



Report of Cooperative Education

Equipment and Procedure Design for Used Oil Analysis

at Sutaiyo Lube Distributor Co., Ltd.

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A Report Submitted in Partial Fulfillment of the Requirements
for the Degree of Bachelor of Engineering (Petrochemical Engineering),
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รายงานสหกิจศึกษาฉบับสมบูรณ์

การออกแบบเครื่องมือและกระบวนการสำหรับการวิเคราะห์น้ำมันใช้แล้ว

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รายงานนี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรวิศวกรรมศาสตรบัณฑิต

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สถาบันเทคโนโลยีพระจอมเกล้าเจ้าคุณทหารลาดกระบัง

ปีการศึกษา 2560

Cooperative Title: Equipment and Procedure Design for Used Oil Analysis at Sutaiyo Lube Distributor Co., Ltd.

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Company: Sutaiyo Lube Distributors Co., Ltd.

Abstract

Equipment and procedure design for used oil analysis at Sutaiyo Lube Distributor Co., Ltd. made for support new project, "Lubefix". They require laboratory room and used oil analysis process. Including the improvement used oil analysis in mobile car for support on customer company site. The used oil testing is analyzed general properties in preliminary for used oil which continue using or changing new oil to systems. All tests are consisting of particulate contamination testing, total acid number testing, water in oil testing viscosity testing and colors testing. These testing are referenced by ASTM which refer on equipment work instruction.

This thesis represents work instruction of oil analysis equipment that show equipment using and how to analyze results. Sample used oil analysis report form for shown the results of testing and recommend which oil state to customer. Before and after installed furniture in laboratory room increase comfortable systematic working process which advantage for lab working personnel. Unfortunately, Sutaiyo do not have enough funding to create lab mobile car. Because a new Sutaiyo's plant is obstructing in Rayong and is requiring more budget. Manager and other staffs are summarized that the new plant is more important and more necessary than lab mobile car. Then, lab mobile car is waiting to build in next year.

Keywords: Oil analysis, Laboratory, Mobile car

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CHAPTER I

INTRODUCTION

1.1 Background

Sutaiyo, a strategic distributor of Mobil lubricants, has been a leader in providing products and services of industrial and automotive lubricants in the region for more than 30 years. To meet customers' growing demands, we offer high-quality lubricants to many diverse industries including energy, metal work, steel, general manufacturing, food and beverage, aviation, textile, marine. In the automotive sector, Sutaiyo offers installation workshop, commercial fleet, and retail outlets. In addition, we have developed a strong customer network and provides a high-level standard of customer service.

Sutaiyo Lube Distributors Co., Ltd. have expanded planning to Rayong, to operate new project "Lubefix" which design instantly used oil analysis but not true operate yet. Desire laboratory with equipment to analyze used oil and the report to customers. In experimental phase, try this project in phrapradaeng. And moving to Rayong when ready to operate in Lubefix project.

1.2 Objectives

- 1.2.1 To determine Thai equipment's procedures
- 1.2.2 To design used oil analysis report
- 1.2.3 To simulate laboratory room for used oil analysis
- 1.2.4 To design used oil analysis mobile car

1.3 Scopes of Work

- 1.3.1 Study general properties of used oil
- 1.3.2 Study equipment which testing properties of used oil
- 1.3.3 Study requirements of laboratory room
- 1.3.4 Study mobile car building process

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1.4 Expected Outputs

1.4.1 Thai work instruction

1.4.2 The used oil analysis report

1.4.3 The laboratory room

1.4.4 The mobile car



CHAPTER II

LITERATURE REVIEW

Laboratory room is oil analysis for help to solve the customer problems. For example, wear problem, NG parts, overheating, etc. Basic test for lubricants, hydraulic oils mostly. Each apparatus is portable equipment which use on-site. After testing, sent the oil analysis report to customer.

2.1 Technical requirements [ISO/IEC 17025:2005]

ISO/IEC 17025:2005 specifies the general requirements for the competence, impartiality and consistent operation of laboratories. And applicable to all organizations performing laboratory activities, regardless of the number of personnel. Laboratory customers, regulatory authorities, organizations and schemes using peer-assessment, accreditation bodies, and others use ISO/IEC 17025:2005 in confirming or recognizing the competence of laboratories.

Technical requirements in laboratory has many factors which determine the correctness and reliability of the tests and/or calibrations performed by a laboratory. These factors include contributions from human factors, accommodation and environmental conditions, test and calibration methods and method validation, equipment, measurement traceability, sampling, the handling of test and calibration items

2.2 Standard Specification for mineral hydraulic oils [ASTM D 6158]

Hydraulic oil types classification including general properties which hydraulic oil should have. In addition, represent requirements table for any types of hydraulic oil.

2.3 Particulate Contamination Test [ASTM D 6217]

A measured volume of about 100 ml of used oil is vacuum filtered through 0.45 μm cellulose membranes. When the level of particulate contamination is high or of a nature that induces slow filtration rates, may be required to complete filtration in a reasonable time. After the filtration has been completed, the membranes are washed with solvent, dried and kept in petrislide.

NAS 1638 and ISO 4406 standard are commonly used to count particle contamination in fluid or oil. Particle Contamination will cause and accelerate wear of

system components. The rate at which damage occurs is dependent on the internal clearance of the components within the system, the size and quantity of particles present in oil and system.

