

# Determination of FOS TAC value in Biogas plants



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#### Use

The determination of the **volatile organic acids (FOS)** and **total inorganic carbon (TAC)** or buffer monitor the fermentation process in biogas reactors.

Determination of FOS/TAC value is performed as an endpoint titration to two pH endpoint using sulphuric acid. The FOS, TAC and the ratio of FOS/TAC are automatically calculated and displayed.

The FOS TAC method is stored as default method inside the TitroLine® 6000/7000/7750 titrators

#### **Appliances**

Titrator: TL 6000 M2/50 consists of

- Basic device
- Magnetic stirrer TM 235
- 50 mL exchange unit WA 50, with brown glass bottle for titrant complete
- pH combination electrode A 7780 DIN ID

#### **Electrodes**

- see above
- Calibration: DIN buffer pH= 4.00 and pH= 7.00

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#### Reagents

Titrant: H2SO4 0.05 mol/L

Titer: Possible with TRIS (Tris (hydroxymethyl)-aminomethan)

#### Description

#### Calibration

The pH combination electrode is calibrated in technical buffer pH=4.00 and pH= 7.00 or in DIN buffer pH= 4.01 and pH= 6.87.

Example of the calibration documentation:

#### Calibration

#### Buffers used

pH buffer 1: TEC\_4.000 pH buffer 2: TEC\_7.000

#### Measured values

pH buffer 1: TEC\_4.000 165.6 mV / 23.4 °C pH buffer 2: TEC\_7.000 -11.2 mV / 23.0 °C

#### Calibration data

 Slope:
 99.4 % / -58.8 mV/pH

 Zero point:
 pH 6.81 / -11.2 mV

 Temperature:
 23.4 °C (a)

 Date and time:
 07.03.13 / 15:04

#### Determination of the exact concentration of the standard solution (option)

The exact concentration of the  $H_2SO_4$  0.05 mol/L titrant can be determined using a titrimetric standard Tris (hydroxymethyl)-aminomethan. TRIS is dried in a desiccator before the titer determination overnight at room temperature.

The standard method for HCI/H2SO4 titrant ("titer HCI) is stored as a default method inside the TitroLine® 6000/7000/7750 titrators. With EDIT/F3 - Default method you can load this method. It is only necessary to change the name of the method and the factor F2 in the calculation formula:

Factor 2 (F2): 1000.0000 Factor 1 (F1): 1.0000

The factor 1 (F1) should be changed to 2.

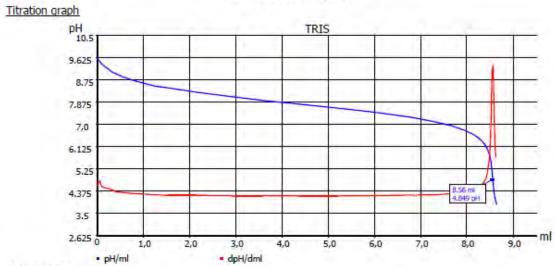
In a 100 or 150 ml, 0.2 to 0.3g TRIS are weighed accurately and dissolved in 60/80 mL of dist. water with stirring. It is titrated with  $H_2SO_4$  0.05 mol/L.

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### Documentation example for standard titration of H2SO4/HCI with TRIS

#### **GLP** documentation



#### Method data

Method name: Titre HCl Titration duration: 3 m 8 s
End date: 13.09.12 End time: 14:39:30

#### Titration data

Sample ID: TRIS Weight: 0.1038 g Start pH: pH 9.590 End pH: pH 3.864 Start temperature: 25.0 °C (m) End temperature: 25.0 °C (m)

Zero point: pH 6.83 / -10.0 mV Slope: 100.6 % / -59.5 mV/pH

EQ: 8.560 ml / pH 4.849 Titre; 0.1001 mol/l

#### Calculation formula

Titre: (W\*F2)/((EQ1-B)\*M\*F1) -> M103

Mol (M): 121.14000

 Weight (W):
 man
 Factor 2 (F2):
 1000.0000

 Blank value (B):
 0.0000 ml
 Factor 1 (F1):
 1.0000

Statistics: Off

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#### Method for standard titration of H2SO4/HCI with TRIS

Method data overall view

Method name: Titre HCl Created at: 09/13/12 14:23:02
Method type: Automatic titration Last modification: 09/13/12 14:27:56

Measured value: pH Damping settings: None Titration mode: Dynamic Documentation: GLP

Dynamic: Steep

Measuring speed / drift: Normal: minimum holding time: 02 s

maximum holding time: 15 s

Measuring time: 02 s

Drift: 20 mV/min

Initial waiting time: 0 s
Titration direction: Decrease
Pretitration: Off

End value: 2.500 pH EQ: On (1)

