

Titer Determination of  $\text{Ce}(\text{SO}_4)_2$  0.1 mol/L

Titer determination of  $\text{Ce}(\text{SO}_4)_2$  by redox titration with di-sodium oxalate.

<b>Sample</b>	Di-sodium oxalate 20 – 40mg
<b>Compound</b>	Di-sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$ M = 134.01, z = 2
<b>Chemicals</b>	1 M Sulfuric acid (5 mL) Conc. Sulfuric acid (98%)
<b>Titrant</b>	Cerium(IV) sulfate, $\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$ c ( $\text{Ce}(\text{SO}_4)_2$ ) = 0.1 mol/L
<b>Standard</b>	--
<b>Indication</b>	DMi140-SC (Combined Platinum ring electrode)
<b>Chemistry</b>	$2\text{Ce}^{4+} + \text{H}_2\text{C}_2\text{O}_4 \rightarrow 2\text{Ce}^{3+} + 2\text{CO}_2 + 2\text{H}^+$
<b>Calculation</b>	$R = m / (\text{VEQ} \cdot c \cdot C)$ , $C = M / (10 \cdot p \cdot z)$  VEQ = Titrant consumption in mL c = Concentration of the titrant m = Mass of the sample in g z = Equivalent number of standard p = Purity of standard %
<b>Waste disposal</b>	Precipitate cerium with sodium hydroxide and filter it. Classify as special waste.
<b>Author, Version</b>	Robin Isyas, IMSG, Version 2.0 Revised: C. De Caro, MSG AnaChem

## Preparation and Procedures

**CAUTION**

- Use safety goggles, a lab coat and wear gloves. If possible, work in a fume hood.
- Ensure sufficient cleaning of sensor after each titration.

**Preparation of 0.1 M Cerium sulphate:**

- Accurately weigh 40.43g of Cerium sulfate in 1000 mL volumetric flask
- Add 500 mL water, and add slowly 28 mL 98%  $\text{H}_2\text{SO}_4$ , dissolve and make up the volume with deionised water.
- Keep it overnight to stand, and then filter it using sintered glass crucible.

**Sample titration :**

- Weigh accurately 20 – 40 mg of di-sodium oxalate in beaker.
- Dispense 5 mL 1M sulfuric acid from additional dosing unit.
- Dispense 50mL water from membrane pump.
- Perform titration at 70°C using DH100 heating system.
- Titrate with 0.1 mol/L  $\text{Ce}(\text{SO}_4)_2$ .
- Color change observed colorless to yellow.
- After completion of each sample, electrode, stirrer and titration tubes will be rinsed by deionized water by means of membrane pump.
- Electrode is again cleaned by water in condition beaker placed on sample changer.

## Remarks

- Rinsing and conditioning of the electrode is crucial to achieve accurate and precise results.
- It is recommended not to dip the tip of the burette in to the sample in order to avoid diffusion.
- This method allows a fully automated analysis procedure. The method can be easily modified for manual operation. Select "Manual stand" in the method function "Titration stand".

## Instruments

- Titration Excellence T50/T70/T90  
(Other Titrators: manual operations and method changes may be necessary)
- XP205 Balance
- 10 mL DV1010 glass burette (ME- 51107501)
- 1 additional dosing unit (ME- 51109030)
- DH100 Heating system (ME- 51108780)

## Accessories

- Rondo 20 with PowerShower<sup>TM</sup> (ME- 51108003)
- Glass Titration beakers (ME- 00101446)
- LabX<sup>®</sup> pro titration software