2.4 Wear particle analysis

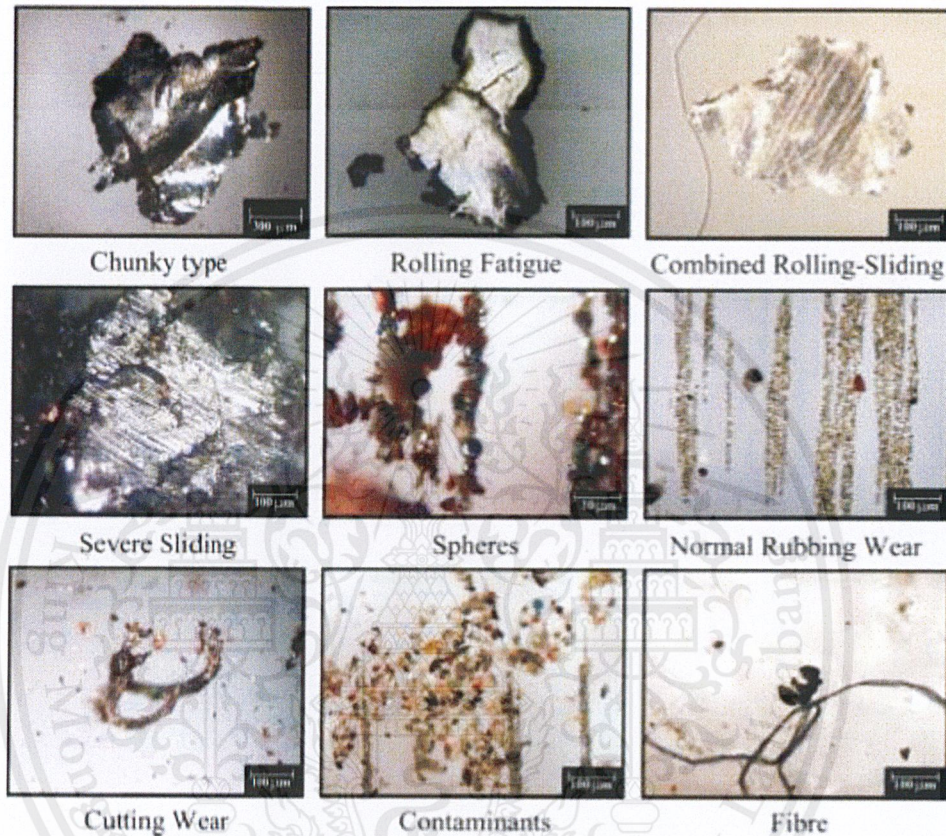


Figure 2.1 types of wear contamination

This picture show particulate which identified to be wear cannot accuracy result of in real laboratory, different power of microscope shown different image resolution.

2.5 Total Acid Number Test [ASTM D 974]

The sample is dissolve in a mixture of toluene and isopropyl alcohol containing small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic acid solution, respectively, to the end point indicated by color change of the added p-naphtholbenzein solution (orange in acid and green-brown in base). To determine the strong acid number, a separate portion of the sample is extracted with hot water and the aqueous extract is titrated with potassium hydroxide solution, using methyl orange as an indicator.

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2.6 Water in oil Test [ASTM D 1744]

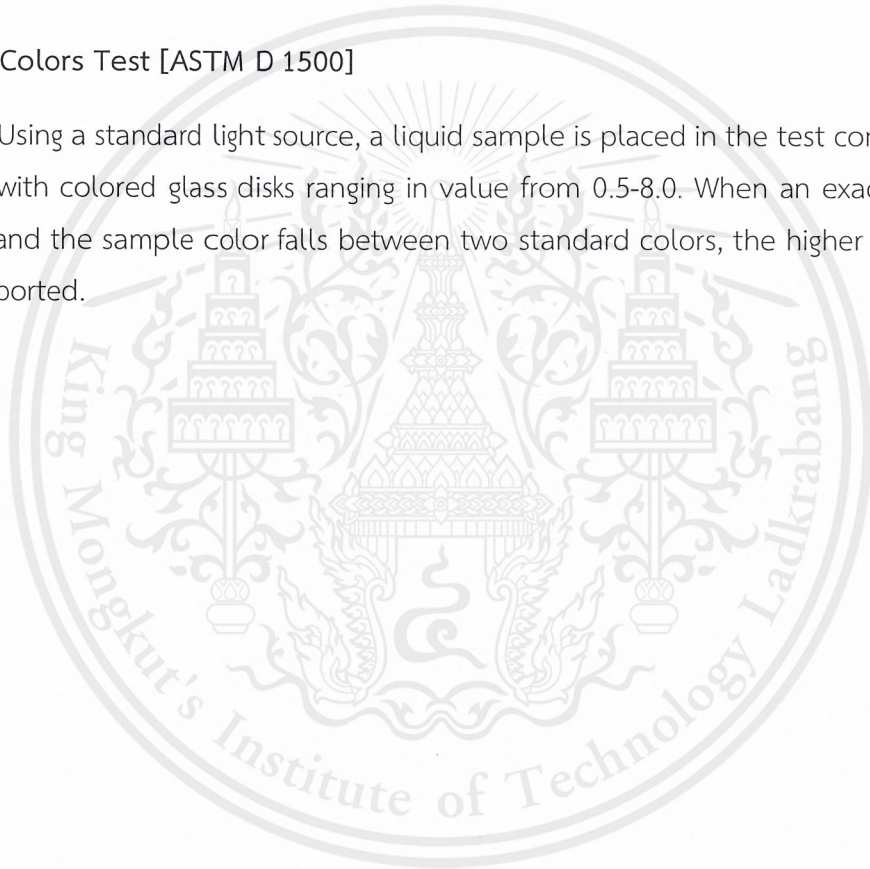
The material to be analyzed is titrated with standard Karl Fischer reagent to an electrometric end point.

2.7 Viscosity Test [ASTM D 445]

The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

2.8 Colors Test [ASTM D 1500]

Using a standard light source, a liquid sample is placed in the test container and compared with colored glass disks ranging in value from 0.5-8.0. When an exact match is not found and the sample color falls between two standard colors, the higher of the two colors is reported.



