Application Bulletin

Of interest to:	General analytical chemistry, alternative energy	P 1, 10

Monitoring of the phosphoric acid concentration in a fuel cell membrane production line

Summary

In fuel cells, hydrogen and oxygen react in a controlled manner to efficiently generate electrical power and heat.

The picture below shows the fuel (e.g. natural gas) entering the reformer, where it is transformed into hydrogen (orange spheres). At the anode catalyst, hydrogen is split into protons (H^+) and electrons (e^-).

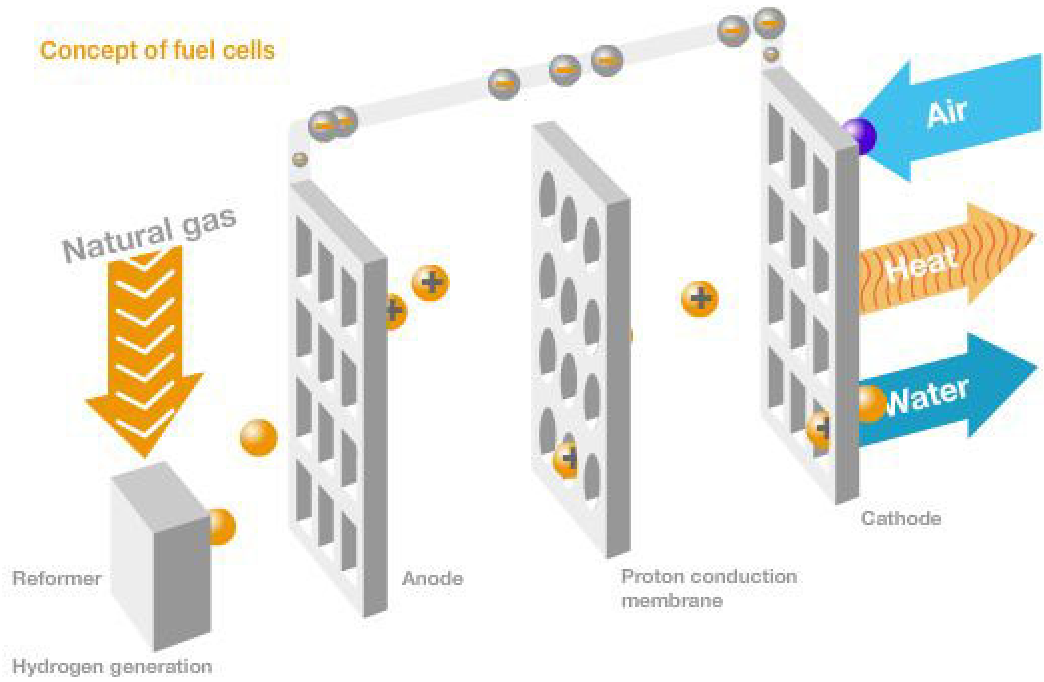


Figure 1: concept of fuel cells[1]

The membrane of a fuel cell is permeable for protons, but not for electrons. Thus, the electrons flow through the external circuit – including a power consumer such as a motor – and return to the cathode side of the fuel cell. On the cathode, oxygen gas (blue spheres) takes up electrodes and protons to form water – the primary exhaust emission of a fuel cell. In order to achieve a reasonable power level, many separate membrane electrode assemblies must be combined to form a fuel-cell stack.

The ProcessLab analysis system described here records and documents the concentration of the phosphoric acid in the reservoir. The concentration has to be within a defined range (± 2% of a target value). This guarantees a constant quality of the membrane. During the stretching of the membrane this concentration will increase, so the solution in the reaction trough has to be diluted with water to reach a specified concentration.

The combination of analytical method, control of the production line, as well as the intuitive handling via the well-arranged user interface allows complete process control.

