

CHEMICAL & ENERGY ANALYSIS

ANALYSIS OF FURANIC COMPOUNDS IN TRANSFORMER OIL BY AGILENT 1290 INFINITY BINARY UHPLC

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INTRODUCTION

Furanic compounds are generated by the degradation of cellulosic materials used in the solid insulation systems of electrical equipment. These compounds are oil soluble to an appreciable degree which leads to migration into the insulating liquid so that high concentrations or unusual increases in the concentrations in oil may indicate cellulose degradation from aging or incipient fault conditions. The individual furanic compounds that may be identified and quantified include the following:

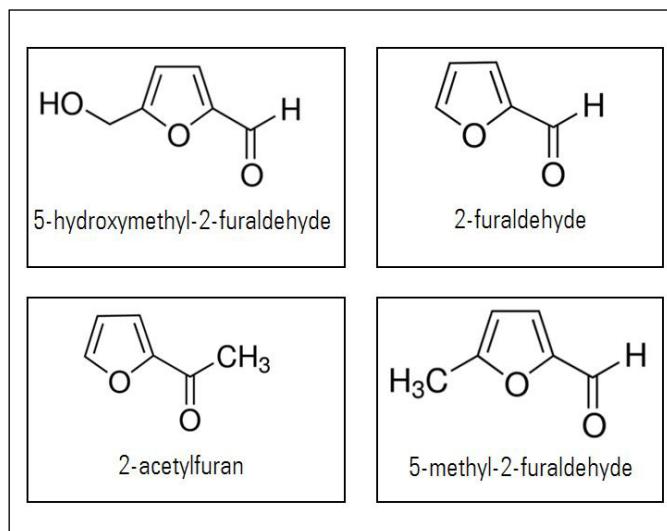
1. 5-hydroxymethyl-2-furaldehyde
2. 2-furaldehyde
3. 2-acetylfuran
4. 5-methyl-2-furaldehyde

Furanic compounds are typically present from 50 ppb to 9000 ppb according to the age of the transformer. Therefore, direct analysis and quantification of these compounds serves as an indicator of the age and health of the transformer. Since the concentration is directly related to the degradation of paper insulation inside the transformer the analysis might serve as an early warning of a catastrophic failure if ignored. Generally if a transformer is one to five years old, 30 ppb to 50 ppb furanic compounds may present in the oil; if it's five to ten years old then 50 ppb to 1000 ppb, ten to twenty years old 1500 ppb to 4000 ppb, and if more than twenty years old then concentration may go as high as 9000 ppb.

Chemically, furan belongs to the class of organic compounds of the heterocyclic aromatic series characterized by a ring structure composed of one oxygen atom and four carbon atoms. The simplest member of the furan family is furan itself, a colorless, volatile, and somewhat toxic liquid that boils at 31.36° C (88.45° F).



Chemical structures of furans:



Experimental

Standard HPLC methods for the analysis of furanic compounds typically consume significant quantities of solvents per run and are time consuming; the analyses are 25 to 30 minutes long and consume roughly 40 to 45 ml mobile phase. By taking advantage of 1.8 μm particle size column and high power range of the Agilent 1290 Infinity Binary pump, an ultrafast UHPLC method has been developed that takes 5.5 minutes or less and saves 90 % solvent compared to conventional methods.

Standards of 5-hydroxymethyl-2-furaldehyde, 2-furaldehyde, 2-acetylfuran, 5-methyl-2-furaldehyde were procured from Sigma Aldrich and dissolved in 100% acetonitrile to make the stock solution.

Sample extraction

2ml of 100% HPLC grade Acetonitrile was used for extraction of furans from 10ml of transformer oil samples. The mixture was subjected to vortex for 10 minutes and allowed to settle into two phases, eliminate, and make; the top portion was the furan extract and the bottom portion was non polar components.

The extract was diluted or injected neat as required to bring the peak area within the calibrated range. The appropriate dilution factor was applied in calculating the actual furan concentration in the oil.

