# Complexation of Molybdenum(VI) with Ethylenediaminediacetic Acid in Different Water + Methanol Solutions[†]

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The stability constants for the reaction of molybdenum(VI) with ethylenediaminediacetic acid (EDDA) and protonation constants were determined at $T = 298$ K and constant ionic strength of 0.1 mol·dm$^{-3}$ sodium perchlorate in five different compositions of water + methanol by a combination of potentiometric and UV spectrophotometric methods. Stability and protonation constant variations versus the composition of different water + methanol mixtures have been reported, and their values have been calculated on the basis of the continuous variation method.

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## Introduction

Many efforts have been made to understand solvent effects in terms of the solvent polarity. It is easy to understand solvent polarity qualitatively, but it is difficult to express it quantitatively. According to idealized theories, the solvent dielectric constant (i.e., the relative permittivity, $\varepsilon_r$) is often used as a quantitative measure of solvent polarity. Also, very often it has been found that there is no correlation between the relative permittivity (or its different functions such as $1/\varepsilon_r$, $(\varepsilon_r - 1)/(2\varepsilon_r + 1)$, etc.) and the logarithms of rate or equilibrium constants of solvent-dependent chemical reactions. On the other hand, solvent dipole moments are not sufficient for measuring the solvent polarity because the charge distribution of a solvent molecule may not only be given by its dipole moment but also by its quadrupole or higher multipole moments.[1] Therefore, it is stated that the polarity of a solvent is determined by its solvation capability or (solvation power) for reactants and activated complexes as well as for molecules in their ground and excited states.[2,3] Thus, solvation power depends on all specific and nonspecific intermolecular forces between solvent and solute molecules which include Coulomb interactions between ions, directional interactions between dipoles, inductive, dispersion, hydrogen bonding, and charge-transfer forces, as well as solvophobic interactions. A literature survey reveals that in spite of several papers about thermodynamics and kinetics of solutions with different protic and aprotic solvents[4−29] there are no reports on the complexation of Mo(VI) with EDDA in different aqueous solutions of methanol. The present study deals with the aforementioned system, and the results have also been compared with similar complexes reported in the literature.

## Experimental Section

***Reagents.*** All chemicals were analytical reagent grade. Sodium perchlorate, perchloric acid, sodium hydroxide, and sodium molybdate were purchased from E. Merck and EDDA from Fluka and were used without further purification. Dilute perchloric acid solution was standardized against $KHCO_3$.[30,31] In all experiments, double-distilled water with a specific conductance equal to (1.3 ± 0.1) $\mu$S·cm$^{-1}$ has been used.

**Table 1. Protonation Constants $K_1$ and $K_2$ of EDDA in Different Volume Fractions, $w$, of Methanol at $T = 298$ K and $I = 0.1$ mol·dm$^{-3}$**

| $W$ | log $K_1$ | log $K_2$ | ref |
|---|---|---|---|
| 0.20 | 10.69 ± 0.03 | 8.44 ± 0.01 | this work |
| 0.25 | 10.75 ± 0.02 | 8.57 ± 0.04 | this work |
| 0.30 | 10.82 ± 0.05 | 8.65 ± 0.01 | this work |
| 0.35 | 10.88 ± 0.01 | 8.73 ± 0.02 | this work |
| 0.40 | 10.95 ± 0.01 | 8.77 ± 0.02 | this work |
| 0.0 | 10.10 ± 0.02 | 8.12 ± 0.01 | 33 |
| 0.0 | 10.67 ± 0.03 | 7.45 ± 0.04 | 25 |
| 0.0 | 9.60 | 6.51 | 43[a] |
| 0.0 | 9.62 | 6.55 | 44[a] |
| 0.0 | 9.63 | 6.55 | 45[a] |
| 0.0 | 9.67 | 6.57 | 46[a] |
| 0.0 | 9.57 | 6.48 | 47[a] |
| 0.0 | 9.58 | 6.59 | 48[a] |
| 0.0 | 9.77 | 6.69 | 49[b] |
| 0.0 | 9.69 | 6.72 | 50[c] |
| 0.0 | 10.06 | 7.18 | 30[d] |

[a] 0.1 M $KNO_3$. [b] 1.0 M $NaClO_4$. [c] 1.0 M KCl. [d] 3.0 M $NaClO_4$.