Features/general information

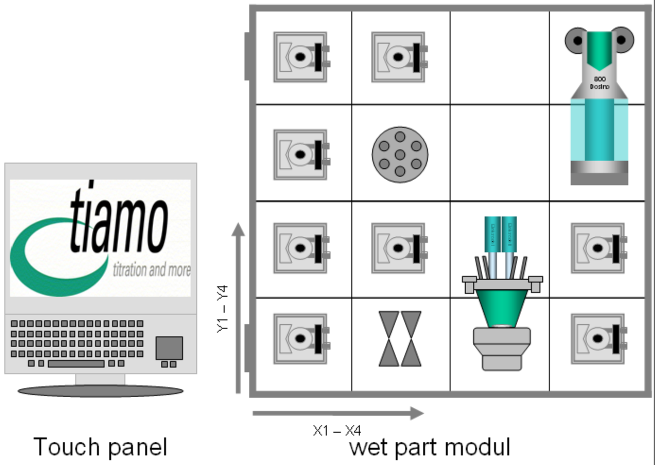
- Monitoring of the H_3PO_4 concentration at several sampling points
- One-button analysis
- System check method
- Start method for semi-automated calibration of the pH electrode used
- Very easy to operate for semi-skilled staff
- One sample cycle includes:
  rinsing (titration vessel)
  measuring (titration of the phosphoric acid with sodium hydroxide)
  calculating the current concentration
  calculating the runtime of the dilution pumps
  start of dilution
- Liquid-level sensors monitor the reagent level (waste, reservoir trough and titration reagent) and make ProcessLab wait for manual intervention if necessary
- Additional terminals for the I/O controller allow external communication like transmission of a result to a PCS. An “out of limit” alarm can be set to switch on any external device.

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

Used Accessories (some are optional)

- 1 x 2.875.0010; 875 ProcessLab Base Unit L, 1 Metrohm Dosing & Measuring Controller (incl. IPC, I/O controller and TFT or keyboard terminal)
- 1 x 6.7201.000; Measuring amplifier
- 1 x 2.800.0010; 800 Dosino
- 1 x 6.3032.220; Buret 20 mL
- 8 x 6.7205.0X0; Peristaltic pump
- 1 x 6.0262.100; Ecotrode plus
- 2 x 6.7202.100; Digital input 4 DI 24 V DC
- 4 x 6.7202.200; Digital output 4 DO 24 V DC
- 6.7207.0X0; Reagent containers (2.5, 5, 10 and 20 L available), incl. level sensor

Wet part layout



X1	Y1	aspiration pump sample point1, 320mL/min
	Y2	aspiration pump sample point2, 320mL/min
	Y3	regulation pump sample point1, 320mL/min
	Y4	regulation pump sample point2, 320mL/min
X2	Y1	solenoid valve
	Y2	transfer pump to sample loop vessel, 120mL/min
	Y3	micro sample valve with fix sample loop (appr. 250µL)
	Y4	transfer pump to titration vessel, 120mL/min
X3	Y1 Y2	titration vessel with pH-electrode
	Y3	Spare (aspiration sample point3)
	Y4	spare (regulation sample point3)
X4	Y1	aspiration pump titration vessel, 320mL/min
	Y2	rinsing pump titration vessel, 120mL/min
	Y3	IDE, 10mL, c(NaOH)=0,1 mol/L
	Y4	electrode connection

Wet part setup

Reagents

- Titrant c(NaOH) = 0.1 mol/L
- Buffer pH = 4 and 7 for calibration of the pH electrodes
- Demineralized water for transferring the sample, cleaning the vessel and diluting the phosphoric acid in the reservoir trough

Calibration and storage of the sensors

- The pH electrodes need to be calibrated on a regular basis, e.g. once a week. The calibration interval is controlled by the software.
- When not used the pH electrode is stored in storage solution (250 mL; 6.2323.000)

