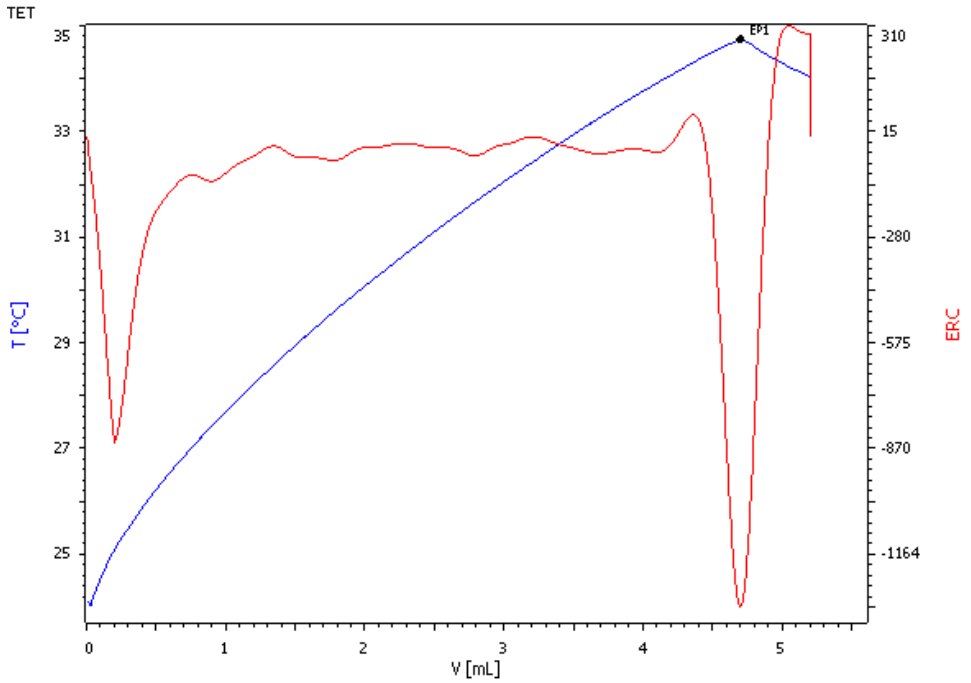
Titration Application Note H-142

Determination of metal-organic compounds



Metal-organic compounds are commonly used in organic chemistry, for example as Grignard reagents or as strong bases (e.g., butyl lithium compounds). The knowledge of the exact content of reactive species allows to better plan the required amounts for reactions preventing the waste of material or too low yields.

This Application Note describes the analysis of metal organics by thermometric titration using 2-butanol as titrant. Due to the strongly exothermic nature of the reaction between 2-butanol with metal-organic compounds, a fast and quantitative analysis of these substances is possible.

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Method description

Sample

Butyllithium compounds (e.g., s-BuLi)

Metalorganic compounds (e.g., Grignard reagents)

Sample preparation

No sample preparation is necessary.

Configuration

859 Titrotherm	2.859.0010
800 Dosino, 2×	2.800.0010
804 Ti Stand	2.804.0040
8.02 Rod stirrer	2.802.0010
Cable USB A – mini-DIN 8-pin	6.2151.000
Titration vessel lid automation	6.1414.080
Titration vessel / 20–90 mL	6.1415.220
Dosing unit 20 mL (Titrant)	6.3032.220
Dosing unit 50 mL (Solvent)	6.3032.250
Stacking frame	6.2065.000
Bottle holder for Dosinos	6.2061.010
Thermoprobe for 859 Titrotherm	6.9011.020

Solutions

Solvent	Toluene, water-free 5 g molecular sieve per 1 L is added 1 day prior to the analyses. The remaining water content should be less than 10 ppm.
Titrant	c(2-butanol) = 1 mol/L in xylene 5 g molecular sieve per 1 L is added 1 day prior to the analyses. The solution is covered with argon. The remaining water content should be less than 10 ppm.
Inter gas	Argon

Analysis

Due to the reactive nature of these samples the titrations have to be carried out under inter gas and all used solvents have to be pre-treated with molecular sieve.

Approx. 15 mL solvent is pre-dosed into the titration vessel. Then 1 to 4 g sample is injected to the titration vessel using a syringe. The sample weight is determined using the back-weighing procedure.

The titration is carried out under a constant stream of inter gas (argon) with c(2-butanol) = 1 mol/L until after the first exothermic end point. After each titration the electrode and buret tips are rinsed with solvent.

Parameters

Mode	TET
Stirrer speed	8
Dosing rate	2 mL/min
Filter factor	80
Damping until	0.2 mL
Stop volume	20 mL
Stop slope	< -0.3 °C/mL
Stop slope active after	1 mL
Volume after stop	0.5 mL
Reaction type	Exothermic
EP criterion	-200

Results

Table 1: Results for 3 batches of s-BuLi, expected content 1.87 mmol/g. (n = 9)

Batch No.	Content / (mmol/g)	s(rel) / %
1	1.8228	0.66
2	1.8659	0.64
3	1.8044	0.54

Table 2: Other metal-organic compounds tested with this method (feasibility test, n = 1)

Sample	Content / (mmol/g)
Butyllithium	3.33
Dimethylaminopropyllithium	0.75
Lithium-bis(ethylhexyl)amine	1.17
Allylmagnesiumbromide	1.16
Benzylmagnesiumchloride	1.16

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Version 1, published in January 2018

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