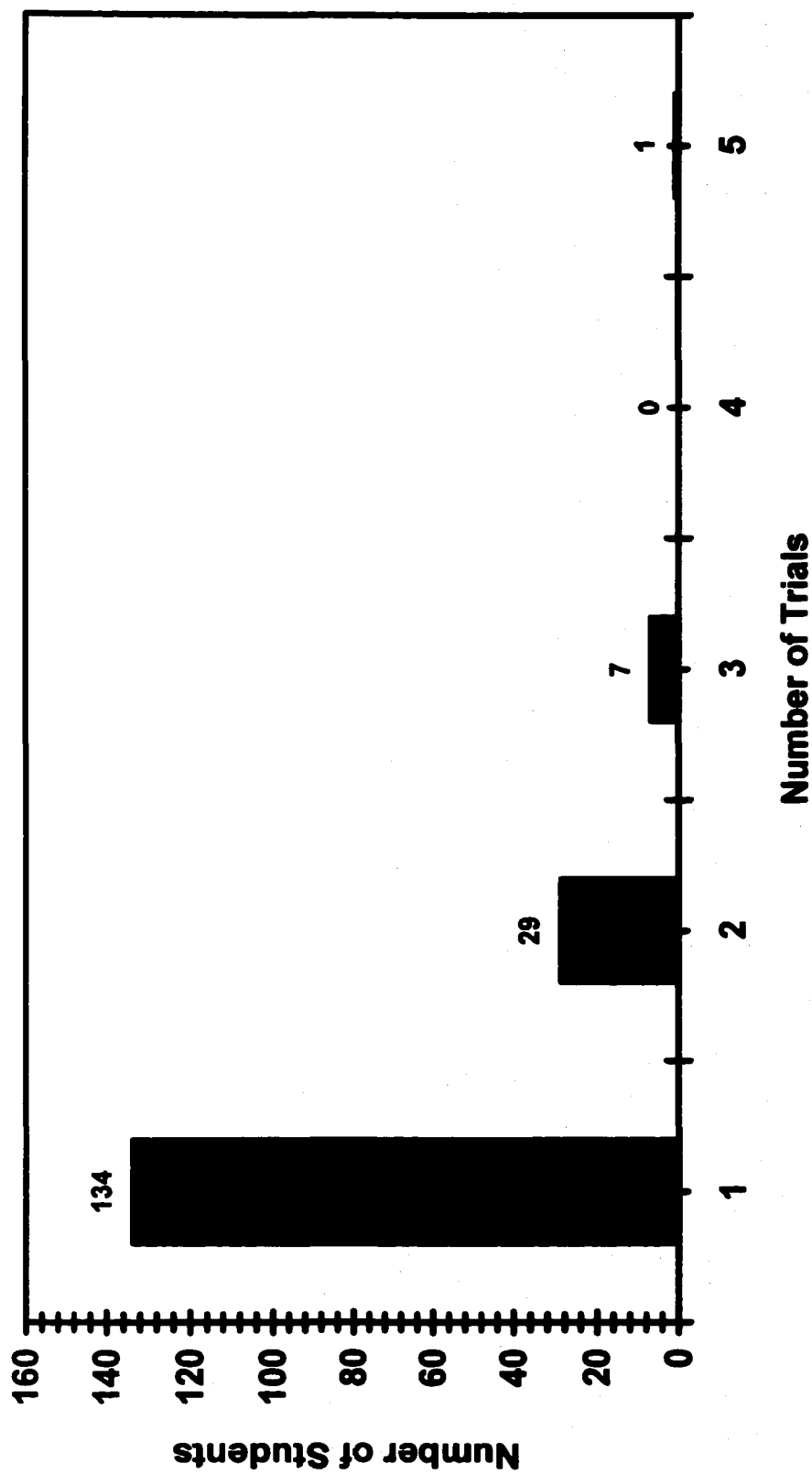


Figure 19. Number of Preparations per Student for 0.0800 M $\text{Ni}(\text{NO}_3)_2$ Solutions

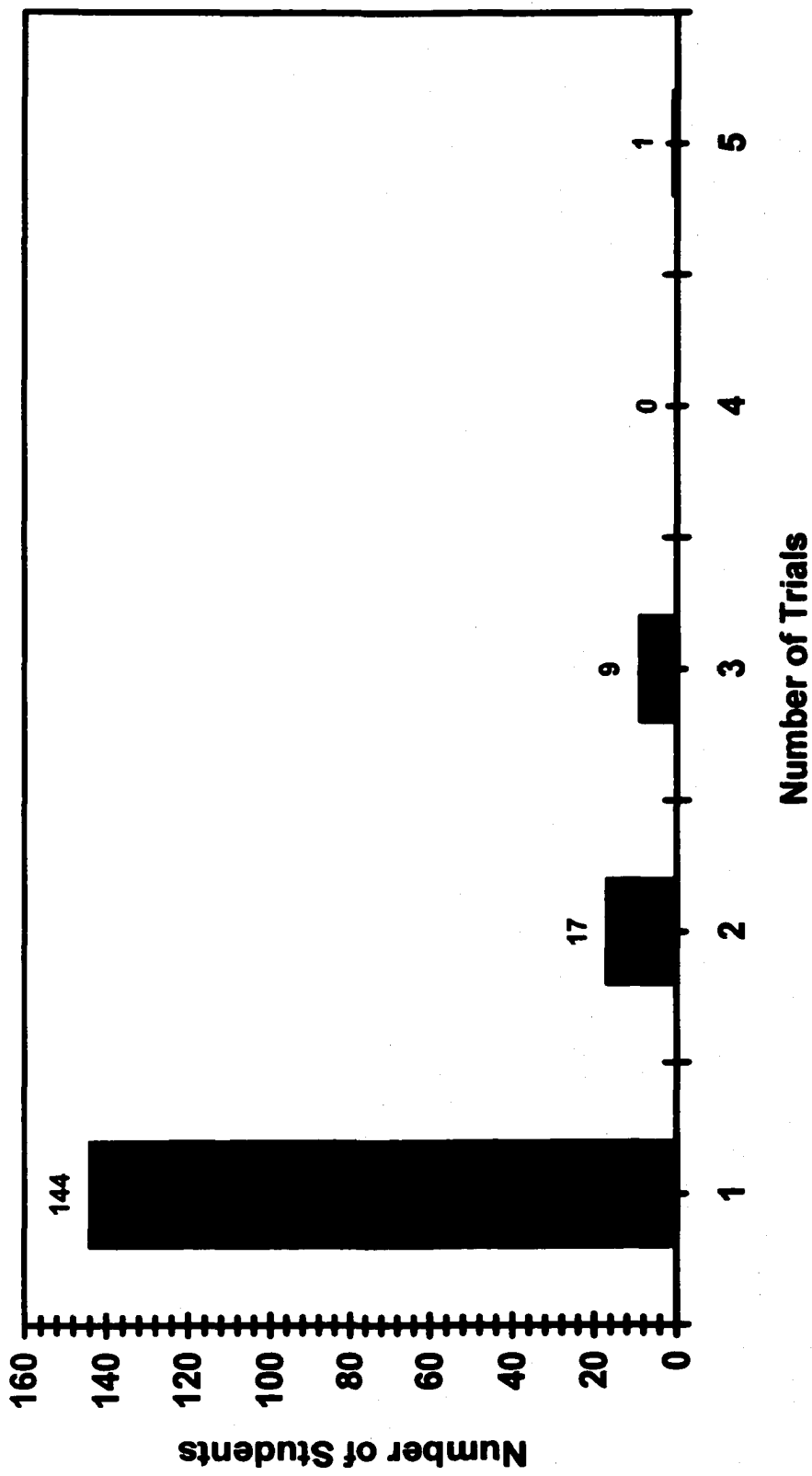


Figure 20. Number of Preparations per Student for 0.0600 M KCl Solutions

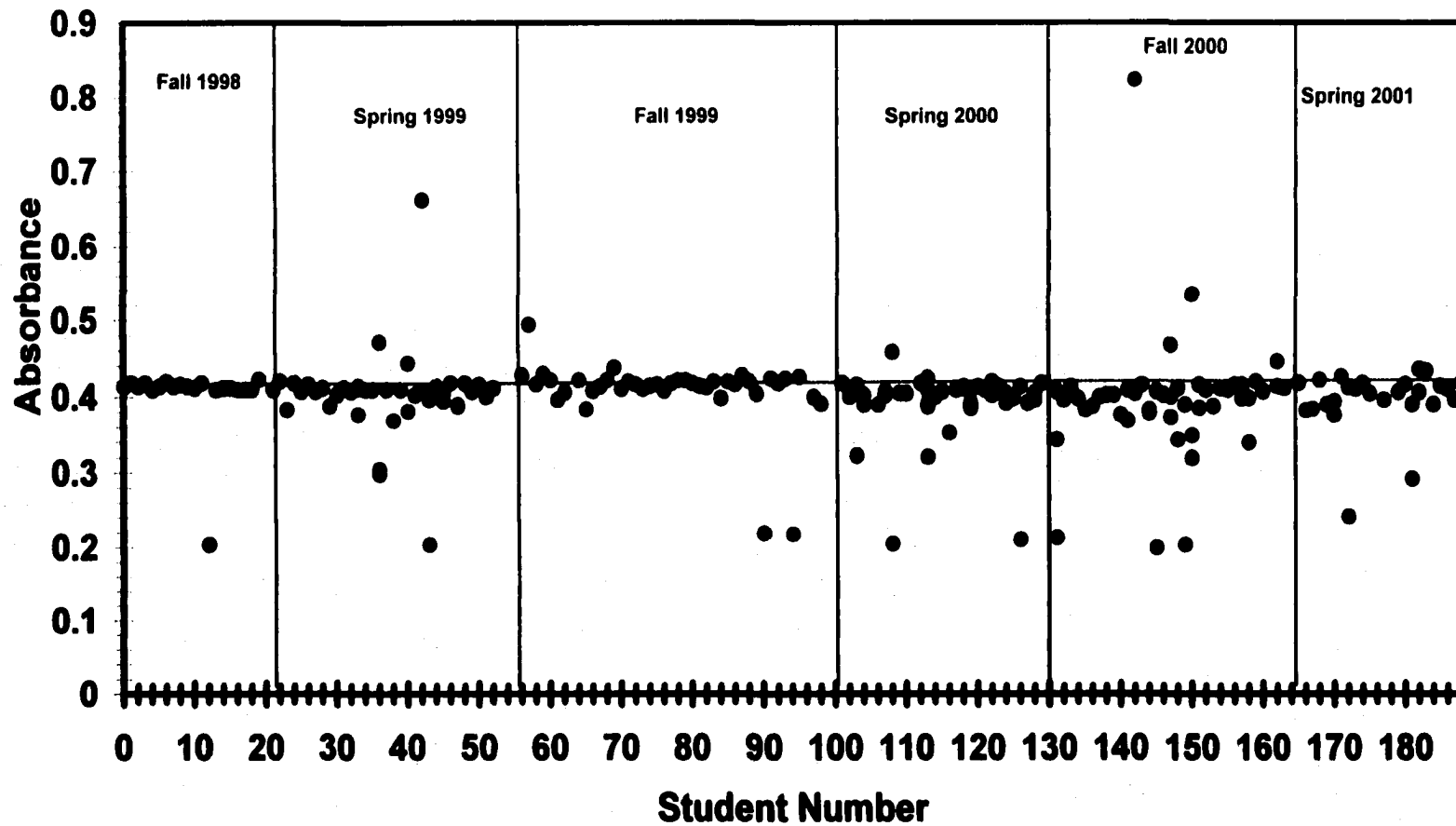


Figure 21. Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions
Fall 1998 - Spring 2001

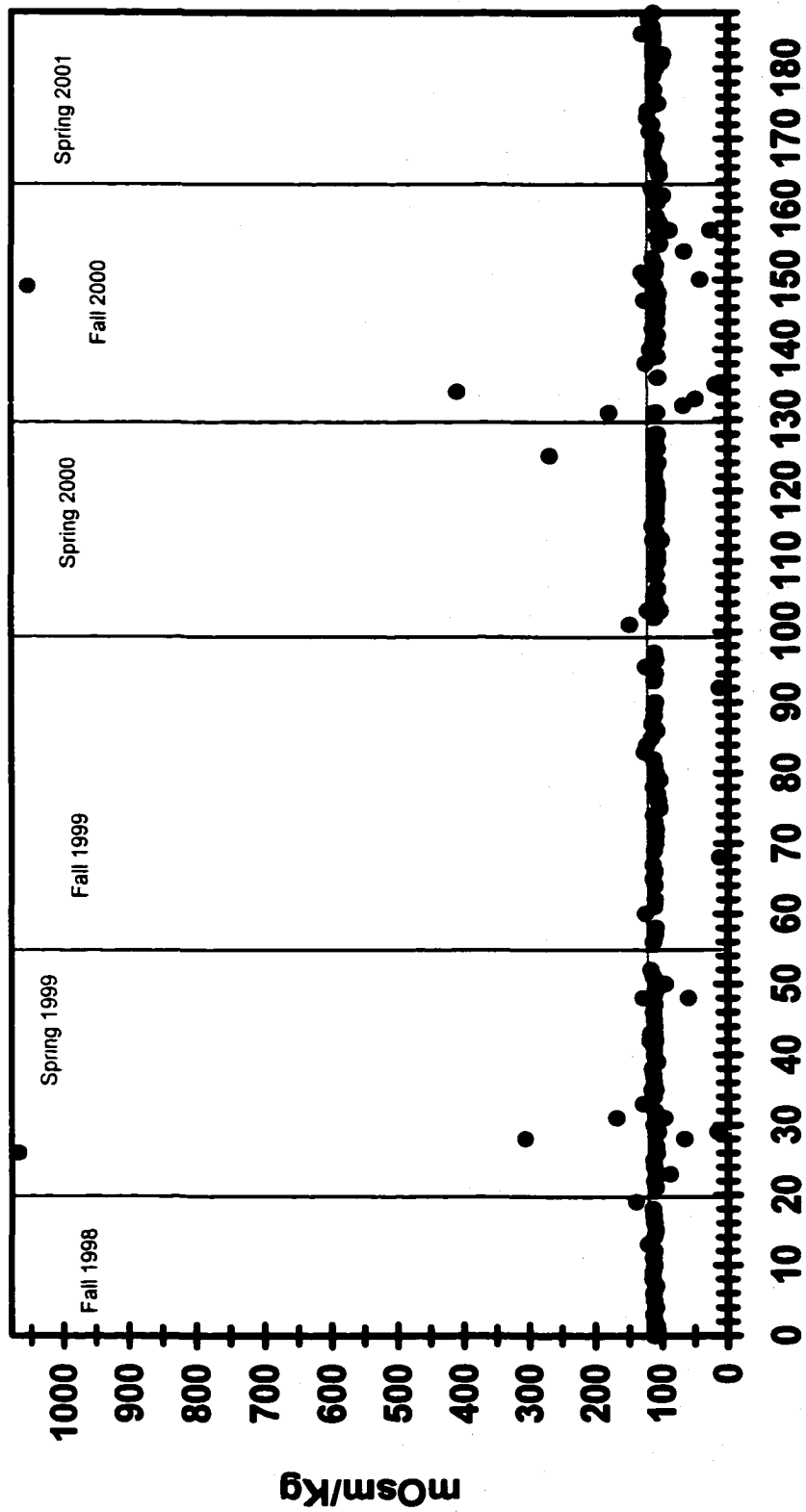


Figure 22. Osmette Data for 0.0600M KCl solutions
 Fal 1998 to Spring 2001

attracting students who were perhaps not as well prepared for independent work in an open laboratory environment. In any case, it is difficult to know with certainty the real cause of this difference in students' performance from semester to semester. Nonetheless, most students obtained instant gratification from their laboratory technical performance as they "constructed" their own learning environment. They viewed their results in diluting solutions to the correct concentration as a real accomplishment. Moreover, the interactive learning from the instrument feedback assisted by an instructor-partner was a more appealing learning method because it placed students in center stage. The results of this research demonstrated that self-evaluation with instrumental feedback is a useful tool in helping students apply the knowledge they have acquired in lectures to the practice of chemistry. In a recent editorial in the *Journal of Chemical Education* entitled "When is an Experiment a Success," John W. Moore [49] wrote: "Any experiment has a considerable value, provided that we can learn from it how to create new, successful approaches to teaching. Even if we are completely satisfied with a course", he stressed, "there is certainly room for variation and experimentation. Without such innovations, no matter how small", he concluded, "teaching would be less interesting and fun."

Recommendations for Future Study

The quantitative analysis laboratory at Middle Tennessee State University is operated on an open-lab format. The enrollment is usually separated into two sections. Students in each section are briefed separately, but they may attend either lab briefing and work in the open-lab setting. This format presents a problem for experiments that require two distinct sections of students to be used as experimental and control groups because the two

sections of students intermingle at will. Change in format is unlikely, so a cooperative effort with another institution should be considered so that the other institution's section of the quantitative analysis laboratory could serve as control group. Both demographic and summative (exit) questionnaires should be revised to assist in ease of analysis. A method should be devised to explore whether or not previous experience in making careful measurements of any type translates into students' more accurate preparation of solutions. A good question would be to ask if students had prepared solutions (before) in another course (including high school level course) and then see if what had been previously learned translates into increased accuracy in measurement. Also, some existing experiments involving solution preparation before and after the serial dilution experiment should be evaluated in order to assess whether or not students' solution making skills have improved sufficiently to permanently enhance their overall laboratory techniques.

Appendix A

Preparation of Standard Solutions and Serial Dilution Handouts

Solutions of nickel nitrate

Experiment 1. Colored primary and secondary standard solutions.

Stock solution provided: 1.000 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ primary standard solution.

(Solution 1) Prepare 50.00 mL of 0.100 M by diluting 1.000 M primary standard solution.

(Solution 2) Prepare 50.00 mL of 0.050 M solution by diluting solution 1 (serial dilution).

Label solution 1 and solution 2 with Exp. 1, your name, the concentration, the date. Tape the label in place with Scotch tape, and turn in solutions 1 and 2.

Experiment 2. Colored secondary standard solutions—Concentrations checked by photometry

Use 1.000 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ primary standard solution.

(Solution 3) Prepare 50.00 mL of 0.200 M solution by diluting 1.000 M primary standard solution.

(Solution 4) Prepare 25.00 mL of 0.0800 M solution by diluting solution 3.

Use the Spectronic 20 to check the absorbance of solution 4 vs. the 0.0800 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ standard solution provided, and prepare solutions 3 and 4 again if needed.

Label solution 3 and solution 4 with Exp. 2, your name, the concentration, the date. Tape the label in place with Scotch tape, and turn in solutions 3 and 4.

Solutions of potassium chloride**Experiment 3.** Colorless primary and secondary standard solutions.

(Solution 1) Prepare 100 mL of 1.500 M KCl solution from dry, weighed KCl solid.

(Solution 2) Prepare 25.00 mL of 0.600 M solution by diluting solution 1.

(Solution 3) Prepare 50.00 mL of 0.120 M solution by diluting solution 2.

Label solution 2 and solution 3 with Exp. 3, your name, the concentration, the date. Tape the label in place with Scotch tape, and turn in solutions 2 and 3.

Experiment 4. Colorless solutions—Concentrations checked by osmometry:

Use solution 1 from experiment 3. 1.500 M KCl solution from dry, weighed KCl solid.

(Solution 4) Prepare 50.00 mL of 0.150 M solution by diluting solution 1.

(Solution 5) Prepare 25.00 mL of 0.0600 M solution by diluting solution 4.

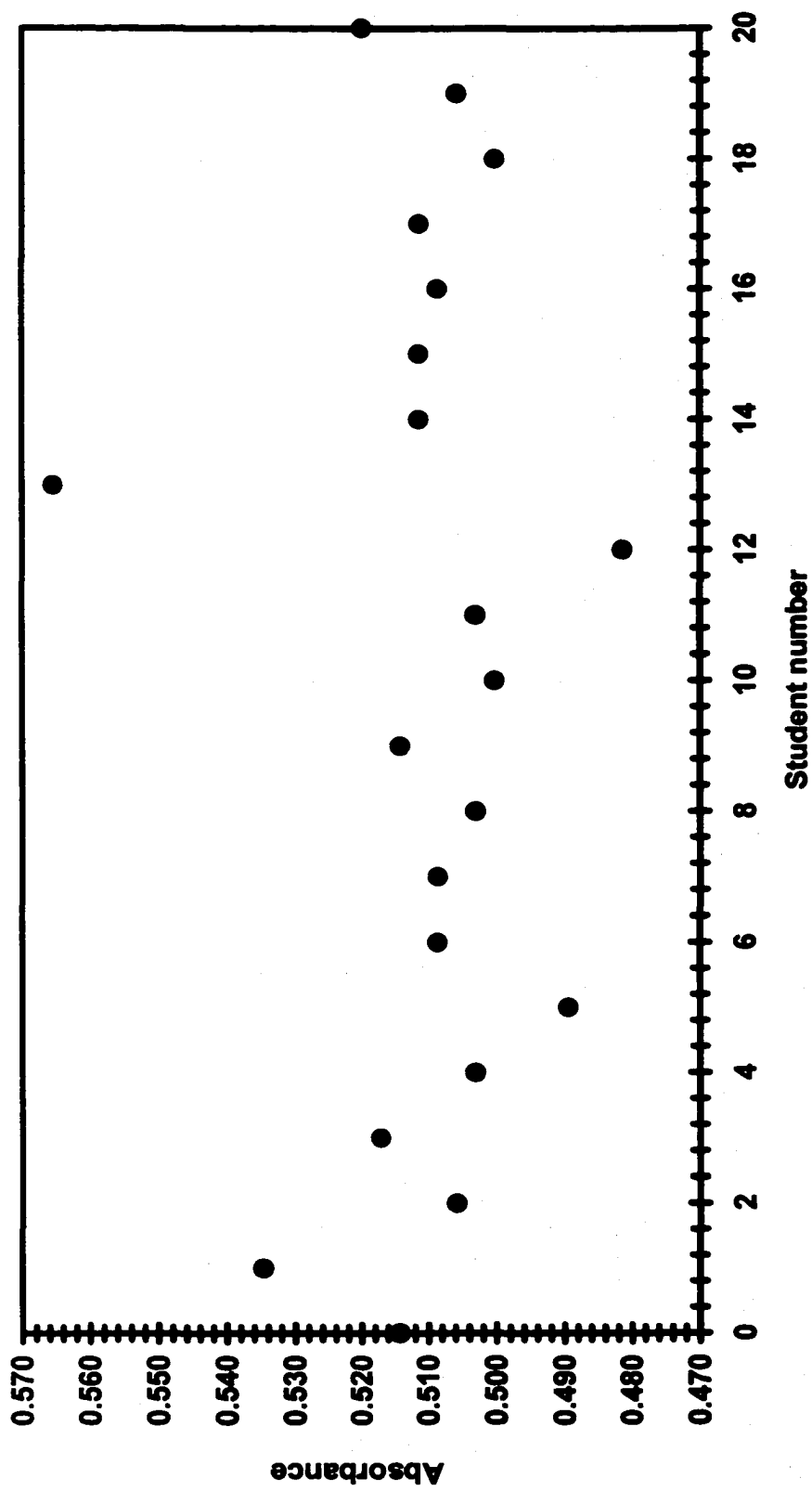
Use the osmometer to compare solution 5 with the 0.0600 M standard KCl solution provided and prepare solutions 4 and 5 again if needed.

Label solution 4 and solution 5 with Exp. 4, your name, the concentration, the date. Tape the label in place with Scotch tape, and turn in solutions 4 and 5.

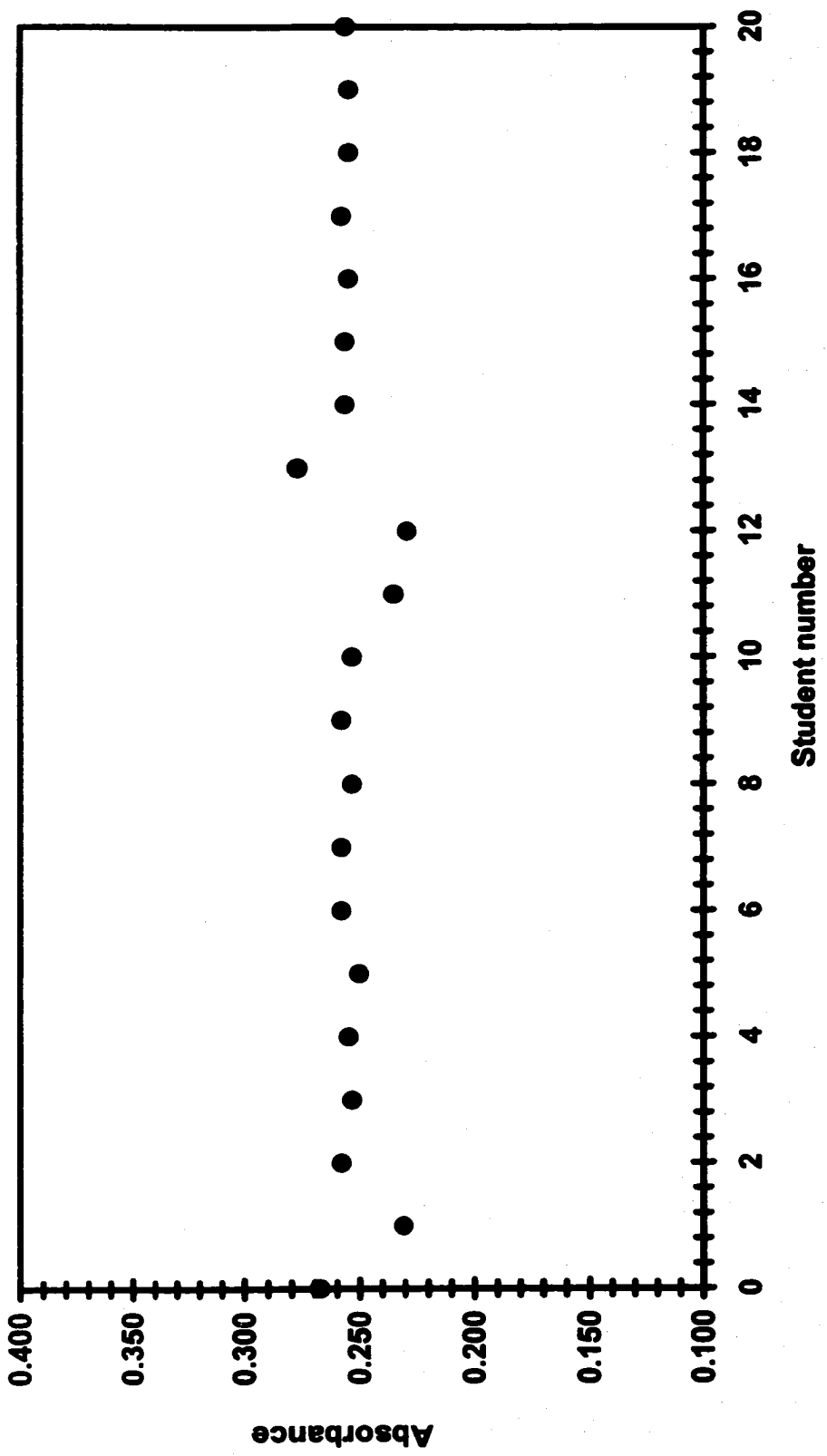
Appendix B

Spectrophotometric and osmometric readings of students' solutions are plotted here, and classified according to semester as Appendix B1 for fall 1998, through Appendix B6 for spring 2001.

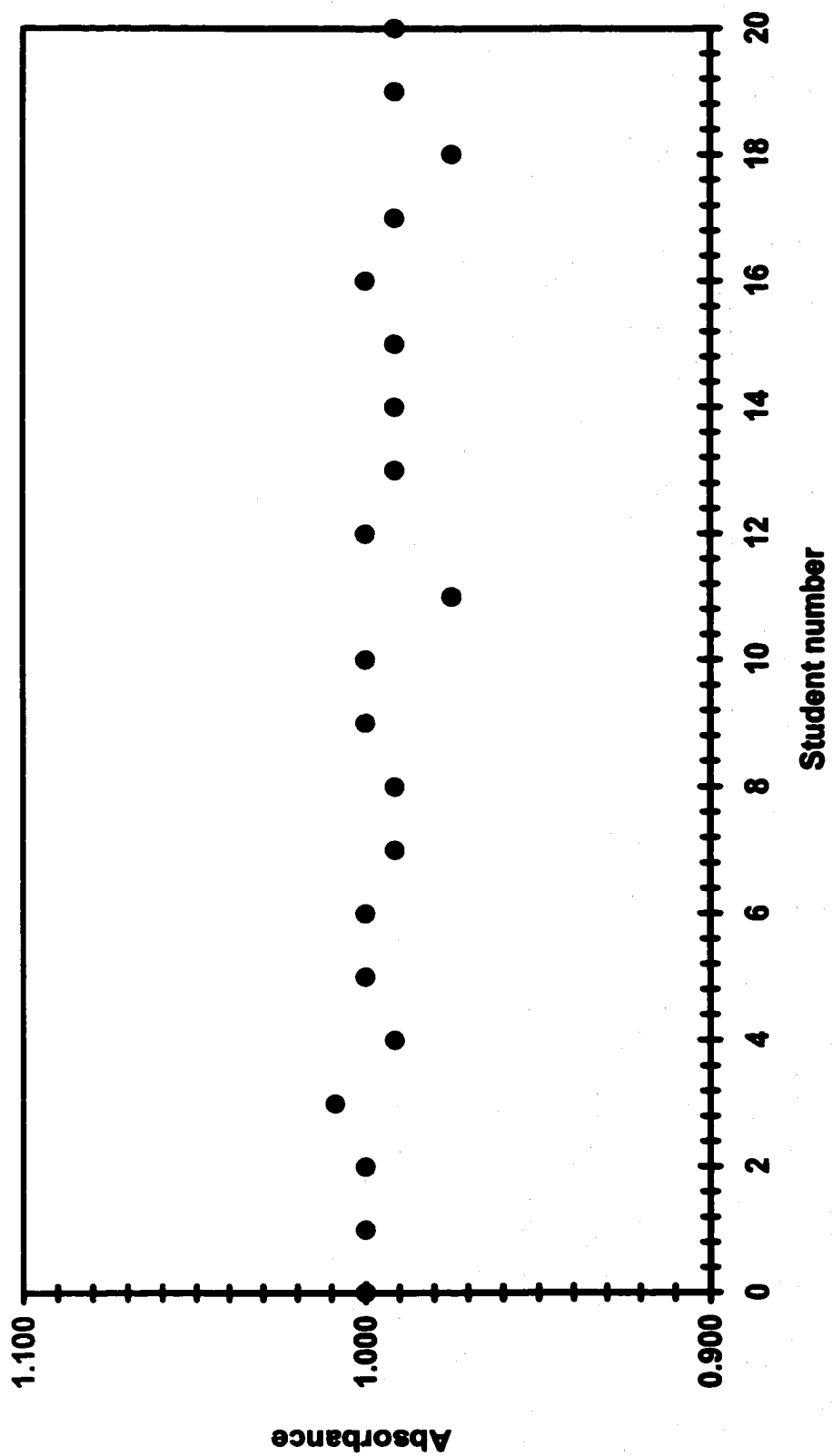
Appendix B1
Instrumental Reading Graphs for Fall Semester, 1998

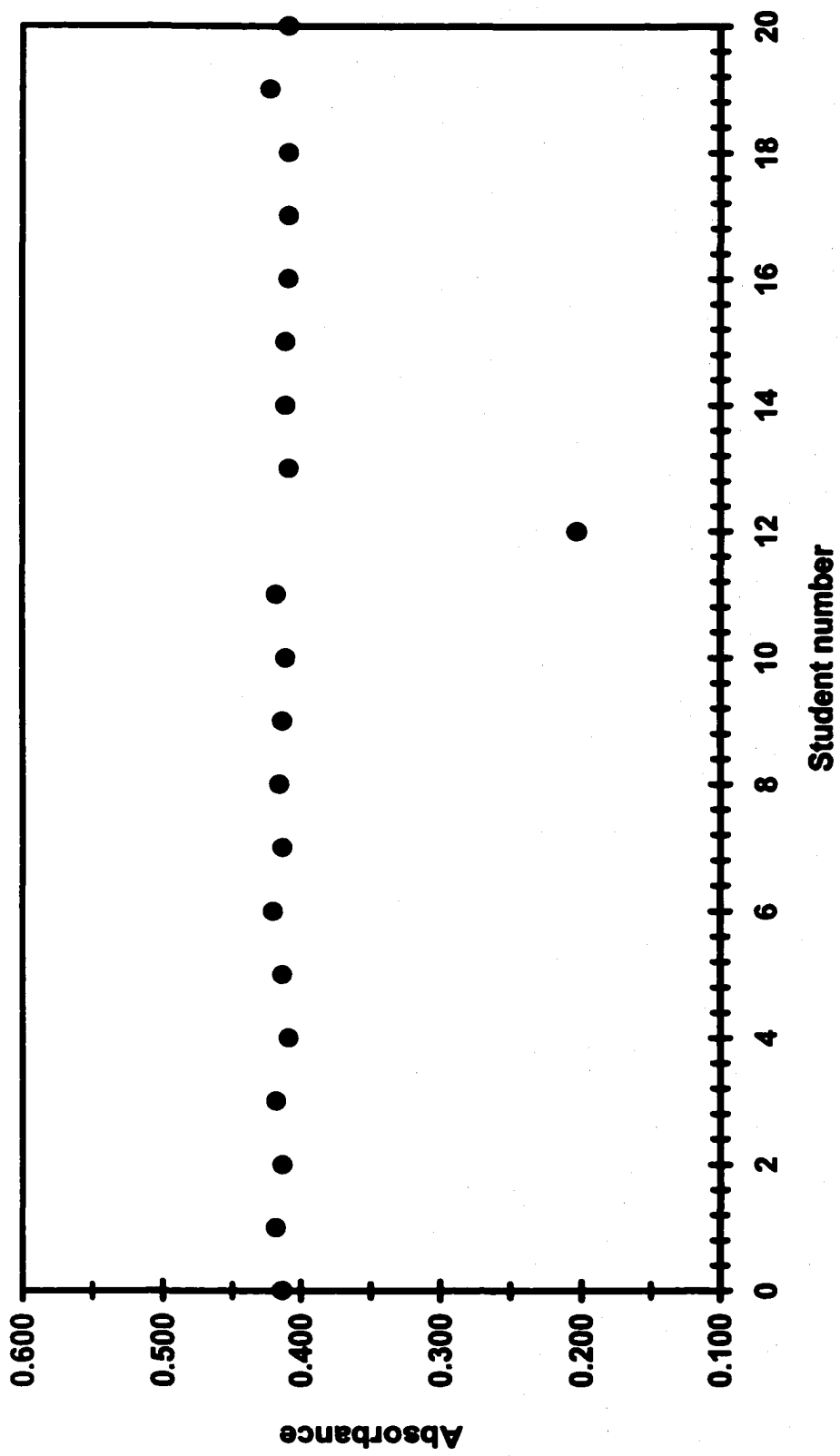


Spectronic 20 Data for 0.100 M Ni(NO₃)₂ Solutions, Fall 1998

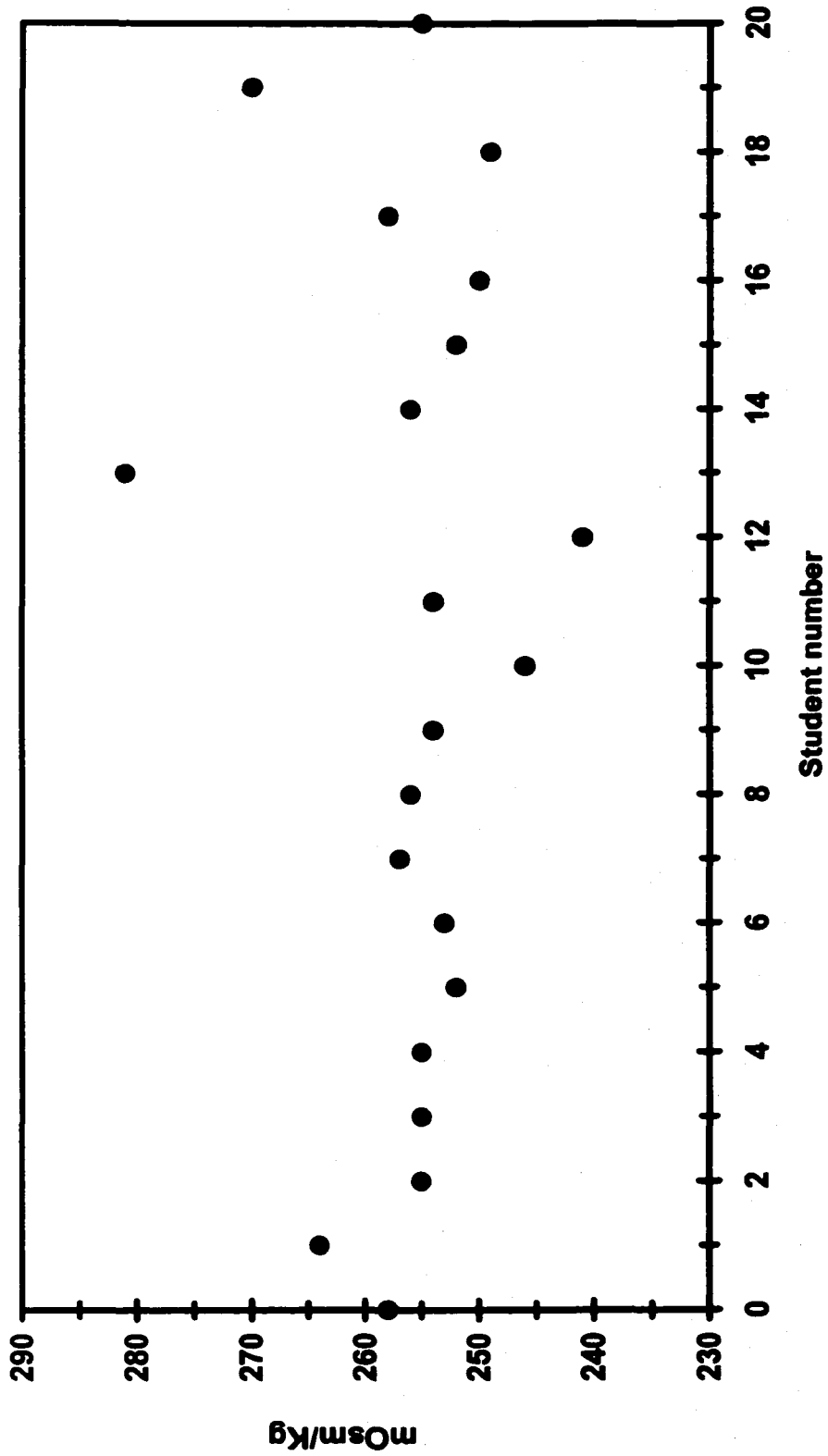


Spectronic 20 Data for 0.0500 M Ni(NO₃)₂ Solutions, Fall 1998

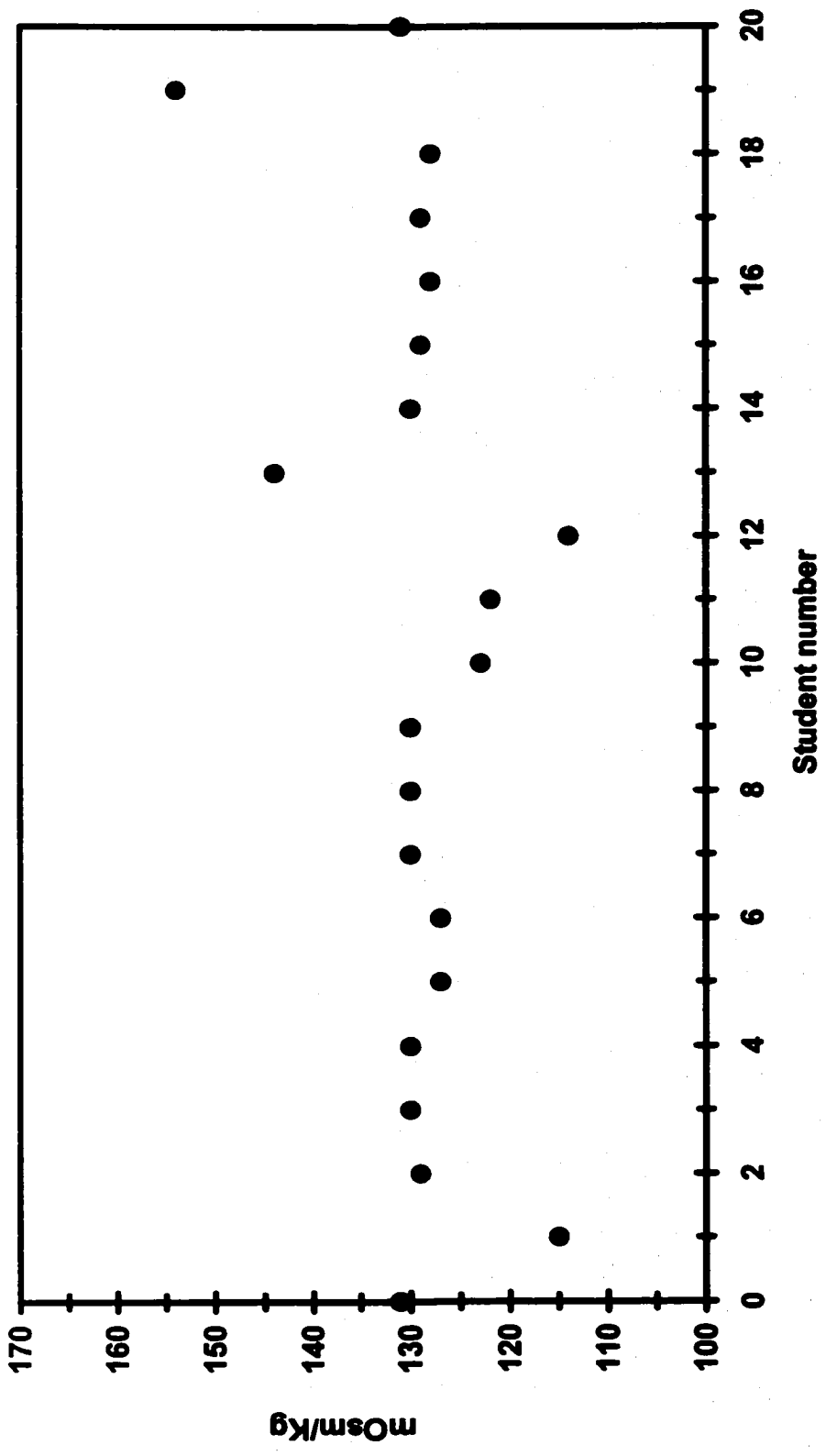
Spectronic 20 Data for 0.200M Ni(NO₃)₂ Solutions, Fall 1998



