National Institute of Standards & Technology

Report of Investigation

Reference Material 8509

Moisture in Methanol

Reference Material (RM) 8509 is intended primarily for use in the calibration of instruments and the evaluation of methods used for moisture determination. A unit of RM 8509 consists of five ampoules, each containing approximately 5 mL of methanol solution with a trace concentration level of water.

Analytical Results

The reference value is based on the results of an interlaboratory comparison exercise conducted among 13 laboratories and NIST using the coulometric Karl Fischer Method. In addition, NIST measurements using the volumetric Karl Fischer Method were performed to provide an independent assessment of both the moisture content as well as the homogeneity of the RM lot. It is important to note that this material is **not** a Standard Reference Material* and the reference value is method dependent.

Reference Mass Concentration Value of Moisture: 93 mg/kg ± 13 mg/kg*

*The reference value and uncertainty describe an interval which is at least a 95 % confidence interval for the moisture concentration in each ampoule of RM 8509. The uncertainty includes the uncertainty due to the interlaboratory comparison exercise measurements as well as a trend observed within the ampoule fill sequence.

Expiration of Reference Value: The reference value of RM 8509 is valid, within the measurement uncertainty specified, until December 31, 2027, provided the RM is handled in accordance with instructions given in this Report of Investigation (see Instructions for Use). This reference value is nullified if the RM is damaged, contaminated, or modified.

The coordination of the technical measurements was under the direction of S.A. Margolis and S.A. Wise of the NIST Analytical Chemistry Division.

Preparation and analytical measurements at NIST were performed by S.A. Margolis, J.C. Williams, and L.C. Sander of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller and L.M. Gill of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, analysis, and issuance of this RM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: June 12, 1997*
8/21/95 (original certificate date)
*This revision reports an expiration date and editorially changed "recommended" to "reference" value

Thomas E. Gills, Chief
Standard Reference Materials Program

INSTRUCTIONS FOR USE

Handling: The ampoules are sealed under dry argon. This material is hygroscopic; therefore, after opening the ampoules, extreme caution should be exercised to avoid contamination with moisture (e.g., dry measuring devices and containers must be used).

Storage and Use: Ampoules should be stored at room temperature. For the reference value to be valid within the stated uncertainty, samples of the material for analysis should be withdrawn from the ampoules immediately after opening, transferred into oven-dried measuring devices, and used without delay. The reference value is not applicable to ampoules that have been stored after opening, even if the ampoules are resealed.

PREPARATION AND ANALYSIS

Preparation of RM 8509: Methanol, as supplied from the manufacturer, typically contains trace levels of water. The nominal moisture level was determined in several lots of HPLC-grade methanol and the appropriate lot was selected for ampouling. Therefore, the reference moisture value reflects the content after processing at NIST and not necessarily the moisture content when obtained from the manufacturer. Approximately 12 L (three bottles) of the selected lot were poured into a 18.9 L (5-gal) bottle containing dry argon, and the solution stirred for 30 min. The argon headspace above the liquid was maintained with argon from an attached Teflon®[1] bag containing dry argon. As the level of the solution decreased in the bottle over the course of the ampouling, argon replaced the fluid. In this way a sealed system was maintained to minimize moisture contamination from the air. Aliquots of 5 mL were dispensed automatically into argon-filled, dried ampoules and then sealed.

Analytical Methods: An interlaboratory comparison exercise among 13 laboratories and NIST was conducted to determine the moisture content of RM 8509. All analyses in the interlaboratory comparison exercise were performed using coulometric Karl Fischer instrumentation and reagents routinely used in the participating laboratories. Each cooperating laboratory analyzed a single sample from each of two ampoules of RM 8509. The participating laboratories are listed below.

Homogeneity Study: Samples from 40 ampoules, selected throughout the fill sequence, were measured at NIST in a random order over four days using the volumetric Karl Fischer method. A statistically significant increase in moisture content with fill sequence was detected in the homogeneity study. A 9 mg/kg change in concentration was predicted by the homogeneity study over the entire population of ampoules of RM 8509 and was included in the total uncertainty of the reference value. The results from the volumetric Karl Fisher method were also used as an independent method to confirm the round robin results obtained by the use of the coulometric Karl Fischer method.