CHAPTER III

RESEARCH METHODOLOGY

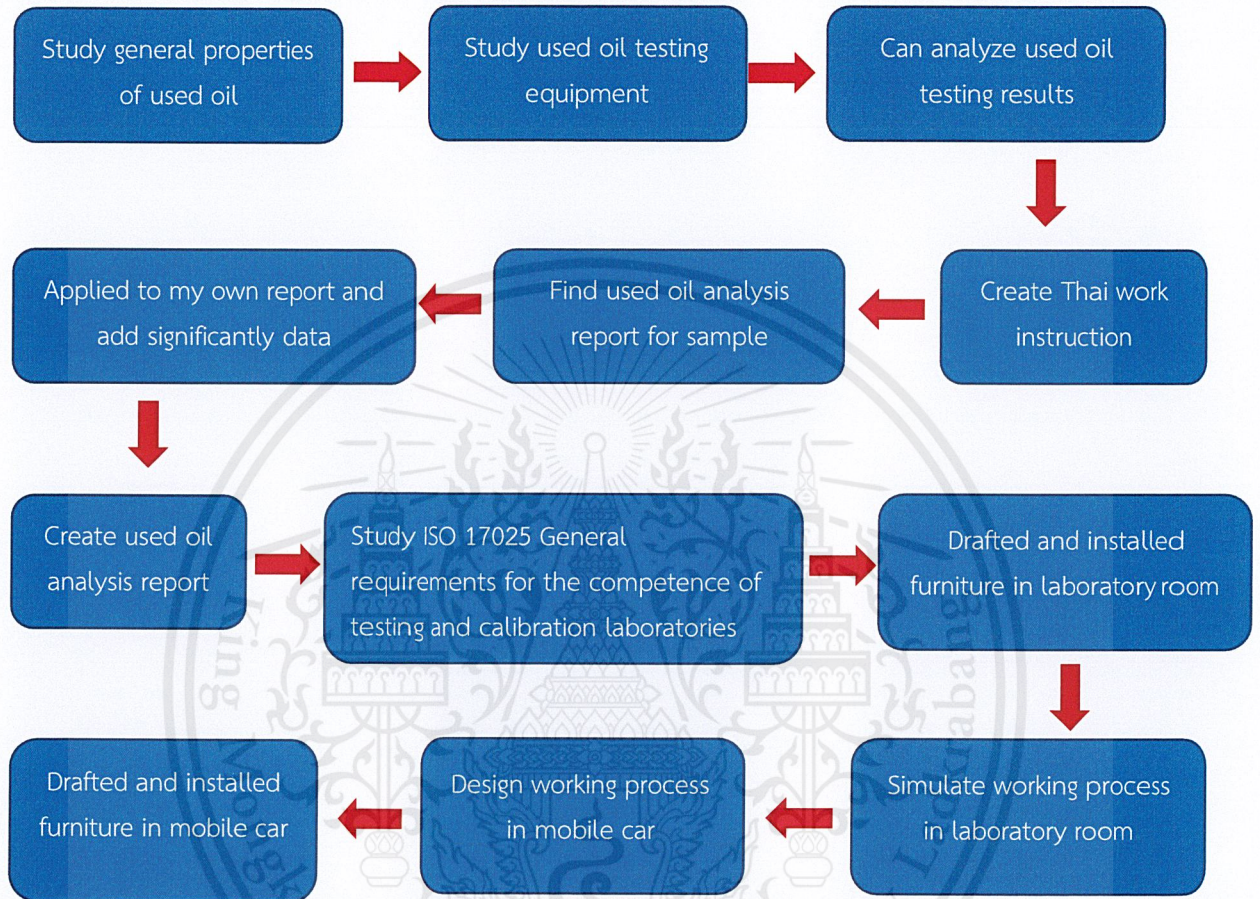


Figure 3.1 Methodology flow chart

3.1 Study General properties of used oil

Used oil has many general properties. For primary analysis which know oil state and instantly solve to customer are:

1. NAS 1638 class or ISO 4406 class
2. Total acid number
3. Water saturated
4. Viscosity
5. Colors

3.2 Study used oil testing equipment

Primary analysis, using equipment as follow:

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1. Filtration set
2. Microscope
3. TAN test kit
4. Water in oil test kit
5. Ball test kit
6. Light source

3.3 Can analyze used oil testing results

Analyze with standard and work instruction as follow:

- ASTM D 445:2005, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
- ASTM D 974:2005, Standard Test Method for Acid and Base Number by Color-Indicator Titration
- ASTM D 1500:2005, Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
- ASTM D 1744:2005, Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent
- ASTM D 6158:2005, Standard Specification for Mineral Hydraulic Oils
- ASTM D 6217:2005, Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration

3.4 Design Thai work instruction

3.4.1 Particulate Contamination Test

- Chemicals: Used oil and Heptane
- Equipment: filtration set (Glass funnel, Anodized aluminum clamp, stainless steel support screen, PTFE gasket, glass support base includes silicone stopper, receiver flask, silicone fixing sucker), cellulose membrane, volumetric cylinder and microscope
- Procedure:
 1. Shake the sample container vigorously for about 30 seconds. Pour oil used 100 mL into volumetric cylinder.

2. Prepare filtration set. Place cellulose membrane in filtration set.

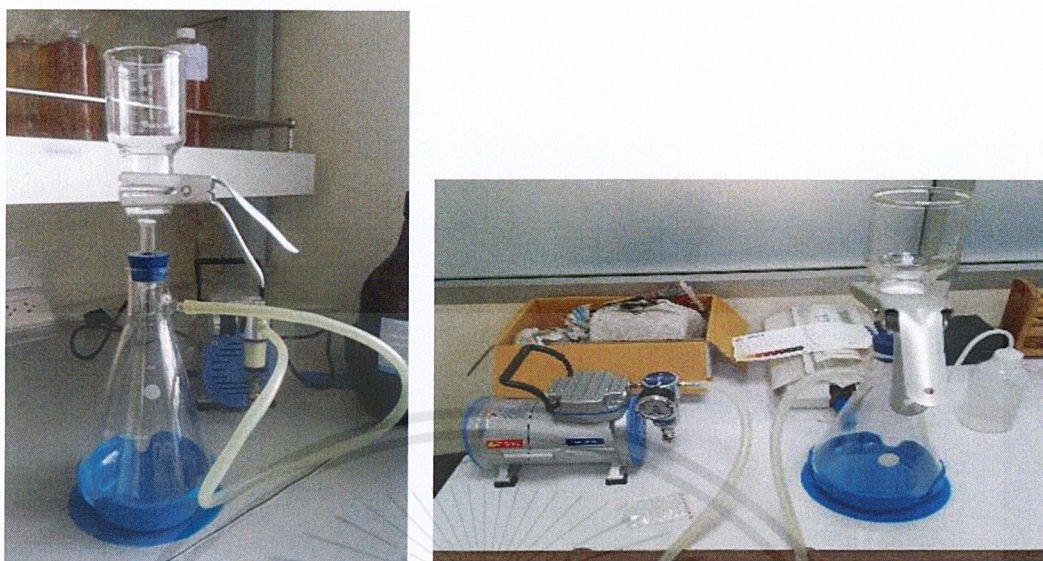


Figure 3.2 Filtration set

3. Start the vacuum, pour used oil from the volumetric cylinder to the glass funnel.

4. Continue transferring 100 mL increments of used oil to the glass funnel. When all the used oil has been filtered, or if filtration slows requires greater than 10 min for complete filtration, then remove the glass funnel from filter set and pour used oil which uncomplete filtration into volumetric cylinder and record the rest used oil volume in mL.

5. If all the used oil has been filtered, thoroughly rinse volumetric cylinder and pour the rinses in glass funnel with heptane.

