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Measuring Methods for Solubility of Gases in Insulation Liquids

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Since 2003 he is with ABB AG, Transformer Service in Halle, Germany, where he has hold different national and international positions. Since 2010 he is the general manager of the Transformer Service Workshop in Halle with more than 200 employees. He is member of VDE, IEEE, DKE K 182 insulation liquids and CIGRÉ as liason officer A2 - IEC TC 10 and active in different working Groups. He is the author or co-author of more than 100 publications and owner of more than 20 patents in Asset Management, Diagnostic Methods, Monitoring and High Voltage Testing.





Measuring Methods for Solubility of Gases in Insulation Liquids

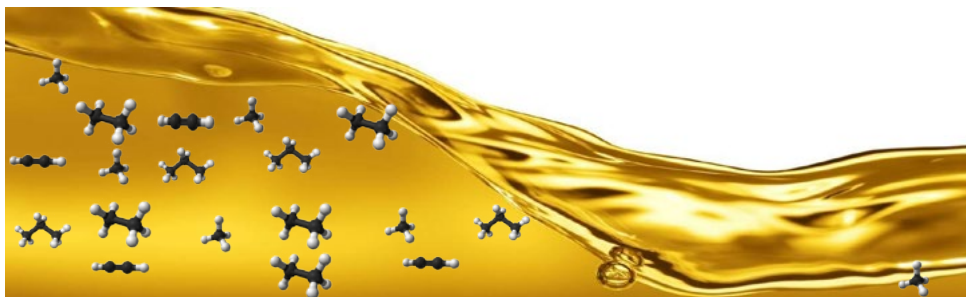
Measuring Methods for Solubility of Gases in Insulation Liquids



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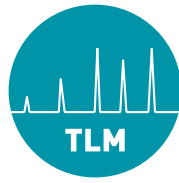


Motivation




- Knowing the gas solubility in insulating fluids is needed, because
 - it is applied for DGA Headspace method
 - it governs the gas diffusion into the liquid phase or vice versa in hermetic sealed transformers
 - enables a comparison of the undissolved trapped gases in the transformer Buchholz relay and those dissolved in oil, which could deliver information about transformer health condition
- No standardized method to quantitatively determine the gas solubility in liquid insulations

ASTM D2780-92 being already withdrawn!





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Motivation





IEEE Std C57.104-2008
IEEE Guide for the Interpretation of Gases Generated in Oil-Immersed Transformers

4) Various diagnostic techniques, such as key gases, Dissolved Gas Ratio, and Keygas ratios
 4) Instruments for detecting and determining the amount of combustible gases present
 0. A bibliography of related literature

1.2 Limitations

Other techniques for the detection and the measurement of gases have been established. However, it must be recognized that the analysis of these gases and interpretation of their significance is, at this time, not a science but an art subject to variability. Their presence and quantity are dependent on equipment variables such as type, location, and management of the tank, reliability and degree of maintenance of various gases in the type of the generation system, the type and rate of oil circulation, the kinds of material in contact with the fault and fault, variables associated with the sampling and measuring procedure themselves. Because of the variability of acceptable gas limits and the significance of various gases and generation rates, a consensus is difficult to obtain. The principal obstacle in the development of final interpretations is an exact relation in the lack of positive correlation of the flash-identifying gases with faults found in actual transformers.

The result of various ASTM testing procedures indicates that the analytical procedure for gas analysis are difficult, have some variation, and can be widely inaccurate, especially between laboratories. A methodological facilities available. However, whether used separately or as complements to one another, the procedures discussed in this guide all provide the operator with useful information concerning the solubility of the equipment.

2. Normative references

The following referenced documents are indispensable for the application of this document (i.e., they must be understood and used, so each referenced document is cited in text and its relationship to this document is explained). For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments or corrigenda) applies.

ASTM D 923, Standard Practice for Sampling Electrical Insulating Liquids.¹
 ASTM D 2943, Standard Test Method for Gas Content of Insulating Oils.

