

# THE 4th ASCOPE LABORATORY TEST CORRELATION PROGRAMME FOR FUELS BY SAMPLE CODES : A.2/X/82, D.2/Y/82

by  
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## ABSTRACT

*The properties of commercial products are measured by standardized laboratory test methods to check their conformance to specification.*

*Two or more measurements of some property of a specific sample by any given test method usually will not give precisely the same answer.*

*Therefore, Laboratory test correlation of the specific sample is necessary to be done on the expected precision, accuracy of the test results and the uniformity of interpretative, to step on the credibility of the testing laboratory.*

*This is a paper on the conduct and the results of the fourth ASCOPE (ASEAN Council on Petroleum) Correlation Programme for fuels were carried out on two kinds of samples.*

## 1. INTRODUCTION

The first ASCOPE Laboratory Test Correlation Programme for Fuel was launched in 1979, with Indonesia as Coordinator, in this case Laboratory of "Lemigas" Oil and Gas Technology Development Centre. (Since 1984 became "Lemigas" Research and Development Centre for Oil and Gas Technology - Ed.)

The second one was carried out in 1980 and the evaluation of the results was discussed in the first Workshop ASCOPE Lab-Correlation Programme held in Singapore, in 1980.

After the third Programme in 1981, in the Second Workshop was held in Kualalumpur the consensus among participating laboratories proposed to increase the frequency of the correlation programme to twice a year.

This is the first correlation programme for fuels in 1982, i.e. the 4<sup>th</sup> ASCOPE Laboratory Test Programme for Fuels which were carried out on fuels by sample codes A.2/X/82 and D.2/Y/82.

The following tests was used in the correlation programme, fuel by sample code A.2/X/82 Jet fuel type :

- Appearance not for correlation, it for identification only.
- Specific Gravity at 60/60°F, ASTM D 1298
- Flash Point, "ABEL" °F IP 170
- Smoke Point, mm IP 57
- Freezing Point, °C ASTM D 2386
- Distillation (group 4) ASTM D 86

Fuel by sample code D.2/Y/82 Fuel Oil Type :

- Specific Gravity at 60/60°F, ASTM D 1298
- Kinematic Viscosity at 50°C, cSt ASTM D 445
- Pour Point, °F ASTM D 97
- Flash Point PM. CC, °F ASTM D 93
- Calorific Value, Btu/lb ASTM D 240
- Sulfur Content, wt % ASTM D 1551
- Conradson Carbon Residue, wt % ASTM D 189

## II. CORRELATION PROGRAMME EXECUTION

### A. Programme Coordinator

The programme coordinator for this correlation programme is Indonesia, in this case LEMIGAS Laboratory.

The coordinator is responsible for the conduct of the programme, including the preparation of the samples, their expeditions, collections of test results, processing the results, and presenting them in a report.

### B. Country Coordinators

To coordinate the participating laboratories in each member country, a country coordinator was nominated.

The country coordinators are responsible for receiving the samples from the programme coordinator, distributing the samples to participating laboratories, collection of results from participating laboratories, and transmitting them to programme coordinator.

### C. Participating Laboratories

As nominated by the member countries, 14 laboratories participated in this the correlation programme. Included 6 laboratories in Indonesia, 3 in Malaysia, 2 in the Philippines, 1 in Singapore and 2 in Thailand.

To maintain confidentiality of result each participating laboratory was designated with an identification number known only to itself.

### D. Test samples and Instructions

Samples consisting of one gallon each of a jet fuel type and a fuel oil type were prepared by the programme coordinator. These samples were sent to the participating laboratories through the respective country coordinators.

Also included in this shipment is a document, giving detailed instruction to the participating laboratory. It was requested in this instruction that the results be sent to the Programme Coordinator in the forms provided, complete with the laboratory identification number, through the respective country coordinator, within one month after the receipt of the samples.

These samples were sent by air from Jakarta in November 1981.

## III. RESULTS OF THE CORRELATION

### A. Test Results

All data will be displayed in a uniform array, the measured property values of the sample type are to be listed as columns and the participating laboratories in rows :

#### 1. 4<sup>th</sup> Correlation Programme for Fuels

Test results, received from 15 participating laboratories are listed in Table 1 and 2.

These results were processed in statistically following the procedure discribed below.

### B. Statistical Treatment of Test Result

Results from test methods other than those requested in the programme were not included in the calculation of average (see Table 5).

These results are compiled and outliers are rejected after determination of the rejection region and accepted region through rest of hypothesis, and using Grubb's T Factors.

The step are :

#### 1. Average

Average of a set of results is obtained by dividing the sum of all the results by the number of the results.

$$\bar{X} = \frac{1}{n} \sum_{i=1}^n x_i \quad X_1, X_2, X_n \text{ results}$$

n, number of results.

## 2. True Value

True value is the value towards which the average of the results obtained in many laboratories tends.

