

Determination of Stability Constants of Chlorocadmium Complexes in Water from Electromotive Force Measurements

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A potentiometric method involving a three-electrode cell was applied to determine the concentration stability constants (K'_n) of chlorocadmium complexes in water at 298.15 K and ionic strength $I = 1 \text{ mol dm}^{-3}$. Initially, calibration diagrams were derived for the cadmium and chloride ion, based on the measurements of the potential difference between the glass and the cadmium-amalgam electrode for known Cd^{2+} ion concentrations, and between the glass and AgCl / Ag electrode for known Cl^- ion concentrations. To determine the concentrations of free Cd^{2+} and Cl^- ions by means of calibration diagrams, potential differences between the same electrodes were measured in the three-electrode cell by changing the concentration of total chlorides while keeping the concentration of cadmium ions constant. These data were used to determine the concentration stability constants of the formed complexes CdCl^+ , CdCl_2 , and CdCl_3^- by applying Leden's graphical method and three numerical methods: nonlinear regression without scaling, nonlinear regression with scaling, and orthogonal polynomials. The two latter methods gave identical constants: 24.6 (CdCl^+), 68 (CdCl_2), and 69 (CdCl_3^-). The obtained concentration stability constants were compared with the available data for the same complexes obtained from the electromotive force measurements (e.m.f.) of the concentration cell with transference, calculated using the same methods. As a result, it was possible to compare both experimental

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methods applied in determining the concentration stability constants of chlorocadmium complexes in water medium at 298.15 K and ionic strength $I = 1 \text{ mol dm}^{-3}$.

Key words: direct potentiometry, three-electrode cell, chlorocadmium complexes, concentration stability constants.

INTRODUCTION

The complexation of CdCl_2 in water,^{1,2,3} and in mixtures of water and an organic component^{3,4,5} was investigated by measuring the electromotive force (e.m.f.) of the concentration cell with transference in solutions of different ionic strengths and at different temperatures. For optimal processing of the potentiometric data, while calculating the concentration stability constants, K'_n , besides using the method of successive graphical extrapolations^{1,2} within Leden's iterative procedure, several other calculating methods were derived: the method of orthogonal polynomials,³ and the method of nonlinear regression without scaling⁶ and with scaling.^{7,8}

Although sophisticated calculating methods were applied, there are two basic disadvantages of this experimental procedure: the impossibility to measure the concentration of free chloride ions, $[\text{Cl}^-]$, and the existence of a junction potential. Namely, in the iterative procedure, the chlorides were adjusted simultaneously with the concentration stability constants starting from the same e.m.f. values that were used for the calculation of the free Cd^{2+} ion concentration, $[\text{Cd}^{2+}]$. The junction potential is a component of the measured e.m.f. of the cell and it is evaluated by the Henderson equation.

To avoid the above mentioned disadvantages, the »direct potentiometry« method was applied in this work. Namely, there is a standardized method in literature⁹ for the determination of chloride ions in various water samples. The chloride ion concentration is determined directly from the e.m.f. measurements of a cell, consisting of an AgCl / Ag electrode reversible to the Cl^- ion and a glass electrode as reference. However, it is first necessary to perform a potentiometric titration with standard chloride solutions in order to obtain a calibration diagram E versus $\log([\text{Cl}^-] / c^\circ)$, where $c^\circ = 1 \text{ mol dm}^{-3}$. In analogy, it is possible to determine the concentration of free Cd^{2+} ions by measuring the potential difference between the cadmium-amalgam electrode, $\text{Cd}(\text{Hg})$, using a glass electrode as reference. Here again, the calibration diagram E versus $\log([\text{Cd}^{2+}] / c^\circ)$ is derived from the potentiometric titration data.

The complexation reaction of Cd^{2+} and Cl^- is then followed by adding the chlorides into the three-electrode cell containing a solution of constant con-

centration of total cadmium, $c_{\text{tot}}(\text{Cd}^{2+})$, and by measuring the potential difference between the AgCl/Ag and the glass electrode, and between the Cd(Hg) and the glass electrode. Consequently, there is no junction potential in these cells. The concentration of free Cd^{2+} and Cl^- ions is determined from the measured e.m.f. values using the calibration diagram. These data enable calculation of the concentration stability constants of chlorocadmium complexes using the same calculating methods as those applied using the concentration cell with transference.

