

CHM 152LL LAB POLICIES Gilletti Spring 2022



INSTRUCTOR: Dr. Paul Gilletti EMAIL: gilletti@mesacc.edu
OFFICE: Building 15 PS-209 PHONE: Office 480.461-7685
Web Page: <https://gillettichemistry.com> (Many of my
Powerpoint presentations and old quizzes and exams are
available)

As per MCCC CD rules, you are required to wear a mask in any building on any campus.

Office Hrs: Virtual by email appointments. Monday: 11:00-11:50 live in PS225 and virtual 2:00-2:50 appointment required, Tuesday 1:30-2:30 virtual or in person, Wednesday 2:30-3:30 virtual, Thursday 1:30-2:30, Friday (appointment only).

To make an appointment, email me through canvas at paudy84101@mesacc.edu. Please indicate your questions and/or problems and times during which you will be available. If possible, a screenshot of your question may help. I will respond as soon as possible with an appointment. Conferences can then be setup **On-line (Webex or Zoom)**.

Experiments: You must download and print experiments from the lab manual before you come to class. Please download all labs and save to hard drive, thumb drive or CD at the beginning of the semester so that if our server is down, you will still be able to print out your lab when you need them. Download files from canvas Modules. There is material to help with the labs on my web page: <https://gillettichemistry.com>

Schedule: You will be given a schedule, but the experiment schedule is also available on the above web site.

CELL PHONES: MAY NOT BE USED DURING CLASS, NOT EVEN TEXTING.

Quizzes may be given at the beginning of the period. The quizzes will be a combination of prelab (from the lab to be performed) and post lab (from the lab just handed in) questions.

LAB NOTEBOOKS: You will be required to keep a laboratory notebook. **Specific details are listed on the on-line laboratory manual site**, but a general set of instructions are given here. There is a sample lab notebook in the instrument lab in the back of our lab. It is a good example of how a notebook should be kept.

- | |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1. Before coming to lab you must print out and read the experiment. <u>Then do items 2-6 BEFORE coming to lab.</u> |
| 2. Create a table of contents at the beginning of your lab manual and keep track of pages used by listing titles and page numbers in the lab manual. The table of contents must be kept up to date each week. |
| 3. On the starting page of each experiment in the laboratory manual, write a 1-3 sentence purpose statement. |
| 4. List the MSDS information for the lab. |
| 5. Write a brief experimental procedures section. |
| 6. Create tables for the data you plan to collect, <i>if you are unsure, wait and do during lab as needed.</i> |
| 7. Before you leave the lab, obtain the instructor's signature. |
| 8. Leave blank pages to perform examples of each calculation. (This will prove to be very useful on the OPEN LABORATORY NOTEBOOK final exam) |

9. YOUR LABORATORY NOTEBOOK WILL BE GRADED ON ORGANIZATION AND DATA TAKING AND GENERAL RECORD KEEPING!

LAB DUE DATES: Each lab will be due at the **BEGINNING** of the lab meeting on the ***scheduled report due date*** or it will be considered late. If you encounter problems with the write-up please see me **BEFORE IT IS DUE**. It is strongly suggested that you **begin writing up your lab as soon as possible** and while the lab is fresh in your mind, and allowing time to get help as needed.

DATA should be recorded in **INK DIRECTLY** in your laboratory notebook. Changes are made by drawing a single line through the data, placing your initials near the cross out, and then writing correct data.

Example: ~~0.1503 cm~~ PFG 0.1603 cm

All numbers less than 1 must begin with a 0 not just a decimal point.

Example: 0.5 is correct not .5 (There have been cases of patients dying due to doctors having written a prescription with just the decimal and not the 0.xx).

Significant figures in data and calculations will be graded so be sure to use all significant digits (i.e. 5.0 vs 5 or 5.00).

****BEFORE you leave the lab, you must obtain the instructor's initials on your data.**

ACADEMIC DISHONESTY POLICY: See the current MCCD student handbook on the academic dishonesty policy. Academic dishonesty may include: representation of the work of other's as one's own, use of unauthorized assistance in academic work, failure to cite sources used, copying the work of another student on any form of a test, helping others cheat, etc. Repercussions can be found in the student handbook and range from a warning to dismissal from the course with a failing grade.

