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ASTM COMMITTEE D02.08 ON VOLATILITY OF PETROLEUM AND PETROLEUM PRODUCTS

RESEARCH REPORT RR:D02 – XXXX*

INTERLABORATORY TEST STUDY ON FLASH POINT: COMPARISON BETWEEN ASTM D6450-99, ASTM D93 A, AND PROPOSED NEW SETTINGS FOR THE METHOD USED IN D6450 (NEW CCCFP)

* Designation number will be assigned by ASTM Headquarters

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1. INTRODUCTION

Several ASTM standards for the determination of the flash point of petroleum and petroleum products exist. An important method is ASTM D93 Procedure A because it is has to be used for the specification of Diesel and aviation turbine fuels.
In 1999, a new method was published under the designation D6450-99. This new method uses less sample than D93, has a continuously closed sample cup, introduces air into the closed test chamber by means of a pump, uses electric arc ignition and detects flash point by an increase in pressure in the test chamber. The heating rate is the same as in D93 A. The method is also known as Continuously Closed Cup Flash Point method (CCCFP).
An interlaboratory study was conducted in 1997 (RR:D02-1464). The sample set consisted of three pure chemicals, one pure hydrocarbon, three fuels, and three lubricating oils. The samples were tested according to method D93 A as well as according to the proposed new method that later became D6450.
This study showed a fair agreement between ASTM D93 A and the proposed method, but the repeatability and reproducibility of D93 A was bigger than the published values.
Based on the results of this study, the repeatability and reproducibility of the new method were derived, and the method was later accepted and published as ASTM D6450-99.
It later turned out that for samples that were contaminated with small concentrations of material with a much lower flash point (e.g. diesel with small amounts of gasoline or lubricating oil containing diesel fuel), D6450 results were higher than results obtained by test method D93 A. The observed differences were, depending on the nature and concentration of the contaminant, up to 8°C.
The manufacturer of the D6450 apparatus informed ASTM about this offset in 2000. They informed about research to find modifications to the D6450 method in order to reduce the bias between D6450 and D93 A even for such contaminated samples. The Task Group on CCCFP was therefore not disbanded.
In December 2000, data were presented that suggested that with a change of some parameters in D6450, the difference to D93 results for contaminated samples could be made much smaller. Additionally, the results of a study conducted in five different Austrian laboratories were presented. The sample set for this test program consisted of fuels, lubricating oils, and contaminated diesel fuels. The data of this small study indicated that the modifications lead in fact to a very good agreement between the new, modified CCCFP method and D93 A.
Based on the results of this preliminary study, the Task Group decided to conduct an interlaboratory study to generate data that would serve as a basis to decide if D6450 should be modified. It was agreed that the sample set shall include contaminated fuels and lubricating oils and that the samples shall be tested using D6450-99, D93 A, and the proposed new CCCFP method.
Michael Palmer, Petrolab, volunteered to organize the study.
The test program started in October 2001. By December 2001, all but one participant had returned their data. The data and the results of the data analysis were presented to the Task Group at the ASTM D02 meeting in June 2002.

2. D6450 AND THE PROPOSED NEW CCCFP METHOD

In D6450, a sample volume of 1 mL is placed in a sample cup with a total volume of 4 mL. The cup is pressed against an oven plate, thus forming a closed but not sealed chamber, and heated from above at a rate of 5.5 °C/min. The temperature of the sample is monitored by a thermocouple that dips into the liquid sample. The sample is stirred by a magnetic stirrer. The ignition source is an electric arc delivered between two electrodes. The gap between the electrodes is 2.5 mm, and the duration of the arc is 43 ms per arc, delivering an energy of 3 mJ per ignition. The ignition frequency is 1 ignition per °C temperature increase of the sample.

After each arc, 1.5 ml air are introduced into the vapor space by means of a small pump in order to deliver oxygen that might have been consumed to the vapor space.

The pressure in the closed chamber is monitored with a pressure transducer. If the vapors do not ignite after an ignition, the pressure in the cup does not increase markedly after when the electric arc is delivered. If, however, a significant fraction of the vapors are ignited and burn, both temperature and pressure increase very rapidly in the chamber. A steep pressure increase above a threshold value is therefore the indication of a flash point in D6450. The threshold value is set to 20 kPa.

The proposed changes to the existing D6450 are summarized in Table 2.1 and Table 2.2.

The rationale for these changes are:

1) In case of a contaminated material, the increased sample volume provides for a bigger reservoir of trace materials.
2) The amount of air introduced into the chamber has to be adjusted in order not to dilute vapors of contaminants too much.
3) The duration of the arc and hence the energy per arc has to be reduced in order not too burn off vapors of trace contaminats already before the flash point is reached.
4) The heating rate has to be lowered so that there is more time for vapors of trace contaminats to be formed after an ignition.

TABLE 2.1 Summary of test conditions for D6450-99 and New CCCFP

PARAMETER	D6450-99	New CCCFP
Pressure threshold (kPa)	20 ± 0.5	20 ± 0.5
Ignition frequency ($°C^{-1}$)	1	1
Stirres speed (rev/min)	260 ± 10	260 ± 10
Chamber volume (mL)	4 ± 0.2	7 ± 0.3
Sample volume (mL)	1 ± 0.1	2 ± 0.2
Introduced air (mL)	1.5 ± 0.5	T-dependent (see Table 2.2)
Arc duration (ms)	43 ± 3	19 ± 2
Arc energy (mJ)	3 ± 0.5	1.3 ± 0.3
Heating rate (°C/min)	5.5 ± 0.5	2.5 ± 0.3

TABLE 2.2 Volume of introduced air as a function of sample temperature for New CCCFP

Sample temperature range (°C)	introduced volume of air (ml)
Below 80	0
80 to below 150	0.5 ± 0.15
150 to below 200	1 ± 0.2
200 to below 300	1.5 ± 0.3

300 and above	2 ± 0.4

3. TEST PROGRAM OBJECTIVES

The objectives of the interlaboratory test program were:

1) To determine the bias relative to D93 A for the new CCCFP method and for D6450-99.

2) To test if the new CCCFP method has a smaller bias relative to D93 A than D6450 for samples contaminated with a small amount of material of considerably lower flash point.

3) To determine the repeatability (r) and reproducibility (R) for the new CCCFP method, based on the sample set used in the study.

