Thermo. Titr. Application Note No. H-036

Title:	Determination of Free Fatty Acids (FFA) in olive oil

Scope:	Determination of free fatty acids (FFA) in oils.

Principle:	The oils are dissolved in a mixture of toluene and 2-propanol (1:1) and titrated with *c*(KOH) = 0.1 mol/L in 2-propanol.

Sample:	Oleic acid, Olive oil
Sample Preparation:	No sample preparation was necessary. The samples could be weighed directly into the titration vessels.

Reagents:	- KOH in 2-propanol, 0.1 mol/L, Merck 1.05544.1000 - Benzoic acid, puriss. ≥ 99.5 %, Fluka 12349 - Toluene, purum ≥ 99.0%, Fluka 89682 - 2-propanol, purum > 99.0%, Fluka 59310 - Oleic acid, pract., Fluka

Method:	**Basic experimental parameters for FFA determination:** Titrant delivery rate (mL/min): 1 No. of endothermic endpoints: 1 Data smoothing factor: 90 **Procedure:** The sample was weighed directly into the titration vessel. Approximately 25 mL of the solvent (1:1 mixture of toluene and 2-propanol) were added. After 5 sec of stirring the mixture was titrated with *c*(KOH) = 0.1 mol/L to the first exothermic endpoint. **Titer determination of KOH in 2-propanol:** Benzoic acid was dried for 2 hours at 105℃ and coo led down in a desiccator. Exactly 0.3352 g of the benzoic acid were weighed into a 200 mL-volumetric flask and made up to the mark with distilled water. Then 10, 15, 20 and 25 mL of the solution were dosed into the titration vessel. Dist. water was added to reach a volume of approx. 35 mL. The sample size (in mmol) was then plotted on the x-axis with corresponding volumes of titrant on the y-axis. A linear regression was performed. The molarity of the NaOH-solution is the reciprocal of the gradient. In this instance, the y-intercept was not used as the method blank, due to the need to match the sample matrix.

Results:

- Oleic acid

Sample size [g]	Volume of titrant [mL]	FFA [%]
0.2151	7.735	100.95
0.2236	8.040	100.95
0.1688	6.032	100.25
0.1767	6.386	101.41
0.1573	5.688	101.43
0.1560	5.576	100.25
0.1288	4.649	101.18
0.1233	4.421	100.49
0.1058	3.836	101.55
0.1088	3.905	100.54
Mean value **SD** **RSD**		100.90% 0.492% 0.49%

Fig. 1: Results of the determination of FFA in oleic acid

- Olive oil

Sample size [g]	Volume of titrant [mL]	FFA [%]
12.0522	1.251	0.290
14.3130	1.480	0.289
9.9720	1.023	0.287
16.5299	1.686	0.286
17.9919	1.837	0.286
18.3100	1.875	0.287
15.9473	1.611	0.283
14.1140	1.432	0.284
12.0272	1.223	0.284
9.9525	1.011	0.284
Mean value **SD** **RSD**		0.286 % 0.0024% 0.86%

Fig. 2: Results of the determination of FFA in olive oil

Titer determination of KOH in 2-propanol:

Sample size [mL]	Sample size [mmol]	Volume of titrant [mL]
10	0.343	3.468
10	0.343	3.490
15	0.274	2.794
15	0.274	2.798
20	0.206	2.122
20	0.206	2.109
25	0.137	1.404
25	0.137	1.414

Fig. 3: Results of the titer determination of KOH in 2-propanol

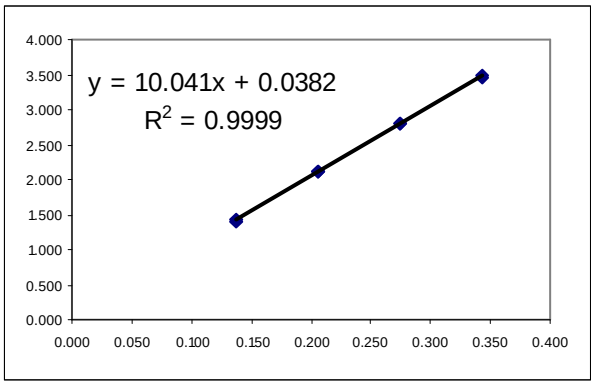

Fig. 4: Regression analysis to determine the concentration of KOH in 2-propanol

Molarity = 1/gradient = 1/10.041 = 0.0996 mol/L

Calculation:

$$FFA\,(\%) = \frac{(EP1 - blank) \times Conc\,(KOH) \times Titer\,(KOH) \times MW\,(Oleic\ acid) \times 100}{Sample\ size \times 1000}$$

with:

EP1	= First endpoint
blank	= Method blank
Conc(KOH)	= Concentration of the KOH in 2-propanol
Titer(KOH)	= Titer of the KOH in 2-propanol
MW(x)	= Molecular weight of the analyte
100	= Calculation factor for %
1000	= Conversion factor for L

Thermometric Titration Plot:

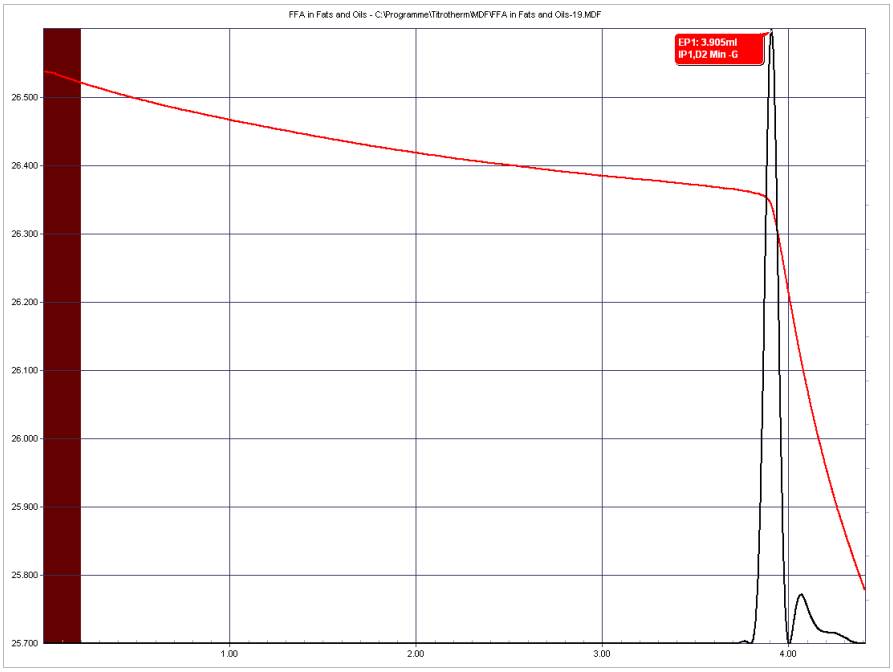

Fig. 5: Example curve of the determination of oleic acid

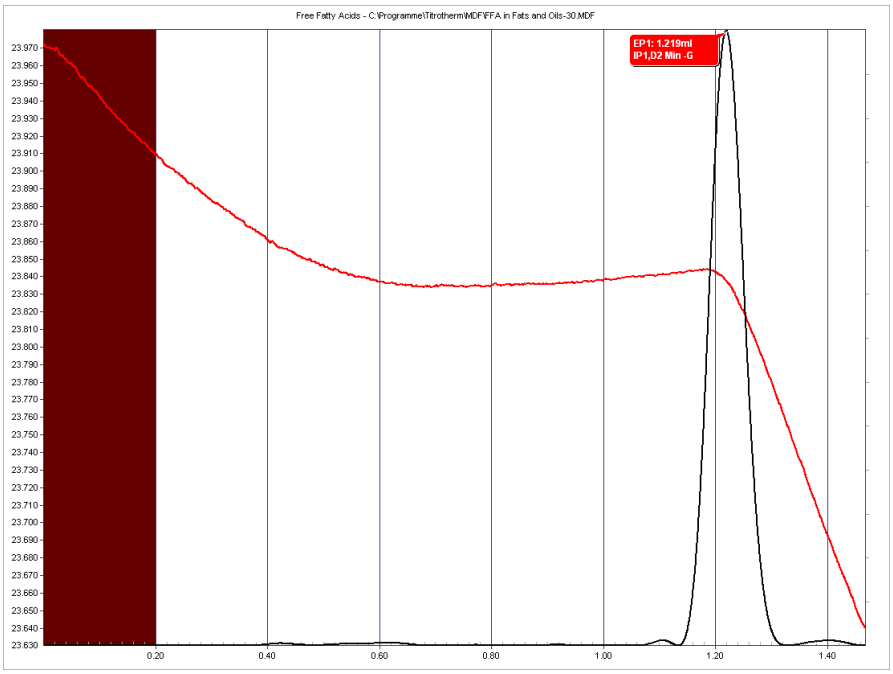

Fig. 6: Example curve of the determination of olive oil

Legend:
Red = solution temperature curve
Black = second derivative curve
Brown area = Endpoints in this area are ignored