ปัจจัยที่มีผลต่อความแม่นและความเที่ยงในการวิเคราะห์กรดอ่อนผสม โดยใช้เทคนิคทางโพเทนชิโอเมตรี และการวิเคราะห์ความถดถอยแบบหลายตัวแปรเชิงเส้น

นาย ไชยวัฒน์ ไชยสุต

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FACTORS AFFECTING THE ACCURACY AND PRECISION OF POTENTIOMETRIC TITRATION OF WEAK ACID MIXTURES BY MULTIPLE LINEAR REGRESSION ANALYSIS

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พิมพ์ต้นฉบับบทคัดย่อวิทยานิพนธ์ภายในกรอบสีเขียวนี้เพียงแผ่นเดียว

หอสมุดกลาง จุฬาลงกรณ์มหาวิทยาลัย

ไชยวัฒน์ ไชยสุต : ปัจจัยที่มีผลต่อความแม่นและความเที่ยงในการวิเคราะห์ กรดอ่อนผสมโดยใช้เทคนิคทางโพเทนชิโอเมตรี และการวิเคราะห์ความถดถอยแบบหลายตัวแปรเชิงเส้น (FACTORS AFFECTING THE ACCURACY AND PRECISION OF POTENTIOMETRIC TITRATION OF WEAK ACID MIXTURES BY MULTIPLE LINEAR REGRESSION ANALYSIS.) อ. ที่ปรึกษา : อ.ดร. มิตร ปทีปวณิช, อ. ที่ปรึกษาร่วม : ผศ.ดร. อุษา กล้ากสิกิจ, 264 หน้า. ISBN 974-634-018-2

การวิเคราะห์หาปริมาณของกรดอ่อนสองตัวที่ผสมกัน สามารถทำได้โดยการใช้โพเทนชิโอเมตริกไทเทรชันด้วยเบสแก่ และหาปริมาตรที่จุดสมมูลของกรดอ่อนแต่ละตัวโดยใช้วิธีวิเคราะห์ความถดถอยแบบหลายตัวแปรเชิงเส้น ได้มีการปรับปรุงสมการที่ใช้ให้มีความถูกต้องมากขึ้นโดยใช้ค่าคงที่ของการแตกตัวของกรดซึ่งคำนวณได้จากแอคติวิตี้ของไอออนต่าง ๆ (Ka°) แทนค่าคงที่ที่ได้จากความเข้มข้น (Ka) ความแม่นและความเที่ยงของผลลัพธ์ของปริมาตรที่จุดสมมูลขึ้นกับปัจจัยหลายประการดังนี้ 1) ความแตกต่างของค่าคงที่การแตกตัวของกรดทั้งสอง (ΔpKa)ซึ่งให้ผลแม่นยำถ้า ΔpKa อยู่ในช่วงระหว่าง 0.93 และ 5.02 ดังแสดงในการทดลองครั้งนี้ 2) อัตราส่วนความเข้มข้นเริ่มต้นของกรดทั้งสอง (X) ควรอยู่ในช่วงระหว่าง 0.1 และ 15 หรือในกรดผสมที่มีความชันของกราฟการไทเทรตไม่มากเกิน 1.90 3) ความเบี่ยงเบนมาตรฐานของการวัดความเป็นกรดด่างและปริมาตรเบสที่ใช้ ซึ่งสัมพันธ์กับความชันของกราฟการไทเทรต เมื่อความชันของกราฟการไทเทรตมีค่ามากจะส่งผลให้ความเบี่ยงเบนมาตรฐานมีค่ามาก 4) ช่วงข้อมูลที่ใช้ในการแปลผล พบว่าวิธี B เป็นวิธีที่เหมาะสมที่สุดในการเลือกช่วงข้อมูลเนื่องจากไม่รวมค่าเบี่ยงเบนจาก Gran plots ของกรดเดี่ยวแต่ละตัว 5) การควบคุม Ionic strength ของสารละลายอย่างเคร่งครัดเป็นสิ่งที่จำเป็นและสำคัญ เนื่องจากส่งผลต่อค่า Liquid junction potential และทำให้เกิดความแปรปรวนในการหาค่าแอคติวิตี้ โดยเฉพาะอย่างยิ่งในคู่กรดผสมที่อยู่ในรูปกรดที่แตกตัว และมีค่า X=15 พบว่ามีการเปลี่ยนแปลงของค่า Ionic strength จากเริ่มต้นประมาณ 0.15 เป็น 0.10 เมื่อการไทเทรตสิ้นสุด (เปลี่ยนประมาณ 50%) 6) สภาวะการทดลอง ต้องมีการควบคุมอย่างระมัดระวัง เช่นการเปลี่ยนแปลงของอุณหภูมิ, การป้องกันการละลายของก๊าซคาร์บอนไดออกไซด์จากอากาศโดยการผ่านก๊าซไนโตรเจนเหนือสารละลายขณะไทเทรต, การปรับมาตราฐานของอิเลคโทรดก่อนการไทเทรตแต่ละครั้ง นอกเหนือจากนี้ได้ศึกษาทางทฤษฎีถึงความเที่ยงของการหาปริมาตรที่จุดสมมูลของกรดเดี่ยวแต่ละตัวในกรดอ่อนผสมโดยใช้วิธี Pointwise และพบว่าผลที่ได้จากการคำนวณทางทฤษฎีสอดคล้องเป็นอย่างดีมากกับผลที่ได้จากการทดลอง