4. TEST METHODS

The following test methods were used in the interlaboratory test program:

1) ASTM D93 A
2) ASTM D6450-99
3) Proposed new CCCFP method

The participants were requested to have their own copy of D93 and D6450-99.
The new CCCFP is only a modification of some parameter settings on the flash point apparatus and is, apart from these parameter values, equal to D6450. It uses the same apparatus. The only available flash point apparatus capable of performing D6450 is a microprocessor controlled instrument that performs the test automatically. The parameter values can be changed in the software.
Therefore, the participants received Tables 1.1 and 1.2 to inform about the differences between the new CCCFP and D6450.

5. SAMPLE SET DESIGN

The sample set consisted of 13 samples. The type of sample used is shown in Table 5.1.

TABLE 5.1 Sample Set

SAMPLE TYPE	SOURCE
Jet A (1)	Mobil Refinery, Torrance, CA (donated)
Jet A(2)	Chevron Refinery, Richmond, CA (donated)
Diesel (1)	Mobil Refinery, Torrance, CA (donated)
Diesel (2)	Chevron Refinery, Richmond, CA (donated)
Diesel (3)	Hess (purchased retail)
Lubricating Oil (1)	Mobil (purchased retail)
Lubricating Oil (2)	Chevron Refinery, Richmond, CA (donated)
2-Stroke Oil	Mobil (purchased retail)
Anisole	Aldrich Chemicals (purchased)
Jet A (1) w/ 0.25 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 0.4 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 0.4 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 2.0 V% biodiesel	Prepared by Petrolab (Biodiesel from Mark IV Consultants, donated)
Diesel (2) w/ 0.3 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Diesel (1) w/ 0.5 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Lube Oil (1) w/ 3 V% diesel	Prepared by Petrolab

The contaminated samples were prepared by adding the respective percentages of the contaminant volumetrically to the pure samples contained in large drums.
The samples were homogenized by rolling the sample containers. After that, samplese were filled in 250 mL glass bottles.
Per participating laboratory and sample, three 250m mL bottles were filled, one for each method so that each method could be run on a fresh sample. In addition, four to nine additional bottles were filled with different samples for the respective participants, so that duplicate measurements were performed by each participant for each method. For the sample Jet A (1), four bottles per participant were filled, and it was run twice by each participant by the new CCCFP method as a blind duplicate set. The duplicates were added to generate data for the calculation of repeatability.
The sample bottles were labelled only with a number and the approximate flash point in order to enable the user to set the initial temperature.
The numbering was random, so that the participants did not know which sample they tested, nor which samples were the duplicates.
Details about the test samples were not provided to the participants in order to minimize effects due to prior knowledge of the samples.
Each participanting laboratory was requested to purchase Dodecane locally and use it as qualification sample before the test program samples were tested.
The sample set thus consisted of 19 samples for D6450-99 and D93 A (dodecane, 16 test samples, 2 blind duplicates) and 20 samples for the new CCCFP method (dodecane, 16 test samples, 3 blind duplicates).

6. PARTICIPATING LABORATORIES

The following 9 laboratories participated in the test program (the order of the participants listed here is not the order of laboratories used in the presentation of the data later in this report):

Mike Jordan
Motiva - DE
2000 Wrangle Hill Rd
Delaware City, DE 19706
Phone: 302-836-6528

Gary Hine
Coastal Eagle Point (El Paso)
PO Box
Westville, NJ 08093
Phone: 856-853-3161

Erna Beal
Naval Research Program
Fuels Section, Code 6181
Washington, DC 20375
Phone: 202-767-2673

Geri Deputy
ITS / Caleb Brett
327 Erickson Ave
Essington, PA 19029
Phone: 610-521-1725

Gretchen Wendtland
Buckeye Pipeline
5002 Buckeye Rd
Emmaus, PA 18049
Phone: 484-232-4548

Michael Palmer
Petrolab Company
874 Albany Shaker Rd
Latham, NY 12110
Phone: 518 424 4833

Andreas Schwarzmann
Grabner Instruments
Dr.-Otto-Neurathgasse 1
1220 Wien
Austria
Phone: +43 1 282 16 27 20 35

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Dr. Alfred Ecker
FICHTE Petroleum Institute
Faradaygasse 3, Arsenal Obj. 214
1030 Wien
Austria
Phone: +43 1 79747 6552

Fay Hutto
MISSISSIPPI STATE CHEMICAL LAB
PO DRAWER CR
PETROLEUM PRODS LAB
MISSISSIPPI STATE, MS 39762-5622
Phone: 662-325-2653

7. INTERLABORATORY TEST PROGRAM INSTRUCTIONS

The following text was distributed to the participants of the test program (MINIFLASH is the name of the D6450 apparatus):

FLASH POINT DETERMINATION WITH MINIFLASH

Unpack the instrument and prepare following items for the measurement.

- sample cup for ASTM D 6450 measurement
- sample cup for NEW CCCFP measurement
- stirring magnets
- glass fiber eraser for cleaning the arc pins
- sandpaper file for cleaning the arc pins
- pipettes

Prepare tissue for cleaning the oven and sample cup.

After power on the display shows the main menu

```
****************************************
CCA-FLP     VERS. 4.10  06/11/2000 09:37
*Measure        *Printer          *Setup
****************************************
```

Shift the cursor to ***Measure** and press **TASK**.

```
****************************************
≈ *0_  No:1 *D 6450     Ta=75 Te= 120 C
sample: 1 ml              Toven= 20,3 C
****************************************
```

the measuring menu is displayed.

PLEASE READ THE INSTRUCTIONS FIRST BEFORE YOU START THE MEASUREMENTS.

1) ASTM D 6450:

All parameters are already pre-programmed in program No 1 to measure according to ASTM D 6450, except the start and final temperature

```
****************************************
≈ *0_  No:1 *D 6450    Ti=75 Tf= 120 C
sample: 1 ml              Toven= 20,3 C
****************************************
```

Set the start temperature Ti at least 18°C (32°F) below the expected (on the label of the glass bottle mentioned flash point) flash point and the final temperature 30°C (54°F) to 50°C (90°F) beyond.

The sample has to be stored at temperatures below the expected flash temperature to avoid vapor loss. Furthermore, the instrument does not start the test when the sample temperature is beyond the start temperature.

Procedure:

After programming the start and final temperature for program No 1, press RUN to start the measurement.

If the start temperature is reached the message “fill sample, press RUN” is displayed. Fill the small sample cup labeled with “ASTM D 6450" with 1mL of sample.