Participants in the RM 8509 Interlaboratory Comparison Exercise:

J., Angelo, Exxon Research & Engineering Co., Baytown, TX
R., Cherrillo, Shell Development Co., Houston, TX
J., Greer, United Power Services Inc., Nashville, TN
C., Smith, Cosa Instrument Co., Norwood, NJ
L., Shick, Photovolt, Indianapolis, IN
G., Gauger, McGraw-Edison Power System, Franksville, WI
P., Griffin, Doble Engineering Co., Watertown, MA
R., Deering, Bonneville Power Administration, Vancouver, WA
D., Meyers, S.D. Meyers Inc., Tallmadge, OH
G., Thomas, Big River Electric Corp., Henderson, KY
A., Kaupa, Baltimore Gas & Electric, Baltimore, MD
V., Peterson, Minnesota Power Co., Duluth, MN
D., Graziano, CSC Scientific, Fairfax, VA

[1] Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.

ASTM RESEARCH REPORT

File No. RR:D32-1016 Date: 6/24/85

Title: Supporting Data for D3907, Method of Testing Fluid Cracking Catalysts by Microactivity Test

|___| Designation No.:..... |___| Draft Document:.....

Author: D.R. Peterson

Sponsoring Subcommittee: D32

Submitted by:.....

THE RESEARCH REPORTS FILE

Research Reports are filed in and available from the Information Center at ASTM Headquarters. The Research Report file provides a place for orderly storage and retrieval of data derived from the extensive research carried out in, or sponsored by, the technical committees.

A Research Report Format Guide for developing a research report is available from ASTM Headquarters.

Research Reports are transmitted to Headquarters by the sponsoring committee.

ASTM
1916 Race Street
Philadelphia, PA 19103

D3907: Activity of Fluid Cracking Catalysts by Microactivity Test

Round Robin Data

Lab No.	RR-1	RR-2	RR-3	RR-4	RR-5	RR-6
1	58.4 59.1	65.8	76.1 76.2 78.3	77.6	80.6 81.2	71.7 71.8
2	60.1 60.1	64.4	70.1 71.0 71.7	75.3	77.2 81.8	
3	61.0 60.2	69.5	77.5 79.8 84.6	73.8	77.9 78.1	
4	57.9 60.4	63.1	80.4 81.0 81.4	80.1	84.3 84.7	
5	58.6 59.1	64.5	75.4 75.8 76.1	76.6	81.6 81.8	72.2 72.5
6	58.5 61.5	66.7	74.7 76.6 77.8	76.1	83.5 85.1	
7	61.0 61.8	67.0	73.4 76.8 77.2	78.8	80.6 82.3	68.6 68.3 66.9 70.8
8	59.1 59.3	64.5	74.4 74.7 75.9	76.0	77.6 78.5	71.4 71.2 71.4
9	51.9 55.5	59.0	72.5 73.5 74.8	71.1	77.0 79.9	66.8 66.2 65.1

Participating Laboratories

Akzo
Arco
Elf
Filtrol
Amoco Oil Co.
Catoleum
Engelhard
Pullman Kellogg
Shell

Reduction of inter-laboratory test data to give consensus values of a measured property for multiple test materials.

Test method: Fluid cracking catalysts by microactivity test: D 3907

Property tested: Conversion of gas oil [wt. %]

Material: RR-1

Trial inter-lab variance component: 3.629

Inter	V2	V4	Mean	d(V2)
1	3.629	4.237	59.346	-1.208
2	2.421	2.964	59.409	0.217
3	2.638	3.196	59.396	0.013
4	2.652	3.210	59.395	0.000
5	2.652	3.210	59.395	0.000

Final inter-lab variance component: 2.65229

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	2	58.750	0.245	0.123	2.775	0.129
2	2	60.100	0.000	0.000	2.652	0.135
3	2	60.600	0.320	0.160	2.812	0.127
4	2	59.150	3.125	1.563	4.215	0.085
5	2	58.850	0.125	0.063	2.715	0.131
6	2	60.000	4.500	2.250	4.902	0.073
7	2	61.400	0.320	0.160	2.812	0.127
8	2	59.200	0.020	0.010	2.662	0.134
9	2	53.700	6.480	3.240	5.892	0.061

