PETRONAS LUBRICANTS INTERNATIONAL

Corrosività di lubrificanti e fluidi per gestione termica:

evoluzione degli standard internazionali e delle metodologie di test.

Dr. Fabio Cova Caiazzo, ***Innovation Project Leader,*** ***fabio.Caiazzo@pli-petronas.com***

April 3rd MILAN, UNICHIM "Prodotti Petroliferi e Lubrificanti" Pleanry

Lubricants performances &

importance of corrosivity assessment

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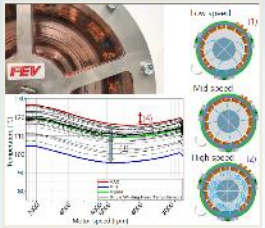
Fluid research and testing capabilities for Fluids Development

In-house tests, with capabilities to customize in accordance with customers' bespoke & technical requirements



Electrical test

- Electrical conductivity ASTM D2624
- Dielectric breakdown voltage IEC 60156
- Relative permittivity, dielectric dissipation factor, resistivity IEC 60247:2004

Compatibility test

Open to customizations

- PCB, integrated microelectronic & ECU components compatibility test

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Fluid development

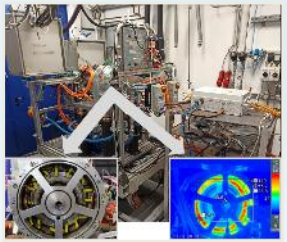
Corrosion test

Open to customizations

- High temperature corrosion bench test
- Wire corrosion test
- Liquid-vapor & vapor phase corrosion test ASTM D130 modified

Thermal test

- Thermal conductivity D7896
- Specific heat capacity ASTM E1269
- Thermo copper In house



Mechanical test

For example, durability test for reduction gear

Dedicated test

For example, battery immersion cooling test rig

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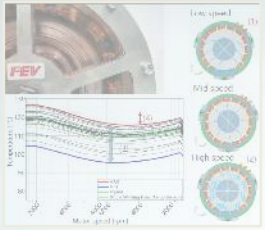
In-house tests, with capabilities to customize in accordance with customers' bespoke & technical requirements



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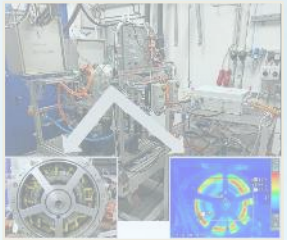


Mechanical test

For example, durability test for reduction gear

Dedicated test

For example, battery immersion cooling test rig

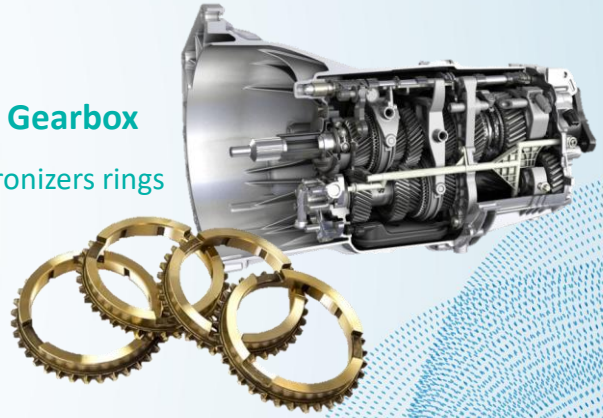
Copper and Copper alloys in automotive hardware

Electric Drive Units

- ✓ Electronic Control Unit (ECU)
- ✓ E-motor windings

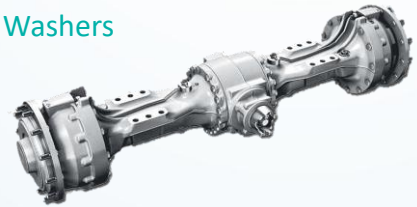
Manual Gearbox

- ✓ Synchronizers rings



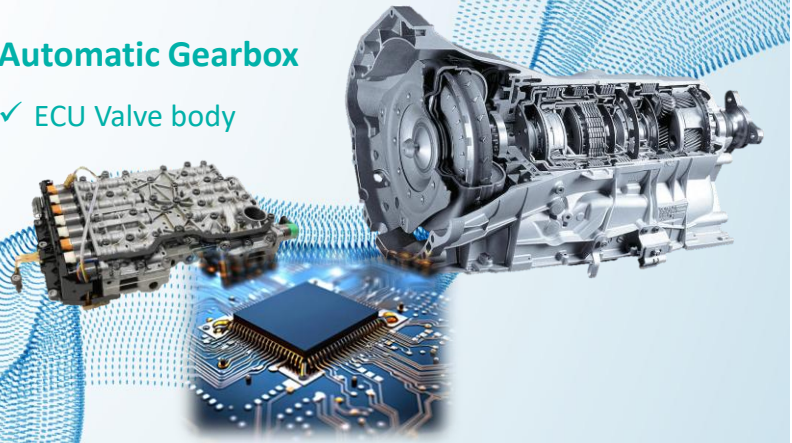
LS Axles and Wet Bake system

- ✓ Sinter Bronze Friction Plates
- ✓ Washers



Automatic Gearbox

- ✓ ECU Valve body



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Copper and metal alloys in thermal management applications

Battery Electric Vehicles

- ✓ Batteries
- ✓ High Power Charger

Data Centers

- ✓ Liquid Immersion Cooling
- ✓ Direct to chip cooling

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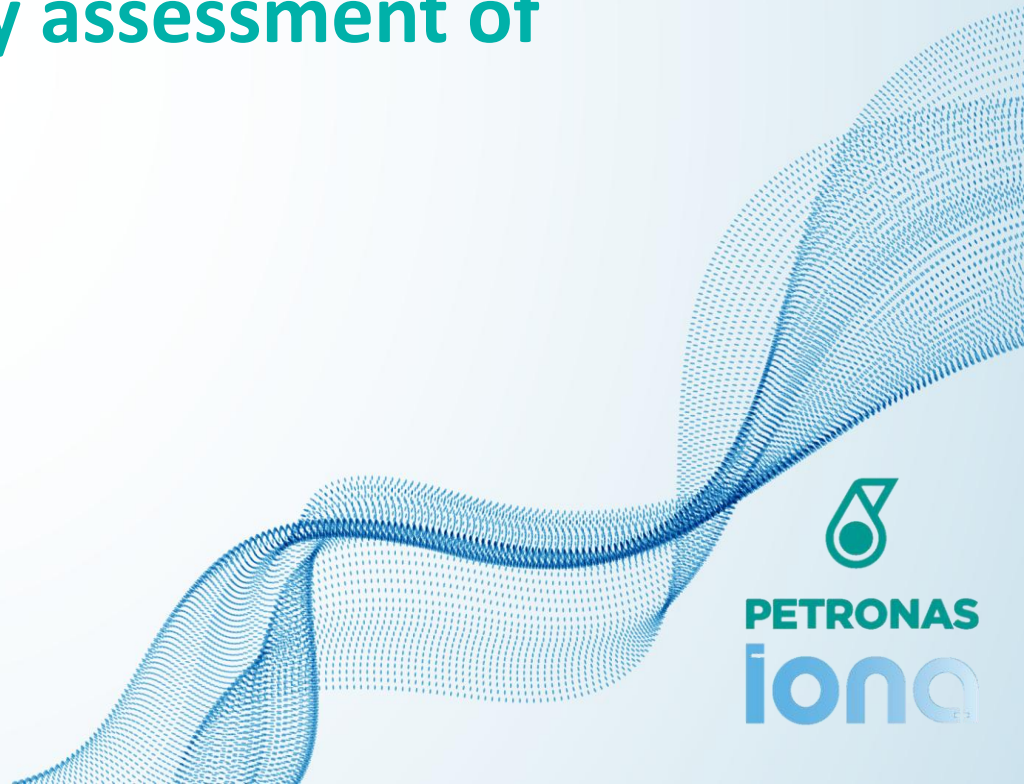
Evolution of Corrosivity assessment of Lubricants & Fluids



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Corrosivity Assessment Evolution:

Methods, Customer Requirements and Standard

ASTM D 130
ASTM D6594
State of the art for corrosivity assessment.