DO NOT PUT THE SAMPLE IN A HOT CUP! IF THE CUP IS STILL HOT COOL IT TO AMBIENT TEMPERATURE!

Put the stirring magnet into the cup.

Place the sample cup into the opening of the tester and close the front door. After pressing RUN five cleaning arcs are initiated and the cup is lifted up to start the measurement.

After the measurement is finished the cup is lowered and the result displayed. Note the flash temperature and the actual barometric pressure on the report sheet and empty the sample cup. It can be easily cleaned with a tissue (see section “Cleaning”).

Clean and dry the electrodes and the oven surface with a tissue and the glass fiber eraser (see section “cleaning”).

Cool the sample cup. Now the instrument is ready for the next test.

2) New CCCFP method:

Switch to program 2 with ↑ on No: 1.

All parameters are already pre-programmed in program No 2 to measure according to the new CCCFP method, except the start and final temperature

```
*****************************************
≈ *0_  No:2 *CCCFP  Ti=75 Tf= 120 C
sample: 2 ml              Toven= 20,3 C
*****************************************
```

Set the start temperature Ti at least 18°C (32°F) below the expected (on the label of the glass bottle mentioned flash point) flash point and the final temperature 30°C (54°F) to 50°C (90°F) beyond.

The sample has to be stored at temperatures below the expected flash temperature to avoid vapor loss. Furthermore, the instrument does not start the test when the sample temperature is beyond the start temperature.

Procedure:

1) After programming the start and final temperature for program 2, press RUN to start the measurement.
2) If the start temperature is reached the message “fill sample, press RUN” is displayed. Fill the larger sample cup labeled with “NEW CCCFP" with 2mL of sample.

DO NOT PUT THE SAMPLE IN A HOT CUP! IF THE CUP IS STILL HOT COOL IT TO AMBIENT TEMPERATURE!

3) Put the stirring magnet into the cup.
4) Place the sample cup into the opening of the tester and close the front door. After pressing RUN five cleaning arcs are initiated and the cup is lifted up to start the measurement.
5) After the measurement is finished the cup is lowered and the result displayed. Note the flash temperature and the actual barometric pressure on the report sheet and empty the sample cup. It can be easily cleaned with a tissue (see section “cleaning”).
6) Clean and dry the electrodes and the oven surface with a tissue and the glass fiber eraser (see section “cleaning”).
7) Cool the sample cup. Now the instrument is ready for the next test.

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Cleaning:

Oven:

Please clean the surface of the oven plate well with tissue. **Do not scratch**.
The surface must be free of residuals and scratches for good heat contact and correct arcing.
Do not bend the temperature sensor and the arc pins.

Tilt back the instrument, open the front door and perform the cleaning procedures as described below:

Clean the arc pins and oven surface carefully with tissue after each measurement. Do not bend the temperature sensor and the arc pins.

Use the glass fiber eraser to clean the arc pins. This is necessary to regain a metal surface at the tips for easy ignition.
Use additionally the delivered sandpaper coated file for cleaning when the message „clean arc pins" appears during the test.

Sample cup

Empty the cup and use a tissue to wipe the cup dry.

Error Message

If the arc pins are corroded or heavily dirty then it is possible that an arc between the pins can not be produced anymore. Then the pressure increase is zero and the instrument gives a warning as following:

```
****************************************
*⌉_ S      DIESEL        Toven=116.0 F
clean arc pins          Tsample=125.6 F
****************************************
```

Press STOP to break the measurement and clean the arc pins (electrodes) very well with the delivered **sandpaper file**. Clean the pins like with the glass fiber eraser (see section cleaning) until a metal surface is regained.

8. DATA REPORT FORMS

The following tables show the data report forms as received by the nine participating laboratories. The order of laboratories is not the same as the order in the list of participants in Section 6.
The participants were also requested to report the manufacturer and type of the D93 A apparatus used for the tests.
Laboratory number 5 returned only data for method D93 A, but not for D6450 and New CCCFP.

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ASTM D6450 Round Robin Report Sheet

LABORATORY 1

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	178	81	Dodecane	58	77	Dodecane	58	79
A1	135	142	61	B1	35	42	C1	130	147
A2	125	138	59	B2	52	70	C2	50	70
A3	310	406	208	B3	135	159	C3	30	43
A4	110	117	47	B4	34	49	C4	30	46
A5	100	106	41	B5	37	52	C5	45	62
A6	100	102	39	B6	185	198	C6	195	209
A7	120	133	56	B7	35	63	C7	25	39
A8	105	117	47	B8	40	60	C8	41	58
A9	305	390	199	B9	195	223	C9	30	47
A10	110	122	50	B10	35	47	C10	35	49
A11	105	115	46	B11	45	65	C11	30	47
A12	140	147	64	B12	50	64	C12	180	196
A13	280	289	143	B13	27	41	C13	40	55
A14	100	111	44	B14	29	42	C14	35	49
A15	110	117	47	B15	45	62	C15	27	41
A16	130	136	58	B16	35	47	C16	45	60
A20	140	144	62	B26	82	58	C17	35	48
A24	105	108	42	B33	75	43	C24	45	60
A27	110	113	45				C30	30	49
A29	115	120	49				C34	27	43

ASTM D93 : Please reference manufacturer and auto / manual
MANUAL

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ASTM D6450 Round Robin Report Sheet

LABORATORY 2

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	180	82	Dodecane	58	76	Dodecane	58	80
A1	135	143	62	B1	35	44	C1	130	143
A2	125	149	65	B2	52	70	C2	50	69
A3	310	320	160	B3	135	156	C3	30	44
A4	110	128	53	B4	34	45	C4	30	48
A5	100	117	47	B5	37	47	C5	45	64
A6	100	103	39	B6	185	210	C6	195	224
A7	120	127	53	B7	35	52	C7	25	36
A8	105	110	43	B8	40	64	C8	41	59
A9	305	315	157	B9	195	210	C9	30	42
A10	110	115	46	B10	35	49	C10	35	52
A11	105	126	52	B11	45	67	C11	30	51
A12	140	148	64	B12	50	62	C12	180	196
A13	280	288	142	B13	27	37	C13	40	56
A14	100	106	41	B14	29	43	C14	35	50
A15	110	98	37	B15	45	65	C15	27	48
A16	130	145	63	B16	35	53	C16	45	60
A28	310	326	163	B26	35	54	C17	35	48
A33	105	102	39	B30	45	65	C36	35	50
				B31	50	62			