Analysis

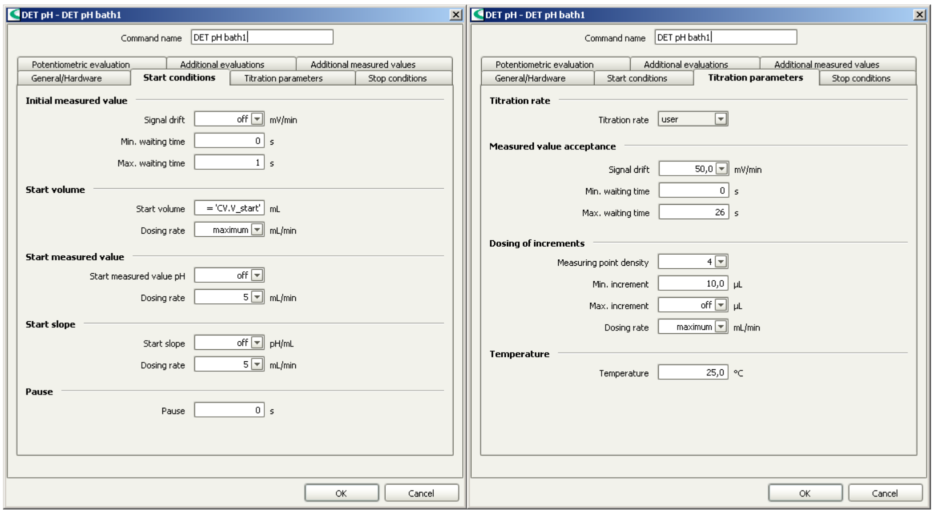
System start / System check

Prior to the first analysis the system has to be checked using a method which prepares the dosing unit, cleans the vessel and executes a first titration of a standard solution (e.g. c(HCl)= 0,1 mol/L). The acid has to be added manually.

Determination of phosphoric acid

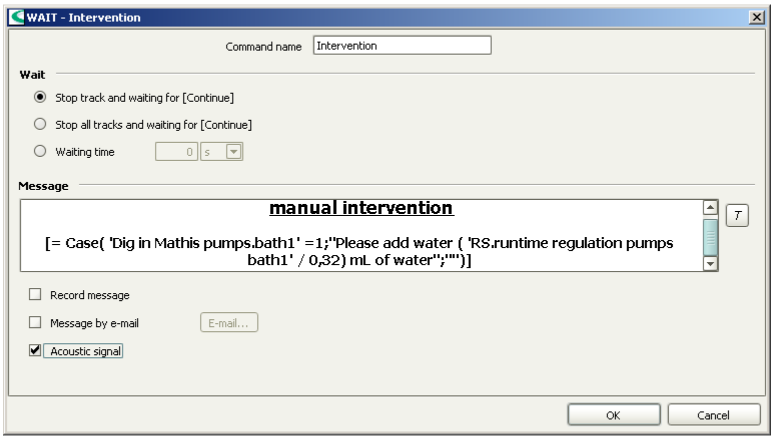
After sampling the sample is pumped through a loop sampling system (LSS), equipped with a loop of about 250 µL. The exact volume of the loop was calibrated by titration of a standard solution. The sample pump is located behind the valve and the loop.

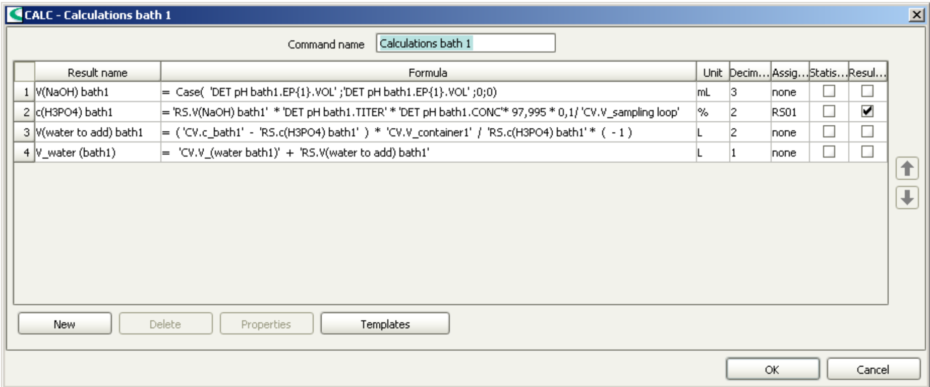
After flushing of the LSS the pump stops, the LSS changes to the vessel position. The sample is then transferred with a transfer pump into the titration vessel. The titration with NaOH starts immediately and is carried out with the following parameters:



After end of the determination the vessel is rinsed.

