## SI Analytics

## Application

a xylem brand

## Determination of Acid number and free fatty acids (FFA) in fats and oils

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## Use

The method is suitable for edible fats and oils such as butter, olive, palm or sunflower oil. The acid number is the quantity of base, expressed in milligrams of potassium hydroxide, that is required to neutralize all acidic constituents present in 1 g of sample. The calculation of the \% FFA depends on the titrated type of sample.

## Appliances

- Titrator: TL 7000/TL 7750 M1
- Basic device
- Magnetic stirrer TM 235
- 10 mL Exchange unit WA 10, with amber glass bottle for the titrant, complete


## Electrodes

- Electrode: N 6480 eth
- Electrolyte: L 5034 (LiCl/ethanol)
- Calibration: n.a.


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## Reagents

- Titrant: KOH $0.1 \mathrm{~mol} / \mathrm{I}$ in IPA (2-propanol). Also KOH $0.1 \mathrm{~mol} / \mathrm{I}$ in ethanol
- Titer determination: Potassium hydrogen phthalate
- Solvent: Ethanol/diethyl ether (1:1)


## Description

Determination of the exact concentration of the KOH titrant
We recommend ready to use KOH titrants. The exact concentration of the $\mathrm{KOH} 0.1 \mathrm{~mol} / \mathrm{l}$ can be determined using the titrimetric standard potassium hydrogen phthalate.

In a 150 mL beaker, 0.2 g of the standard are weighed accurately and dissolved in 80 mL of dist. water with stirring. It is titrated with the $0.1 \mathrm{~mol} / \mathrm{KOH}$ solution.

Repeat the standardization two times. The average value is stored automatically in the exchangeable unit.


Pic. left: titer

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Page 1: Curve and result: Titer determination

## GLP documentation

Itration graph


Method data

## Method name: <br> End date:

Iltration data
Sample ID:

EQ:
$4.933 \mathrm{ml} /-91.2 \mathrm{mV}$
Titer:
$0.1032 \mathrm{~mol} / \mathrm{l}$

## Calculation formula

| Titer: | $\left(\right.$ W $^{*}$ F2 $) /\left((\text { EQ1-B })^{*} M^{*}\right.$ F1 $)->$ M103 |
| :--- | :--- |
| Mol (M): | 204.22000 |


| Weight (W): | man |
| :--- | :--- |
| Blank value (B): | 0.0000 ml |
| Statistics: | Off |

Factor 2 (F2):
1000.0000
Factor 1 (F1): $\quad 1.0000$

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Page 2: Method parameters Titer determination:

## Method data overall view

Method name:
Method type:
Measured value:
Titration mode:
Dynamic:

Titer KOH
Automatic titration
mV
Dynamic

Steep

| Measuring speed / drift: | Normal: |
| :--- | :--- |
|  |  |
|  |  |
| Initial waiting time: | 0 s |
| Titration direction: | Decrease |
| Pretitration: | Off |
| End value: | Off |
| EQ: | On (1) |
| Slope value: | Steep |

minimum holding time: 02 s
maximum holding time: 15 s
Measuring time: 02 s
Drift: $\quad 20 \mathrm{mV} / \mathrm{min}$

Value:
700

## Dosing parameter

| Dosing speed: | $100 \%$ | Filling speed: |
| :--- | :--- | :--- |
| Maximum dosing volume: | 50.00 ml |  |

## Unit values

| Unit size: | 10 ml |
| :--- | :--- |
| Unit ID: | 00072696 |
| Reagent: | TBA Hydroxid |
| Batch ID: | 1.0265 |
| Concentration [mol/l]: | 0.10320 |
| Determined at: | $09 / 20 / 120: 57: 27$ |
| Expire date: | $04 / 12 / 12$ |
| Opened/compounded: | $10 / 19 / 11$ |
| Test according ISO 8655: | $12 / 01 / 10$ |
| Last modification: | $09 / 21 / 1215: 13: 56$ |

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## Titration of the sample

Weigh the sample in a 100 ml beaker and add at least 50 ml of the solvent mixture to the sample. If necessary heat the solution to dissolve the sample.

The sample weight should be calculated and selected that the titration amount is not more than 5 ml because of the long titration time.

For acid numbers between 0.2 and 1 the sample amount should be about $10-20 \mathrm{~g}$.
For acid numbers between 1 and 10 the sample amount should be about $1-3 \mathrm{~g}$.
Place the beaker on the magnetic stirrer and start the titration method. After the titration rinse the electrode and burette tip with solvent. For each set of samples perform a blank titration with 50 ml of the titration solvent.

## Result calculation

The enclosed titration example shows the calculation of the result in $\mathrm{mg} \mathrm{KOH} / \mathrm{g}$ sample (acid number).