**Table 2. Corrected Absorbance, $A_c$, Data for the Mo(VI) + EDDA System at $T = 298$ K, pH = 6.00, $I = 0.1$ mol·dm$^{-3}$, and Different Wavelengths and Volume Fraction of 0.20 of Methanol**

| | $A_c$ | | |
|---|---|---|---|
| mole fraction of Mo(VI) | 260 nm | 265 nm | 270 nm |
| 0.00 | 0.0000 | 0.0000 | 0.0000 |
| 0.05 | 0.1619 | 0.1170 | 0.0847 |
| 0.10 | 0.3160 | 0.2300 | 0.1691 |
| 0.20 | 0.5242 | 0.3627 | 0.2538 |
| 0.40 | 0.7331 | 0.4766 | 0.3124 |
| 0.50 | 0.7661 | 0.4985 | 0.3266 |
| 0.60 | 0.7329 | 0.4788 | 0.3168 |
| 0.80 | 0.4562 | 0.3129 | 0.2191 |
| 0.90 | 0.2445 | 0.1551 | 0.0993 |
| 0.95 | 0.1503 | 0.0964 | 0.0619 |
| 1.00 | 0.0000 | 0.0000 | 0.0000 |

***Measurements.*** All measurements were carried out at 298 K and ionic strengths of 0.1 mol·dm$^{-3}$ sodium perchlorate. A Metrohm pH-meter, 744, was used for pH measurements. The pH-meter has a sensitivity of 0.01 units. The hydrogen ion concentration was measured with a Metrohm combination electrode, model 6.0228.000. A 0.01 mol·dm$^{-3}$ perchloric acid solution containing 0.09 mol·dm$^{-3}$ sodium perchlorate (for adjusting the ionic strength to 0.1 mol·dm$^{-3}$) was employed as a standard solution of hydrogen ion concentration. The change in liquid junction potential[30] was calculated from eq 1

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$$\text{pH (real)} = \text{pH (measured)} + a + b[\text{H}^+]\ \text{(measured)} \qquad (1)$$

$a$ and $b$ were determined by measurement of the hydrogen ion concentration for two different solutions of $HClO_4$ with sufficient $NaClO_4$ to adjust the ionic media.[30] Spectrophotometric measurements were performed with a Varian Cary 300 UV−vis spectrophotometer with Pentium 4 computer in the UV range (260 to 270) nm in thermoregulated matched 10 mm quartz cells. The measurement cell was of the flow type. A Masterflux pump allowed circulation of the solution under study from the potentiometric cell to the spectrophotometric cell so the absorbance and pH of the solution could be measured simultaneously. Calibration of the glass electrode for different methanol mixtures has been done according to the literature.[4,32] $B$ is the value from the pH meter for the methanol mixture and is related to $[H^+]$ by using the following equation

$$-\log[\text{H}^+] = B + \log \mu_\text{H} \qquad (2)$$

In this research, the values of $B$ were obtained in different methanol mixtures containing a known concentration of HCl and $NaClO_4$ to give a constant ionic strength of $0.1\ \text{mol}\cdot\text{dm}^{-3}$.[4] The difference between the logarithm of known hydrogen ion concentrations and the corresponding values of $B$ was used to calculate values of the correction term $\log \mu_\text{H} = \log(\mu_\text{H}^0 \gamma_+)$.[4] The value of $\mu_\text{H}^0$ is independent of ionic concentration but is dependent on solvent composition. $\gamma_+$ is the mean activity coefficient of perchloric acid in the solvent mixtures.[4]

For each experiment, two solutions of Mo(VI) + ligand were prepared with the same concentration, but the ionic strength of the first was maintained with sodium perchlorate and that of the second with sodium hydroxide or perchloric acid. The pH of the first solution was adjusted with the second one. The second solution consists of metal + ligand + NaOH for increasing pH, while for decreasing pH the second one consists of metal + ligand + $HClO_4$. The absorbance of the first solution was measured after adjusting the pH. In all cases, the procedure was repeated at least three times, and the resulting average values and corresponding deviations from the average are shown in the text and tables.