## Results

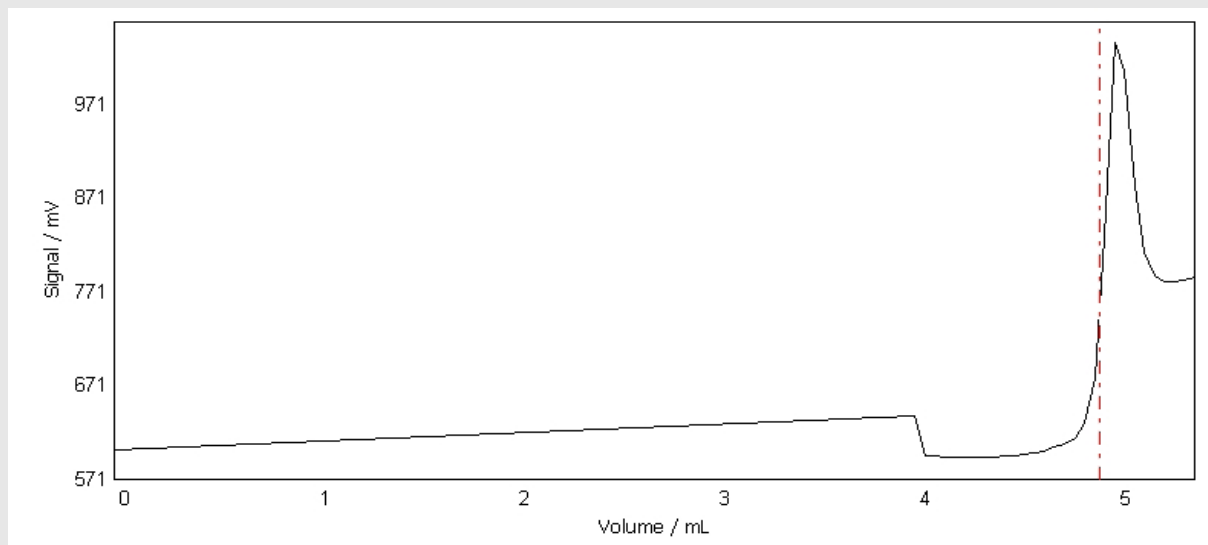
### All results

Method-ID	TitreCericSulphate
Sample	Disodium oxalate (1/6)
R1 (1/6)	0.99782
Sample	Disodium oxalate (2/6)
R1 (2/6)	0.99949
Sample	Disodium oxalate (3/6)
R1 (3/6)	0.99963
Sample	Disodium oxalate (4/6)
R1 (4/6)	0.99755
Sample	Disodium oxalate (5/6)
R1 (5/6)	0.99690
Sample	Disodium oxalate (6/6)
R1 (6/6)	0.99707

### Statistics

Method-ID	TitreCericSulphate
R1	6
Samples	6
Mean	0.9981
s	0.00120
srel	0.120 %

## Titration curve

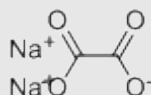


## Table of measured values

	Volume mL	Increment mL	Signal mV	Change mV	1st deriv. mV/mL	Time s	Temperature °C
	0.0000	NaN	603.2	NaN	NaN	0	25°C
	4.0000	4.0000	639.0	35.8	NaN	12	25°C
	4.0500	0.0500	596.6	-42.4	NaN	48	25°C
	4.1000	0.0500	596.1	-0.5	NaN	52	25°C
	4.1500	0.0500	595.4	-0.7	NaN	58	25°C
	4.2000	0.0500	594.8	-0.6	-73.30	63	25°C
	4.2500	0.0500	594.6	-0.2	44.63	68	25°C
	4.3000	0.0500	594.4	-0.2	1.30	73	25°C
	4.3500	0.0500	594.5	0.1	6.40	78	25°C
	4.4000	0.0500	595.3	0.8	10.54	83	25°C
	4.4500	0.0500	595.9	0.6	13.63	88	25°C
	4.5000	0.0500	596.7	0.8	20.72	93	25°C
	4.5500	0.0500	597.7	1.0	26.12	98	25°C
	4.6000	0.0500	599.3	1.6	28.13	103	25°C
	4.6500	0.0500	601.5	2.2	6.71	108	25°C
	4.7000	0.0500	605.3	3.8	-69.84	113	.....
	4.7500	0.0500	608.4	3.1	-14.44	118	25°C
	4.8000	0.0500	615.3	6.9	664.92	123	25°C
	4.8500	0.0500	630.3	15.0	1529.96	128	25°C
	4.9000	0.0500	680.1	49.8	1946.35	133	25°C
EQP	4.925293	NaN	755.4	NaN	1951.10	NaN	NaN
	4.9500	0.0500	829.0	148.9	1723.07	138	25°C
	5.0000	0.0500	1037.2	208.2	976.40	143	25°C
	5.0500	0.0500	1006.8	-30.4	-4.61	148	NaN
	5.1000	0.0500	883.5	-123.3	-868.00	153	25°C
	5.1500	0.0500	814.1	-69.4	-1254.08	158	25°C
	5.2000	0.0500	787.7	-26.4	NaN	163	25°C
	5.2500	0.0500	782.0	-5.7	NaN	168	25°C
	5.3000	0.0500	782.1	0.1	NaN	173	25°C
	5.3500	0.0500	783.2	1.1	NaN	178	25°C
	5.4000	0.0500	787.4	4.2	NaN	183	25°C

## Comments

- The titration runs slowly and is therefore performed at temperature of 70°C. Use glass beakers.
- Di-sodium oxalate is used as primary standard.