ASTM D93 : Please reference manufacturer and auto / manual

PETROTEST PMA-4 Automatic

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ASTM D6450 Round Robin Report Sheet

LABORATORY 3

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	173	78	Dodecane	58	77	Dodecane	58	77
A1	135	149	65	B1	35	43	C1	130	144
A2	125	145	63	B2	52	73	C2	50	67
A3	310	350	177	B3	135	160	C3	30	43
A4	110	116	47	B4	34	49	C4	30	48
A5	100	110	43	B5	37	51	C5	45	63
A6	100	112	44	B6	185	205	C6	195	221
A7	120	138	59	B7	35	60	C7	25	44
A8	105	122	50	B8	40	62	C8	41	59
A9	305	344	173	B9	195	212	C9	30	48
A10	110	128	53	B10	35	53	C10	35	52
A11	105	119	48	B11	45	71	C11	30	50
A12	140	143	62	B12	50	66	C12	180	195
A13	280	298	148	B13	27	50	C13	40	58
A14	100	119	48	B14	29	43	C14	35	52
A15	110	98	37	B15	45	64	C15	27	47
A16	130	135	57	B16	35	48	C16	45	61
A29	115	136	58	B20	35	42	C17	35	50
A33	105	119	48				C25	195	224
A35	135	129	54				C27	41	56
							C35	45	64

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ASTM D93 : Please reference manufacturer and auto / manual

ISL AUTOMATED

ASTM D6450 Round Robin Report Sheet

LABORATORY 4

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	189	87	Dodecane	58	77	Dodecane	58	77
A1	135	149	65	B1	35	42	C1	130	146
A2	125	145	63	B2	52	72	C2	50	65
A3	310	313	156	B3	135	165	C3	30	44
A4	110	123	51	B4	34	51	C4	30	47
A5	100	113	45	B5	37	49	C5	45	56
A6	100	107	42	B6	185	200	C6	195	217
A7	120	136	58	B7	35	60	C7	25	37
A8	105	121	49	B8	40	61	C8	41	71
A9	305	301	149	B9	195	224	C9	30	45
A10	110	125	52	B10	35	49	C10	35	50
A11	105	121	49	B11	45	72	C11	30	49
A12	140	152	67	B12	50	66	C12	180	196
A13	280	290	143	B13	27	46	C13	40	53
A14	100	115	46	B14	29	45	C14	35	49
A15	110	100	38	B15	45	n/a	C15	27	40
A16	130	134	57	B16	35	53	C16	45	n/a
A22	405	321	161	B33	29	46	C17	35	49
							C23	30	49
							C26	25	39
							C28	30	47

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									C30	80	48

ASTM D93 : Please reference manufacturer and auto / manual
ISL AUTOMATED

ASTM D6450 Round Robin Report Sheet

LABORATORY 5

ASTM D93 PenskyMartens					**ASTM D6450 CCCFP**				**Alternate Method**		
Sample ID	Estimated FP (F)	Result (F)	Result (C)		Sample ID	Initial Temp (C)	Result (C)		Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	177	81		Dodecane	58			Dodecane	58	
A1	135	142	61		B1	35			C1	130	
A2	125	132	56		B2	52			C2	50	
A3	310	410	210		B3	135			C3	30	
A4	110	116	47		B4	34			C4	30	
A5	100	106	41		B5	37			C5	45	
A6	100	100	38		B6	185			C6	195	
A7	120	130	54		B7	35			C7	25	
A8	105	112	44		B8	40			C8	41	
A9	305	385	196		B9	195			C9	30	
A10	110	116	47		B10	35			C10	35	
A11	105	112	44		B11	45			C11	30	
A12	140	160	71		B12	50			C12	180	
A13	280	290	143		B13	27			C13	40	
A14	100	110	43		B14	29			C14	35	
A15	110	116	47		B15	45			C15	27	
A16	130	136	58		B16	35			C16	45	
A21	130	136	58		B29	35			C17	35	

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A27	110	112	44				C21	50	
A28	380	390	199						
A29	115	118	48						

ASTM D93 : Please reference manufacturer and auto / manual
MANUAL

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ASTM D6450 Round Robin Report Sheet

LABORATORY 6

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	185	85	Dodecane	58	77	Dodecane	58	80
A1	135	146	65	B1	35	43	C1	130	145
A2	125	149	65	B2	52	71	C2	50	70
A3	310	305	152	B3	135	154	C3	30	44
A4	110	120	49	B4	34	47	C4	30	48
A5	100	108	42	B5	37	49	C5	45	71
A6	100	102	39	B6	185	202	C6	195	223
A7	120	134	57	B7	35	55	C7	25	36
A8	105	116	47	B8	40	63	C8	41	60
A9	305	370	188	B9	195	204	C9	30	44
A10	110	125	52	B10	35	49	C10	35	50
A11	105	117	47	B11	45	67	C11	30	49
A12	140	150	66	B12	50	62	C12	180	198
A13	280	285	141	B13	27	39	C13	40	56
A14	100	114	46	B14	29	43	C14	35	50
A15	110	122	50	B15	45	65	C15	27	47
A16	130	139	59	B16	35	53	C16	45	59
A20	140	144	62	B24	37	53	C17	35	48
				B29	35	47	C20	130	149
							C22	30	43
							C27	41	61
							C31	180	195

ASTM D93 : Please reference manufacturer and auto / manual

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ISL AUTOMATED

ASTM D6450 Round Robin Report Sheet

LABORATORY 7

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	182	83	Dodecane	58	77	Dodecane	58	80
A1	135	148	64	B1	35	42	C1	130	146
A2	125	137	58	B2	52	68	C2	50	70
A3	310	315	157	B3	135	164	C3	30	44
A4	110	120	49	B4	34	52	C4	30	48
A5	100	110	43	B5	37	50	C5	45	64
A6	100	102	39	B6	185	197	C6	195	220
A7	120	132	56	B7	35	61	C7	25	38
A8	105	119	48	B8	40	61	C8	41	69
A9	305	290	143	B9	195	225	C9	30	44
A10	110	124	51	B10	35	50	C10	35	50
A11	105	117	47	B11	45	65	C11	30	46
A12	140	148	64	B12	50	66	C12	180	195
A13	280	297	147	B13	27	47	C13	40	57
A14	100	112	44	B14	29	48	C14	35	47
A15	110	124	51	B15	45	68	C15	27	39
A16	130	142	61	B16	35	48	C16	45	61
A24	105	109	43	B31	50	67	C17	35	49
				B34	45	68	C29	35	50
							C31	180	197
							C34	27	37