6. Keep cellulose membrane dry in the petrislide.

7. When it dries, analyze NAS 1638 and ISO 4406 by see though microscope.

- Results analysis

- NAS 1638 specifies five range; 5-15 micron, 15-25 micron, 25-50 micron, 50-100 micron and smaller than 100 micron. Each size range is assigned a class number based on the number of particles present, and the overall class is the range which has the highest count. Such as Count on Size Range 5-15 micron is 50100 particles, NAS 1638 Class : 8, Size Range 15-25 micron is 4500 particles, NAS 1638 Class : 7, Size Range 25-50 micron is 1300 particles, NAS 1638 Class : 8, Size Range 50-100 micron is 100 particles,

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NAS 1638 Class : 7, Size Range smaller than 100 micron is 20 particles , NAS 1638 Class : 7.
So The overall NAS 1638 Class = 8

NAS- Class	Number of Particles						Amount of Contamination (ACFID)
	2 - 5 μm	5 - 15 μm	15 - 25 μm	25 - 50 μm	50 - 100 μm	>100 μm	[mg/l]
00	625	125	22	4	1	0	—
0	1,250	250	44	8	2	0	0.01
1	2,500	500	88	16	3	1	—
2	5,000	1,000	178	32	6	1	—
3	10,000	2,000	356	63	11	2	—
4	20,000	4,000	712	126	22	4	0.1
5	40,000	8,000	1,425	253	45	8	—
6	80,000	16,000	2,850	506	90	16	0.2
7	160,000	32,000	5,700	1,012	180	32	0.5
8	320,000	64,000	11,400	2,025	360	64	1
9	640,000	128,000	22,800	4,050	720	128	3
10	1,280,000	256,000	45,600	8,100	1,440	256	5
11	2,560,000	512,000	91,200	16,200	2,880	512	7 - 10
12	5,120,000	1,024,000	182,400	32,400	5,760	1,024	20
13	—	2,048,000	364,800	64,800	11,520	2,048	40
14	—	4,096,000	729,600	129,600	23,040	4,096	80

Table 3.1 NAS Class

- ISO 4406 specifies three size ranges; size range smaller than 4 micron, size range smaller than 6 micron, size range smaller than 14 micron. The three scale numbers are reported with a backslash between them. Such as ISO: 18/ 16/13 represent particles smaller than 4 micron substitute code 18 (between 1300-2500), particles smaller than 6 micron substitute code 16 (between 320-640) and particles smaller than 14 micron substitute code 13 (between 40-80).

ISO- Class	Number of Particles		ISO- Class	Number of Particles	
	more than	up to & including		more than	up to & including
0	0,5	1	15	16,000	32,000
1	1	2	16	32,000	64,000
2	2	4	17	64,000	130,000
3	4	8	18	130,000	250,000
4	8	6	19	250,000	500,000
5	16	32	20	500,000	1,000,000
6	32	64	21	1,000,000	2,000,000
7	64	130	22	2,000,000	4,000,000
8	130	250	23	4,000,000	8,000,000
9	250	500	24	8,000,000	16,000,000
10	500	1,000	25	16,000,000	32,000,000
11	1,000	2,000	26	32,000,000	64,000,000
12	2,000	4,000	27	64,000,000	130,000,000
13	4,000	8,000	28	130,000,000	250,000,000
14	8,000	16,000			

Table 3.2 ISO Class

ISO/NAS/SAE code comparison table

The comparisons relate to particle count data only. To confirm to any particular standard reference should be made to the recommended experimental procedure.

ISO/DIS 4406 BS 5540/4 codes	Defence Std. 05/42		NAS 1638	SAE 749
	Table A	Table B		
13/11/08			2	
14/12/09			3	0
15/13/10			4	1
16/14/09		400F		
16/14/11			5	2
17/15/09	400			
17/15/10		800F		
17/15/12			6	3
18/16/10	800			
18/16/11		1300F		
18/16/13			7	4
19/17/11	1 300	2000		
19/17/14			8	5
20/18/12	2 000			
20/18/13		4400F		
20/18/15			9	6
21/19/13	4 400	6300F		
21/19/16			10	
22/20/13	6 300			
22/20/17			11	
23/21/14	15 000			
23/21/18			12	
24/22/15	21 000			
25/23/17	100 000			

Figure 3.3 Comparison table between ISO, NAS and SAE

- Wear analysis

Wear in used oil found that equipment or machine have the problem.

3.4.2 Total Acid Number

- Chemicals : Used oil, Reagent D and TAN titrant
- Equipment : volumetric bottle, syringe



Figure 3.4 Total Acid Number testing set

- Procedure

1. Measure 20 mL of reagent D in volumetric bottle.
2. Ensure colors of reagent D which should be green color. If not, add TAN titrant by one drop until green color.

3. Stir used oil sample and syringe 1 mL of used oil sample to volumetric bottle.

4. If solution in volumetric is green, continue to step 5. If not, add TAN titrant by one drop while stirring until green color.

5. Count amount of TAN titrant's drop.

- Results analysis

Calculate total acid number by equation

TAN titrant drops x factor = TAN mgKOH/g

If used TAN titrant 22 drops and used oil 1 mL

$22 \times 0.1 = 2.2 \text{ mgKOH/g}$

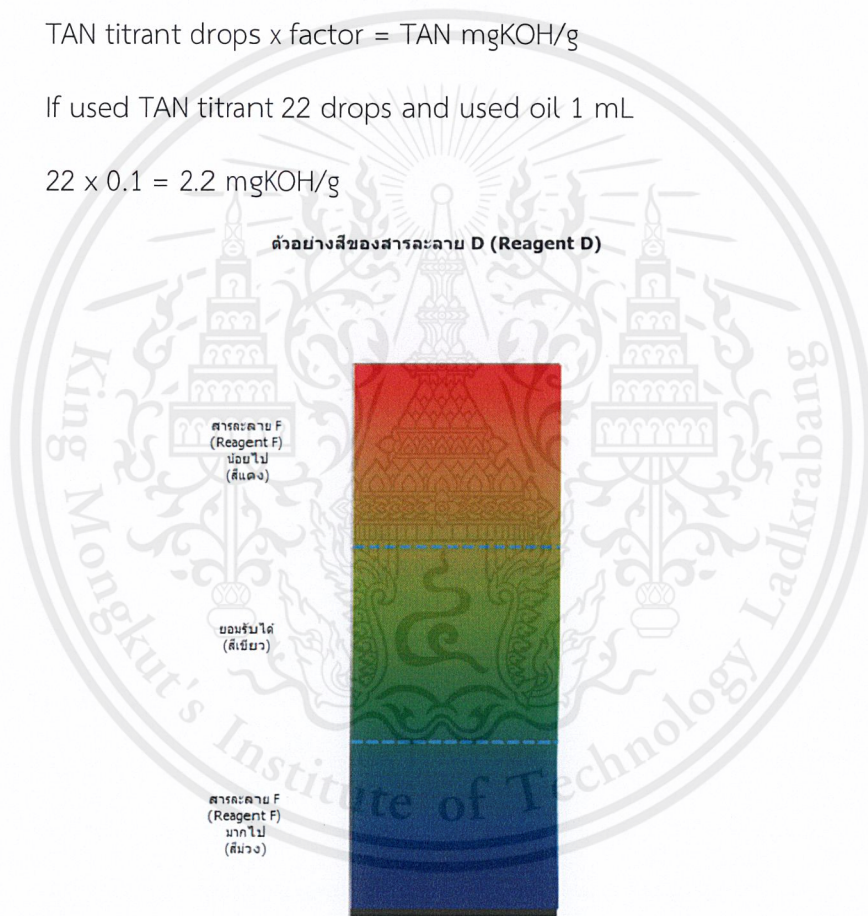


Figure 3.5 Reagent D's Color

3.4.3 Water Saturated

- Chemicals : Used oil, Easyship and Reagent S
- Equipment : Water test kit (include ring) and syringe
- Procedure

