Study Committee A2

Power Transformers & Reactors

Paper A2-11064_2022

TESTING CHALLENGES WITH ESTER INSULATING LIQUIDS

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Motivation

- there are still many open questions about the test methods/diagnostic procedures of ester insulating liquids
- discussion of some important test methods (still missing in the standards) :
    - calibration and interpretation of DGA
    - monitoring of inhibitors
    - compatibility between different ester Insulation liquids
    - detection of mineral oil contamination

➢ DGA- relative calibration method

- by using a known mineral oil calibration curve, it is possible to construct a calibration curve for any other insulating liquid- by using *relative slope factors* ***F***

what is needed

- fault gas free oil:

  +inhibited isoparaffinic/naphthenic and uninhibited oil
  +synthetic and natural ester **+**silicon oil
- specific gas mixtures in at least three concentrations
- glovebox/rotating table
- gaschromatograph

how it works

- floating the rotating table with specific gas mixtures
- fault gas free oil is filled to a defined ratio in the vial after a current time
- the vial is closed hermetic and analyzed in the GC via Head Space

prerequistes

- Linear calibration curves (with zero point as origin=>blind value analysis is necessary)
- Equal oil/gas ration
- Equal conditioning and extraction temperatures

The following diagrams show the dispersion of acetylene and ethylene:

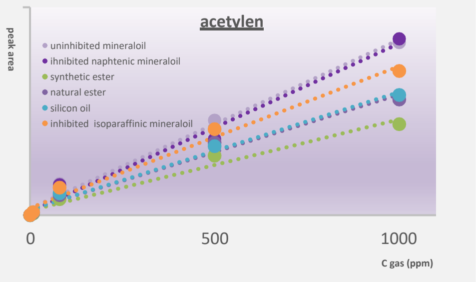

Fig. 1: *. calibration curves for acetylene with different insulating liquids and gas concentrations*

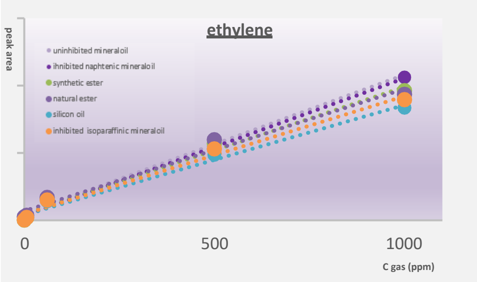

Fig. 2: *. calibration curves for ethylene with different insulating liquids and gas concentrations*

- the dispersion for acetylene is much higher than for ethylene=> more significant differences in the gas solubility of the tested insulating liquids for the gas acetylene than for the gas ethylene
- this is expressed in the **value of F**

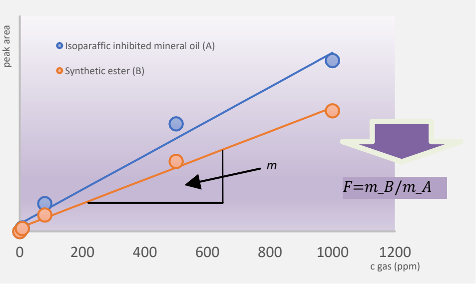

Fig. 3: *. determination of the factor F*

creating a calibration curve

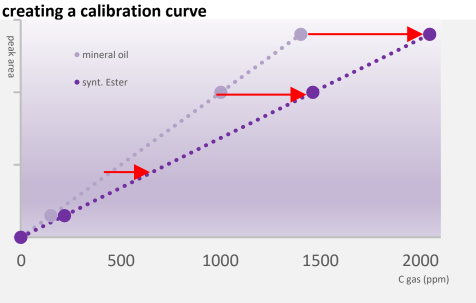

Fig. 4: *creating a calibration curve*

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continued

- multiplying the gas concentrations in mineral oil with the value 1/F to get a calibration curve for each other insulating liquid

F<1: better soluble in this liquid than in mineral oil

***Tab. 1:** examples for F multiplication factors for the investigated insulating liquids*

F	isoparaffic/ inhib. mineral oil	non-inhib. mineral oil	naphthenic/ inhib. mineral oil	synth. ester	soya natural ester	silicon liquid
CO_2	1,00	1,02	1,04	0,86	0,70	0,80
C_2H_4	1,00	1,11	1,10	1,03	0,98	0,91
C_2H_2	1,00	1,10	1,09	0,69	0,76	0,81
C_2H_6	1,00	1,11	1,08	1,13	0,93	0,98
C_3H_6	1,00	1,09	1,10	1,10	0,99	1,05
C_3H_8	1,00	1,11	1,11	1,17	1,10	1,09
H_2	1,00	1,11	1,11	1,17	1,10	1,09
CH_4	1,00	0,98	0,95	0,98	0,98	0,84
CO	1,00	0,97	0,97	0,93	1,00	0,90

- the determined factors for mineral oils are very similar, independent of the mineral oil constitution.
- e.g., acetylene is better soluble in silicon liquids, synthetic and natural esters than in mineral oil

