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SQN AI-18.18 Page 36 Revision 1

SEQUOYAH FINAL EVENT REPORT

REPORT NO. II-89-049

FINAL EVENT REPORT CONCERNING

Representative Sampling of Diesel Fuel Storage Tanks

Routing	Original (Signature/Date)	Rev. A (Init./Date)	Rev. B (Init./Date)	Rev. C (Init./Date)
Event Manager	Any Finn 18-23-09	/	/	/
Plant Manager or Duty Plant Manager	(Maron, 8-23-8	<u> </u>	/`	
Site Director or Duty Site Director	No Contolesky	/	/	· · ·
Other	/	/	/	
ther documents <u>AQR SQP890457</u> ,	s (i.e., CAQR's, LER's, etc. , PRO 1-89-187, PRO 1-89-189) generated a	s a result of	this event:
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PAGE 1 of 5

Executive Summary

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On 08/16/89 at 1109, during a evaluation of a Nuclear Experience Review Report concerning a problem at another nuclear facility encountered in obtaining representative samples of diesel generator fuel oil, it was determined that the samples taken at Sequoyah did not comply with the Technical Specifications (LCO 3.8.1.1.d) . The plant Technical Specification requires sampling of the 7-day storage tanks to be performed as outlined in ASTM D270-1975. Sampling of all the 7-day storage tanks was initiated to determine fuel oil quality and reestablish operability of the diesel generators.

Operations personnel initiated Potential Reportable Occurrence (PRO) Report No. 1-89-187. Preliminary Event Report (PER) No. II-89-049 (Attachment A) was performed by the Sequoyah Chemistry and Environmental Group. This occurrence was determined to be reportable to the Nuclear Regulatory Commission (NRC). An investigation, including Root Cause Analysis (Attachment B), was performed. The root causes were determined to be (1) Review during design did not consider sampling. (2) Inadequate review of system design during procedure evaluation.

A detailed summary of findings and recommended corrective actions are provided in Section V of this report.

II. <u>Description of the Event</u>

A. Initial Conditions

Unit 1 and 2 were in Mode 1 at 100 percent power.

B. Sequence of Events

The following sequence of events provide a detailed narrative of all actions and occurrences:

- 08/10/89 ≈1500 Chemistry personnel were notified of a potential problem associated with sampling of diesel fuel storage tanks (7-day tanks). Chemistry was given a 10 day period to respond to the NER (NER OR 3491). At this time chemistry personnel believed that the design allowed recirculation of the tanks, and that the NER did not apply to Sequeval.
- 08/14/89 0800- This time frame was utilized to verify the NER 1500 did not apply to Sequoyah and to prepare the appropriate response. However, during the evaluation it was determined that each 7-day storage tank was designed with four horizontal cylindrical tanks, side-by-side, approximately 85' long and 6' diameter. These tanks are connected to each other at each end on the top and bottom by a 12" section of pipe. ThFI000259 recirculation was inadequate in that only a portion of the two center tanks were attended.

PAGE 2 of 5

TT. Description of the Event (continued)

1500-	An evaluation of system design was		
2100	conducted by the chemistry section	and	a CAQ
	(Attachment C) written to document	the	finding.

08/15/89 0800- A review of the NER, CAQ, 7-day tanks design, Sequoyah procedures, etc., set in place the preparation of an Action Plan (SQA 211) to verify the quality of diesel fuel in the 7-day storage tanks.

> 1109 Entered LCO 3.8.1.1.d due to inadequate verification of D/G seven day tank fuel oil, SR 4.0.3., allowed 24 hours until action requirement are in effect (PRO 1-89-187).

The Action Plan provided for removal of each 7-day storage tanks manhole covers and sampling per ASTH D270-1975. The first sampling was followed by the removal of water, sludge, and particles, and additional sets of samples were obtained.

Samples were transfered to Chattanooga Power Services Center for analysis per Technical Specification requirements.

- 2130 Chattanooga Power Service Center notified Sequoyah of initial results for analysis of Water and Sediment and Viscosity. Tank 2AA initially failed the Water and Sediment limit at .25% by volume (Limit of .05). The second sample taken after pumping was within the acceptance criteria (Attachment E).
- 08/16/89 1109- LCO 3.8.1.1.d, 24-hour clock expired, NRC was notified and Notice of an Unusual Event declared at 1129.
 - 1430 Chemistry received the results from the insolubles analysis from Chattanooga Power Service Center. All results of the diesel fuel were within specifications, and applicable LCO exited.
- C. Immediate Corrective Actions
 - 1. Chemistry obtained samples from each of the four tanks that makeup the individual 7-day tank. All sample results were within the specific acceptance criteria except 2AA.
 - 2. Each of the tanks were checked for water and if detected, the water was removed by pumping and resampled.

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PAGE 3 of 5

III. Analysis of the Event

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A. Evaluation of Plant Systems/Components

The design of the fuel oil storage tanks does not provide an appropriate sampling mechanism to comply with ASTM D270-1975. This ASTM requires samples from the bottom, middle, and top of each tank.

Also the design will not allow the mixing of chemical additives through-out the four tanks making up a 7-day tank. The only provisions for mixing is using the transfer pump which takes suction from the bottom end of one tank and returns the fuel to the same end of a second tank. Two of the four tanks that make up the 7-day tank were not in the recirculation path. See Attachment D print number 47W310-6 for further details.

B. Evaluation of Personnel Performance

This section is not applicable.

C. Safety Implications

The procedure for sampling the diesel fuel oil used in the emergency diesel generators, does not ensure that the diesel generators could be maintained in-service if needed. The fuel oil quality can result in either a diesel generator not starting or being operated properly.

D. Previous Similar Events

The section is not applicable due to this evaluation resulting from an NER.

IV. Root Cause Statements and Their Associated Root Cause Codes

Performance of the Root Cause Analysis (Attachment B) resulted in the following determinations:

ROOT CAUSE

CODE

- 1. Review during design did not consider sampling. JC
- 2. Inadequate review of system design during procedure JI evaluation.
- V. Finding and Corrective Actions
 - A. Findings
 - 1. During the design or construction no means was provided for sampling the 7-day storage tanks as required by technical specification section LCO 3.8.1.1.d.

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PAGE 4 of 5

Finding and Corrective Actions

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- A. Findings (continued)
 - Interpretation of the requirements for sampling failed to identify that dip sampling was the only acceptable method to satisfy ASTM D270-1975. Recirculation sampling was not allowed.
- B. Corrective Actions

The listed corrective action will be the responsibility of G. L. Fiser, Chemistry and Environmental Superintendent and will be completed by September 30, 1989.

- 1. The Sequoyah Technical Instruction for sample collection, TI-16 section A.10.2 and TI-37 logsheet numbers 37 and 38 will be revised to ensure sampling is performed per ASTM D270-1975.
- 2. The Sequoyah Surveillance Instruction SI-116 for quarterly diesel . generator fuel oil sampling will be revised to ensure sampling and testing is performed per ASTM D270-1975. CAQR No. SQP890457 will also track these corrective actions.
- 3. Sequoyah Technical Specifications addressing diesel fuel will be evaluated to determine if a Technical Specification change is needed to reflect present-day sampling and analysis techniques and requirements.
- The design of the seven day storage tanks will be evaluated for possible changes to facilitate sampling and enhance recirculation.
 (A Design Change Request will be submitted if warranted.)

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C. Concurrence by Responsible Organizations I hereby concur with the above stated corrective actions, and agree to complete those actions for which my organization is responsible by the required due date.

Fiser

VI. Description of the Investigation

The investigation team consisted of the following members:

Don Adams, Chemistry, Event Manager

Don Amos, Chemistry, Investigator

Wayne Reid, Chemistry, Investigator

Vernon Shanks, Chemistry, Investigator

All investigative actions as stated in the Preliminary Event Report were completed.

PAGE 5 of 5

VI. <u>Description of the Investigation (continued)</u>

An evaluation of a Nuclear Experience Review report concerning a problem another nuclear power facility encountered in obtaining representative samples of diesel generator fuel oil was being performed. During the investigation it was found that the 7-day tanks are made-up of four individual cylindrical tanks connected at both ends. Also it was learned that during the recirculation process suction was being taken from the bottom of one tank and returned to the same end of a second tank. Prior to this investigation it was believed that the recirculation met the intent of ASTM D270-1975.

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APPENDIX E

PRELIMINARY	EVENT	REPORT
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	PRELIMINARY EVENT REPORT Page 1 of 4 REPORT DATE: 08-17-89 REPORT NO. II-89-049
1.	Plant: <u>Sequoyah</u> Unit(s) Affected: <u>1 and 2</u>
	Discovery (Date/Time): 08/14/89 1500 Event (Date/Time):
	System(s) Affected: <u>Emergency Diesel Generators</u>
	Component(s) Affected: 7-day Storage Tanks (Fuel Oil)
	Plant Condition at Time of Discovery/Event: <u>Both units in Mode 1</u>
	Procedure(s) Involved: <u>SI-116, SI-294, TI-16, TI-37 and SOI-18.1</u>
	Radiological Emergency Plant Classification (if applicable): <u>NA</u> Event Classification: NA
	Position of Supervisor Directly Responsible for the Work/Evolution Leading to
	the Condition or Event: <u>Chemistry and Environmental Superintendent</u>
	the condition of Event. <u>Chemistry and Environmental Superintendent</u>
	Location of Event: <u>Diesel Generator Building</u>
	Date(s) of Any Known Previous Occurrences: NA
	bace(b) of may move revious occurrences. ma
·.	NRC Notification Determination 3. NRC Reportable
1	(phone call): (written report)?
	Not Required No
	Need Further Info/Review X Yes Type/No. LER
	X NRC Call Date/Time8-15-89 1109 Due Date: 09-14-89
	a. Per 10 CFR 50 Further Investigation
	Section: 10CFR50.73 (J)(1) Required
	Comments: Comments:
4.	Nuclear Experience Review:
	Database Review for Similar Events Required? YES NO _X
	Nuclear Network Entry Required? YES NO X
5.	Description of Condition/Event: (For personnel injuries, include the
	individual's name, nature of the injury, and how the individual was
	dispositioned).