## Results and Discussion

***Complexation of Molybdenum(VI) with EDDA. Theory and Calculation.*** According to our knowledge, the protonation equilibria of EDDA have been studied in different kinds of background electrolytes of aqueous solutions. Protonation constant values were obtained at different mole fractions of methanol on the basis of the following equilibria (L = EDDA)[33]

$$\text{H}^+ + \text{L}^{2-} \rightleftharpoons \text{HL}^- \qquad K_1 = \frac{[\text{HL}^-]}{[\text{H}^+][\text{L}^{2-}]} \qquad (3)$$

$$\text{H}^+ + \text{HL}^- \rightleftharpoons \text{H}_2\text{L} \qquad K_2 = \frac{[\text{H}_2\text{L}]}{[\text{H}^+][\text{HL}^-]} \qquad (4)$$

The values of protonation constants at different ionic strengths and aqueous solutions of methanol together with the values reported in the literature are gathered in Table 1. $MoO_4^{2-}$ will bind with this tridentate ligand as a 1:1 complex[30,33]

$$\text{MoO}_4^{2-} + \text{EDDA}^{2-} + 2\text{H}^+ \rightleftharpoons \text{MoO}_3\text{EDDA}^{2-} + \text{H}_2\text{O} \qquad (5)$$

$$\beta_{112} = \frac{[\text{MoO}_3\text{EDDA}^{2-}]}{[\text{MoO}_4^{2-}][\text{EDDA}^{2-}][\text{H}^+]^2} \qquad (6)$$

Using the continuous variations method, the absorbances of solutions of the $MoO_4^{2-}$ complex at total concentration of $9.6\cdot10^{-3}\ \text{mol}\cdot\text{dm}^{-3}$ in the UV range (260 to 270) nm at a constant pH = 6.00 have been determined. Stability constants have been calculated at different mole fractions of methanol by combination of the following equations according to our previous works for aqueous solutions[33−40]

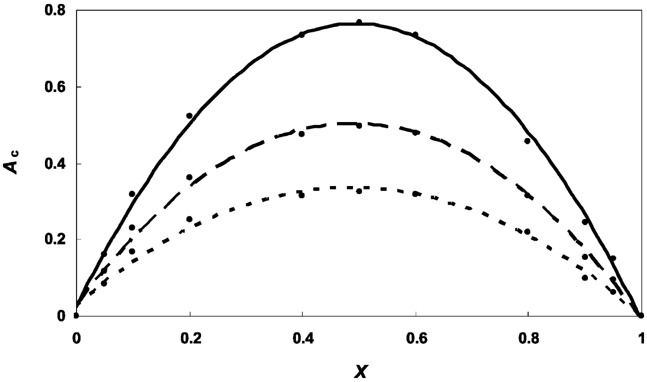

**Figure 1.** Corrected absorbance data, $A_c$, for $MoO_3EDDA^{2-}$ vs the mole fraction of Mo(VI), $X$, at $T$ = 298 K, an ionic strength of $0.1\ \text{mol}\cdot\text{dm}^{-3}$ $NaClO_4$, volume fraction of 0.20 of methanol, and different wavelengths: —, 260 nm; — — —, 265 nm; and - - - -, 270 nm. All of the lines have been obtained on the basis of the best fit to corrected absorbance data.