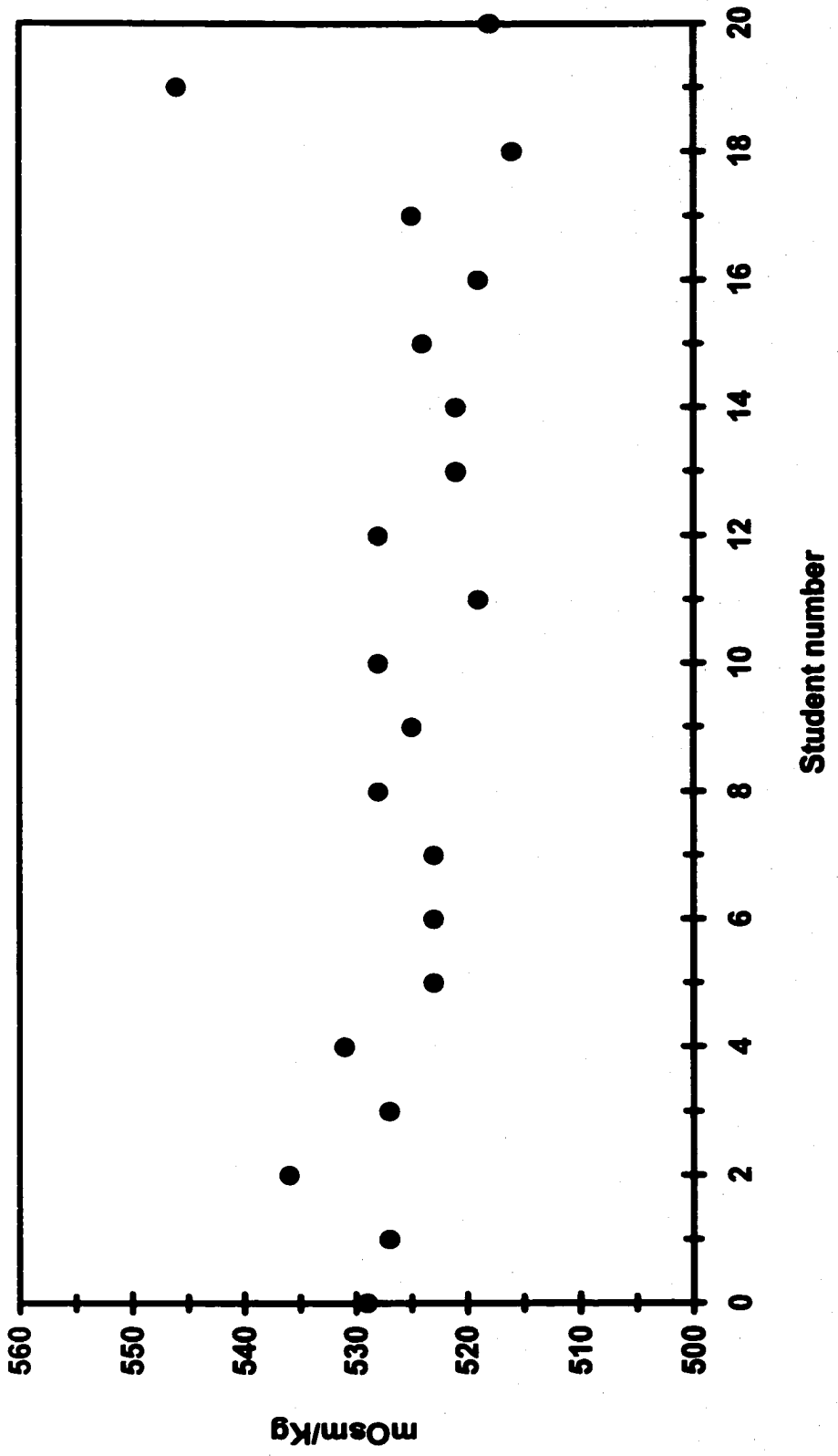
Spectronic 20 Data for 0.0800 M $\text{Ni}(\text{NO}_3)_2$ Solutions, Fall 1998



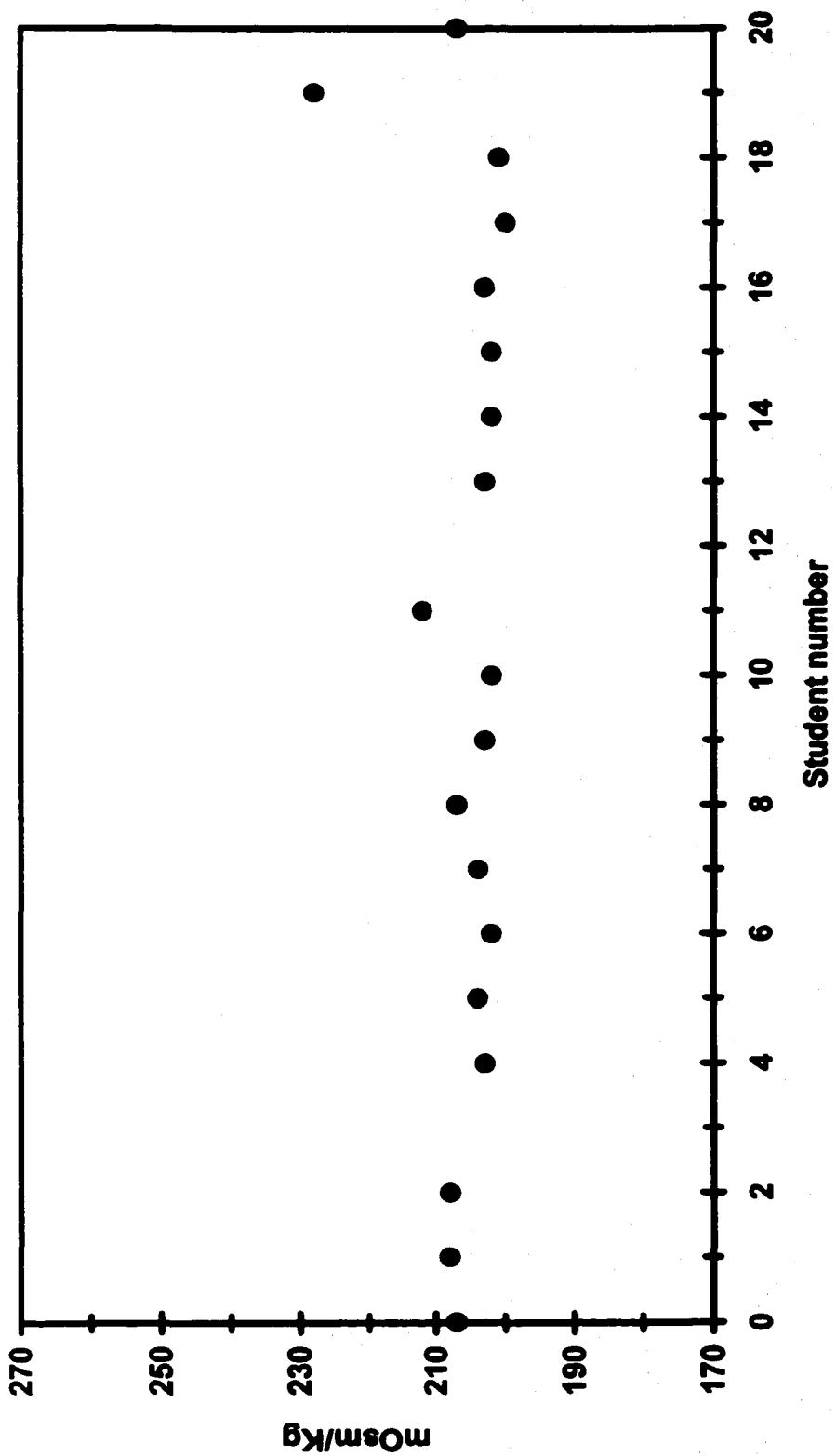
Osmette Reading for 0.100 M Ni(NO₃)₂ Solution, Fall 1998



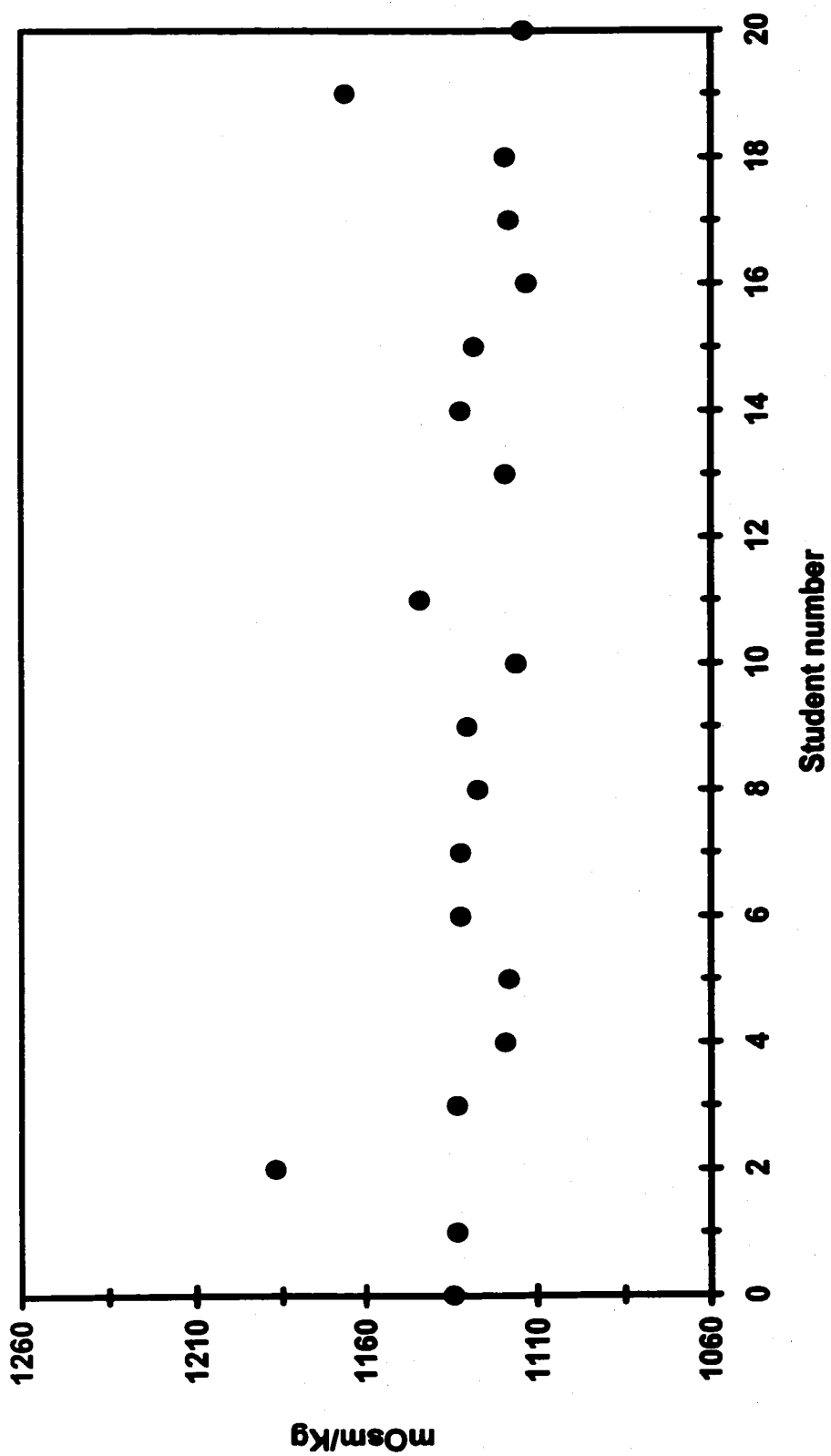
Osmette Reading for 0.050 M Ni(NO₃)₂ Solution, Fall 1998



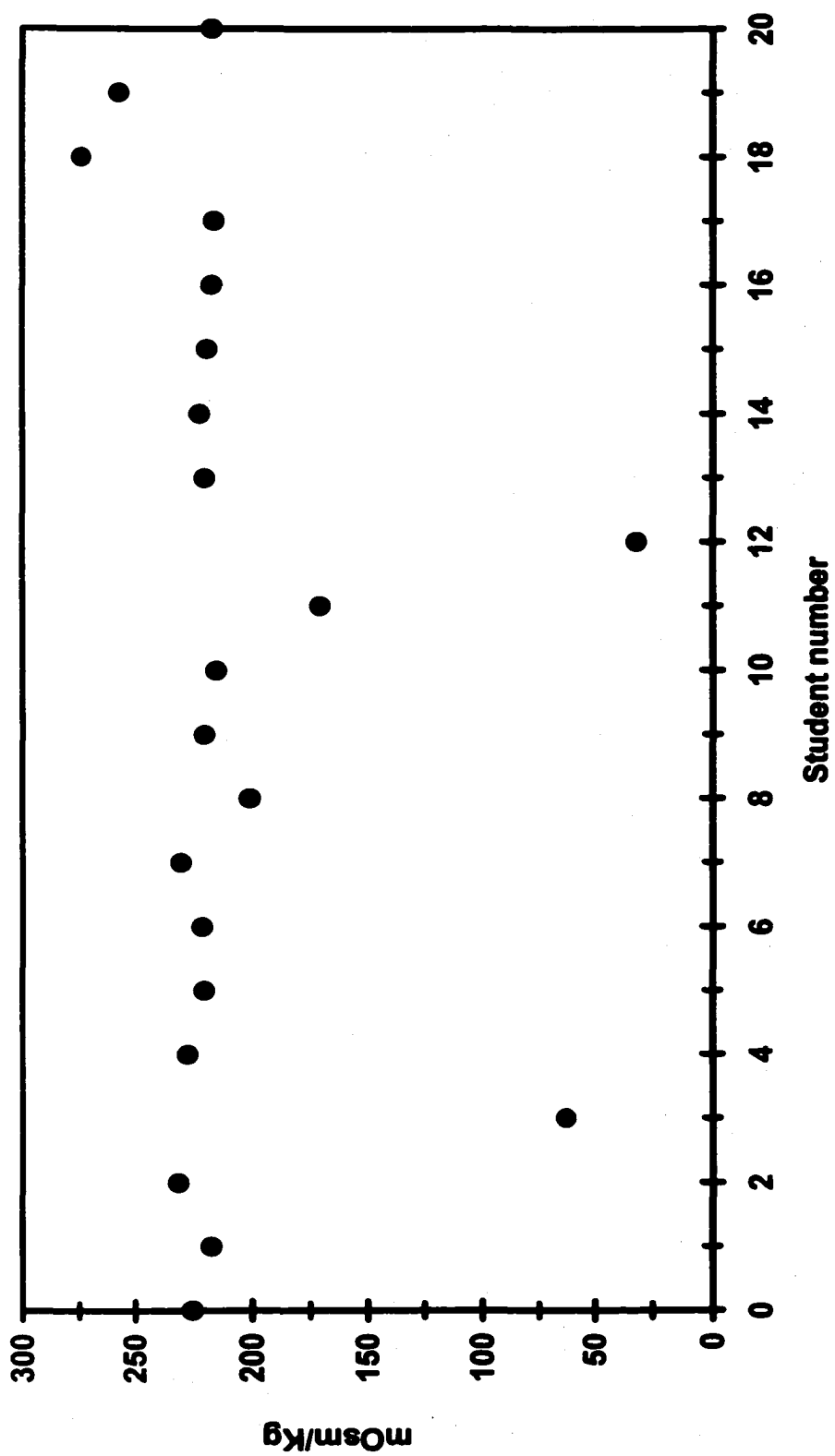
Osmette Reading for 0.200 M Ni(NO₃)₂ Solution, Fall 1998



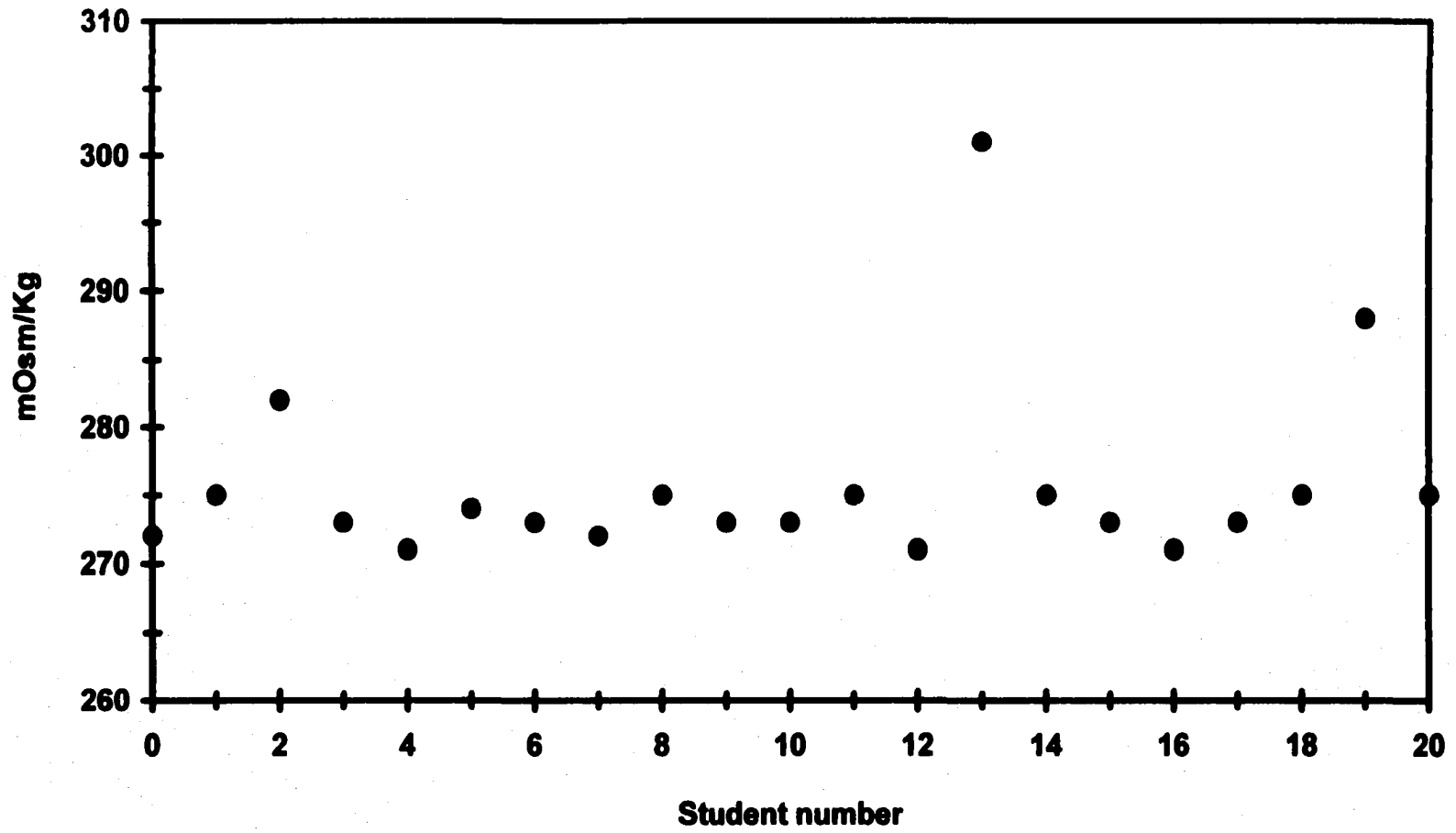
Osmette Reading for 0.080 M Ni(NO₃)₂ Solution, Fall 1998



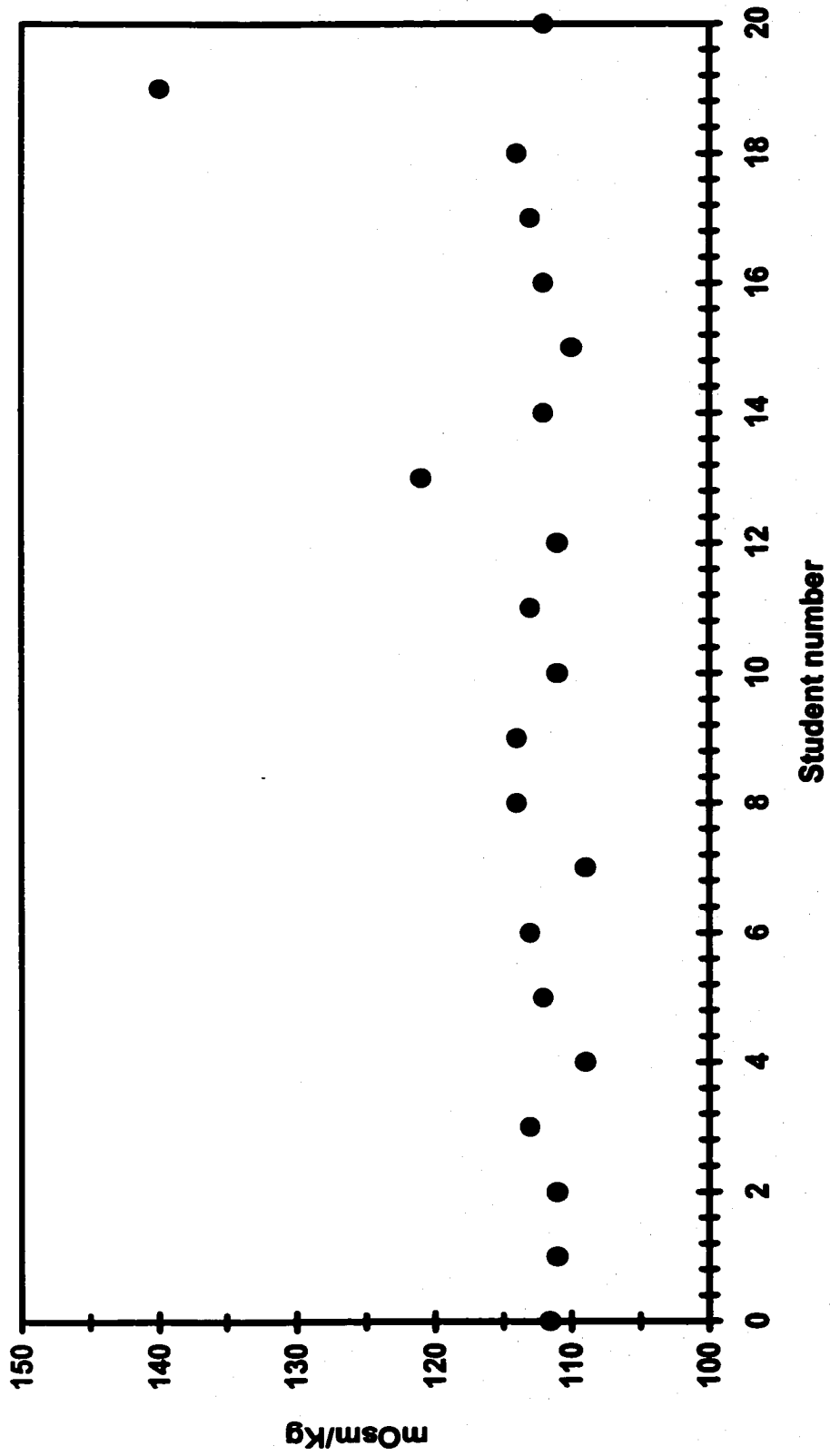
Osmette Reading for 0.600 M KCl Solution--View 2, Fall 1998



Osmette Reading for 0.120 M KCl Solution, Fall 1998



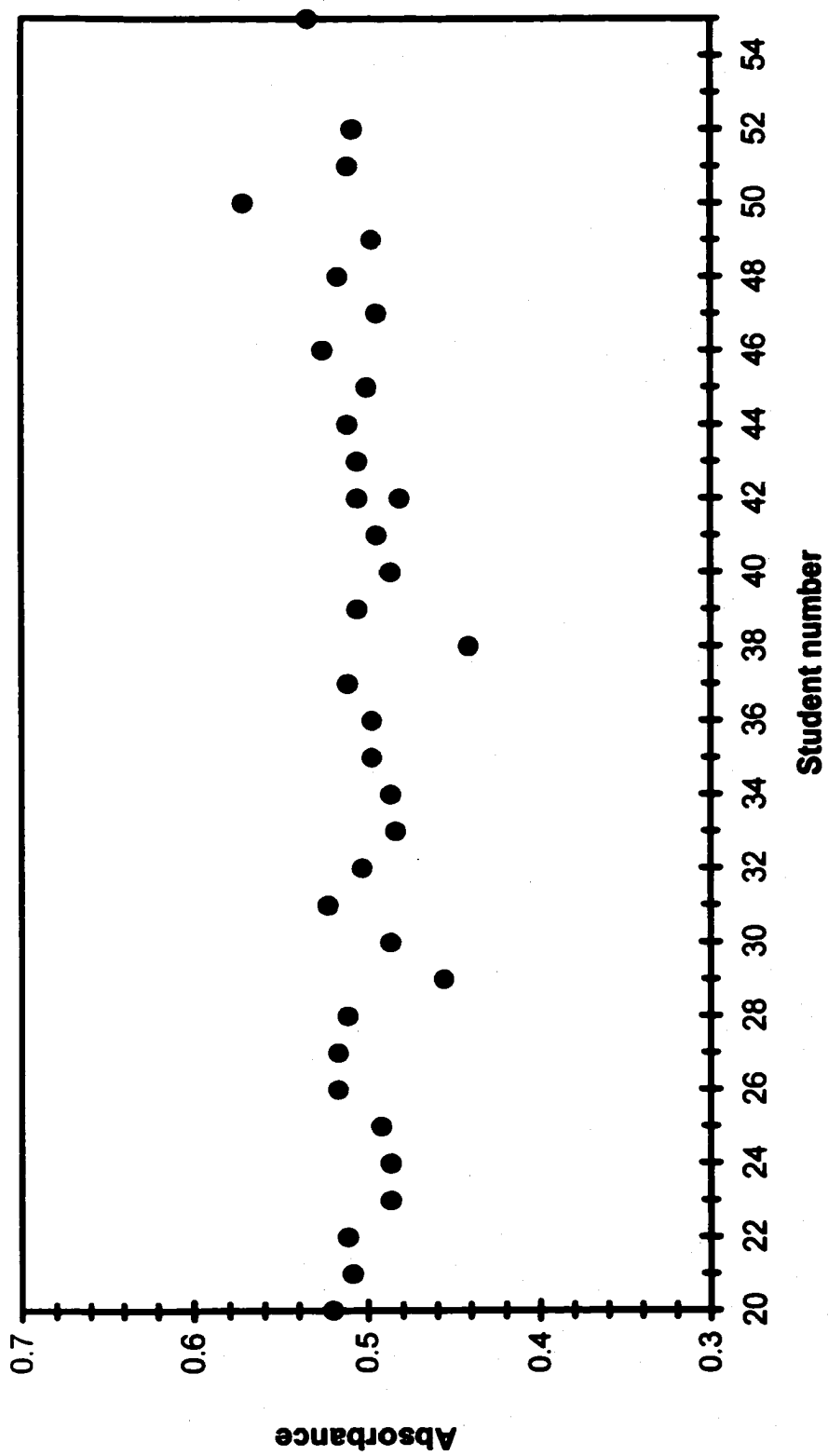
Osmette Reading for 0.150 M KCl Solution, Fall 1998



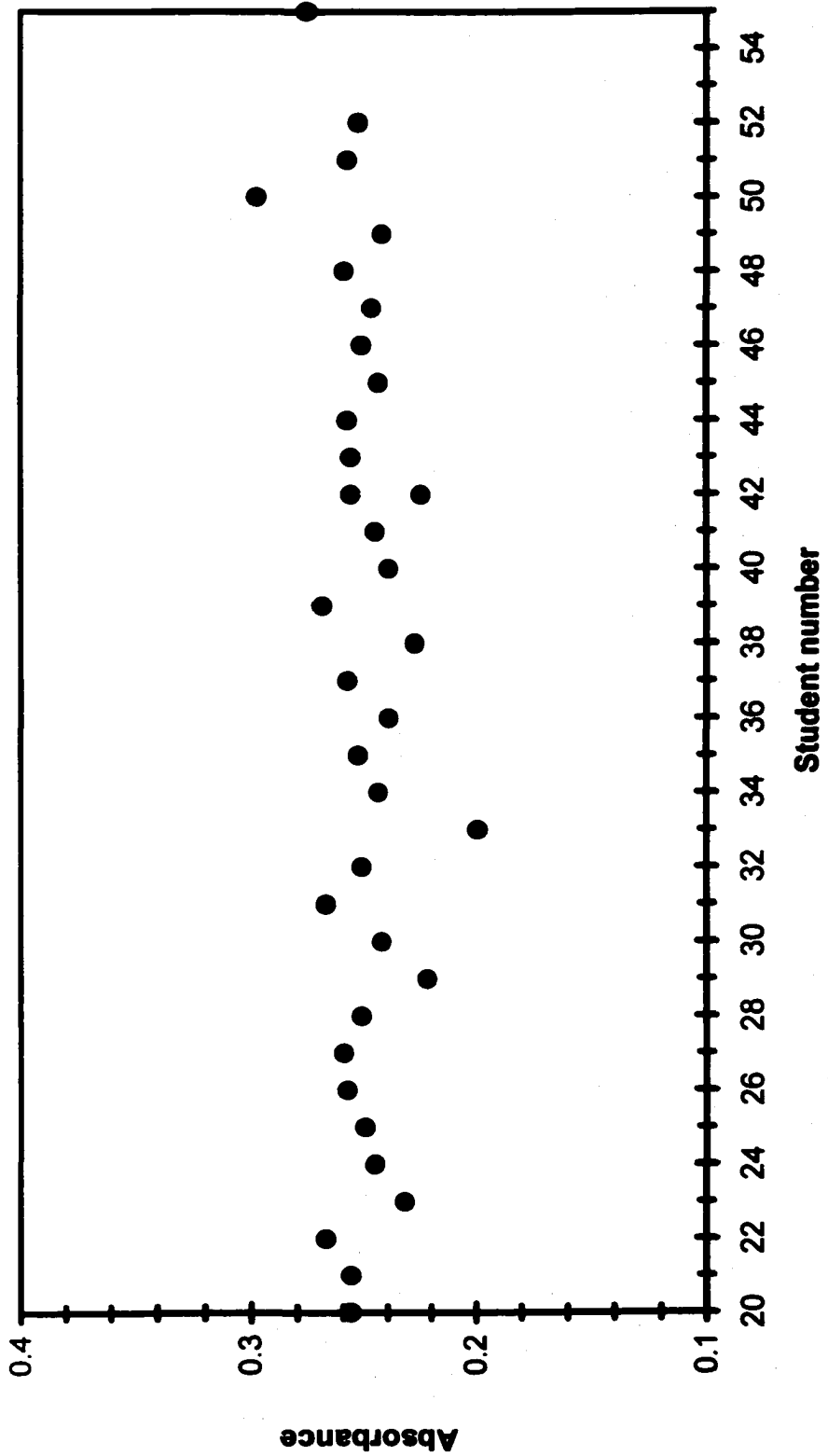
Osmette Reading for 0.0600 M KCl, Fall 1998

Appendix B2

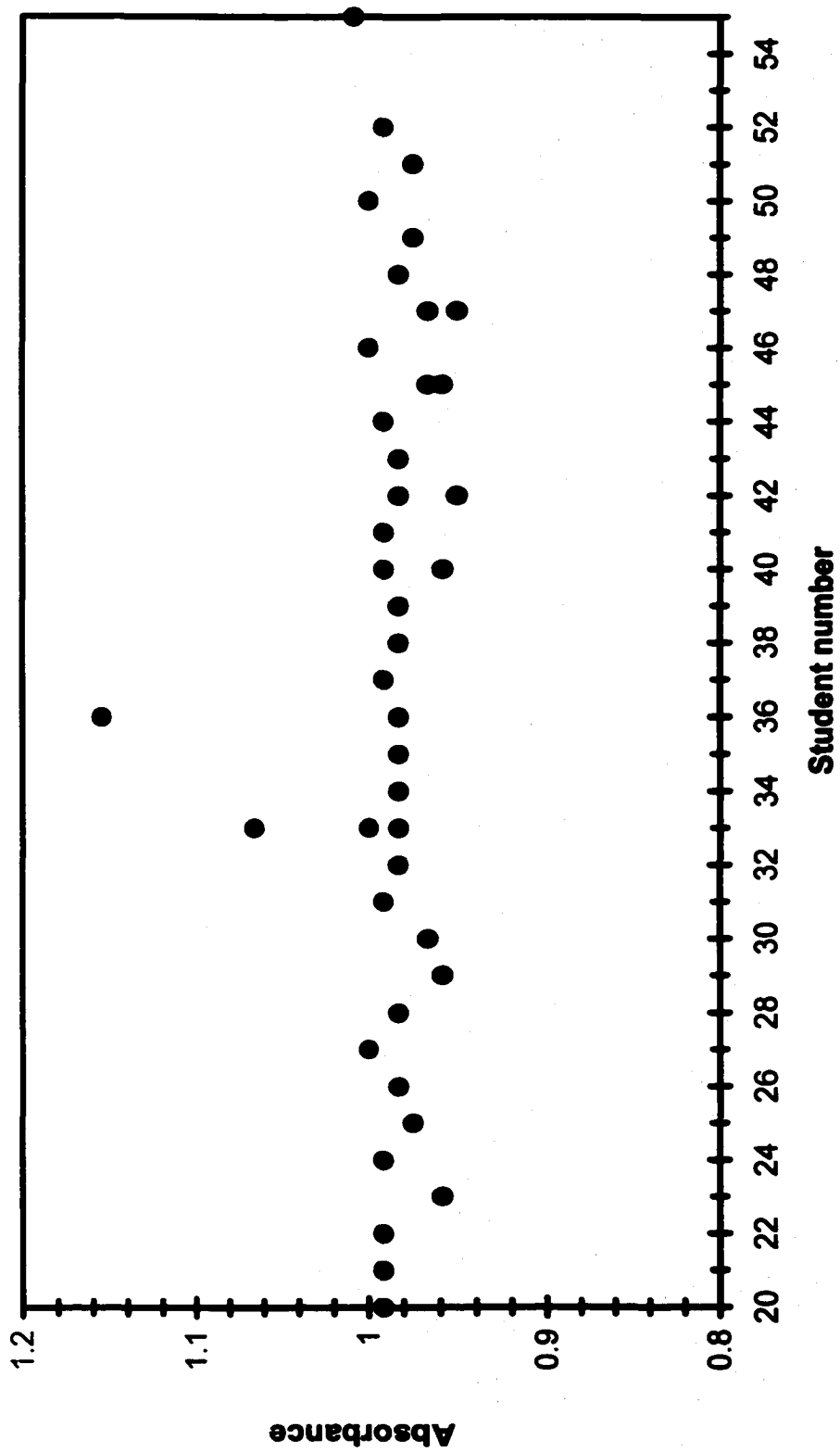
Instrumental Reading Graphs for Spring Semester, 1999