## 3. Deviation :

Deviation is a constant or systematic error frequently present in test work. It differs from random error and manifests itself as a persistent positive or negative deviation from the known or true value.

$$D_i = X_i - \bar{X}$$

## 4. Variance (unbiased estimate of population variance)

Variance is a measure of the dispersion of a set of accepted results around their average. It is equal to the sum of the squares of the deviation of each result from the average.

$$S^2 = \frac{1}{n-1} \sum_{i=1}^n (X_i - \bar{X})^2$$

## 5. Standard deviation

Standard deviation is a measure of a dispersion of a set of accepted results around their average, equal to the square root of the variance.

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (X_i - \bar{X})^2}$$

6. "T" factor according to Grubb's rejection criteria is found from Grubb's table which lists T against at 99% confidence level.

## 7. Outlier

Outlier is a result far enough in magnitude from other results so as to be considered not a part of a set.

Rejection of outliers if,

$$[X_i - \bar{X}] > S X T, \text{ measurement } (X_i) \text{ is rejected}$$

## 8. Accepted measurement :

$$[X_i - \bar{X}] \leq S X T,$$

measurement ( $X_i$ ) is accepted as belonging to the population.

All test values that fall within plus or minus two standard deviations of the group are considered statistically equal, precision-wise. Values which exceed the two standard deviations but are within acceptable limits on the basis of Grubb's Criterion for 99 percent probability are included in the computation. Rejected values are not included in computation.

Table 1  
Laboratory Result of 4<sup>th</sup> Correlation Programme on Fuels  
Sample Code : A.2/X/82 (Jet Fuel Type)

Determination	Methods	Lab. No. 01		Lab. No. 02		Lab. No. 03		Lab. No. 04		Lab. No. 05		Lab. No. 06		Lab. No. 07	
		Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2
Appearance		Conforms	Conforms	Conforms	Conforms	Clear	Clear	Clear	Clear	Clear	Clear	-	-	Clear	Clear
Specific Gravity at 60/60°F	°F ASTM D 1298	0.7918	0.7923	0.7908	0.7908	0.7911	0.7912	0.7909	0.7909	0.7913	0.7911	0.7902	0.7904	0.7908	0.7906
Flash Point "Abel"	°F IP 170	109	109	109	109	108	107	107	107	110	110	110	112	108	107
Smoke	mm IP 57	20	19	22	22	25	24	26	26	23.0	24.0	21	21	22	23
Freezing Point	°C ASTM D 2386	-	-	-34	-34	-37	-37	-36.0	-36.0	-35.5	-35.5	-36	-36	-34	-34
Distillation :	ASTM D 86														
Initial Boiling Point,	°C (group) 4)	140	150	154	154	145	143	150.0	151.0	153	153	150	151	153	150
20 % recovered at	-	172	173	173	173	169	170	171.0	171.0	169.5	170	170	171	173	172
50 % recovered at	-	187	189	190	190	186	189	186.0	187.0	187	187.5	186	186	190	190
90 % recovered at	-	223	225	227	226	224	225	221.0	222.0	223	223	224	224	227	227
Recovered at 200°C	vol-%	69	68	63.0	63.5	67	66	70.0	70.0	68.0	67.5	67.0	67.5	65	64
Final boiling Point	°C	294	293	248	247	246	249	249.0	250.0	250	246.5	247	248	253	252
Residue	vol-%	1.0	1.0	1.5	1.5	1.5	1.5	0.5	0.5	1.0	1.5	1.0	1.0	1.5	1.5
Low	vol-%	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1.0	0.5	1.0	1.0	1.5	0.5

Table 1 (Continued)

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Average
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	
		3) Conforms	3) Conforms	Bright n Clear	Bright n Clear	Clear	Clear			Bright/ Clear	Bright/ Clear			Clear n bright	Clear n bright	Clear	Clear	
		0.7913	0.7914	0.7910	0.7910	0.7901	0.7901			0.7886	0.7905	0.7919	0.7919	0.7913	0.7909	0.7918	0.7914	0.7910
		106	108	109	111	108	108			2) 110	2) 114	106	106	1) 109	1) 109	2) 112	2) 112	109
		23	23	28	28	25	24			24	26	25	26	25.0	25.0	26	26	24.0
		-53	-54	-54.0	-55.0	-55	-54			-38	-38	-56.5	-57.0	-53.5	-53.5	-56.0	-56.0	-55.5
		152	150	152	152	157	157			140	144	152.0	152.0	155.0	156.5	150.0	153.5	151.0
		172	170	170	169	175	176			173	170	171.5	172.0	173.0	172.5	173.0	173.0	171.5
		189	188	186	186	193	192			187	185	187.5	187.5	189.5	190.0	189.0	189.5	188.0
		228	226	224	223	233	232			225	221	224.0	225.0	226.5	227.5	228.0	228.0	225.5
		64.5	66	66	69	60.0	59.0			70	71	67.5	68.0	64.5	64.5	65.0	65.0	66.5
		255	254	265	248	255	253			249	245	253.5	254.0	251.0	251.0	256.0	255.5	250.5
		1.4	1.5	1.0	1.0	1.3	1.3			1.0	0.5	1.0	0.5	1.0	1.0	0.5	0.5	1.0
		0.6	0.5	NE	NE	0.5	0.5			1.0	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