WITHDRAWAL: See your student schedule in *my.maricopa.edu* for the Last Day to Withdraw without an Instructor Signature for each class in which you are enrolled (This is the first seven weeks from when the class started). After that time your instructor's signature is required. (Refer to the Important Deadlines for Students to determine the Last Day Student Initiated Withdrawal will be accepted.) . Either a withdrawal passing (W) or a withdrawal failing (Y) may be given, based upon student performance***.

STUDENTS WITH DISABILITIES: If you have a documented disability, including a learning disability, and would like to discuss possible accommodations, please contact the MCC Disabilities Resources and Services Office at [480-461-7447](tel:480-461-7447) or email drsfrontdesk@mesacc.edu.

Students with disabilities must have an equally effective and equivalent educational opportunity as those students without disabilities. Students experiencing difficulty accessing course materials because of a disability are expected to contact the course instructor so that a solution can be found that provides all students equal access to course materials and technology.

Information for Pregnant or Parenting Students: If you are a pregnant or parenting student you are protected under Title IX regarding classroom accommodations. Please request your accommodations through the MCC Disabilities Resources and Services Office at [480-461-7447](tel:480-461-7447) or email drsfrontdesk@mesacc.edu.

MCC Early Alert Program (EARS)

Mesa Community College is committed to the success of all our students. Numerous campus support services are available throughout your academic journey to assist you in achieving your educational goals. MCC has adopted an Early Alert Referral System (EARS) as part of a student success initiative to aid students in their educational pursuits. Faculty and Staff participate by alerting and referring students to campus services for added support. Students may receive a follow up contact from various campus services as a result of being referred to EARS. Students are encouraged to participate, but these services are optional.

Early Alert Web Page with Campus Resource Information can be located at:

<http://www.mesacc.edu/students/ears> or

On the MCC Home Page, click on "Current Students" and then click on EARS Student Resources under "Services" which will take you to the EARS Web Page including the MCC Resources Page.

Note: There are two sample lab reports on line. One is on my web page, the other is a link in the CHM152LL schedule page.

GRADING:

Each Lab is worth 50 Pts. Formal lab reports are required. See the on-line lab manual and the handout for instructions on writing formal reports. Quizzes = 10 points each.

Lab Notebook Quality (graded at the end of the semester) = 50 Pts.

Lab final = 100 points. (~600 total points)

Late 1 day: -2.5 points Late 2 days: -5 points Late 3 days: -10 points
Late 4 days: -20 points Late 5 days: -40 points Late 6 days: KEEP IT!

90-100 A; 80-89 B; 70-79 C; 60-69 D; <59 F.

(Note, missing a lab = at least 50 points off grade total and lowers your grade by ~ 10%, which is a FULL LETTER GRADE.) It behooves you to not miss any labs, see me if you must miss or have missed a lab.

Missing or failing to turn in three labs will result in an F or W for the course.

Numerical Grade Comments for lab reports: *These are SOME of the things that I look for when I grade your report. Usually I will just write these numbers on your report for you to reference.*

No.	Comment
1	*****Refer to the format of the sample article.
2	Abstract is missing. The abstract should summarize the experiment <u>and the results obtained</u> . The abstract is the last thing you write but is presented first. It should only be 3 to 5 sentences long. Be sure to include <u>chemical equations</u> and your unknown numbers.
3	Grammar. Sentence needs to be reworded.
4	Verb tense is not consistent
5	Define abbreviations when first introduced
6	Use proper format for footnotes and references
7	Don't write in first person (no I or we)
8	Significant figures. Read and record instruments to the proper place (one place past smallest markings on the instrument) and use sig. fig. rules in reporting calculation results.
9	Write out chemical equations for all reactions and number the equations in sequential order throughout the paper.
10	Safety information and MSDS data is missing
11	State your Unknown number
12	Data table must be typed and have a title (i.e. Table 1. Time (s) and Concentration (M))
13	Set up is correct, but there is a calculation error.
14	What are the units?
15	Okay based, on the above error
16	Stoichiometry, look at ratios in the equations
17	Good job!
18	Calculate the percent error.
19	Graph needs a title (i.e. Figure 1. Time vs. Distance)
20	Label graph axis.
21	Scale the graph properly
22	Attach carbon copies from lab manual as appendices
23	Spelling!
24	Written as mL not ml
25	Conclusion: Discuss the CHEMISTRY involved and the results obtained in the experiment (including numerical values). Also compare your results to any accepted values available.
26	Does your answer(s) make sense?