**Table 3. Average Values of $\log \beta_{112}$ at pH = 6.00, $I$ = 0.1 $\text{mol}\cdot\text{dm}^{-3}$ $NaClO_4$, and Different Volume Fractions, $w$, of Methanol for the Complexation of Molybdenum(VI) with EDDA, $T$ = 298 K**

| $w$ | $\log \beta_{112}$ | ref |
|---|---|---|
| 0.20 | 21.71 ± 0.08 | this work |
| 0.25 | 21.75 ± 0.10 | this work |
| 0.30 | 21.82 ± 0.14 | this work |
| 0.35 | 22.10 ± 0.14 | this work |
| 0.40 | 22.50 ± 0.15 | this work |
| 0.0 | 21.66 | 33 |
| 0.0 | 19.18 | 51[a] |
| 0.0 | 19.69 | 30[b] |

[a] 0.2 M KCl. [b] 3.0 M $NaClO_4$.

$$A_\text{c} = A_\text{obs} - \varepsilon_0[\text{MoO}_4^{2-}] \qquad (7)$$

$$[\text{C}] = \frac{A_\text{c}}{\varepsilon_1} \qquad (8)$$

$$C_\text{M} = [\text{MoO}_4^{2-}] + [\text{C}] \qquad (9)$$

$$C_\text{L} = [\text{Ligand}] + [\text{C}] \qquad (10)$$

$A_c$, $A_{obs}$, and $\varepsilon_0$ are the corrected absorbance of the complex, the observed absorbance, and the molar absorptivity of $MoO_4^{2-}$, respectively. Continuous variations data for the Mo(VI) + EDDA system at pH = 6.00, $I = 0.1\ \text{mol}\cdot\text{dm}^{-3}$, and different wavelengths and aqueous solutions of methanol are gathered in Tables 2 and A1 to A4 in the Appendix and the plot in Figure 1. The molar absorptivity values of the complex, $\varepsilon_1$, were calculated at low mole fraction of the metal, where essentially all the metal ions were in the form of a complex.[33] [C] is the concentration of the complex.[33] The values of stability constants together with the literature values are shown in Table 3.

***Comparison with Literature Data.*** The authors are not aware of any previous publication of stability constants for reaction 5 in any alcohol/water media. Gharib[6] has reported solvent effects on the complexation of molybdenum(VI) with nitrilotriacetic acid (NTA) in different aqueous solutions of methanol and obtained a similar pattern for stability constant changes with increasing mole fraction of methanol. Our results are similar to the literature.[6] In contrast, complexation of molybdenum(VI) with NTA has also been studied in aqueous solutions of propanol,[16] but the parameter patterns[41,42] are different from the Gharib paper.[6] Another report in the literature[25] is about the solvent effects on the complexation of tungsten(VI) with EDDA in different aqueous solutions of propanol in which stability constants increase with increasing mole fractions of propanol.

## Conclusions

It was shown that the values of protonation and stability constants change as a function of different percents of methanol at constant ionic strength of 0.1 mol·dm$^{-3}$ sodium perchlorate. The values increase and finally reach a maximum at a volume fraction of 0.40 of methanol. Finally, it can be concluded that protonation and stability constants increase with a decrease in the polarity of the mixture in the desired range of methanol concentration.

## Appendix

**Table A1. Corrected Absorbance, $A_c$, Data for the Mo(VI) + EDDA System at $T$ = 298 K, pH = 6.00, $I$ = 0.1 mol·dm$^{-3}$, and Different Wavelengths and Volume Fraction of 0.25 of Methanol**

| | $A_c$ | | |
|---|---|---|---|
| mole fraction of Mo(VI) | 260 nm | 265 nm | 270 nm |
| 0.00 | 0.0000 | 0.0000 | 0.0000 |
| 0.05 | 0.1610 | 0.1179 | 0.0860 |
| 0.10 | 0.2881 | 0.1992 | 0.1406 |
| 0.20 | 0.4782 | 0.3227 | 0.2181 |
| 0.40 | 0.6877 | 0.4375 | 0.2778 |
| 0.50 | 0.7514 | 0.4905 | 0.3248 |
| 0.60 | 0.6430 | 0.4029 | 0.2505 |
| 0.80 | 0.3881 | 0.2475 | 0.1559 |
| 0.90 | 0.1915 | 0.1200 | 0.0739 |
| 0.95 | 0.0966 | 0.0618 | 0.0409 |
| 1.00 | 0.0000 | 0.0000 | 0.0000 |