NER OE-3491 (08/10/89), an evaluation was performed on 08/14/89 on the sampling procedure used for the 7-day diesel fuel oil storage tanks. It was found that the sampling procedure required a recirculation to be performed prior to sampling, to provide a representative sample. The ASTM methods specified in the Plant Technical Specifications (ASTM D270-1975) does not specify this type of sampling. It was also determined that the 7-day storage tanks were four individuals cylindrical tanks connected at both ends and that during the recirculation process suction was being taken from the bottom of one tank and return to the same end of a second tank. This process does not provide adequate recirculation of all four tanks.

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APPENDIX E

PRELIMINARY EVENT REPORT

PRELIMINARY EVENT REPORT Page 4 of 4 REPORT DATE: 08-17-89 REPORT NO. II-89-049 9. Approvals ١ Event Manager Organization: Chemistry G. L Fiser Typed Plant Manager or Duty Plant Manager C. Mason Č. Typed Site Director or Duty Site Director т. LaPoint Typed

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SQN **S0A84** Page 11 Revision 8 ATTACHMENT 1 Page 2 of 8 . REPORTABLE OCCURRENCE PART "A" POTENTIAL PRO NO. 1-89-187 ORIGINATOR NR THOMAS 6371 OPS 08/15/89 / Name Section Ext. Date DESCRIPTION OF EVENT: ENTERED LCO 3.8.1.1.d AT 1109 DN 8/15/89 DUE TO INADEQUATE VERIFICATION OF D/G SEVEN DAY TANK FUEL OIL PER SI-116.ALL FOUR D/Gs ARE INOP SINCE REPRESENTATIVE SAMPLES HAVE NOT BEEN TAKEN.SR 4.0.3 IS ALLOWING 24 HRS UNTIL THE ACTION STATEMENT REQUIREMENTS ARE IN EFFECT. HAS A CAOR BEEN INITIATED ?: NO CAGR NO. N/A TIME/DATE EVENT DISCOVERED 1109 08/15/89 METHOD OF DISCOVERY OPS INFORMED BY MANAGEMENT LCO ENTERED OR INVOLVED 3.8.1.1 ____ TIME/DATE LCO ENTERED_1109 08/15/89 TIME AND DATE LCD EXITED 1 PERSONNEL INVOLVED: OPS MAINT (Check more than one if appropriate) Specify PROCEDURES INVOLVED IN THE EVENT SI-116 (Workplan, SI, MR, WR, GOI, SOI, etc.) EQUIPMENT INVOLVED (Component ID) _D/G 7-DAY TANK IS THIS ITEM 10CFR50.49 RELATED ?: NO PLANT ACTIVITIES PRIOR TO EVENT BOTH UNITS IN MODE 1 RPS or ESF ACTUATED: NO N/A (as a result of the event) List what actuated or what should have actuated

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:	ATTACHME Page 3 o POTENTIAL REPORTA PART "A" (co	f B BLE OCCURRENCE ntinued) PR	O NO. <u>1-89</u> -187
SAFE	TY-RELATED EQUIPMENT OUT OF SERVI	CE AT TIME OF EVENT EQUIPMENT NA	:
·	$\begin{array}{c} 3.7.12\\ \text{COMMON} & \underline{3.3.3.10}\\ 3.4.3.2\\ \text{UNIT} & \underline{3.5.1.1}\\ \underline{3.4.3.2}\\ \text{UNIT} & \underline{3.5.1.1}\\ \text{UNIT} & \underline{3.5.1.1}\\ \end{array}$	FIRE BARRIERS 0-RM-90-101 PURV 1-LT-63-109 FURV	• ••••••••••••••••••••••••••••••••••••
	ATOR ACTION TAKEN ON EVENT: D LCO 3.8.1.1 AND SR 4.0.3	<u>2-LT-63-99</u>	
WAS	1 STATUS <u>1</u> Mode <u>100%</u> Power 2 STATUS <u>1</u> Mode <u>100%</u> Power THE REP INITIATED (IF YES, 1 HOUR ICABLE 10CFT50.73 (if known) <u>10C</u>	<u>2235</u> Pressure NRC NOTIFICATION):	578F Temp 578F Temp NO
NRC NRC	NOTIFIED: NO · If yes, PERSON NOTIFIED: <u>N/A</u> RS NOTIFIED	10CFR	N/A
OPER PORS	ATIONS DUTY SPECIALIST: NO DUTY MANAGER: NO	PLANT MANAGER NRC RESIDENT:	
WR/M STA	R WRITTENTO CORRECT PROBLEM: NO MULTUN 18-15-85 Date	NUMBER N SOS <u>Clandar</u>	1A ww.c. 1. <u>S-15-5-</u> Date

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SQN SQA84 Page 13 **Revision** 8 ATTACHMENT 1 Page 4 of 8. POTENTIAL REPORTABLE OCCURRENCE PRO NO. 1-89-187 PART "B" This PRO has been reviewed and, based on the information above, appears to potentially be _____ Reportable _____ Not Reportable. 10CFR Requirement applicable 50.73 (5)(1) Should a CAQR be initiated? _____ Yes No If yes, refer to AI-12, Part III. CAQR No. (if known) Basis: There is a possibility The methodology of testing was incorrect. This would show a proceedual deficiency that all Four D/G3 inaped ۳. 18-16-89 PRS ENGINEER DATE

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	· ·	SGN SQAB4 Page 10 ATTACHMENT 1 Page 1 of 8 POTENTIAL REPORTABLE OCCURRENCE	· · · · · ·
		PRO NUMBER <u>1-89-189</u> <u>ROUTING</u> ORIGINATOR <u>G STRICKLAND</u> <u>1 08/16/89</u> SHIFT OPERATIONS SUPERVISOR <u>REVENUE & F.C. 17</u> PRS ENG <u>OTE Sandla 1 e/17/89</u> <u>for J. KENT</u>	•
	This event is:	NOT REPORTABLE REPORTABLE Report No.	** ·
Ú		PRS Supervisor Date	<u> </u>
	Package Distributio Original - Sequoyah cc: PRS PRO Notebo cc: Regulatory Lic generic items	on (when completed) n Document Control poks ensing Experience Review Coordinator (potentially only)	· · ·

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Page 11 Revision 8	
ATTACHMENT 1	
Page 2 of 8 POTENTIAL REPORTABLE OCCURRENCE	
PART "A" PRO NO. <u>1-89-189</u>	
ORIGINATOR <u>G STRICKLAND / 6371 / STA / 08/16/89</u> Name Ext. Section Date	• • • • •
DESCRIPTION OF EVENT:	.
AT 1109, 8/16/89, UNIT 1 & 2 ENTERED NOUE DUE TO D/G'S DECLARED INOP.	· ·
HOUR PHONE CALL TO NEC FOR FO TO A LAND TO THE	
	6055/16/51
LUGS ATTACHED. 1-1.50 entered LCD 37.11.1. Action 5.	
· ·	•
	•
HAS A CADR BEEN INITIATED?: YES CADR NO.	. ·
TIME/DATE EVENT DISCOVERED 1109 08/16/89	- .
METHOD OF DISCOVERY <u>CHEM LAB NOTIFICATION</u>	
LCO ENTERED OR INVOLVED 3.8.1.1.D TIME/DATE LCO ENTERED 1107 08/16/89)
TIME AND DATE LCO EXITED //	
PERSONNEL INVOLVED: _ OPS	
(Check more than one if appropriate) TECHNICAL SUPPORT Specify	· · ·,
PROCEDURES INVOLVED IN THE EVENT	•
(Workplan, SI, MR, WR, GDI, SDI, etc.)	•
EQUIPMENT INVOLVED (Component ID)	_
IS THIS ITEM 10CFR50.49 RELATED?: NO	
PLANT ACTIVITIES PRIOR TO EVENT 11 & 2 100%	
RPS or ESF ACTUATED: NO	
(as a result of the event) List what actuated or what should have actuated	•