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1. Switch on and set equipment.
2. Clean and dry the equipment.
3. Stir reagent S and measure 20 mL of reagent S to container.
4. Pour Easyship and 5 mL of used oil to center container.

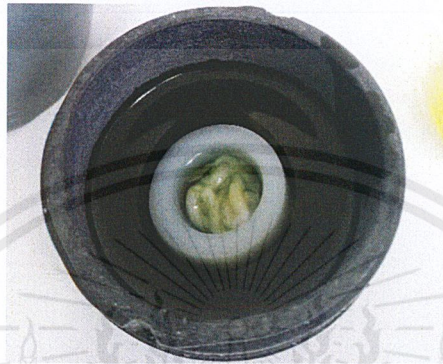


Figure 3.6 Easyship, used oil and ring in center container

5. Place ring to help stirring. Then, close lid and stir about 3 minutes

- Results analysis

Notice in screen, represent amount water in ppm.



Figure 3.7 Equipment screen represent amount of water

3.4.4 Viscosity

- Chemicals : Used oil

- Equipment : Viscosity test kit (Include 3 balls, mirror and cylinder)

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- Procedure

1. Measure used oil density and set viscosity equipment.
2. Pour used oil for a little and choose ball size to cylinder.
3. fill used oil until full and measure used oil temperature.
4. Instantly flip cylinder over and set the timer of ball flow to bottom cylinder by mirror.

- Results analysis

Do the test 3 times and find average value. After that, fill data and notice the results

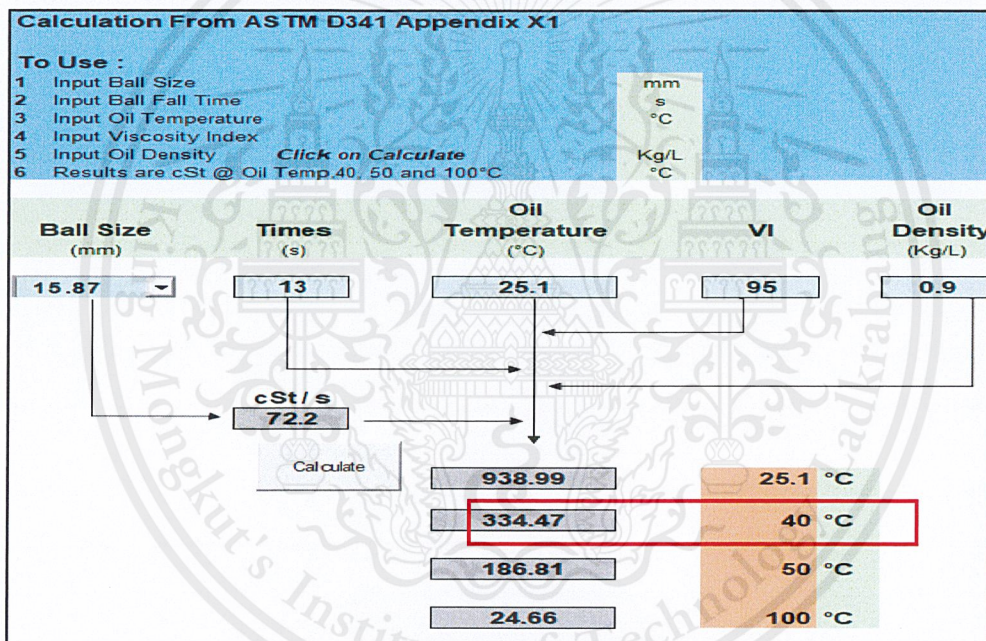


Figure 3.8 Viscosity calculation in program screen

3.4.5 Colors

- Chemicals : Used oil
- Equipment : light source, test tube

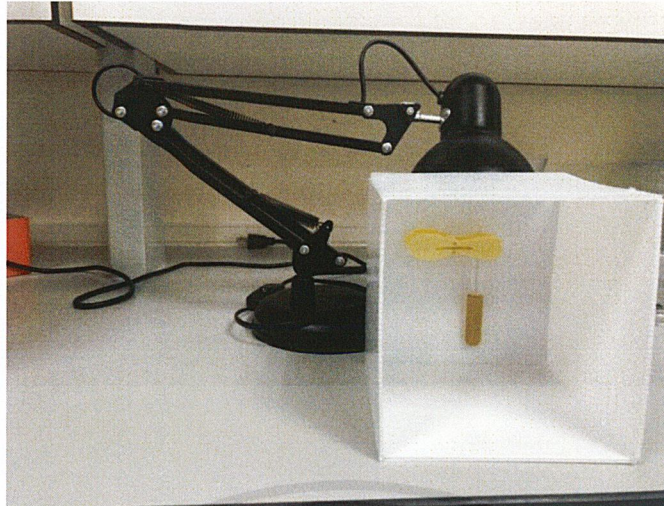


Figure 3.9 Colors testing kit

- Procedure

1. Measure the used oil sample about 4 ml and pour into test tube.
2. Switch on the light source.
3. Compare the color of the sample with the standard.

- Results analysis

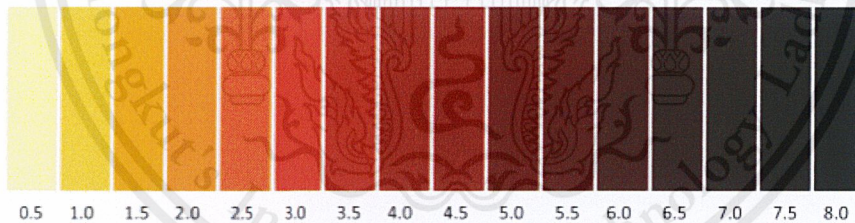


Figure 3.10 Demonstrative ASTM D 1500 scale

3.5 Find used oil analysis report for sample

Search and study sample oil analysis report for design used oil analysis report.