ASTM D93 : Please reference manufacturer and auto / manual

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TANAKA AUTOMATED

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ASTM D6450 Round Robin Report Sheet

LABORATORY 8

ASTM D93 PenskyMartens				ASTM D6450 CCCFP			Alternate Method		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	172	78	Dodecane	58	77	Dodecane	58	78
A1	135	145	63	B1	35	42	C1	130	146
A2	125	145	63	B2	52	70	C2	50	70
A3	310	424	218	B3	135	158	C3	30	42
A4	110	118	48	B4	34	50	C4	30	46
A5	100	108	42	B5	37	50	C5	45	63
A6	100	102	39	B6	185	194	C6	195	217
A7	120	136	58	B7	35	59	C7	25	42
A8	105	129	54	B8	40	61	C8	41	57
A9	305	387	197	B9	195	211	C9	30	46
A10	110	124	51	B10	35	47	C10	35	48
A11	105	117	47	B11	45	67	C11	30	47
A12	140	151	66	B12	50	64	C12	180	194
A13	280	289	143	B13	27	42	C13	40	55
A14	100	113	45	B14	29	44	C14	35	49
A15	110	120	49	B15	45	61	C15	27	41
A16	130	142	61	B16	35	48	C16	45	59
A23	110	118	48	B25	185	200	C17	35	48
A33	105	113	45	B30	45	66	C24	45	62
							C28	30	47

ASTM D93 : Please reference manufacturer and auto / manual

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MANUAL

ASTM D6450 Round Robin Report Sheet

LABORATORY 9

ASTM D93 PenskyMartens				**ASTM D6450 CCCFP**			**Alternate Method**		
Sample ID	Estimated FP (F)	Result (F)	Result (C)	Sample ID	Initial Temp (C)	Result (C)	Sample ID	Initial Temp (C)	Result (C)
Dodecane	175	180	82	Dodecane	58	77	Dodecane	58	78
A1	135	148	64	B1	35	40	C1	130	147
A2	125	138	59	B2	52	73	C2	50	69
A3	310	405	207	B3	135	165	C3	30	42
A4	110	122	50	B4	34	49	C4	30	47
A5	100	108	42	B5	37	50	C5	45	63
A6	100	102	39	B6	185	200	C6	195	206
A7	120	134	57	B7	35	54	C7	25	40
A8	105	116	47	B8	40	59	C8	41	60
A9	305	367	186	B9	195	223	C9	30	47
A10	110	124	51	B10	35	46	C10	35	50
A11	105	116	47	B11	45	63	C11	30	48
A12	140	158	70	B12	50	53	C12	180	197
A13	280	284	140	B13	27	37	C13	40	56
A14	100	116	47	B14	29	37	C14	35	50
A15	110	118	48	B15	45	58	C15	27	43
A16	130	138	59	B16	35	45	C16	45	59
A21	130	missed	missed	B25	185	198	C17	35	48
A23	110	122	50				C32	40	57

ASTM D93 : Please reference manufacturer and auto / manual

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MANUAL

9. STATISTICAL DATA SUMMARY

9.1. Results by method for the different sample types

TABLE 9.1 Flash point results for method D93 A

Sample #	Sample Type	LAB1	LAB2	LAB3	LAB4	LAB5	LAB6	LAB7	LAB8	LAB9	Mean	St. Dev.
	Dodecane	81	82	78	87	81	85	83	78	82	81,9	2,93
A1	Diesel(2)	61	62	65	65	61	63	64	63	64	63,1	1,54
A2	Diesel(1)	59	65	63	63	56	65	58	63	59	61,2	3,27
A3	Lube Oil (2)	208	160	177	156	210	152	157	218	207	182,8	27,58
A4	Jet A(2)	47	53	47	51	47	49	49	48	50	49,0	2,06
A5	Jet A(2) w/ 0.4% gasoline	41	47	43	45	41	42	43	42	42	42,9	1,96
A6	Jet A(2) w/ 0.7% gasoline	39	39	44	42	38	39	39	39	39	39,8	1,92
A7	Diesel(2) w/ 0.3% gasoline	56	53	59	58	43	57	56	58	57	55,2	4,89
A8	Diesel(1) w/ 0.5% gasoline	47	43	50	49	44	47	48	54	47	47,7	3,24
A9	Lube Oil (1)	199	157	173	149	196	188	143	197	186	176,4	21,80
A10	Jet A(1)	50	46	53	52	47	52	51	51	51	50,3	2,35
A11	Jet A(1) w/ 0.25% gasoline	46	52	48	49	44	47	47	47	47	47,4	2,19
A12	2-Cylinder Oil	64	64	62	67	71	66	64	66	70	66,0	2,96
A13	Lube Oil (1) w/ 3.0% diesel	143	142	148	143	143	141	147	143	140	143,3	2,60
A14	Anisole	44	41	48	46	43	46	44	45	47	44,9	2,15
A15	Jet A(2) w/ 2.0% Biodiesel	47	37	37	38	47	50	51	49	48	44,9	5,82
A16	Diesel(3)	58	63	57	57	58	59	61	61	59	59,2	2,05

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TABLE 9.2 Flash point results for method D6450 - 99

Grabner Miniflash Flash Point Data (ASTM D6450-99)