Note

Kinetics of the molybdate and tungstate catalyzed oxidation of iodide by hydrogen peroxide

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United States Naval Academy, Annapolis, MD 21402, USA

Received 27 November 1998; accepted 4 February 1999

Abstract

Several studies of the Mo(VI) and W(VI) catalyzed reaction of H_2O_2 with various substrates have been documented previously. The experiments described herein attempt to settle some apparent contradictions in these reports. Specifically, the oxidation of I^- with H_2O_2 in the presence of molybdate or tungstate ion in an acid solution was studied. The rate law for this system was found to be $rate_i = k_{cat}[MoO_4^{2-}][I^-] + k_{uncat}[H_2O_2][I^-]$ with values of $k_{cat} = 3.87 M^{-1} s^{-1}$ and k_{uncat} of $9.5 \times 10^{-3} M^{-1} s^{-1}$ (at 22.8°C). The values of ΔH^\ddagger for the molybdate catalyzed and uncatalyzed reactions were found to be 39.4 and 56.8 kJ mol $^{-1}$, respectively. Similarly, the value of k_{cat} was found to be 32.7 M $^{-1} s^{-1}$ and ΔH^\ddagger was found to be 41.1 kJ mol $^{-1}$ (at 21.8°C) for the same system catalyzed with tungstate rather than molybdate. © 1999 Elsevier Science S.A. All rights reserved.

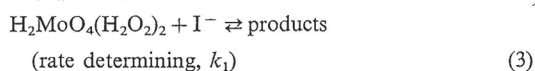
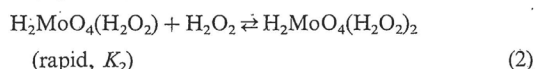
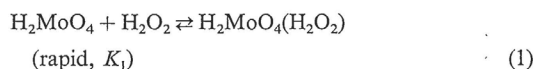
Keywords: Catalysis; Iodide oxidation; Hydrogen peroxide; Molybdate; Tungstate

1. Introduction

Several studies of the Mo(VI) and W(VI) catalyzed reaction of H_2O_2 with various substrates have been reported [1–8]. One of the first detailed studies was that of the oxidation of I^- with H_2O_2 in the presence of molybdate ion in an acid solution. This study by Garcia and Lara [1] found that, in 0.08 M H_2SO_4 solution, the rate law for the reaction is:

$$rate = k[MoO_4^{2-}]^{0.5}[H_2O_2]^{0.5}[I^-]^{1.2}$$

Later, Smith and Kilford [2] studied the same reaction with I^- and H^+ in excess over H_2O_2 . They found that under these conditions the reaction was neither first nor second order in H_2O_2 . A slight dependence on H^+ was found in the range 0.025–0.20 M. They concluded that their results could best be explained by the sequence:



with $k_1 = 3.3 \times 10^2 M^{-1} s^{-1}$. No activation parameters were given. Studies by Thompson and co-workers [6] have also shown that the formation of the proposed peroxy complexes have large formation constants.

Recently, it has been reported [7] that the reaction between H_2O_2 and an excess of I^- in the presence of molybdate in an acid solution follows the rate law:

$$rate = k_2[Mo(VI)][H_2O_2]$$

with the formation of either a mono or diperoxy complex being the rate determining step which is followed by the rapid oxidation of I^- .

We have decided to investigate this apparent contradiction by employing an iodide clock technique to measure the true initial rate of reaction. We have

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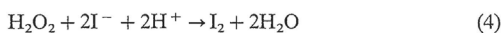
Table 1
Volumes of reagents (ml added to 25.0 ml buffered $\text{Na}_2\text{S}_2\text{O}_3$ stock solution) and reaction times from varying $[\text{I}^-]$ and $[\text{H}_2\text{O}_2]$

Flask number	0.060 M KI	Distilled H_2O	0.040 M H_2O_2	2.0×10^{-3} M Na_2MoO_4	Reaction time (s) at 22.8°C
1a	25	29	20	1.0	55.8
1b	25	30	20	0.0	113.7
2a	25	19	30	1.0	45.0
2b	25	20	30	0.0	78.6
3a	25	0	50	1.0	33.1
3b	25	0	50	0.0	47.3
4a	15	9	50	1.0	56.1
4b	15	10	50	0.0	79.9
5a	10	14	50	1.0	83.0
5b	10	15	50	0.0	118.0

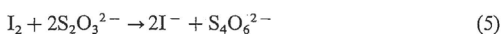
chosen to work in an acetate buffered (pH 4.5) medium where there is little interference by the acid promoted, uncatalyzed (by MoO_4^{2-}) reaction [9,10].