¹ASTM publications are available from the American Society for Testing and Materials, 100 Bar Harbor Drive, West Conshohocken, PA 19380-1524, USA (http://www.astm.org).

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IEEE Guide for the Interpretation of Gases Generated in Oil-Immersed Transformers

C57.104™

IEEE Power and Energy Society
Sponsored by
Transformers Committee


IEEE
3 Park Avenue
New York, NY 10161-4007, USA
2 February 2008

IEEE Std C57.104™-2008
Revision of
IEEE Std C57.104-1997


Many techniques for the detection and the measurement of gases have been established. However, it must be recognized that analysis of these gases and interpretation of their significance is, at this time, not a science but an art subject to variability.

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Motivation



■ Gas solubility coefficients provided by IEEE C57.104 and IEC 60599 are inconsistent resulting in misleading DGA interpretations

Gas	k (IEC 60599)	k (IEEE C57.104)	Difference in %
N ₂	0.091	0.0745	22.1
H ₂	0.056	0.0429	30.5
O ₂	0.172	0.138	24.6
CO	0.132	0.102	29.4
CO ₂	1.09	0.9	21.1
CH ₄	0.429	0.337	27.3
C ₂ H ₆	2.82	1.99	41.7
C ₂ H ₄	1.84	1.35	36.3
C ₂ H ₂	1.24	0.938	32.2

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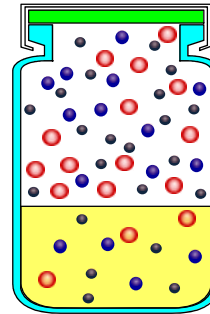


Ostwald Coefficient



- Henry's law:
 - The ratio of partial pressure to mole fraction of gas in solution is a constant
- Ostwald coefficient
 - The solubility of a gas is the volume of gas dissolved per volume of liquid when the gas and liquid are in equilibrium at the specified partial pressure of gas and at the specified temperature

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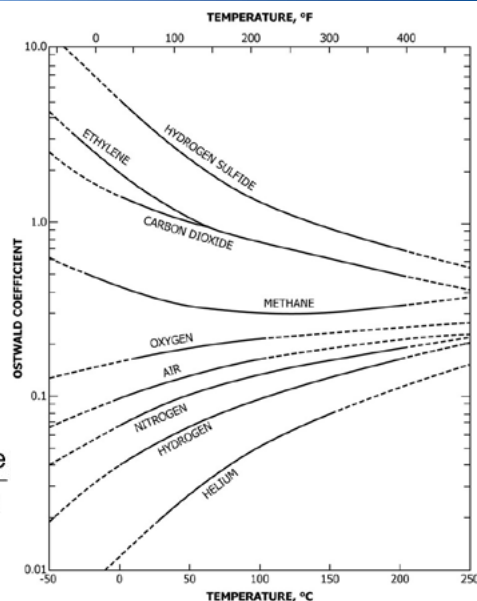
Ostwald Coefficient



- Ostwald Coefficient dependencies
 - Pressure
 - Temperature
 - Moisture
 - Solvent and solute compositions
 - ...?

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$$k = \frac{\text{concentration of gas in liquid phase}}{\text{concentration of gas in gas phase}}$$





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Glass Syringe Method



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- **Measurement procedure**
 - A 50ml glass syringe filled with 30ml insulation liquid
 - Residual gas volume decreased to less than 0.3%
 - 20ml pure key gas was injected
 - The syringe kept in a heating chamber at 25°C for 72h
 - And subjected to the periodical shaking



- **Dilemma**
 - Gas leakage at elevated temperature (75°C)

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Measuring of Dissolved Gases



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- **Full-Vacuum degassing using TOGA GC**
 - Establishment of vacuum ranging from 2 to 200mbar for app. 2min
 - Complying with the standard IEC 60567
 - Detection of extracted gases through Thermal Conductivity Detector (TCD) and a Flame Ionization Detector (FID)