Instrumentation and method

The Agilent 1290 Infinity UHPLC system with the following configurations were used for analysis:

Hardware Description
G4220A , Agilent 1290 Infinity Binary Pump
G4226A , Agilent 1290 Infinity Autosampler
G1316C , Agilent 1290 Infinity Thermostatted Column Compartment
G4212A , Agilent 1290 Infinity Diode Array Detector, Flow cell: 10 mm path length, 1 μL

Method Parameters

Column	Eclipse plus C-18 RRHD, 2.1 mm x100 mm x1.8 µm
Flow	0.8 mL/min
Column oven	40°C
Detector	40 Hz, 282 nm/4nm, ref- 400/100 nm. Slit 4
Injection volume	1 µL, wash time 10 second
Run Time	5.50 min

Method Parameters

Time[min]	A[%]	B[%]	Flow[ml/min]
0.00	90.00	10.00	0.8
2.50	90.00	10.00	0.8
4.20	0.00	100.00	0.8
4.30	90.00	10.00	0.8
5.50	90.00	10.00	0.8

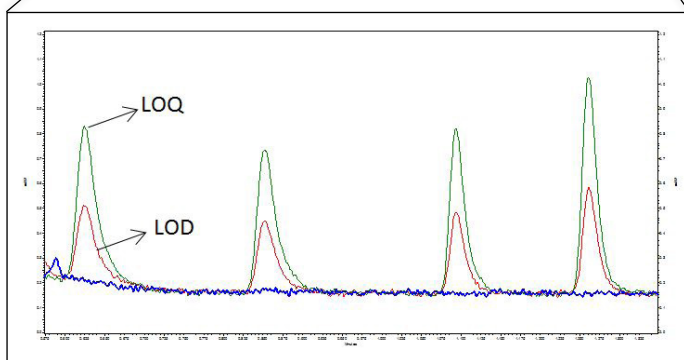
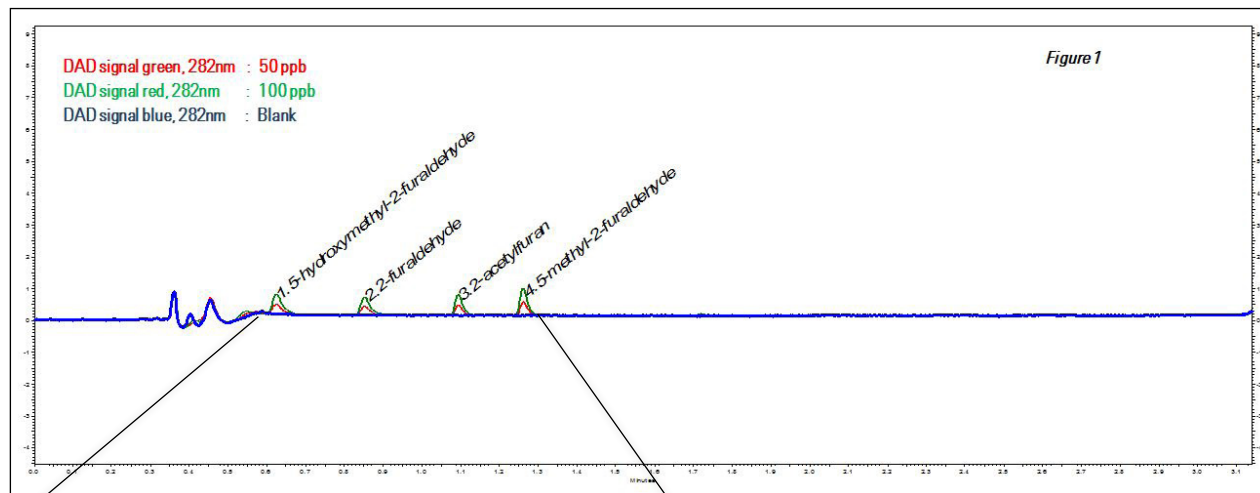
Mobile phase A: Milli Q Water

Mobile phase B: Acetonitrile HPLC grade

Consumption of mobile phase per run: 4.4 mL per run

Limit of detection [LOD] & Limit of quantitation [LOQ]

50 ppb was observed as detection limit for all four furanic compounds (Fig: 1), 100 ppb was observed as limit of quantitation.