2. Experimental

The reaction studied is a variation of the familiar iodine clock reaction. The reaction is:



For each reaction mixture studied, the initial reaction rate was determined by adding starch and a small, known quantity of sodium thiosulfate to the iodide ion solution before adding hydrogen peroxide. Upon mixing the hydrogen peroxide with the iodide ion solution, the molecular iodine initially produced Eq. (4) is immediately consumed by the thiosulfate according to the reaction:



However, after the small, known quantity of thiosulfate is exhausted, the iodine produced will complex with the starch resulting in a blue color in the reaction solution. The interval between final reagent mixing and the first appearance of a blue color throughout the solution provides the time data for the mixture.

2.1. Stock solutions

Since the rate of reaction of hydrogen peroxide with iodide ion is greatly influenced by small amounts of contaminants, care must be taken to insure the cleanliness of all glassware.

A buffer-starch stock solution of $\text{Na}_2\text{S}_2\text{O}_3$ was used in all reactions. It was made by combining 25 ml of 0.01 M $\text{Na}_2\text{S}_2\text{O}_3$, 25 ml of buffer (1 M $\text{HC}_2\text{H}_3\text{O}_2$ and 1 M $\text{NaC}_2\text{H}_3\text{O}_2$, pH 4.5), 25 ml of a 2% starch solution, and 175 ml of distilled water.

2.2. Timed reactions

2.2.1. Order in H_2O_2 and I^-

To determine the order in H_2O_2 , 25 ml of the $\text{Na}_2\text{S}_2\text{O}_3$, buffered, starch stock solution was transferred to a 250 ml Erlenmeyer flask followed by a constant amount of Na_2MoO_4 and KI solution. Distilled water was added as indicated in Table 1. To begin each reaction, varying amounts of H_2O_2 solution were added, quickly mixed and the time noted. At the first appearance of the blue starch- I_2 complex, the time was again noted. From this time and the change in thiosulfate concentration (from 2.5×10^{-4} to 0 M), the overall initial rate of each reaction was determined. By

Table 2
Catalyzed rate^a dependence upon H_2O_2 and I^- at pH 4.5, 22.8°C, $[\text{MoO}_4^{2-}] = 2.0 \times 10^{-5}$ M

$[\text{H}_2\text{O}_2]_i$ (M)	$[\text{I}^-]_i$ (M)	10^{-6} Catalytic rate ($\text{M}^{-1} \text{s}^{-1}$)
0.008	0.015	1.14
0.012	0.015	1.19
0.020	0.015	1.14
0.020	0.009	0.67
0.020	0.006	0.44

^a Total rate-uncatalyzed rate = catalyzed rate.

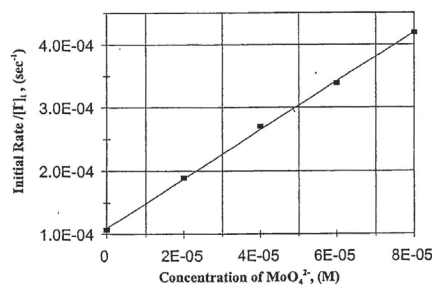


Fig. 1. Dependence of initial rate on MoO_4^{2-} concentration at constant $[\text{H}_2\text{O}_2]$ ($= 0.1155$ M) and 22.8°C.

subtracting the uncatalyzed rates (those obtained from the b flasks) from the overall rates (those obtained from the a flasks), one obtains the initial catalyzed rates of reaction. These initial rates for the Mo(VI) catalyzed reactions, along with initial concentrations are given in Table 2.

2.2.2. Order in Mo(VI) and W(VI)

The catalyzed reactions were run as before except that varying amounts of MoO_4^{2-} (or WO_4^{2-}) were added (Table 3). In the case of the rapid WO_4^{2-} catalyzed reactions, it was found that it was best to add the Na_2WO_4 solution to the H_2O_2 solution before mixing.