2016 2017 2018 2019 2020 2021 2022 2023 2024 2025

Compatibility with Copper and other metals: ASTM D130 & D6594

Designation: D130 – 19

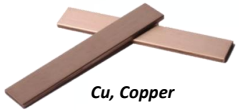
ASTM D130

Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test[1]

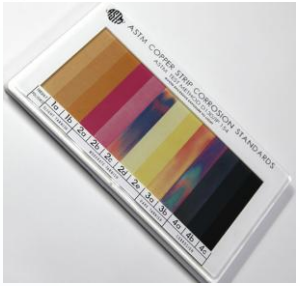
This standard is issued under the fixed designation D130; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

Federation of Societies for Paint Technology Standard No. DI-28-65
British Standard 4351



Cu, Copper



Report

- Test Duration (hrs.)
- Temperature (°C)
- Classification category (1,2,3 or 4)
- Classification description (a, b or c)

\+ **Easy to execute**

\+ **Well accepted industry std**

\- **Not Quantitative**

\- **No kinetic information**

Industry accepted modifications to ASTM D 130:

I. Measure of Cu released by means of ICP-AES (ASTM D5185) was introduce to provide quantitative information.

II. Extension of the test to 1000 hrs + Measure of Cu released over the time was introduce to provide kinetic information.

Designation: D6594 – 20ε1

ASTM D6594

Standard Test Method for Evaluation of Corrosiveness of Diesel Engine Oil at 135 °C[1]

This standard is issued under the fixed designation D6594; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε1 NOTE—Editorially updated TMC governance information in June 2022.

Report

- Test Duration (hrs.)
- Temperature (135°C)
- Cu released (ICP-AES ASTM D5185)
- Pb released (ICP-AES ASTM D5185)
- Cu Rating ASTM D130 Classification category (1,2,3 or 4)
- Cu Rating ASTM D130 Classification description (a, b or c)

\+ **Easy to execute**

\+ **Well accepted industry standard**

\+ **Quantitative**

\- **No kinetic information**

Cu, Copper

Phosphor Bronze

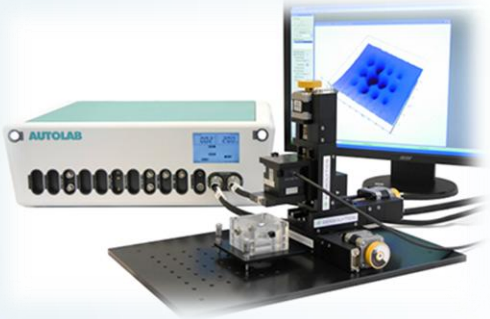
Sn, Tin

Pb, Lead

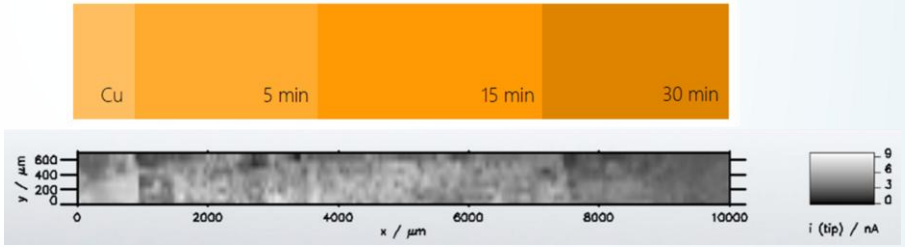
Limitations and misinterpretations of: ASTM D130[1]

In presence of surface-active molecules, as corrosion Inhibitors, ASTM D130 interpretation can be misleading

Benzotriazole base molecules are the most typical Corrosion inhibitor used in Lubricants industry to protect Cu and Cu alloys

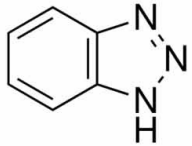


Scanning Electrochemical Microscopy



Tarnishing degree

Corrosion Current



Adsorption of Benzotriazole on Cu

[1]*Scanning electrochemical microscopy (SCEM) Principle and Application.*

Dr. Michaela Nabel. Metrohm Users Meeting 2019, Zofinghen.

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Wire resistance method for measuring the carrion of copper by lubricating fluids.
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Michel P Gahagan et al./International Journal of Automotive Engineering 7 (2016) 115-120

Research Paper 20164092

New Insights on the Impact of Automatic Transmission Fluid (ATF) Additives on Corrosion of Copper

- The Application of a Wire Electrical Resistance Method -

Michel P Gahagan [1)] Gregory J Hunt [2)]

1)-2) Lubrizol Ltd., The Knowle, Nether Lane, Hazelwood, Derbyshire, DE56 4AN, UK

Received on July 4, 2016

Presented at the JSAE Annual Congress on May 27, 2016

LUBRICATION SCIENCE
Lubrication Science 2017; **29**:279–290
Published online 9 December 2016 in Wiley Online Library (wileyonlinelibrary.com). DOI: 10.1002/ls.1368

Wire resistance method for measuring the corrosion of copper by lubricating fluids

Gregory J. Hunt*,†, Michael P. Gahagan and Mitchell A. Peplow

Lubrizol Limited, The Knowle, Nether Lane, Hazelwood, Derby DE56 4AN, UK

Bethan Warren[1*], Gregory J. Hunt[2†], Michael Bryant[1], Anne Neville[1], Ardian Morina[1], Michael Gahagan[2]

[1] Institute of functional surfaces, School of Engineering, Leeds University, LS2 9JT, UK
[2] Lubrizol Limited, The Knowle, Nether Lane, Hazelwood, Derby DE56 4AN, UK
*Corresponding author, email: bethan@djwarren.co.uk
†Email: Gregory.hunt@lubrizol.com

Automatic transmission fluid corrosion inhibitor interactions with copper

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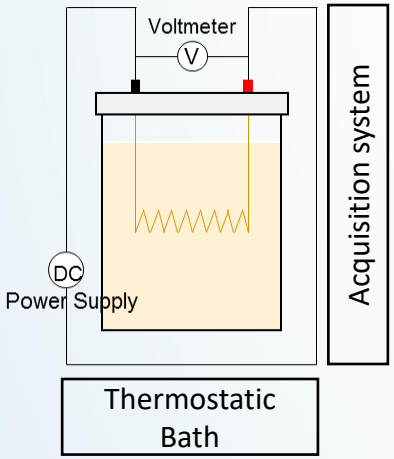
Wire resistance method[2,3,4]: Principles

Despite the method is not yet an ASTM standard, **PLI** recognized its potentialities and **decided to invest resources to implement it** in house as **key method for e-fluids development** and technology selection.

