



National Institute of Standards & Technology

Report of Investigation

Reference Materials 8589 and 8590

Fluid Cracking Catalysts (8589) and High Sulfur Gas Oil Feed (8590)

Prepared by the American Society for Testing and Materials

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(This is not a Certified Reference Material)

The Reference Material (RM) 8589 consists of 6 units, 50 grams each, of equilibrium FCC Catalysts RR1-RR6 and RM 8590 consists of 946 mL (1 quart) of the High Sulfur Gas Oil Feed (Amoco Oil No. FCC 893). These are intended for use in determining the activity of FCC Catalysts by Microactivity Test D3907-87 [1]. They are meant for use primarily by petroleum refining industries and catalysts suppliers for this industry.

RMs 8589-8590 are being distributed by NIST through a cooperative program between the National Institute of Standards and Technology and ASTM Committee D32 on Catalysts (Subcommittee D32.04 on Catalytic Properties). The cooperating laboratories used ASTM Standard Test Method D 3907 [1].

Fluid Cracking Catalysts by Microactivity Test

Test method D 3907 covers the determination of the activity of equilibrium or laboratory-deactivated fluid cracking catalyst or both. This is evaluated on the basis of weight percent conversion of gas oil in a microactivity unit. An interlaboratory study was conducted in which the conversion of gas oil was measured in 6 separate test materials in 9 separate laboratories. Not all materials were measured by all laboratories. ASTM Standard Practice E 691 [2], modified for non-uniform test sets, was followed for the data reduction. The results of the round robin tests (9 participants) are:

Reference Material 8589

<u>Sample No.</u>	<u>Conversion of Gas Oil (consensus, wt %)</u>	<u>95% Repeat. Limit (within lab. S)</u>	<u>95% Reprod. Limit (between lab. S)</u>
RR-1	59.39	2.54 (4.3)	5.18 (8.7)
RR-2	64.94	2.77 (4.3)	8.09 (12.5)
RR-3	70.07	1.19 (1.7)	7.32 (10.4)
RR-4	76.06	2.64 (3.5)	8.66 (11.4)
RR-5	76.16	2.77 (3.6)	7.38 (9.7)
RR-6	80.87	2.79 (3.4)	7.20 (8.9)

Pairs of test results obtained by the procedure described in the study are expected to differ in absolute value by less than $2.77 \cdot S$, where $2.77 \cdot S$ is the 95% probability limit on the difference between two test results, and S is the appropriate estimate of standard deviation. Definitions and usage are given in Standard Practices E 456 and E 177, respectively. Supporting data and statistical analyses of within-laboratory and between-laboratory reproducibility are presented in the accompanying Research Report RR:D32-1016. The procedure described is without known bias.

Reference Material 8590

Notice and Warning to Users

Handling: The Gas Oil Feed is a vacuum distillate, of which similar materials have been reported to have mutagenic and/or carcinogenic properties; therefore, this material should be handled with care. Use proper methods for disposal of waste.

Use: The Gas Oil Feed should be warmed slightly to ensure that any wax in the oil is brought into solution.

The feed characterization of Amoco Oil No. FCC 893 is attached to the accompanying Research Report.

REFERENCES

1. ASTM Standard Test Method for Fluid Cracking Catalysts by Microactivity Test, D 3907-87. Annual Book of ASTM Standards, Vol. 05.03, 1991.
2. ASTM Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test, E691-79. Annual Book of ASTM Standards Vols 06.03, 08.03, 14.02, 1991.

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:.....

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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 + UOPK	4.42 at 98.9°C(210 °F) 12.02	