The above processes were repeated for the catalyzed and uncatalyzed reactions at a low temperature. To maintain the low temperature, the flasks containing all reagents were kept in a constant temperature bath prior to use. Once the iodine clock reactions were begun, the flasks were still kept at a constant, low temperature. No attempt was made to maintain conditions of constant ionic strength as changes in ionic strength are reported to have no effect upon the rate of reaction [2,7].

3. Results and discussion

Our results (given in Tables 2 and 3) show that the rate of I^- oxidation at pH 4.5 in the presence of small catalyst quantities of MoO_4^{2-} can be expressed by the rate law:

$$\text{rate}_i = k_{\text{cat}}[\text{MoO}_4^{2-}][\text{I}^-] + k_{\text{uncat}}[\text{H}_2\text{O}_2][\text{I}^-]$$

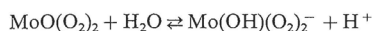
Plots of the initial rate/ $[\text{I}^-]_i$ versus $[\text{MoO}_4^{2-}]$ at constant $[\text{H}_2\text{O}_2]_i$ (see Fig. 1) were linear ($r^2 = 0.9996$) with a slope = $k_{\text{cat}} = 3.87 \text{ M}^{-1} \text{ s}^{-1}$ and intercept = 1.1×10^{-4} at 22.8°C . Dividing the intercept by $[\text{H}_2\text{O}_2]_i$ gives a value for k_{uncat} of $9.5 \times 10^{-3} \text{ M}^{-1} \text{ s}^{-1}$ which is in excellent agreement with the value in the literature [9]. Data obtained at 0.3°C also proved linear ($r^2 = 0.9997$) with a slope = $k_{\text{cat}} = 1.03 \text{ M}^{-1} \text{ s}^{-1}$ and an intercept = 1.64×10^{-5} . Dividing the intercept by $[\text{H}_2\text{O}_2]_i$ gives a value for $k_{\text{uncat}} = 1.42 \times 10^{-3} \text{ M}^{-1} \text{ s}^{-1}$. These results yield a ΔH^\ddagger of 39.4 kJ mol^{-1} for the molybdate catalyzed reaction and a ΔH^\ddagger for the uncatalyzed reaction of 56.8 kJ mol^{-1} . While the former result is in poor agreement with the value reported by Karunakaran and Muthukumaran [7] of 20.6 kJ mol^{-1} , the later is in good agreement with the value reported (53.8 kJ mol^{-1}) by Liebafsky and Mohamud [9]. Similar results were obtained with the W(VI) catalyzed reaction between I^- and H_2O_2 (Table 3). k_{cat} at 21.8°C was $32.7 \text{ M}^{-1} \text{ s}^{-1}$ ($r^2 = 0.9970$) while at 0.4°C k_{cat} was $8.8 \text{ M}^{-1} \text{ s}^{-1}$ ($r^2 = 0.9970$). This yields a ΔH^\ddagger for the catalyzed reaction of 41.1 kJ mol^{-1} . It should be noted that the Mo(VI) catalyzed reaction has a slightly lower activation energy even though the W(VI) is a superior catalyst. Thus the kinetic advantage

Table 3
Volumes of reagents (ml added to 25.0 ml buffered $\text{Na}_2\text{S}_2\text{O}_3$ stock solution) and reaction times (s) for MoO_4^{2-} and WO_4^{2-} Studies

Flask number	0.060 M KI	Distilled H_2O	0.040 M H_2O_2	2.0×10^{-3} M Na_2MoO_4	Reaction time at 22.8°C	Reaction time at 0.3°C	2.5×10^{-4} M Na_2WO_4	Reaction time at 21.8°C	Reaction time at 0.4°C
<i>Catalyzed</i>									
1	25	19	30	MoO_4^{2-} 1	44.0	324	WO_4^{2-} 1	43.7	210
2	25	18	30	2	30.9	251	2	28.6	130
3	25	17	30	3	24.6	219	3	22.0	98.5
4	25	16	30	4	19.9	195	4	18.8	80.5
<i>Uncatalyzed</i>									
5	25	20	30	0	77.9	480			

of W(VI) has over Mo(VI) must be due to a more favorable activation entropy. This result has been observed previously on Mo(VI) and W(VI) catalyzed reactions of H_2O_2 [3]. We are unable to offer a suitable explanation for this result. It should be noted that the k_{uncat} for the uncatalyzed reactions obtained from this W(VI) plot is again in excellent agreement with the values in the literature [9].