Finally, the values obtained for the concentration stability constants at 298.15 K and ionic strength $I = 1 \text{ mol dm}^{-3}$ were compared with the reported values³ from the e.m.f. measurements data of the concentration cell with transference calculated by the orthogonal polynomials method. In this work, for a more precise comparison, the concentration stability constants were calculated from the same data using other calculating methods, such as the method of nonlinear regression with and without scaling, and the classical graphical Leden's method.

EXPERIMENTAL

The experimental section involves direct potentiometry and consists of three steps:

- the calibration diagram for cadmium ions,
- the calibration diagram for chloride ions,
- potentiometric titration in the three-electrode cell to follow the complexation of the Cd^{2+} and Cl^- ions.

In order to derive the calibration diagram for cadmium ions, it was necessary to prepare two stock solutions, labelled A₁ and B. Stock solution A₁ contained 0.01 mol dm⁻³ Cd(ClO₄)₂, 0.01 mol dm⁻³ HClO₄, and $(I - 0.04) \text{ mol dm}^{-3}$ NaClO₄. Stock solution B was composed of 0.01 mol dm⁻³ HClO₄ and $(I - 0.01) \text{ mol dm}^{-3}$ NaClO₄. In this work, all solutions had an ionic strength of $I = 1 \text{ mol dm}^{-3}$.

Solution A₁ was prepared as described earlier³ from the following reagents: CdO (*p.a.* purity grade, »Lafoma«), HClO₄ 70% (*w*) (*p.a.* purity grade, »Merck«), and NaClO₄ · H₂O (*p.a.* purity grade, »Riedel-de-Haën«), while solution B was prepared in the usual way. All stock solutions were prepared using redistilled water.

The cell for measuring the potential difference between the glass electrode and the Cd(Hg) electrode was a glass vessel of about 300 cm³ volume with the Pt-wire fused in at the bottom. One end of the wire was covered with Cd(Hg) in the vessel, and the other end was inserted into a narrow side tube to establish electrical contact with the measuring instrument across a Wood alloy and a Cu-wire. The silicone stopper on the vessel had openings for the glass electrode, a fine tube for the introduction of nitrogen into the solution, and for the burette. Nitrogen was previously purified and saturated with water vapour.¹⁰

Preparation of the Cd(Hg) electrode ($w(\text{Cd}) = 11\%$) was described earlier.¹⁰

The reference glass electrode HES 0201 was produced by »Iskra«.

Measurements of the potential difference between the Cd(Hg) and the glass electrode were performed by a pH-meter (MA 5741, Iskra) of 0.1 mV sensitivity. The cell temperature was maintained constant to 298.15 ± 0.02 K. Starting with the thermostating and while reading the e.m.f. values, a slow stream of nitrogen was being passed through the potentiometric cell. Nitrogen was also used to mix the solution.

E.m.f. measurements for the calibration diagram of Cd^{2+} ion started by transferring, with a warm dropper, a small amount of warm Cd(Hg) and a specific amount of solution B into the cell. The glass electrode was then immersed into the solution. After thermostating for half an hour, the required volume of solution A_1 was added using a burette in order to ensure the necessary Cd^{2+} ion concentration. After 10 minutes, the potential difference between the amalgam electrode and the reference glass electrode was measured, and the readings were repeated every five minutes until the value became constant. The procedure continued by adding a new amount of solution A_1 . A calibration curve for cadmium was then obtained from the recorded potential differences at a defined Cd^{2+} ion concentration.

To obtain the calibration diagram for chlorides, stock solution B was used as well as stock solution A_2 . Stock solution A_2 contained 0.70 mol dm^{-3} NaCl, 0.01 mol dm^{-3} HClO_4 , and $(I - 0.71) \text{ mol dm}^{-3}$ NaClO_4 . Reagent NaCl was *p.a.* purity grade, »Kemika«.

The cell for measuring the potential differences between the glass and the AgCl / Ag electrode was a wider glass tube of about 300 cm^3 volume having a silicone stopper with four openings. The latter were meant for the AgCl / Ag electrode, the glass electrode, the burette, and a small tube for the passage of extra pure nitrogen. Nitrogen was treated as before.

The preparation and requirements for the AgCl / Ag electrode were described in Ref. 11, and the potential difference between the electrodes was measured by a Feussner potentiometer. The passage of N_2 and the maintenance of temperature while thermostating were as previously described.