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SQN SDA84 Page 12 Revision 8	
ATTACHMENT -1 Page 3 of 8	
FOTENTIAL RÉPORTABLE OCCURRENCE PART "A" (continued) PRO NO. <u>1-89-189</u>	
SAFETY-RELATED EQUIPMENT OUT OF SERVICE AT TIME OF EVENT:	
LCO NO. EQUIPMENT NAME	
COMMON 3.7.12 3.3.3.10	
UNIT 1 3.5.1.1	
UNIT 2 3.4.3.2 3.5.1.1	
OPERATOR ACTION TAKEN ON EVENT:	
COMPLYING WITH I CO 3.8.1.1.0 AND 3.0.5 , for protection commited	
fine prop to HEFT Syster De-43.711.1 27 his please	
Cali mule To Nill at 1411 5/16/17 (65) 5/16/81	
UNIT 1 STATUS <u>1</u> Mode <u>100%</u> Power <u>2235</u> Pressure <u>578F</u> Temp	
UNIT 2 STATUS <u>1</u> Mode <u>100%</u> Power <u>2235</u> Pressure <u>578F</u> Temp	
WAS THE REP INITIATED (IF YES, 1 HOUR NRC NOTIFICATION): YES	
APPLICABLE 10CFT50.73 (if known) N/A	
NRC NOTIFIED: YES IF yes, 1 HR 10CFR 72.A.1.I BIII	
NRC PERSON NOTIFIED: BOB SFENCE TIME 1129	
OTHERS NOTIFIED	
CPERATIONS DUTY SPECIALIST: YES PLANT MANAGER: YES	
PORS DUTY MANAGER: NO NRC RESIDENT: YES	
WR/MR WRITTEN TO CORRECT/PROBLEM: NO NUMBER N/A	
STA Silver 18/1: (7) SOS MOD marche 1 8-16-7-7 Date	

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SON FILE PACKAGE NO. 18 AI-18 Page 18 of 33 N.S. NUCLEAR REGULATORY COMMISSION OPERATIONS CEN. Page 39 Appendix A EVENT NOTIFICATION HORKSHEET Revision 47 KOTIFICATION TIKE FACILITY OR ORCANIZATION דזאט CALLER'S NAKE TELEPHONE NUMBER (FOR Call 0-142 المهزبة بتركح 13.55 Since EVENT CLASSIFICATION CAUSE OF FAILURE EVENT CATEGORY INITIATION SIGNAL GENERAL ENERGENCY REACTOR TRIP/SCRAM HECHNICAL SITE AREA EMERGENCY ESF ACTUATION ELECTRICAL ALTRT ECCS ACTUATION PERSONNEL ERROR UNUSUAL EVENT PROCEDURE INADEOUACY SAFETY INJECTION FLOW 3:8.1.1.64 50.72 NON-EMERGENCY LCO ACTION STATEMENT OTHER loil surfle 3. 0. 1 214 SECURITY/SAFEGUARDS 140 OTHER TRANSPORTATION EVENT ZONE SYSTEN: 62 EVENT TIME EVENT OTHER: 1105 DATE コン COMPONENT: And EVENT DESCRIPTION AT 1108, 5/16/88, all 4 D/6's declared insp A. NOUE was entered at 1105. The reason for The DIG's being declared map was due to the chemistry section determining that the recorculation with the DIG field oil tunks did not adequately record all the fuel bit in the Tanks and, Therefore, The sirve requirements incre not being mot. 1105 Stist the plant stilized SR 4.2.3 which allouis 24 hours TE complete sure tests. AT 1:09 16 the 24 hours to complete sure testing The fuel s. I has been sumplied & The - visicosity has passed. Till Test بالمن المتر - le hars 1. reguines Sevenent . خ-م POWER PRIOR TO EVENT (2): 1000 YFS_ If NO. Explain above Did all systems function as required ? CURRENT POWER OR MODE NO . لر تن سر د If YES, Explain above. Anything "unusual" or not understood ? Sumpling 13/15 foret or 1 OUTSIDE AGENCY OR PERSONNEL NOTIFIED BY LICENSEE STATE(S): F1000273 Cal ; 100 SIDENT 125 WELL BE :10 +-0 ESTIMATE TIME TO PESTART: NODE OF CREPATION TILL CORRECTION: PRESS PELEASE ومرود المسارقة والمرود والمستعد المراجع والمراجع والمراجع والمراجع • • • • ADDITICNAL INFORMATION ON BACK ۰. and all a service of the second s

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SQN FILE PACKAGE NO. 18 AI-18 U.S. NUCLEAR REGULATORY COMMISSION Page 18 of 33 OPERATIONS CEXT Page 39 EVENT NOTIFICATION WORKSHEET Appendix A **Revision 47** TELEPHONE NUMBER (FOF CALL 5 KOTIFICATION TIKE FACILITY OR ORCAHIZATION UNIT CALLER'S NAME 59111 EVENT CLASSIFICATION CAUSE OF FAILURE rl N EVENT CATEGORY INITIATION SIGNAL GENERAL EMERGENCY RECHNICAL REACTOR TRIP/SCRAM SITE AREA EMERGENCY ELECTRICAL ESF ACTUATION ALTRT PERSONNEL ERROR ECCS ACTUATION UNUSUAL EVENT PROCEDURE INADEQUACY SAFETY INJECTION FLOW 50.72 NON-EMERGENCY 140 44 LCO ACTION STATEMENT OTHER 140 2440 SECURITY/SAFEGUARDS OTHER TRANSPORTATION -EVENT SYSTEH: EVENT TIME ZONE RONTIE \mathcal{N} EVENT OTHER: DATE COMPONENT: At 1451, CX.Ted LCO3.8.1.1. d 3.0.5 /NOUE based on results of D/1 Frel oil Called NRC, Bus Sperse accepta Ste ndate + cancel the NOLE Also informed 24 phone call AFD's my due to D/6's my 623 07 475 1s may doe - LCS 3.7.11.1, requires phone cell to foll in Iwerday doy" + 14 days. Enterel LO3. 7.11.1 at 0524 for SI-234.2 HPEP's in STOP. ; All Y HPEP's inpat 1103 5/16 don't D/G pro Slens.; 1 11! ; Corrently that oper at 1451 when Old 200 51-234.6 mul for YES It NO. Explain above. POWER PRIOR TO EVENT (3): Did all systems function as required ? If YES, Explain above, NO CURRENT POWER OR MODE Anything "unusual" or not understood ? . . OUTSIDE AGENCY OR PERSONNEL CORRECTIVE ACTION(S) NOTIFIED BY LICENSEE STATE(S) : F1000275 IQCAL : 1. LOLAT 165 "0 WELL BE ESTIMATE TIME TO RESTART: THER MODE OF OPERATION TILL CORRECTION: a na shi shi a shi a shi a shi a shi PRESS PELEASE . . . ADDITIONAL INFORMATION ON DACK · . - · · ويستعرب والمتعرب الاستريك سريد المريم المراويون والمريد ويدهون ال × •

	SQN SQA84
,	Page 13 Revision 8
	ATTACHMENT 1
	Page 4 of 8 POTENTIAL REPORTABLE OCCURRENCE
	PRO NO. 1-89-189

	PART "B"
	This PRO has been reviewed and, based on the information above, appears to potentially be Reportable Not Reportable.
	10CFR Requirement applicable
	Should a CAQR be initiated? Yes No ALREADY WAITTEN Sps signary
	If yes, refer to AI-12, Part III. CAQR No (if known)
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Attachment 1 Page 1 of 2 SON SQA 211 Page 7 Revision O UNIT ACTION PLAN Action Plan Leader_ Date 0/15/89 Ext: 6930 DON Amos I. Statement of problem/condition Specification SR 4.8. 1.1.2. hnica 1975. ASTM -2270 II. Objectives action Arne 271-1975. TM M 97 77 ASTM . 227 70 ٢. III. Tasks to be performed ama an aid 14 : IV. Responsible Organization/Tasks : Responsible Organization/Individual/Phone No. · Task nation Reb Richi 7421 4 751-4277 Re l Action Plan Leader m 0239M/IRG/mjf FI000277

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Attachment 1 Page 2 of 2

UNIT ACTION PLAN

SON SQA 211 Page 8 Revision 0

V. Time Line of Action Plan:

Remove the montile Covers on each of the 4 compartments of the applicable 7-day storage tail (WR 271 830)

(2) obtain 3 somples of the lowa end of each form (4) compartments making up the 7-daing storage tank (total of 12 samples) litilize ASTM-D270.1975

Cupar a 1-liter -Composite for the 7- day tack from the 12 samples. Per ASTM

Jran samples (4) to Chatt. Power Service Caster for analysis . (T.S requirements)

Complete water and sedimant, binematei viscosity prior to impinity level and notify SNP personnel

Complete impirity level analysis any with SNP chimisty.

Mainterana return markde covers to proper locations

8 Chemistry crotify Operations, monagement sete. of results. -

Action Plan Leader: 18/15/89 Approval Authority: Revision:

0239M/IRG/mjf

Attachment 2 Page 1 of 5

SQN SQA 211 Page 9 Revision 0

RESPONSIBLE ORGANIZATION TASK ACTION PLAN

Konustry Responsible Organization Initiator Douline Date 8/15/89 Prejob Analysis: Α. Make a clear statement of the task to be performed. 1. the I storage tan 7-day diesel Sue tan 5 11ñ 2. Define the objectives of the task. lac m !se -0270-0975-77 ASTM 2274-フわ ; • Define Scope: Consider interconnected systems, related programs, interactive procedures etc., when trying to establish scope. A thorough evaluation of scope will result in an enhanced picture of the task to be performed. з. 7-0 ton 220en m STM-D975-77 274-70

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RESPONSIBLE ORGANIZATION TASK ACTION PLAN

- B. Consider the following items and provide a brief statement to determine their relevance to the task for which the Action Plan is developed.
- 1. Evaluate the task to identify obstacles and methods to resolve them. Special conditions necessary for completion of the task should be evaluated under this heading.