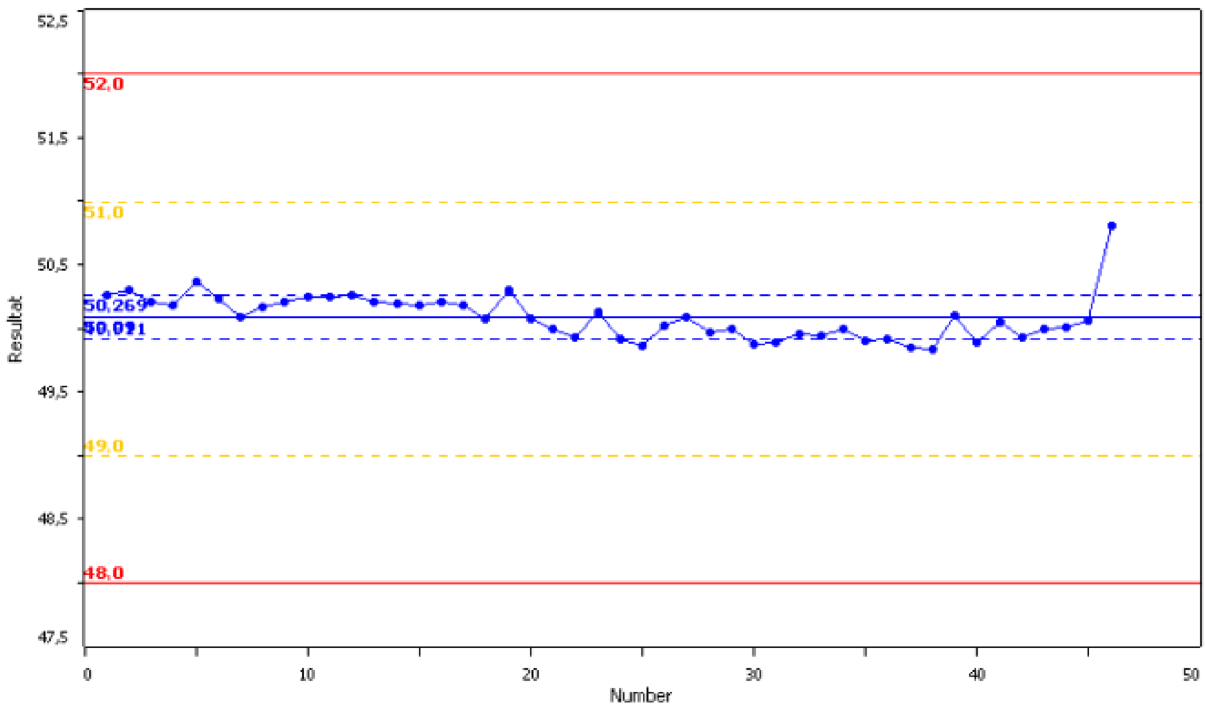
Depending on the amount of H_3PO_4 in the sample, the runtime of the dilution pump is calculated. If it is below the target value, no dilution solution (water) is added to the reservoir trough. This could be done within a defined concentration range (e.g., target value 50 ± 2%). If the concentration is higher than 52% a manual intervention is necessary. The signal is linked with the controller of the production line via digital output signals. A status signal prompts the user to have a closer look at the ***tiamo***™software. A display message shows the next steps required to resume monitoring.





The results for each sample are calculated immediately, shown as a report in the workplace view and finally stored in the database.

Control chart view



Statistics
Mean value 50.09 %
s(abs.) = 0.179 %
s(rel.) = 0.36 %
n = 46

Clean Up

After a production batch is finished, the system is automatically cleaned up by calling a clean-up method: the NaOH Dosing Unit will be emptied and the titration vessel cleaned up.

Reference

[1] Source: http://www.basf-fuelcell.com
http://en.wikipedia.org/wiki/Fuel_cell