Note : 1). Converted from deg. C

2). Flash point by Tag Closed Tester, ASTM D.56

3). Visually Clear, bright and free from solid matter and undissolved water at normal ambient temperature

Table 2  
Laboratory Result of 4<sup>th</sup> Correlation Programme on Fuels  
Sample Code : D.2/X/82 ( Fuel Oil Type )

Determination	Methods	Lab. No. 01		Lab. No. 02		Lab. No. 03		Lab. No. 04		Lab. No. 05		Lab. No. 06		Lab. No. 07	
		Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2
Specific Gravity at 60/60°F	ASTM D 1298	0.9146	0.9153	0.9132	0.9132	0.9137	0.9128	0.9141	0.9141	0.9146	0.9145	0.9096	0.9100	0.9129	0.9100
Kinematic Viscosity at 50°C	dlr ASTM D 445	18.80	18.91	18.336	18.405	19.13	19.32	18.518	18.463	19.04	18.92	17.8	17.9	18.79	18.80
Pour Point	°F ASTM D 97	50	45	45	45	32	32	45	45	45	40	50	50	40	40
Flash Point FM CC	°F ASTM D 93	230	230	235	235	241	241	226	228	230	235	234	239	240	239
Calorific value Gross	Btu/lb ASTM D 240	-	-	1) 19,188	1) 19,188	-	-	18,999	18,600	19,150	19,150	-	-	18,891	18.8
Sulfur Content	wt-% ASTM D 1551	1.31	1.35	1.90	1.90	1.85	1.84	6) 2.31	6) 2.34	1.98	1.89	1.8	1.7	1.73	1.68
Conradson Carbon Residue	wt-% ASTM D 189	4.4	4.3	4.37	4.38	4.76	4.79	4.45	4.47	4.99	4.83	5.4	5.3	4.32	4.17

Table 2 (Continued)

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Average		
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2			
		0.9130	0.9129	0.914	0.913	0.9129	0.9135			10)	10)	0.9125	0.9129	0.9120	0.9130	0.9129	0.9128	0.9141	0.9141	0.9133
		18.69	18.46	18.5	19.0	18.61	18.70			17.3	17.8	18.83	18.89	18.99	18.69	19.03	19.225	18.63		
		40	35	35	35	40	40			40	40	50	50	5) 43	5) 37	40	45	42		
		235	230	234	232	224	226			225	222	228	230	5) 237	5) 232	231	234	230		
		-	-	-	-	18,865	18,895			19,050	19,100	2) -	2) -	-	-	7) -	7) -	18,954		
		1.64	1.60	8) 2.1*	8) 2.0*	2.00	2.10			2.10	2.08	3) 2.19*	3) 2.26*	4) 2.16*	4) 2.18*	6) 2.58*	6) 2.51*	1.80		
		4.48	4.25	5) 4.4*	5) 4.8*	4.94	4.90			5.3	5.2	4.7	5.0	5.0	4.9	4.55	4.84	4.73		

Note : 1). Calculated figure, reference Table 7 – Petroleum Product Handbook

2). No equipment

3). Sulfur content determined using Flask Combustion Method (IP.242)

4). Method IP.336

5). Converted from deg. C

6). ASTM D.129

7). Apparatus is out of order

8). Tested by telsec x-ray fluorescence

9). Converted from ramsbottom carbon residue

10). Reported API Gravity at 60/60°F

\*). Not used in average

Table 1 ( Continued )

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Average
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	
		3) Conforms	3) Conforms	Bright & Clear	Bright & Clear	Clear	Clear			Bright/ Clear	Bright/ Clear			Clear & bright	Clear & bright	Clear	Clear	
		0.7913	0.7914	0.7910	0.7910	0.7901	0.7901			0.7896	0.7905	0.7919	0.7919	0.7913	0.7909	0.7918	0.7914	0.7910
		108	108	109	111	108	108			2) 110	2) 114	106	106	1) 109	1) 109	2) 112	2) 112	109
		23	23	28	28	25	24			24	26	25	26	23.0	23.0	26	26	24.0
		-53	-54	-54.0	-55.0	-55	-54			-58	-58	-56.5	-57.0	-53.5	-53.5	-56.0	-56.0	-55.5
		152	150	152	152	157	157			140	144	152.0	152.0	155.0	156.5	150.0	151.5	151.0
		172	170	170	169	175	176			173	170	171.5	172.0	173.0	172.5	173.0	173.0	171.5
		189	188	186	186	193	192			185	185	187.5	187.5	189.5	190.0	189.0	189.5	188.0
		228	226	224	223	223	222			223	221	224.0	225.0	226.5	227.5	228.0	228.0	225.5
		64.3	66	68	69	80.0	59.0			70	71	67.5	68.0	64.5	64.5	65.0	65.0	66.5
		255	254	245	248	251	253			249	245	253.5	254.0	251.0	251.0	256.0	255.5	250.5
		1.4	1.5	1.0	1.0	1.5	1.5			1.0	0.5	1.0	0.5	1.0	1.0	0.5	0.5	1.0
		0.6	0.5	Nil	Nil	0.5	0.5			1.0	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