Sample #	Sample Type	LAB1	LAB2	LAB3	LAB4	LAB5	LAB6	LAB7	LAB8	LAB9	Mean	St. Dev.
	Dodecane	77	76	**77**	77		77	77	77	**77**	**76,9**	**0,35**
B1	Anisole	42	44	43	42		43	42	42	40	**42,3**	**1,16**
B2	2-Cylinder Oil	70	70	73	72		71	68	70	73	**70,9**	**1,73**
B3	Lube Oil w/ 3.0% diesel	159	156	160	165		154	164	158	165	**160,1**	**4,19**
B4	Jet A(1) w/ 0.25% gasoline	49	45	49	51		47	52	50	49	**49,0**	**2,20**
B5	Jet A(1)	52	47	51	49		49	50	50	50	**49,8**	**1,49**
B6	Lube Oil (1)	198	210	205	200		202	197	194	200	**200,8**	**4,98**
B7	Diesel(1) w/ 0.5% gasoline	63	52	60	60		55	61	59	54	**58,0**	**3,85**
B8	Diesel(2) w/ 0.3% gasoline	60	64	62	61		63	61	61	59	**61,4**	**1,60**
B9	Lube Oil (2)	223	210	212	224		204	225	211	223	**216,5**	**8,12**
B10	Jet A(2)	47	49	53	49		49	50	47	46	**48,8**	**2,19**
B11	Diesel(1)	65	67	71	72		67	65	67	63	**67,1**	**3,04**
B12	Diesel(2)	64	62	66	66		62	66	64	53	**62,9**	**4,32**
B13	Jet A(2) w/ 0.7% gasoline	41	37	50	46		39	47	42	37	**42,4**	**4,84**
B14	Jet A(2) w/ 0.4% gasoline	42	43	43	45		43	48	44	37	**43,1**	**3,09**
B15	Diesel(3)	62	65	64	n/a		65	68	61	58	**63,3**	**3,25**
B16	Jet A(2) w/ 2.0% Biodiesel	47	53	48	53		53	48	48	45	**49,4**	**3,16**

TABLE 9.3 Flash point results for new CCCFP method

New CCCFP Grabner Miniflash Settings Flash Point Data

Sample #	Sample Type	LAB1	LAB2	LAB3	LAB4	LAB5	LAB6	LAB7	LAB8	LAB9	Mean	St. Dev.
	Dodecane	79	80	77	77		80	80	78	78	**78,6**	**1,30**
C1	Lube Oil (1) w/ 3.0% diesel	147	143	144	146		145	146	146	147	**145,5**	**1,41**
C2	2-Cylinder Oil	70	69	67	65		70	70	70	69	**68,8**	**1,83**
C3	Anisole	43	44	43	44		44	44	42	42	**43,3**	**0,89**
C4	Jet A(1) w/ 0.25% gasoline	46	48	48	47		48	48	46	47	**47,3**	**0,89**
C5	Diesel(2)	62	64	63	56		71	64	63	63	**63,3**	**4,06**
C6	Lube Oil (2)	209	224	221	217		223	220	217	206	**217,1**	**6,49**
C7	Jet A(2) w/ 0.7% gasoline	39	36	44	37		36	38	42	40	**39,0**	**2,88**
C8	Diesel(1)	58	59	59	71		60	69	57	60	**61,6**	**5,29**
C9	Jet A(2)	47	42	48	45		44	44	46	47	**45,4**	**2,00**
C10	Jet A(1)	49	52	52	50		50	50	48	50	**50,1**	**1,36**
C11	Diesel(1) w/ 0.5% gasoline	47	51	50	49		49	46	47	48	**48,4**	**1,69**
C12	Lube Oil (1)	196	196	195	196		198	195	194	197	**195,9**	**1,25**
C13	Diesel(2) w/ 0.3% gasoline	55	56	58	53		56	57	55	56	**55,8**	**1,49**
C14	Jet A(1) DUPLICATE SET	49	50	52	49		50	47	49	50	**49,5**	**1,41**
C15	Jet A(2) w/ 0.4% gasoline	41	48	47	40		47	39	41	43	**43,3**	**3,58**
C16	Diesel(3)	60	60	60	n/a		59	61	59	59	**59,7**	**0,76**
C17	Jet A(2) w/ 2.0% Biodiesel	48	48	50	49		48	49	48	48	**48,5**	**0,76**

a. Results of the duplicate measurements for the three different methods

TABLE 9.4 Results for the blind duplicates for method D93 A

ASTM FLASH POINT BLIND DUPLICATES				
	ASTM D93 A			
LAB	**BLIND DUPLICATES**	**ASTM D93**		**+/-**
LAB2	Anisole	41	39	*2*
LAB3	Anisole	48	48	*0*
LAB8	Anisole	45	45	*0*
LAB5	Diesel (1)	61	58	*3*
LAB1	Diesel (1) w/ 0.5% gasoline	47	45	*2*
LAB5	Diesel (1) w/ 0.5% gasoline	44	44	*0*
LAB1	Diesel (2)	61	62	*-1*
LAB6	Diesel (2)	65	62	*3*
LAB3	Diesel (3)	57	54	*3*
LAB1	Jet A (1)	50	49	*1*
LAB3	Jet A (1)	53	58	*-5*
LAB5	Jet A (1)	47	48	*-1*
LAB8	Jet A (2)	48	48	*0*
LAB9	Jet A (2)	50	50	*0*
LAB1	Jet A (2) w/ 0.4% gasoline	41	42	*-1*
LAB7	Jet A (2) w/ 0.4% gasoline	43	43	*0*
LAB2	Lube Oil (1)	157	163	*-6*
LAB5	Lube Oil (1)	196	199	*-3*
LAB4	Lube Oil (2)	156	161	*-5*

TABLE 9.5 Results for the blind duplicates for method D6450 - 99

ASTM FLASH POINT BLIND DUPLICATES

ASTM D6450 - 99

LAB	BLIND DUPLICATES	ASTM D6450		+/-
LAB3	Anisole	43	42	*1*
LAB8	Diesel (1)	67	66	*1*
LAB7	Diesel (2)	66	67	*-1*
LAB7	Diesel (3)	68	68	*0*
LAB2	Diesel(1)	67	65	*2*
LAB1	Diesel(1) w/ 0.5% gasoline	63	58	*5*
LAB2	Diesel(1) w/ 0.5% gasoline	52	54	*-2*
LAB2	Diesel(2)	62	62	*0*
LAB6	Jet A (1)	49	53	*-4*
LAB6	Jet A (2)	49	47	*2*
LAB5	Jet A(2)			*0*
LAB1	Jet A(2) w/ 0.4% gasoline	42	43	*-1*
LAB4	Jet A(2) w/ 0.4% gasoline	46	45	*1*
LAB8	Lube Oil (1)	194	200	*-6*
LAB9	Lube Oil (1)	200	198	*2*

TABLE 9.6 Results for the complete blind duplicate set for the new CCCFP method

New CCCFP Blind Duplicate Complete Set

LAB	BLIND DUPLICATES	Modified		+/-
LAB1	JET A (1)	49	49	0
LAB2	JET A (1)	52	50	2
LAB3	JET A (1)	52	52	0
LAB4	JET A (1)	50	49	1
LAB5	JET A (1)			0
LAB6	JET A (1)	50	50	0
LAB7	JET A (1)	50	47	3
LAB8	JET A (1)	48	49	-1
LAB9	JET A (1)	50	50	0