The e.m.f. measurements used for the calibration diagram of the chloride ion were started by adding a specific amount of solution B into the cell. The AgCl / Ag electrode and the glass electrode were then immersed into the cell. After thermostating for half an hour, the required volumes of solution A_2 were added with a burette, and readings were taken as described for cadmium. The calibration diagram for the chloride ion was drawn from the measured e.m.f. values at known Cl⁻ concentrations.

The potentiometric titration in the three-electrode cell, the so-called »working« measurement, required, besides solution A_1 , the preparation of stock solution C composed of 0.01 mol dm^{-3} $\text{Cd}(\text{ClO}_4)_2$, 0.01 mol dm^{-3} HClO_4 , 0.80 mol dm^{-3} NaCl, and $(I - 0.84) \text{ mol dm}^{-3}$ NaClO_4 . Solution C was prepared as described in Ref. 3 from previously described reagents.

An identical and uniform pH in all stock solutions (A_1 , A_2 , B, C), which is a prerequisite for the use of a glass electrode as a potential reference, was achieved by precisely adding the same volume of standardized HClO_4 (0.4 mol dm^{-3}), followed by dilution in a volumetric flask to 1 dm^3 . The pH of the stock solutions was checked by

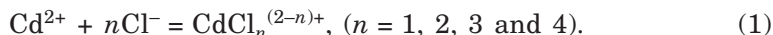
means of a combined glass electrode (HEC 0102). The pH-meter was previously calibrated using the standard buffer pH = 2.

The three-electrode cell was identical to the cadmium amalgam cell, however the silicone stopper had an extra opening for the AgCl / Ag electrode besides the three openings mentioned earlier. First, the cadmium amalgam and a certain volume of solution A₁ were placed into the cell. The AgCl / Ag electrode and the glass electrode were then immersed into the solution, and the cell was blown-through with nitrogen and thermostatted for half-an-hour. Then, precise volumes of solution C, calculated with respect to the required concentration of total chlorides ($c_{\text{tot}}(\text{Cl}^-)$), were added into the cell. The potential difference between the amalgam and the glass electrode (E_1), and between the AgCl / Ag and the glass electrode (E_2) was then separately measured following the same time intervals as earlier. The equilibrium concentration of the Cd²⁺ and Cl⁻ ions was directly determined from the calibration diagram using the E_1 and E_2 values previously obtained.

Since the glass electrode served as the reference electrode in all three steps of the experimental procedure, the stability of its response was checked against a chosen AgCl / Ag electrode. The potential difference between these two electrodes was measured in a solution containing 0.01 mol dm⁻³ HClO₄, 0.1 mol dm⁻³ NaCl, and 0.89 mol dm⁻³ NaClO₄ at 298.15 K before and after the experimental procedure. During the whole experiment (about 10 hours), the glass electrode showed excellent stability.

RESULTS AND DISCUSSION

The Cd²⁺ and Cl⁻ ions form complexes in aqueous solution according to the following relations:



The expression for the concentration equilibrium constants (concentration stability constant) is as follows:

$$K'_n = \frac{[\text{CdCl}_n^{(2-n)+}]/c^\circ}{\{[\text{Cd}^{2+}]/c^\circ\}\{[\text{Cl}^-]/c^\circ\}^n}. \quad (2)$$

The expression in brackets represents the equilibrium concentration of the involved species.

To obtain the concentration stability constants by the method of nonlinear regression, the equation

$$\frac{x - [\text{Cd}^{2+}]}{[\text{Cd}^{2+}]} = F\{[\text{Cl}^-]\} = K'_1[\text{Cl}^-] + K'_2[\text{Cl}^-]^2 + K'_3[\text{Cl}^-]^3 + K'_4[\text{Cl}^-]^4 \quad (3)$$

was used; scaling was performed by multiplying the same equation by weighing factor $[\text{Cd}^{2+}]/x$, and before applying the method of orthogonal polynomials,

equation (3) was divided by the concentration of free chloride ions.⁵ In the above equation, x denotes the concentration of total cadmium (0.01 mol dm^{-3}).