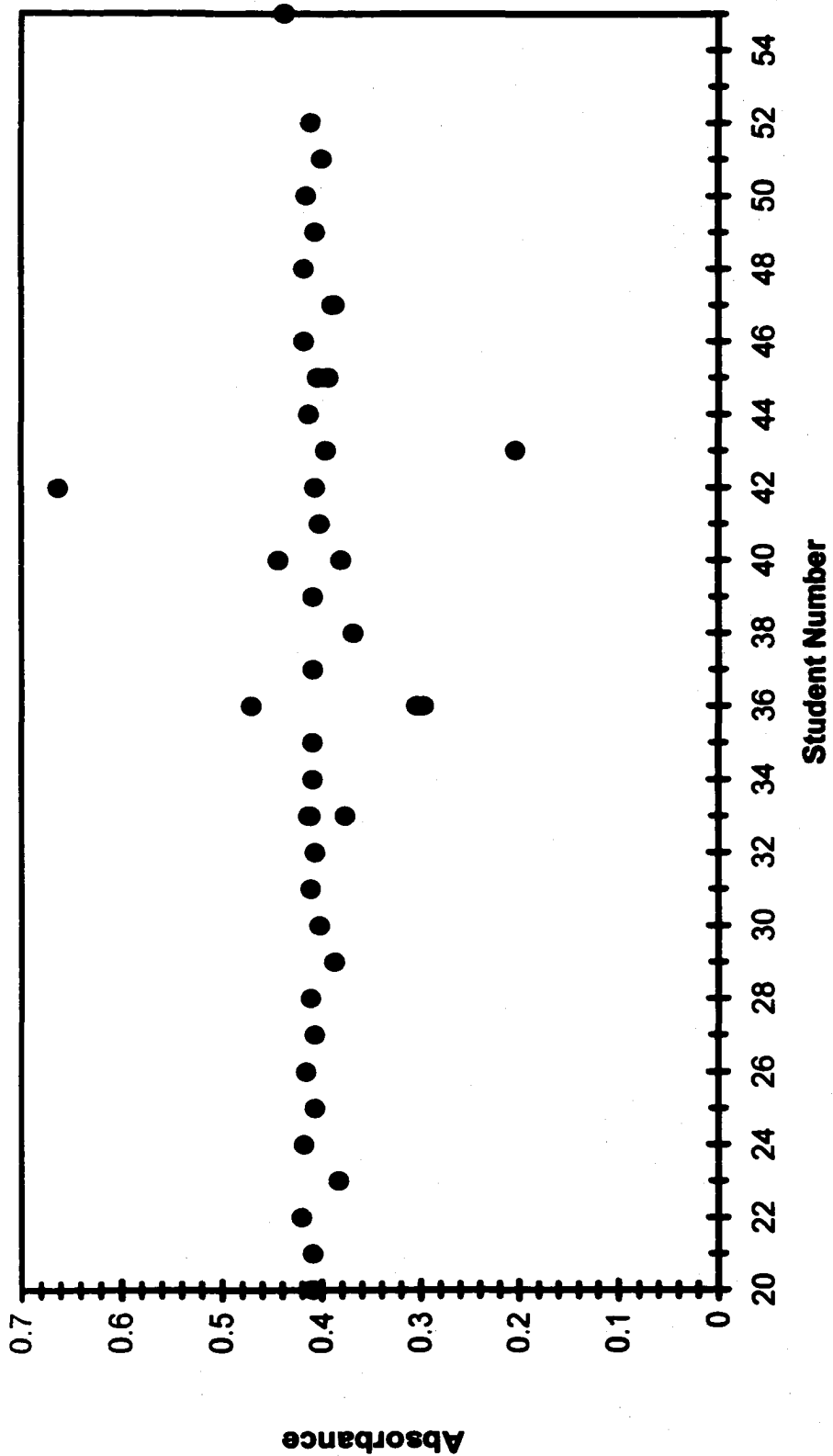


Spectronic 20 Data for 0.100 M Ni(NO₃)₂, Spring 1999

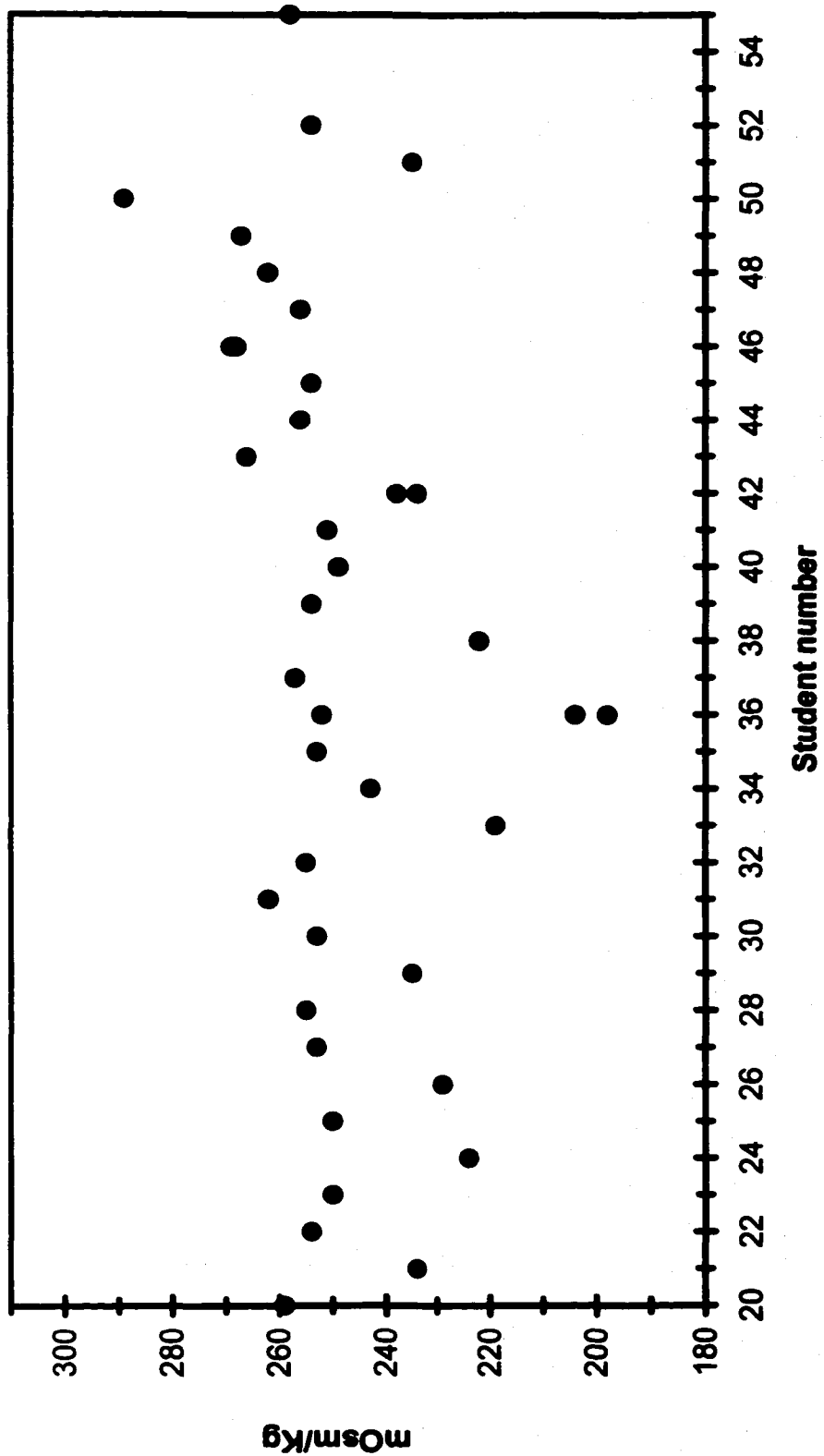


Spectronic 20 Data for 0.0500 M Ni(NO₃)₂, Spring 1999

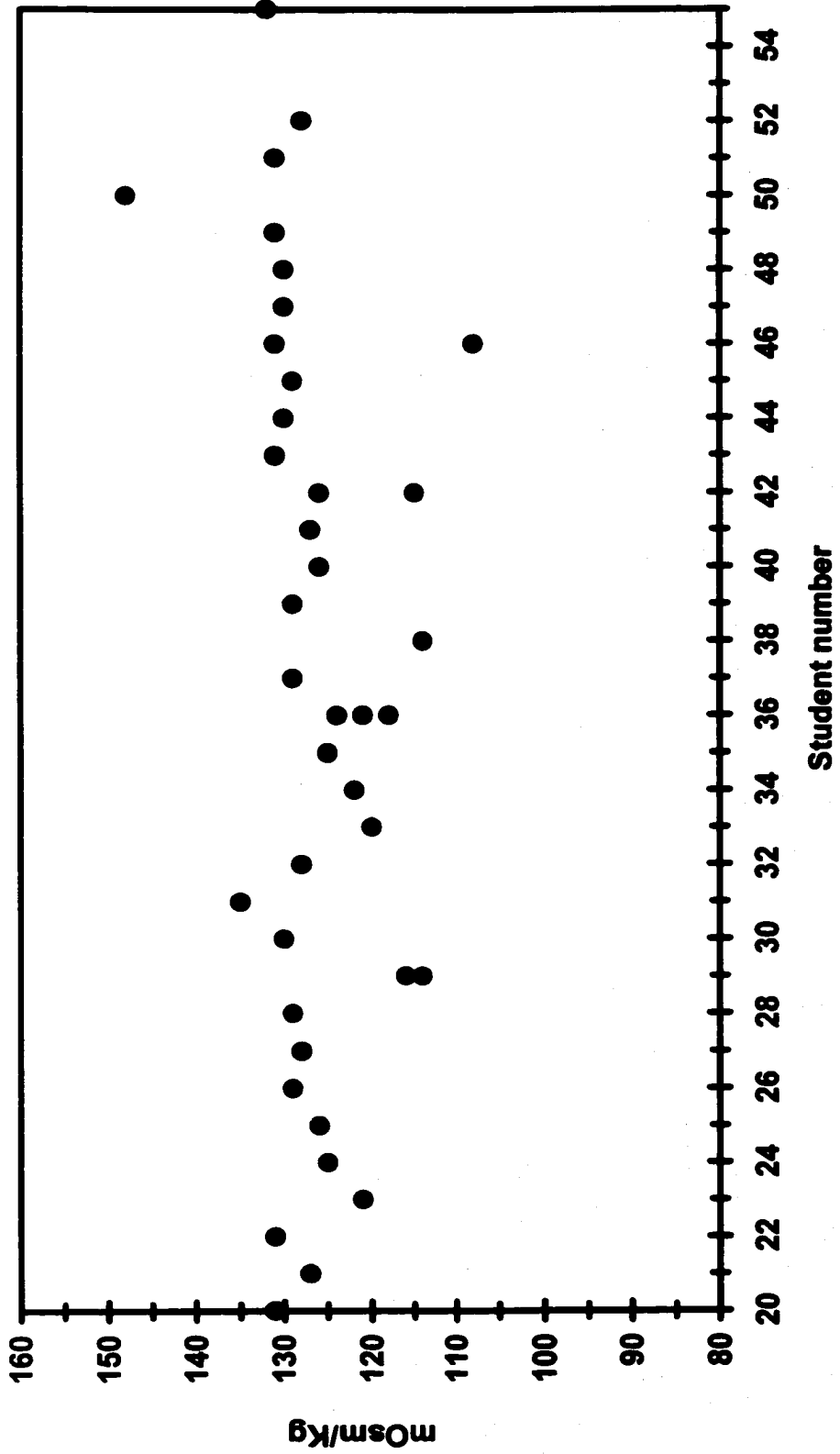
Spectronic 20 Data for 0.200 M Ni(NO₃)₂, Spring 1999



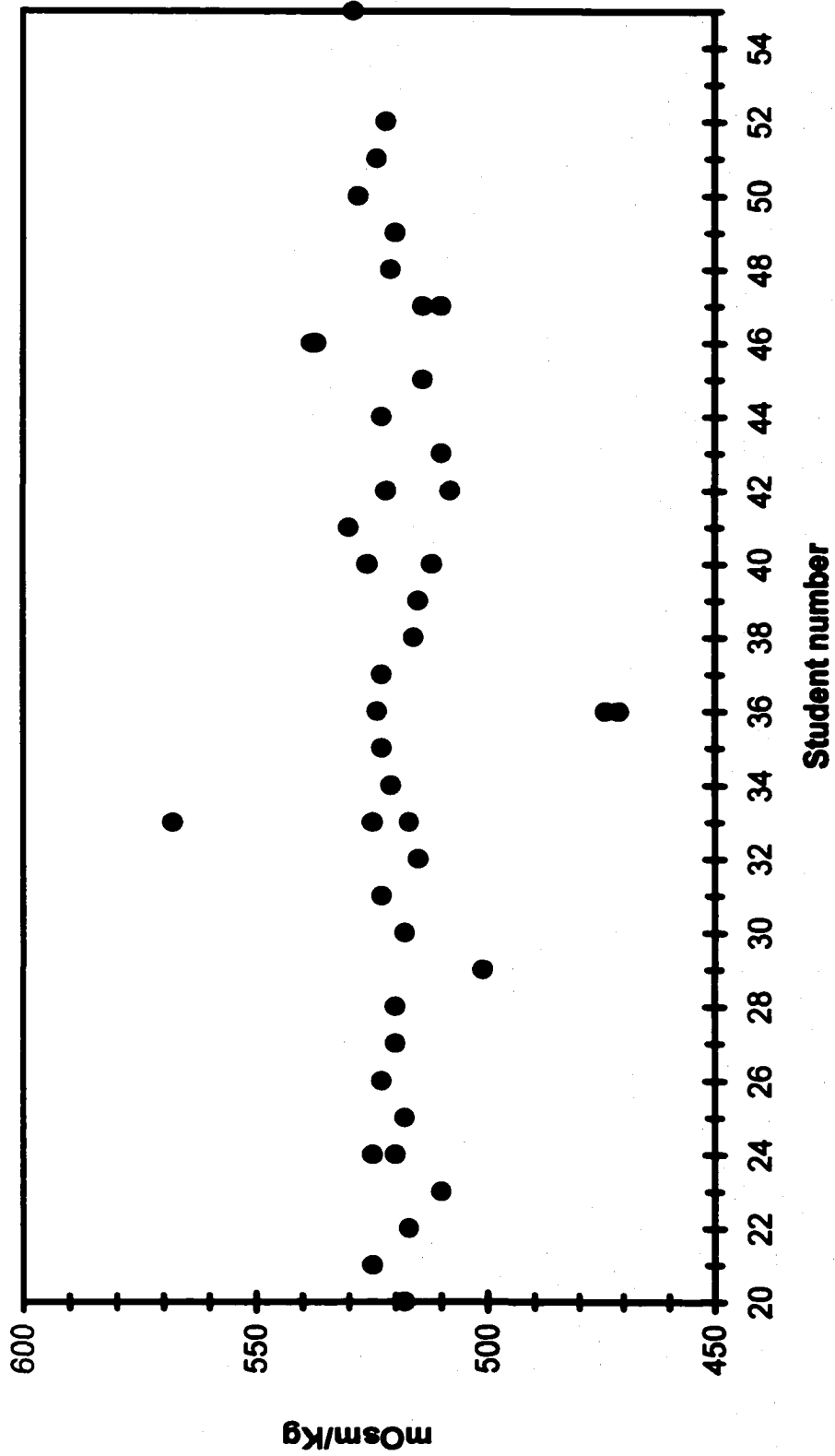
Spectronic 20 Data for 0.0800 M Ni(NO₃)₂, Spring 1999



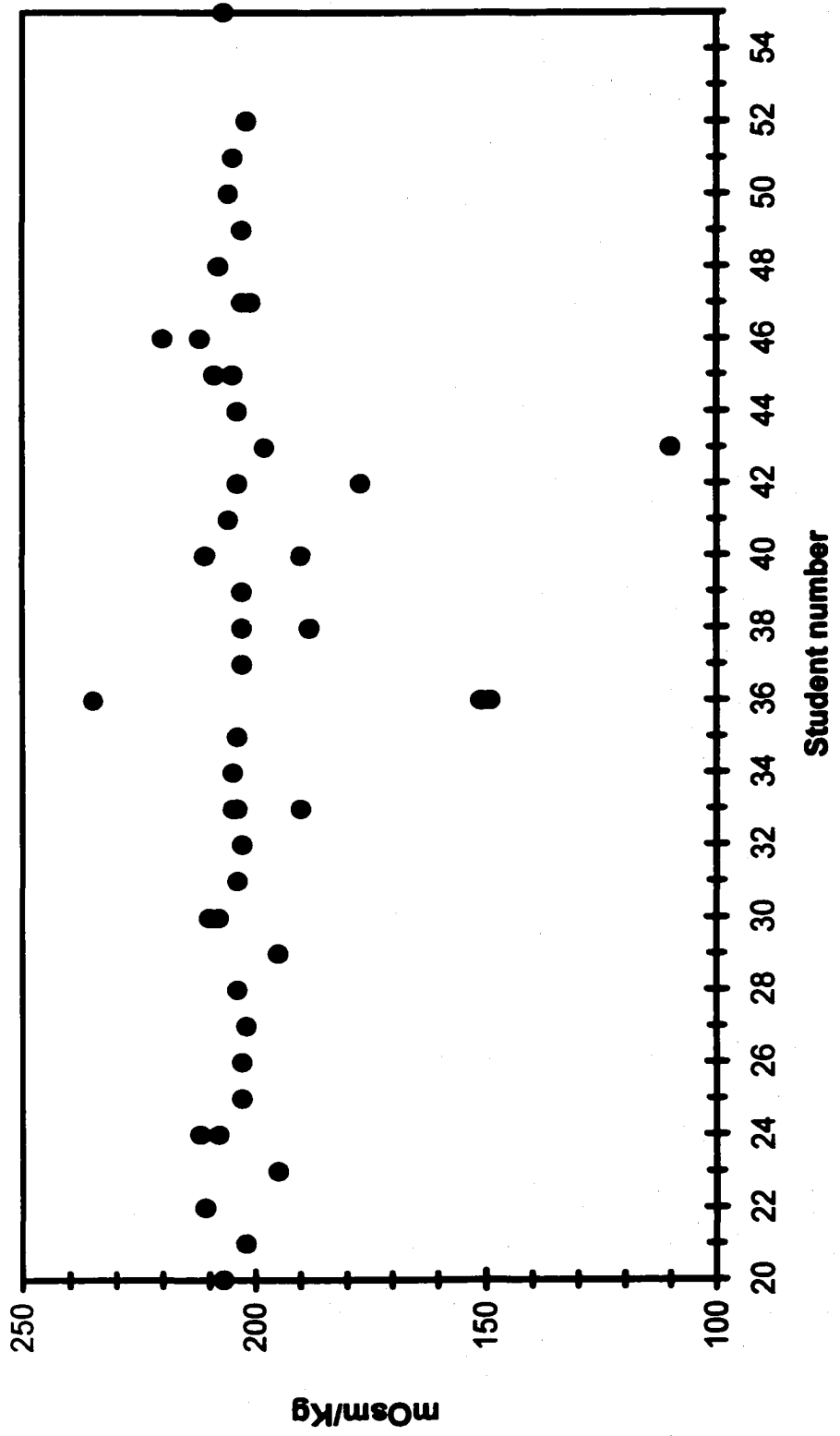
Osmometer Readings for 0.100 M Ni(NO₃)₂, Spring, 1999



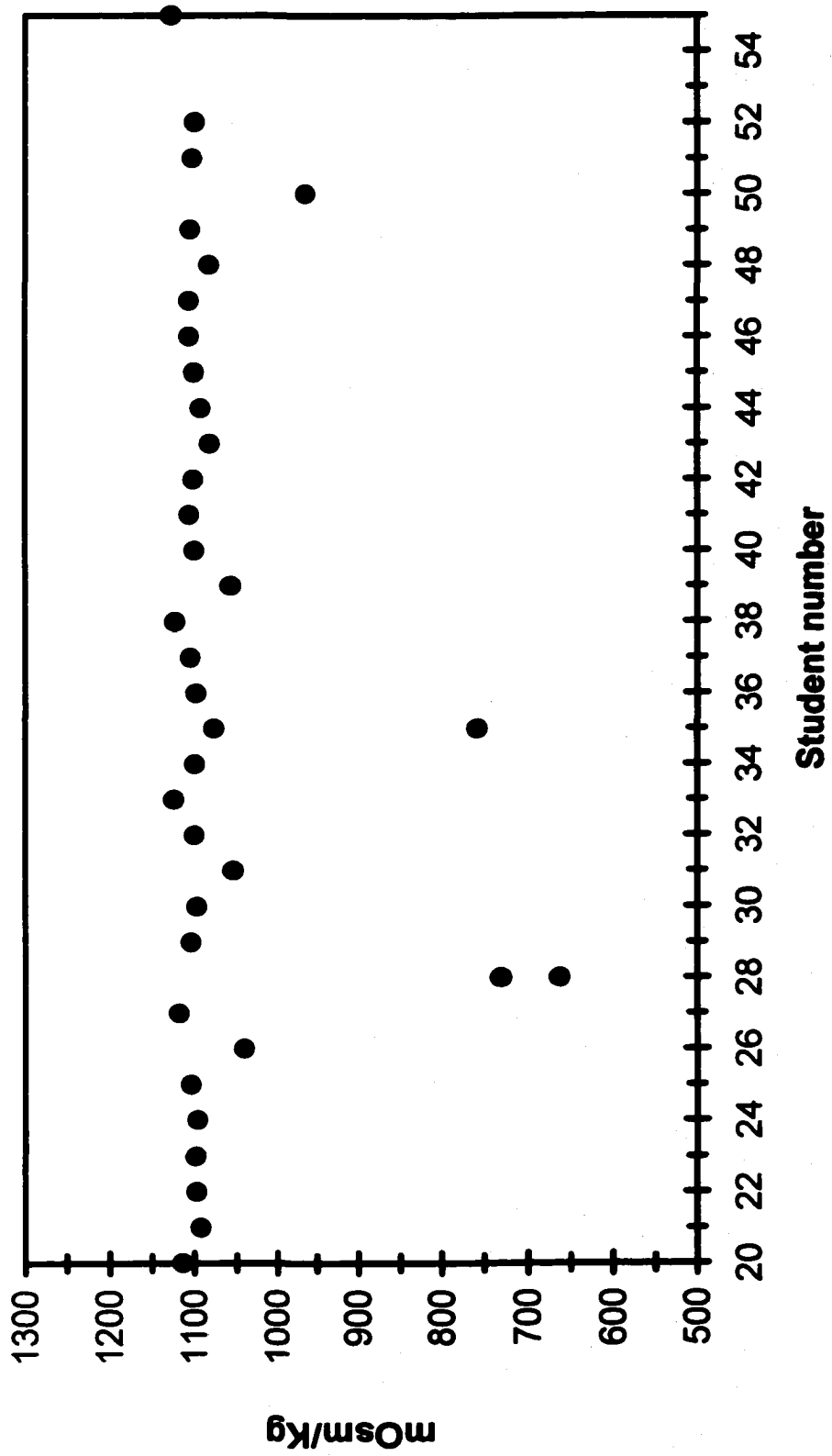
Osmette Readings for 0.0500 M Ni(NO₃)₂, Spring 1999



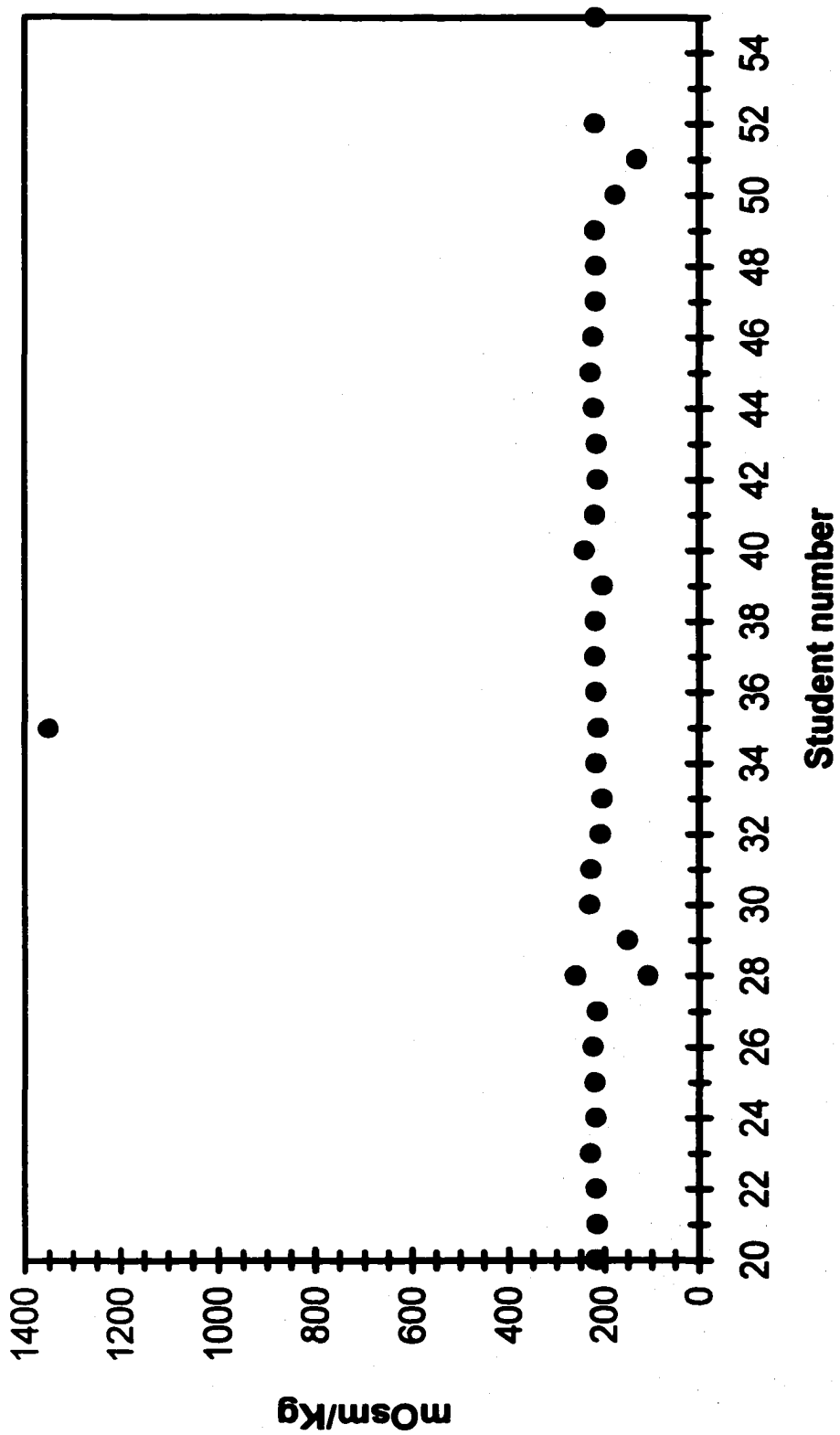
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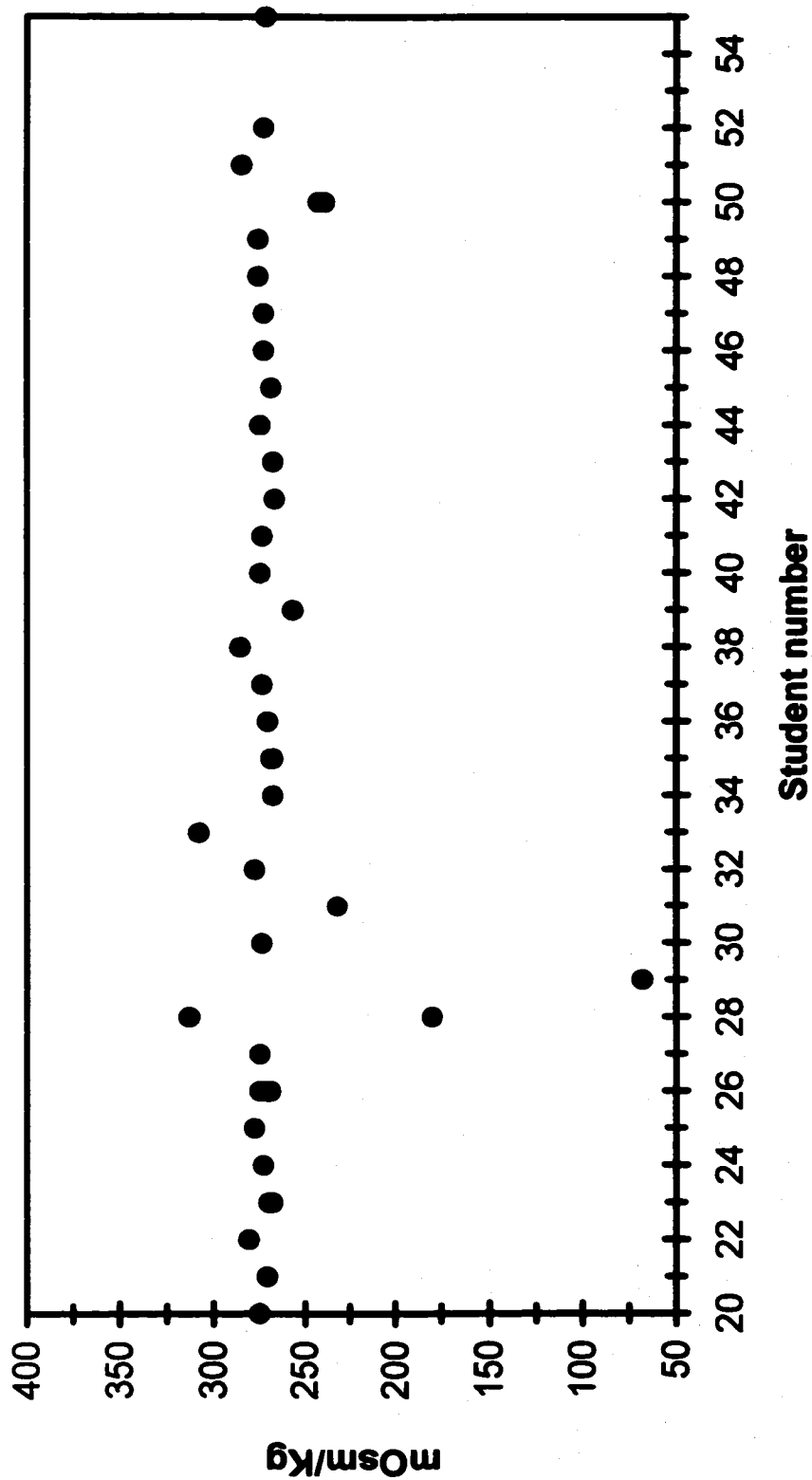
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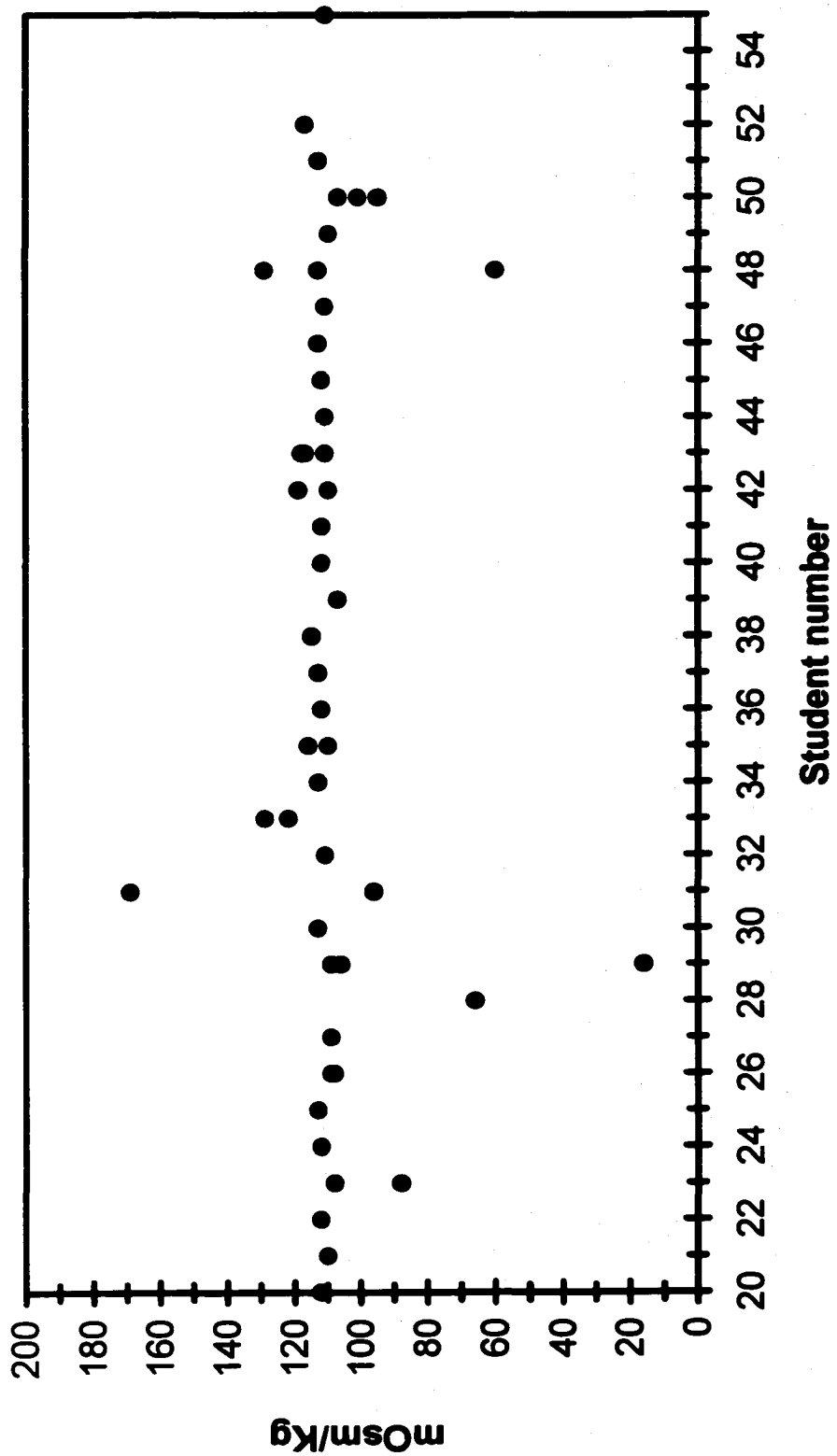
Osmette Readings for 0.600 KCl, Spring, 1999



Osmometer Readings for 0.120 M KCl, Spring, 1999



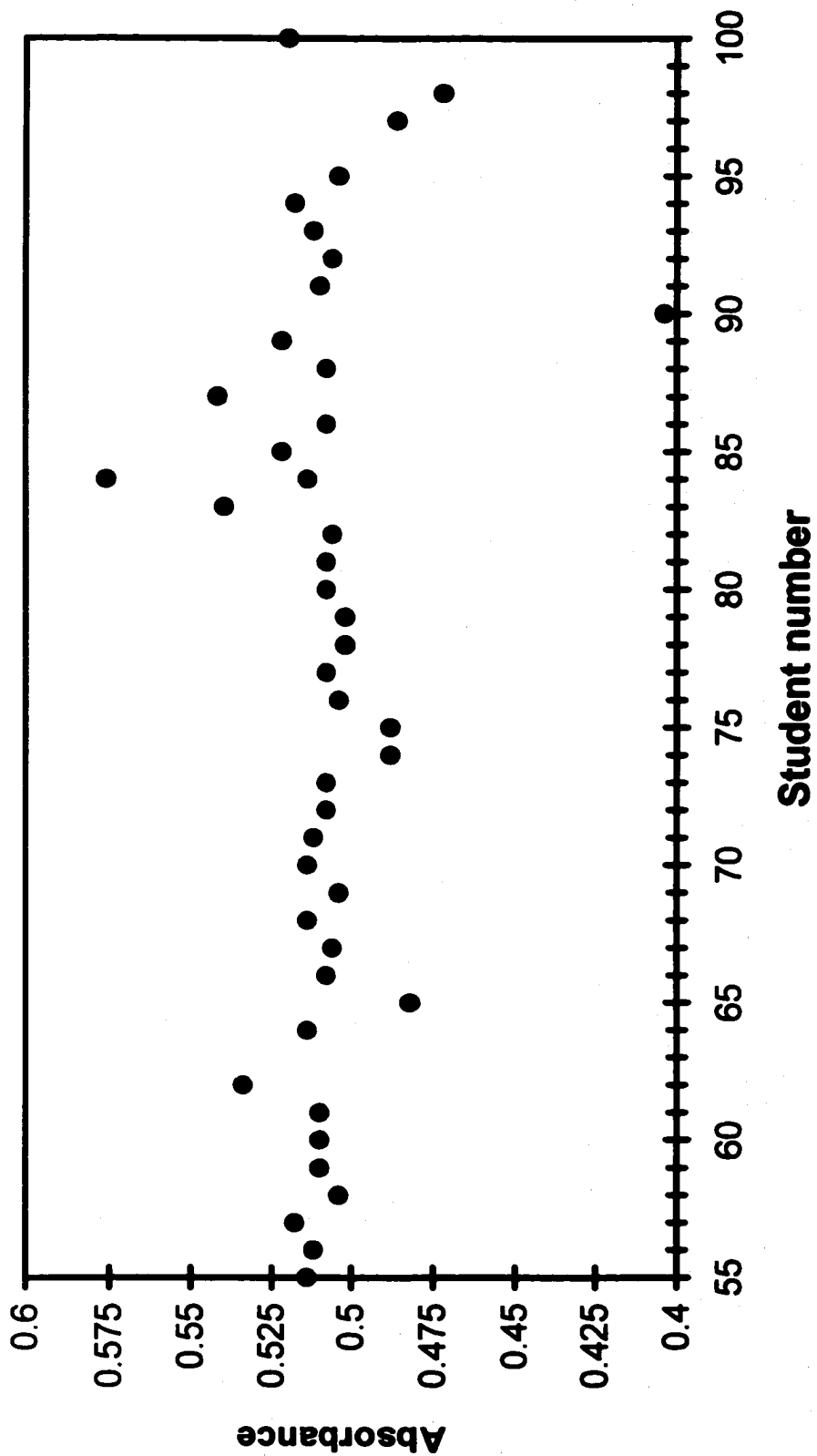
Osmometer Readings for 0.150 M KCl, Spring, 1999

