

1.0 Title:

Total Oil Content in aqueous metalworking fluids

2.0 Control Number:

3-WET-070-A

3.0 Authored By:

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4.0 Validated By:

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5.0 Effective Date:

November 1, 2004

6.0 Date of Previous Revision:

None

7.0 Standard Methods Referenced:

None

8.0 Applicable matrices:

Soluble and Semi-synthetic oil in aqueous medium

9.0 Detection Limits and Procedures:

1-100%

10.0 Scope and Application:

This test method covers the determination of total oil (tramp plus Metalworking fluid oil) in aqueous based fluids.

11.0 Summary of the Method:

The aqueous sample is homogenized and an aliquot obtained in a centrifuge tube. Sulfuric acid is added to split the oil. The treated sample is kept at 60 deg. C for 24 hours and subsequently centrifuged. The top layer volume is read and divided by the charge volume to compute total oil.

12.0 Definitions:

None

13.0 Interferences:

This procedure is applicable for soluble oils, semi-synthetics and tramp only

14.0 Safety:

Refer to the Chemical Hygiene Plan located in the QA office and specific MSDS for information regarding handling of reagents utilized in this procedure.

15.0 Equipment and Supplies:

50ml graduated centrifuge tube
Natural convection oven
Centrifuge with 50ml head

16.0 Maintenance and Preventative Maintenance:

None

17.0 Reagents and Standards:

ACS grade Concentrated sulfuric acid

18.0 Sample Collection, Preservation, Shipment, and Storage:

Not applicable

19.0 Quality Control:

Analysis on one or more samples in the batch should be performed in at least duplicate. The RSD value for the multiple sample analysis must be 10% or less to validate the batch.

A laboratory control sample of known oil content in water should be analyzed at a 5% sample frequency. The value of the LCS sample should not exceed 20% of nominal value.

20.0 Calibration and Standardization:

Not applicable

21.0 Procedure:

Homogenize the sample by rapid shaking for 30 seconds.

Immediately transfer 45ml to the centrifuge tube.

Add 5ml concentrated sulfuric acid to the centrifuge tube (caution: exothermic)

Cap the centrifuge tube and invert several times to disperse the sulfuric acid.

Place centrifuge tube upright in oven set at 60 deg. C for a period of 24 hours.

Remove centrifuge tube from oven and inspect. If more 5mls or more split layer (top layer) is present or an emulsion exists above the split, the sample will need to be reprepared or processed with a smaller charge volume. If less than 5ml split layer is present, centrifuge the sample for 5minutes at 2000 RPM.

Remove centrifuge tube from centrifuge and record split layer volume

22.0 Calculations:

Total Oil Content (% v/v) = [Split layer volume (ml) / charge volume (ml)] X 100

23.0 Method Performance:

This method is capable of measuring total oil at 1% to 100% with resolution of 1%

24.0 Pollution Prevention:

Not applicable

25.0 Data Assessment and Acceptance Criteria for Quality Control Measures:

Duplicate readings should not exceed RSD of 10%
LCS readings should be between 80 and 120%

26.0 Corrective Actions for Out of Control Data:

Repeat the testing on the affected samples.

27.0 Contingencies for Handling Out of Control or Unacceptable Data:

Samples for which duplicate values can not obtain RSD 10% will be qualified as such on the final report

28.0 Waste Management:

Dispose of oil waste in the non-hazardous oil waste drum

29.0 Revision History:

November 1, 2004 – Draft document

30.0 Modifications to Cited Methods:

None

31.0 References:

None

32.0 Any Tables, Diagrams, Flowcharts and Validation Data:

None.

33.0 Demonstration of Capability:

Not applicable.