Values of K'_n , obtained from e.m.f. data for a concentration cell³ using the method of orthogonal polynomials (II), are given in Table I. In this work, new K'_n values were calculated from the same data, but using other methods, such as the classical graphical Leden's method (I), as well as the method of nonlinear regression without scaling (III) and with scaling (IV).⁵ These are also shown in Table I. Table I also presents the corresponding standard deviations derived in a manner described in literature.^{12a} Parameters K'_1 and K'_2 of the original polynomial in method II were derived as linear combinations of parameters of Forsythe orthogonal polynomials,^{12a} so that their standard deviations can be calculated according to Ref. 12b. The correlation coefficient, R^2 , measures the goodness of fit of the corresponding polynomial to the experimental data. Values are calculated according to an example in the literature¹³ and presented in the same Table.

TABLE I

Concentration stability constants of chlorocadmium complexes in water, calculated by different methods from the e.m.f. of the concentration cell with transference³ at an ionic strength of $I = 1 \text{ mol dm}^{-3}$ and 298.15 K, including the values of the respective correlation coefficient R^2

	Method				Ref. 14
	I	II	III	IV	
K'_1	21.7	23.0 ± 0.8	25.7 ± 1.4	23.3 ± 0.8	22.4 ± 1.0
K'_2	51	51 ± 10	26 ± 11	51 ± 10	50 ± 12
K'_3	60	50 ± 24	102 ± 21	45 ± 25	32 ± 15
R^2		0.9925	0.9998	0.9936	

The values in Table I are fairly consistent. Even Leden's laborious method, which accumulates errors into higher constants, agrees well with the numerical methods. Again, the concordance with the literature data (sixth column) is good. The only discrepant values are those for K'_2 and K'_3 of nonlinear regression (III), as already established in *t*-butanol + water mixtures.⁵

In order to determine the concentration stability constants by means of »direct potentiometry«, it was first necessary to perform measurements for the calibration diagrams. Namely, the e.m.f. values of the cell Cd(Hg) and glass electrode (E_1) for different known concentrations of free cadmium were measured as presented in Table II. In this case, the free Cd^{2+} ion concentra-

TABLE II

Potential differences between the Cd(Hg) and glass electrode (E_1) for known concentrations of Cd^{2+} ion, and between the AgCl/Ag and glass electrode (E_2) for known concentrations of Cl^- ion in water at an ionic strength of $I = 1.0 \text{ mol dm}^{-3}$ and 298.15 K

$\frac{[\text{Cd}^{2+}]}{\text{mol dm}^{-3}}$	$\frac{E_1}{\text{V}}$	$\frac{[\text{Cl}^-]}{\text{mol dm}^{-3}}$	$\frac{E_2}{\text{V}}$
0.0005	0.9694	0.02	0.1804
0.0010	0.9600	0.06	0.2087
0.0015	0.9560	0.10	0.2213
0.0020	0.9523	0.14	0.2299
0.0025	0.9499	0.18	0.2362
0.0030	0.9475	0.22	0.2411
0.0035	0.9455	0.26	0.2453
0.0040	0.9440	0.30	0.2488
0.0045	0.9425	0.34	0.2519
0.0050	0.9412	0.38	0.2546

tion equals the concentration of total cadmium, because the Cd^{2+} ion does not form complexes with the ClO_4^- ion. The same table shows the e.m.f. values of the cell AgCl/Ag and glass electrode (E_2) for different known concentrations of free chloride. All of these values were obtained in water medium for an ionic strength of $I = 1 \text{ mol dm}^{-3}$ at 298.15 K.

Plots of E_1 against $\log([\text{Cd}^{2+}]/c^\circ)$ and of E_2 against $\log([\text{Cl}^-]/c^\circ)$ are given in Figures 1 and 2, and they denote the calibration dependence for cadmium, and for chloride ions, respectively. The straight line equations, obtained by the least-squares method, and the corresponding correlation coefficients are

$$E_1 / \text{V} = 0.8763 - 0.0282 \log([\text{Cd}^{2+}] / c^\circ), \quad (4)$$

$$(R^2 = 0.9987)$$

$$E_2 / \text{V} = 0.2792 + 0.0579 \log([\text{Cl}^-] / c^\circ). \quad (5)$$

$$(R^2 = 0.9999)$$

The straight line slopes for cadmium (-28.2 mV) and for chlorides (57.9 mV) are in quite a good agreement with the theoretically predicted values of -29.58 mV and 59.15 mV , respectively.