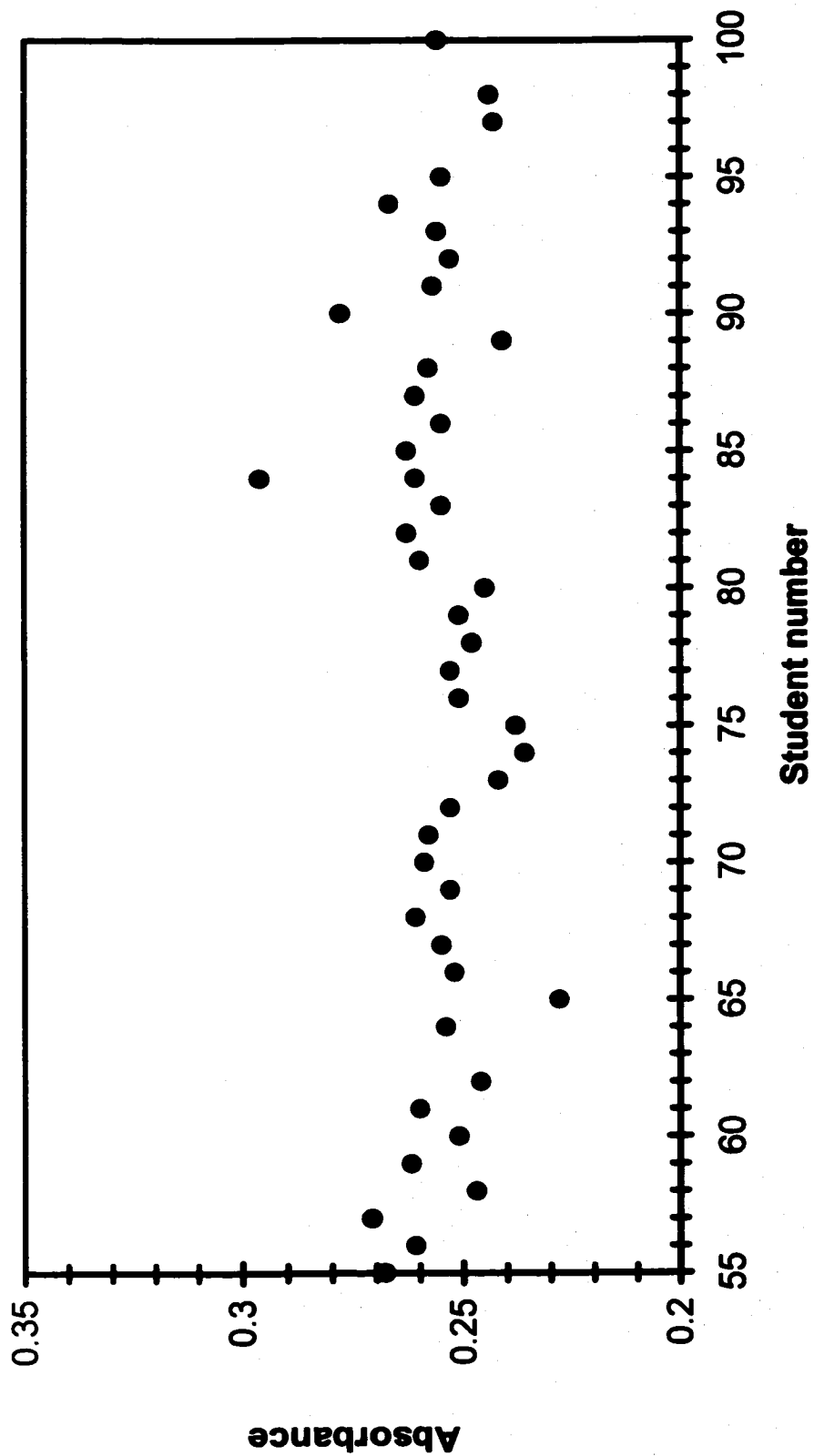


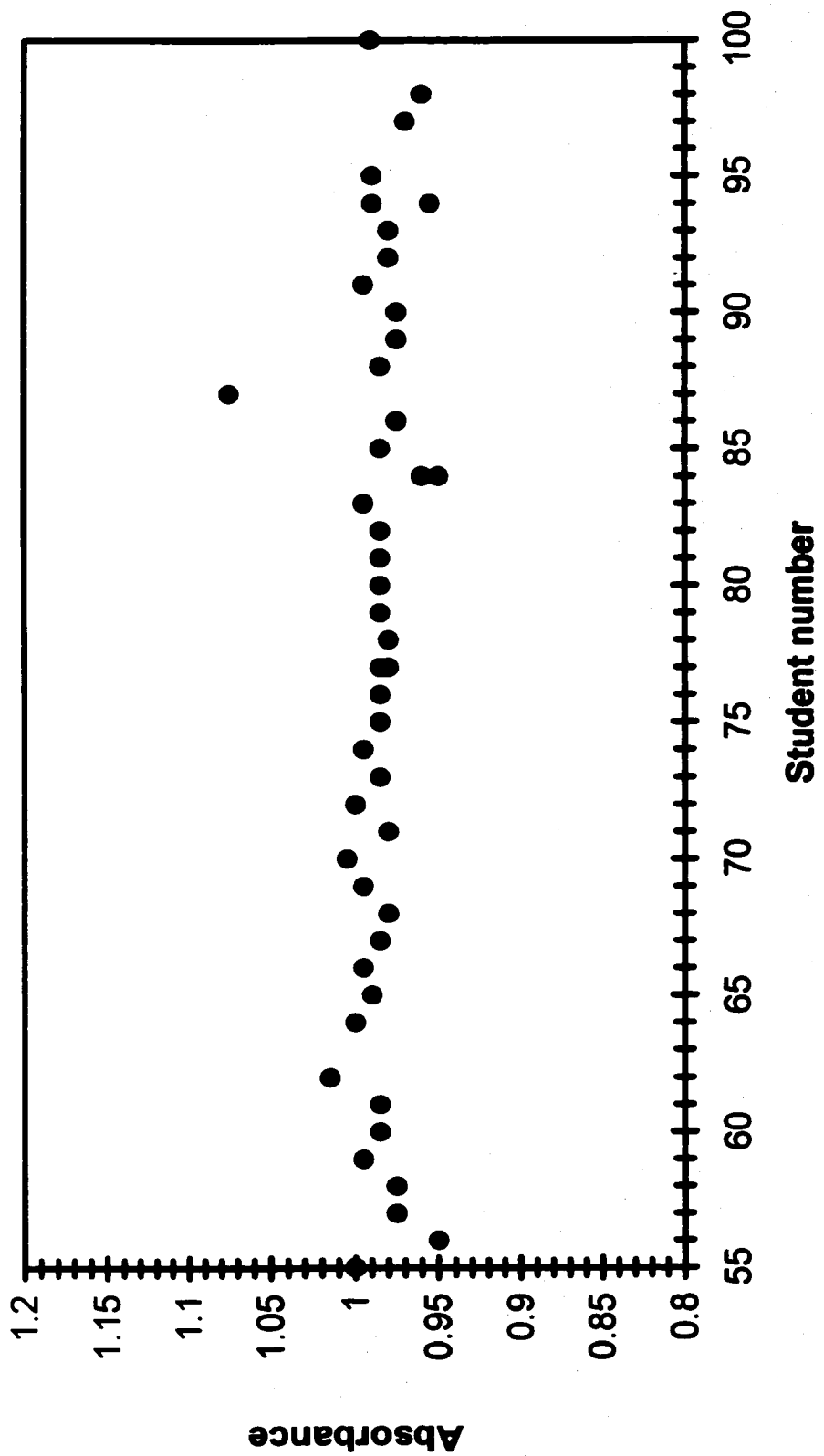
Osmometer Readings for 0.0600 M KCl, View 2, Spring, 1999

Appendix B3

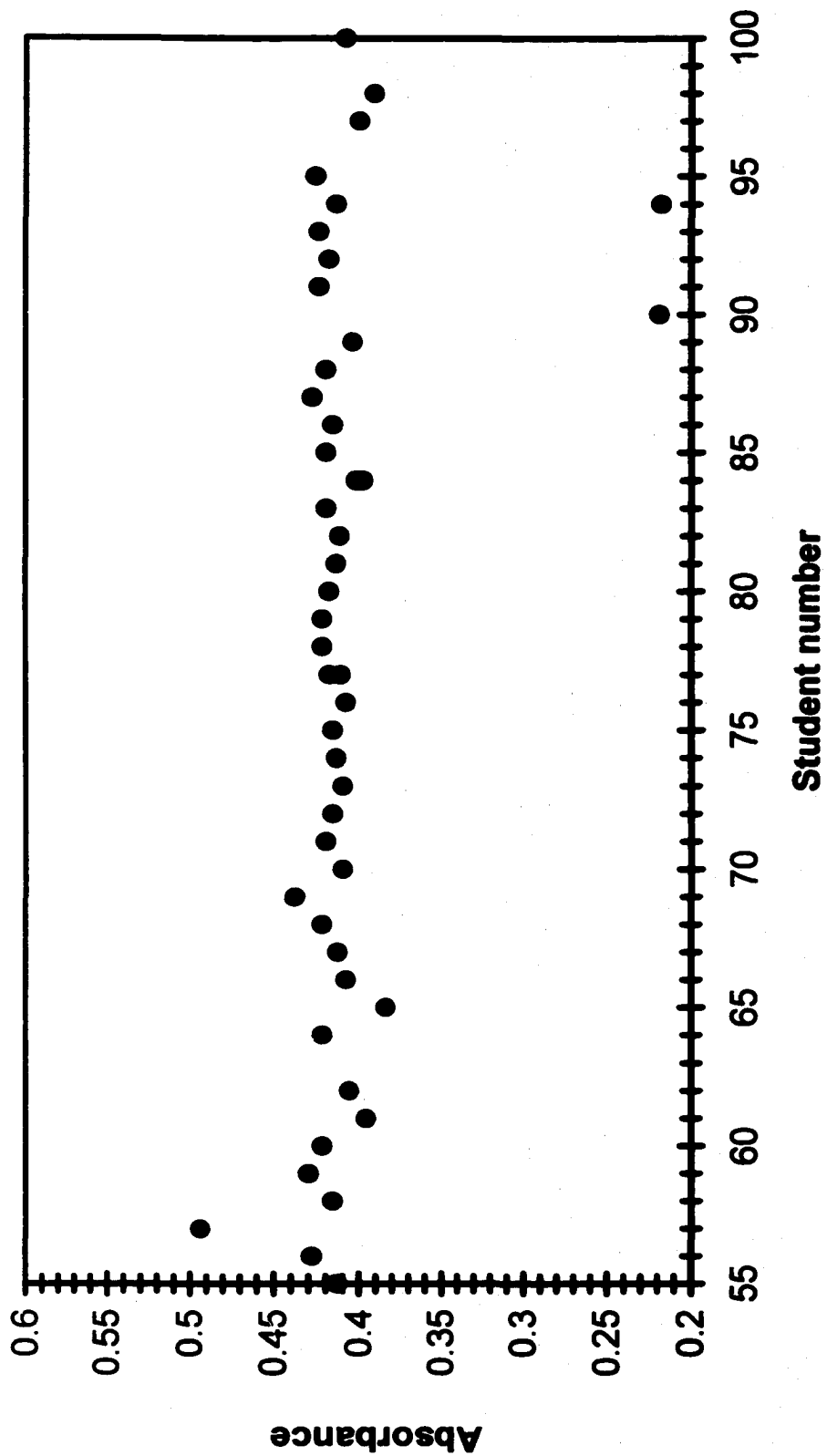
Instrumental Reading Graphs for Fall Semester, 1999

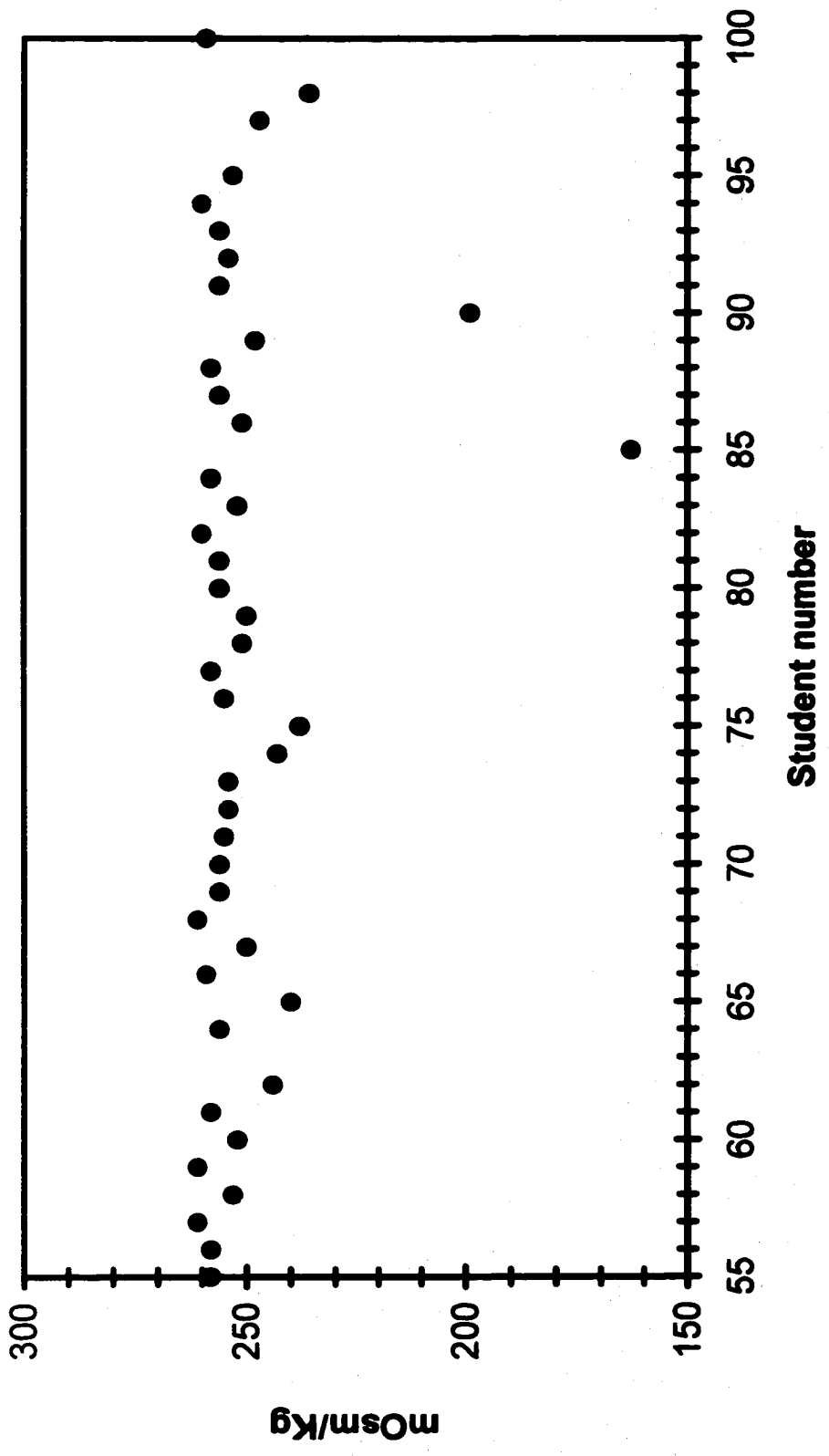
Spectronic 20 Data for 0.100M Ni(NO₃)₂ Solutions, Fall, 1999, View 2

Spectronic 20 Data for 0.0500M Ni(NO₃)₂ Solutions, Fall 1999, View 2

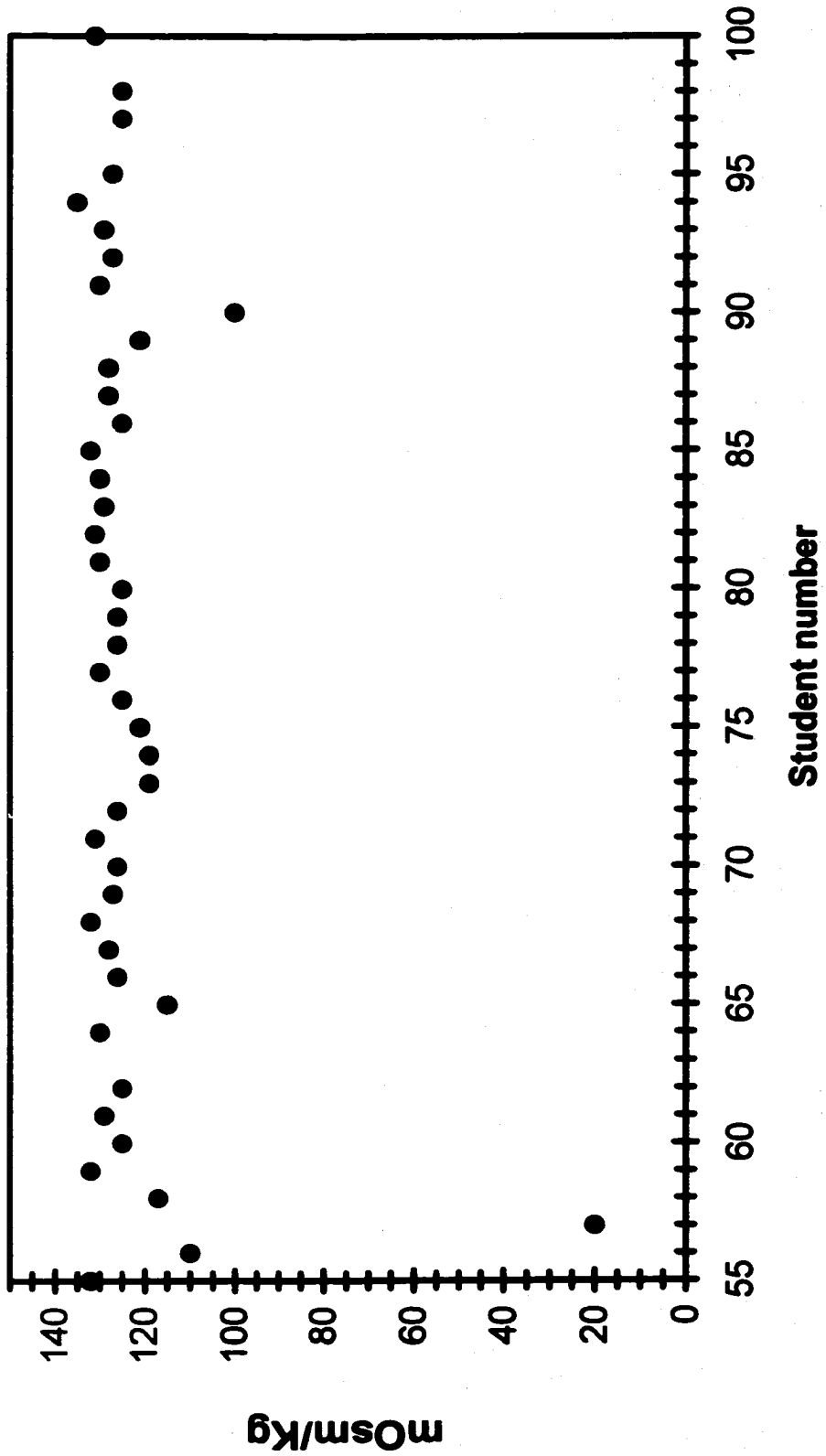


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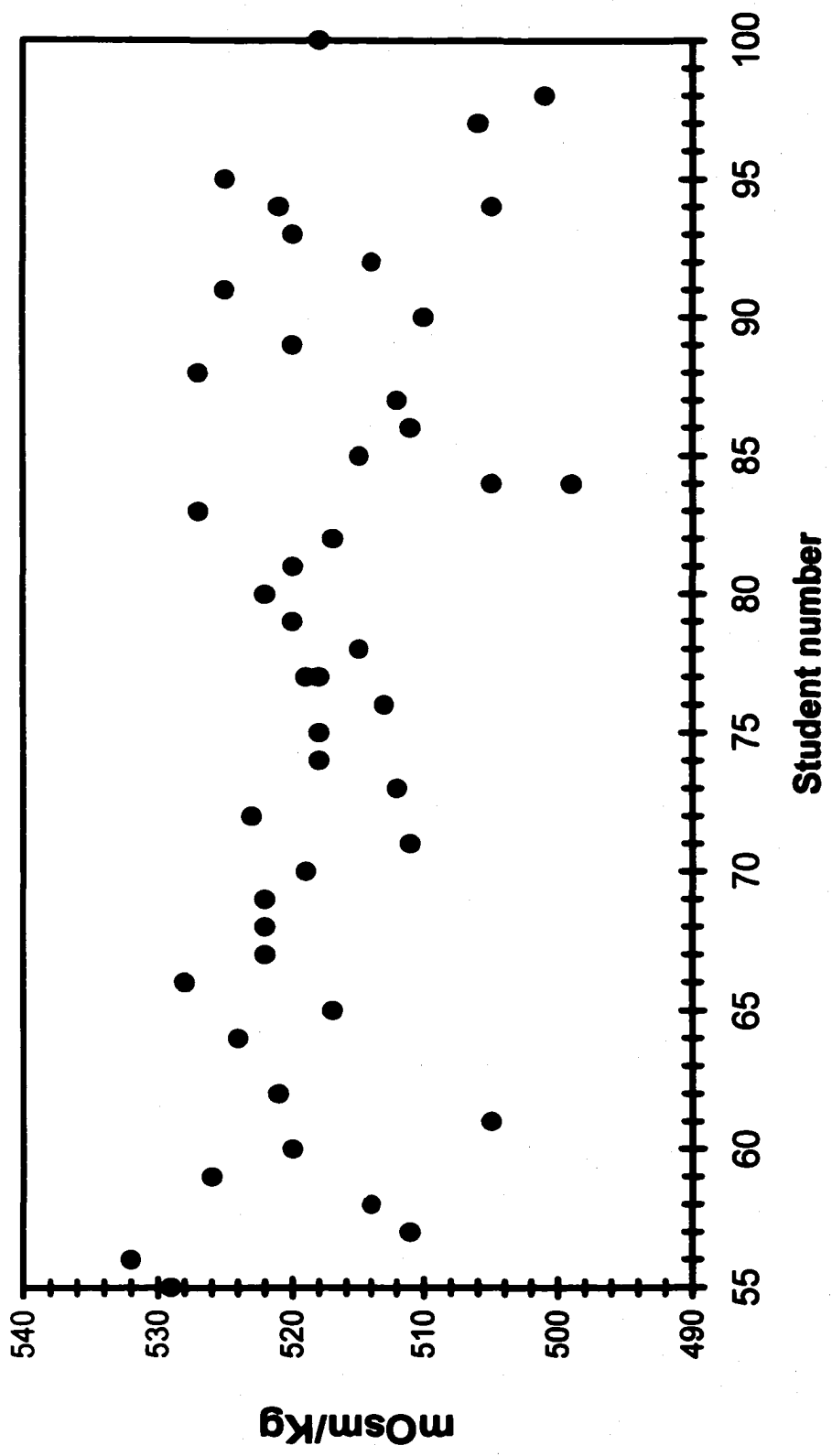
Spectronic 20 Data for 0.080M Ni(NO₃)₂ Solutions, Fall 1999, View 2



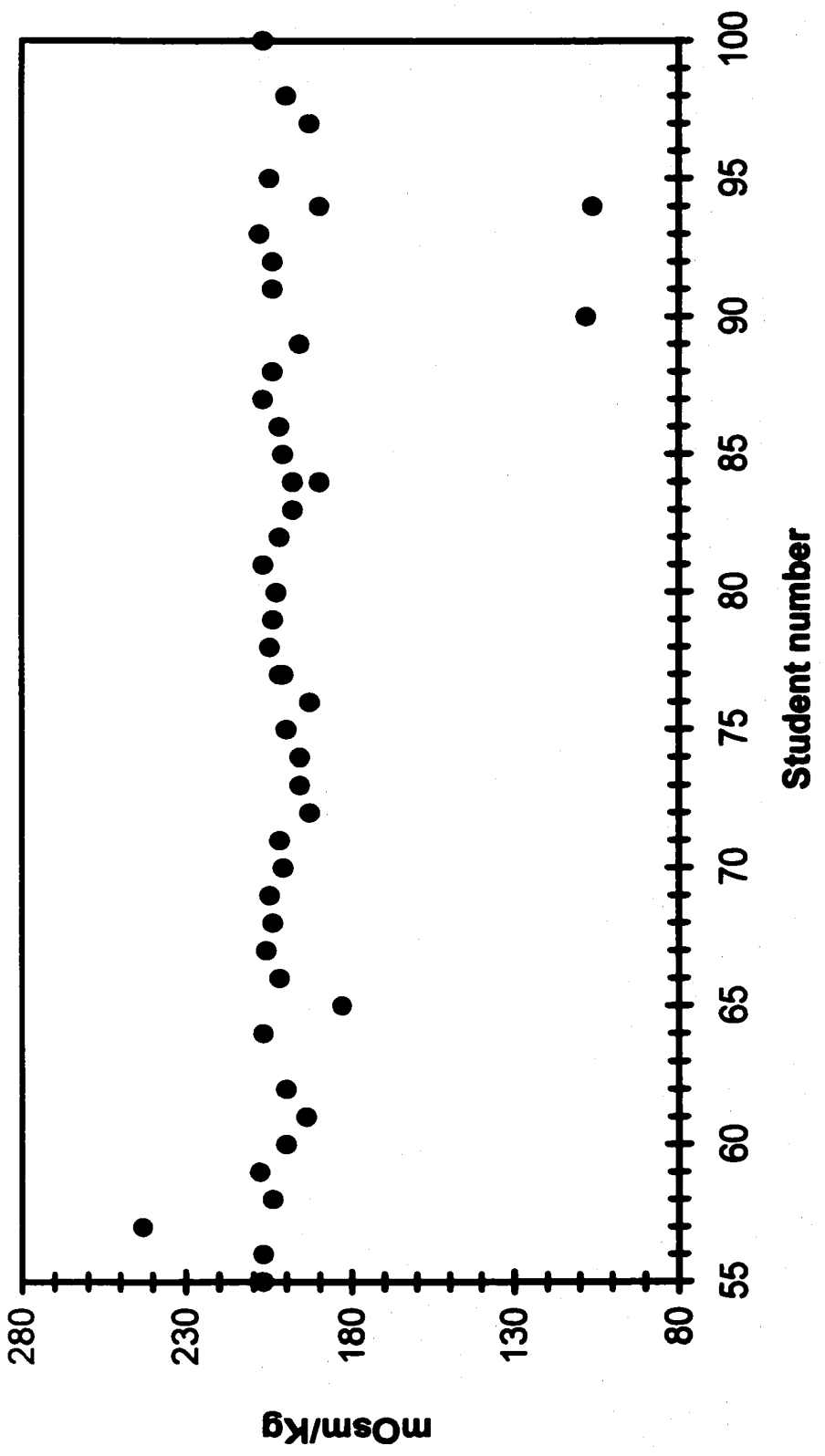
Osmometer Readings for 0.100 M Ni(NO₃)₂, Fall, 1999



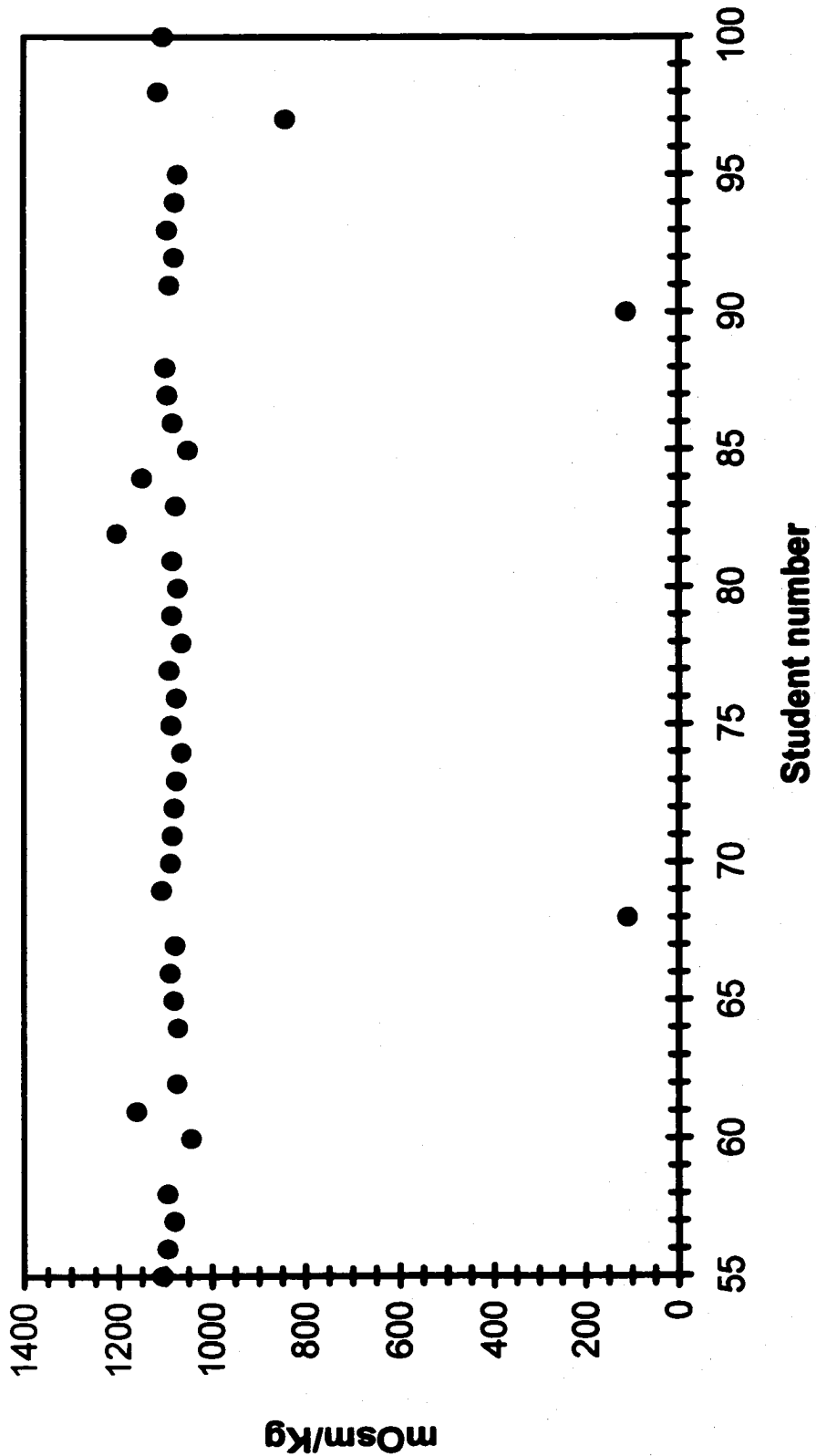
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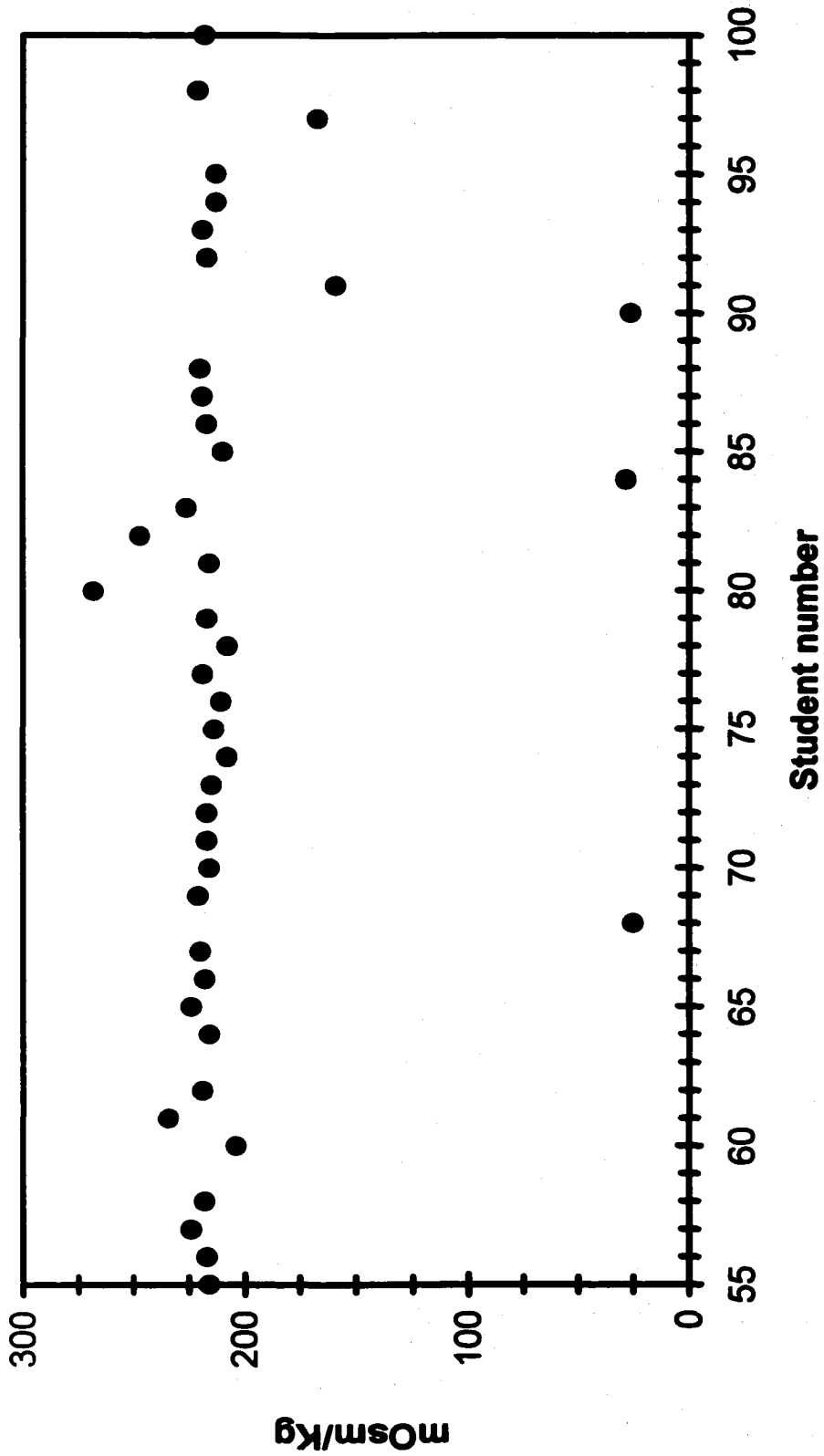
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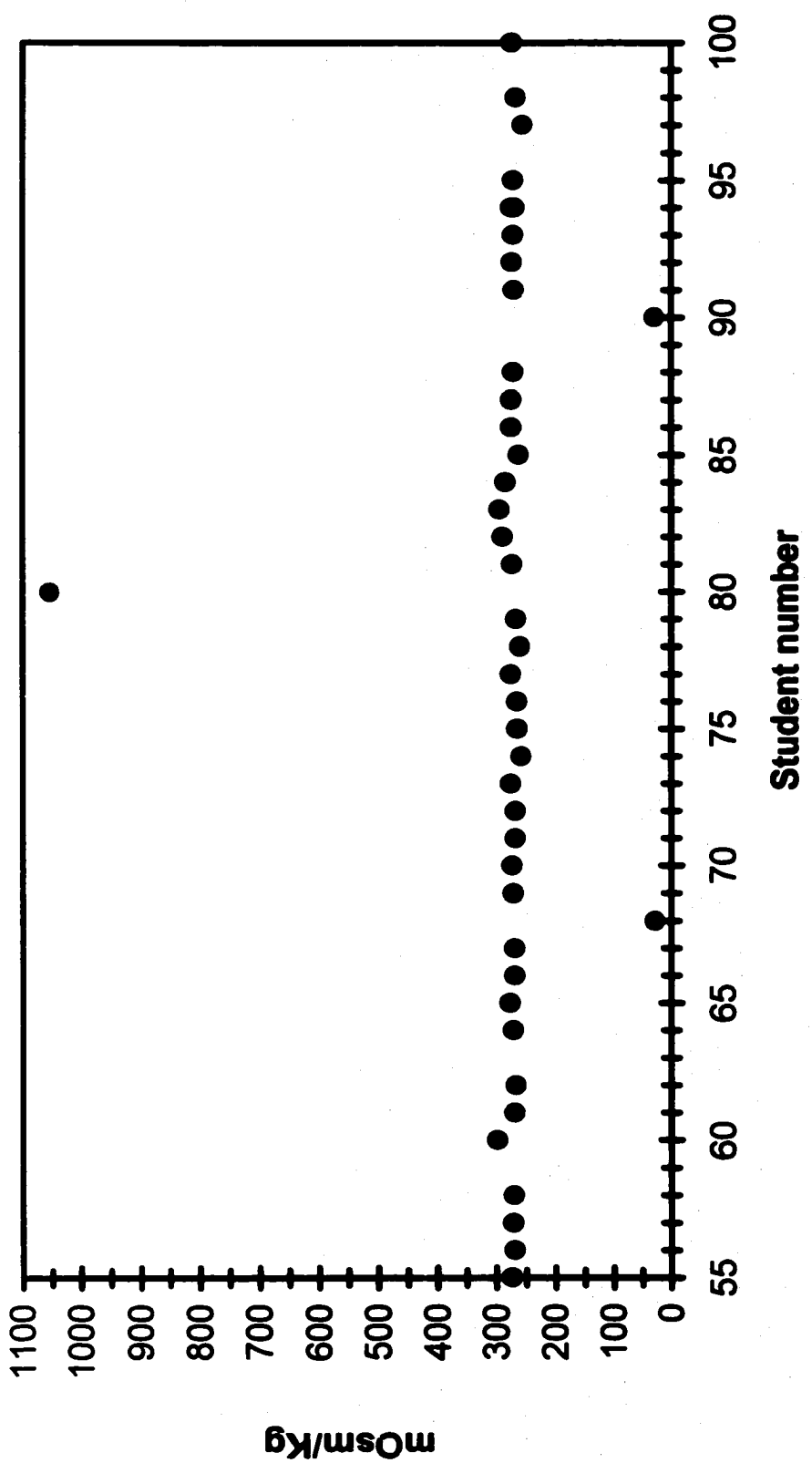
Osmometer Readings for 0.0800 M Ni(NO₃)₂, Fall, 1999