One item to be noted is that our k_{cat} for the Mo(VI) catalyzed reaction is smaller than that reported by Smith and Kilford [2]. This may be due to the differences in the pH and the reaction:

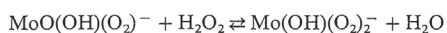


At higher pH values the species $\text{Mo}(\text{OH})(\text{O}_2)_2^-$ should be favored. Ghirm and Thompson have shown this species to be a much less reactive species than $\text{MoO}(\text{O}_2)_2$ [3]. This lowering of the rate of Mo(VI) peroxide catalyzed reaction at higher pH has also been noted before [11].

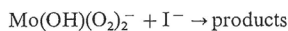
Thus, our results are essentially in agreement with the mechanism originally proposed by Smith and Kilford, i.e. at pH 4.5:



(rapid, K_1) (6)

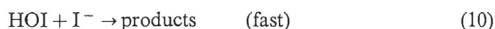
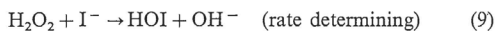


(rapid, K_2) (7)



(rate determining) (8)

along with the uncatalyzed reaction



However, our rate law is somewhat less complex than those reported earlier [1,2]. The reason for this appears to be, that under the conditions we have chosen, $[\text{H}_2\text{O}_2] \gg [\text{Mo}(\text{VI})]$, the equilibrium proposed in steps 1 and 2 always lie far to the right. This keeps the concentration of the active catalytic species constant and equal to $[\text{MoO}_4^{2-}]_i$. This greatly simplifies the rate law for the catalytic path. This large excess of H_2O_2 does lead to a large contribution to the overall rate by the uncatalyzed pathway: so much so that at times, the

uncatalyzed pathway proceeds at a faster rate than the catalyzed pathway. For example, it can be noted that in flask 5 (Table 1) the catalyzed rate is $0.45 \times 10^{-6} \text{ M}^{-1} \text{ s}^{-1}$ while the uncatalyzed rate is $1.05 \times 10^{-6} \text{ M}^{-1} \text{ s}^{-1}$. This is a direct result of the small quantity of catalyst used.

One last item to be addressed is the rate law and ΔH^\ddagger reported by Karunakaran and Muthukumaran [7]. As mentioned previously, they reported

$$\text{rate} = k[\text{Mo}(\text{VI})][\text{H}_2\text{O}_2] \quad \text{with } \Delta H^\ddagger = 20.6 \text{ kJ mol}^{-1}$$

We believe the reason for the difference in their results and those being reported here, is due to the different initial conditions. They used a large excess of I^- and small quantities of H_2O_2 and Mo(VI). Apparently, under these conditions, the formation of the active Mo(VI) species is rate limiting. This leads to a rate law that is first order in H_2O_2 and zero order in I^- . Our conditions are $[\text{I}^-] \approx [\text{H}_2\text{O}_2] \gg [\text{Mo}(\text{VI})]$. This favors the rapid formation of the catalytic species ($\Delta H^\ddagger = 20.6 \text{ kJ mol}^{-1}$) and makes step (8) ($\Delta H^\ddagger = 39.4 \text{ kJ mol}^{-1}$) rate limiting.

We have also investigated the effect of Mo(VI) upon the reaction of t-butyl hydroperoxide and peroxydisulfate ion with I^- and have found no catalysis. This result is consistent with the above proposed catalytic complex ions with two η^2 equatorial peroxo ligands [12].

References

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CHEMISTRY INFORMATION

Please complete and return at the time of the next class meeting.

Semester: Fall Year: 2021 Course: CHM152LL Section: 22975

Name: _____ Phone: _____

e-mail: _____

Major **and** CAREER Goal*: _____

Number of Hours You Are Working per Week (Please consider family responsibilities and volunteer hours when considering your course load below): _____

Number of Credit Hours You are Taking: _____

Previous **Chemistry** Courses Taken: Date Taken Name and/or Location of School

High School Course(s): _____

College **Chemistry** Course(s) : _____

Highest Level Math you have completed: _____

Math Currently being taken: _____

Future Chemistry courses that you are planning to take at MCC: _____

Write a brief statement detailing the combination of work ethic, ability, time commitment, and grades that are required to succeed in the major and career goal that you listed above.

I have received the syllabus and am responsible for its content _____ (signature)