ภาควิชา เคมี ลายมือชื่อนิสิต ไชยวัฒน์ ไชยสุต

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บทคัดย่อวิทยานิพนธ์ภายในกรอบสีเขียวนี้พิมพ์

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Potentiometric titrations in which mixtures of two weak acids were titrated with a strong base, and in which multiple linear regression analysis was used to evaluate the equivalent volume of each single weak acid were discussed. The multiple linear equations derived were corrected by using thermodynamic dissociation constant (Ka°) instead of concentration dissociation constant (Ka). The accuracy and precision of the resulting values of equivalent volume depended on various parameters as followed: 1) Difference between pKa value (ΔpKa), in which the accurate result could be obtained if ΔpKa was between 0.93 and 5.02 as presented in this experiment. 2) Initial concentration ratios (X), should be in the range between 0.1 and 15 or in the acid mixtures which the slope of any point of titration curve was not more than 1.90. 3) Standard deviation of pH measurement and volume of base depended on the slope of the titration curve; therefore, the more value of slope of titration curve was, the more standard deviation obtained. 4) Titration data range, Method B was the most appropriate method for choosing titration data range since it was not include the deviation from Gran plots of each single acid. 5) Strictly controlled ionic strength of solution was necessary and important since it affected the liquid junction potential and caused deviation in activity coefficient, especially in the ionized acid mixtures which X=15, ionic strength changed from initial about 0.15 to 0.10 at the end of titration (50% change). 6) Experimental conditions must be carefully controlled ;e.g., temperature variation, prevention of solvation of carbon dioxide gas from atmosphere by passing nitrogen gas over the titration solution being titrated. In addition, the theoretically precisions of the equivalent volume of each single acid in weak acid mixtures were also studied by Pointwise method and found that the theoretical results corresponded very well with experimental results.

ภาควิชา เภสัชเคมี ลายมือชื่อนิสิต Chaiyavat Chaiyasut

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96 Titration curve of the mixture of p-nitrophenol and pralidoxime chloride in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 5.....256

97 Titration curve of the mixture of p-nitrophenol and pralidoxime chloride in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 10.....257

98 Titration curve of the mixture of p-nitrophenol and pralidoxime chloride in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 15.....257

99 Titration curve of the mixture of p-nitrophenol and pralidoxime chloride in 0.1M potassium chloride solution

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100 Titration curve of the mixture of p-nitrophenol and pralidoxime chloride in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 0.1.....258

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102 Titration curve of the mixture of pralidoxime chloride and boric acid in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 1.....259

103 Titration curve of the mixture of pralidoxime chloride and boric acid in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 5.....260

104 Titration curve of the mixture of pralidoxime chloride and boric acid in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 10.....260

105 Titration curve of the mixture of pralidoxime chloride and boric acid in 0.1M potassium chloride solution with 0.1 N

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106 Titration curve of the mixture of pralidoxime chloride and boric acid in 0.1M potassium chloride solution with 0.1 N sodium hydroxide solution.The approximate initial concentration ratio is (X) = 0.1.....261
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ABBREVIATION

ml	Milliliter
meq	Milliequivalence
g	Gram
V	Volume of titrant (ml)
Ve	Volume of titrant at equivalent point (ml) (equivalent volume, ml)
Ve_A	The equivalent volume of weak acid A
Ve_B	The equivalent volume of weak acid B
Vo	Initial volume of weak acid solution (ml)
N	Normality of titrant
Ka	Dissociation constant of acid
Ka^o	Thermodynamic dissociation constant of acid
Kw	Ionization constant of water
Ka_A	Dissociation constant of acid A
Ka_B	Dissociation constant of acid B
Ka^o_A	Thermodynamic dissociation constant of acid A
Ka^o_B	Thermodynamic dissociation constant of acid B
C_{HA}	Concentration of weak acid, HA
Co_A	Initial concentration of weak acid, A
Co_B	Initial concentration of weak acid, B
[a]	Concentration of substance a (Molar concentration of a)
{a}	Activity of substance a
Ci	Concentration of ion i
Zi	Charge of ion i

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$\gamma_{\pm}$	The activity coefficient
ε_{H_2O}	The static dielectric constant of water
δ^+,δ^-	The contributions of the cation and anion, respectively
σ_X, S.D.	Standard error, standard deviation of variable x
σ^2_X	Variance of variable x
%C.V.	Coefficient variation
r_X	True residual of X = observed value of x - true value of x
f	Titration parameter = VN/VoCo
x	Concentration ratio = Co_B/Co_A = Ve_B/Ve_A
r	Dilution parameter = Co_A/N
Gran plots	Modified gran plots of titration data prior to equivalent point which accounted for autoprotolysis of water, also called G plot.

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