Experimental Set-up



Outputs

$$Resistance = \frac{\rho Cu_T l}{\pi r^2}$$

$$r = \sqrt{\frac{\rho Cu_T l}{\pi \, Resistance}}$$

r Copper wire radius (µm)

ρCu_T *Resistivity of Copper at Testing Temp. T (Ωm)*

T Temperature (°C) → *Const. (Imposed/Measured)*

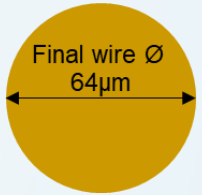
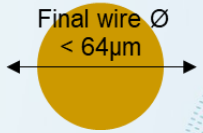
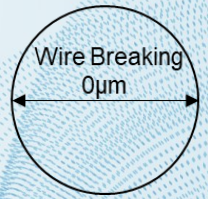
$\rho Cu_{20°C}$ *Resistivity of Copper at 20°C (Ωm)* → *Const.*

α *Coefficient of thermal expansion of Cu* → *Const.*

$$\rho Cu_T = \rho Cu_{20°C} + \alpha(T - 20)\rho Cu_{20°C}$$

l Copper wire length (m) → *Const.*

Practical meaning of the outputs

Ω Constant	Ω Constant Increase	Ω Exponential Increase
Ø Constant	Ø Reduction	Wire braking
		
Corrosivity: No/Low	Corrosivity: Low/Medium	Corrosivity: High
Cu compatibility: ++ High	Cu compatibility: +/- Medium/TBD	Cu compatibility: -- Poor

[2] **New Insight on the Impact of Automatic Transmission Fluid (ATF) Additives on Corrosion of Copper**. *Michel P. Gahagan et al. International Journal of Automotive Engineering 7 (2016) 115-120*

[3] **Wire resistance method for measuring the carrion of copper by lubricating fluids**. *Gregory J. Hunt et al. Lubrication Science 29 (2017) 279-280*

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Corrosivity Assessment Evolution:

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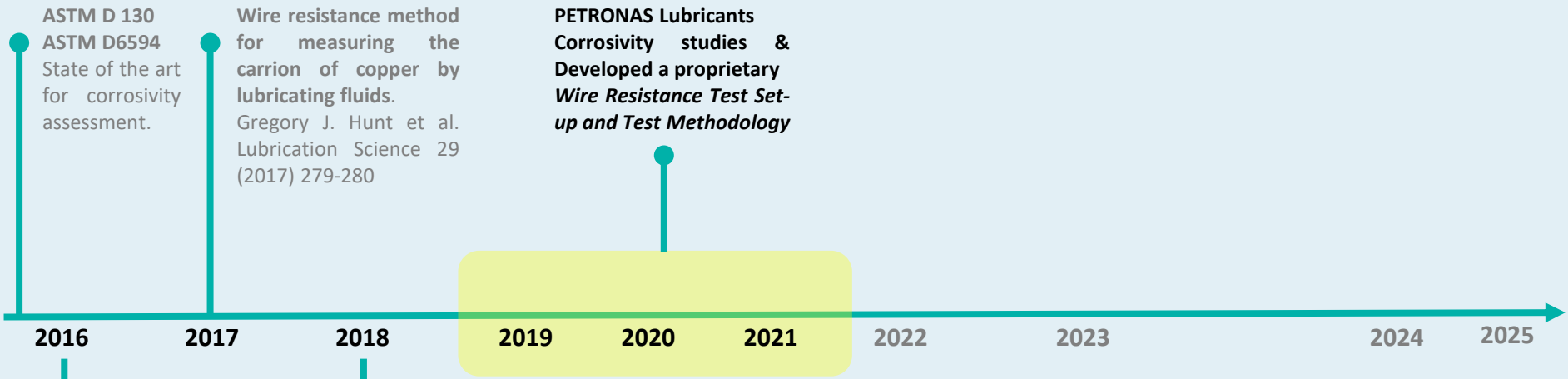
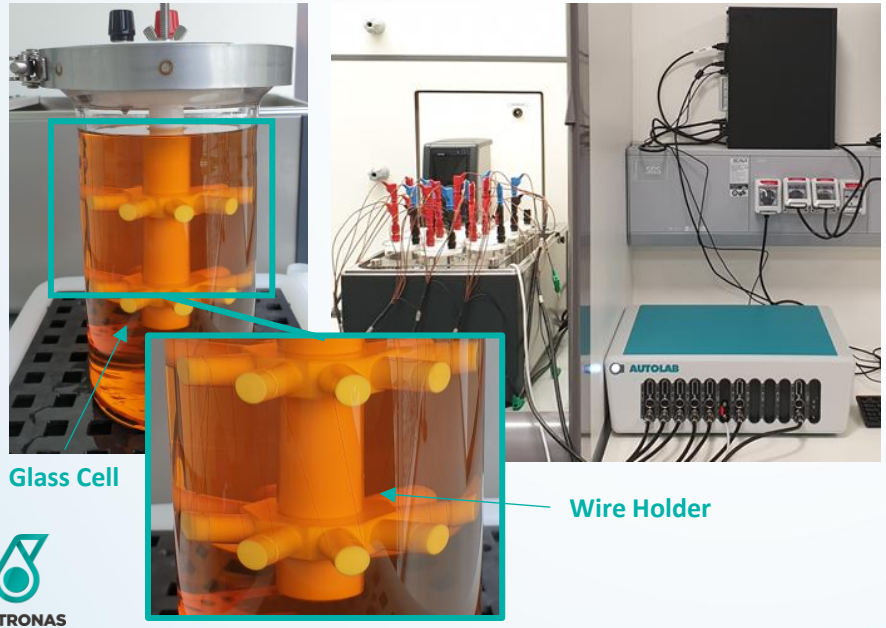


Wire resistance method: PLI Measurement set-up

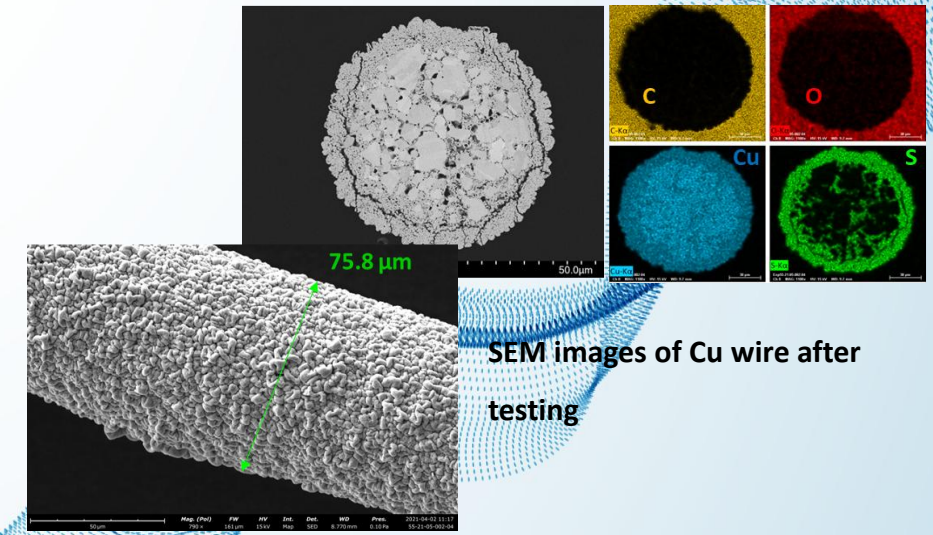
Electrochemical Measurement Set-up

PLI Test Rig has been implemented since 2019, and it meet SAE J 3200 requirements. The testing temperature range is 50°C -160°C and the test duration can vary from 72 hrs up to 1000 hrs. The method is applicable to several metals (e.g. Cu, SS, Al, Au etc.).



