Application Bulletin

Of interest to:	General analytical chemistry, organic chemistry, pharmaceutical industry	P 1, 3, 4

Determination of acidic and alkaline solids by non-aqueous titration

Summary

The analysis of acids and bases is a well-known basic task in analytical chemistry. With this fully automated system only the sample needs to be taken and weighed in by the user. ProcessLab does the rest autonomously.

For the determination of the acid or base content an automated ProcessLab system is used. Liquid handling of the needed solvents is done using the buret and pumps which ProcessLab provides. Sample identification is done with a barcode reader.

A certain amount of sample is weighed in and the result automatically transferred to *tiamo*. Depending on the type of sample, *tiamo* starts and performs an acid or base titration with the corresponding non-aqueous solvent.

Features/General information

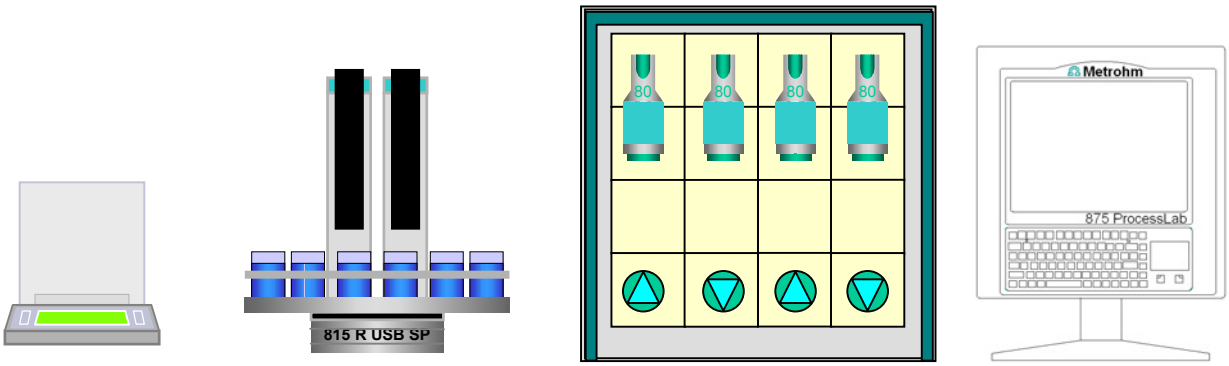
- Acid and base determinations in one system, non-aqueous
- Barcode reader offers automatic method selection by using predefined bar-codes
- Balance integration makes operating easy and prevents typing errors
- System can be used for content determination of any suitable material, e.g. raw material or finished products
- Optional liquid level sensors monitor the reagent level and call for manual intervention when required
- Additional terminals for the I/O controller allow to transmit or receive external signals, for example a result which is sent to a remote PCS (process control system). An incoming signal can be a density value from a density meter.

This system is very flexible and can be adapted very easily to any specific needs. If necessary an additional reagent cabinet is available and can be placed directly under ProcessLab. It has sufficient space for all reagents and makes ProcessLab even more comfortable.

Used Accessories (only important parts listed)

- 1 x 2.875.0010; 875 ProcessLab Base Unit L, 1 Metrohm Dosing & Measuring Controller (incl. IPC, I/O controller and TFT/Keyboard terminal)
- 2 x 6.7201.000; Measuring amplifier
- 4 x 2.800.0010; 800 Dosino
- 2 x 6.3032.220; Buret 20 mL
- 2 x 6.3032.250; Buret 50 mL
- 4 x 6.7205.0X0; Peristaltic pump
- 1 x 2.815.0110; 815 Robotic USB Sample processor XL 2 towers and 2 pumps
- 2 x 2.786.0040; Swing Head
- 2 x 6.0229.100; Solvotrode (1 x 6.2312.010 LiCl in EtOH, 1 x 6.2320.000 TeaBr in Eth.-glyc.)
- Different input and output terminals, e.g. digital or analog output
- Barcode reader, USB
- Reagent containers if needed (2.5, 5, 10 and 20 L), incl. level switch
- Reagent cabinet
- Balance which is supported by *tiamo*

Wet part setup & System view



Reagents

- Titrant c(TBAOH) = 0.1 mol/L in 2-propanol/methanol
- Titrant c($HClO_4$) = 0.1 mol/l in acetic acid
- Solvents according to sample, e.g. acetic acid and acetone
- Buffer pH=4 and 7 for calibration of the electrodes

Calibration and storage of the Solvotrode

- The electrode needs to be calibrated on a regular basis, e.g. once a week
- When not used the Solvotrode is stored in the corresponding electrolyte.
- In an environment where ProcessLab is running 24 hrs a day it is necessary to check the electrode more often than in a normal lab area.