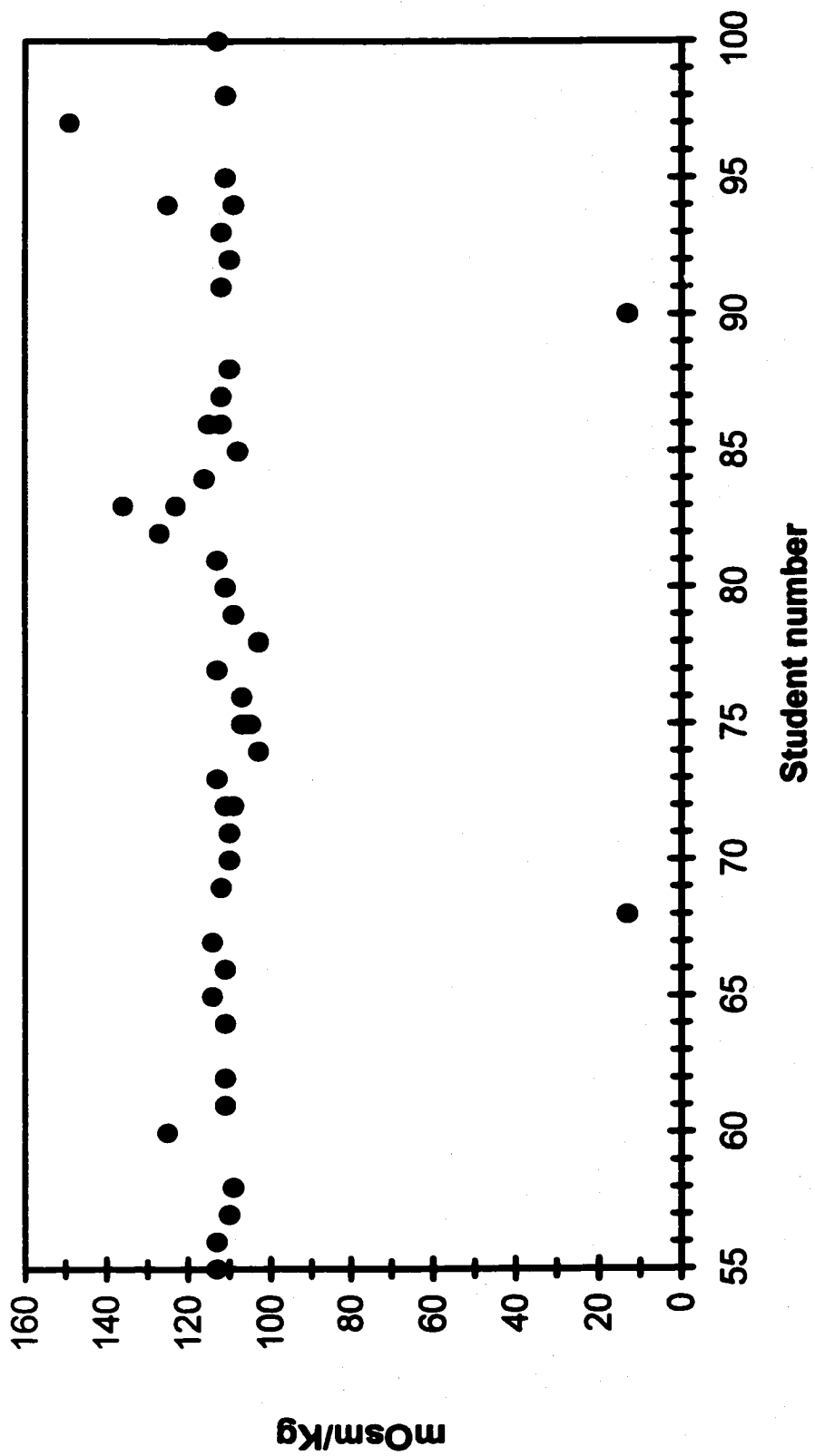
Osmometer Readings for 0.600 M KCl, Fall, 1999



Osmometer Readings for 0.1200 M KCl, Fall, 1999



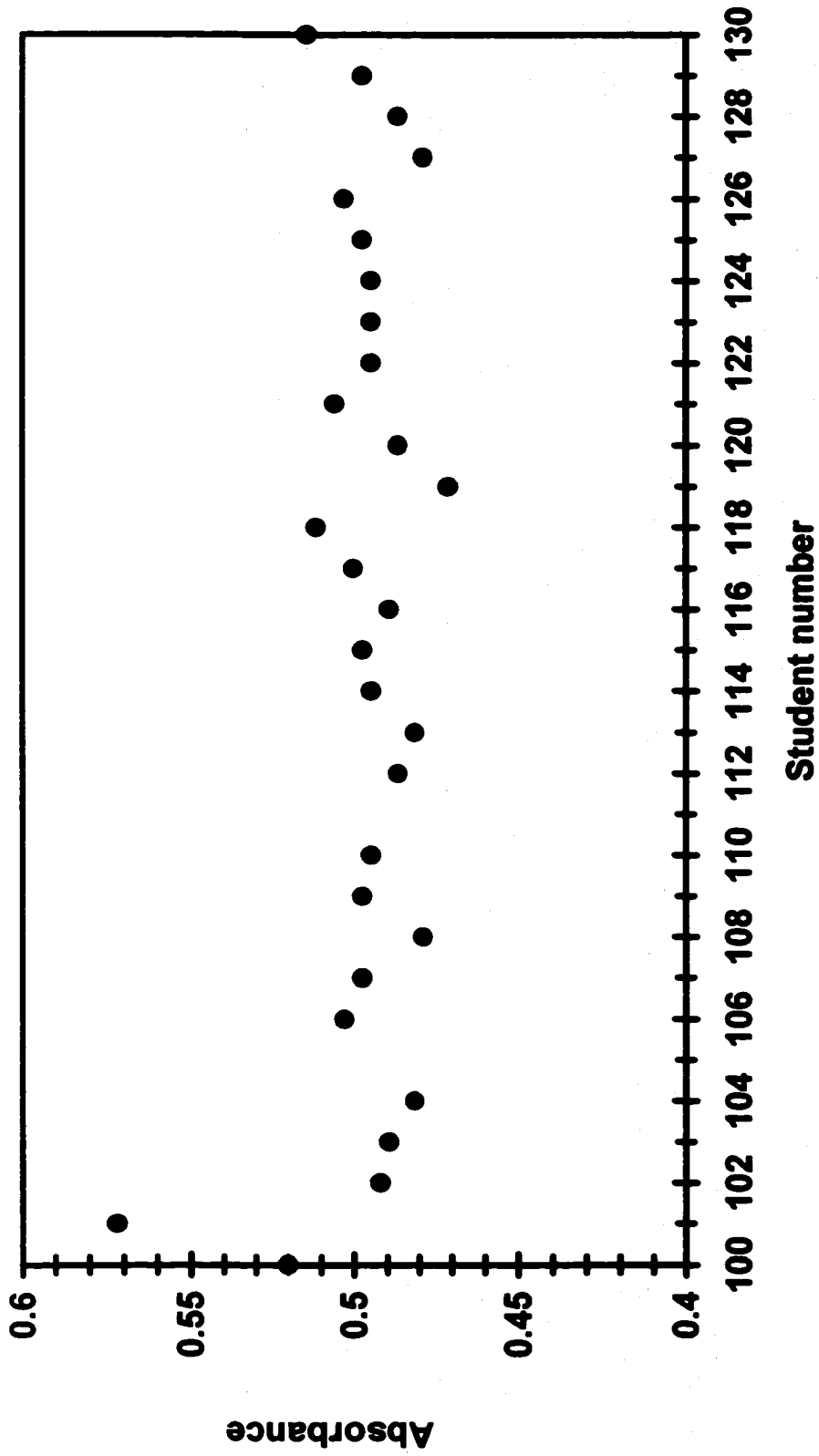
Osmometer Readings for 0.1500 M KCl, Fall, 1999



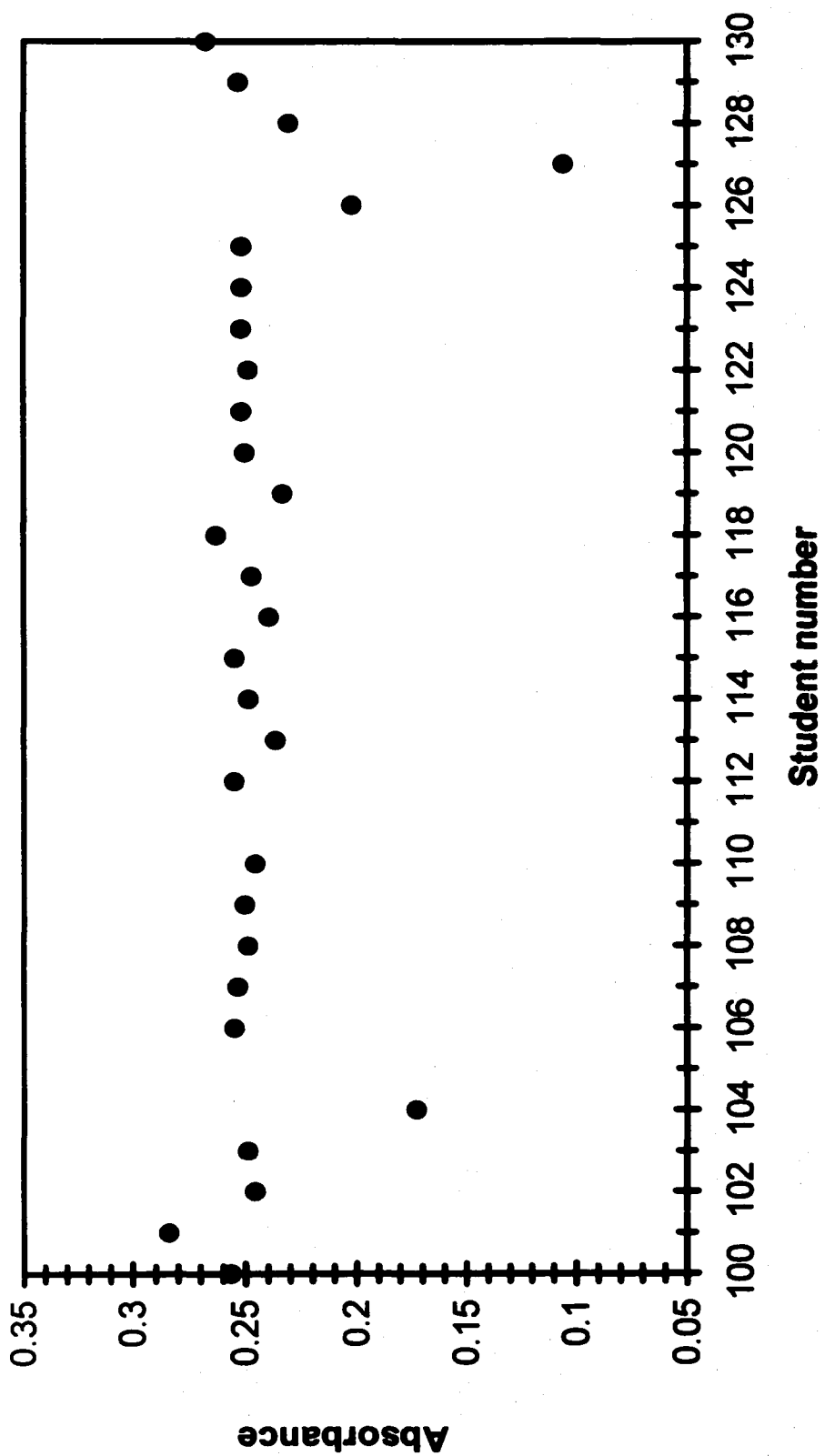
Osmometer Readings for 0.0600 M KCl, Fall, 1999

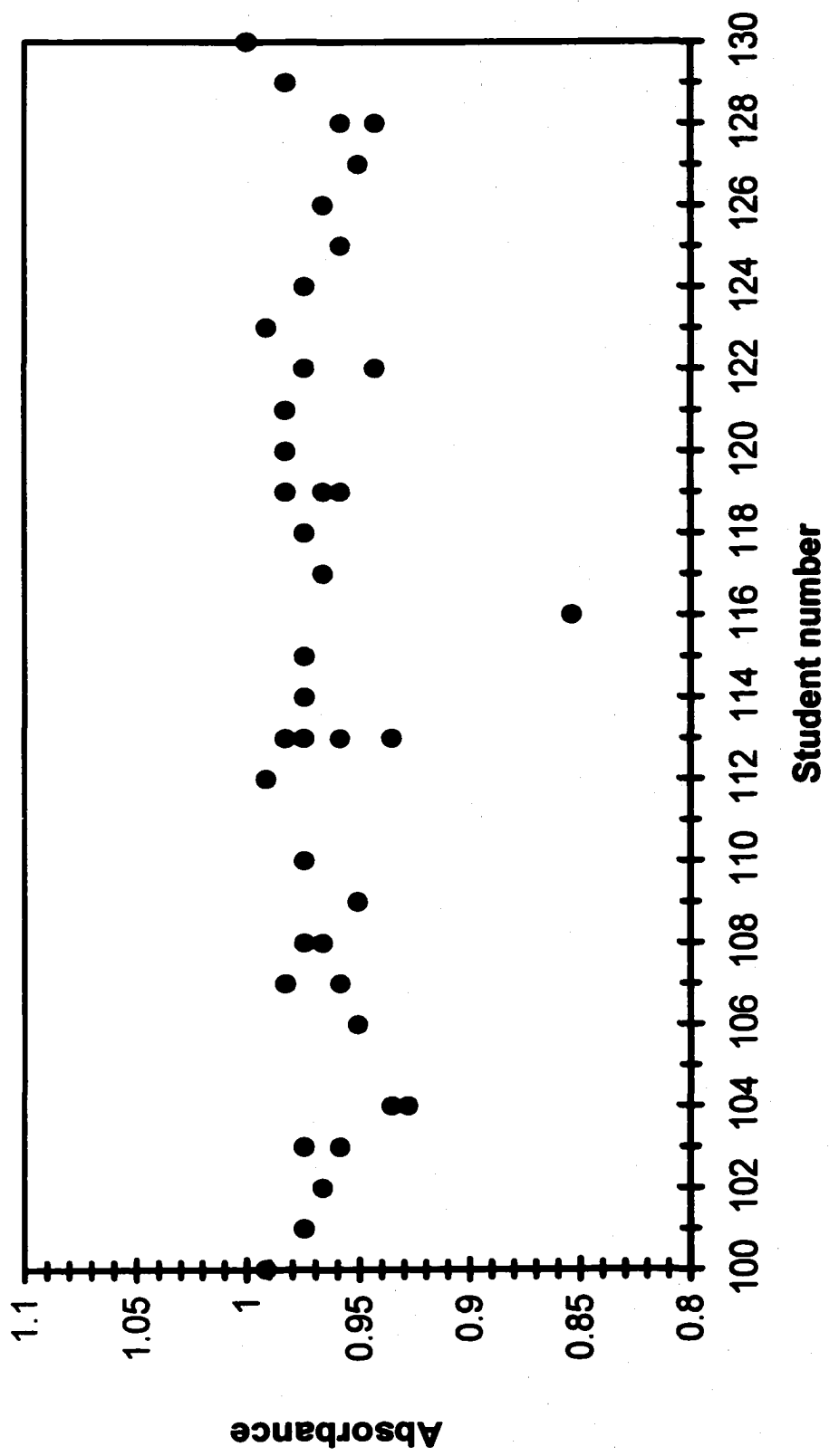
Appendix B4

Instrumental Reading Graphs for Spring Semester, 2000

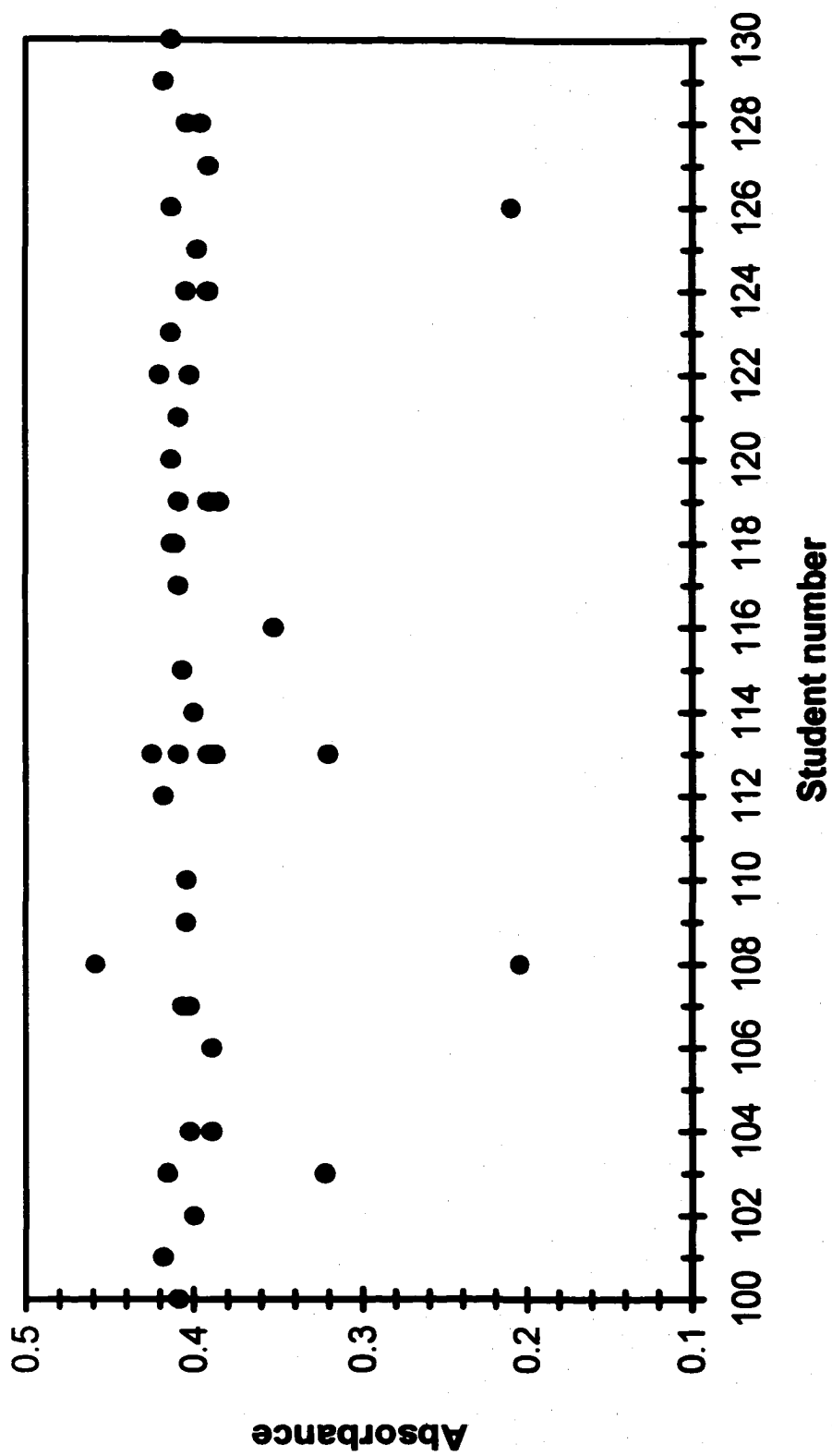


Spectronic 20 Data for 0.100M Ni(NO₃)₂ Solutions, Spring 2000

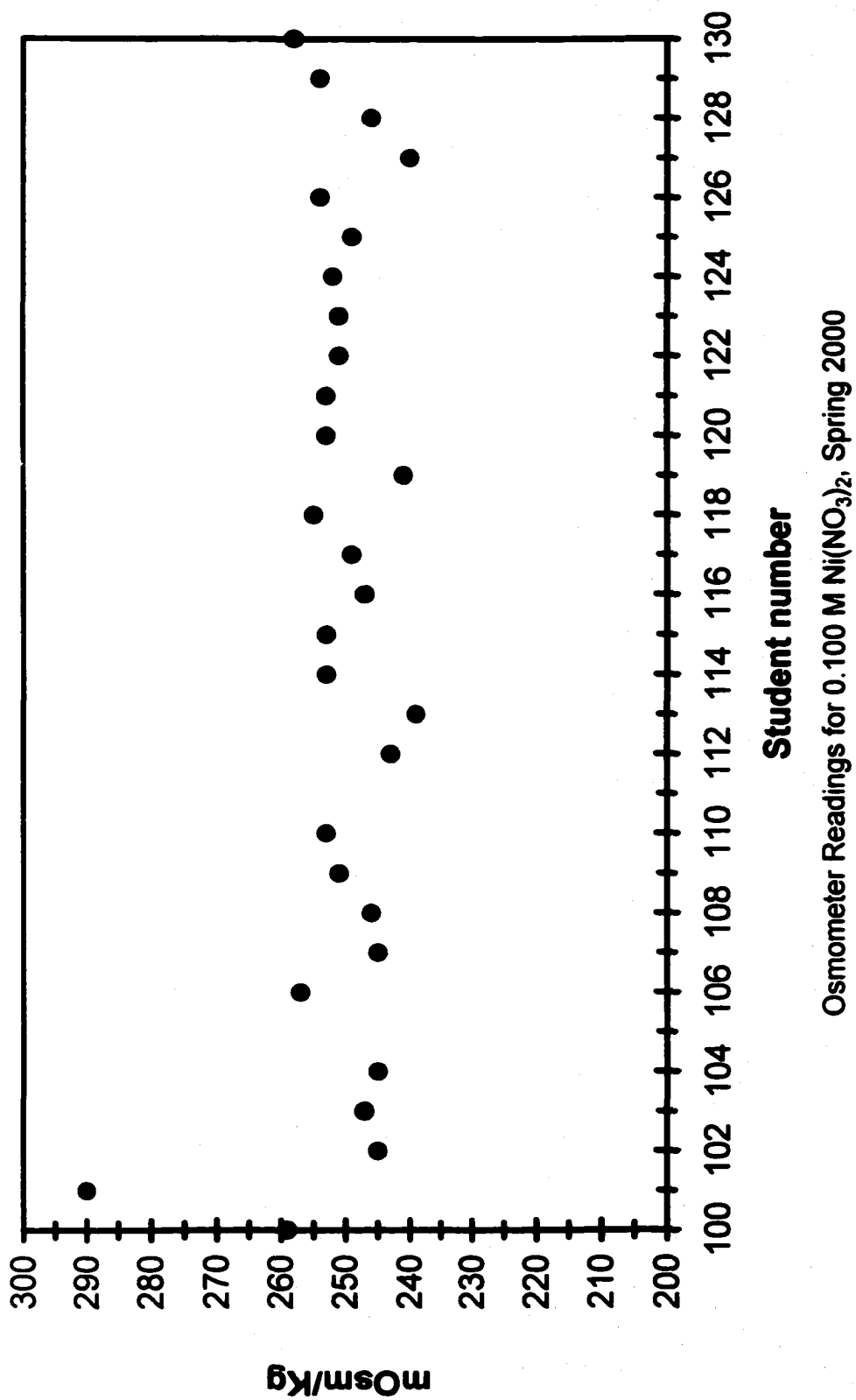
Spectronic 20 Data for 0.0500M Ni(NO₃)₂ Solutions, Spring 2000