Scanning Electron Microscopy (SEM) & Energy Dispersive X-Ray Analysis (EDX), EOT investigation

PLI decided to invest in a Benchtop SEM + EDX (Thermo Fisher Phenom XL) to maximise the outputs of the tests and to achieve a deep understanding of the corrosion phenomena taking place on the copper wire surface.



SEM images of Cu wire after testing

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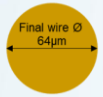
Ω Constant

Ø Constant

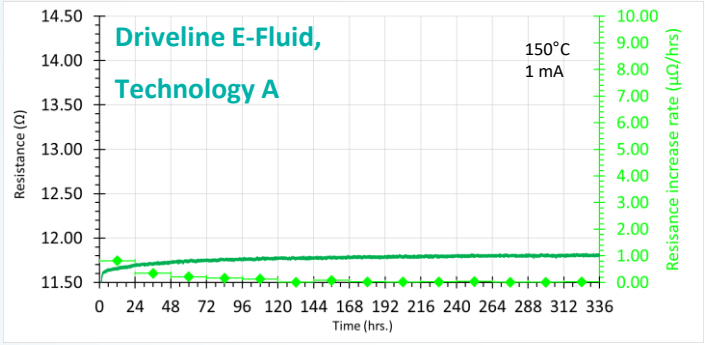


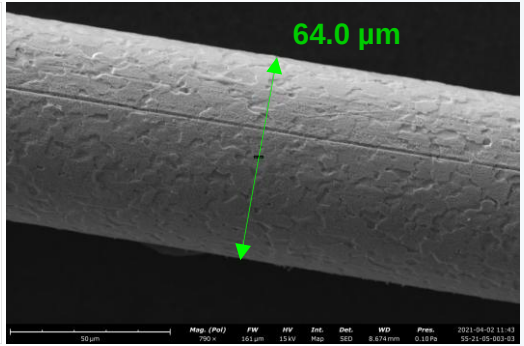
Corrosivity:

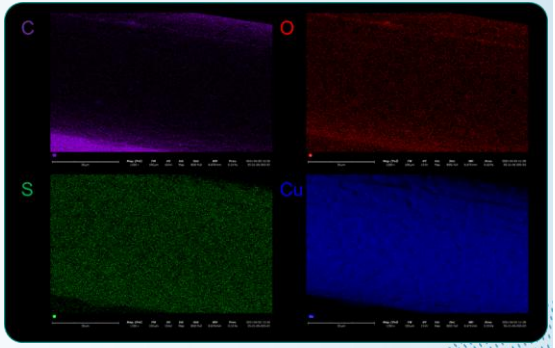
No/Low

Cu compatibility:

++ High







Ω Constant Increase

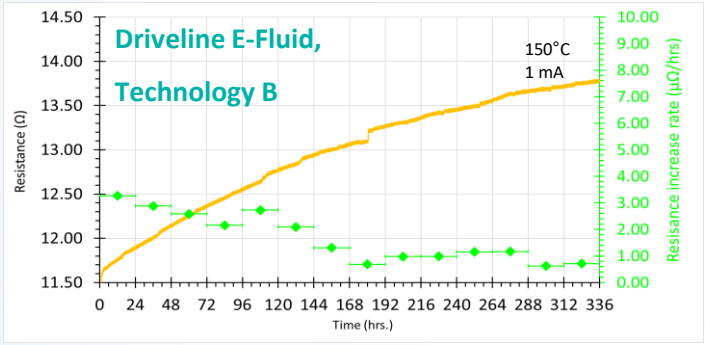
Ø Reduction

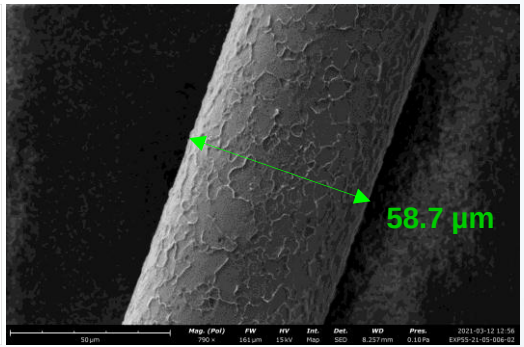
Corrosivity:

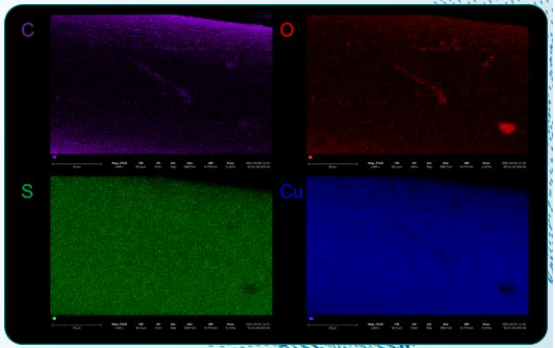
Low/Medium

Cu compatibility:

+/- Medium/TBD







Ω Exponential Increase

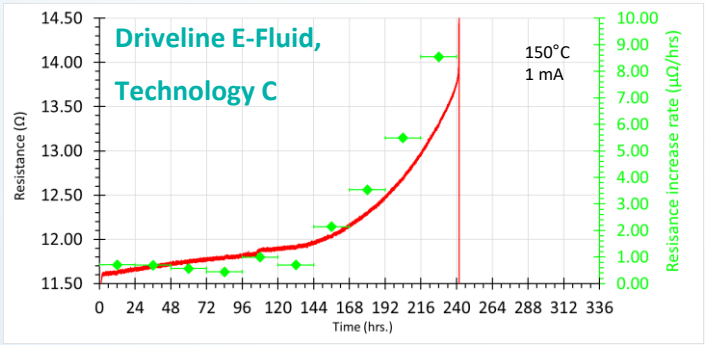
Wire braking

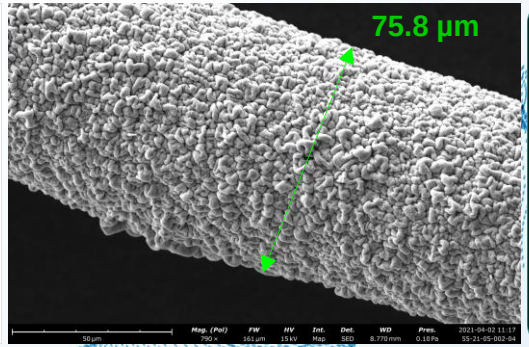
Corrosivity:

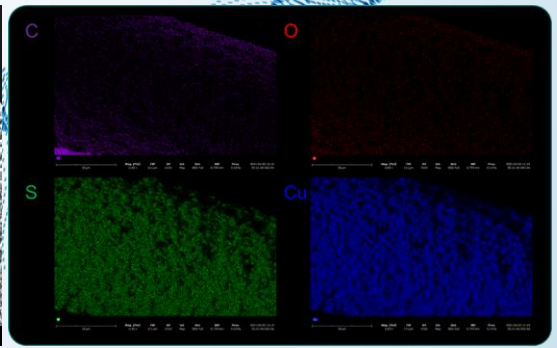
High

Cu compatibility:

-- Poor







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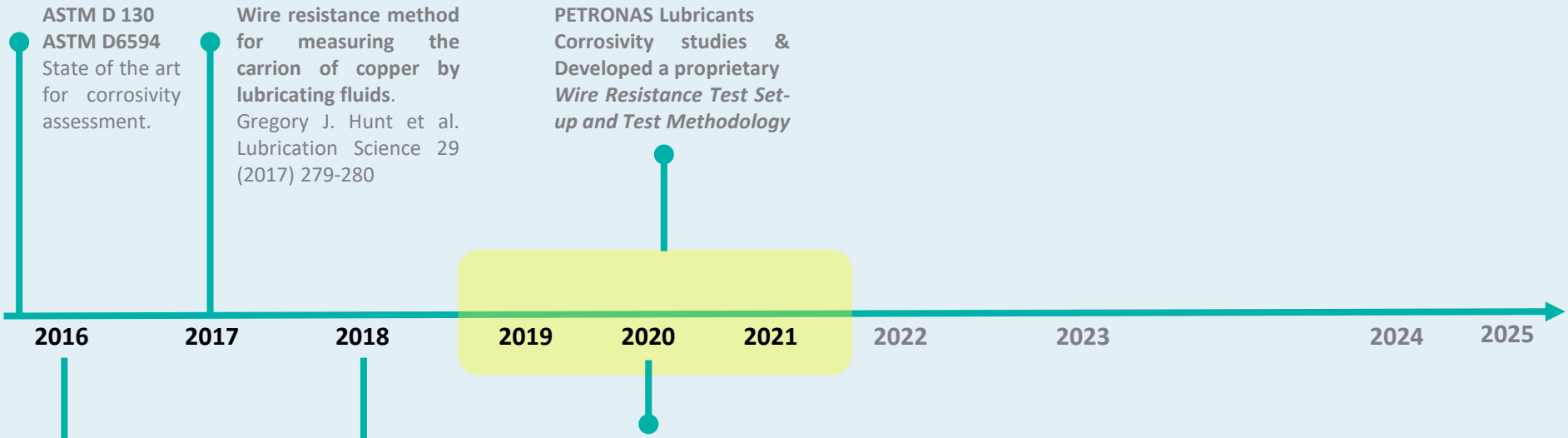
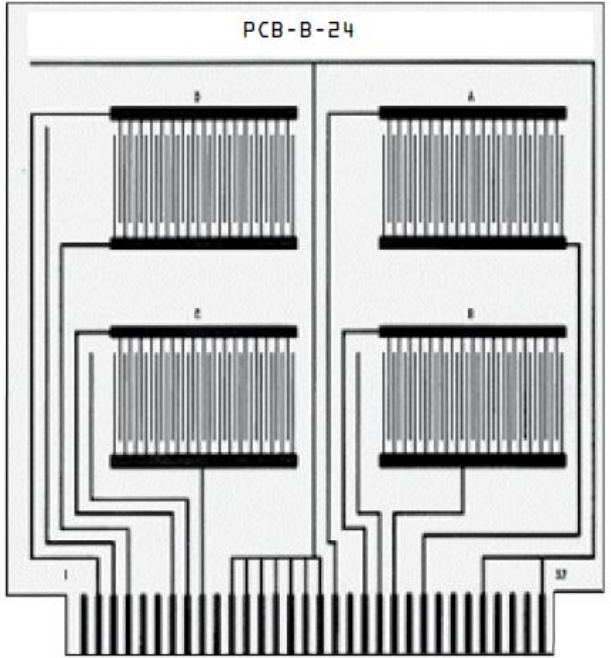


Surface insulation resistance Test (SIR)

PCB-B-24 ICP standard test board

SIR (surface insulation resistance) is an important test in determining the long-term electrical reliability of flux residues in most electronics devices. The primary purpose of SIR testing is to understand the impact of flux residues on the electrical reliability of a device, specifically the conductive and corrosive nature of the residue, regardless of whether the residue is removed or allowed to remain on the board post-reflow. SIR can be a little difficult to understand because it provides a measurement of conductivity of the residue in “ohms per square”. People are often thrown by a “unit-less” value. Ohms per square what - square centimeter, square inch, square meter In fact, it can be whatever unit you would like as long as it is a square measure of area.

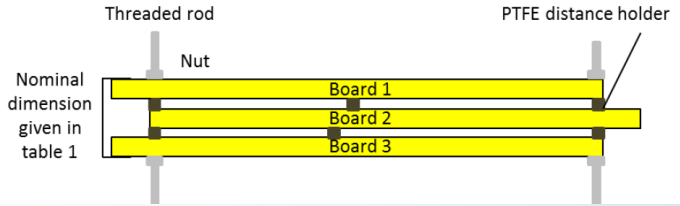


Understanding SIR. Chris Nash, Technical Support Engineer - Eric Bastow, Senior Technical Support Engineer. Indium Corporation. https://www.ipc.org/system/files/technical_resource/E7%26S17_01.pdf

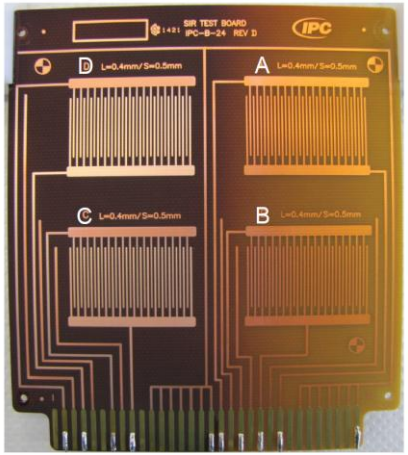
PETRONAS

Surface insulation resistance Test (SIR)

Good Reference Fluid after 1000 hours test at 150°C, 120 µm board gap.



Board 1



No Buildup of Conductive Sediments

Diagram 1: Electrical Resistance of Board 1, Structure A & B (Gas)

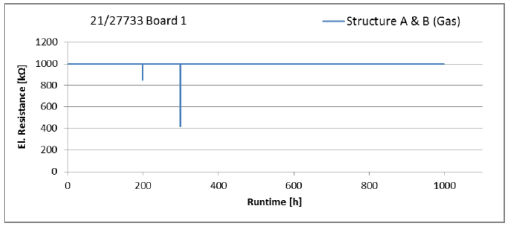


Diagram 2: Electrical Resistance of Board 1, Structure C & D (Oil)

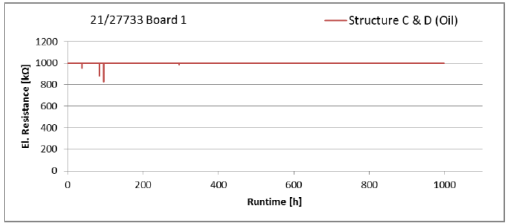
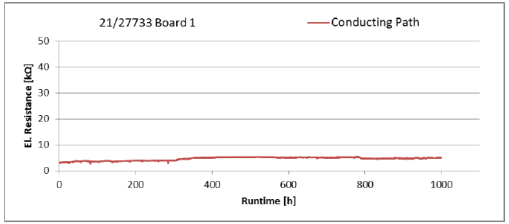
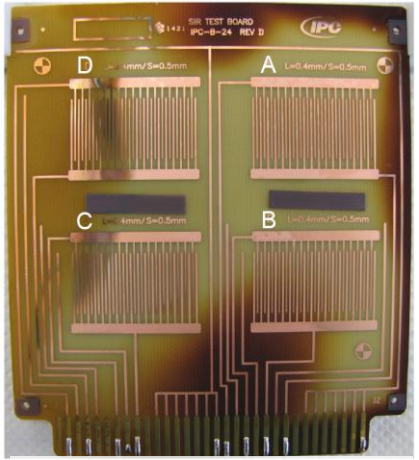


