

Methods for testing biodegradation of lubricants

Umweltverträgliche Schmierstoffe 24.11.2010-26.11.2010

TAE Esslingen

The Coordinating European Council

for the Development of performance tests for fuels, lubricants and other fluids

„CEC is an Industry-based organisation which develops Test Methods for the performance testing of Automotive Engine Oil, Fuels & Transmission Fluids.“

engine tests	rig tests	laboratory tests
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The Coordinating European Council
for the Development of Performance Tests for Fuels, Lubricants and Other Fluids

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Quality Surveillance of CEC

The quality standards of CEC are very high:

- **Steady monitoring of results coming from mandatory tests with Reference Oils**
- **Active Surveillance group for each test method**
 - Discussion and removal of arising problems
 - Arrange and conduct regular Round Robin Tests
- **Every participating laboratory must be certified according to ISO 17025**
- **Online Test Monitoring System since 2009**
- **Several Support Groups to assist Development Groups in difficult questions**

Biodegradation Test Procedure

CEC-L-33-A-93 (unsupported)

Preparation of flasks:

0-day flasks	21-day flasks	poisoned flasks
3 flasks	3 flasks	2 flasks
no loss → 100%	biotic and abiotic loss	abiotic loss

Extraction of flasks:

with Freon or Tetrachloroethylene

→ **Determination
of oil loss**

Analysis of extract:

Infrared spectroscopy

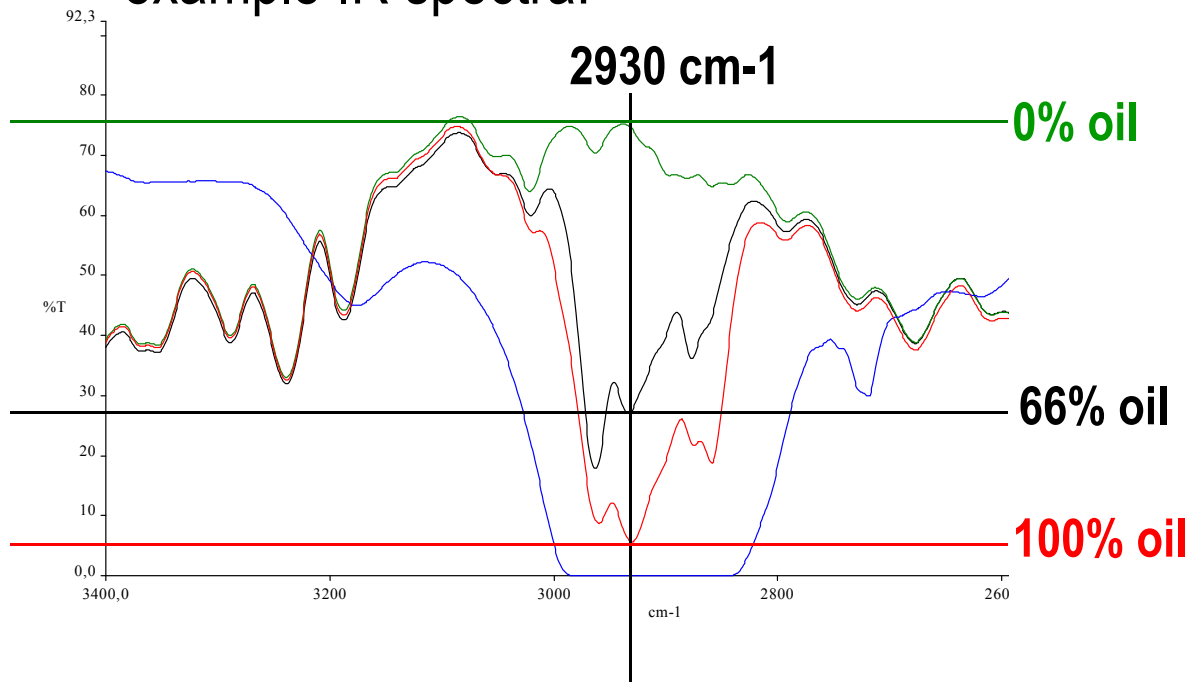
CEC-L-33-A-93

Analysis

■ Extraction solvent must not have C-H-bonds

- only a few solvents can be used such as 1,1,2-Trichlorotrifluoroethane (Freon) or Tetrachloroethene

example IR spectra:



Freon, without oil

21 day flask

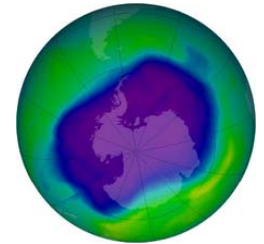
0 day flask

solvent which contain

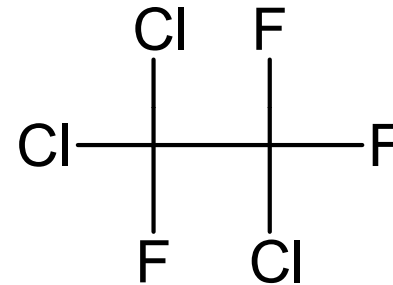
C-H bonds → not applicable

CEC-L-33-A-93

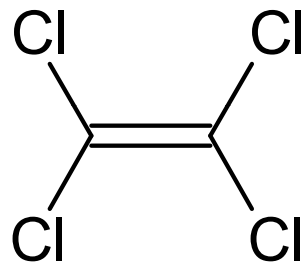
Problems



- Freon destroys ozone in atmosphere (it is a CFC!!)
- laboratory work with Freon is difficult
 - volatile liquid with high density
- Freon is very expensive (750 € / 2,5 L)



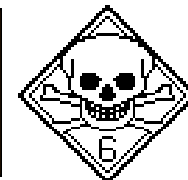
- Tetrachloroethene is toxic



N



Xn



Alternative analysis

CEC-TDG-L-103

Using gas chromatography instead of infrared spectroscopy
like in DIN EN ISO 9377-2 (H 53):

Determination of hydrocarbon oil index (hydrocarbons in water)

infrared spectroscopy - IR	gas chromatography - GC
determination of C-H bonds because of absorbance of infrared light → only a few solvents applicable	determination of C-H bonds because of combustion after separation in components → many solvents applicable

Alternative analysis

CEC-TDG-L-103

- In a mini round robin test CEC-TDG-L103 showed good correlation between GC and IR analysis
- In this test the results obtained from GC analysis were even more precise than the results obtained from IR analysis

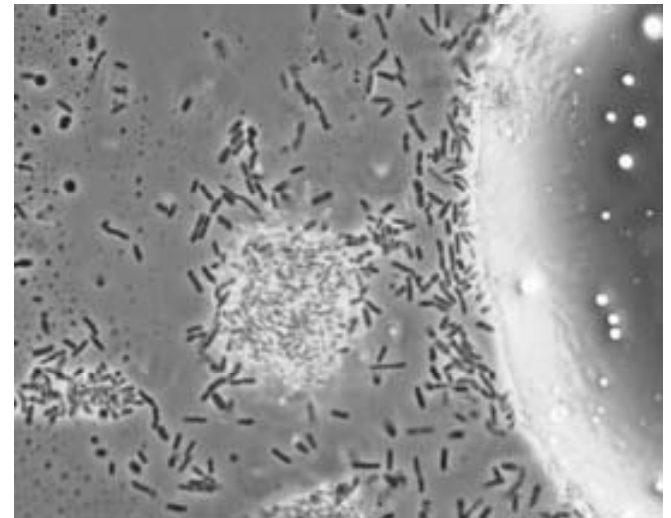
	IR	GC
R (RL110)	6,2	3,5
R (TMP-Ester)	8,4	5,1

Second Problem: Sample introduction

CEC-TDG-L-103

For a fast biodegradation the sample must be fine dispersed in the water phase. Bacteria cannot enter in oil phase, only oil/water interface can be used as food.

In CEC-L-33-A-93 mother solution solvent was Freon. Due to it's high density it kept the test oil on the bottom flask and led to a fine dispersion of oil in water.



Second Problem: Sample introduction

CEC-TDG-L-103

How can oil be dispersed in water without Freon?

possible solutions	possible problems
using a solid carrier, e.g. glass fibres or sand	extraction of oil residues from carrier not in all cases possible
using polar solvents for mother solution, e.g. esters	oil sample solubility ?
using detergents or emulsifiers	extraction step difficult → phase separation!
ultrasonic sound	only short-term efficiency

Results of method development CEC-TDG-L-103

Results of method development CEC-TDG-L-103:

Sample preparation in polar solvent → homogenous dispersion

Reduction of sample size → less chemicals necessary

Extraction with a hydrocarbon → not dangerous for the environment

GC-analysis → high precision



unpolar hydrocarbon



polar ether

CEC-TDG-L-103

time schedule

- **Conclusion of phase 1** **End of 2010**
- **Approval of phase 1** **Q1 2011**
- **Round Robin Test (phase 2)** **Q2 2011**
- **Conclusion of phase 2** **Q3 2011**
- **Approval of phase 2** **Q4 2011**