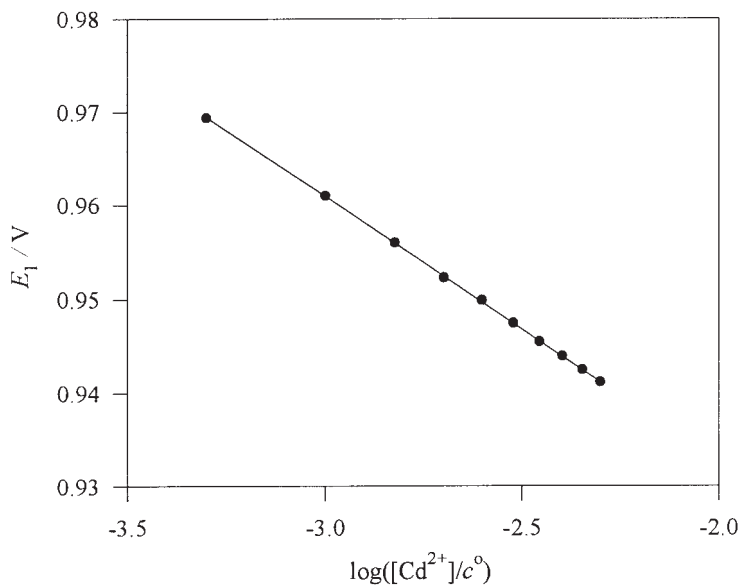


Figure 1. Variation of the potential difference between the Cd(Hg) and glass electrode (E_1/V) versus logarithm of Cd^{2+} ion concentration ($Cd(ClO_4)_2$) in water media at an ionic strength of $I = 1.0 \text{ mol dm}^{-3}$ and 298.15 K (according to data from Table II).

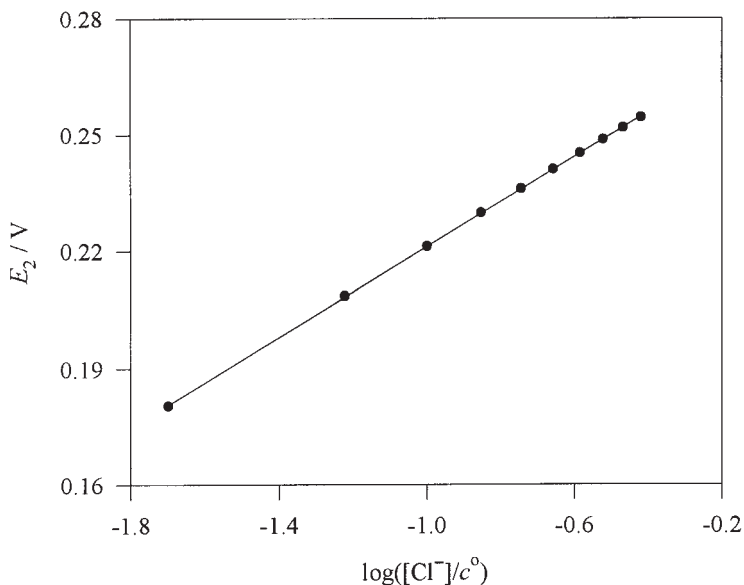


Figure 2. Variation of the potential difference between the AgCl / Ag and glass electrode (E_2/V) versus logarithm of Cl^- ion concentration (NaCl) in water media at an ionic strength of $I = 1.0 \text{ mol dm}^{-3}$ and 298.15 K (according to data from Table II).

TABLE III

Potential differences between the Cd(Hg) and glass electrode (E_1), AgCl / Ag and glass electrode (E_2), and equilibrium concentrations of Cd^{2+} and Cl^- ions for different concentrations of total chloride ($c_{\text{tot}}(\text{Cl}^-)$) in water at an ionic strength of $I = 1.0 \text{ mol dm}^{-3}$ and 298.15 K

$\frac{c_{\text{tot}}(\text{Cl}^-)}{\text{mol dm}^{-3}}$	$\frac{E_1}{\text{V}}$	$\frac{E_2}{\text{V}}$	$\frac{[\text{Cd}^{2+}]}{\text{mol dm}^{-3}}$	$\frac{[\text{Cl}^-]}{\text{mol dm}^{-3}}$
0.075	0.9460	0.2112	0.00337	0.0670
0.100	0.9491	0.2187	0.00262	0.0902
0.150	0.9544	0.2294	0.00170	0.1380
0.200	0.9587	0.2370	0.00120	0.1866
0.252	0.9627	0.2430	0.00086	0.2371
0.300	0.9658	0.2475	0.00067	0.2838
0.350	0.9688	0.2515	0.00052	0.3327

The results of e.m.f. measurements in the three-electrode cell for different concentrations of NaCl ($c_{\text{tot}}(\text{Cl}^-)$) are shown in Table III together with the corresponding values of the equilibrium concentrations of Cd^{2+} and Cl^- ions obtained from the calibration diagrams.