The calculation of the \% FFA value depends on the titrated sample. For many oil and fat samples the molecular weight of the oleic acid $(282 \mathrm{~g} / \mathrm{mol})$ is used.
$\%$ FFA $=(E Q 1-B) * 282 * T * 100 /(1000 * W)$
EQ1: ml consumption at the equivalence point
B : ml consumption for the blank titration
282: molecular weight of oleic acid in $\mathrm{g} / \mathrm{mol}$
T : concentration of the KOH titrant (e.g. $0.1 \mathrm{~mol} / \mathrm{l}$ )
100: per 100 g sample
1000: conversation
W : sample weight in g

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Blank titration page 1: Curve and result

## GLP documentation

Titration graph


Method data

| Method name: | Blank AN |
| :--- | :--- |
| End date: | 30.04 .13 |

$\begin{array}{ll}\text { Titration duration: } & 6 \mathrm{~m} 14 \mathrm{~s} \\ \text { End time: } & 11: 44: 44\end{array}$
Titration data

| Sample ID: | Solvent |  |  |
| :--- | :--- | :--- | :--- |
| Start mV: | 23.2 mV | End $\mathrm{mV}:$ | -79.3 mV |
|  |  |  |  |
| EQ: | $0.099 \mathrm{ml} /-46.3 \mathrm{mV}$ | Blank: | 0.099 ml |

## Calculation formula

Blank:
EQ1 -> M02

Statistics:
Off

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Blank titration page 2: method

## Method data overall view

| Method name: | Blank AN |
| :--- | :--- |
| Method type: | Automatic |
| Measured value: | mV |
| Titration mode: | Linear |
| Linear steps: | 0.010 ml |
|  |  |
|  |  |
|  |  |
| Measuring speed / drift: | 12 s |
|  |  |
|  |  |
| Initial waiting time: | 10 s <br> Titration direction: <br> Pretitration: |
| Decrease <br> End value: | Off <br> Off <br> EQ: |

Dosing parameter

| Dosing speed: | $100.00 \%$ | Filling speed: | 30 s |
| :--- | :--- | :--- | :--- |
| Maximum dosing volume: | 0.30 ml |  |  |
| Unit values |  |  |  |
| Unit size: | 10 ml |  |  |
| Unit ID: | 00072696 |  |  |
| Reagent: | TBA Hydroxid |  |  |
| Batch ID: | 1.0265 |  |  |
| Concentration [mol/l]: | 0.10350 |  |  |
| Determined at: | $09 / 21 / 12$ 22:27:50 |  |  |
| Expire date: | $04 / 12 / 12$ |  |  |
| Opened/compounded: | $10 / 19 / 11$ |  |  |
| Test according ISO 8655: | $12 / 01 / 10$ |  |  |
| Last modification: | $09 / 21 / 12$ 15:28:02 |  |  |

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Sample titration page 1: Curve and result

## GLP documentation

Titration graph


## Method data

$$
\begin{array}{ll}
\text { Method name: } & \text { Acid number } \\
\text { End date: } & 30.04 .13
\end{array}
$$

Titration data

## Sample ID: <br> Start mV:

EQ:

## Olive oil

123.5 mV
$0.548 \mathrm{ml} /-47.4 \mathrm{mV}$

Titration duration:
End time:

3 m 33 s
12:19:19

AN mg KOH/g:
0.260

Weight:
10.03650 g

End mV:
$-94.6 \mathrm{mV}$

AN mg KOH/g:
(EQ1-B) ${ }^{*} \mathrm{~T}^{*} \mathrm{M}^{*} \mathrm{~F} 1 /\left(\mathrm{W}^{*} \mathrm{~F} 2\right)$
0.0990 ml (M02)
$\begin{array}{ll}\text { Blank value (B): } & 0.0990 \\ \text { Factor } 1 \text { (F1): } & 1.0000\end{array}$
Factor 2 (F2): $\quad 1.0000$

## Calculation formula

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Sample titration page 2: method

## Method data overall view

| Method name: | Acid number | Created at: | 04/29/13 16 |
| :---: | :---: | :---: | :---: |
| Method type: | Automatic titration | Last modification: | 04/29/13 16 |
| Measured value: | mV | Damping settings: | strong |
| Titration mode: | Linear | Documentation: | GLP |
| Linear steps: | 0.050 ml |  |  |
| Measuring speed / drift: | User-defined: | minimum holding time: | 07 s |
|  |  | maximum holding time: | 20 s |
|  |  | Measuring time: | 04 s |
|  |  | Drift: | $10 \mathrm{mV} / \mathrm{min}$ |
| Initial waiting time: | 10 s |  |  |
| Titration direction: | Decrease |  |  |
| Pretitration: | Off |  |  |
| End value: | Off |  |  |
| EQ: | On (1) |  |  |
| Slope value: | Flat | Value: | 120 |

## Dosing parameter

| Dosing speed: | $100.00 \%$ |
| :--- | :--- |
| Maximum dosing volume: | 6.00 ml |
| Unit values |  |
| Unit size: | 10 ml |
| Unit ID: | 00072696 |
| Reagent: | TBA Hydroxid |
| Batch ID: | 1.0265 |
| Concentration [mol/l]: | 0.10350 |
| Determined at: | $09 / 21 / 12$ 22:27:50 |
| Expire date: | $04 / 12 / 12$ |
| Opened/compounded: | $10 / 19 / 11$ |
| Test according ISO 8655: | $12 / 01 / 10$ |
| Last modification: | $09 / 21 / 1215: 28: 02$ |

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## Notes

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

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