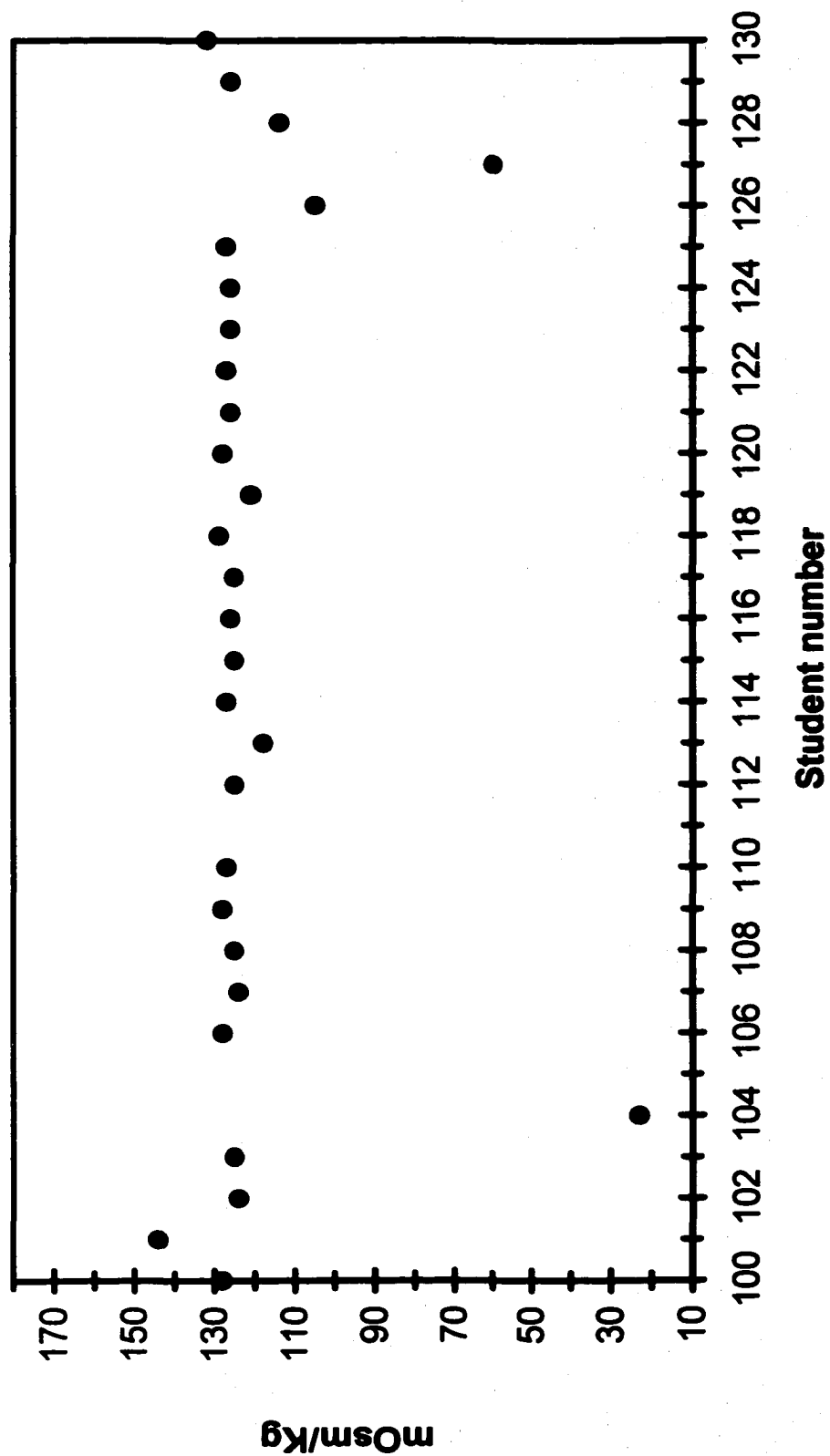


Spectronic 20 Data for 0.200M Ni(NO₃)₂ Solutions, Spring 2000

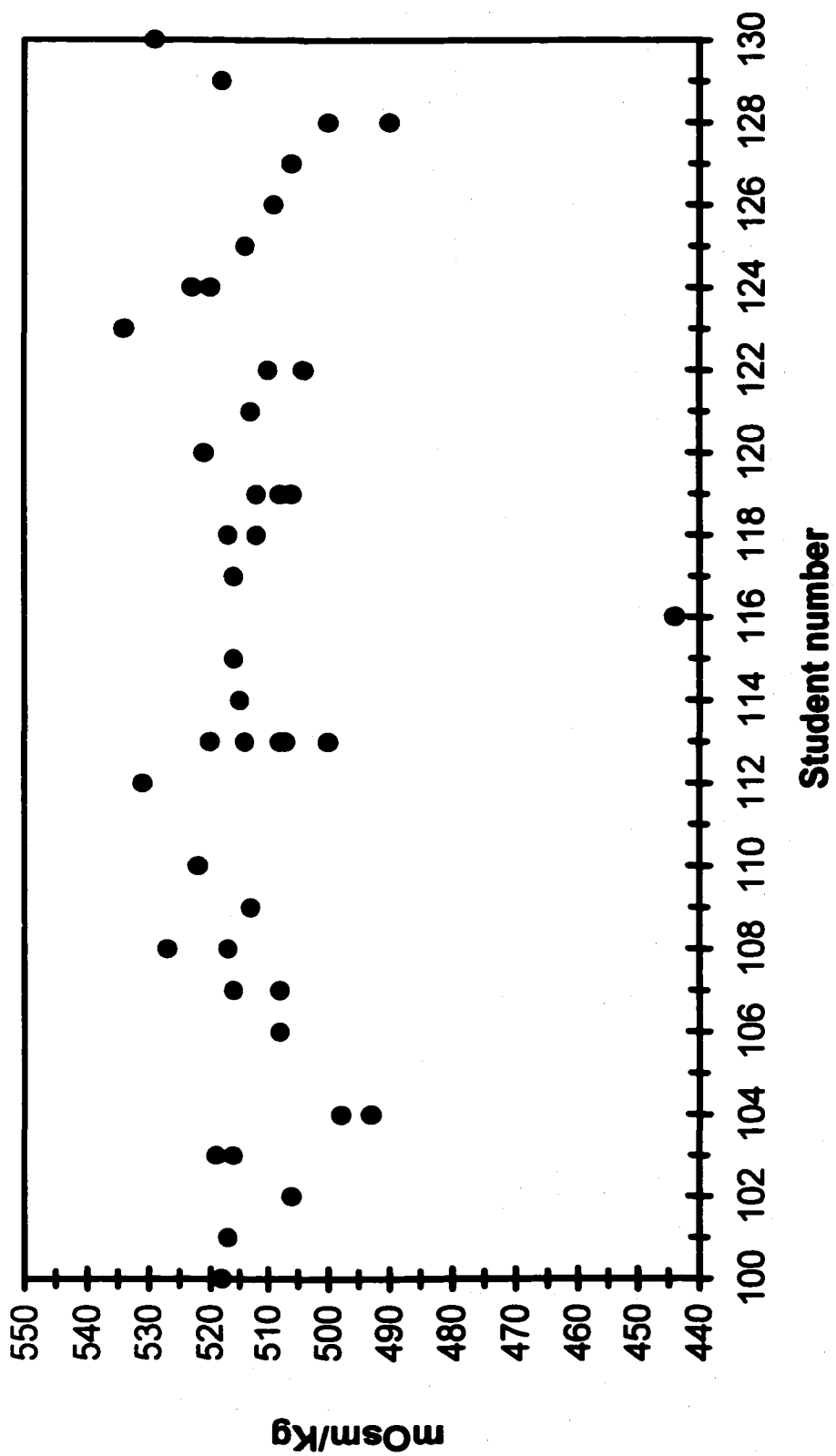


Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions, Spring 2000



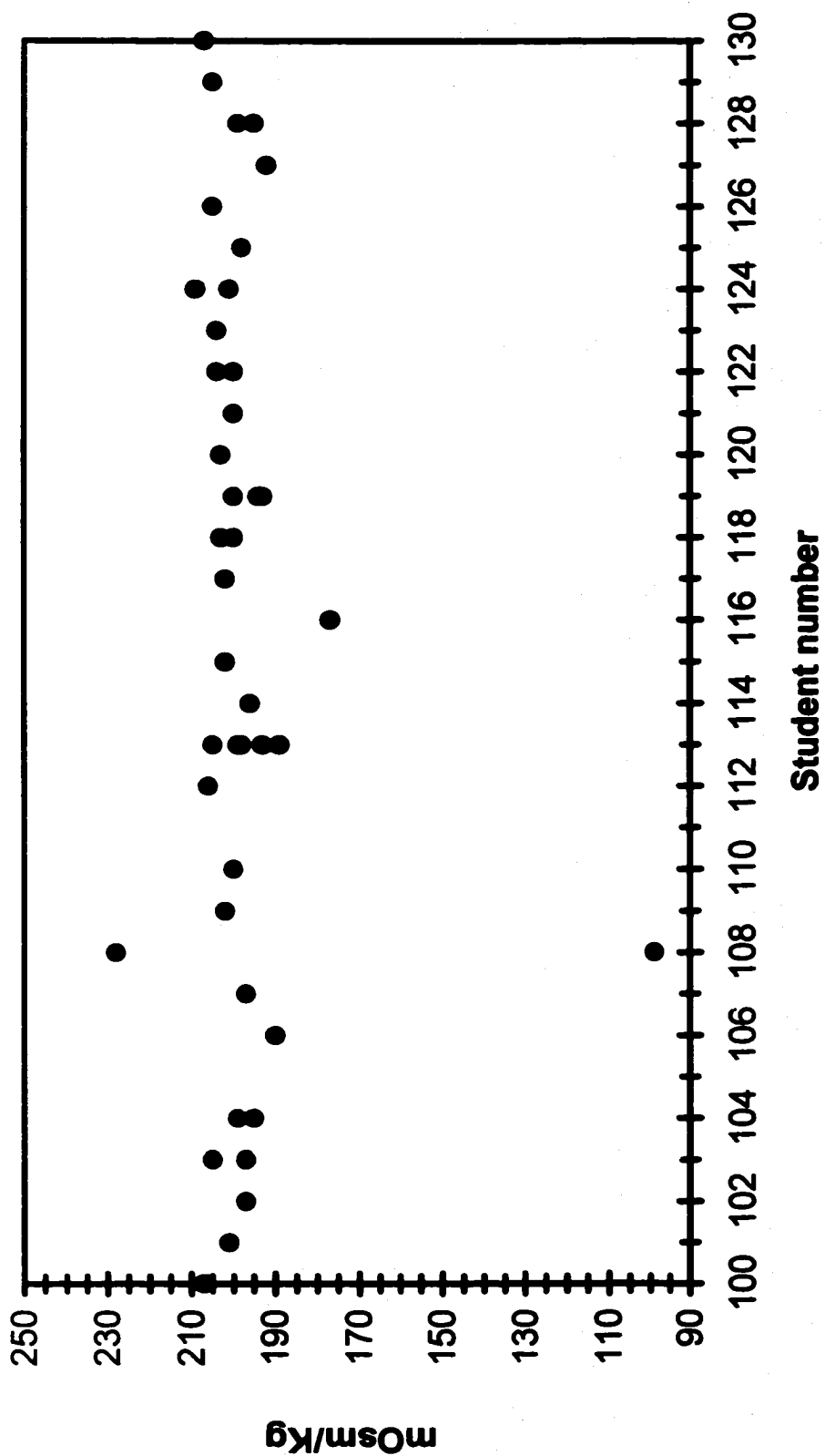


Osmometer Readings for 0.0500 M Ni(NO₃)₂, Spring 2000, View 1

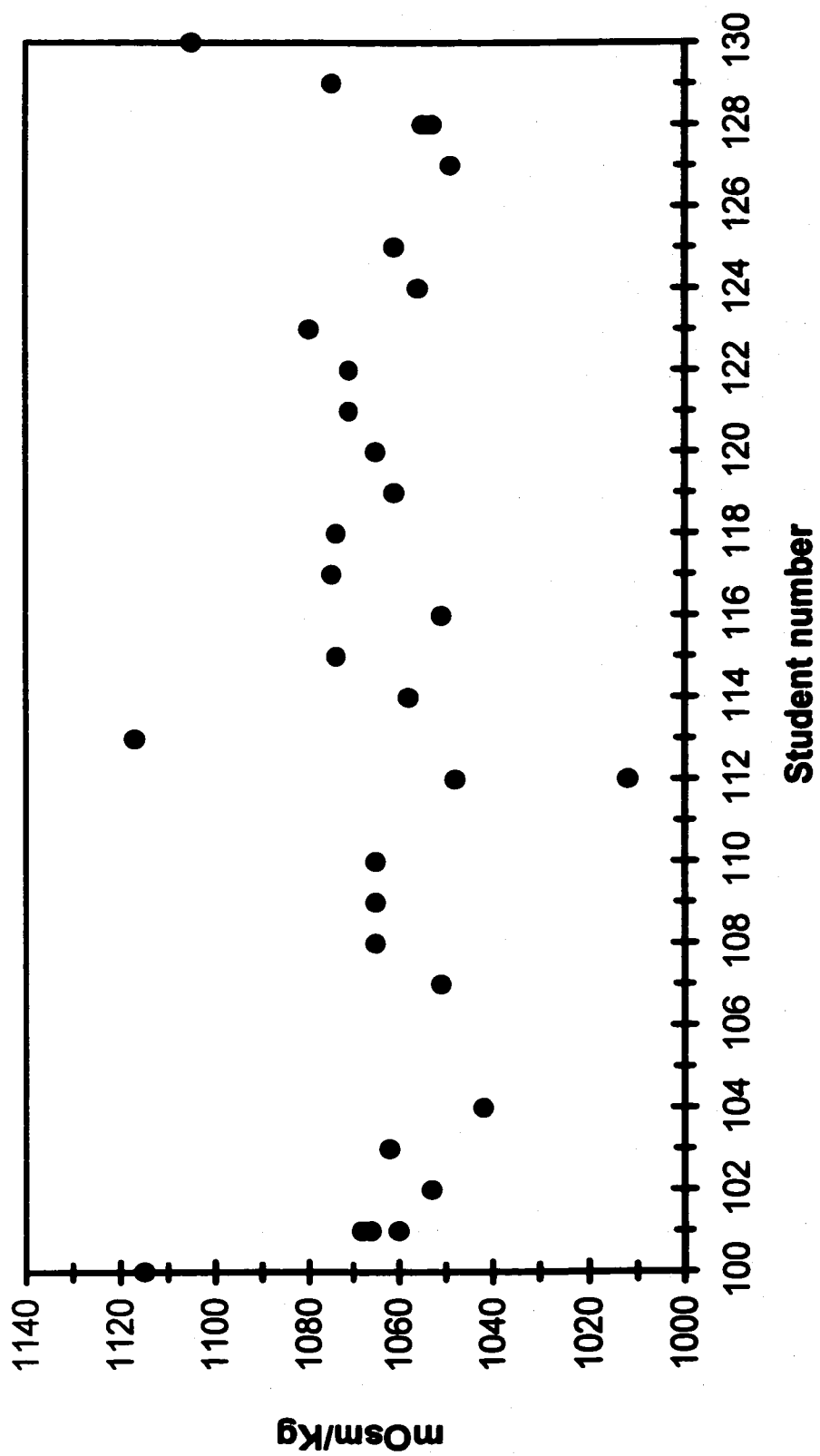


Student number

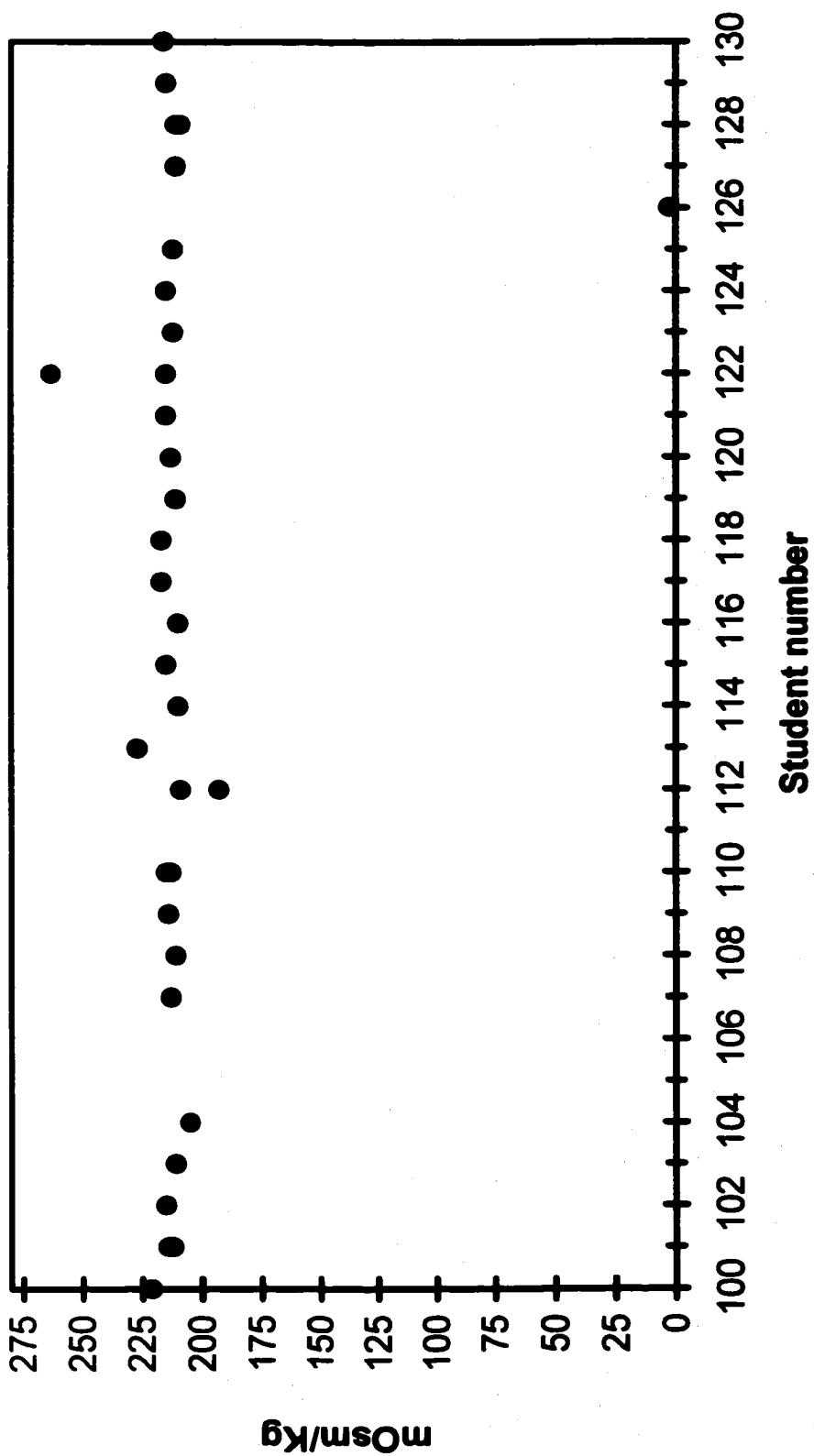
Osmometer Readings for 0.200 M Ni(NO₃)₂, Spring 2000, View 1



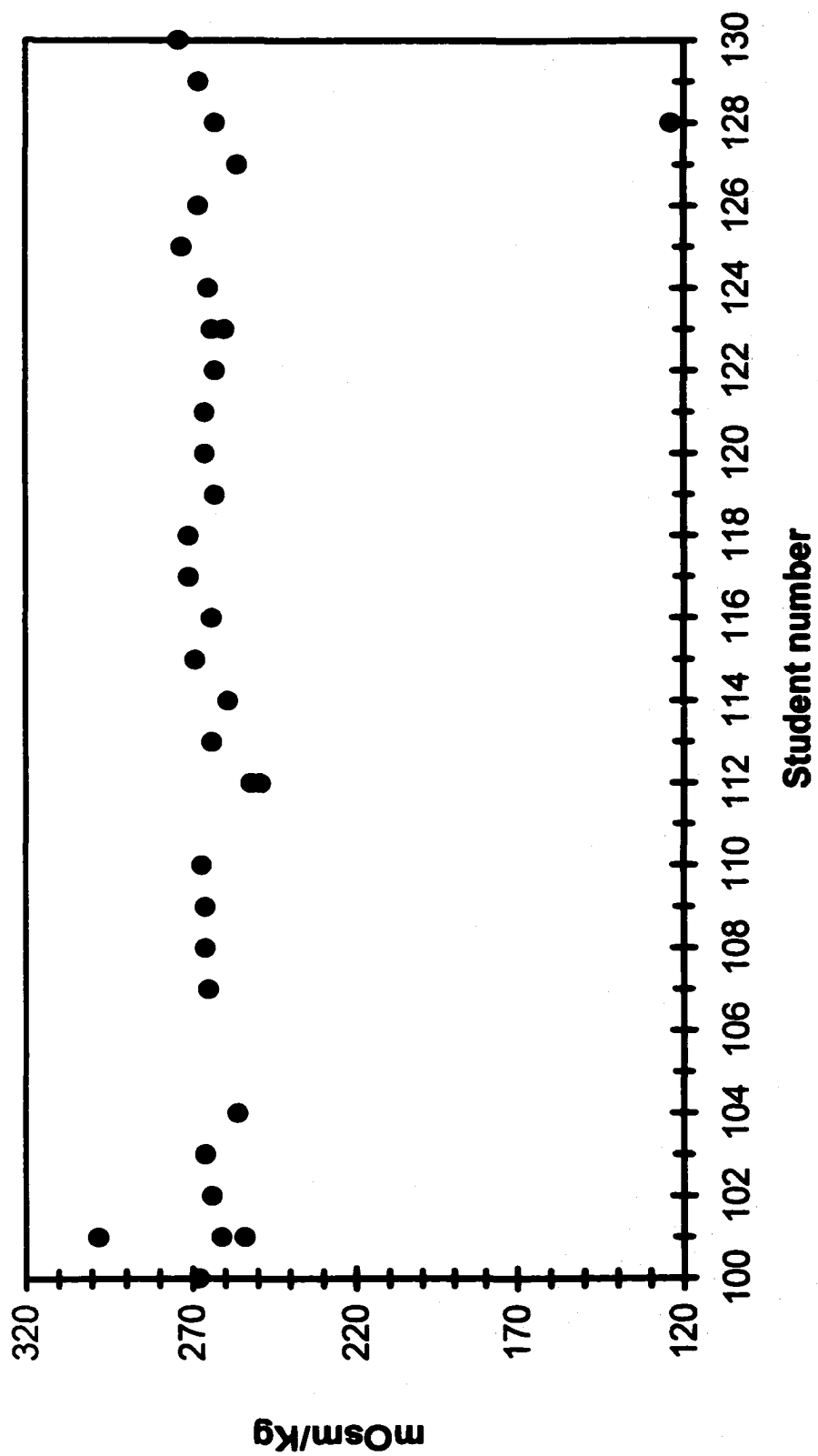
Osmometer Readings for 0.0800 M Ni(NO₃)₂, Spring 2000, View 1



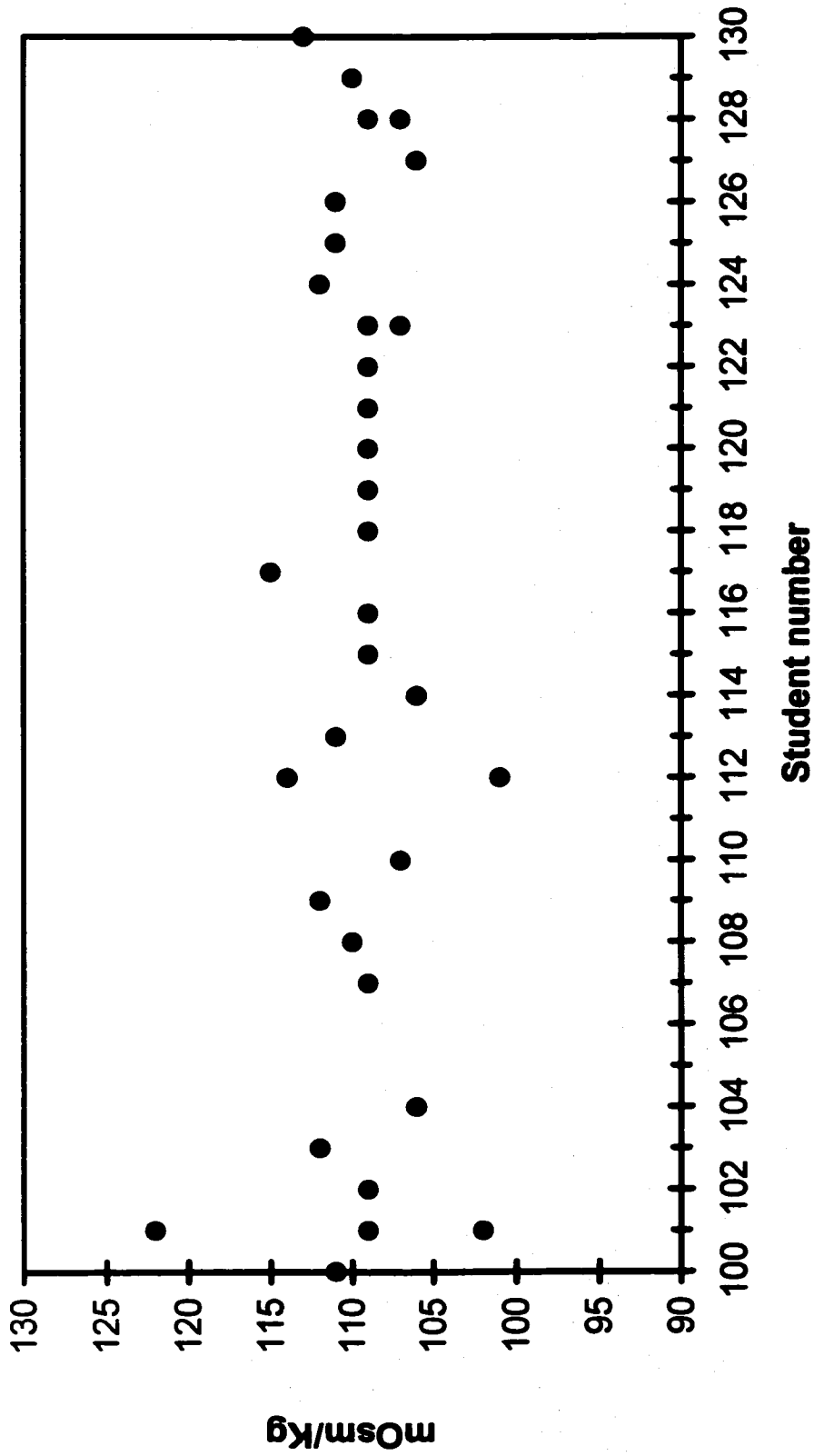
Osmometer Readings for 0.600 M KCl, Spring 2000, View 2



Osmometer Readings for 0.1200 M KCl, Spring 2000, View 1



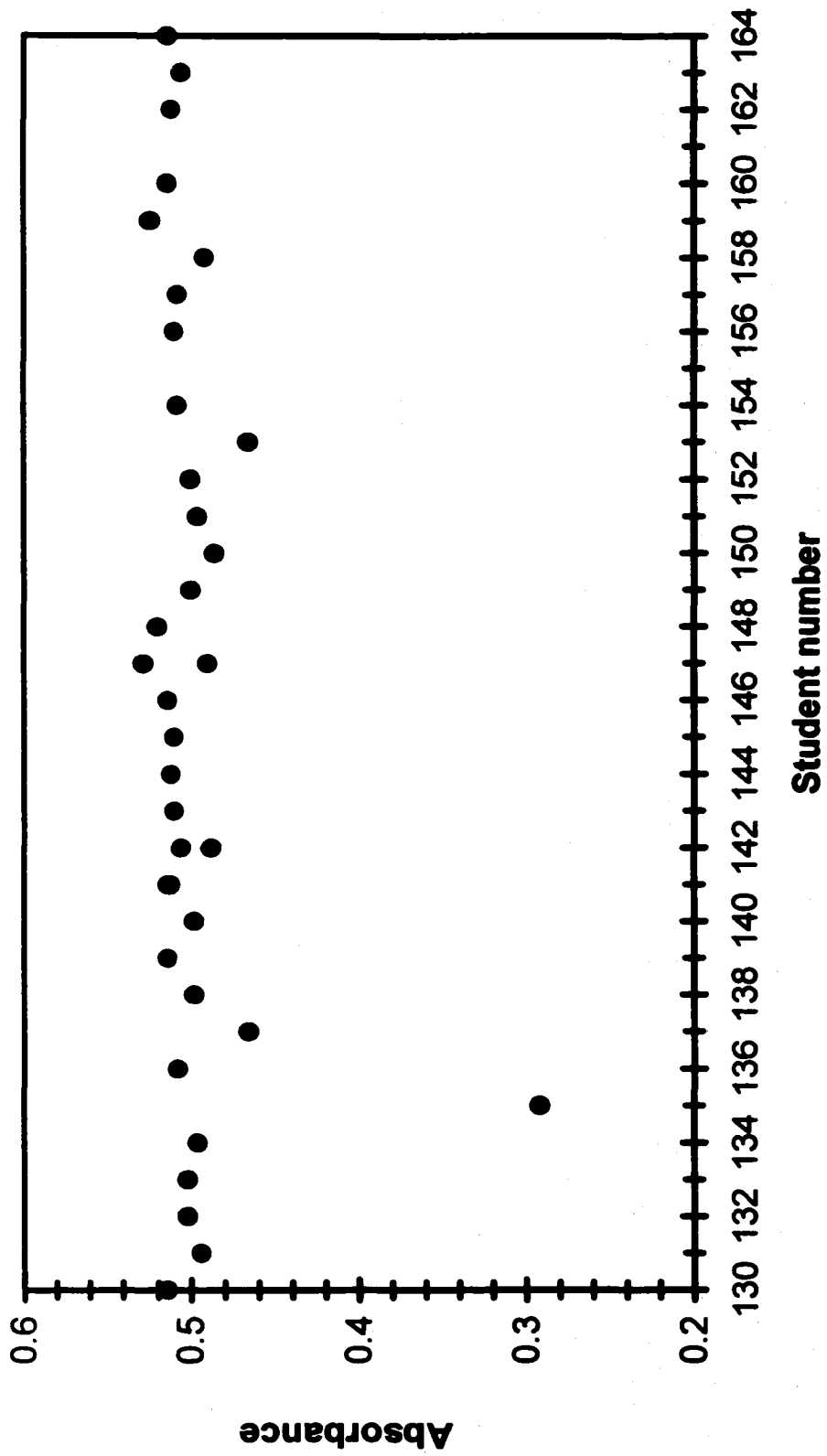
Osmometer Readings for 0.1500 M KCl, Spring 2000, View 1



Osmometer Readings for 0.0600 M KCl, Spring 2000, View 2

Appendix B5

Instrumental Reading Graphs for Fall Semester, 2000

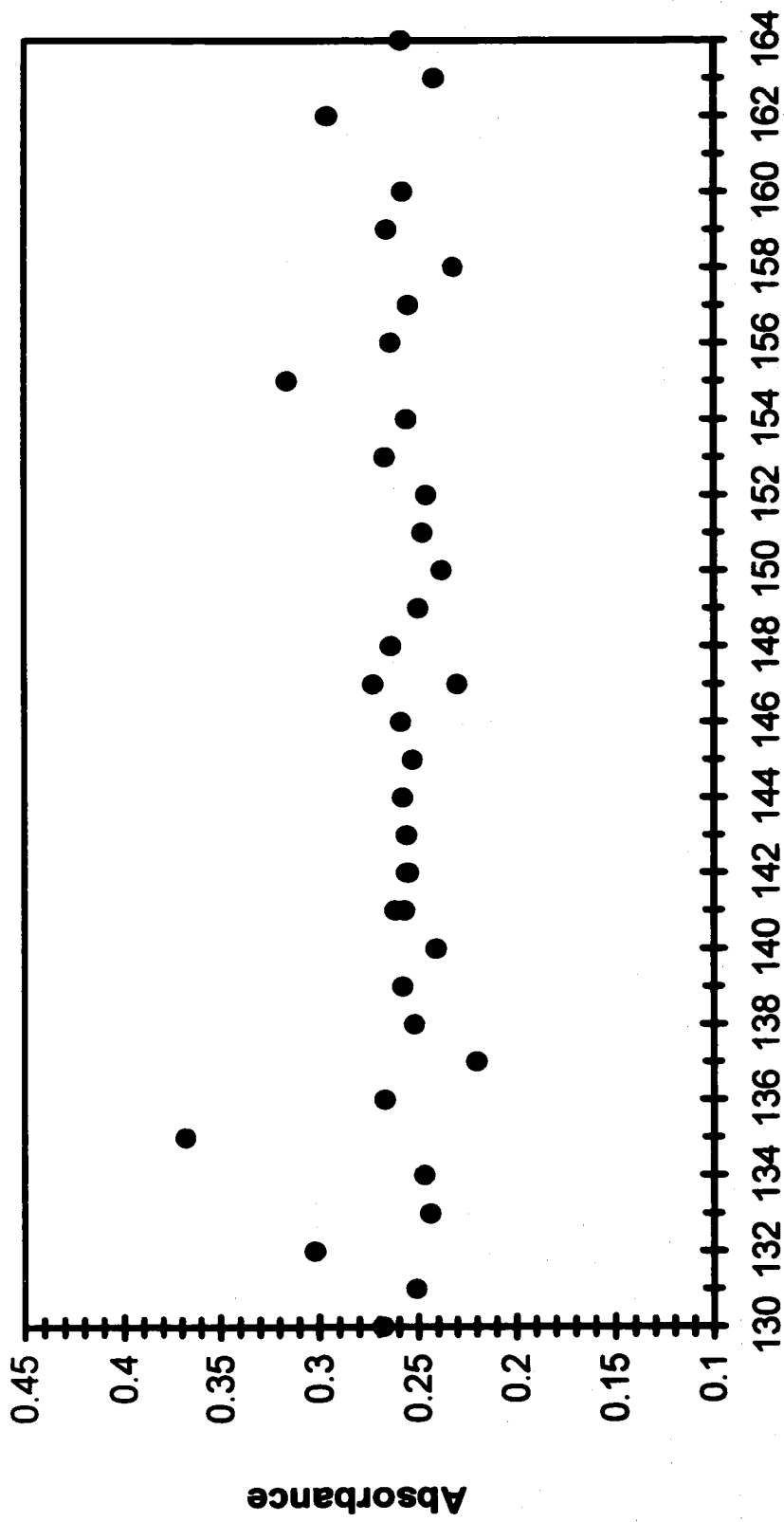


Spectronic 20 Data for 0.100M Ni(NO₃)₂ Solutions, Fall 2000

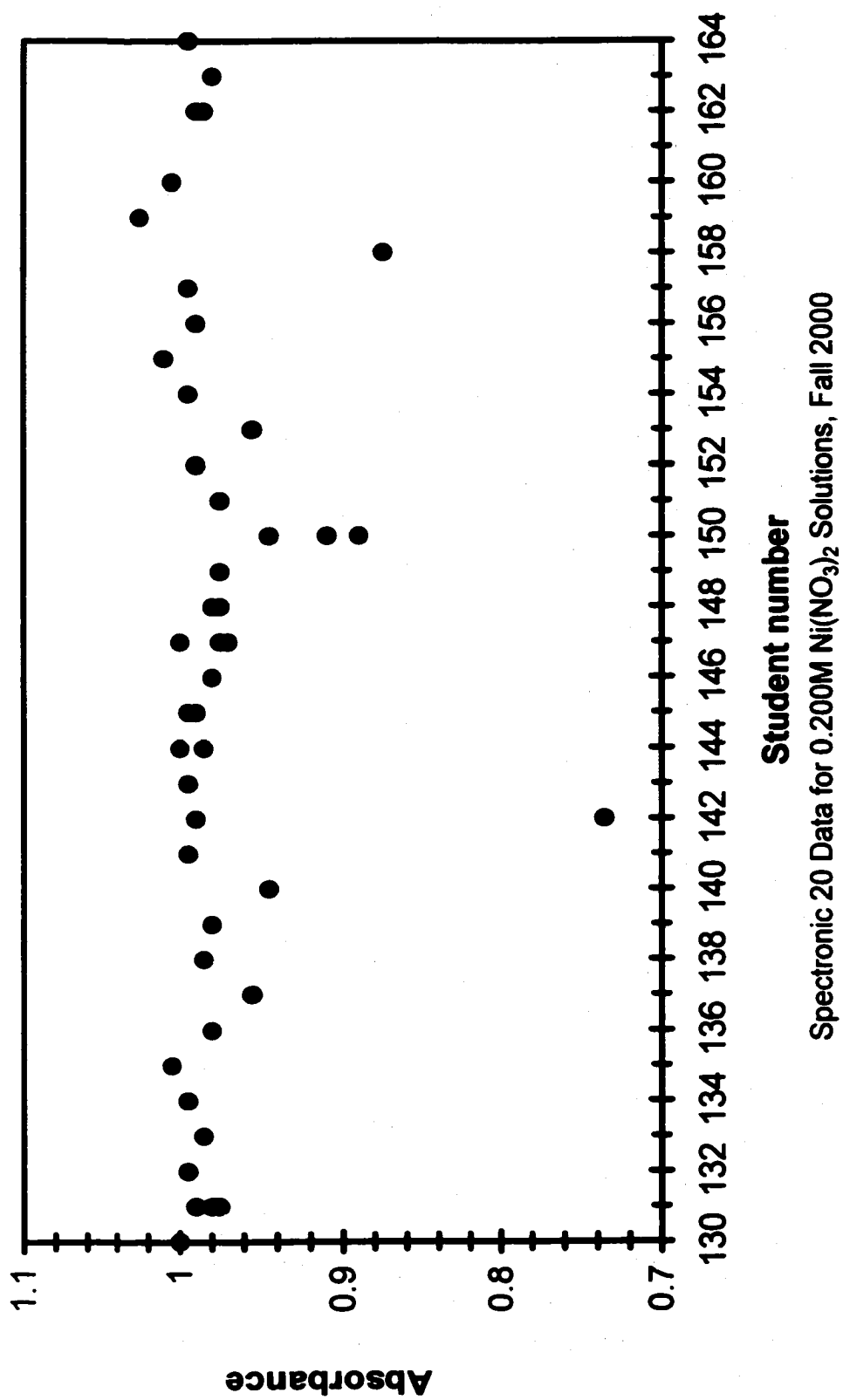
Student number

Absorbance

130 132 134 136 138 140 142 144 146 148 150 152 154 156 158 160 162 164

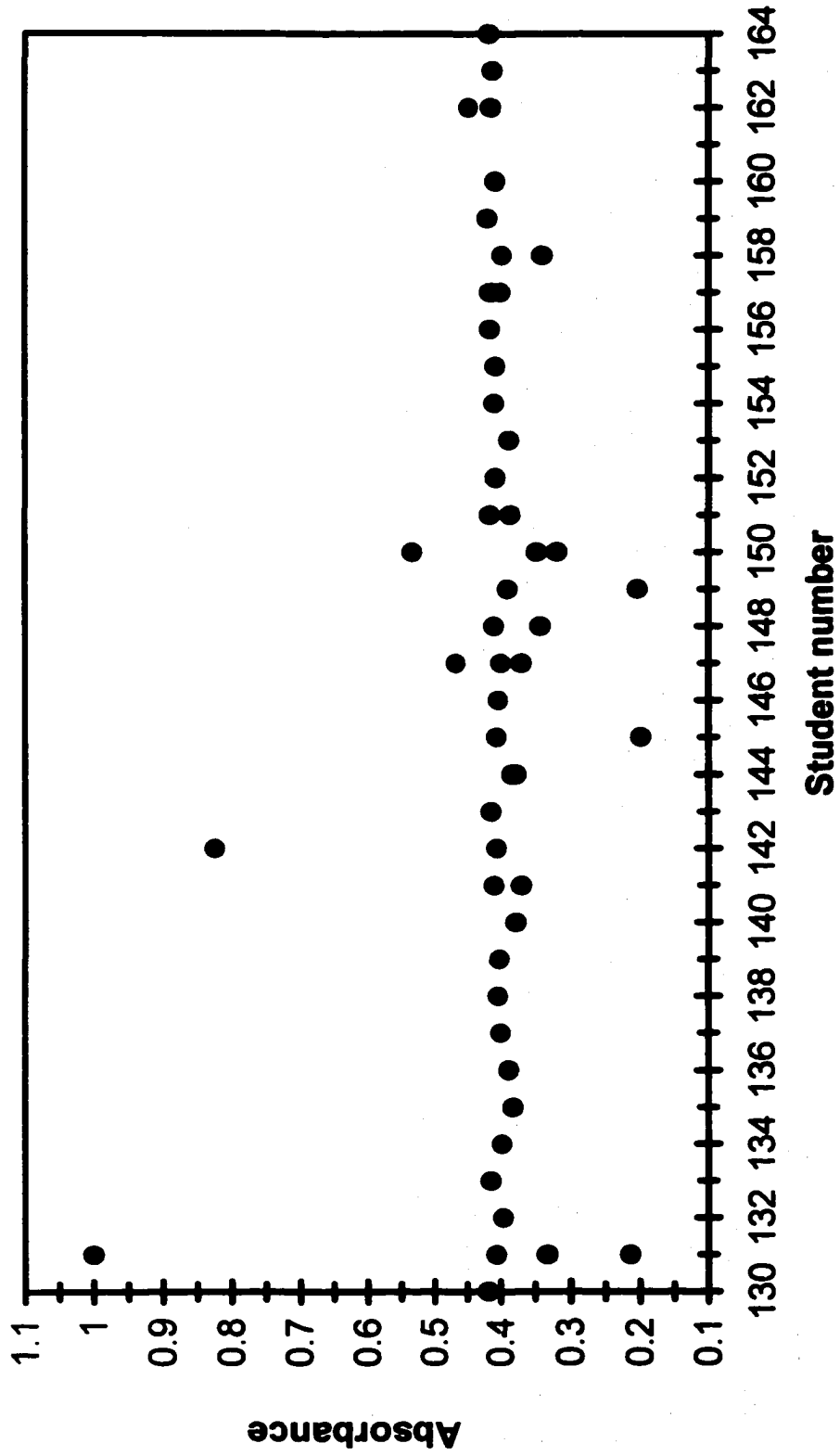


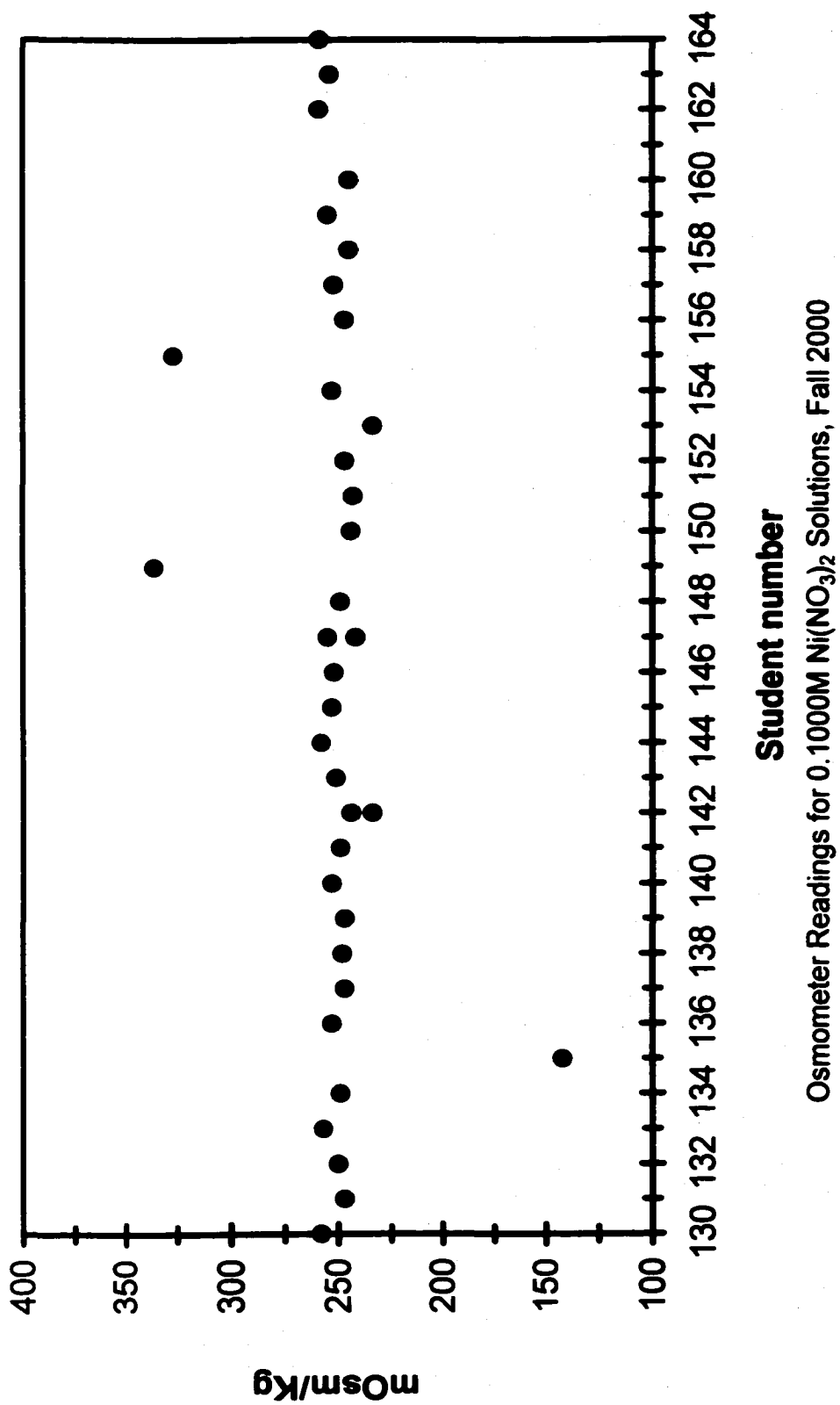
Student number
Spectronic 20 Data for 0.0500M Ni(NO₃)₂ Solutions, Fall 2000

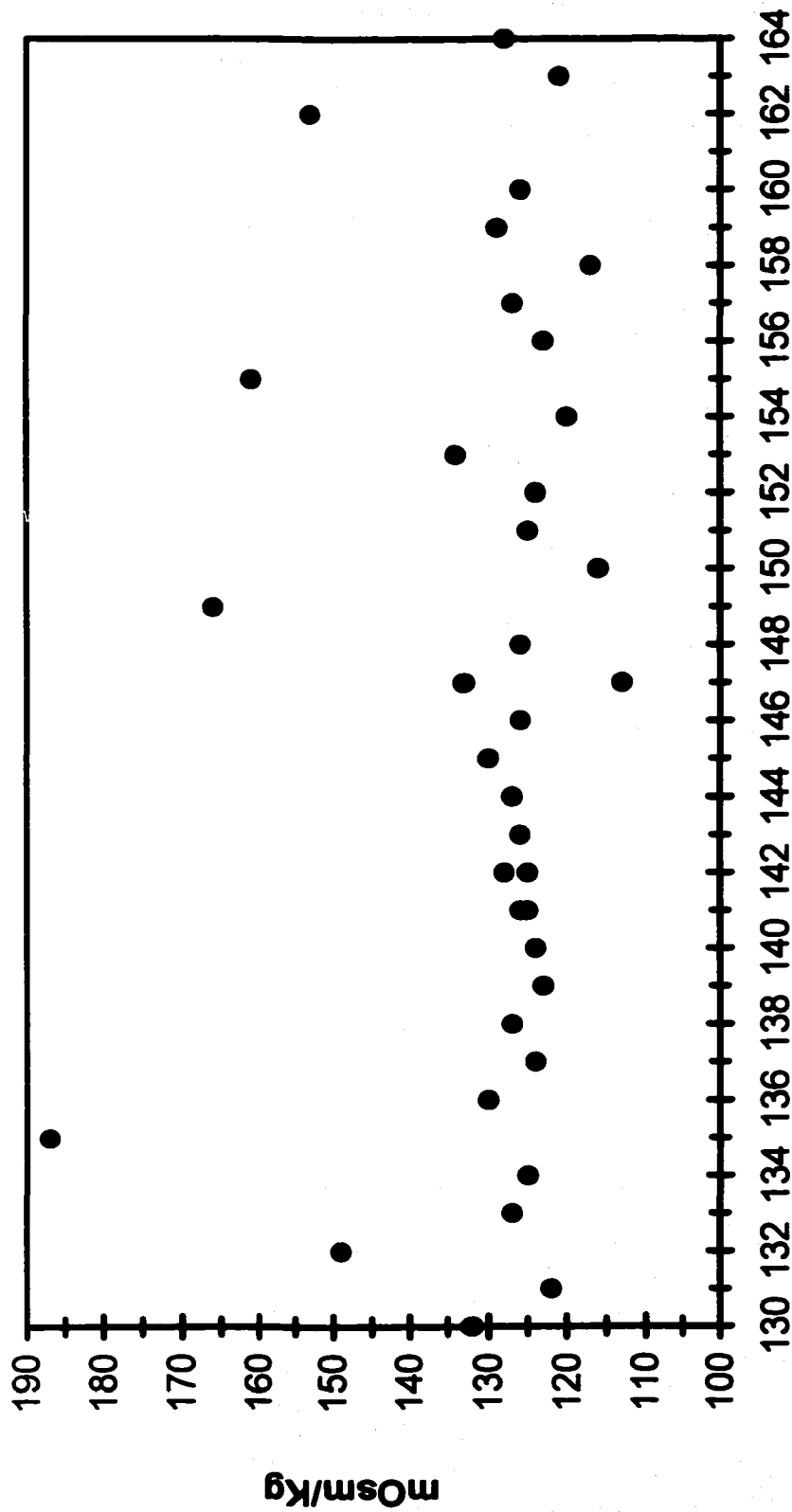


Student number

Spectronic 20 Data for 0.200M Ni(NO₃)₂ Solutions, Fall 2000

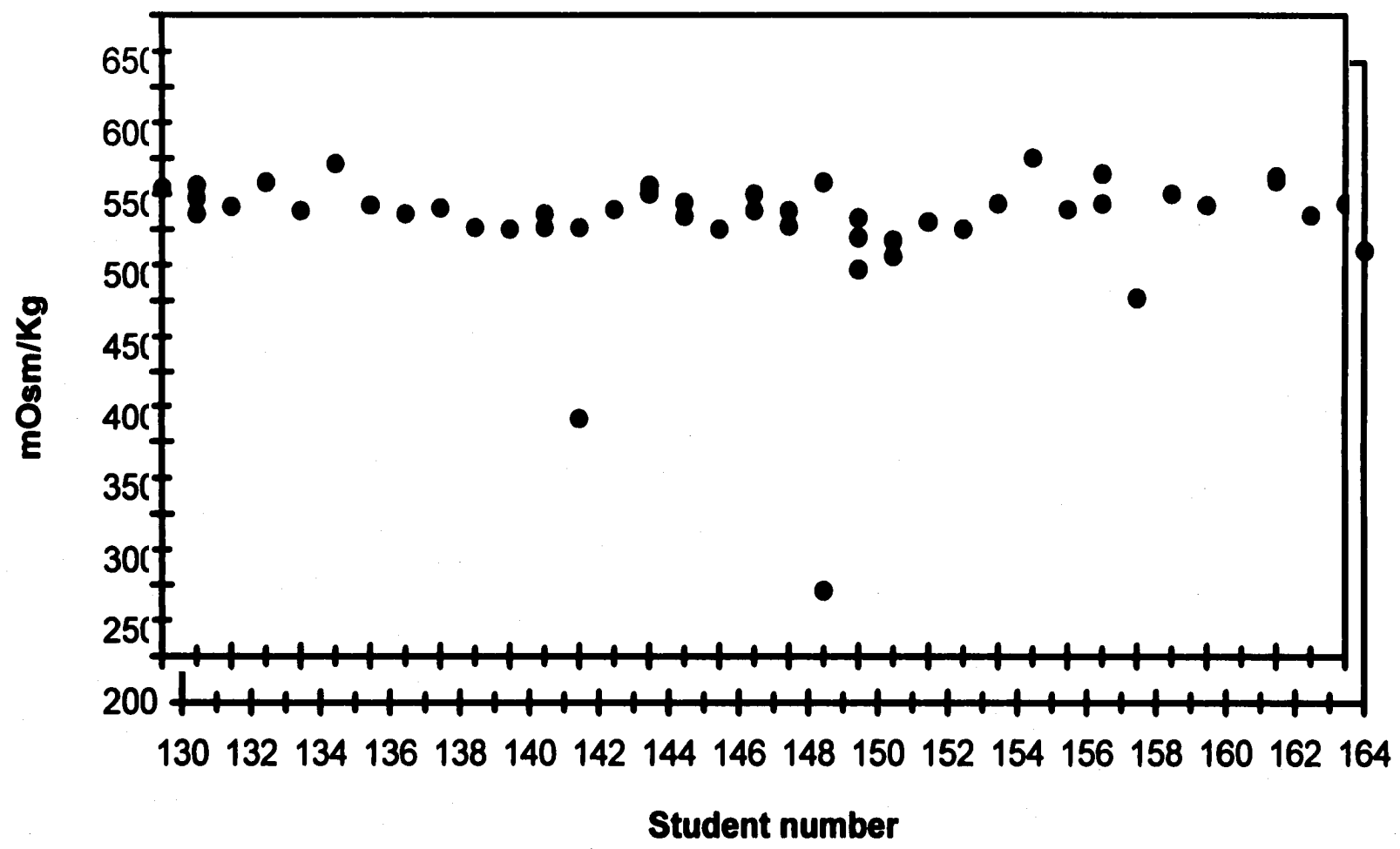
Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions, Fall 2000