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New Method



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- **Test set-up for gas saturation in liquid**
 - The vessel was filled with 150ml of mineral insulating fluid
 - Liquid was treated at room temperature again at a vacuum level of 0.05mbar for 180min to ensure the removal of gas traces dissolved in the investigated oil
 - The pressure of the key gas was regulated at 1020mbar at the temperature of 25°C for 72h
 - Measuring the dissolved gases using a full vacuum degassing unit connected to the GC

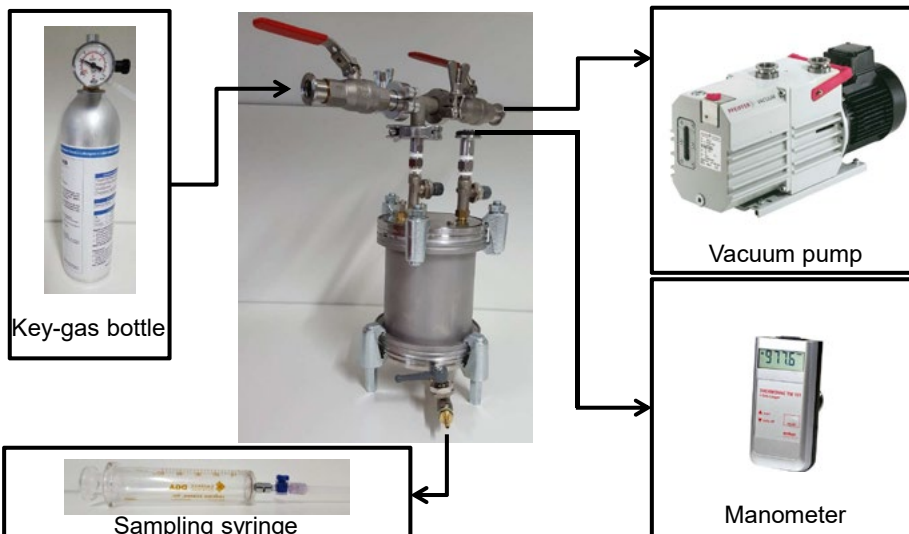


New Method




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- **Test set-up for gas saturation in liquid**



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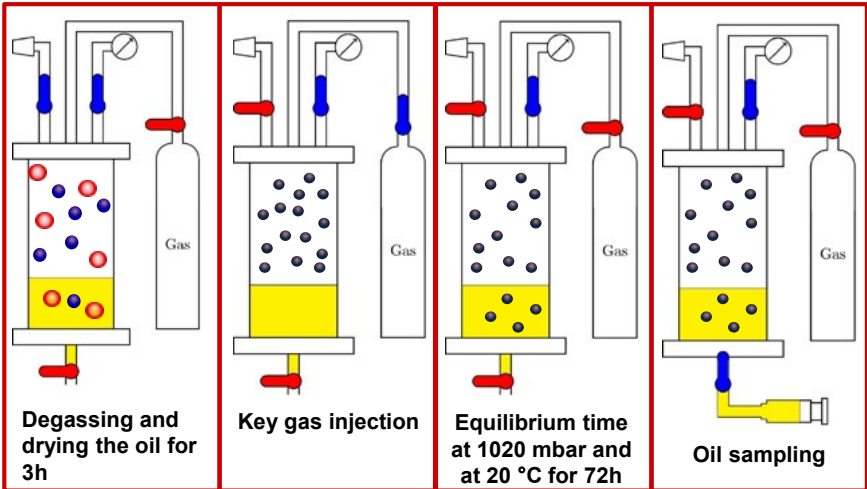