Diagram 3: Electrical Resistance of Board 1, Conducting Path



Board 2



No Dendrites Formation

Diagram 4: Electrical Resistance of Board 2, Structure C & D (Gas)

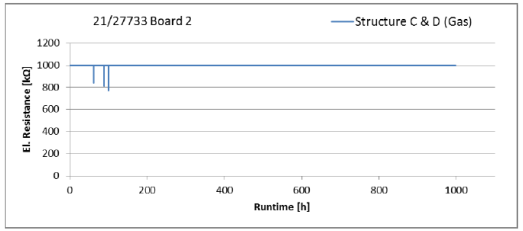


Diagram 5: Electrical Resistance of Board 2, Structure A & B (Oil)

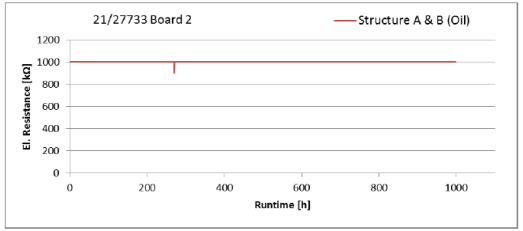
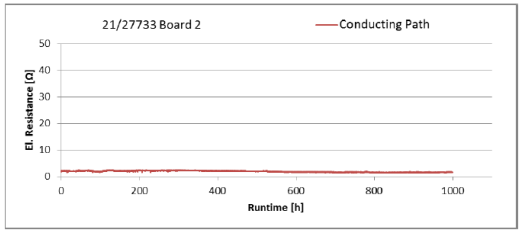


Diagram 6: Electrical Resistance of Board 2, Conducting Path



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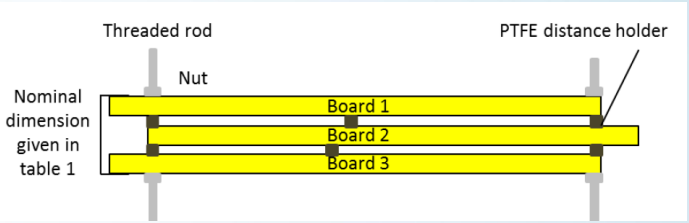
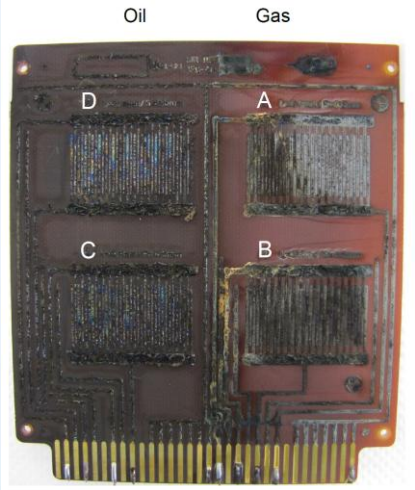
Surface insulation resistance Test (SIR)

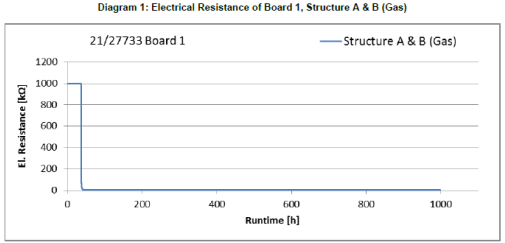
Good Reference Fluid after 1000 hours test at 150°C, 120 µm board gap.