Mostly oil analysis reports have:

- name and address of customer company
- name and address of lab for contact
- equipment type and unit ID

- all testing and results of testing which notification by colors
- regularly sampling data for create oil trend

3.6 Applied to my own report and add significantly data, then create used oil analysis report

In addition, add sampling person and sampling date to keep for evidence. specially add recommendation for the best resolve machine problem to customer company in this report.

3.7 Study ISO 17025:2005, General requirements for the competence of testing and calibration laboratories

Good laboratory will consider general technical requirements. Many factors determine the correctness and reliability of the tests and/or calibrations performed by a laboratory. These factors include contributions from

- Personnel
- Accommodation and environmental conditions
- Test and calibration method validation
- Equipment
- Measurement Traceability
- Sampling
- Handling of test and calibration items
- Reporting the results

3.8 Drafted and installed furniture in laboratory room

There are shelves which keep sample used oil, lockers for keep equipment and sink that wash glassware and others equipment. Ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement and facilitate correct performance of the tests and/or calibrations.

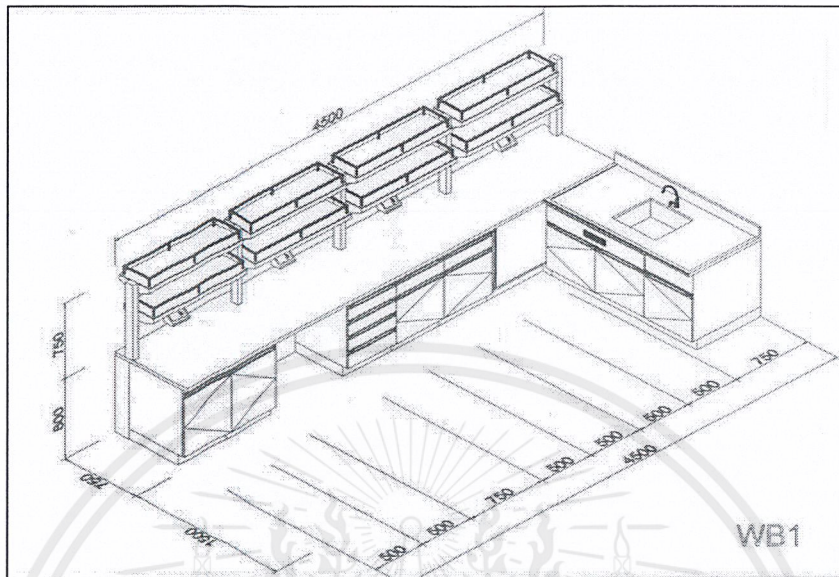


Figure 3.11 Drafted laboratory room

3.9 Simulate working process in laboratory room

- Used oil analysis process

1. Register bottle code and bottle number before sampling oil
2. Sampling 300 ml used oil and filling data on bottle sticker
3. Testing used oil by require issues from customer, all testing procedure are in work instruction

4. Record testing value in test sheet

5. Keep test sheet data fill in report

- Cleaning and removing process

1. Removing resist oil from testing to waste bottle
2. Close and unplug testing equipment and machine

3. Wash glassware and other equipment by using dishwashing liquid and fresh water

*If metal or stainless equipment rinse with heptane

4. Lay in basket and wait for dry

5. Rinse with heptane and store in locker

** Should clean equipment and glassware before and after testing by rinse with heptane. Ensure cleanliness equipment and corrective results.

3.10 Design working process in mobile car

Like working process in laboratory room, just different storage. Add printer to instantly print out report to customer.

3.11 Drafted and installed furniture in mobile car

Draft and specified mobile car to maker. There are cabinets for equipment storage, desk with locker for place laptop and small printer, two chairs, workbench in bottom car, aperture for big equipment storage, air conditioner, television and other facilitation that need for mobile car.

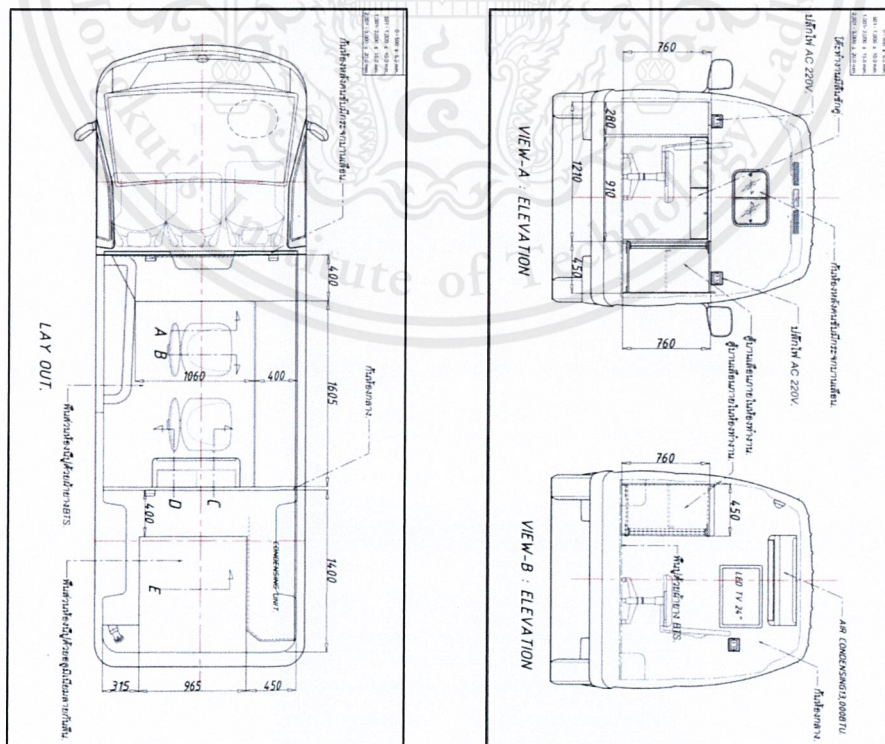


Figure 3.12 Drafted Mobile car

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CHAPTER IV

RESULTS AND DISCUSSION

1. Thai equipment's procedure

Fabricate Thai equipment's procedures with cleaning and removing process. DLE team will use this data to test and clean used oil for work instruction.