- Note : 1). Converted from deg. C  
 2). Flash point by Tag Closed Tester, ASTM D.56  
 3). Visually Clear, bright and free from solid matter and undissolved water at normal ambient temperature

Table 2  
 Laboratory Result of 4<sup>th</sup> Correlation Programme on Fuels  
 Sample Code : D.2/X/82 ( Fuel Oil Type )

Designation	Methods	Lab. No. 01		Lab. No. 02		Lab. No. 03		Lab. No. 04		Lab. No. 05		Lab. No. 06		Lab. No. 07	
		Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2
Specific Gravity at 60/60°F	ASTM D 1298	0.9146	0.9153	0.9122	0.9132	0.9127	0.9128	0.9143	0.9141	0.9146	0.9145	0.9096	0.9100	0.9129	0.9100
Kinematic Viscosity at 30°C	dm ASTM D 445	18.60	18.91	18.236	18.425	18.13	19.12	18.218	18.463	19.04	18.91	17.8	17.9	18.79	18.80
Pour Point	°C ASTM D 97	50	45	45	45	32	32	45	45	40	40	39	50	40	40
Flash Point FM CC	°C ASTM D 93	230	230	235	235	241	241	226	228	230	235	224	220	240	235
Calorific value Gross	Btu/lb ASTM D 240	-	-	19,168	19,168	-	-	18,509	18,600	19,150	19,150	-	-	18,891	18.8
Sulphur Content	wt-% ASTM D 1331	1.31	1.35	1.90	1.90	1.83	1.84	6) 2.51*)	6) 2.54*)	1.98	1.89	1.8	1.7	1.73	1.66
Conradson Carbon Residue	wt-% ASTM D 189	4.4	4.3	4.37	4.38	4.76	4.79	4.45	4.47	4.99	4.83	3.4	5.3	4.22	4.17

Table 2 ( Continued )

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Average
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	
		0.9130	0.9129	0.914	0.913	0.9129	0.9135			100	100	0.9120	0.9120	0.9129	0.9128	0.9141	0.9141	0.9133
		18.49	18.46	18.5	19.0	18.61	18.70			17.3	17.8	18.83	18.89	18.99	18.69	19.01	19.225	18.63
		40	35	35	35	40	40			40	40	50	50	5) 43	5) 37	40	45	42
		235	230	214	212	224	226			225	222	228	230	5) 237	5) 232	233	234	230
		-	-	-	-	18,865	18,895			19,050	19,100	2) -	2) -	-	-	7) -	7) -	18,954
		1.44	1.60	8) 2.1*)	8) 2.0*)	2.00	2.10			2.10	2.06	3) 2.19*)	3) 2.26*)	4) 2.16*)	4) 2.18*)	6) 2.58*)	6) 2.51*)	1.80
		4.48	4.25	9) 4.4*)	9) 4.8*)	4.94	4.90			5.3	5.2	4.7	5.0	5.0	4.9	4.55	4.84	4.73

- Note : 1). Calculated figure, reference Table 7 – Petroleum Product Handbook  
 2). No equipment  
 3). Sulphur content determined using Flask Combustion Method (IP.242)  
 4). Method IP.336  
 5). Converted from deg. C  
 6). ASTM D.129  
 7). Apparatus is out of order  
 8). Tested by telsec x-ray fluorescence  
 9). Converted from ramsbottom carbon residue  
 10). Reported API Gravity at 60/60°F  
 \*) . Not used in average

## IV. EVALUATION

### A. 4<sup>th</sup> Correlation Programme for Fuels

The results of the computation processing of this programme is shown in table 3 and 4 Deviation of each result from the average values is shown in table 5 and 6, the following evaluation as below.

#### 1. Concerning to the sample code A.2/X/82, jet fuel type

##### a. Specific Gravity at 60/60°F

9(nine) test results exceed standard deviation viz the results coming from Participating Lab. 0.01, 06, 11, 13, 14 and 16, which are however not be rejected as outlier being still within the permitted limits. Hence, all the list results from all the participating laboratories are satisfactory.

##### b. Flash Point "ABEL"

6 (six) test results exceed standard deviation viz the test results coming from Participating Lab. No. 06, 13 and 16, which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

##### c. Smoke Point

6 (six) test results exceed standard deviation viz the test results coming from Participating Lab. No. 01, 06 and 10, which are however not be rejected as outliers being still within the permitted limits. These results are satisfactory.

##### d. Freezing Point

5(five) test results exceed standard deviation viz the test results coming from Participating Lab No. 09.1, 13 and 15, but are within acceptable limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

##### e. Distillation, IBP

8(eight) test results exceed standard deviation viz the test result coming from Participating Lab. No. 01.1, 03, 11, 13 and 15.2 which however

are still within the permitted limits and are not to be rejected as outliers. Hence the test results are satisfactory.