RT	Compound	LOD, 50 ppb (S/N)	LOQ, 100 ppb (S/N)
0.62	5-hydroxymethyl-2-furaldehyde	5.9	11.3
0.85	2-furaldehyde	5.7	10.3
1.09	2-acetylfuran	6.5	12.3
1.26	5-methyl-2-furaldehyde	8.3	16.0

Calibration

Calibration has been done from 50 ppb to 9000 ppb on column for seven levels and good correlation has been achieved.

- L1 – 50 ppb
- L2 – 100 ppb
- L3 – 500 ppb
- L4 – 1000 ppb
- L5 – 3000 ppb
- L6 – 6000 ppb
- L7 – 9000 ppb

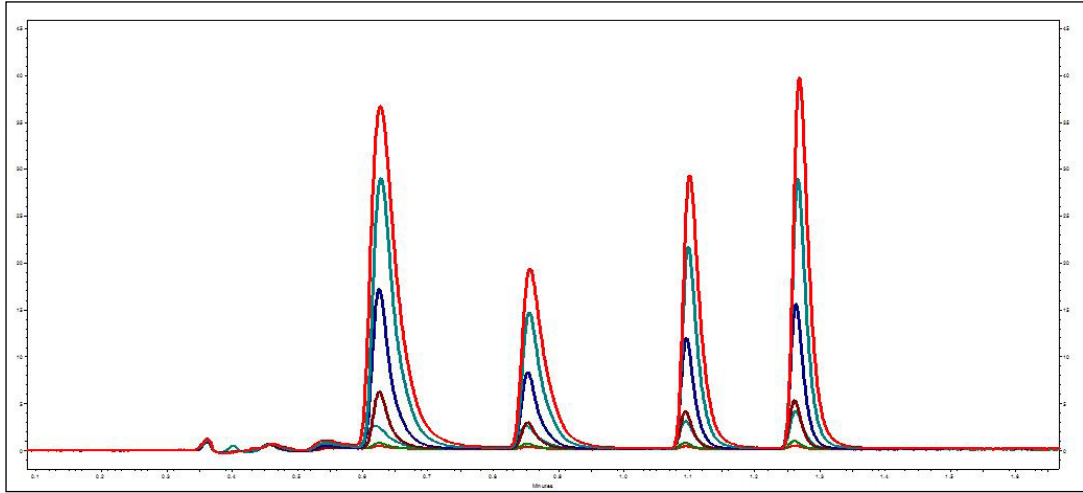
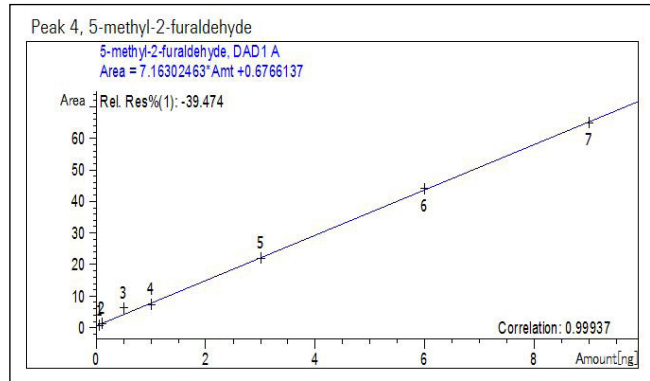
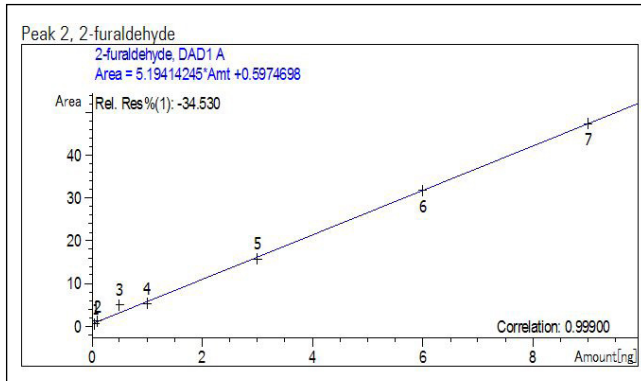
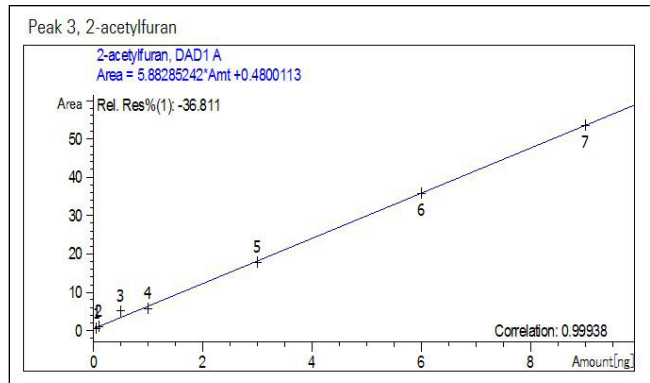
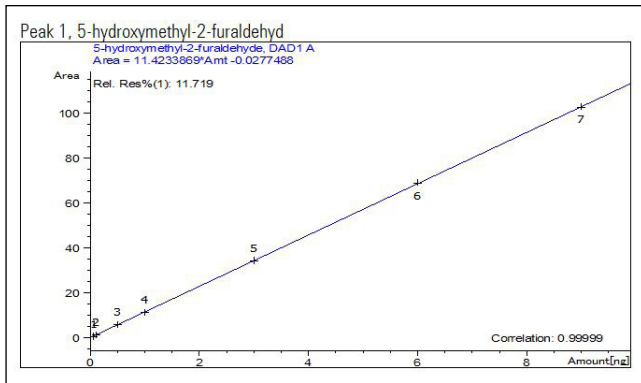