Mony mosite I be collected somples. 10- ASTM-DZ10-1925

- 2. Perform a task safety analysis. Review each item to be performed in the task and ensure that both personnel and equipment safety is addressed. <u>HCI-HM5</u> "Slotage Use and Handleir of Alandlo
- 3. Evaluate the environment in which work is performed. This will identify time frames that can be spent in areas, types of equipment that can be used, etc.

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Attachment 2 Page 3 of 5

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SON SOA 211 Page 11 Revision 0

RESPONSIBLE ORGANIZATION TASK ACTION PLAN

If data is to be collected what methods will be used for analysis? Root cause Assessment is to be performed, plan to use SQA-186. 5. If lle compared to crted a.C. we aint T.S. -. Plan for procedure interface by selecting, reviewing, and placing them in proper sequence. (Prepare new procedures if required). 6. <u>n/A-</u> •• 7. Evaluate personnel, equipment, and procedural interfaces that are required. .• NIA .. . : ; | Identify the level and detail of documentation necessary to ensure proper closure of the Action Plan. Determine methods used to ensure clarity and 8. verify adequacy. M. ms ficed results 0239M/IRG/mjf FI000281

Attachment 2 Page 4 of 5

SON SOA 211 Page 12 Revision 0

RESPONSIBLE ORGANIZATION TASK ACTION PLAN

Address the communication interfaces that are necessary to complete your Action Plan activities. 9. hemister Osuction maintenance . . : 10. Evaluate staff and schedule to ensure that the performance of the Action Plan can be carried out effectively. Determine what materials are needed or could be needed and either obtained or schedule as appropriate. 11. .-.• A Ň . . :-• ~ . . Consider any special test or maintenance equipment that may be needed to complete the plan. 12. : 0239M/IRG/mjf

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Attachment 2 Page 5 of 5

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C.

SON SQA 211 Page 13 Revision O

RESPONSIBLE ORGANIZATION TASK ACTION PLAN

Establish the sequence of events to be performed in chronological order. Use enough flexibility to make changes that will have a minimal impact on the task. Consider the possible alternate or parallel paths the evaluation can take (USE A LOGIC FLOW TREE WHERE THE COMPLEXITY OF THE ACTION PLAN MAKES A SEQUENTIAL CHRONOLOGICAL PLAN INADEQUATE).

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Designation: D 270 - 65 (Reapproved 1975)

American National Standard 211,33-1966 American National Standards Institute Method 8001-Federal Tast Method Standard No. 791b

Standard: 2546

Standard Method of SAMPLING PETROLEUM AND PETROLEUM PRODUCTS¹

This Standard is issued under the fixed designation D 270, the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. This is also a standard of the American Petroleum Institute issued under the fixed designation API 2546.

This method was adopted as a joint ASTM-API standard in 1965.

This method has been approved for use by agencies of the Department of Defense and for listing in the DOD Index of Specifications and Standards.

1. Scope

1.1 This method covers procedures for obtaining representative samples of stocks or shipments of crude petroleum and petroleum products. except electrical insulating oils, and butane, propane, and other pétroleum products that are gases at atmospheric temperature and pressure.

NOTE 1-The procedures described in this method may also find application in sampling most noncorrosive liquid industrial chemicals

Note 2-The procedure for sampling liquefied petroleum gases is described in ASTM Method D-1265. Sampling Liquefied Petroleum (LP) Gases.² the procedure for sampling electrical insulating oils in ASTM Method D 923, Sampling Electrical Insulating Liquids:³ and the procedure for sampling natural gas in ASTM Method D 1145, Sampling Natural Gas.

NOTE 3-The values stated in U.S. customary units are to be regarded as the standard. Metric couivalents have been rationalized where tolerances are considered non-critical.

2. Summary of Method

2.1 Samples of petroleum and petroleum products are examined by various methods of test for the determination of physical and chemical characteristics. It is accordingly necessary that the samples be truly representative of the petroleum or petroleum products in ouestion. The precautions required to ensure the representative character of the samples are numerous and depend upon the type of material being sampled, the tank, carrier, container or line from which the sample is being obtained, the type and cleanliness of the

sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their application is presented in Table 1. Each procedure is suitable for sampling a number of specific materials under definite storage, transportation, or container conditions. The basic principle of each procedure is to obtain a sample or a composite of several samples in such manner and from such locations in the tank or other container that the sample or composite will be truly representative of the petroleum or petroleum product.

3. Description of Terms

3.1 Average Sample is one that consists of proportionate parts from all sections of the container.

3.2 All-Levels Sample is one obtained by submerging a stoppered beaker or bottle to a point as near as possible to the draw-off level, then opening the sampler and raising it at arate such that it is about three-fourths full (maximum 85 percent) as it emerges from the liquid. An all-levels sample is not necessarily an average sample because the tank volume

Current edition effective Aug 31, 1965. Originally is-sued 1927. Replaces D 270 - 61

In 1965, this method was rewritten as a joint method with the American Petroleum Institute to take care of features on ASTM and API methods for sampling

Annual Book of ASTM Standards, Part 23.

Annual Book of ASTM Standards. Part 40

* Annual Book of ASTM Standaras. Part 26

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This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and the API Central Committee on Petroleum Measurement.

may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

NOTE 4—The tube sampling procedure. Section 22, may be used to obtain an all-levels sample from a barrel or drum.

3.3 Running Sample is one obtained by lowering an unstoppered beaker or bottle from the top of the oil to the level of the bottom of the outlet connection or swing line, and returning it to the top of the oil at a uniform rate of speed such that the beaker or bottle is about three-fourths full when withdrawn from the oil.

3.4 Spot Sample is one obtained at some specific location in the tank by means of a thief, bottle, or beaker.

3.5 Top Sample is a spot sample obtained 6 in. (150 mm) below the top surface of the liquid (Fig. 1).

3.6 Upper Sample is a spot sample taken at the mid-point of the upper third of the tank contents (Fig. 1).

3.7 Middle Sample is a spot sample obtained from the middle of the tank contents (Fig. 1).

3.8 Lower Sample is a spot sample obtained at the level of the fixed tank outlet or the swing line outlet (Fig. 1).

3.9 Clearance Sample is a spot sample taken 4 in. (100 mm) below the level of the tank outlet (Fig. 1).

3.10 Bottom Sample is one obtained from the material on the bottom surface of the tank, container, or line at its lowest point.

3.11 Drain Sample is one obtained from the draw-off or discharge valve. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

3.12 Water and Sediment Sample is one obtained with a thief to determine the amount of nonmerchantable material at the bottom of the tank.

3.13 Composite Sample is one made up of equal portions of two or more soot samples obtained from a tank. The term also applies to a series of line samples obtained from a free-flowing pipe line. (See Fig. 1 for location of spot samples.)

3.14 Single Tank Composite Sample 15

used in sampling petroleum products and is a blend of the upper, middle, and lower samples. For a tank of uniform cross section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal cylindrical tank, the blend consists of the three samples in the proportions shown in Table 2.

3.15 Multiple Tank Composite Sample (Ships. Barges, etc.), is a mixture of individual all-levels samples from the several compartments each of which contains the same grade of petroleum material. The mixture is blended in proportion to the volume of material in each compartment.

3.16 Composite Spot Sample is a blend of spot samples mixed in equal proportions for testing. Tests may also be made on the spot samples before blending and the results averaged. Spot samples from crude oil tanks are collected as follows:

3.16.1 Three-Way—On tanks larger than 1000-barrel (160-m³) capacity which contain in excess of 15 ft (5 m) of oil, samples should be taken at the upper, middle, and lower, or outlet, connection of the merchantable oil, in the order named. On tanks of 1000-barrel capacity and under, this method may be used also.

3.16.2 Two-Way—On tanks larger than 1000-barrel capacity which contain in excess of 10 ft (3 m) and up to 15 ft (5 m) of oil. samples should be taken at the upper and lower, or outlet, connection of the merchantable oil, in the order named. On tanks of 1000-barrel capacity and under, this method may also be used.

3.17 Middle Spot Sample—On tanks larger than 1000-barrel capacity containing 10 ft (3 m) or less of crude oil, one spot sample should be taken as near the center of the vertical column of oil as possible.

3.18 Continuous Sample is one obtained from a pipeline in such manner as to give a representative average of a moving stream.

3.19 Dipper Sample is one obtained by placing a dipper or other collecting vessel into the path of a free-flowing stream so as to collect a definite volume from the full cross section of the stream at regular time intervals for a constant rate of flow, or at time intervals varied in proportion to the rate of flow.

3.20 Mixed Sample is one obtained after

mixing or vigorously stirring the contents of the original container, and then pouring out or drawing off the quantity desired.

3.21 Tube of Thief Sample is one obtained with a sampling tube or special thief, either as a core sample or spot sample from a specified point in the container.

3.22 Borings Sample is one obtained by collecting the chips made by boring holes with a ship auger from top to bottom of the material contained in a barrel, case, bag, or cake.