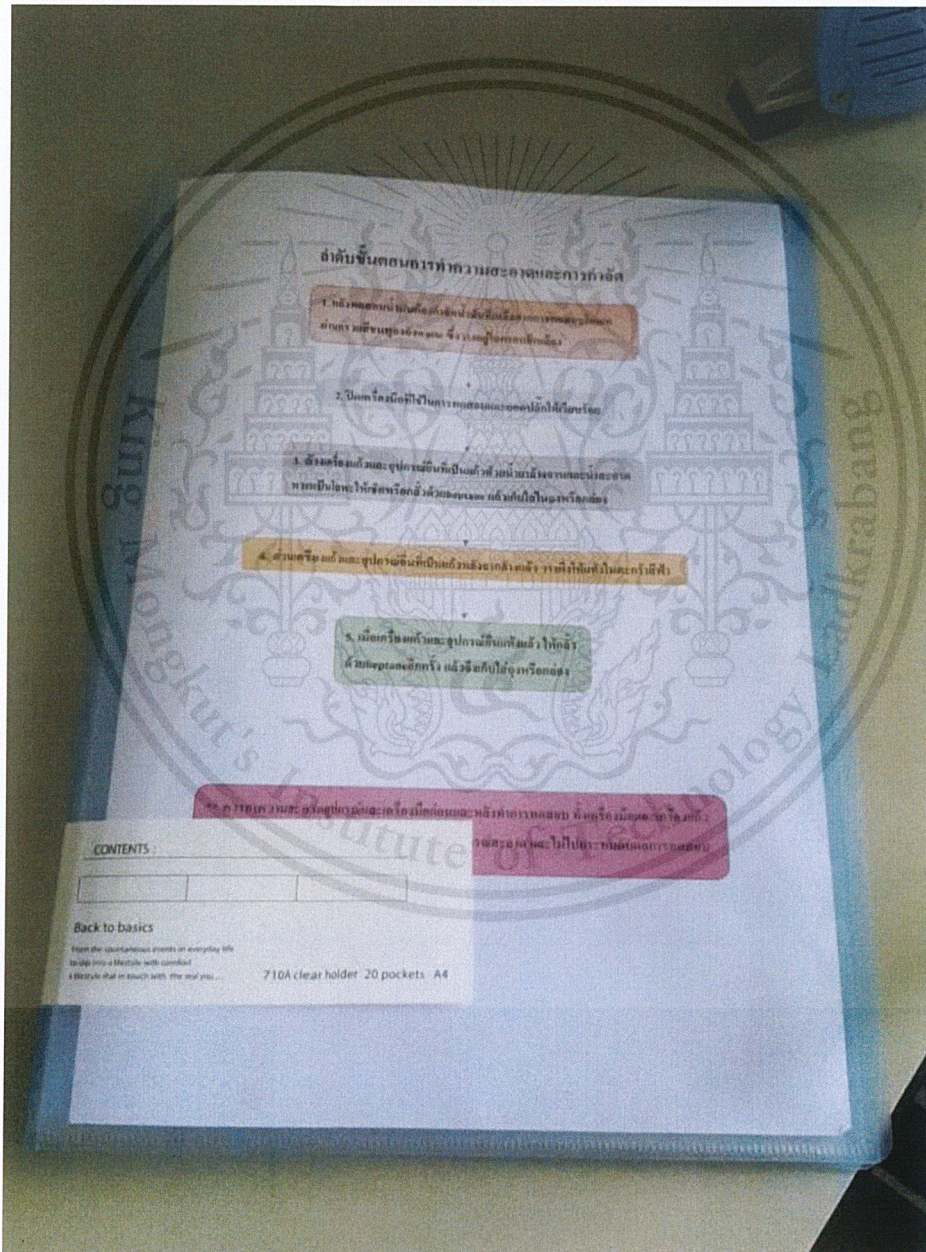


Figure 4.1 Thai used oil work instruction

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2. Used oil analysis report

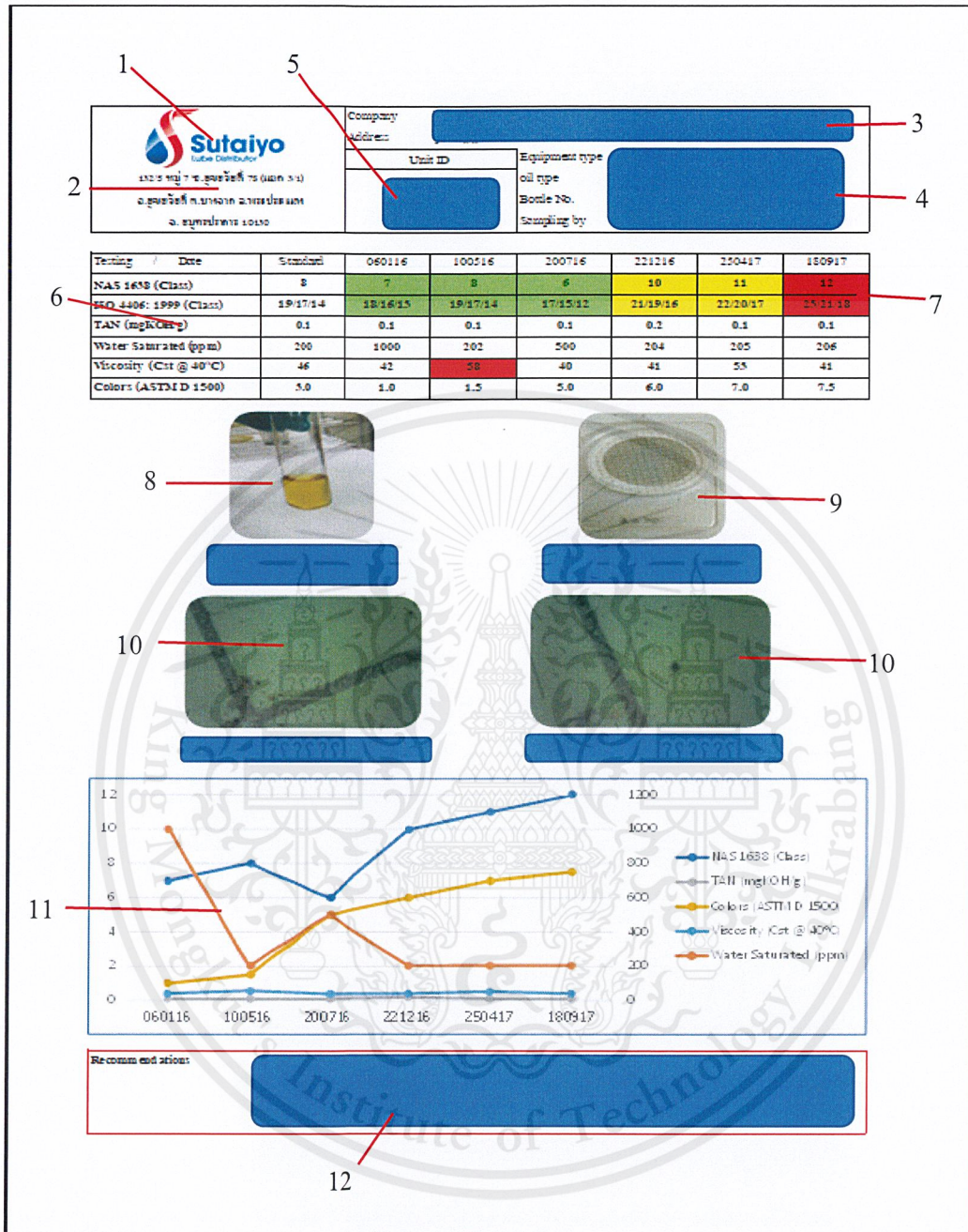


Figure 4.2 Sample used oil analysis report

This sample report consists of

1. Logo
2. Company address
3. Customer address
4. Customer equipment information (Including equipment type, oil type, bottle number and who sampling oil)

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5. Unit ID
6. Used oil testing
7. Used oil testing results with color notification
8. Oil's color in test tube
9. Oil's color after filtergram in petrislide
10. Filtergram result from 200x microscope
11. Trend line
12. Suggesting block

Sample report will show necessary data which customer should know and suggest to solve their problem about their used oil.

3. Laboratory room

To simulate testing process in laboratory room, will installed furniture made nice and tidy laboratory room. Moreover, increase systematic process used oil testing and analysis. Add waste bottles for keep used oil after testing before removing process and fire extinguisher for ensure safety or use in emergency condition.



Figure 4.3 Laboratory room before installed furniture lab



Figure 4.4 Laboratory room after installed furniture lab



Figure 4.5 Waste bottles and fire extinguisher in laboratory room

4. Mobile car

To support new project, lab mobile car is good choice for used oil testing on customer site. Unfortunately, Sutaiyo create new plant in Rayong then take much more money budget. Finally, Sutaiyo team consider that new plant is more necessary than mobile car. Sutaiyo will create mobile car in next year or new plant finished.

CHAPTER V

CONCLUSION

Thai equipment's procedures, used oil analysis report, laboratory room and mobile car are support new project in Sutaiyo Lube Distributors Co., Ltd. Create Thai equipment's procedure for used oil testing work instruction. Design used oil analysis report to show testing results of general properties and suggest how to solve used oil problem for customer. Simulate primary laboratory process and installed furniture in laboratory room. This company do not have budget and time enough to build mobile car.

SUGGESTION

Used oil laboratory should have hood and grease trap. Hood is important for laboratory room which use volatile substances to purify circulate air with air suction. Volatile substances will cumulate in personal and may be severe fatalities. Grease trap is installed under sink for trap grease before release to environment. If grease trap does not installed, river which organism habitat will be rotten and difficult to regain. In addition, should be control document in laboratory follow as ISO/IEC 17025:2005 standard. Always update document to be current information for easy personal working.

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- ASTM D 974:2005, Standard Test Method for Acid and Base Number by Color-Indicator Titration

- ASTM D 1500:2005, Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

- ASTM D 1744:2005, Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent

- ASTM D 6158:2005, Standard Specification for Mineral Hydraulic Oils

- ASTM D 6217:2005, Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration

- ASTM D 4057:2005, Standard Practice for Manual Sampling of Petroleum and Petroleum Products



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APPENDIX A

Sampling

Manual sampling is sampling procedure in customer company and take back to test in laboratory room