Figure 2: Overlay of linearity chromatogram L1-L7

Calibration curves for four furanic compounds



Precision of Method

Precision of method has been tested for consecutive ten replicate injections at 500 ppb concentration. Figure 3 shows the overlay of ten consecutive runs

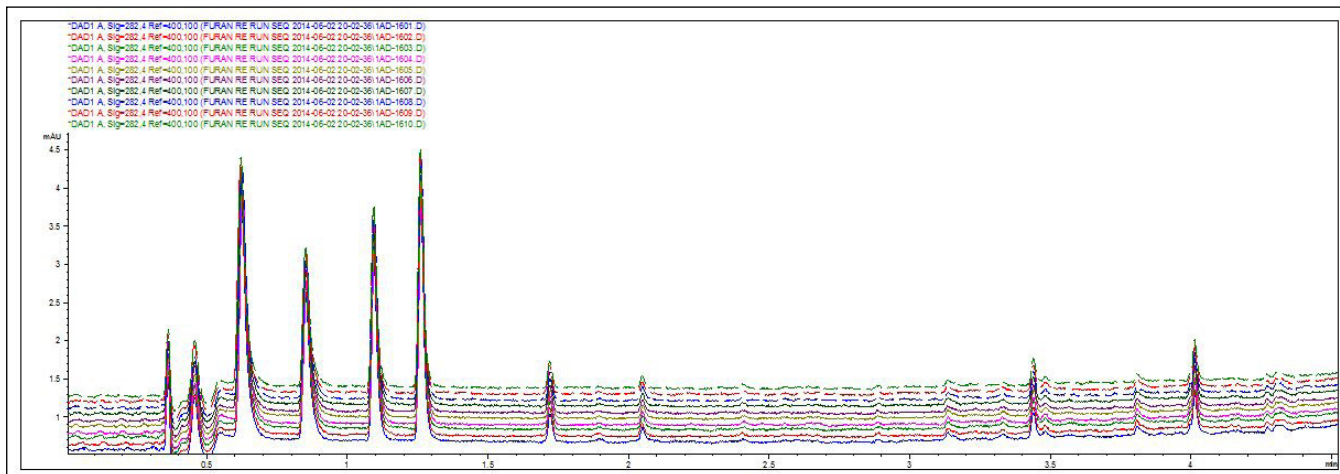
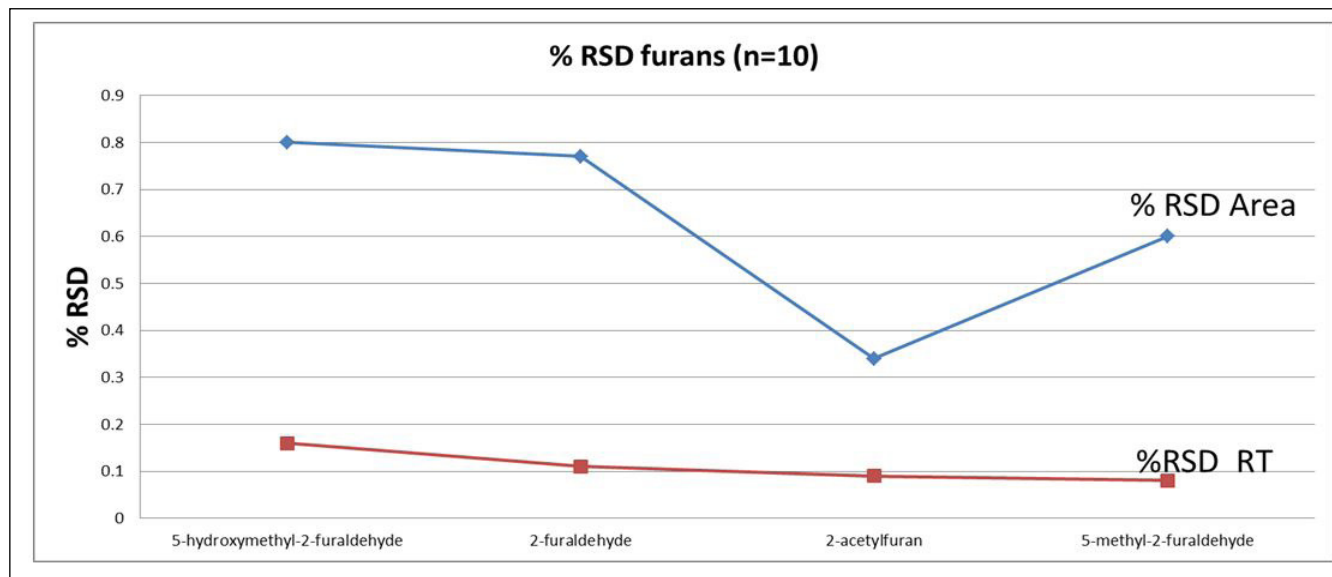


Figure 3: Overlay chromatogram of ten replicates [500ppb]



CONCLUSION

A fast method for the analysis of 4 furanic compounds in transformer oil in 5.50 minutes with a detection limit of 50 ppb and quantitative limit of 100 ppb has been developed based upon the Agilent 1290 Infinity UHPLC. Good linearity was observed from 50 ppb to 9000 ppb with a linear correlation of 0.999 for all four compounds. The method was tested for precision with ten replicate runs which gave a maximum of 0.2 % and 0.8 % RSD for RT and area respectively.

REFERENCE:

Liquid-liquid extraction procedure Method-A, Standard Test Method (ASTM) for Furanic Compounds in Electrical-Insulating Liquids by High Performance. Liquid Chromatography, Designator: 05837

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