Thermo. Titr. Application Note No. H-040

Title:	Determination of HCl (ppm range) in silicone oil

Scope:	Determination of low level contents around 10 ppm of HCl in silicone oil.

Principle:	The silicone oils were dissolved in a mixture of toluene and 2-propanol (1:1) and titrated with *c*(KOH) = 0.01 mol/L in 2-propanol.

Sample:	Different silicone oil samples
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 - Paraformaldehyde, purum > 95.0%, Fluka 76240

Method:	**Basic experimental parameters for the HCl determination:** Titrant delivery rate (mL/min): 1 No. of endothermic endpoints: 1 Data smoothing factor: 80 Stirring rate: 10 **Procedure:** The samples were weighed directly into the titration vessel. 40 mL of the solvent (1:1 mixture of toluene and 2-propanol) and approx. 0.5 g of paraformaldehyde were added. After 5 sec of stirring the mixture was titrated with *c*(KOH) = 0.01 mol/L (50 mL of KOH 0.1 mol/L were diluted with 2-propanol to 500 mL) to the first exothermic endpoint. To determine the method blank for sample 1 and 2 the sample sizes [in g] were plotted on the x-axis with the corresponding volumes of titrant [in mL] on the y-axis. A linear regression was carried out and the y-intercept corresponds to the method blank. **Determination of the concentration of KOH in 2-propanol:** Benzoic acid was dried for 2 hours at 105℃ and coo led down in a desiccator. Exactly 0.3154 g of the benzoic acid were weighed into a 100 mL-volumetric flask, approx. 5 mL of 2-propanol were added to dissolve the benzoic acid and the solution made up to the mark with dist. water. Then different amounts of the solution (0.5, 0.75, 1.0 and 1.25 mL) were dosed into the titration vessel and solvent was added to reach a volume of approx. 35 mL. The volumes of the titrant were then plotted on the x-axis with the corresponding sample sizes (in mmol) on the y-axis. A linear regression was performed. The slope of the resulting curve represents the concentration of the NaOH-solution.

Results:

Sample size [g]	Volume of titrant [mL]	HCl [ppm]
12.1170	0.648	8.31
14.1120	0.705	8.54
10.9020	0.619	8.30
13.1420	0.670	8.24
8.2940	0.562	8.50
12.0860	0.644	8.20
Mean value **SD** **RSD**		8.35 ppm 0.140 ppm 1.68 %

Fig. 1: Results of the determination of HCl, Sample 1

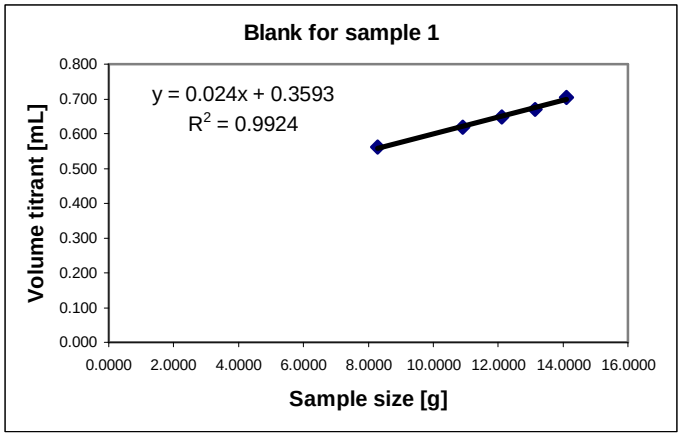


Fig. 2: Blank determination, Sample 1

Sample size [g]	Volume of titrant [mL]	HCl [ppm]
12.0060	0.533	10.97
13.0620	0.572	11.10
15.2530	0.640	11.07
14.0000	0.584	10.66
10.8920	0.509	11.33
14.1780	0.668	12.60
10.6320	0.522	12.02
Mean value **SD** **RSD**		11.39 ppm 0.677 ppm 5.94 %

Fig. 3: Results of the determination of HCl, Sample 2

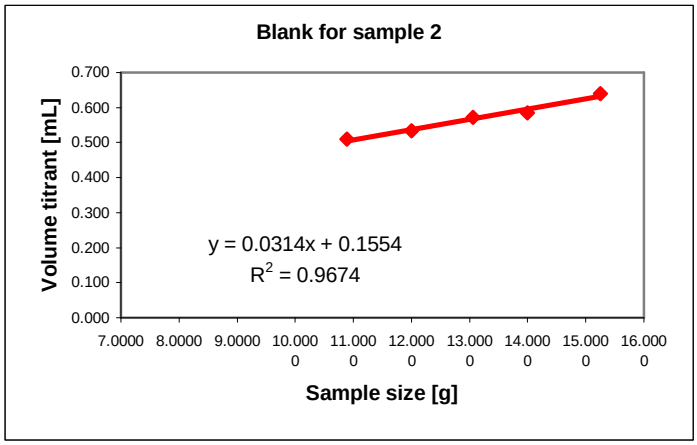


Fig. 4: Blank determination, Sample 2

Titer determination of KOH in 2-propanol:

Sample size [mL]	Sample size [mmol]	Volume of titrant [mL]
0.5	0.0129	1.730
0.5	0.0129	1.736
0.75	0.0194	2.391
0.75	0.0194	2.443
1.0	0.0258	3.020
1.0	0.0258	3.020
1.25	0.0323	3.789
1.25	0.0323	3.782

Fig. 5: Results of the titer determination of KOH in 2-propanol

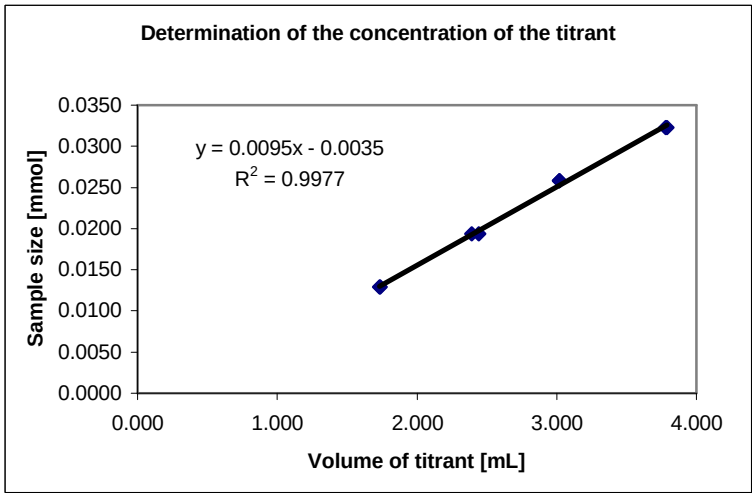
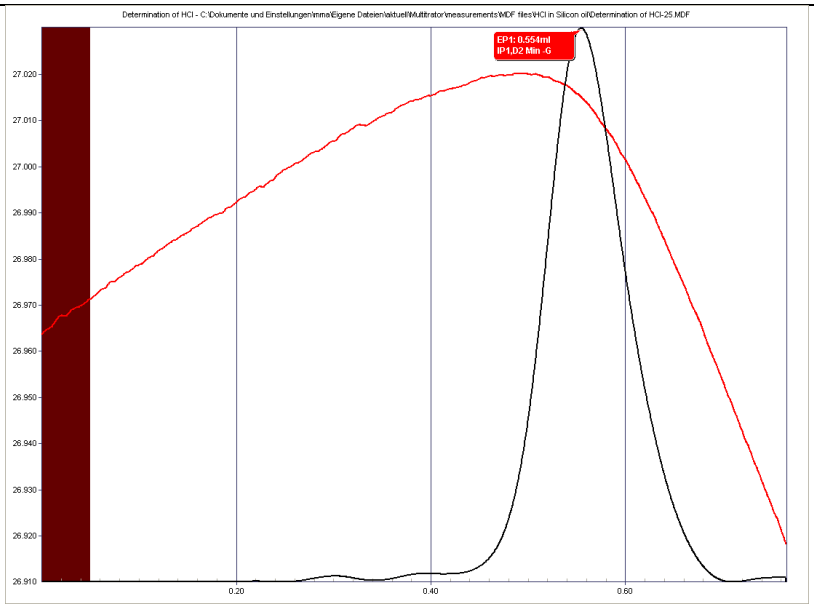


Fig. 6: Regression analysis to determine the concentration of KOH in 2-propanol

Concentration = slope of linear regression = 0.0095 mol/L

Calculation:	$$ppm\,HCl = \frac{(EP1 - blank) * conc(KOH) * 36.46 * 1000}{sample\,size}$$ with: EP1 = First endpoint [mL] blank = Method blank [mL] Conc(KOH) = Concentration of the KOH in 2-propanol [mol/L] 36.46 = Molecular weight of HCl [g/mol] 1000 = Conversion factor for ppm [mg/g] sample size = size of the sample [g]
Thermometric Titration Plot:	 **Fig. 7:** Example curve of the HCl content in silicone oil Legend: Red = solution temperature curve Black = second derivative curve Brown area = Endpoints in this area are ignored