Final intra-lab variance component: 0.841

Test laboratories: 9 Degrees of freedom: 9

Consensus mean value	59.39 wt%
Consensus S (intra)	0.92 wt%
Consensus S (inter)	1.63 wt%
Consensus S (total)	1.87 wt%

95% Repeatability limit	2.54 wt%	4.3% of consensus value
95% Reproducibility limit	5.18 wt%	8.7% of consensus value

Material: RR-2

Trial inter-lab variance component: 6.396

Iter	V2	V4	Mean	d(V2)
1	6.396	7.396	64.944	0.982
2	7.378	8.378	64.944	0.148
3	7.525	8.525	64.944	0.003
4	7.528	8.528	64.944	0.000
5	7.528	8.528	64.944	0.000

Final inter-lab variance component: 7.528

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	2	65.800	2.000	1.000	8.528	0.111
2	2	64.400	2.000	1.000	8.528	0.111
3	2	69.500	2.000	1.000	8.528	0.111
4	2	63.100	2.000	1.000	8.528	0.111
5	2	64.500	2.000	1.000	8.528	0.111
6	2	66.700	2.000	1.000	8.528	0.111
7	2	67.000	2.000	1.000	8.528	0.111
8	2	64.500	2.000	1.000	8.528	0.111
9	2	59.000	2.000	1.000	8.528	0.111

Final intra-lab variance component: 1.000

Test laboratories: 9 Degrees of freedom: 9

Consensus mean value	64.94 wt%
Consensus S (intra)	1.00 wt%
Consensus S (inter)	2.74 wt%
Consensus S (total)	2.92 wt%

95% Repeatability limit	2.77 wt% 4.3% of consensus value
95% Reproducibility limit	8.09 wt% 12.5% of consensus value

Material: RR-3

Trial inter-lab variance component: 7.389

Inter	V2	V4	Mean	d(V2)
1	7.389	8.140	76.045	1.243
2	8.632	9.399	76.061	0.224
3	8.856	9.626	76.064	0.006
4	8.862	9.631	76.064	0.000
5	8.862	9.631	76.064	0.000

Final inter-lab variance component: 8.86196

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	3	76.867	1.543	0.514	9.376	0.114
2	3	70.933	0.643	0.214	9.076	0.118
3	3	80.633	13.123	4.374	13.236	0.081
4	3	80.933	0.253	0.084	8.946	0.120
5	3	75.767	0.123	0.041	8.903	0.120
6	3	76.367	2.443	0.814	9.676	0.111
7	3	75.800	4.360	1.453	10.315	0.104
8	3	75.000	0.630	0.210	9.072	0.118
9	3	73.600	1.330	0.443	9.305	0.115

Final intra-lab variance component: 0.906

Test laboratories: 9 Degrees of freedom: 18

Consensus mean value	76.06 wt%
Consensus S (intra)	0.95 wt%
Consensus S (inter)	2.98 wt%
Consensus S (total)	3.13 wt%

95% Repeatability limit	2.64 wt%	3.5% of consensus value
95% Reproducibility limit	8.66 wt%	11.4% of consensus value

Material: RR-4

Trial inter-lab variance component: 5.316

Iter	V2	V4	Mean	d(V2)
1	5.316	6.316	76.156	0.688
2	6.004	7.004	76.156	0.083
3	6.087	7.087	76.156	0.001
4	6.088	7.088	76.156	0.000
5	6.088	7.088	76.156	0.000

Final inter-lab variance component: 6.088

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	2	77.60	2.000	1.000	7.088	0.111
2	2	75.30	2.000	1.000	7.088	0.111
3	2	73.80	2.000	1.000	7.088	0.111
4	2	80.10	2.000	1.000	7.088	0.111
5	2	76.60	2.000	1.000	7.088	0.111
6	2	76.10	2.000	1.000	7.088	0.111
7	2	78.80	2.000	1.000	7.088	0.111
8	2	76.00	2.000	1.000	7.088	0.111
9	2	71.10	2.000	1.000	7.088	0.111

Final intra-lab variance component: 1.000

Test laboratories: 9 Degrees of freedom: 9

Consensus mean value	76.16 wt%
Consensus S (intra)	1.00 wt%
Consensus S (inter)	2.47 wt%
Consensus S (total)	2.66 wt%