##### f. Distillation, 20 pct rec.

5(five) test results exceed standard deviation viz the test results coming from Participating Lab No. 03.1, 05.1, 10.2 and 11, which are however not be rejected as outliers being still within the permitted limits. These results are satisfactory.

##### g. Distillation, 50 pct rec.

Only 3 (three) test results exceed standard deviation viz test results coming from Participating Lab. No. 11 and 13.2 which however are still within the permitted limits and are not to be rejected as outliers. Hence the test results are satisfactory.

##### h. Distillation, 90 pct rec.

5(five) test results exceed standard deviation viz test results coming from Participating Lab. No. 04, 11, 13.2 but are within acceptable limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

##### i. Distillation, FBP

8(eight) test results exceed standard deviation viz test results coming from participating Lab. No. 03.1, 05.2, 09.1, 10.1, 11.1, 13.2 and 16 which however are still not be rejected as outliers being still within the permitted limit. Hence, the test results are satisfactory.

##### j. Distillation, rec at 200 deg. C

7(seven) test results exceed standard deviation viz test results coming from participating Lab. No. 02.1, 04, 11 and 13 which however are still not be rejected as outliers being still within the permitted limit. Hence, the test results are satisfactory.

Table 3  
Test Results of 4<sup>th</sup> Correlation Programme on Fuels  
Sample Code : A.2/X/82 ( Jet Fuel )

NO. DESCRIPTION	MEAN AVERAGE DEVIATION	STANDARD DEVIATION	T FAC	CRITICAL STANDARD DEVIATION	99 PCT CONFIDENCE LOW LIM	INTERVAL UP. LIM	CRITICAL VALUE		NO. OF RES
							LOWER LIM	UPPER LIM	
1. SPECIFIC GRAVITY AT 60/60 DEG F	0.7910	0.00062	3.070	0.00190	0.7907	0.7913	0.7891	0.7929	28
2. FLASH POINT "ABEC" DEG F	109.0000	1.92450	3.070	5.90822	107.9926	110.0074	103.0918	114.9082	28
3. SMOKE POINT P.P	24.0000	2.24433	3.070	6.89011	22.8251	25.1749	17.1059	30.8901	28
4. FREEZING POINT DEG C	-55.3269	1.43407	3.030	4.34765	-56.1120	-54.5418	-59.6746	-50.9793	26
5. DISTILLATION, IBP DEG C	150.9643	4.00677	3.070	12.30077	148.8668	153.0617	138.6635	163.2050	28
6. DISTILLATION, 20 PCT REC AT DEG C	171.6964	1.74451	3.070	5.35564	170.7832	172.6096	166.3408	177.0521	28
7. DISTILLATION, 50 PCT REC AT DEG C	188.1964	1.98298	3.070	6.08774	187.1584	189.2345	182.1087	194.2842	28
8. DISTILLATION, 90 PCT REC AT DEG C	225.3571	2.88629	3.070	8.46091	223.8462	226.8680	216.4962	234.2180	28
9. DIST. REC AT 200 DEG C (VOL PCT)	66.3393	2.89653	3.070	8.89234	64.8230	67.8555	57.4469	75.2316	28
10. DISTILLATION, FBP (DEC C)	250.6250	3.38741	3.070	10.39934	248.8517	252.3982	240.2256	261.0242	28
11. DISTILLATION, RESIDUE (VOL PCT)	1.0857	0.38269	3.070	1.17487	0.8854	1.2860	-0.0892	2.2006	28
12. DISTILLATION, LOSS (VOL PCT)	0.5393	0.23308	3.070	0.71555	0.4173	0.6613	-0.1763	1.2548	28

Table 4  
Test Results of 4<sup>th</sup> Correlation Programme on Fuels  
Sample Code : D.2/X/82 ( Fuel Oil )

NO. DESCRIPTION	MEAN AVERAGE DEVIATION	STANDARD DEVIATION	T FAC	CRITICAL STANDARD DEVIATION	99 PCT CONFIDENCE LOW LIM	INTERVAL UP. LIM	CRITICAL VALUE		NO. OF RES
							LOWER LIM	UPPER LIM	
1. SPECIFIC GRAVITY AT 60/60 DEG F	0.9133	0.00122	3.070	0.00374	0.9127	0.9140	0.9096	0.9171	28
2. KIN C VISCOSITY AT 122 DEG T (CST)	18.6262	0.45955	3.070	1.41080	18.3857	18.8668	17.2154	20.0370	28
3. FOUR POINT, (DEG F)	41.9286	5.41553	3.070	16.62566	39.0936	44.7635	25.3029	58.2542	28
4. FLASH POINT FMCC (DEG F)	229.6429	7.23782	3.070	22.22011	225.8540	233.4317	207.4227	251.8630	28
5. CALORIFIC VALUE GROSS, (BTU/LB)	18.9537	0.22173	2.530	00.56540	18.7546	19.1527	18.3082	19.5191	12
6. SULPHUR CONTENT, (WT PCT)	1.8017	0.22994	2.820	0.64844	1.6445	1.9588	1.1532	2.4501	18
7. CONRADSON CARBON RESIDUE (WT PCT)	4.7265	0.36072	3.030	1.09298	4.5292	4.9239	3.6335	5.8195	26