New Method




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- Gas saturation process




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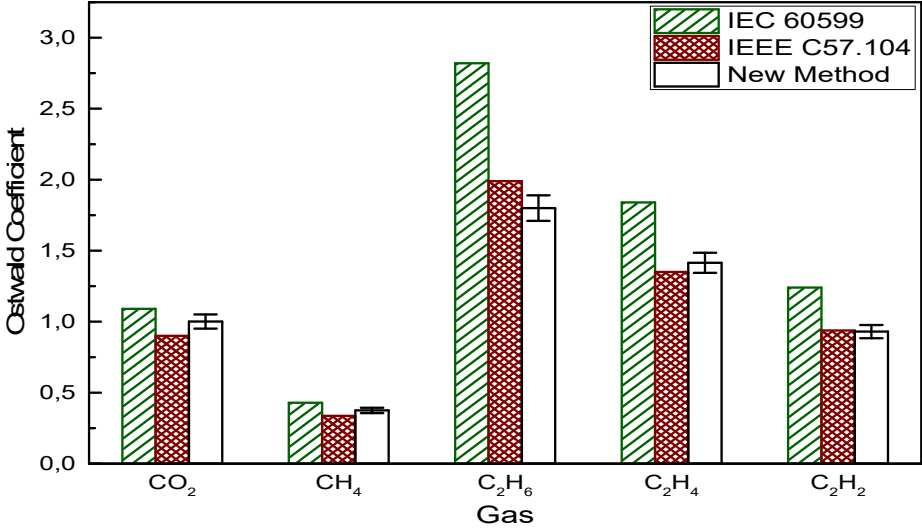


Results



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- The measured results are in between IEC and IEEE standard, but more close to IEEE



Gas	IEC 60599	IEEE C57.104	New Method
CO ₂	~1.1	~0.9	~1.0
CH ₄	~0.4	~0.3	~0.35
C ₂ H ₆	~2.8	~2.0	~1.8
C ₂ H ₄	~1.8	~1.3	~1.4
C ₂ H ₂	~1.2	~0.9	~0.95

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Results

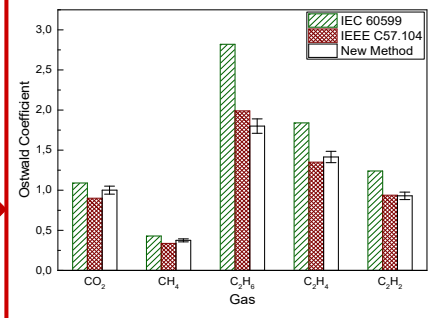


- Ostwald coefficient values by IEEE C57.104 and IEC 60599 are inconsistent
- Measured Ostwald coefficients of key gases in mineral oil is comparable with those provided by IEEE C57.104
- Good reproducibility

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Ostwald coefficient of key gases in mineral oil S2 ZU-I at room temperature and at the pressure of 1020 mbar in comparison to IEC 60599 and IEEE C57.104

Gas	IEC 60599	IEEE C57.104	New Method
N ₂	0,091	0,075	
H ₂	0,056	0,043	
O ₂	0,172	0,138	
CO	0,132	0,102	
CO ₂	1,090	0,900	1,001
CH ₄	0,429	0,337	0,375
C ₂ H ₆	2,820	1,990	1,800
C ₂ H ₄	1,840	1,350	1,415
C ₂ H ₂	1,240	0,938	0,930



Conclusion



- Gas solubility coefficients provided by IEEE C57.104 and IEC 60599 are inconsistent resulting in misleading DGA interpretations
- No standardized method to quantitatively determine this dynamic property of the binary gas-liquid system
- Gas solubility measurement on oil S2 ZU-I has verified the applicability of the new proposed method
- Establishment of a new standard method for determination of Ostwald coefficient to substitute ASTM standard D2780-92 being already withdrawn

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Measuring Methods for Solubility of Gases in Insulation Liquids



Outlook



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- **Verification of this approach should be subjected to a round robin test**
- **Influence of the following parameters should be investigated**
 - **Water content**
 - **Aging status of the oil (more importantly dispersed cellulose fibers)**
 - **Cross-influence of gases**
- **Ostwald coefficients for all important insulation liquids to be determined**
 - **Synthetic and natural esters**
 - **Silicone liquid**
 - **Etc....**