➢ DGA- evaluation of synthetic esters

- synthetic ester in transformers of offshore wind farms or traction applications usually operate at high temperatures
- consider not only generally known rules for gas formation of electrical and thermal faults, but also thermo-oxidative gas formation ("stray gas formation") of the liquid itself in connection with the presence of copper
- use of different, experimental schemata for condition assessment for transformers filled with ester
- **BUT:** faults can rarely be placed in one classification
  - only limited use for real cases
- interpretation via combination of threshold values and gas quotients makes sense
- due to different operation, construction, cooling etc. values cannot be postulated identically for different transformer fleets
  - 95% and 99% limits (for transformer fleet of interest) are useful
- the following main types of faults are known from the evaluation of mineral oils:
  - PD – partial discharge faults,
  - D – arc discharge faults
  - T – thermal faults (T1: <300°C; T2: >300°C and <700°C; T3: >700°C)

***Tab. 2:** proposed criteria for PD,D and T faults in synthetic esters*

fault	key gas value	key gas ratios
PD	H_2≥100 ppm	CH_4/H_2≤0,2 C2H2/C2H4≤0,1
D	H2≥50 ppm and C2H4≥10 ppm	CH4/H2≤1 C2H2/C2H4≥1 C2H4/C2H6≥2
T	C2H4 ≥ 50 ppm	CH4/H2≥0,3 C2H2/C2H4≤0,1 C2H4/C2H6≥1

comparison of the interpretation schemes between mineral oil and synthetic ester

- direct comparison between the used evaluation schemes for mineral oil and synthetic ester do not show significant differences for the major types of faults

***Tab. 3:** comparison between the ratio criteria for mineral oil and for synthetic esters for PD, D and T faults*

case	C_2H_2/C_2H_4		CH_4/H_2		C_2H_4/C_2H_6	
	mineral oil	synth. ester	mineral oil	synth. ester	mineral oil	synth. ester
PD	NS	≤ 0,1	< 0,1	≤ 0,2	< 0,2	NS
D1	>1	≥ 1	0,1 to 0,5	≤1	>1	≥2
D2	0,6 -2,5		0,1 to 1		>2	
T1	NS	≤0,1	>1	≥0,3	<1	≥1
T2	<0,1		>1		1-4	
T3	<0,2		>1		>4	

➢ ADDITIVES IN ESTER INSULATING LIQUIDS AND THEIR MONITORING

- additives: slows down the aging process
- oxidation inhibitors and metal passivators are typical additives
- inhibitors for ester liquids are mostly phenol-based with a high flash point

***Fig. 5:** examples for inhibitors used in ester liquids*

- metal passivators protect the copper parts built into a transformer

Irgamet 39 BTA

***Fig. 6:** examples for passivators used in ester liquids*

- metal passivators protect the copper parts built into a transformer

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continued

- to present, there is no recognized method for quantifying additives in ester liquids
- a new (HPLC) method especially for ester insulating liquids has been developed
    - **first step:** liquid-liquid extraction of the additives with acetonitrile
    - **second step:** analysis of the extract by HPLC

optimization possibility for the HPLC:

- gradient of mobile phase: acetonitrile and water (1:1) -> 100% acetonitrile
- preferred wavelength: 273nm

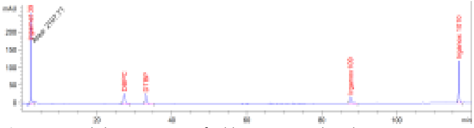

Fig. 7: *typical chromatogram of additives in ester liquids*

➢ COMPATIBILITY OF ESTER INSULATING LIQUIDS

- the compatibility assessment requires knowledge on used inhibitors and general liquid composition
- the miscibility and compatibility between the commercial synthetic esters on the market is usually provided
- in case of natural ester, a special emphasis shall be placed on susceptibility to oxidation

➢ DETECTION OF MINERAL OIL CONTAMINATION

- mineral oil contamination in ester liquids may:
    - reduce the biodegradability
    - reduce considerably the fire point

complicated tests

simple on-site test:
like **refraction index**

- a quick refraction index test (applicable even on site) can give useful information, whether contamination by another insulating liquid has taken place.
- examples of refractive indices of some insulating liquids are shown in the next table

Tab. 3: *examples for refractive indices*

n_D^{20}	**mineral oil (naphthenic type)**	**synthetic ester**	**silicon insulating liquid**
	1,4710	1,4522	1,4028

➢ CONCLUSIONS

- using a known mineral oil calibration curve, it is possible to construct a calibration curve for any other insulating liquid using relative slope factors F
- experimentally determined factors are very similar to those for mineral oil
    - evaluation schemes for these liquids are similar to those of mineral oil
- separate DGA evaluation for synthetic ester filled transformers is useful
- a suitable method for quantifying additives in ester insulating fluids using high pressure liquid chromatography was introduced
- some other preliminary studies on compatibility of ester types and detection of mineral oil contaminants are presented.