Table 5  
Deviation from Average  
Sample Code : A.2/X/82 ( Jet Fuel Type )

Determination	Methods	Lab. No. 01		Lab. No. 02		Lab. No. 03		Lab. No. 04		Lab. No. 05		Lab. No. 06		Lab. No. 07	
		Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2
Appearance															
Specific Gravity at 60/60 <sup>o</sup> F	ASTM D 1298	+0.0008	+0.0013	-0.0002	-0.0002	+0.0001	+0.0002	-0.0001	-0.0001	+0.0003	+0.0001	-0.0008	-0.0006	-0.0002	-0.0004
Flash Point "Abec"	IP 110	0	0	0	0	- 1	- 2	- 2	- 2	+ 1	+ 1	+ 1	- 3	- 1	- 2
Smoke Point	IP 57	- 4	- 5	- 2	- 2	+ 1	0	+ 2	+ 2	- 1	0	- 3	- 3	- 2	- 1
Freezing Point	<sup>o</sup> C ASTM D 2386	-	-	+ 1.5	+ 1.5	- 1.5	- 1.5	- 0.5	- 0.5	0	0	- 0.5	- 0.5	+ 1.5	+ 1.5
Distillation :		ASTM D 86													
Initial Boiling Point,	<sup>o</sup> C (group 4)	-11.0	- 1.0	+ 3.0	+ 3.0	- 6.0	- 6.0	- 1.0	0	+ 2.0	+ 2.0	- 1.0	0	+ 2.0	- 1.0
20 % recovered at	"	+ 0.5	+ 1.5	+ 1.5	+ 1.5	- 2.5	- 1.5	- 0.5	- 0.5	- 2.0	- 1.5	- 1.5	- 0.5	+ 1.5	+ 0.5
50 % recovered at	"	- 1.0	+ 1.0	+ 2.0	+ 2.0	- 2.0	+ 1.0	- 2.0	- 1.0	- 1.0	- 0.5	- 2.0	- 2.0	+ 2.0	+ 2.0
90 % recovered at	"	- 2.5	- 0.5	+ 1.5	+ 0.5	- 1.5	- 0.5	- 4.5	- 3.5	- 2.5	- 2.5	- 1.5	- 1.5	+ 1.5	+ 1.5
Recovered at 200 <sup>o</sup> C	vol-%	+ 2.5	+ 1.5	- 3.5	- 3.0	+ 0.5	- 0.5	+ 3.5	+ 3.5	+ 1.5	+ 1.0	+ 0.5	+ 1.0	- 1.5	- 2.5
Final Boiling Pt Point	<sup>o</sup> C	+ 3.5	+ 2.5	- 2.5	- 3.5	- 4.5	- 1.5	- 1.5	- 0.5	- 0.5	- 4.0	- 3.5	- 2.5	+ 2.5	+ 1.5
Residue	vol-%	0	0	+ 0.5	+ 0.5	+ 0.5	+ 0.5	- 0.5	- 0.5	0	+ 0.5	0	0	+ 0.5	+ 0.5
Loss	vol-%	0	0	0	0	0	0	0	0	+ 0.5	0	+ 0.5	+ 0.5	0	0

Table 5 ( Continued )

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Grubb's Limit
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	
		+0.0003	+0.0004	0	0	-0.0009	-0.0009			-0.0014	-0.0005	+0.0009	+0.0009	+0.0003	-0.0001	+0.0008	+0.0004	0.0019
		-1	-1	0	+2	-1	-1			+1	+5	-3	-3	0	0	+3	+3	6
		-1	-1	+4	+4	+1	0			0	+2	+1	+2	+1	+1	+2	+2	7
		+2.5	+1.5	+1.5	+0.5	+0.5	+1.5			-2.5	-2.5	-1.0	-1.5	+2.0	+2.0	-0.5	-0.5	4.5
		+1.0	-1.0	+1.0	+1.0	+6.0	+6.0			-11.0	-7.0	+1.0	+1.0	+4.0	+5.5	-1.0	+0.5	12.5
		+0.5	-1.5	-1.5	-2.5	+3.5	+4.5			+1.5	-1.5	0	+0.5	+1.5	+1.0	+1.5	+1.5	5.5
		+1.0	0	-2.0	-2.0	+5.0	+4.0			-1.0	-3.0	-0.5	-0.5	+1.5	+2.0	+1.0	+1.5	6.0
		+2.5	+0.5	-1.5	-2.5	+7.5	+6.5			-2.5	-4.5	-1.5	-0.5	+1.0	+2.0	+2.5	+2.5	9.0
		-2.0	-0.5	+1.5	+2.5	-6.5	-7.5			+3.5	+4.5	+1.0	+1.5	-2.0	-2.0	-1.5	-1.5	9.0
		+4.5	+3.5	-5.5	-2.5	+4.5	+2.5			-1.5	-5.5	+3.0	+3.5	+0.5	+0.5	+5.5	+5.0	10.5
		+0.4	+0.5	0	0	+0.5	+0.5			0	-0.5	0	-0.5	0	0	-0.5	-0.5	1.0
		+0.1	0	-0.5	-0.5	0	0			+0.5	0	0	0	0	0	0	0	0.5