The equilibrium concentration of Cd^{2+} and Cl^- ions served to calculate the concentration stability constants, K'_n , using the four methods mentioned earlier. The calculation then proceeded in one step, since the chlorides were obtained directly from the experiments. Standard deviations of the parameters and the correlation coefficient were calculated in the same way as for the concentration cell (Table I). The results are given in Table IV.

TABLE IV

Concentration stability constants, K'_n , of the chlorocadmium complexes in water calculated by different methods from »direct potentiometry« data at an ionic strength of $I = 1.0 \text{ mol dm}^{-3}$ and 298.15 K, including the values of the respective correlation coefficient R^2

Method	I	II	III	IV
K'_1	25.0	24.6 ± 0.5	24.7 ± 1.1	24.6 ± 0.5
K'_2	63	68 ± 6	66 ± 10	68 ± 6
K'_3	75	69 ± 15	73 ± 21	69 ± 16
R^2		0.9993	0.9999	0.9976

It can be seen from Table IV that three complex-species were also identified by direct potentiometry, *i.e.* CdCl^+ , CdCl_2 , and CdCl_3^- . The concentration stability constants obtained by different calculating methods are in very good agreement; moreover, methods II and IV yield identical values, differing only slightly from method III. Considering of their standard deviations, preference could undoubtedly be given to the methods of orthogonal polynomials (II) and nonlinear regression with scaling (IV).

A comparison of the results obtained by both experimental techniques (Tables I and IV) indicates that the direct potentiometry data are better correlated with the corresponding functions and the stability constants are obtained with greater precision. There is a satisfactory agreement of all K'_1 values. The K'_2 and K'_3 values obtained by method II and IV display a slight difference and those obtained by method III are in total disagreement.

In conclusion, it seems that direct potentiometry is a more reliable experimental technique in the investigated system. Further research will therefore be of interest, particularly in mixed water-organic solvents.

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SAŽETAK

Određivanje konstanti stabilnosti klorokadmijevih kompleksa u vodi iz mjerenja elektromotorne sile

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Potenciometrijskom metodom uz upotrebu trielektrodnog članka određene su koncentracijske konstante stabilnosti (K'_n) klorokadmijevih kompleksa u vodenoj otopini ionske jakosti $I = 1 \text{ mol dm}^{-3}$ pri 298,15 K. Prvo su izrađeni baždarni dijagrami za kadmijeve i kloridne ione i to na osnovi mjerenja potencijalne razlike između staklene i kadmij-amalgamske elektrode za poznate koncentracije Cd^{2+} -iona, odnosno između staklene i AgCl / Ag elektrode za poznate koncentracije Cl^- -iona. Da bi se s pomoću baždarnih dijagrama odredile koncentracije slobodnih kadmijevih i kloridnih iona, mjerene su potencijalne razlike između istih elektroda u trielektrodnom članku, promjenom koncentracije ukupnih klorida uz konstantnu koncentraciju kadmija. Iz tih su podataka izračunane koncentracijske konstante stabilnosti nastalih kompleksa CdCl^+ , CdCl_2 i CdCl_3^- , i to upotrebom Ledenove grafičke metode te triju numeričkih metoda: nelinearne regresije bez skaliranja, nelinearne regresije sa skaliranjem i ortogonalnih polinoma. Dvije posljednje metode dale su identične konstante, naime: 24,6 (CdCl^+), 68 (CdCl_2) i 69 (CdCl_3^-). Dobivene koncentracijske konstante stabilnosti uspoređene su s objavljenim podacima za iste komplekse dobivenim iz mjerenja e.m.s. koncentracijskog članka s prijenosom, a izračunane istim metodama. To je, nadalje, omogućilo usporedbu samih eksperimentalnih metoda određivanja koncentracijskih konstanti stabilnosti klorokadmijevih kompleksa u vodenom mediju pri 298,15 K i ionskoj jakosti $I = 1 \text{ mol dm}^{-3}$.