3.23 Grab Sample is one obtained by collecting loose solids in equal quantities from each part or package of a shipment and in sufficient amount to be representative of all sizes and components.

3.24 Grease Sample is one obtained by scooping or dipping a quantity of soft or semiliquid material, such as grease, from a package in such a manner that the material on the scoop or dipper is representative of the material in the package.

APPARATUS

4. Sample Containers

4.1 Container Specifications-Sample containers may be clear or brown glass bottles, or cans. The clear glass bottle is advantageous because it may be examined visually for cleanliness, and also allows visual inspection of the sample for free water or solid impurities. The brown glass bottle affords some protection from light. The only cans permissible are those with the scams soldered on the exterior surfaces with a flux of rosin in a suitable solvent. Such a flux is easily removed with gasoline, whereas many others are very difficult to remove. Minute traces of flux may contaminate the sample so that results obtained on tests for dielectric strength, resistance to oxidation, and sludge formation may be erroneous. Sample containers for manual sampling of crude petroleum should be vaportight, equipped with delivery tube extending through the top to within ^{1/2} in. (12 mm) of the bottom, and with a funnel and positive closure to allow for submerged filling.

4.2 Container Closure—Cork or glass stoppers. or screw caps of plastic or metal, may be used for glass bottles; screw caps only shall be used for cans to provide a vaportight closure seal. Corks must be of good quality,

clean and free from holes and loose bits of cork. Never use rubber stoppers. Contact of the sample with the cork may be prevented by wrapping tin or aluminum foil around the cork before forcing it into the bottle. Glass stoppers must be a perfect fit. Screw caps must be protected by a fork disk faced with tin or aluminum foil, or other material that will not affect petroleum or petroleum products.

4.3 Cleaning Procedure-All sample containers must be absolutely clean and free of water, dirt, lint, washing compounds, naphtha, or other solvents, soldering fluxes or acids, corrosion, rust, and oil. Before using a container, rinse it with Stoddard solvent or other naphtha of similar volatility. (It may be necessary to use sludge solvents to remove all traces of sediment and sludge from containers previously used.) Then wash the container with strong soap solution, rinse it thoroughly with tap water, and finally with distilled water. Dry either by passing a current of clean, warm air through the container or by placing it in a hot dust-free cabinet at 104 F (40 C) or higher. When dry, stopper or cap the container immediately. In the ordinary field. sampling of crude petroleum, washing with soap and rinsing with water may be eliminated.

5. Sampling Apparatus

5.1 Sampling apparatus is described in detail under each of the specific sampling procedures. Clean, dry, and free all sampling apparatus from any substance that might contaminate the material, using the procedure described in 4.3.

SAMPLING INSTRUCTIONS AND PRECAUTIONS

6. Time and Place of Sampling

6.1 Crude Petroleum: By mutual agreement, samples may be taken either from tanks or from pipelines. The pipeline samples may be obtained by either manual or mechanical methods as described in Sections 18 to 20, inclusive.

6.1.1 Stationary Tanks-Samples may be taken from tanks by mutual agreement as follows: composite spot, middle spot, all-levels. running samples, or by sample cocks. Additional samples may be taken as deemed necessary.

6.1.2 Skip or Barge Tanks—Samples of crude petroleum shall be taken as follows:

6.1.2.1 From the shore tanks before loading, and both before and after discharging. These shall be all-levels samples as described in 3.2 or running samples as described in 3.3.

6.1.2.2 From the pipeline during loading and discharging. These shall be taken by the procedures given in the Sections 18 to 20.

6.1.2.3 From the ship's tanks after loading and before discharging. An all-levels sample as described in 3.2 or a running sample as described in 3.3 shall be obtained from the ship's cargo tanks as follows:

Number of Grades	Minimum Number of Samples
1	3 compartments
Number of Compart- ments Containing One Grade	
l to 2	each compartment
3 to 6	2 compartments
7 or more	3 compartments

6.1.3 Except as specifically exempted, when loading a ship, shore tank samples shall be "official." However, ship's tank samples shall also be tested for water and sediment, and for other aspects of quality, when required; the results of these tests, together with tests of shore tank samples, shall be shown on the cargo certificate. Except when specifically exempted, when discharging a ship, ship's tank samples shall be "official." By mutual agreement, line samples may be used for the official samples, but samples from shore and ship tanks are preferred.

6.2 Finished Products—When loading or discharging finished products, take samples from both shipping and receiving tanks, and from the pipeline if required.

6.2.1 Ship or Barge Tanks—Sample each product after the vessel is loaded or just before unloading.

6.2.2 Tank Cars—Sample the product after the car is loaded or just before unloading.

NOTE 5—When taking samples from tanks suspected of containing flammable atmospheres, precautions should be taken to guard against ignitions due to static electricity. Metal or conductive objects, such as gage tapes, sample containers, and thermometers, should not be lowered into or suspended in, a compartment or tank which is being filled or immediately after cessation of pumping. A waiting period of approximately 1 min will generally permit a substantial relaxation of the electrostatic charge; under certain conditions a longer penod may be deemed advisable.

6.2.3 Package Lots (Cans. Drums. Barrels. or Boxes)—Take samples from a sufficient number of the individual packages to prepare a composite sample which will be representative of the entire lot or shipment. Select at random the individual packages to be sampled. The number of such random packages will depend upon several practical considerations, such as:

6.2.3.1 The tightness of the product specifications.

6.2.3.2 The source and type of the material and whether or not more than one production batch may be represented in the lot, and

6.2.3.3 Previous experience with similar shipments, particularly with respect to the uniformity of quality from package to package.

6.2.3.4 In most cases, the number specified in Table 3 will be satisfactory.

7. Obtaining Samples

7.1 Directions for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgment are necessary to ensure samples that represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as in cold weather, or when handling materials at high temperature, or for reasons of safety. Select wiping cloths so that lint is not introduced, contaminating samples.

7.2 As many petroleum vapors are toxic and flammable, avoid breathing them or igniting them from an open flame or a spark produced by static.

7.3 When sampling relatively volatile products (more than 2 lb (0.14 kgf/cm²) Reid vapor pressure (Rvp)), the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be upended into the opening of the sample container and remain in this position until the contents have

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been transferred so that no unsaturated air will be entrained in the transfer of the sample.

7.4 When sampling nonvolatile liquid products (2 lb (0.14 kgf/cm²) Rvp or less), the sampling apparatus shall be filled and allowed to drain before drawing the actual sample. If the actual sample is to be transferred to another container, the sample container shall be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

8. Handling Samples

8.1 Volatile Samples—It is necessary to protect all volatile samples of petroleum and petroleum products from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when the material is being transferred. When samples of more than 16 lb (1.12 kgf/cm²) Rvp are being obtained, be sure to use containers strong enough to meet local safety regulations. After delivery to the laboratory, volatile samples should be cooled before the container is opened.

8.2 Light-Sensitive Samples—It is important that samples sensitive to light, such as gasoline containing tetraethyllead, be kept in the dark, if the testing is to include the determination of such properties as color. tetraethyllead and inhibitor contents. sludgeforming characteristics, stability tests, or neutralization value. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately. It is a definite advantage to use covered cardboard cartons into which the sample bottles may be placed immediately after collection.

8.3 Refined Materials—Protect highly refined products from moisture and dust by placing paper, plastic, or metal foil over the stopper and the top of the container.

8.4 Container Outage—Never completely fill a sample container, but allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling and the probable maximum temperature to which the filled container may be subjected.

9. Shipping Samples

9.1 To prevent loss of liquid and vapors

during shipment, and to protect against moisture and dust, cover the stoppers of glass bottles with plastic caps that have been swelled in water, wiped dry, placed over the tops of the stoppered bottles, and allowed to shrink tightly in place. The caps of metal containers must be screwed down tightly and checked for leakage. Postal and express office regulations applying to the shipment of flammable liquids must be observed.

10. Labeling Sample Containers

10.1 Label the container immediately after a sample is obtained. Use waterproof and oilproof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing, and handling. Include the following information:

10.1.1 Date and time (the period elapsed during continuous sampling and the hour and minute of collection for dipper samples).

10.1.2 Name of the sampler,

10.1.3 Name or number and owner of the vessel, car, or container,

10.1.4 Brand and grade of material, and

10.1.5 Reference symbol or identification number.

SAMPLING PROCEDURES

11. General Information

11.1 The standard sampling procedures described in this method are summarized in Table 1. Alternative sampling procedures may be used if a mutually satisfactory agreement has been reached by the parties involved. Such agreement shall be put in writing and signed by authorized officials.

BOTTLE OR BEAKER SAMPLING

12. Application

12.1 The bottle or beaker sampling procedure is applicable for sampling liquids of 16 lb (1.12 kgf/cm²) Rvp or less in tank cars. tank trucks. shore tanks. ship tanks. and barge tanks. Solids or semiliquids that can be liquefied by heat may be sampled by this procedure, provided they are true liquids at time of sampling.

13. Apparatus

13.1 A suitable sampling bottle or beaker.

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as shown in Fig. 2, is required. Recommended uses and diameter of openings in the bottle or beaker are given in Table 4.