Slope value: Steep Value: 700

Dosing parameter

Dosing speed: 100 % Filling speed: 30 s

Maximum dosing volume: 50.00 ml

Unit values

 Unit size:
 20ml

 Unit ID:
 10039005

 Reagent:
 HCl 0.1 mol/L

 Batch ID:
 no Charge

 Concentration [mol/l]:
 0.10070

Determined at: 12/05/11 19:18:45

Expire date: 08/18/12
Opened/compounded: 09/10/11
Test according ISO 8655: 05/10/11

Last modification: 09/13/12 14:35:18

Device information

Device: TitroLine 7000

Serial number: 00012 Software version: 1230 Titre\_HCl\_13\_09\_12-14\_36\_21.pdf

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#### Titration of the sample

The sample is either centrifugated 20 min or filtrated before use. It is also important that a larger amount of the sample is homogenized before filtration/centrifugation. The sample should be treated always in the same manner.

20 ml of the filtrated sample (or less) are pipetted in a beaker and diluted with water that the pH electrode and titration tip can immerse correctly.

Select the FOS TAC method and start the titration.

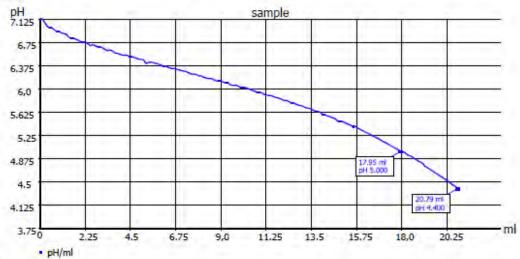
#### Result example:

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#### GLP documentation

#### Titration graph



#### Method data

Method name: FOS TAC Titration duration: 7 m 9 s
End date: 08.01.13 End time: 17:39:42

#### Titration data

Pattern: 20.00000 ml
Start pH: pH 7.148 End pH: pH 4.390
Start temperature: 25.0 °C (m) End temperature: 25.0 °C (m)

Zero point: pH 6.88 / -6.7 mV Slope: 97.5 % / -57.7 mV/pH

EP1: 17.950 ml / pH 5.000 TAC: 4487.50

EP2: 20.788 ml / pH 4.400 FOS: 2280.54

FOS/TAC: 0.51

#### Calculation formula

TAC: (F1/V)\*EP1\*F2

FOS: ((F1/V)\*(EP2-EP1)\*F3-F4)\*F5

FOS/TAC: (F6\*F7)/(F8\*F9)

20,0000 ml (f) Pattern (V): Factor 1 (F1): 20,0000 Factor 2 (F2): 250.0000 Factor 3 (F3): 1.6600 Factor 4 (F4): 0.1500 Factor 5 (F5): 500.0000 Factor 6 (F6): 2280.54 (FOS) Factor 7 (F7): 1.0000 Factor 8 (F8): 4487.50 (TAC) Factor 9 (F9): 1,0000

Statistics: Off

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#### Method

The method is available as default method in the TL 6000/7000/TL 7750 titrator and ready to use.

#### Method data overall view

 Method name:
 FOS TAC
 Created at:
 01/08/13 17:30:29

 Method type:
 Automatic titration
 Last modification:
 01/08/13 17:32:28

Measured value: pH Damping settings: None Titration mode: End pt. Documentation: GLP Linear steps: 0.050 ml

Measuring speed / drift: Normal: minimum holding time: 02 s

maximum holding time: 15 s

Measuring time: 02 s

Drift: 20 mV/min

Initial waiting time: 0 s
Titration direction: Decrease
Pretitration: Off

Endpoint 1: pH 5.000 delta endpoint 1: pH 0.400

Endpoint delay 1: 5 s
Endpoint 2: pH 4.400 delta endpoint 2: pH 0.400
Endpoint delay 2: 5 s

Dosing parameter

Dosing speed: 40 % Filling speed: 30 s Maximum dosing volume: 50.00 ml

Unit values

 Unit size:
 20ml

 Unit ID:
 10039005

 Reagent:
 HCl 0.1 mol/L

 Batch ID:
 no Charge

 Concentration [mol/l]:
 1.66666

Determined at: 01/07/13 21:41:57

Expire date: 08/18/12
Opened/compounded: 09/10/11
Test according ISO 8655: 05/10/11

Last modification: 01/07/13 13:42:17

Device information

Device: TitroLine 7000 Serial number: 10003645

Software version: 1301 FOS\_TAC\_08\_01\_13-17\_32\_33.pdf 2/2

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#### **Notes**

If you have any questions on the application, you can feel free to contact us..

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