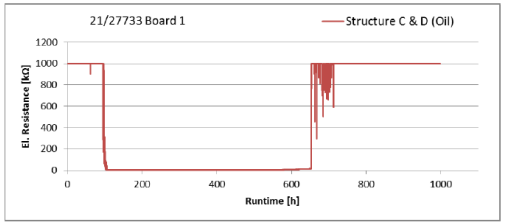


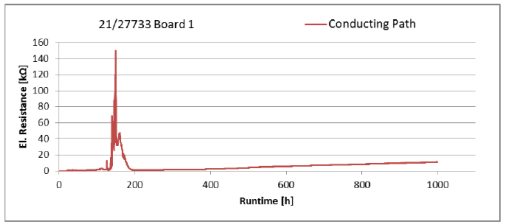
Board 1



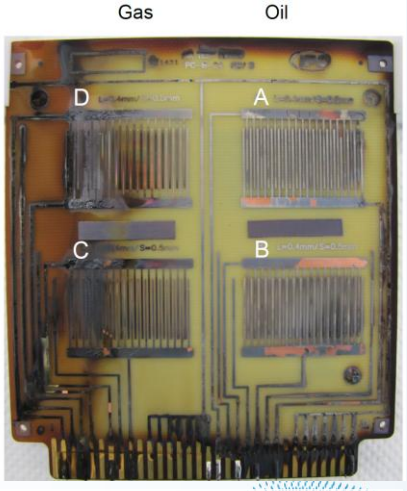
Buildup of Conductive Sediments



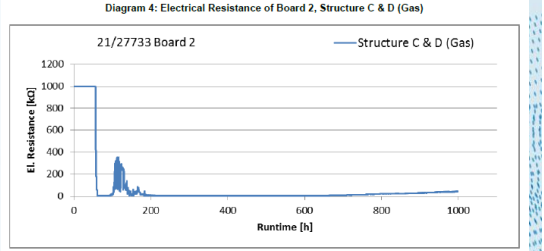


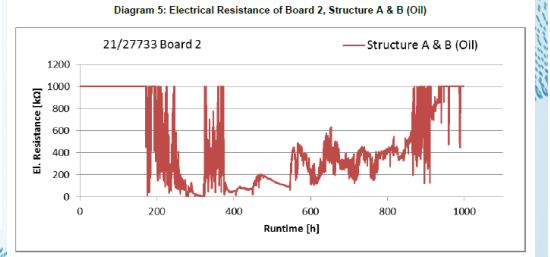


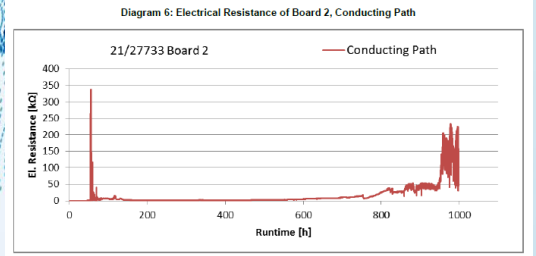
Board 2



Dendrites Formation







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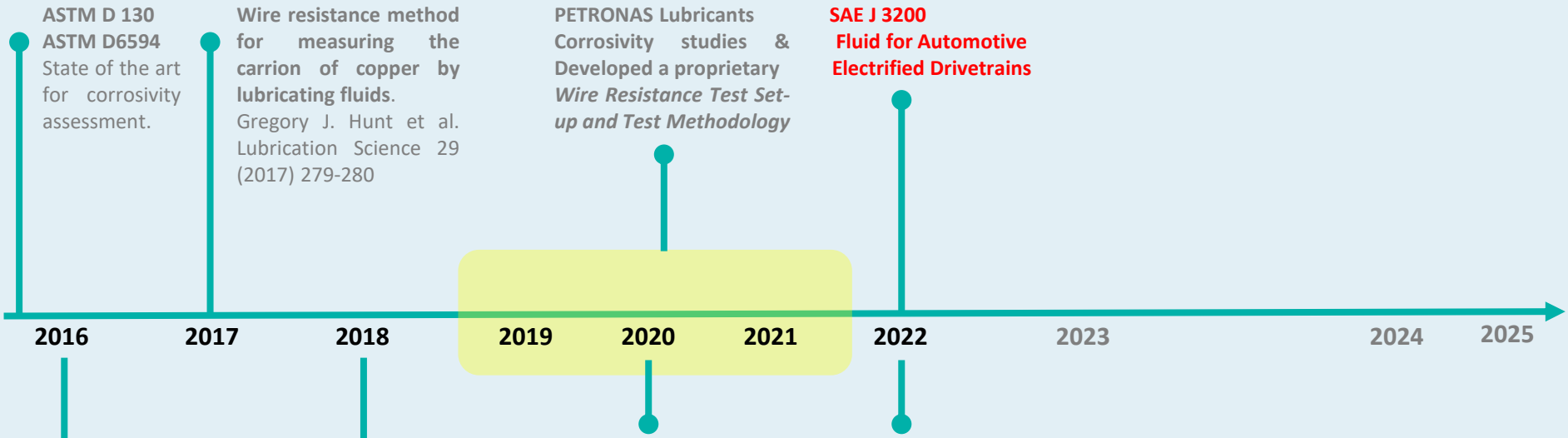
Corrosivity Assessment Evolution:

Methods, Customer Requirements and Standard



SAE J3200

Fluid for Automotive Electrified Drivetrains

Chapter

5.1 Electrical Properties

5.2 Heat Transfer Properties

5.3 Copper Corrosion and Conductive Deposit Properties

5.3.1 Wire Corrosion Test

5.3.2 Conductive Deposit Test

5.3.3 Extended ASTM D 130 Copper Corrosion Test

5.3.4 Vapor Phase Copper Strip Test

5.4 Oxidant Properties

5.6 Material Compatibility Properties

5.7 High-Speed Aeration Properties

5.6 New Wear Concerns

SAE INTERNATIONAL

SURFACE VEHICLE INFORMATION REPORT	J3200™	OCT2022
	Issued	2022-10
Fluid for Automotive Electrified Drivetrains		

COPPER CORROSION AND CONDUCTIVE DEPOSIT PROPERTIES			
Test Method(s)	**Measured Properties**	**Recommended Equipment**	**Units**
Wire Corrosion Test US Army MilSpec / FTM XXXX (TBD) ASTM Subcommittee 9	Rate of copper wire corrosion	Wire corrosion test - WCT Tannas Co. / Lubrizol Patent pending	Radius change in Angstroms
Conductive Deposit Test US Army MilSpec / FTM XXXX (TBD) ASTM Subcommittee 9	Rate of change in resistance of copper traces over time, both in the fluid and vapor states at 150 °C	Conductive ceposit test CDT - Tannas Co. Patent pending	Rate of change in resistance
Extended ASTM D130: Needs addendum or new method ASTM Subcommittee 5	Copper strip corrosion levels measured after exposure at 150 °C	Per standard	ppm copper visual rating
Vapor Phase Copper Test New method ASTM Subcommittee 5	Extension of copper corrosion in response to vapor conditions at 100 °C	Test method lists parts to assemble	Visual rating from chart

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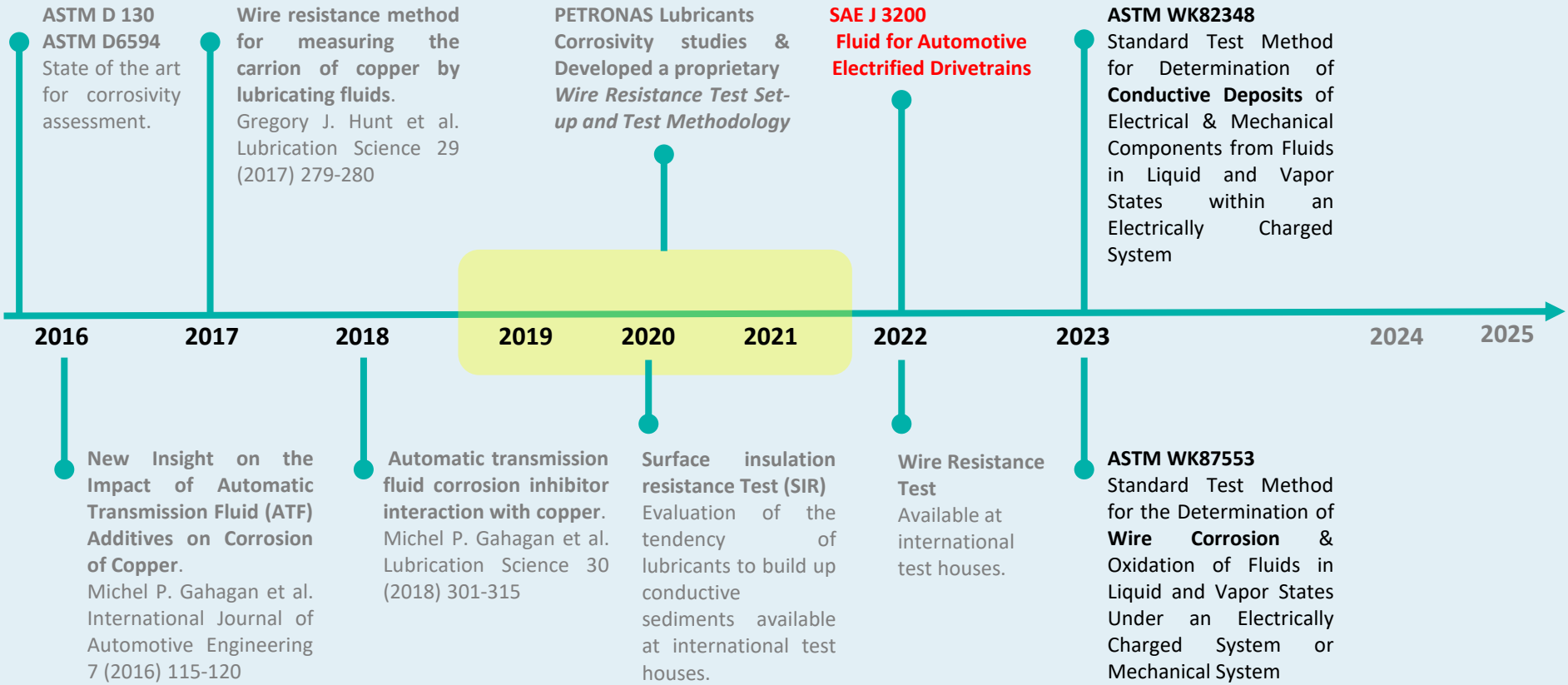
Corrosivity Assessment Evolution:

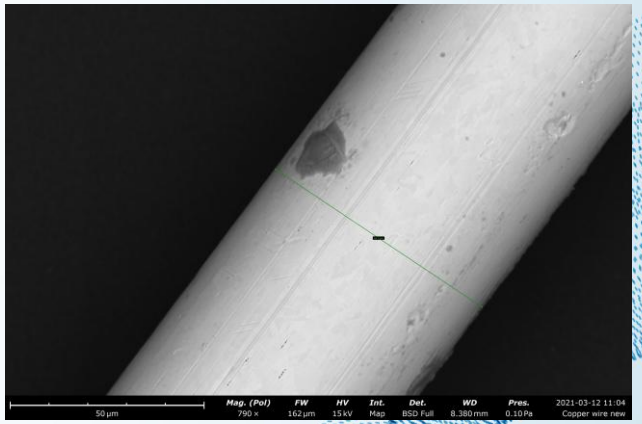
Methods, Customer Requirements and Standard



ASTM WK87553 – ASTM Subcommittee: D02-09

New Test Method for the Determination of Wire Corrosion and Oxidation from Fluids in Liquid and Vapor States Within an Electrically Charged or Mechanical System

SCOPE: This procedure covers the **basic operation of the Wire Corrosion Test**, which is used to monitor the **corrosiveness** of the test fluid by **monitoring changes in resistance in the wires under test**. The change in resistance is proportional to the change in diameter of the wire. The wire may be copper or other material where corrosion is of concern. The general test represents the degree of protection an oil has on the bare copper connective wire, or other conductive material, in automatic transmission gearbox systems or other industry applications. The test equipment has been adapted to collect both solution corrosion data and vapor corrosion data, simulating effects on the copper containing components in a gearbox, engine or other application where the materials is subject to immersion in a fluid and potentially having a vapor state. Testing temperatures within temperatures from ambient to 180 °C for periods of up to 72 hours or more.



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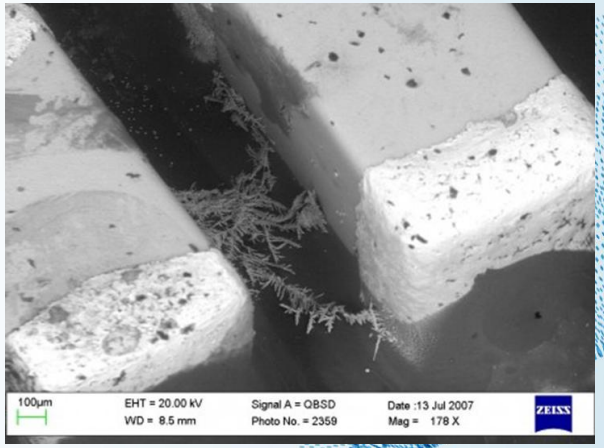
ASTM WK82348 → ASTM D8544 Subcommittee: D02-09

Standard Test Method for Determination of Conductive Deposits of Electrical and Mechanical Components from Fluids in Liquid and Vapor States within an Electrically Charged System

SCOPE: This test method covers the basic operation of the **conductive deposit test**. The apparatus is utilized to monitor the **formation of dendrites and deposits that are conductive** and may form under oxidation conditions of fluids used in electric vehicles and other industries where electronics are involved. The oxidation conditions can vary from 80 °C to 180 °C with very specific circuit boards, under power, and monitoring changes in conductance over time. Both the liquid and vapor areas are monitored for this condition over time. Variables in temperature, voltage, and time can be altered according to the industry need. A reported index based on the rate of deposit formation may be implemented. The typical testing is performed at 150 °C over a period of up to 500 h with a monitored voltage applied.



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Corrosivity Assessment Evolution:

Methods, Customer Requirements and Standard

2016

ASTM D 130
ASTM D6594
State of the art for corrosivity assessment.

2017

Wire resistance method for measuring the carrion of copper by lubricating fluids.
Gregory J. Hunt et al. Lubrication Science 29 (2017) 279-280

2018

New Insight on the Impact of Automatic Transmission Fluid (ATF) Additives on Corrosion of Copper.
Michel P. Gahagan et al. International Journal of Automotive Engineering 7 (2016) 115-120

Automatic transmission fluid corrosion inhibitor interaction with copper.
Michel P. Gahagan et al. Lubrication Science 30 (2018) 301-315

2019

2020

PETRONAS Lubricants Corrosivity studies & Developed a proprietary *Wire Resistance Test Set-up and Test Methodology*

Surface insulation resistance Test (SIR)
Evaluation of the tendency of lubricants to build up conductive sediments available at international test houses.

2021

2022

SAE J 3200
Fluid for Automotive Electrified Drivetrains

Wire Resistance Test
Available at international test houses.

2023

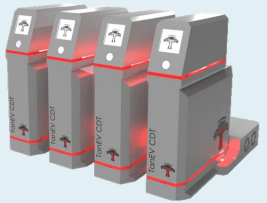
ASTM WK82348
Standard Test Method for Determination of **Conductive Deposits** of Electrical & Mechanical Components from Fluids in Liquid and Vapor States within an Electrically Charged System

ASTM WK87553
New Test Method for the Determination of **Wire Corrosion and Oxidation** from Fluids in Liquid and Vapor States Within an Electrically Charged or Mechanical System

2024

ASTM D8544

Tannas Equipment
Tan EV CDT
Tan EV WCT



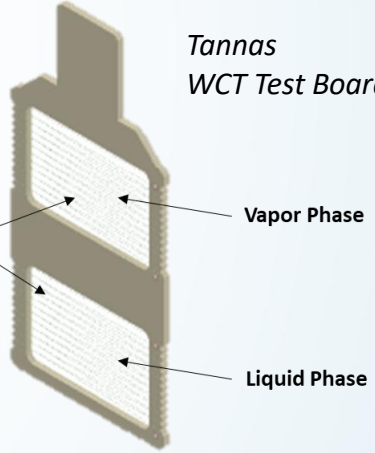
2025

Tannas – Tan EV WCT test Equipment (Round Robin ILS 1869 On-going)

New Test Method for the Determination of Wire Corrosion and Oxidation from Fluids in Liquid and Vapor States Within an Electrically Charged or Mechanical System

Tannas
Tan EV WCT

Tannas
WCT Test Board



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PETRONAS
iona

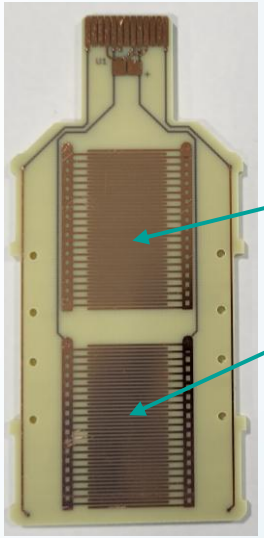
Tannas – Tan EV CDT test Equipment (Round Robin ILS 1857 On-going)

Recommended test equipment for SAE J 3200 and ASTM D8544

PETRONAS Lubricants International

is a member of of ILS 1857 Round Robin

Tannas
Tan EV CDT



Tannas
CDT Test Board

PCB Board
Voltage Loops

Vapor Phase

Liquid Phase

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ASTM D6594
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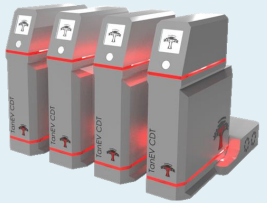
2024

ASTM D8544

Tannas Equipment
Tan EV CDT
Tan EV WCT



2025

Thank You for your attention

Questions?