14. Procedure

14.1 All-Levels Sample-Lower the weighted, stoppered bottle or beaker as near. as possible to the draw-off level, pull out the stopper with a sharp jerk of the cord or chain and raise the bottle at a uniform rate so that it is about three-fourths full as it emerges from the liquid.

14.2 Running Sample—Lower the unstoppered bottle or beaker as near as possible to the level of the bottom of the outlet connection or swing line and then raise the bottle or beaker to the top of the oil at a uniform rate of speed such that it is about three-fourths full when withdrawn from the oil.

14.3 Upper, Middle, and Lower Samples-Lower the weighted, stoppered bottle to the proper depths (Fig. 1) as follows:

Lower sample	level of the fixed tank outlet or the swingline outlet
Middle sample	middle of the tank contents
Upper sample	middle of upper third of the tank con- tents

Pull out the stopper with a sharp jerk of the cord or chain and allow the bottle or beaker to fill completely at the selected level, as evidenced by the cessation of air bubbles. When full, raise the bottle or beaker, pour off a small amount, and stopper immediately.

14.4 Multiple Tank Composite Sample— Prepare a composite sample in the laboratory (not in the field) by mixing portions of the alllevels samples as specified in 3.15.

14.5 Composite Spot Sample—Prepare a composite spot sample by mixing spot samples in equal proportions as specified in 3.16. using either the three-way or the two-way procedure, whichever applies.

14.6 Middle Spot Sample—Obtain this sample in the manner specified in Section 3.17.

14.7 Top Sample—Obtain this sample (Fig. 1) in the same manner as specified in 10.3 but at 6 in..(150 mm) below the top surface of the tank contents.

14.8 Handling—Stopper and label bottle samples immediately after taking them, and deliver to the laboratory in the original sampling bottles.

TAP SAMPLING

15. Application

15.1 The tap sampling procedure is applicable for sampling liquids of 26 lb (1.83 kgf/cm²) Rvp or less in tanks which are equipped with suitable sampling taps or lines. This procedure is recommended for volatile stocks in tanks of the breather and balloonroof type, spheroids, etc. (Samples may be taken from the drain cocks of gage glasses, if the tank is not equipped with sampling taps.) The assembly for tap sampling is shown in Fig. 3.

NOTE 6—If Rvp is more than 16 lb (1.12 kgf/cm^3) but not more than 26 lb (1.83 kgf/cm^3) a cooling bath as shown in 44.2. Fig. 10, shall be used between the tank tap and the sample container to cool the sample and prevent volatilization of low-boiling components.

16. Apparatus

16.1 Tank Taps—The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height and extending at least 3 ft (1 m) inside the tank shell. A standard 14-in. pipe with suitable valve is satisfactory.

16.2 Sample Cocks-Samples of crude petroleum may be taken through sample cocks properly placed in the shell of the tank. The upper sample cock shall be located 18 in. (457 mm) below the top of the tank shell; the lower sample cock shall be located level with the bottom of the outlet connection or at the top of an upturned elbow or other similar fitting if installed on the outlet connection; and the middle sample cock shall be located halfway between the upper and lower sample cocks. An additional cock for the clearance sample should be located 4 in. (100 mm) below the bottom of the outlet connection to determine whether the level of merchantable oil is at least below this point. The sample cocks should be located a minimum of 6 ft (1.3 m) distant circumferentially from the pipeline outlet and drain connections, and 3 ft (2.4 m) from the filling line connection. The sample cocks should be of ¹⁴-in. (19 mm) size, and the lines should be of 4-in, nominal diameter for crude oil of 18 deg API gravity or less. For lighter oil. iz-in. (12.7-mm)-size cocks, with Le-in. nominal diameter lines, should be used. The lines should extend a minimum of 4 in. (102 mm) inside the tank shell. except on floating-roof tanks. where flush installations are necessary. All sample cocks should be equipped with sealable valves and plugged inspection tees.

16.2.1 On tanks of more than 10.000-barrel (1600-m³) capacity, at least two sets of sample cocks shall be installed. located equidistant apart, around the circumference of the tank. Five or more sample cocks should be installed per set, evenly spaced between lower and upper sample levels.

16.3 Tube-A delivery tube that will not contaminate the product being sampled and long enough to reach to the bottom of the sample container is required to allow submerged filling. When a cooling bath is used while tap sampling, a similar suitable tube should be used between the tank tap and the cooler inlet.

16.4 Sample Containers—Use clean. dry glass bottles of convenient size and strength to receive the samples. If the vapor pressure of the product to be sampled is between 16 and 26 lb (1.12 and 1.83 kgf/cm²) Rvp. protect the bottle with a metal cover until the sample is discarded. In some cases, such as the sampling of crude petroleum, metal containers may be used instead of glass bottles.

17. Procedure

17.1 Before a sample is drawn. flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

17.2 When a sample cooler is used during the tap sampling operation. Rush the tap (or gage glass drain cock). Then, using a section of clean tubing, connect the tap to the cooler inlet. Flush the cooler throughly, after which connect the clean delivery tube to the cooler outlet and proceed with the sampling operation.

17.3 In the sampling of crude petroleum, check for merchantable oil at the clearance sample cock. Flush each sample connection until all oil from the previous run has been

removed and the sample lines are filled with fresh oil from the tank.

17.3.1 On tanks of 10,000-barrel (1600-m³) capacity or smaller, samples of equal amounts shall be taken from the lower, middle, and upper sample connections. A measuring cup of proper size may be used to assure the drawing of the proper quantity from each sample cock.

17.3.2 On tanks of more than 10.000-barrel capacity, samples of equal amounts shall be taken from each of the sample connections at each set of sample connections.

17.3.3 All samples shall be mixed in equal proportions for a composite sample, or the samples may be tested separately and the results averaged.

17.3.4 When crude oil in a tank fails to reach the upper or middle sample cocks on a tank equipped with three sample cocks, it is suggested that the sample for the run be obtained as follows: if the level of the oil is nearer the upper sample cock than the middle, two thirds of the sample shall be taken from the middle sample cock and one-third from the lower. If the level of oil is nearer the middle sample cock than the upper, one half of the sample shall be taken from the middle and one half from the lower. If the level of the oil is below the middle sample cock, all of the sample shall be taken from the lower cock.

CONTINUOUS SAMPLING

18. Application

18.1 The continuous sampling procedure is applicable for sampling liquids of 16 (1.12 kgf/cm²) Rvp or less and semiliquids in pipelines, filling lines, and transfer lines. The continuous sampling may be done manually or by using automatic devices.

19. Apparatus

19.1 Sampling Probe—The function of the sampling probe is to withdraw from the flow stream a portion that will be representative of the entire stream. The apparatus assembly for continuous sampling is shown in Fig. 4. Probe designs that are commonly used are as follows:

19.1.1 A tube extending to the center of the line and beveled at a 45-deg angle facing up-

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stream (Fig. 4(a)).

19.1.2 A long-radius forged elbow or pipe bend extending to the center line of the pipe and facing upstream. The end of the probe should be reamed to give a sharp entrance edge. (Fig. 4(b)).

19.1.3 A closed-end tube with a round orifice spaced near the closed end which should be positioned in such a way that the orifice is in the center of the pipeline and is facing the stream as shown in Fig. 4(c).

19.2 Since the fluid pumped may not in all cases be homogeneous, the position and size of the sampling probe should be such as to minimize stratification or dropping out of heavier particles within the tube or the displacement of oil within the tube as a result of variation in gravity of the flowing stream. The sampling probe should be located preferably in a vertical run of pipe and as near as practicable to the point where the oil passes to the receiver. The probe should always be in a horizontal position.

19.2.1 The sampling lines should be as short as practicable and should be cleared before any samples are taken.

19.2.2 A suitable device for mixing the fluid flow to ensure a homogeneous mixture at all rates of flow and to eliminate stratification should be installed upstream of the sampling tap. Some effective devices for obtaining a homogeneous mixture are as follows: reduction in pipe size; a series of baffles: orifice or perforated plate; and a combination of any of these methods.

19.2.3 The design or sizing of these devices is optional with the user, as long as the flow past the sampling point is homogeneous and stratification is eliminated.

19.3 To control the rate at which the sample is withdrawn, the probe or probes should be fitted with valves or plug cocks.

19.4 Automatic Sampling Devices—If mutually agreeable, automatic line-sampling devices that meet the standards set out in 19.5 may be used to withdraw line samples for the purpose of determining gravity, water, and sediment, and other characteristics required in the custody transfer of crude petroleum. These devices may also be used in obtaining samples of petroleum products. The quantity of sample collected must be of sufficient size for analysis, and its composition should be identical with the composition of the batch flowing in the line while the sample is being taken. An automatic sampler installation necessarily includes not only the automatic sampling device that extracts the samples from the line, but also a suitable probe, connecting lines, auxiliary equipment, and a container in which the sample is collected. Automatic samplers may be classified as follows:

19.4.1 Continuous Sampler. Time Cycle (Nonproportional) Types—A sampler designed and operated in such a manner that it transfers equal increments of liquid from the pipeline to the sample container at a uniform rate of one or more increments per minute is a continuous sampler.