Analysis

These parameters vary depending on the type of sample which you use. Please use the same parameters for the titration of standard and sample.

Performing the analysis

The sample is weighed into a beaker and the result is automatically sent to *tiamo* by pressing the "Print" key on the balance. Then the user scans the barcode with the barcode reader and one of the 2 methods is now selected (*tiamo* identifies the pre-defined barcode string). Here the barcode reader is very useful and makes system handling easier.

When all samples have been registered the system is started with the "Start" button in *tiamo*. ProcessLab now performs the titration depending on the method selected.

Alkaline solid determination - Titration with $HClO_4$

Addition of 50 mL acetic acid under stirring of the solution. When the sample dissolved the vessel walls are sprayed with acetic acid to make sure all sample is collected. Then, if the drift is below a certain threshold (here 3 mV/min), the titration starts and proceeds to the first inflection point. After this the vessel is emptied and rinsed. The head now moves to the rinsing position where the electrode, tips and

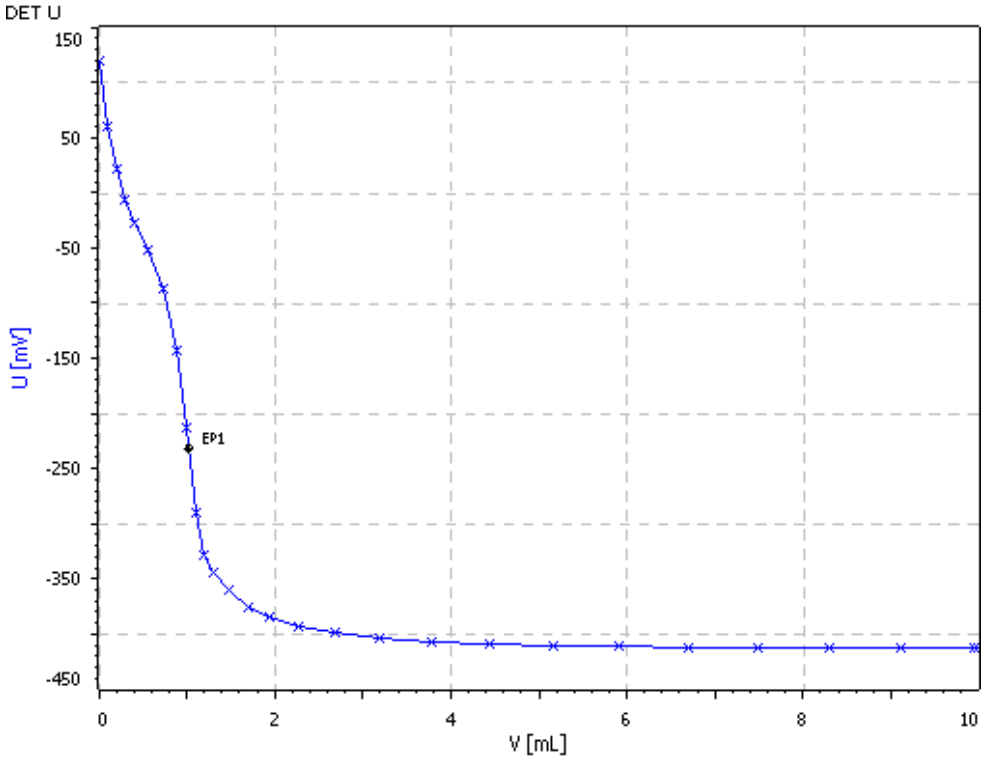
the stirrer are cleaned. The result is now used to calculate the proper content of the sample.

Acidic solid determination - Titration with TBAOH

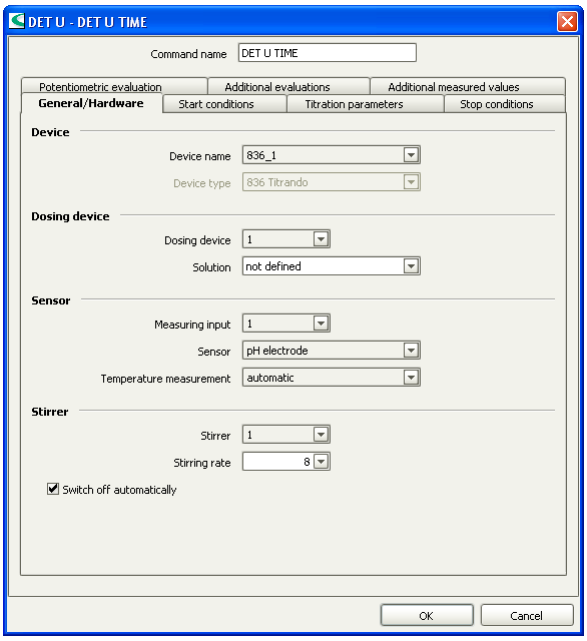
Addition of 50 mL acetone and 20 mL demin. water to dissolve the sample under stirring. Vessel walls are then flushed with demin. water as well to collect all of the sample. In the next step the titration is started and when the endpoint is reached the vessel is cleaned and the head moves to the rinsing position. When finished *tiamo* calculates the results and issues a report.