- Literature:  
Mettler-Toledo Application M015 (V1.0)

## Method

<b>001 Title</b>	Type	Titer determination	<b>Control</b>	Control	User
	Compatible with	T50 / T70 / T90		Titrant addition	Incremental
	ID	TitreCericSulphate		dV	0.05 mL
	Title	Titer 0.1mol/L CeSO4		Mode	Fixed time
	Author	admin		dt	5.0 s
	Date/Time	. . .	<b>Evaluation and recognition</b>	Procedure	Standard
	Modified on	. . .		Threshold	200.0
	Modified by	admin		Tendency	Positive
	Protect	No		Ranges	0
	SOP	None		Add. EQP criteria	Steepest jump
				Number of EQPs	1
<b>002 Sample (Titer)</b>			<b>Termination</b>	At Vmax	10.0
	Titrant	Ce(SO <sub>4</sub> ) <sub>2</sub>		At potential	No
	Concentration	0.1 mol/L		At slope	No
	Standard	Di-sodium oxalate		After number of	
	Type of standard	Solid		recognized EQPs	Yes
	Entry type	weight		Number of EQPs	1
	Lower limit	0.02 g		Combined termination	
	Upper limit	0.04 g		criteria	No
	Correction factor	1.0			
	Temperature	25.0°C	<b>010 Calculation R1</b>	Result	Titer
	Entry	Arbitrary		Result unit	- -
<b>003 Titration stand (Rondo/Tower A)</b>	Type	Rondo/Tower A		Formula	R1=m/(VEQ*c*C)
	Titration stand	Rondo60/1A		Constant	M/(10*p*z)
	Lid handling	No		M	M[Disodium oxalate]
<b>004 Dispense (normal) [1]</b>				z	z[Disodium oxalate]
	Titrant	H <sub>2</sub> SO <sub>4</sub>		Decimal places	5
	Concentration	1		Result limits	No
	Volume	5 mL		Record statistics	Yes
	Dosing rate	60.0 mL/min		Extra statistical func.	No
	Condition	No		Send to buffer	No
				Condition	No
<b>005 Pump</b>			<b>011 Auxiliary instrument</b>	Control type	OutTTL (single pin)
	Auxiliary reagent	WaterClean		Name	DH100 Off
	Volume	50 mL		Mode	Fixed time
	Condition	No		Time	1.0 s
				Condition	No
<b>006 Auxillary instrument</b>	Control type	OutTTL(single pin)	<b>012 Rinse</b>	Auxiliary reagent	WaterClean
	Name	DH100 ON		Rinse cycles	1
	Mode	Fixed time		Vol. per cycle	10
	Time	2 s		Position	Current position
	Condition	No		Drain	No
<b>007 Stir</b>				Condition	No
	Speed	30 %	<b>013 Conditioning</b>	Type	Fix
	Duration	60 s		Interval	1
	Condition	No		Position	Conditioning beaker
				Time	10 s
<b>008 Measure (normal) [1]</b>				Speed	30
	<b>Sensor</b>			Condition	No
	Type	Temperature	<b>014 End of sample</b>		
	Sensor	DH 100	<b>015 Titer</b>	Titrant	Ce(SO <sub>4</sub> ) <sub>2</sub>
	Unit	°C		Concentration	0.1
	<b>Stir</b>			TITER	Mean[R1]
	Speed	30 %		Limits	No
	<b>Acquisition of measured values</b>			Condition	No
	Acquisition	Set value			
	Mode	T > set value			
	Set value	70 °C			
	t(max)	300 s			
	Mean value	No			
	<b>Condition</b>				
	Condition	No			
<b>009 Titration (EQP) [1]</b>					
	<b>Titrant</b>				
	Titrant	Ce(SO <sub>4</sub> ) <sub>2</sub>			
	Concentration	0.1 mol/L			
	<b>Sensor</b>				
	Type	mV			
	Sensor	DM140-SC			
	Unit	mV			
	<b>Temperature acquisition</b>				
	Temperature acquisition	No			
	<b>Stir</b>				
	Speed	50 %			
	<b>Predispense</b>				
	Mode	Volume			
	Volume	4.0			
	Wait time	30 s			