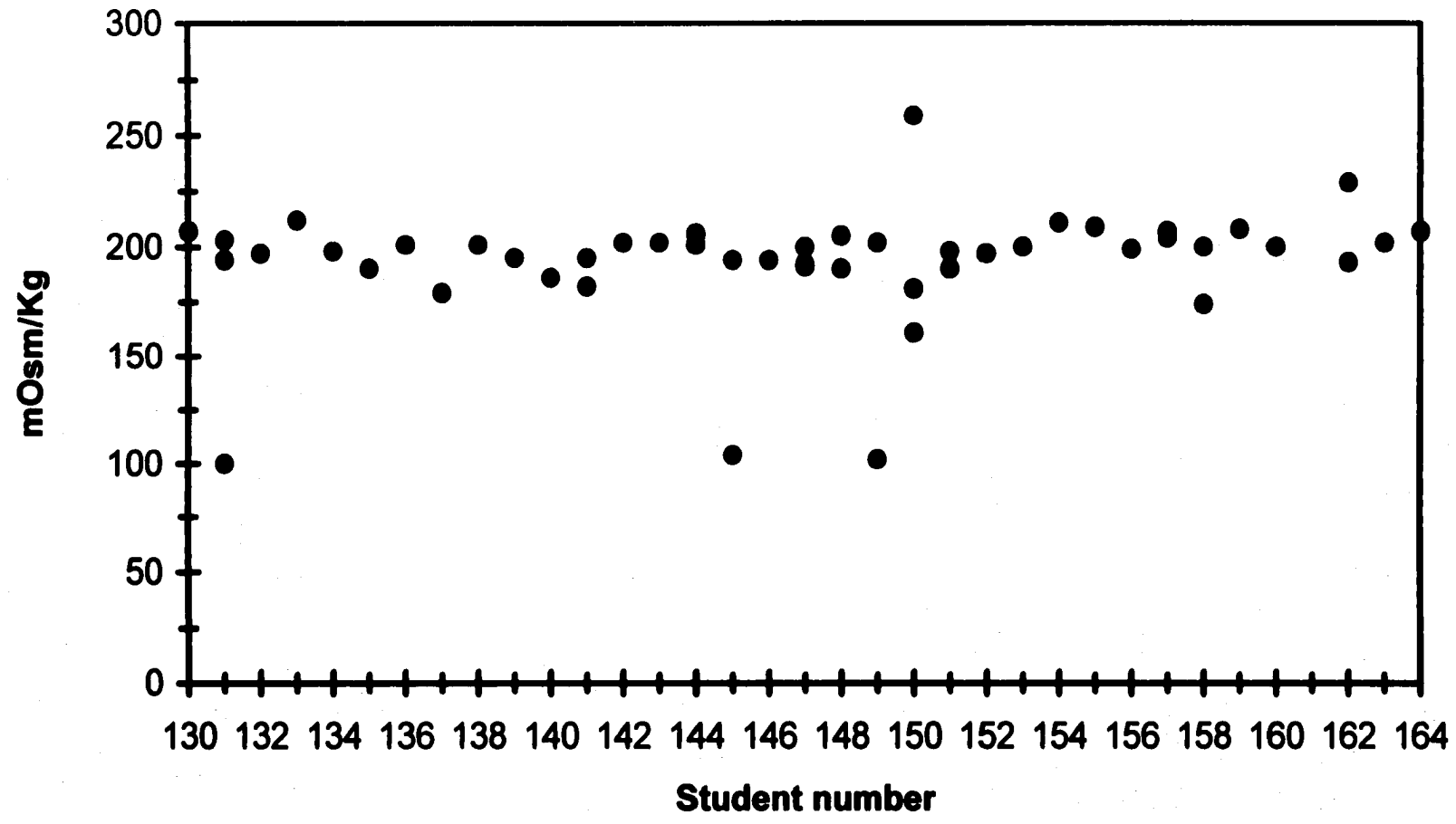


Student number

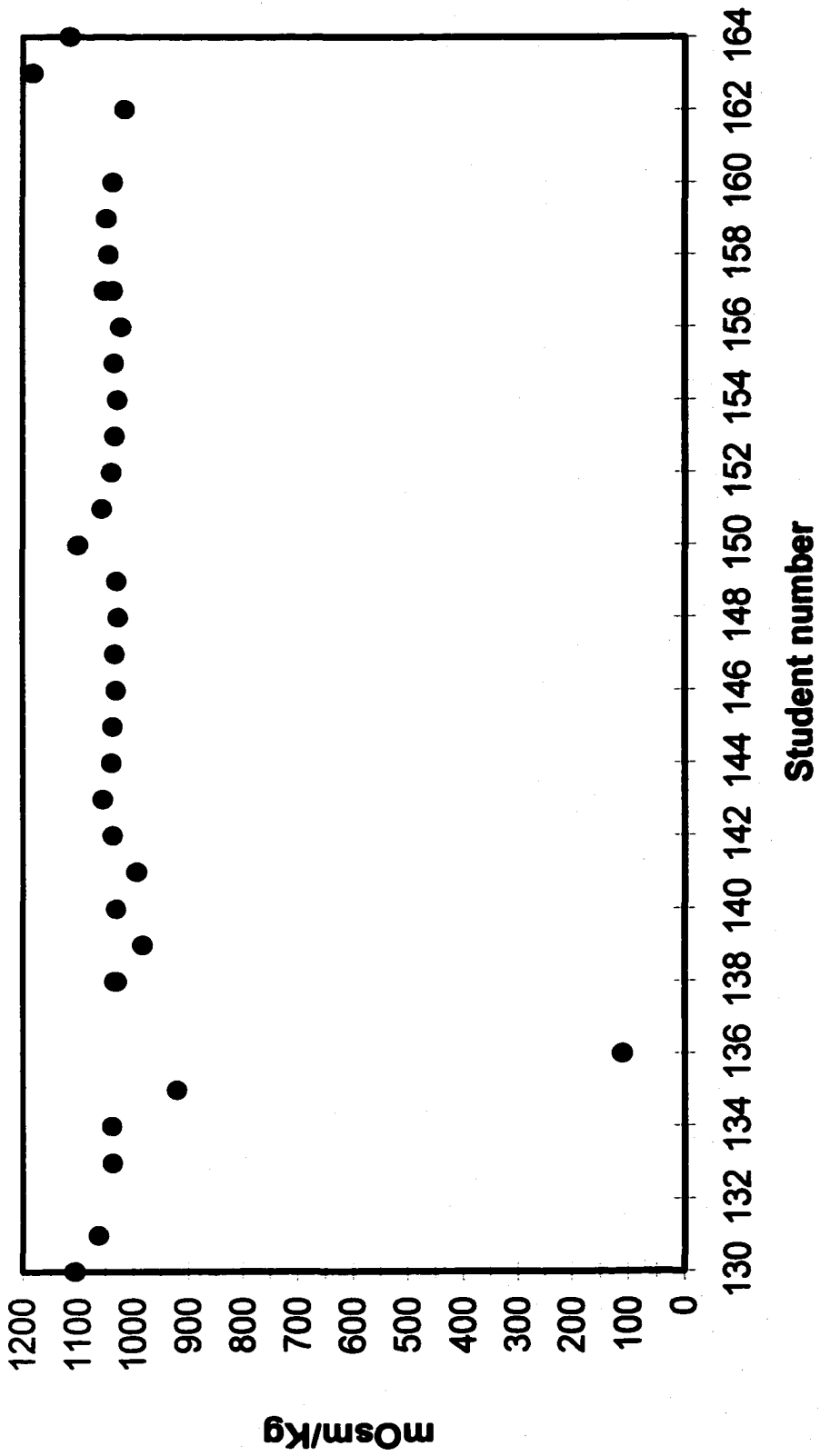
Osmometer Readings for 0.0500M Ni(NO₃)₂ Solutions, Fall 2000



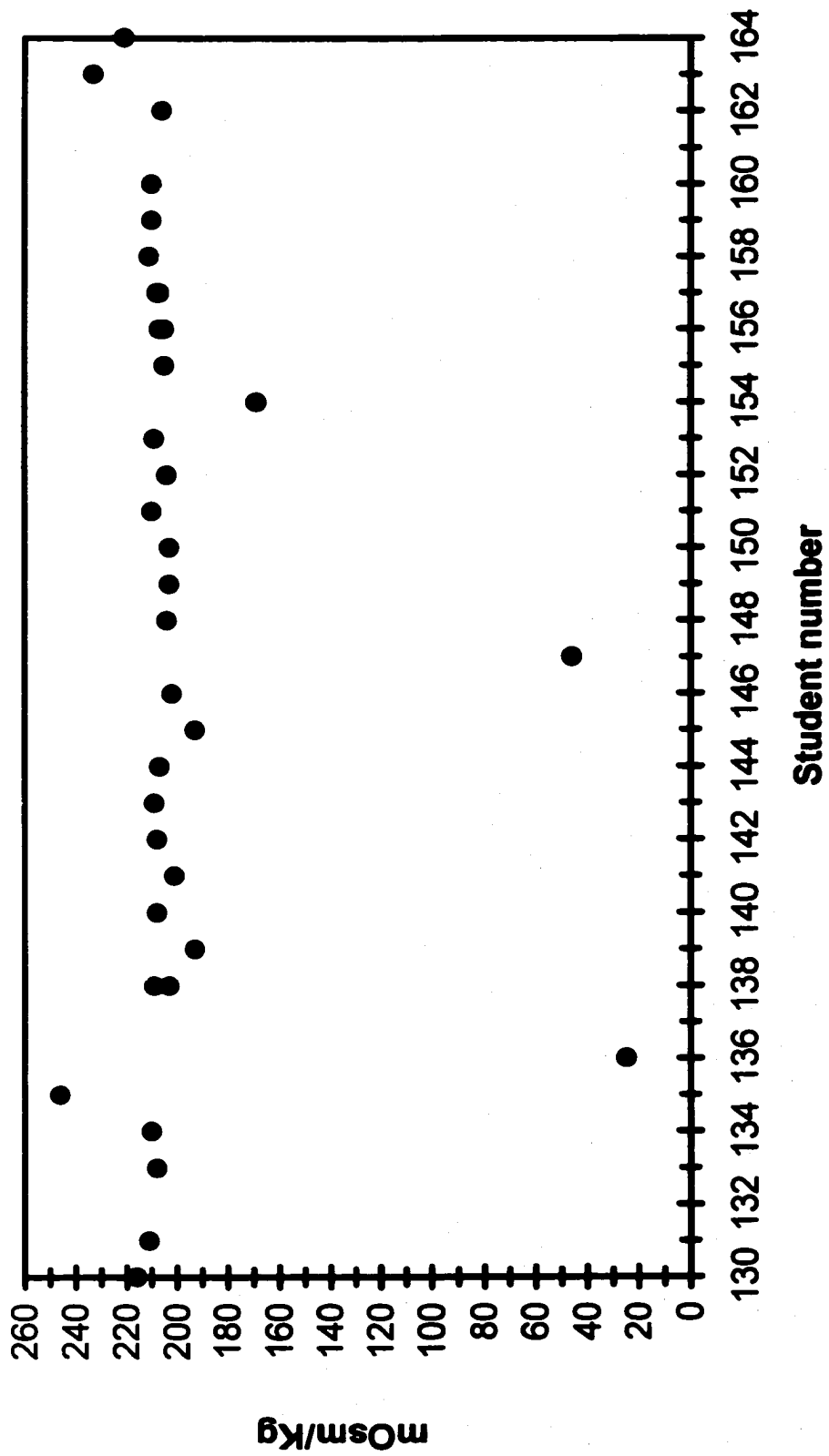
Osmometer Readings for 0.2000M Ni(NO₃)₂ Solutions, Fall 2000



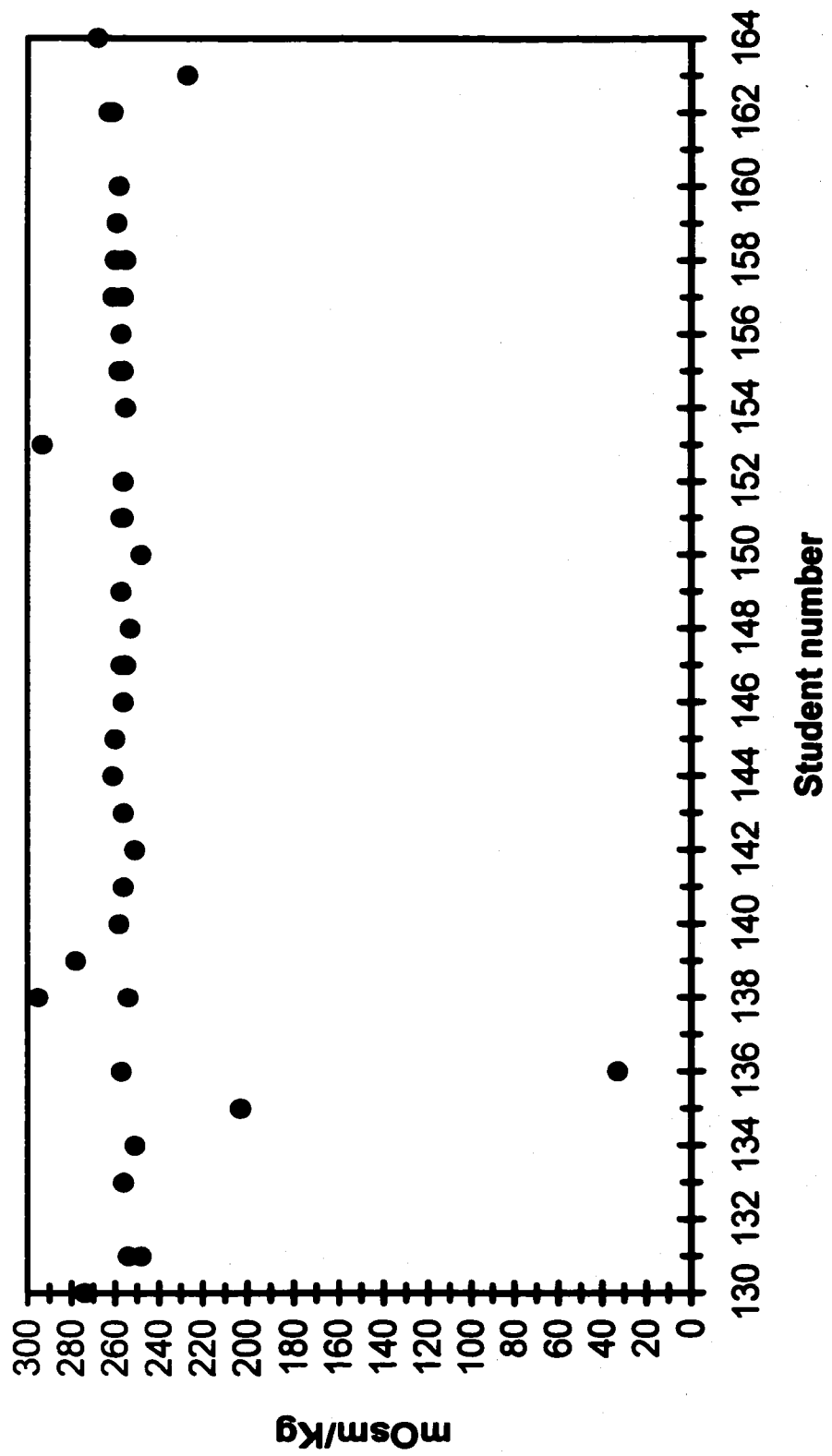
Osmometer Readings for 0.0800M Ni(NO₃)₂ Solutions, Fall 2000



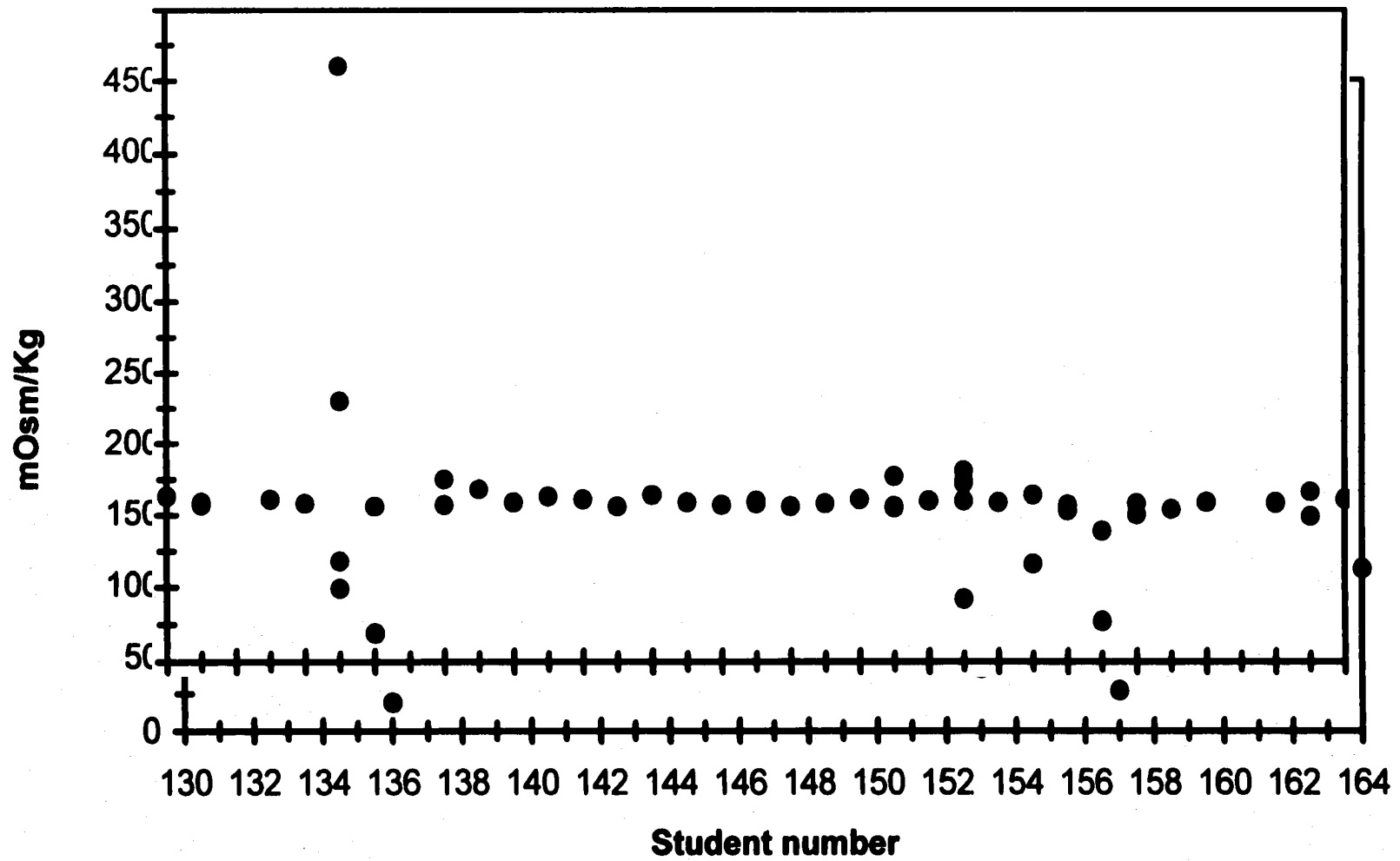
Osmometer Readings for 0.600 M KCl, Fall 2000, View 1



Osmometer Readings for 0.1200 M KCl, Fall 2000, View 1



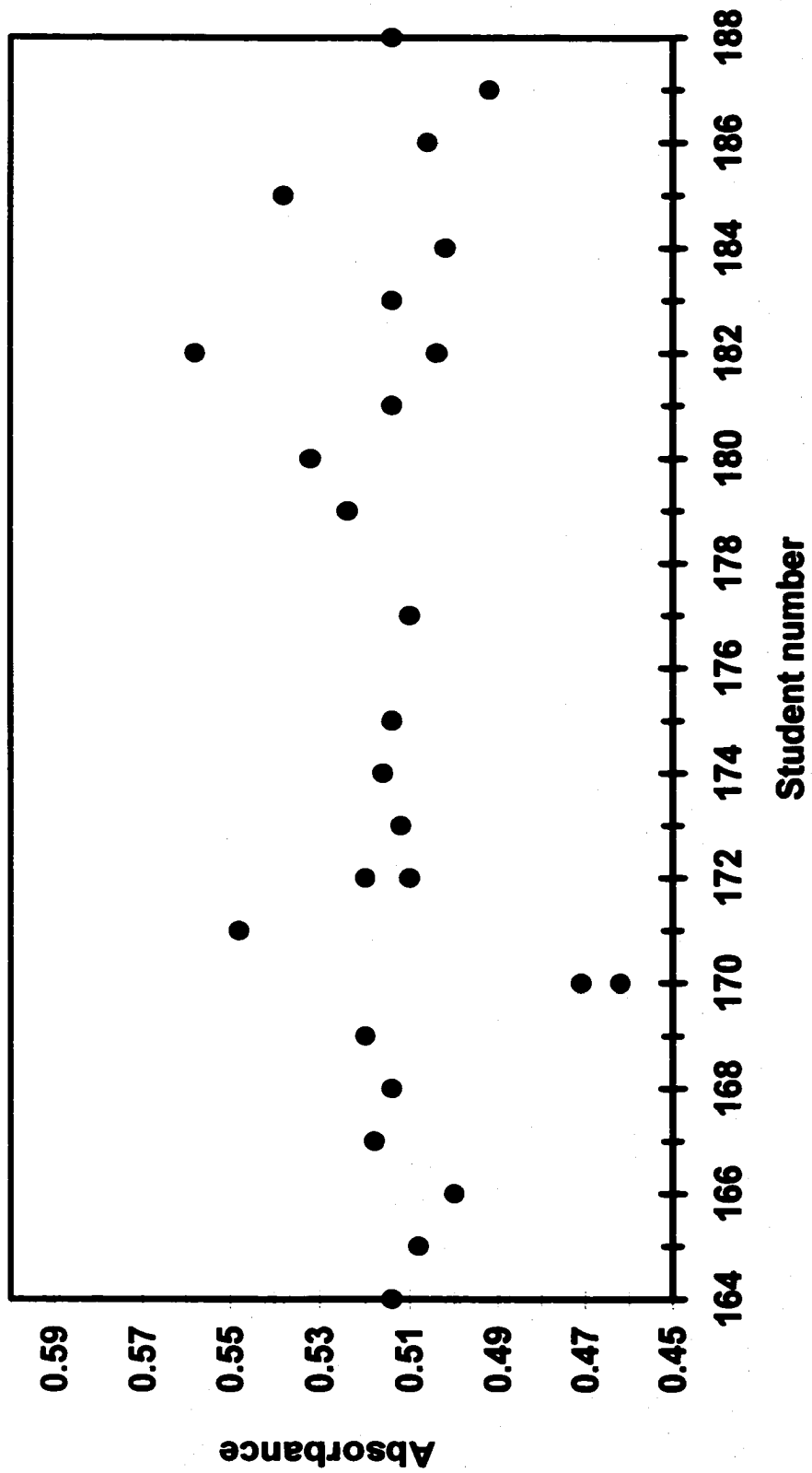
Osmometer Readings for 0.1500 M KCl, Fall 2000, View 1

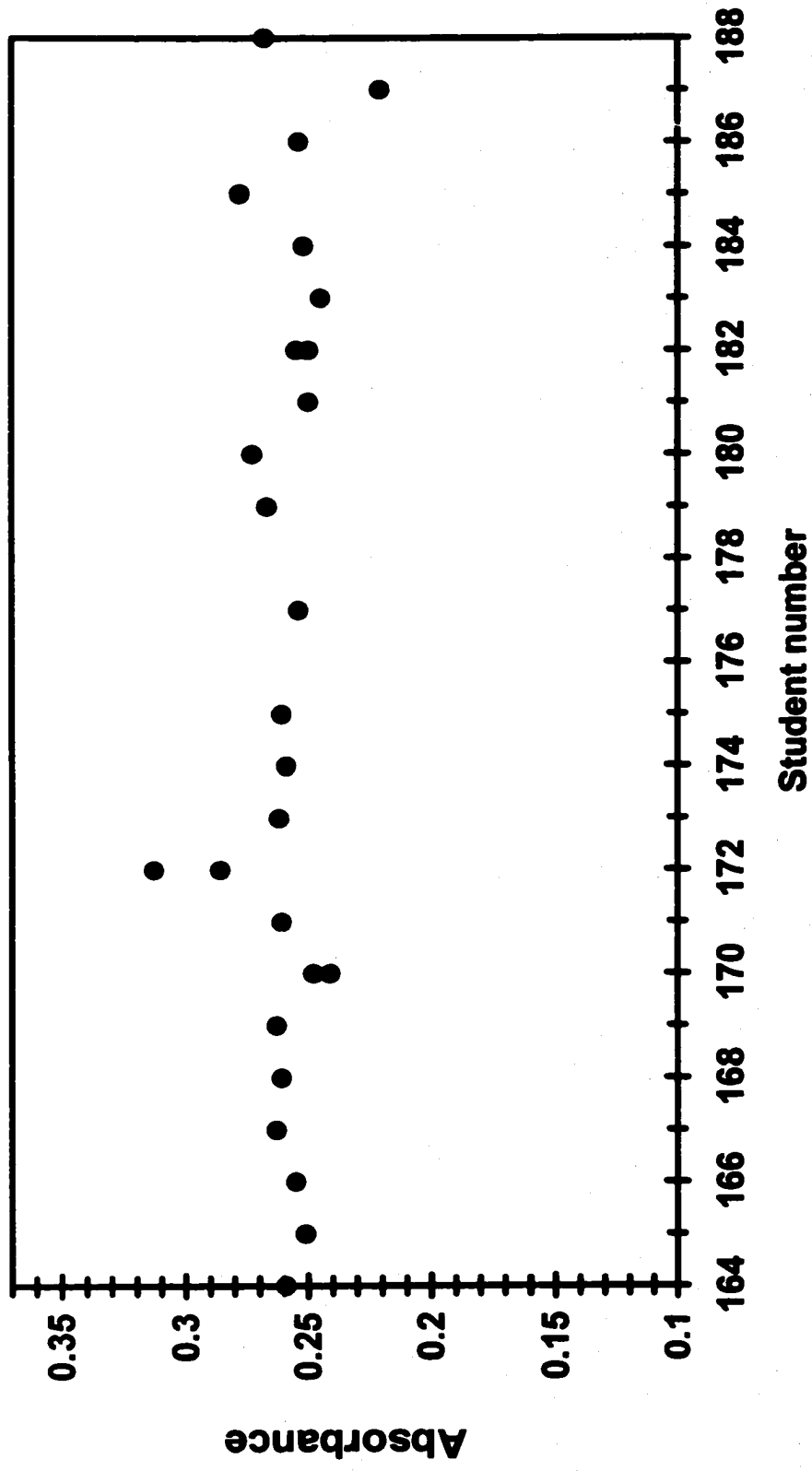


Osmometer Readings for 0.0600 M KCl, Fall 2000, View 2

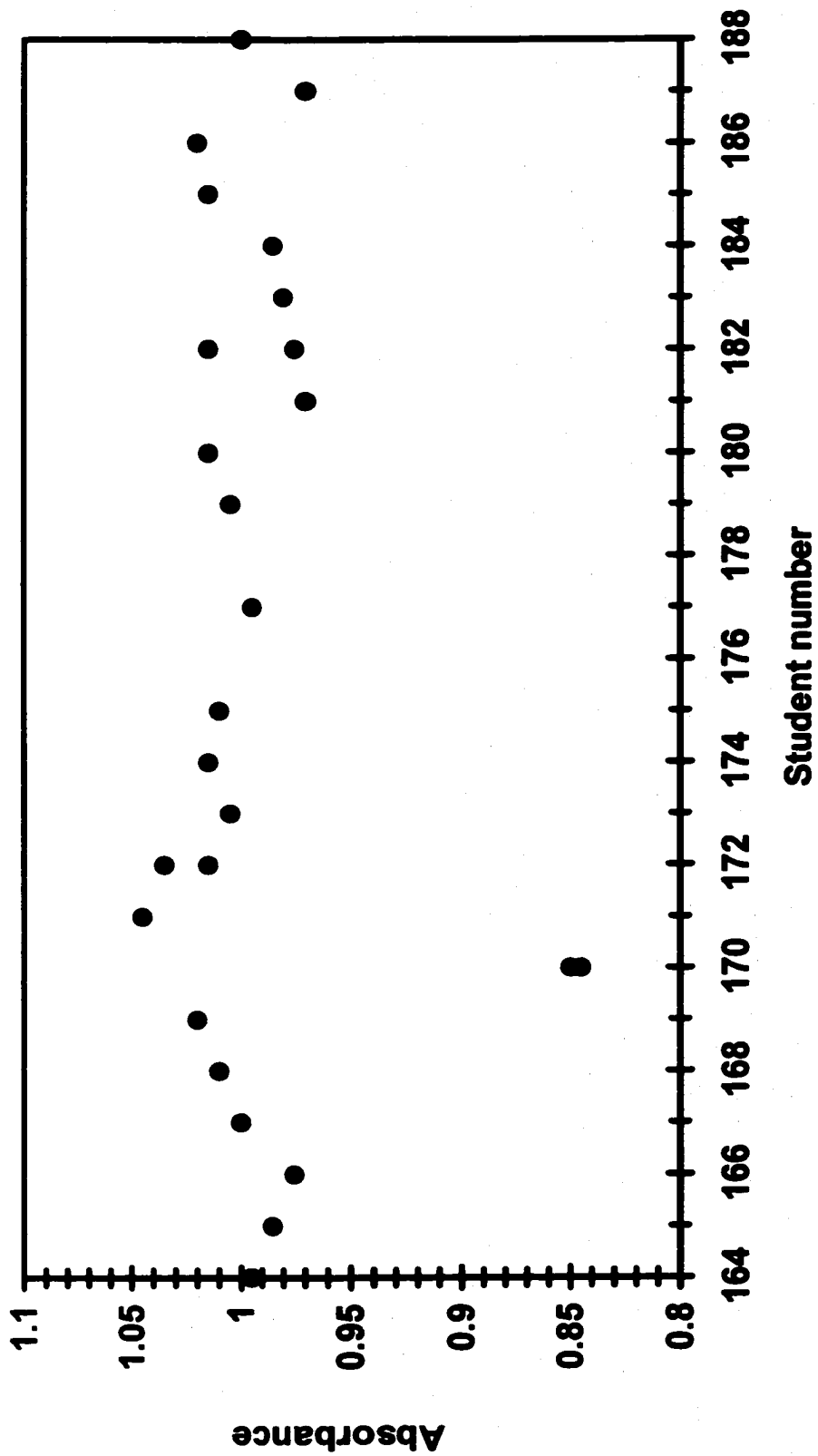
Appendix B6

Instrumental Reading Graphs for Spring Semester, 2001

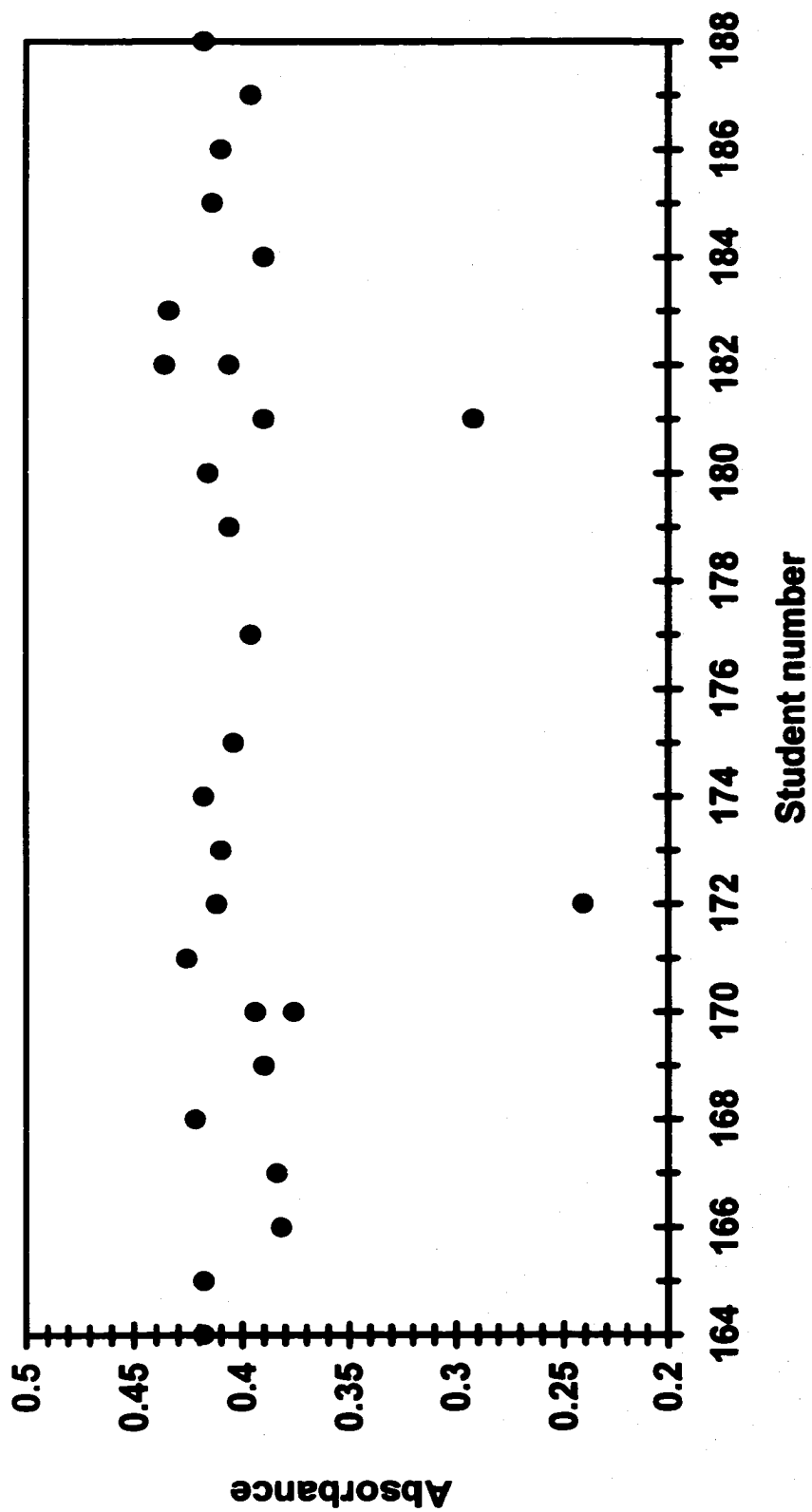
Spectronic 20 Data for 0.100M Ni(NO₃)₂ Solutions, Spring 2001