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TABLE 9.7 Results for the blind duplicates for the new CCCFP method

ASTM FLASH POINT BLIND DUPLICATES

Modified Miniflash New CCCFP

LAB	BLIND DUPLICATES	Modified		+/-
LAB5	2-Cylinder Oil			0
LAB6	Anisole	44	43	1
LAB6	Diesel (1)	60	61	-1
LAB8	Diesel (2)	63	62	1
LAB3	Diesel(1)	59	56	3
LAB1	Diesel(1) w/ 0.5% gasoline	49	47	2
LAB4	Diesel(1) w/ 0.5% gasoline	49	48	1
LAB1	Diesel(2)	60	62	-2
LAB9	Diesel(2) w/ 0.3% gasoline	56	57	-1
LAB3	Diesel(3)	61	64	-3
LAB7	Jet A (1)	50	50	0
LAB8	Jet A (2)	46	47	-1
LAB4	Jet A(1) w/ 0.25% gasoline	47	49	-2
LAB4	Jet A(2)	45	47	-2
LAB1	Jet A(2) w/ 0.4% gasoline	43	41	2
LAB7	Jet A(2) w/ 0.4% gasoline	39	37	2
LAB4	Jet A(2) w/ 0.7% gasoline	37	39	-2
LAB2	Jet A(2) w/ 2.0% Biodiesel	50	48	2
LAB6	Lube Oil (1)	198	195	3
LAB7	Lube Oil (1)	195	197	-2
LAB3	Lube Oil (2)	221	224	-3
LAB6	Lube Oil w/ 3.0% diesel	145	149	-4

b. Results by sample for the different test methods

	Jet A (1)			Duplicate
LAB	D93	D6450	Modified	Modified
1	50	52	49	49
2	46	47	52	50
3	53	51	52	52
4	52	49	50	49
5	47			
6	52	49	50	50
7	51	50	50	47
8	51	50	48	49
9	51	50	50	50
mean	50,3	49,8	50,1	49,5
Std. dev.	2,34	1,18	1,28	1,40

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	Jet A (2)			Jet A (2) w/ 0.4% gasoline			Jet A (2) w/ 0.7% gasoline			Jet A (2) w/ 2% Biodiesel		
LAB	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified
1	47	47	47	41	42	41	39	41	39	47	47	39
2	53	49	42	47	43	48	39	37	36	37	53	36
3	47	53	48	43	43	47	44	50	44	37	48	44
4	51	49	45	45	45	40	42	46	37	38	53	37
5	47			41			38			47		
6	49	49	44	42	43	47	39	39	36	50	53	36
7	49	50	44	43	48	39	39	47	38	51	48	38
8	48	47	46	42	44	41	39	42	42	49	48	42
9	50	46	47	42	37	43	39	37	40	48	45	48
mean	49,0	48,8	45,4	42,9	43,1	43,3	39,8	42,4	39,0	44,9	49,4	40,0
Std. dev.	1,92	2,07	1,89	1,83	3,06	3,46	1,90	4,81	2,88	5,77	3,01	4,29

	Diesel (1)			Diesel (1) w/ 0.5% gasoline			Diesel (2)			Diesel (2) w/ 0.3% gasoline		
LAB	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified
1	59	65	58	47	63	47	61	64	62	56	60	55
2	65	67	59	43	52	51	62	62	64	53	64	56
3	41	71	59	50	60	50	65	66	63	59	62	58
4	63	72	71	49	60	49	65	66	56	58	61	53
5	56			44			61			43		
6	65	67	60	47	55	49	65	62	71	57	63	56
7	58	65	69	48	61	46	64	66	64	56	61	57
8	63	67	57	54	59	47	63	64	63	58	61	55
9	64	63	60	47	54	48	64	63	63	57	59	56
mean	59,3	67,1	61,6	47,7	58,0	48,4	63,3	64,1	63,3	55,2	61,4	55,8

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	Diesel (3)			Lube Oil (1)			Lube (1) w/ 3.0% Diesel			Lube Oil (2)		
Std. dev.	7,60	2,92	5,09	3,23	3,29	1,59	1,41	1,73	4,03	4,88	1,50	1,46
LAB	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified
1	58	62	60	199	198	196	143	159	147	208	223	209
2	63	65	60	157	210	196	142	156	143	160	210	224
3	57	64	61	173	205	195	148	160	144	177	212	221
4	57	n/a	n/a	149	200	196	143	165	146	156	224	217
5	58			196			143			210		
6	59	65	59	188	202	198	141	154	145	152	204	223
7	61	68	61	143	197	195	147	164	146	157	225	220
8	61	61	60	197	194	194	143	158	146	218	211	217
9	59	58	59	186	200	197	140	165	147	207	223	206
Mean	59,2	63,3	60,0	176,4	200,8	195,9	143,3	160,1	145,5	182,8	216,5	217,1
Std. dev.	2,00	3,20	0,82	20,11	4,85	1,25	2,60	4,17	1,28	25,93	7,69	5,61

	Dodecane			Anisole			Two-Cycle Oil			Jet A (1) w/ 0.25% gasoline		
LAB	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified	D93	D6450	Modified
1	81	77	79	44	42	43	64	70	70	46	49	46
2	82	76	80	41	44	44	64	70	69	52	45	48
3	78	77	77	48	43	43	62	73	67	48	49	48
4	87	77	77	46	42	44	67	72	65	49	51	47
5	81			43			71			44		
6	85	77	80	46	43	44	66	71	70	47	47	48
7	83	77	80	44	42	44	64	68	70	47	52	48
8	78	77	78	45	42	42	66	70	70	47	50	46
9	82	77	78	47	40	42	70	73	69	47	49	47
Mean	81,9	76,9	78,6	44,9	42,3	43,3	66,0	70,9	68,8	47,4	49,0	47,3

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Std. dev.	2,92	0,35	1,29	2,12	1,16	0,88	2,86	1,69	1,76	2,12	2,20	0,73

10. PRECISION

The precision data were calculated by Richard M. Stanley, Chevron Information Tech Co.

10.1 Precision for D93 A
The distributions of results for both lubricating oils and for the jet A with biodiesel were distinctively bimodal. Hence these samples were considered to be outliers and have been eliminated.
The precision estimates from the remaining sample set are:
Repeatability r = 4.1 °C
Reproducibility R = 6.9 °C
Compared to the published reproducibility of 0.078 times the flash point in °C, the precision for D93 A estimated from this test program is larger for all samples except one (Lube oil 2).