19.4.2 Continuous Sampler, Flow-Responsive (Proportional) Type—A sampler that is designed and operated in such a manner that it will automatically adjust the quantity of sample in proportion to the rate of flow is a flow-responsive (proportional) sampler. Adjustment of the quantity of sample may be made either by varying the frequency of transferring equal increments of sample to the sample container, or by varying the volume of the increments while maintaining a constant frequency of transferring the increments to the sample container. The apparatus assembly for continuous sampling is shown in Fig. 4.

19.4.3 Intermittent Sampler—A sampler that is designed and operated in such a manner that it transfers equal increments of liquid from a pipeline to the sample container at a uniform rate of less than one increment per minute is an intermittent sampler.

19.5 Standards of Installation—Automatic sampler installations should meet all safety requirements in the plant or area where used, and should comply with American National Standard Code for Pressure Piping, and other applicable codes (ANSI B31.1). The sampler should be so installed as to provide ample access space for inspection and maintenance.

19.5.1 Small lines connecting various elements of the installation should be so arranged that complete purging of the automatic sampler and of all lines can be accomplished effectively. All fluid remaining in the sampler and the lines from the preceding sampling cycle should be purged immediately

before the start of any given sampling operation.

19.5.2 In those cases where the sampler design is such that complete purging of the sampling lines and the sampler is not possible. a small pump should be installed in order to circulate a continuous stream from the sampling tube past or through the sampler and back into the line. The automatic sampler should then withdraw the sample from the sidestream through the shortest possible connection.

19.5.3 Under certain conditions, there may be a tendency for water and heavy particles to drop out in the discharge line from the sampling device and appear in the sample container during some subsequent sampling period. To circumvent this possibility, the discharge pipe from the sampling device should be free of pockets or enlarged pipe areas, and preferably should be pitched downward to the sample container.

19.5.4 To ensure clean, free-flowing lines, piping should be designed for periodic cleaning. When sampling waxy or high-viscosity oils, it may be necessary to heat the lines and sampler.

19.6 Field Calibration-Composite samples obtained from the automatic sampler installation should be verified for quantity performance in a manner that meets with the approval of all parties concerned, at least once a month and more often if conditions warrant. In the case of time-cycle samplers, deviations in quantity of the sample taken should not exceed =5 percent for any given setting. In the case of flow-responsive samplers, the deviation in quantity of sample taken per 1000 barrels of flowing stream should not exceed =5 percent. For the purpose of field-calibrating an installation, the composite sample obtained from the automatic sampler under test should be verified for quality by comparing on the basis of physical and chemical properties, with either a properly secured continuous nonautomatic sample or tank sample. The tank sample should be taken under the following conditions:

19.6.1 The batch pumped during the test interval should be diverted into a clean tank and a sample taken within 1 h after cessation of pumping.

19.6.2 If the sampling of the delivery tank is to be delayed beyond 1 h. then the tank selected must be equipped with an adequate mixing means. For valid comparison, the sampling of the delivery tank must be completed within 8 h after cessation of pumping, even though the tank is equipped with a motor-driven mixer.

19.6.3 When making a normal full-tank delivery from a tank. a properly secured sample may be used to check the results of the sampler if the parties mutually agree to this procedure.

19.7 Receiver—The receiver must be a clean, dry container of convenient size to receive the sample. All connections from the sample probe to the sample container must be free of leaks. Two types of container may be used, depending upon service requirements:

19.7.1 Atmospheric Container—The atmospheric container shall be constructed in such a way that it retards evaporation loss and protects the sample from extraneous material such as rain, snow, dust, and trash. The construction should allow cleaning, interior inspection, and complete mixing of the sample prior to removal. The container should be provided with a suitable vent.

19.7.2 Closed Container—The closed container shall be constructed in such a manner that it prevents evaporation loss. The construction must allow cleaning, interior inspection and complete mixing of the sample prior to removal. The container should be equipped with a pressure-relief valve.

20. Procedure

20.1 Nonautomatic Sample:

20.1.1 Adjust the valve or plug cock from the sampling probe so that a steady stream is drawn from the probe. Whenever possible, the rate of sample withdrawal should be such that the velocity of liquid flowing through the probe is approximately equal to the average linear velocity of the stream flowing through the pipeline. Measure and record the rate of sample withdrawal as gallons per hour. Divert the sample stream to the sampling container continuously or intermittently to provide a quantity of sample that will be of sufficient size for analysis. 20.1.2 In sampling crude petroleum, samples of V_2 pt (0.24 liter) or more should be taken every hour or less—whichever is thought necessary. By mutual agreement, the sample period may be increased to 2-h intervals. It is important that the size of the samples and the intervals between the sampling operations be uniform for each batch of material to be sampled.

20.1.2.1 The sample of crude petroleum should be placed in a closed container and at the end of a 24-h period, or at a predetermined period, the combined samples should be gently, yet thoroughly, mixed and a composite sample taken for test purposes. The sample container should be vapor-tight, equipped with a delivery tube extending through the top to within $\frac{1}{2}$ in. (12 mm) of the bottom, and with a funnel and positive closure for allowing submerged filling. The sample container should be stored in a cool, dry place; exposure to direct sunlight should be avoided.

20.1.2.2 Duplicate line samples obtained for gravity, and water and sediment tests may be taken, according to the instructions for sampling, and the tests made at that time. A record of the results of such tests should be kept and averaged for a 24-h period, or for a predetermined period. The composite or average of hourly samples is acceptable.

20.1.3 Label the sample and deliver it to the laboratory in the container in which it was collected.

20.2 Automatic Sampling-Purge the sampler and the sampling lines immediately before the start of -a-sampling operation. If the sample design is such that complete purging is not possible, circulate a continuous stream from the probe past or through the sampler and back into the line. Withdraw the sample from the side stream through the automatic sampler using the shortest possible connections. Adjust the sampler to deliver not less than one and not more than 40 gal (151 liters) of sample during the desired sampling period. For time-cycle samplers, record the rate at which sample increments were taken per minute. For 'flow-responsive samplers, record the proportion of sample to total stream. Label the samples and deliver them to the laboratory in the containers in which they were collected. Alternatively, in the case of

crude petroleum, the analysis of the sample may be performed in the field.

NOTE 7-When sampling semiliquids, heat the sampler lines, sampler, and receiver to a temperature just sufficient to keep the material liquid and to assure accurate operation of the sampling devices.

DIPPER SAMPLING

21. Application

21.1 The dipper sampling procedure is applicable for sampling liquids of 2 lb (0.14 kgf/cm²) Rvp or less and semiliquids where a free or open discharge stream exists. as in small filling and transfer pipelines (2-in. diameter or less) and filling apparatus for barrels, packages, and cans.

22. Apparatus

22.1 Dipper—Use a dipper with a flared bowl and a handle of convenient length, made of material such as tinned steel that will not affect the product being tested. The dipper should have a capacity suitable for the amount to be collected (Section 23) and must be protected from dust and dirt when not being used.

22.2 Sample Container-Use a clean, dry container of the desired size.

23. Procedure

23.1 Insert the dipper in the free-flowing stream so that a portion is collected from the full cross section of the stream. Take portions at time intervals chosen so that a complete sample proportional to the pumped quantity is collected. The gross amount of sample collected should be approximately 0.1 percent. but not more than 40 gal (105 liters), of the total quantity being sampled. Transfer the portions into the sample container as soon as collected. Keep the container closed. except when .pouring a.dipper portion into it. As soon as all portions of the sample have been collected, close and label the sample container, and deliver it to the laboratory.

TUBE SAMPLING

24. Application

24.1 The tube sampling procedure is applicable for sampling liquids of 2 lb (0.14 kgf/cm²) Rvp or less and semiliquids in drums, barrels, and cans.

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25. Apparatus

25.1 Tube—Either a glass or metal tube may be used, designed so that it will reach to within about v_s in. (3.2 mm) of the bottom and have a capacity of approximately 1 pt (0.5 liters) or 1 qt (1 liter). A metal tube suitable for sampling 50-gal (190-liter) drums is shown in Fig. 5. Two rings soldered to opposite sides of the tube at the upper end are convenient for holding it by slipping two fingers through the rings, thus leaving the thumb free to close the opening.

25.2 Sample Containers-Use clean. dry cans or glass bottles.

26. Procedure

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26.1 Drums and Barrels-Place the drum or barrel on its side with the bung up. If the drum does not have a side bung, stand it upright and sample from the top. If detection of water, rust or other insoluble contaminants is desired. let the barrel or drum remain in this position long enough to permit the contaminants to settle. Remove the bung and place it beside the bung hole with the oily side up. Close the upper end of the clean, dry sampling tube with the thumb, and lower the tube into the oil for a depth of about 1 ft (0.3 m). Remove the thumb, allowing oil to flow into the tube. Again close the upper end with the thumb and withdraw the tube. Rinse the tube with the oil by holding it nearly horizontal and turning it so that the oil comes in contact with that part of the inside surface that will be immersed when the sample is taken. Avoid handling any part of the tube that will be immersed in the oil during the sampling operation. Discard the rinse oil and allow the tube to drain. Insert the tube into the oil again, holding the thumb against the upper end. (If an all-levels sample is desired, insert the tube with the upper end open.) When the tube reaches the bottom, remove the thumb and allow the tube to fill. Replace the thumb. withdraw the tube quickly and transfer the contents to the sample container. Do not allow the hands to come in contact with any part of the sample. Close the sample container: replace and tighten the bung in the drum or barrel. Label the sample container and deliver it to the laboratory.