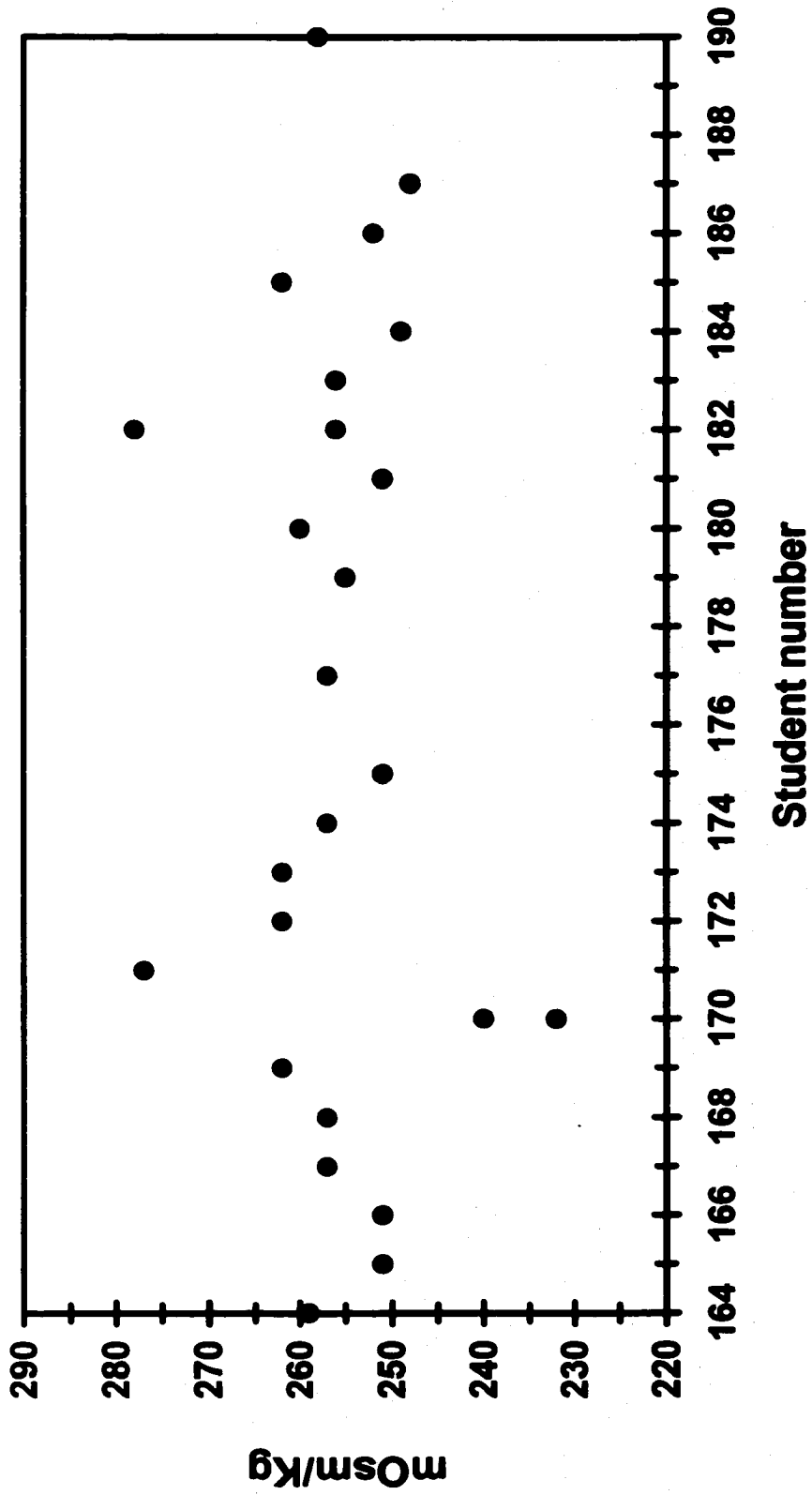


Spectronic 20 Data for 0.0500M Ni(NO₃)₂ Solutions, Spring 2001

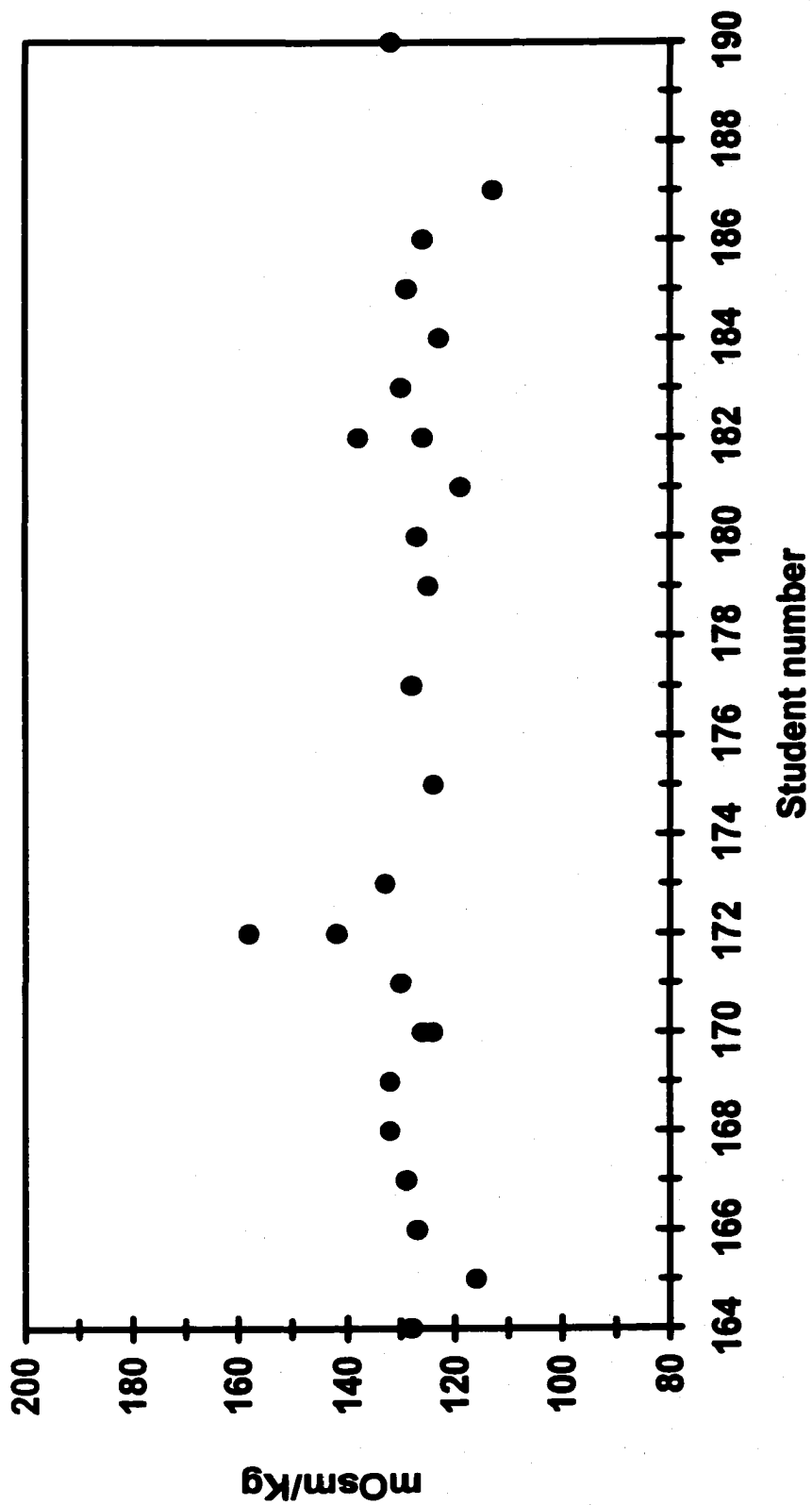


Spectronic 20 Data for 0.200M Ni(NO₃)₂ Solutions, Spring 2001

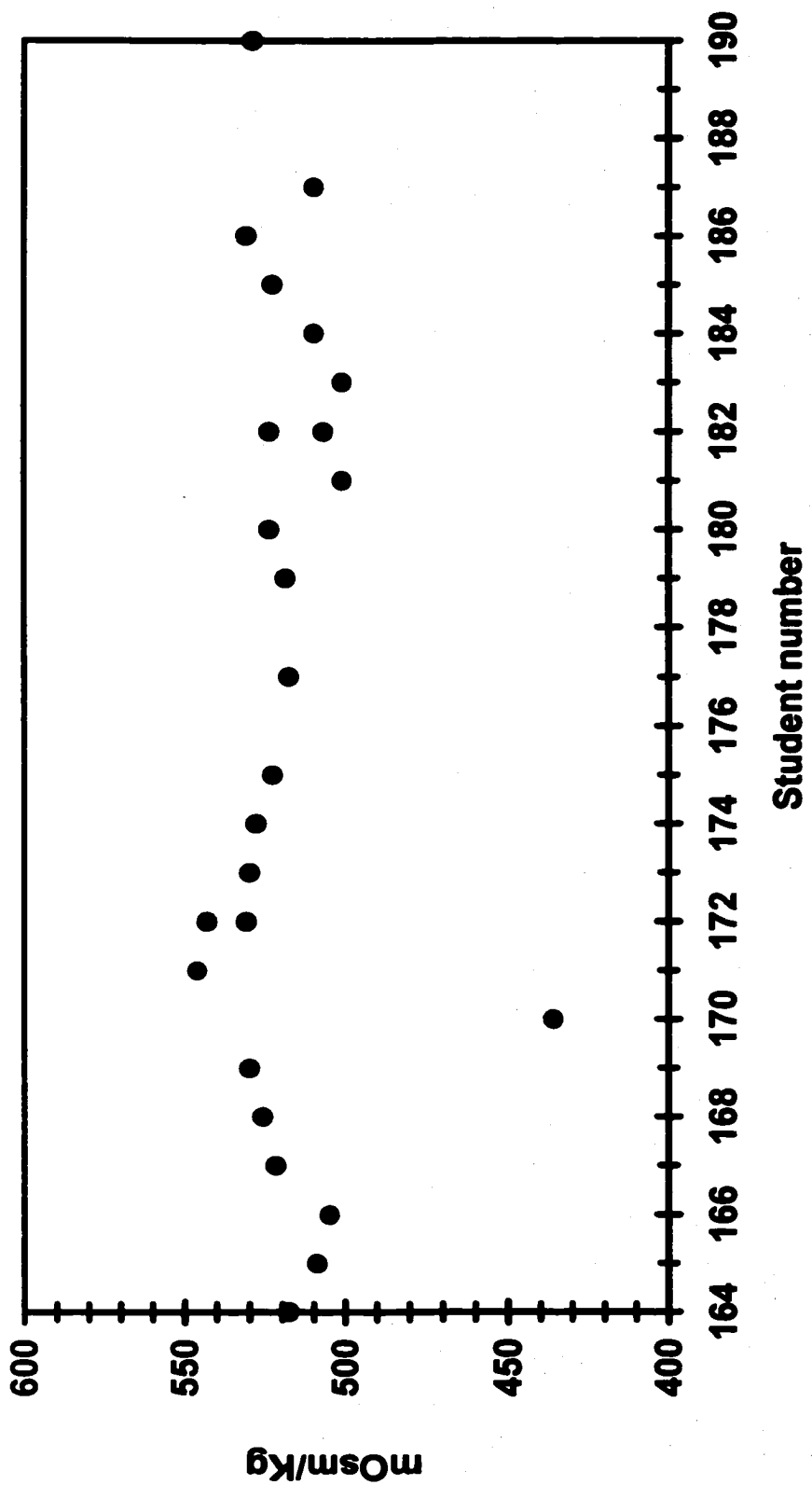
Spectronic 20 Data for 0.0800M Ni(NO₃)₂ Solutions, Spring 2001, View 2



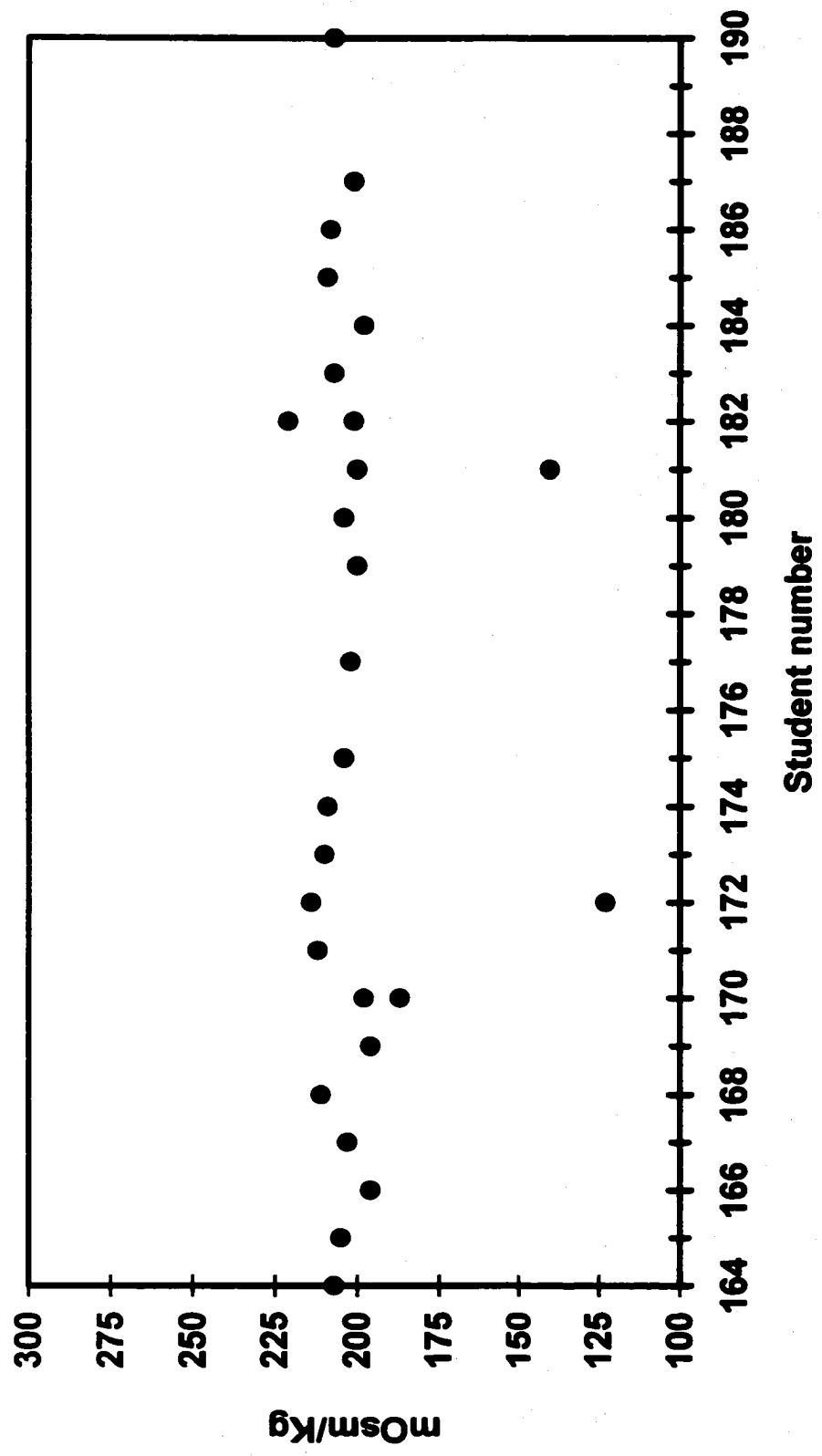
Osmometer Readings for 0.100M Ni(NO₃)₂ Solutions Spring 2001



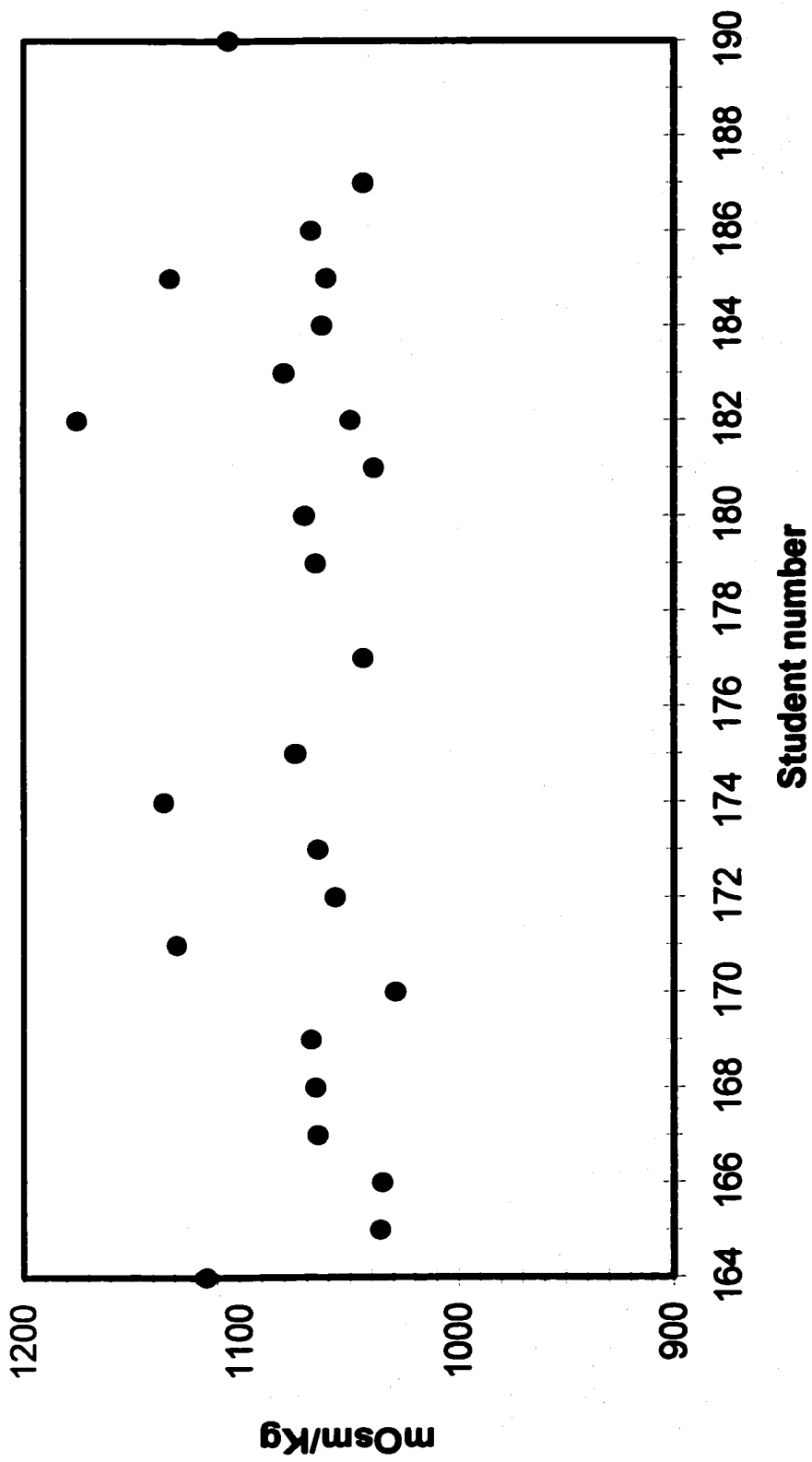
Osmometer Readings for 0.0500M Ni(NO₃)₂ Solutions Spring 2001



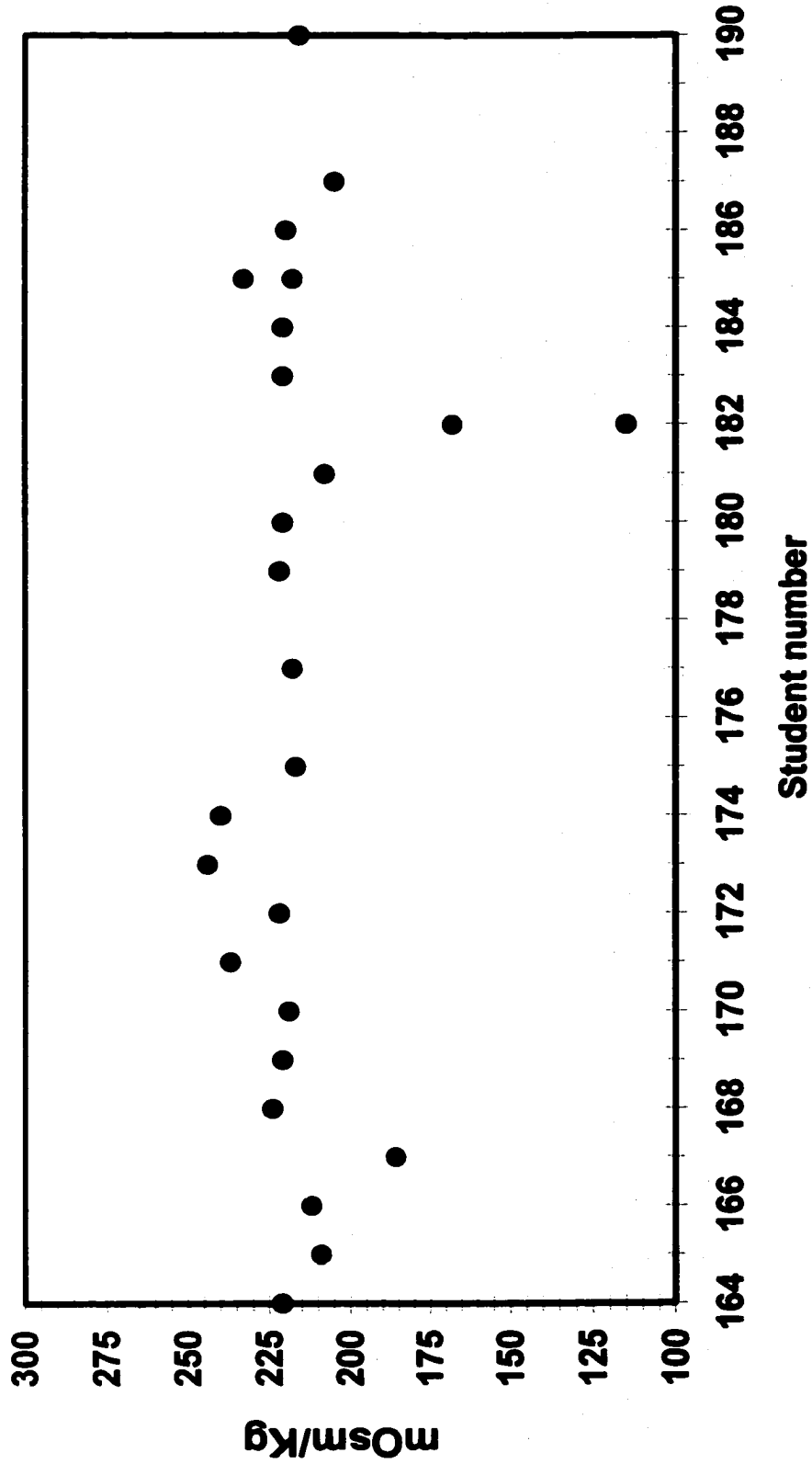
Osmometer Readings for 0.200 M Ni(NO₃)₂ Solutions, Spring 2001



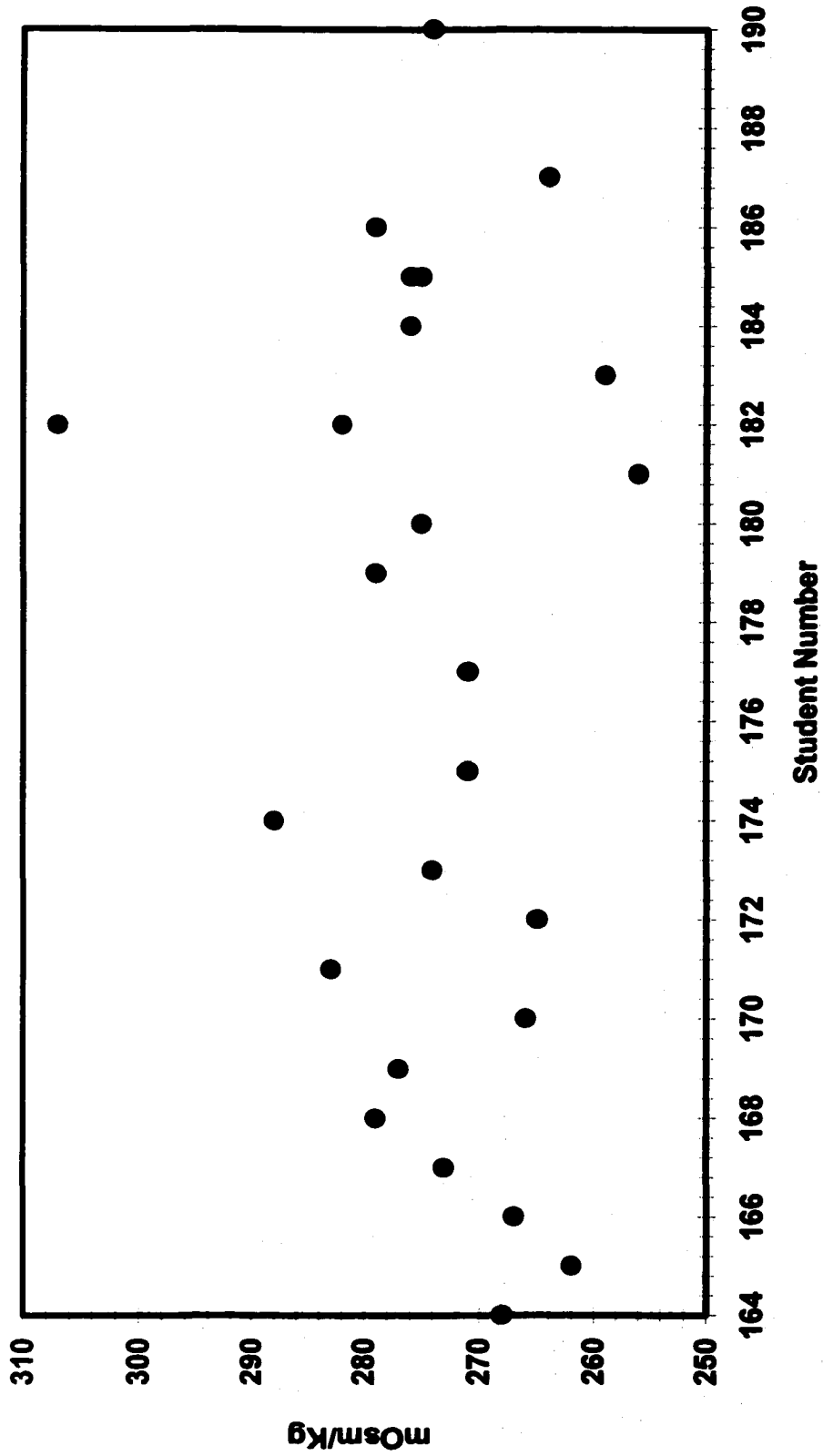
Osmometer Readings for 0.0800M Ni(NO₃)₂ Solutions, Spring 2001



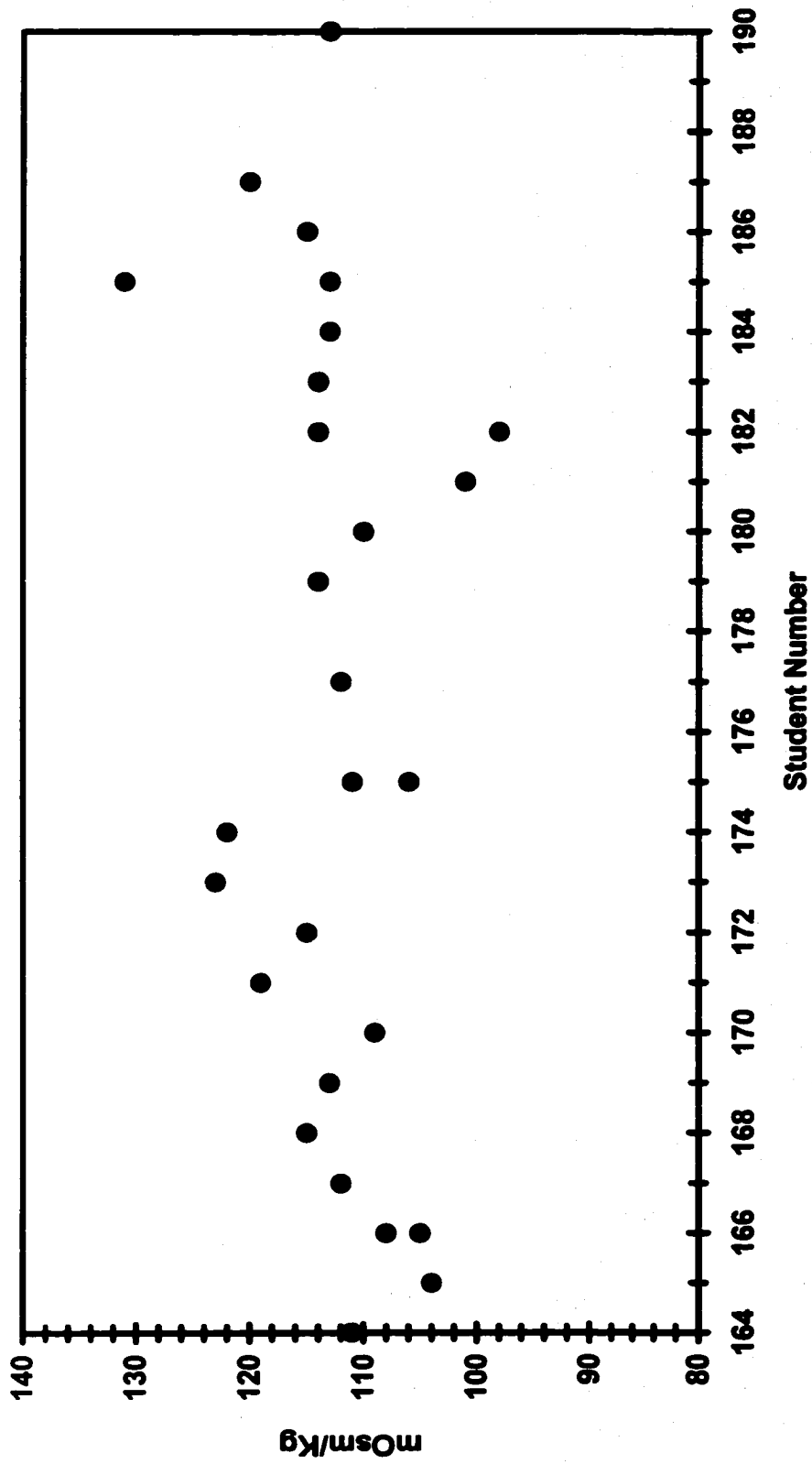
Osmometer Readings for 0.600 M KCl, Spring 2001, View 1



Osmometer Readings for 0.1200 M KCl, Spring 2001, View 1



Osmette Readings for 0.1500M KCl, Spring 2001



Osmette Readings for 0.0600M KCl, Spring 2001

Appendix C

Demographic Questionnaire

Chemistry 222 Questionnaire

The analytical chemistry faculty constantly redesign our courses to make them fit your needs better. Sometimes the changes are small; but sometimes they are extensive. For example, the changes might involve rescheduling laboratory or lecture times, or even changing the topics we cover in class or the kind of lab experiments we do. The questions below are designed to assess your needs. Please spend a few minutes assisting us to make Quant more useful to you.

Name _____ SS# _____

What is your major? _____ Minor? _____

Check the courses you have completed or in which you are currently enrolled.

		Completed	Currently Enrolled		Completed	Currently enrolled
Gen	Chem 121	_____	_____	Pchem	Chem 421	_____
Gen	Chem 122	_____	_____	Pchem	Chem 422	_____
Research I	Chem 250	_____	_____	Instrumental	Chem 423	_____
Organic	Chem 303	_____	_____	Adv Org	Chem 424	_____
Organic	Chem 321	_____	_____	Biochem	Chem 425	_____
Organic	Chem 322	_____	_____	Adv Inorg	Chem 426	_____
Organic	Chem 324L	_____	_____	Polymers	Chem 428	_____
Biochem	Chem 325	_____	_____	Adv Pchem	Chem 429	_____
Research II	Chem 350	_____	_____	Biochem II	Chem 435	_____
Polymers	Chem 400	_____	_____	PS Research	PSci 400	_____
				Research	Chem 450	_____
Pchem	Chem 401	_____	_____	Environmen	Chem 470	_____
Pchem	Chem 402	_____	_____	Detec of	Chem 471	_____
Inorganic	Chem 416	_____	_____	Chem Pollu		
				Other course	Chem _____	_____

What is you class? (circle one) Freshman Sophomore Junior Senior Masters

Do you live on campus? Yes _____ No _____ Campus or local phone number: _____

What is your e-mail address? _____

Do you work at a paid job on campus? Yes _____ No _____ Hours/week? _____

Off campus? Yes _____ No _____ Hours/week? _____

When do you work? Mostly mornings _____. Mostly afternoons (<5:00pm) _____.
Mostly evenings _____. Mostly weekends _____.

Does your work schedule conflict with times the laboratory is open? Yes _____ No _____

We need to know more about your work experience.

Have you ever held a full time job? yes _____ no _____

Have you worked part-time at the same job for several years? yes _____ no _____

Did your work require you to handle cash? yes _____ no _____

Did the job require careful measurements or meticulous handling of data? A carpenter job or pharmacy assistant's work would be examples. yes _____ no _____

What are/were some of the activities required by your full-time or long-term job?

Personal Information: Male _____ Female _____
(optional)

What is your age? _____ Your ethnic background? _____

Are you continuing your education after an absence for work, military service, or to start a family?
Yes _____ No _____

Or have you returned to the University to get a second degree? Yes _____ No _____

What was the major of your previous degree? _____

Do you have family responsibilities? Care of Children? Yes _____ No _____

Care of elderly family member? Yes _____ No _____

What is your marital status? (circle one) Single Married Separated Divorced Widowed

Name _____ SS# _____

General Preferences:

Which do you like better, working in pairs or working alone? Pairs _____ Alone _____

Would you like working on Quant experiments in an even larger group--say four or five students working on a project together? Yes _____ No _____

What days do you attend school or prefer to attend school? Check all that apply:
MWF _____ TuTh _____ All week _____ Weekends _____ Nights _____

Are there any days that would be hard or impossible for you to attend class or lab?

- Please discuss below: (1) Your plans after college (2) Your hobbies, sports, interests (3) Any present worries or problems I should know about (4) Any comments.

Appendix D
Exit Questionnaire

**PREPARATION OF STANDARD SOLUTIONS AND SERIAL DILUTION
EXPERIMENT**

Summative Evaluation Questionnaire

We would like to know your opinion of this experiment. Your answers to the following questions will assist us in revising and improving its content. Your response to this questionnaire is voluntary and your answers will be treated as confidential. Thank you.

1. In general, how well did this experiment help you focus on the important points in the course?

(Very well 5	Reasonably well 3	Poorly) 1
-----------------	----------------------	--------------

2. How would you rate the relative difficulty of this experiment compared to other experiments you have completed in this course (or other labs) this academic term?
(Least difficult Most difficult)

5	4	3	2	1
---	---	---	---	---

3. The activities in previous Quant experiments gave me sufficient practice to do this experiment.

(Strongly agree Agree Neutral Disagree Strongly disagree)

5	4	3	2	1
---	---	---	---	---

4. The content level of the experiment was appropriate for my level of experience.

(SA A N DA SDA)

5	4	3	2	1
---	---	---	---	---

5. Proper time was allocated to this experiment.

(SA A N DA SDA)

5	4	3	2	1
---	---	---	---	---

6. Which instrument was easier to use? Help us explain your choice.

(Spectronic 20)

(Osmometer)

7. Has your ability to prepare solutions improved as a result of this experiment?
 (Yes Somewhat Stayed the same No)
- 5 4 3 1

In general, how would you rate this experiment for the following points?

- | | <u>Excellent</u> | | | | <u>Poor</u> |
|--|------------------|---|---|---|-------------|
| 8. Was it interesting? | 5 | 4 | 3 | 2 | 1 |
| 9. Clarity of handouts | 5 | 4 | 3 | 2 | 1 |
| 10. Helpfulness of instruments | 5 | 4 | 3 | 2 | 1 |
| 11. Organization of experiment | 5 | 4 | 3 | 2 | 1 |
| 12. What did you like least about this experiment? | | | | | |

- 13.. What was the most significant thing you learned from this experiment?

Appendix E

Students' Comments and Responses to the Interviewer

The following is a sampling of students' comments and responses to the interview conducted during the process of the serial dilution experiment.

"This is my fourth trial and I'm wondering if I should use a pipette to measure for some accuracy here"

"Does it matter if I overshoot my meniscus while making the solutions, I mean, how close do I need to get it?"

"I don't recall using a pipette in the past, can you tell me what it looks like?"

"I hate using the pipette because the rubber bulb won't listen to me."

"I have no idea what I did wrong but I bet it had something to do with the flasks I used. The necks weren't big enough to pour from the beakers."

"I can't begin to tell you how much grief that graduated cylinder gave me, I thought I would never finish. You would think there would be a easier way to do this."

"This scares me--my meniscus was right on the mark this morning and now its dropped below the mark. Will evaporation affect my solution readings?"

"I hate pipetting--I used a graduated cylinder instead, that doesn't make a difference ...does it?"

"Isn't the eyedropper the same thing as a pipette?"

"What do these serial dilutions mean? How do I know if my solution is weak enough?"

"I think the cuvette is affecting my solution readings. It looks weaker in the cuvette than it does my flask."

"What difference does it make if I don't use a pipette? I'm used to a beaker. I used one to measure the 10 mLs for this solution."

“I didn’t invert the solutions; was I supposed to? But it looks like it is well mixed. As you can see, it’s turned green already.”

Student Comments after Instrument Feedback

“I am overjoyed that my numbers match Dr. Bonicamp’s. This means I did it perfect.”

“It was stressful for me, especially after I discovered my solutions were not exact-- apparently my precision skills need more practice.”

(The osmometry experiment) “Solidified aspects of colligative properties for me.”

“Using a pipette correctly requires a lot of skill”.

Appendix F**Permission from the Institutional Review Board**

Permission was obtained from MTSU's Institutional Review Board to use students as object of this research. A copy of this letter follows.



Department of Chemistry
Middle Tennessee State University
P O Box 68
Murfreesboro, Tennessee 37132
USA

Phone: (615) 898-2956
Fax: (615) 898-5182

MEMORANDUM

To: Nyanguila Kakolesha

From: George Devendorf 
Basic and Applied Representative to IRB

Date: February 10, 2000

Re: Research Proposal: "Serial Dilution of Solutions and Instrument Feedback"
IRB Protocol #: 00-153

The above named human subjects research proposal has been reviewed and approved. This approval is for one year only. Should the project extend beyond one year or should you desire a change in the research protocol in any way, you must submit a memo describing the proposed changes (including changes in proposed interview scripts, etc.) or reason for extension to your college's IRB representative for review. Best of luck in the successful completion of your research.

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