**Table A2. Corrected Absorbance, $A_c$, Data for the Mo(VI) + EDDA System at $T$ = 298 K, pH = 6.00, $I$ = 0.1 mol·dm$^{-3}$, and Different Wavelengths and Volume Fraction of 0.30 of Methanol**

| | $A_c$ | | |
|---|---|---|---|
| mole fraction of Mo(VI) | 260 nm | 265 nm | 270 nm |
| 0.00 | 0.0000 | 0.0000 | 0.0000 |
| 0.05 | 0.1614 | 0.1172 | 0.0874 |
| 0.10 | 0.3024 | 0.2164 | 0.1547 |
| 0.20 | 0.4958 | 0.3339 | 0.2257 |
| 0.40 | 0.6075 | 0.3853 | 0.2419 |
| 0.50 | 0.7150 | 0.4553 | 0.2884 |
| 0.60 | 0.6187 | 0.3908 | 0.2455 |
| 0.80 | 0.4899 | 0.3088 | 0.1928 |
| 0.90 | 0.2530 | 0.1605 | 0.1003 |
| 0.95 | 0.1081 | 0.0688 | 0.0420 |
| 1.00 | 0.0000 | 0.0000 | 0.0000 |

**Table A3. Corrected Absorbance, $A_c$, Data for the Mo(VI) + EDDA System at $T$ = 298 K, pH = 6.00, $I$ = 0.1 mol·dm$^{-3}$, and Different Wavelengths and Volume Fraction of 0.35 of Methanol**

| | $A_c$ | | |
|---|---|---|---|
| mole fraction of Mo(VI) | 260 nm | 265 nm | 270 nm |
| 0.00 | 0.0000 | 0.0000 | 0.0000 |
| 0.05 | 0.1543 | 0.1073 | 0.0761 |
| 0.10 | 0.2758 | 0.1863 | 0.1264 |
| 0.20 | 0.5052 | 0.3331 | 0.2200 |
| 0.40 | 0.7733 | 0.4896 | 0.3090 |
| 0.50 | 0.7631 | 0.4750 | 0.2919 |
| 0.60 | 0.6794 | 0.4219 | 0.2585 |
| 0.80 | 0.3743 | 0.2360 | 0.1488 |
| 0.90 | 0.1961 | 0.1265 | 0.0795 |
| 0.95 | 0.0496 | 0.0333 | 0.0209 |
| 1.00 | 0.0000 | 0.0000 | 0.0000 |

**Table A4. Corrected Absorbance, $A_c$, Data for the Mo(VI) + EDDA System at $T$ = 298 K, pH = 6.00, $I$ = 0.1 mol·dm$^{-3}$, and Different Wavelengths and Volume Fraction of 0.40 of Methanol**

| | $A_c$ | | |
|---|---|---|---|
| mole fraction of Mo(VI) | 260 nm | 265 nm | 270 nm |
| 0.00 | 0.0000 | 0.0000 | 0.0000 |
| 0.05 | 0.1471 | 0.1028 | 0.0734 |
| 0.10 | 0.2641 | 0.1869 | 0.1329 |
| 0.20 | 0.5737 | 0.3986 | 0.2799 |
| 0.40 | 0.8809 | 0.5765 | 0.3775 |
| 0.50 | 0.8902 | 0.5686 | 0.3623 |
| 0.60 | 0.8318 | 0.5198 | 0.3229 |
| 0.80 | 0.5147 | 0.3257 | 0.2071 |
| 0.90 | 0.2701 | 0.1735 | 0.1118 |
| 0.95 | 0.0992 | 0.0648 | 0.0421 |
| 1.00 | 0.0000 | 0.0000 | 0.0000 |

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