## Thermo. Titr. Application Note No. H-001

## Title: Determination of TAN in Oils

| Scope: | Determination of Total Acid Number (TAN) values in <br> mineral oils and similar fluids. |
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| Principle: | Dissolve oil sample in mixture of toluene and propan-2-ol, <br> add paraformaldehyde and titrate with 0.1 M KOH in <br> propan-2-ol. The endpoint is indicated by a strongly <br> endothermic response caused by the base-catalyzed de- <br> polymerization of paraformaldehyde. |
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| Reagents: | $0.1 \mathrm{~mol} / \mathrm{L} \mathrm{KOH}$ in iso-propanol (standardized) |
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| $50 \%$ A.R. toluene : 50\% A.R. propan-2-ol by volume |  |
|  | A.R. paraformaldehyde fine powder (eg, Sigma-Aldrich <br> cat. no. 158127 ) |


| Method: | Basic Experimental Parameters: |
| :---: | :---: |
|  | Data rate (per second) 20 |
|  | Titrant delivery rate (mL/min.) 1 |
|  | No. of endothermic endpoints 1 |
|  | Data smoothing factor 50 |
|  | Procedure: |
|  | Weigh accurately approximately $0.5-2 \mathrm{~mL}$ oil in a clean dry titration vessel (the aim is to obtain a titre of approx. 1 $\mathrm{mL} \mathrm{KOH})$. Add 30 mL of toluene/propan-2-ol mixture. Add $\sim 0.5-0.6 \mathrm{~g}$ paraformaldehyde (a level $1 / 8^{\text {th }}$ kitchen teaspoon measure is $\sim 0.5 \mathrm{~g}$ ). Titrate to an inflection characterized by a sudden reduction in temperature. |

Results: Analysis of a heavy vehicle hydraulic oil:

| Sample Mass, g | $\mathrm{mL} 0.1 \mathrm{~mol} / \mathrm{L} \mathrm{KOH}$ | TAN mg KOH/g sample |
| :---: | :---: | :---: |
| 1.7447 | 0.875 | 2.60 |
| 1.8842 | 0.940 | 2.60 |
| 1.9237 | 0.960 | 2.61 |
| 1.8487 | 0.924 | 2.60 |
| 1.8494 | 0.924 | 2.60 |
| 1.4029 | 0.718 | 2.60 |
| 0.9727 | 0.519 | 2.60 |
| 0.5049 | 0.304 | 2.60 |
|  | Average | 2.60 |
|  | Standard Deviation | 0.002 |


| Calculation: | TAN $=\mathrm{mg} \mathrm{KOH} / \mathrm{g}$ oil |
| :--- | :--- |
|  | $\left.\therefore T A N=\frac{((\text { titre }, m L-\text { blank }, m L) \times M K O H \times F W K O H}{}\right)$ |
| sample mass, $g$ |  |
|  | Example: |
|  | $T A N=\frac{((0.940-0.071) \times 0.1006 \times 56.11)}{1.8842}=2.60$ |

## Thermometric Titration Plot:



## Legend:

Red $=$ solution temperature curve
Black = second derivative curve

## Determination of titration blank or offset:



Titration blank or offset $=y$-intercept $=0.0705 \mathrm{~mL}$ (which is to be subtracted from each titre)