Table 6  
Deviation from Average  
Sample Code : D.2/X/82 ( Fuel Oil Type )

Determination	Method	Lab. No. 01		Lab. No. 02		Lab. No. 03		Lab. No. 04		Lab. No. 05		Lab. No. 06		Lab. No. 07	
		Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2
Specific Gravity at 60/60°F	ASTM D 1298	+0.0013	+0.0020	-0.0001	-0.0001	+0.0004	+0.0005	+0.0008	+0.0008	+0.0013	+0.0013	-0.0007	-0.0005	+0.0006	+0.0007
Kinematic Viscosity at 50°C, cSt	ASTM D 445	-0.03	+0.28	-0.29	-0.20	+0.59	+0.49	-0.31	-0.17	+0.41	+0.39	-0.83	-0.73	+0.16	+0.13
Pour Point °F	ASTM D 97	+8	+3	+3	+3	-10	-10	+3	+3	+3	-2	+8	+8	-2	-2
Flash Point FM CC °F	ASTM D 93	0	0	+5	+5	+11	+11	-4	-2	0	+5	-6	-10	+10	+5
Calorific value Gross Btu/lb	ASTM D 140	-	-	+214	+214	-	-	-445	-354	+196	+196	-	-	-63	-56
Sulphur Content wt-%	ASTM D 1551	-0.49	-0.45	+0.10	+0.10	+0.03	+0.04	+0.71	+0.74	+0.18	+0.39	0	-0.30	-0.07	-0.12
Conradson Carbon Residue wt-%	ASTM D 189	-0.33	-0.40	-0.36	-0.35	+0.03	+0.06	-0.36	-0.26	+0.26	+0.10	+0.47	+0.57	-0.51	-0.56

Table 6 ( Continued )

Lab. No. 08		Lab. No. 09		Lab. No. 10		Lab. No. 11		Lab. No. 12		Lab. No. 13		Lab. No. 14		Lab. No. 15		Lab. No. 16		Grubb's Limit
Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	Op. 1	Op. 2	
		-0.0003	-0.0004	+0.0007	-0.0003	-0.0004	+0.0002			-0.0010	-0.0004	-0.0003	-0.0003	-0.0004	-0.0005	+0.0008	+0.0008	0.0037
		+0.06	-0.17	-0.13	+0.31	-0.02	+0.07			-1.35	-0.83	+0.20	+0.26	+0.36	+0.06	+0.41	+0.60	1.41
		-2	-7	-7	-7	-2	-2			-2	-2	+8	+8	+1	-5	-2	+3	17
		+5	0	-16	-18	-6	-4			-5	-8	-2	0	+7	+2	+1	+4	22
		-	-	-	-	-89	-59			+96	+146	-	-	-	-	-	-	565
		-0.16	-0.20	+0.30	+0.20	+0.20	+0.30			+0.30	+0.28	+0.39	+0.46	+0.28	+0.38	+0.78	+0.71	0.65
		-0.25	-0.38	+1.67	+2.07	+0.21	+0.17			+0.57	+0.47	-0.03	+0.27	+0.27	+0.17	-0.38	+0.11	1.09

## 2. Concerning to the sample code D.2/X/82, Fuel oil type

### a. Specific Gravity at 60/60°F

5(five) test results exceed standard deviation viz test results coming from participating Lab. No. 01, 05, 1, 06 which however are still not be rejected as outliers being still within the permitted limit.

Hence, the test results are satisfactory.

### b. Kinematic Viscosity at 50 deg. F

7(seven) test results exceed standard deviation viz test results coming from participating Lab. No. 03, 06, 13 and 16.2 which however are still not be rejected as outliers being still within the permitted limit. Hence, the test results are satisfactory.



### c. Flash Point, PM CC.

7(seven) test results exceed standard deviation viz the test results coming from participating Lab. No. 03, 06.2, 07.1, 10, 13.2 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### d. Pour Point

10(ten) test results exceed standard deviation viz the test results coming from participating Lab. No. 01.1, 03, 06, 09.2, 10 and 14 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### e. Calorific Value, Gross

Test results, received from 6 participating laboratories only, 2 (two) test values exceed standard deviation viz the test results coming from participating Lab. No. 04 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### f. Sulphur Content

9(nine) Participating Laboratories determined sulphur content by method ASTM D. 1551. 5(five) test results exceed standard deviation viz test participating Lab. No. 01, 11.2 and 13 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

In this case 10 (ten) test results coming from Participating Lab. No. 04, 10, 14, 15 and 16 determined sulphur content not used the method as prescribed. The position of 5(five) values exceed standard deviation and 4(four) values are rejected.

### g. Conradson Carbon Residue

8(eight) test results exceed standard deviation viz test results coming from Participating Lab No. 01.2, 06, 07, 09.2 and 13 which however are still within the permitted limits and are not to be rejected as outliers.