95% Repeatability limit	2.77 wt%	3.6% of consensus value
95% Reproducibility limit	7.38 wt%	9.7% of consensus value

Material: RR-5

Trial inter-lab variance component: 4.667

Iter	V2	V4	Mean	d(V2)
1	4.667	5.364	80.883	0.875
2	5.542	6.269	80.871	0.180
3	5.722	6.454	80.869	0.006
4	5.727	6.460	80.869	0.000
5	5.727	6.460	80.869	0.000

Final inter-lab variance component: 5.72725

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	2	80.900	0.180	0.090	5.817	0.123
2	2	79.500	10.580	5.290	11.017	0.065
3	2	78.000	0.020	0.010	5.737	0.125
4	2	84.500	0.080	0.040	5.767	0.125
5	2	81.700	0.020	0.010	5.737	0.125
6	2	84.300	1.280	0.640	6.367	0.113
7	2	81.450	1.445	0.723	6.45	0.111
8	2	78.050	0.405	0.203	5.93	0.121
9	2	78.450	4.205	2.103	7.83	0.092

Final intra-lab variance component: 1.012

Test laboratories: 9 Degrees of freedom: 9

Consensus mean value	80.87 wt%
Consensus S (intra)	1.01 wt%
Consensus S (inter)	2.39 wt%
Consensus S (total)	2.60 wt%

95% Repeatability limit	2.79 wt%	3.4% of consensus value
95% Reproducibility limit	7.20 wt%	8.9% of consensus value

Material: RR-6

Trial inter-lab variance component: 5.23

Iter	V2	V4	Mean	d(V2)
1	5.234	5.409	70.09	1.209
2	6.443	6.620	70.078	0.329
3	6.772	6.949	70.075	0.017
4	6.79	6.967	70.075	0.000
5	6.79	6.967	70.075	0.000

Final inter-lab variance component: 6.79

Lab number	Data count	Data mean	Data variance	Intra-lab variance	Total variance	Weight fraction
1	2	71.750	0.005	0.003	6.792	0.205
5	2	72.350	0.045	0.023	6.812	0.206
7	4	68.650	2.603	0.651	7.440	0.187
8	3	71.333	0.013	0.004	6.794	0.205
9	3	66.033	0.743	0.248	7.037	0.198

Final intra-lab variance component: 0.186

Test laboratories: 5 Degrees of freedom: 9

Consensus mean value	70.07 wt%
Consensus S (intra)	0.43 wt%
Consensus S (inter)	2.61 wt%
Consensus S (total)	2.64 wt%

95% Repeatability limit	1.19 wt%	1.7% of consensus value
95% Reproducibility limit	7.32 wt%	10.4% of consensus value

RM 8590

High Sulfur Gas Oil Feed

(Amoco Oil No. FCC 893)

FEED CHARACTERIZATION
OF D-32 ASTM STANDARD MAT FEED

Distillation Data

Vol	ASTM D-1160 Temp, °C (°F)	760 mm TBP Temp, °C (°F)
IBP	81.7 (179)	197.8 (388)
5	141.7 (287)	262.2 (504)
10	165.6 (330)	301.1 (574)
20	190.0 (374)	333.3 (632)
30	211.1 (412)	361.1 (682)
40	228.9 (444)	385.6 (726)
50	248.3 (479)	411.7 (773)
60	270.0 (518)	438.3 (821)
70	292.2 (558)	465.6 (870)
80	317.2 (603)	496.1 (925)
90	347.2 (657)	532.8 (991)
95	367.8 (694)	551.7 (1025)
FBP	378.9 (714)	571.7 (1061)

Pour Point, °C	26.7 °C (80°F)
Gravity, °API	27.6
Vol ABP, °C	414.4 °C (778°F)
Total Nitrogen, ppm	875
Basic Nitrogen, ppm	281
Sulfur, Wt%	0.64
Conradson Carbon, Wt%	0.18
Ramsbottom Carbon, Wt%	0.21
Refractive Index	1.4772 at 67 °C
Aniline Point, °C	83.3°C(182°F)
Viscosity, CS +	4.42 at 98.9°C(210 °F)
UOPK	12.02