10.2 Precision for D6450-99
One outlier was eliminated from Diesel(2), all other results were included in the analysis.
The precision estimates from the remaining sample set are:
Repeatability r = 4.8 °C
Reproducibility R = 8.1 °C
Compared to the published reproducibility of R = 3.1 °C, the precision for D6450-99 estimated from this test program is significantly larger.

10.3 Precision for new CCCFP
Altogether, there were five outliers: Two in Lube oil (2), two in Diesel (1), and one outlier in Diesel (2). All other results were included in the analysis.
The precision estimates from the remaining sample set are:
Repeatability r = 4.1 °C
Reproducibility R = 5.5 °C
If the two lube oil samples are eliminated form the analysis, as was the case for the precision estimate of D93 A, the precision becomes slightly better: r = 3.8°C and R = 5.4°C.

10.4 Comparison to D93 A
The comparison between the different flash point methods used in this test program was performed by Richard M. Stanley, Chevron Information Tech Co., according to D6708, „Standard Practice for Statistical Assessment and Improvement of the Expected Agreement Between Two Methods that Purport to Measure the Same Property of a Material".
The range for these comparisons is restricted from about 40°C to about 145°C because the two samples with a higher flash point, the two lubricating oils, had to be removed from the D93 A data set (See Section 9.4.1.).

10.4.1. D6450–99 and D93 A Comparison
There is a sample specific bias which technically is random, as it passes the Anderson-Darling test.
The bias correction is: FP(D93) = 0.862 * FP(D6450) + 4.95 (in °C)
The Cross Method Reproducibility R = 8.8°C, which is larger than the reproducibility of either method.

10.4.2. New CCCFP and D93 A Comparison
There is a small amount of sample specific bias which is random.
No bias corection is needed.

The Cross Method Reproducibility R = 6.8°C, which is smaller, but not significantly smaller, than the estimate of the D93 A reproducibility.

11. RESEARCH REPORT SUMMARY

The data from the interlaboratory study group indicate that for pure fuels, oils, and chemicals, method D6450-99 show results that are very close to the results obtained by method D93 A. For the test samples that are contaminated with small amounts of material of significantly lower flash point (diesel with gasoline, jet with gasoline, lubricating oil with diesel), the results obtained by method D6450 are higher than the D93 A results. The analysis according to practice D6708 showed that there is significant bias between D6450-99 and D93 A.

The proposed new CCCFP method that is similar to D6450 but uses different test parameters gives results in close agreement to D93 A for pure fuels, oils, and chemicals, like D6450. But also for the contaminated samples, the results obtained by the new CCCFP method are very close to D93 A.
In fact, the analysis according to practice D6708 showed that, on the basis of the sample set used in the interlaboratory study, no statistically significant bias is observed between the proposed new CCCFP and D93 A.

For the two existing ASTM methods D6450 and D93, the repeatability r and and reproducibility R estimated from this test program are worse than the published values. This is, at least partly, explained by the large fraction of contaminated samples used in this study. It is a well known fact that samples of comparatively high flash point that are contaminated with a small amount of material of a much lower flash point are among the most difficult samples in the field of flash point testing.
For D93 A, however, the two samples with a high flash point of 200°C and above had to be eliminated from the data analysis because of a bimodal distribution of the results. Since these samples were pure lubricating oils, contamination cannot account for this behavior.

The results of this interlaboratory study support the expectation that modifying D6450 to the new CCCFP leads, especially for contaminated samples, to a closer agreement between D6450 and D93 A results. No bias between D93 A and the new CCCFP was observed in this test program.

12. ACKNOWLEDGEMENTS

Report Author: Roland Aschauer Grabner Instruments

Statistical Analysis: Richard Stanley, Chevron Texaco Energy Research

Sample Suppliers:

SAMPLE TYPE	SOURCE
Jet A (1)	Mobil Refinery, Torrance, CA (donated)
Jet A(2)	Chevron Refinery, Richmond, CA (donated)
Diesel (1)	Mobil Refinery, Torrance, CA (donated)
Diesel (2)	Chevron Refinery, Richmond, CA (donated)
Diesel (3)	Hess (purchased retail)
Lubricating Oil (1)	Mobil (purchased retail)
Lubricating Oil (2)	Chevron Refinery, Richmond, CA (donated)
2-Stroke Oil	Mobil (purchased retail)
Anisole	Aldrich Chemicals (purchased)
Jet A (1) w/ 0.25 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 0.4 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 0.4 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Jet A (2) w/ 2.0 V% biodiesel	Prepared by Petrolab (Biodiesel from Mark IV Consultants, donated)
Diesel (2) w/ 0.3 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Diesel (1) w/ 0.5 V% gasoline	Prepared by Petrolab (gasoline Hess regular, purchased)
Lube Oil (1) w/ 3 V% diesel	Prepared by Petrolab

Round Robin Participants:

Mike Jordan
Motiva - DE
2000 Wrangle Hill Rd
Delaware City, DE 19706
Phone: 302-836-6528

Gary Hine
Coastal Eagle Point (ElPaso)
PO Box
Westville, NJ 08093
Phone: 856-853-3161

Erna Beal
Naval Research Program
Fuels Section, Code 6181
Washington, DC 20375

Phone: 202-767-2673

Geri Deputy
ITS / Caleb Brett
327 Erickson Ave
Essington, PA 19029
Phone: 610-521-1725

Gretchen Wendtland
Buckeye Pipeline
5002 Buckeye Rd
Emmaus, PA 18049
Phone: 484-232-4548

Michael Palmer
Petrolab Company
874 Albany Shaker Rd
Latham, NY 12110
Phone: 518 424 4833

Andreas Schwarzmann
Grabner Instruments
Dr.-Otto-Neurathgasse 1
1220 Wien
Austria
Phone: +43 1 282 16 27 20 35

Dr. Alfred Ecker
FICHTE Petroleum Institute
Faradaygasse 3, Arsenal Obj. 214
1030 Wien
Austria
Phone: +43 1 79747 6552

Fay Hutto
MISSISSIPPI STATE CHEMICAL LAB
PO DRAWER CR
PETROLEUM PRODS LAB
MISSISSIPPI STATE, MS 39762-5622
Phone: 662-325-2653