In this case 2(two) test results coming from Participating Lab No. 10 are rejected.

## V. CONCLUSION

Conducting to the test data submitted by all participating laboratories and the evaluation result, the following conclusion can be drawn.

### A. In general the test results are satisfactory

The wide disparities in test results are observed in, Flash point PM CC, IBP and FBP distillation, Kinematic Viscosity, Sulphur Content and Calorific value.

### B. About sulphur content, the high results obtained by Bomb Method, such as the results of the Participating Lab No. 04 and 16

It must be noted that ASTM D 129 (Bomb Method) indicated that this method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead.

### C. Test results show that a number of participating laboratories still have not fully adhered to the reporting instruction described by the ASTM test methods

These are evident in the results for : Kinematic Viscosity wherein the result should be rounded to the nearest one part per thousand of the value measured.

There are Participating Lab No. 02, 04, 06, 10, 13, 16 in the 4th Correlation Programme.

## VI. SUMMARY

### The 4<sup>th</sup> Correlation Programme on Fuels

No of participant	
Indonesia	6
Malaysia	3
Philippines	2
Singapore	1
Thailand	2
Total :	14
No of sample	2
No of test correlated	12
No of results	458
Results with different method	12 (2.6%)
No of results exceed standard deviation	104 (23.3%)
No of results are to be rejected	2 (0.45%)

### c. Flash Point, PM CC.

7(seven) test results exceed standard deviation viz the test results coming from participating Lab. No. 03, 06.2, 07.1, 10, 13.2 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### d. Pour Point

10(ten) test results exceed standard deviation viz the test results coming from participating Lab. No. 01.1, 03, 06, 09.2, 10 and 14 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### e. Calorific Value, Gross

Test results, received from 6 participating laboratories only, 2 (two) test values exceed standard deviation viz the test results coming from participating Lab. No. 04 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

### f. Sulphur Content

9(nine) Participating Laboratories determined sulphur content by method ASTM D. 1551. 5(five) test results exceed standard deviation viz test participating Lab. No. 01, 11.2 and 13 which however are still within the permitted limits and are not to be rejected as outliers. Hence, the test results are satisfactory.

In this case 10 (ten) test results coming from Participating Lab. No. 04, 10, 14, 15 and 16 determined sulphur content not used the method as prescribed. The position of 5(five) values exceed standard deviation and 4(four) values are rejected.

### g. Conradson Carbon Residue

8(eight) test results exceed standard deviation viz test results coming from Participating Lab No. 01.2, 06, 07, 09.2 and 13 which however are still within the permitted limits and are not to be rejected as outliers.

In this case 2(two) test results coming from Participating Lab No. 10 are rejected.

## V. CONCLUSION

Conducting to the test data submitted by all participating laboratories and the evaluation result, the following conclusion can be drawn.

### A. In general the test results are satisfactory

The wide disparities in test results are observed in, Flash point PM CC, IBP and FBP distillation, Kinematic Viscosity, Sulphur Content and Calorific value.

### B. About sulphur content, the high results obtained by Bomb Method, such as the results of the Participating Lab No. 04 and 16

It must be noted that ASTM D 129 (Bomb Method) indicated that this method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead.

### C. Test results show that a number of participating laboratories still have not fully adhered to the reporting instruction described by the ASTM test methods

These are evident in the results for : Kinematic Viscosity wherein the result should be rounded to the nearest one part per thousand of the value measured.

There are Participating Lab No. 02, 04, 06, 10, 13, 16 in the 4th Correlation Programme.

## VI. SUMMARY

### The 4<sup>th</sup> Correlation Programme on Fuels

No of participant	
Indonesia	6
Malaysia	3
Philippines	2
Singapore	1
Thailand	2
Total :	14
No of sample	2
No of test correlated	12
No of results	458
Results with different method	12 (2.6%)
No of results exceed standard deviation	104 (23.3%)
No of results are to be rejected	2 (0.45%)

## REFERENCES

- ASTM STANDARDS, 1981 – *Petroleum Products and Lubricants volume 05.01*
- ASTM STANDARDS, 1981 – *Petroleum Products and Lubricants volume 05.02*
- ASTM STANDARDS, 1981 – *Petroleum Products and Lubricants volume 05.03*
- Djasrul Anwar, Drs., September 1980 – *Pengertian dan Penggunaan Metoda Statistik dalam Analisa Data*. Temu Karya Korelasi antar Lab. Jakarta.
- IP Standards for Petroleum and Its Products, 1980-Part 1 Methods for Analysis and Testing*
- IP Standards for Petroleum and Its Products, 1980-Part 2 Method for Analysis and Testing*

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### VI. SUMMARY

#### Number of Participants

Indonesia 2  
Malaysia 2  
Philippines 1  
Singapore 1  
Thailand 2  
Total 9

#### Number of samples

Number of samples 2  
Number of test completed 12  
Number of results 1455  
Number of results with different method 12 (2.0%)  
Number of results reported standard deviation 104 (33.3%)  
Number of results not to be reported 2 (0.45%)