Manual Sampling of Petroleum and Petroleum products [ASTM 4057]

Procedures for manually obtaining samples of petroleum and petroleum products of a liquid, semi-liquid or solid state from tanks, pipelines, drums, barrels, cans, tubes, bags, kettles and open-discharge streams.

Manual sampling Considerations

- Physical and chemical properties tests
- Sampling sequence
- Equipment cleanliness
- Compositing of individual samples
- Sample storage
- Sample handling

Plastic bottle containers made of suitable material may be used for the handling and storage of gas oil, diesel oil, fuel oil and lubricating oil. Bottles of this type should not be used for gasoline, aviation jet fuel, kerosene, crude oil, white spirit, medicinal white oil, and special boiling point products unless testing indicates there is no problem with solubility, contamination or loss of high components.

Sampling handling

- Volatile samples shall be protected from evaporation. Transfer the product from the sampling apparatus to sample container immediately. After delivery to the laboratory, volatile samples should be cooled before the containers are opened.
- Light sensitive samples be kept in dark, if the testing is to include the determination of such properties as color, octane, inhibitor contents. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately.
- Container Outage Adequate sample mixing is difficult if the container is more than 80% full.

Sampling labeling

Label the container immediately after a sample is obtained. Use waterproof and oil proof ink or a pencil hard enough to dent the tag. Soft pencil and ordinary ink markers are subject to obliteration from moisture, oil smearing and handling. Include the following information on the label:

- Date and time
- Name of sampler
- Name and number and owner of the container
- Grade of material
- Reference symbol or identification number

Bottle spot sampling procedure

1. Inspect the sampling bottle, graduated cylinder and sample container for cleanliness and use only clean and dry equipment.
2. Obtain an estimate of the liquid level in the tank. Use an automatic gage or obtain an outage measurement if required.
3. Attach the weighted line to the sample bottle or place the bottle in a sampling cage, as applicable.
4. Insert the cork in the sampling bottle.
5. Lower the sampling assembly to the required location.
6. At the required location, pull out the stopper with a sharp jerk of the sampling line.
7. Allow sufficient time for the bottle to completely fill at the specific location.
8. Withdraw the sampling assembly.
9. Verify the bottle is completely full. If not full, empty the bottle and repeat the procedure beginning with 4.
10. If only this spot sample is required for compositing will be accomplished elsewhere, pour all of the sample into the sample container or discard one-fourth of the sample. If composited samples are required at more than one location, measure out a specific amount of sample with a graduated cylinder and deposit it in the sample container.

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11. Discard the remainder of the sample from the sampling bottle as required.
12. Repeat 3. Through 11. to obtain additional sample volume if only a middle sample is required.
13. Install the closure on the sample container.
14. Disconnect the line from the bottle or remove the sample bottle from the sampling cage, as applicable.
15. Label the sample container.
16. Return the sampling container to the laboratory or other facility for mixing and testing



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