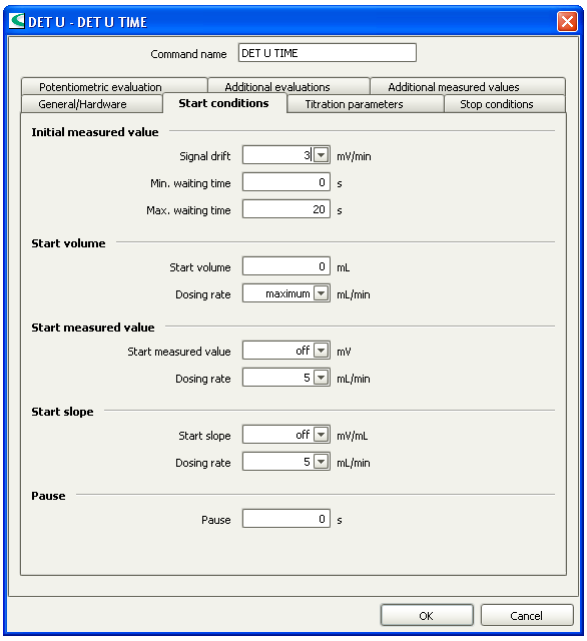
Practical example

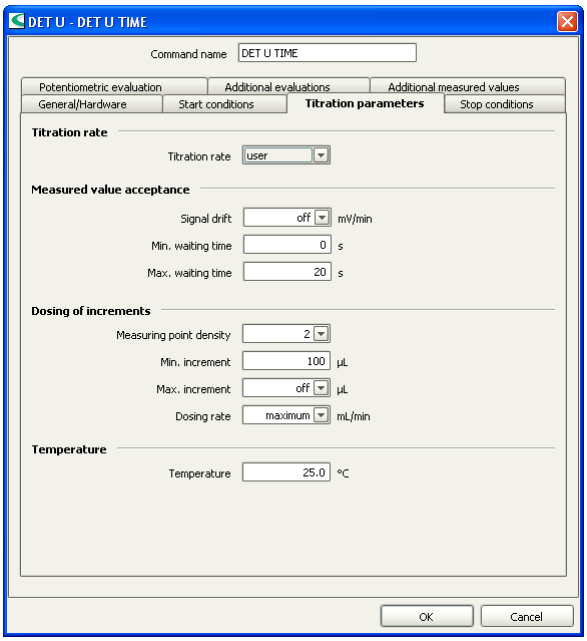
Titration of an acidic substance with TBAOH

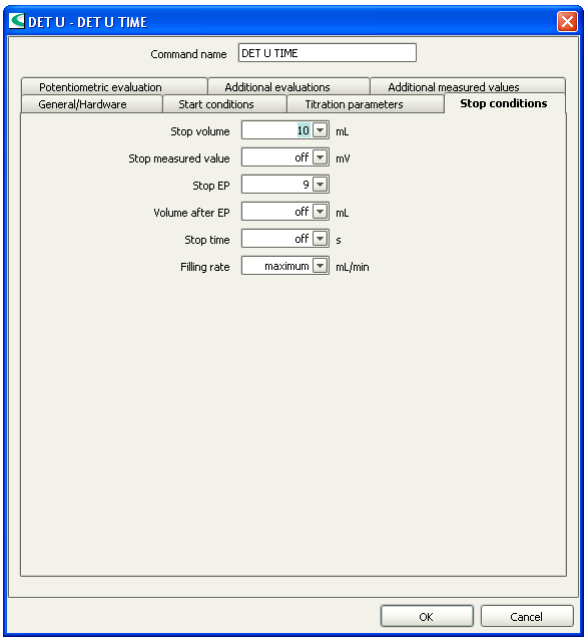


Parameter









Literature

Bruttel, P. (2001), Non-aqueous titration of acids and bases with potentiometric endpoint indication, Metrohm Monograph 8.024.5003

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Haider, Dr. C., Electrodes in Potentiometry, Metrohm Monograph 8.015.5003