26.2 Cans-Obtain samples from cans of 5-gal (19-liter) capacity or larger in the same manner as from drums and barrels (26.1). using a tube of proportionately smaller dimensions. For cans of less than 5-gal capacity. use the entire contents as the sample, selecting cans at random as indicated in Table 3 or in accordance with agreement between the purchaser and the seller.

THIEF SAMPLING

27. Application

27.1 The thief sampling procedure is applicable for obtaining bottom samples (Fig. 1). or of semiliquids in tank cars and storage tanks.

27.2 The thief is also widely used in sampling crude petroleum in storage tanks. In this application it may be used for taking samples at different levels as well as for bottom samples of nonmerchantable oil and water at the bottom of the tank.

28. Apparatus

28.1 Thief-The thief shall be designed so that a sample can be obtained within 1/2 in.. (13 mm) of the bottom of the car or tank. Two types of thiefs are illustrated in Fig. 6. One type is lowered into the tank with valves open to permit the oil to flush through the container. When the thief strikes the bottom of the tank, the valves shut automatically to trap a bottom sample. The other type has a . projecting stem on the valve rod which opens the valves automatically as the stem strikes the bottom of the tank. The sample enters the container through the bottom valve and air is released simultaneously through the top. The valves snap shut when the thiel is withdrawn. A core-type thief similar to that shown in Fig. 6 (b), with a uniform cross section and bottom closure and with a capacity depending upon the size of the sample required, may be used for sampling crude petroleum. The thief should be capable of penetrating the oil in the tank to the required level, mechanically equipped to permit filling at any desired level, and capable of being withdrawn without undue contamination of the contents. The thief may be equipped with the following:

28.1.1 Sample cocks for obtaining samples for the determination of water and sediment.

28.1.2 Extension rods for use in obtaining samples at levels corresponding with require-

ments for high connections or for samples to determine high settled water and sediment levels.

28.1.3 Water and sediment gage for determining the height of water and sediment in the thief.

28.1.4 Windshield to be used when taking the gravity and temperature of the oil.

28.1.5 Opener to break the tension on the valve or slide at any desired level.

28.1.6 A thief cord marked so that sample can be taken at any depth in the vertical cross section of the tank, and

28.1.7 Hook to hang the thief in the hatch vertically.

28.2 Sample Containers—Use clean, dry cans or glass bottles.

29. Procedure

29.1 Lower the clean, dry thief through the dome of the tank car or tank hatch until it strikes the bottom. When full, remove the thief and transfer the contents to the sample container. Close and label the container immediately, and deliver it to the laboratory.

29.2 In the sampling of crude petroleum, lower the clean, dry thief slowly into the oil to the desired depth, trip the thief to secure the sample and raise slowly to avoid agitation. The proper depths for sampling are described in Section 3.

BORING SAMPLING

30. Application

30.1 The boring sampling procedure is applicable for sampling waxes and soft solids in barrels, cases, bags, and cakes when they cannot be melted and sampled as liquids.

31. Apparatus

31.1 Ship Auger-Use a ship auger ¼ in. (19 mm) in diameter, similar to that shown in Fig. 7, and of sufficient length to pass through the material to be sampled.

31.2 Sample Containers—Use clean, widemouth, metal containers or glass jars with covers.

32. Procedure

32.1 Remove the heads or covers of barrels and cases. Open bags and wrappings of cakes. Remove any dirt. sticks. string. or other for-

eign substances from the surface of the material. Bore three test holes through the body of the material, one at the center, the other two halfway between the center and the edge of the package on the right and left sides, respectively. If any foreign matter is removed from the interior of the material during the boring operation, include it as part of the borings. Put the three sets of borings in individual sample containers, label and deliver them to the laboratory.

33. Laboratory Inspection

33.1. If there are any visible differences in the samples, examine and test each set of borings at the laboratory. Otherwise, combine the three sets of borings into one sample. If subdivision of the borings is desired, chill, pulverize (if necessary), mix, and quarter the borings until reduced to the desired amount.

GRAB SAMPLING

34. Application

34.1 The grab sampling procedure is applicable for sampling all lumpy solids in bins. bunkers. freight cars. barrels. bags. boxes. and conveyors. It is particularly applicable for the collection of green petroleum coke samples from railroad cars and for the preparation of such samples for laboratory analysis. Refer to ASTM Method D 346. Sampling Coke for Analysis.⁴ when other methods of shipping or handling are used.

34.2 Place of Sampling—Petroleum coke may be sampled while being loaded into railroad cars from piles or after being loaded into railroad cars from coking drums.

35. Apparatus

35.1 Sample Container.—A polyethylene pail of approximately 10-qt (10-liter) capacity.

35.2 Scoop, stainless steel or aluminum. No. 2 size.

36. Procedure

36.1 Sampling—Lumpy solids are usually heterogeneous and therefore are difficult to sample accurately. It is preferable to take samples during the unloading of cars, or during transit of the material by conveyors. From material in transit, obtain a number of portions at frequent and regular intervals and compine them.

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36.2 When sampling from railroad cars. use one of the following procedures:

36.2.1 Being Loaded from a Pile-Take a full scoop of sample at each of the five sampling points shown in Fig. 8 and deposit in the polyethylene pail. Cover the sample and deliver to the laboratory. Each sampling point shall be located equidistant from the sides of the railroad car.

36.2.2 Railroad Cars After Direct Loading from Coking Drums—At any five of the sampling points shown in Fig 9, take a full scoop of coke from about 1 ft (0.3 m) below the surface and deposit it in the polyethylene pail. Cover the sample and deliver to the laboratory.

36.3 When sampling from conveyors, take one scoop for each 8 to 10 tons (725 to 900 kg) of coke transported. These samples may be handled separately, or composited after all samples representing the lot have been taken.

36.4 When sampling from bags, barrels, or boxes, obtain portions from a number of packages selected at random as shown in Table 3, or in accordance with the agreement between the purchaser and the seller:

36.5 Quartering-Carefully mix the sample and reduce it in size to a convenient laboratory sample by the quartering procedure described in Method D 346. Perform the quartering operation on a hard, clean surface, free from cracks, and protected from rain, snow, wind, and sun. Avoid contamination with cinders. sand, chips from the floor, or any other material. Protect the sample from loss or gain of moisture or dust. Mix and spread the sample in a circular layer, and divide it into quadrants. Combine two opposite quadrants to form a representative reduced sample. If this sample is still too large for laboratory purposes. repeat the quartering operation. In this manner, the sample will finally be reduced to 2 representative, suitable size for laboratory purposes. Label and deliver the sample to the laboratory in a suitable container. Subdivision may be made in the laboratory by using a riffic sampler as described in ASTM Method D 271. Laboratory Sampling and Analysis of Coal and Coke

GREASE SAMPLING

37. Application

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37.1 This method covers practices for ob-

taining samples representative of production lots or shipments of lubricating greases, or of soft waxes or soft bitumens similar to grease in consistency. The procedure is necessarily quite general to cover the wide variety of conditions encountered, and may require modification to meet individual specifications. Proceed in accordance with Sections 4 to 10, particularly those pertaining to precautions, care, and cleanliness, except where they conflict with Sections 38 to 40.

38. Inspection

38.1 If the material is a lubricating grease and inspection is made at the manufacturing plant, take samples from the finished shipping containers of each production batch or lot. Never take grease samples directly from grease kettles, cooling pans, tanks, or processing equipment. Do not sample the grease until it has cooled to a temperature not more than 15 F (8 C) above that of the air surrounding the containers and until it has been in the finished containers for at least 12 h. When the -containers for a production batch of grease are of different sizes, treat the grease in each size of container as a separate lot. When inspection is made at the place of delivery. obtain a sample from each shipment. If a shipment consists of containers from more than one production batch (lot numbers). sample each such batch separately.

38.2 If the material being inspected is of grease-like consistency, but is not actually a lubricating grease, but some mixture of heavy hydrocarbons such as microcrystalline waxes or soft bitumens, it will be permissible to take samples from pans, tanks, or other processing eouipment, as well as from containers of the finished product. The grease sampling method shall be applicable to such stocks only if for some reason it is not possible to apply heat and cenvert the material into a true houid.

39. Sample Size

39.1 Select containers at random from each lot or shipment to give the desired quantity specified in Table 5.

40. Procedure

40.1 Inspection-Examine the opened containers to determine whether the grease is homogeneous, comparing the grease nearest the outer surfaces of the container with that in the center, at least 6 in. (150 mm) below the top surface, for texture and consistency. When more than one container of a lot or shipment is opened, also compare the grease in the respective containers.

40.2 Sampling-If no marked difference in the grease is found, take one portion from the approximate center and at least 3 in. (76 mm) below the surface of each opened container, in sufficient quantity to provide a composite sample of the desired quantity (Table 5). Withdraw portions with a clean scoop, large spoon, or spatula and place them in a clean container. Very soft, semifluid greases may be sampled by dipping with a 1-1b (0.5-kg) can or suitable dipper. If any marked difference in the grease from the various locations of an opened container is found, take two separate samples of about 1 lb each, one from the top surface adjacent to the wall, and the other from the center of the container, at least 6 in. (150 mm) below the top surface. If any marked variations are noted between different containers of a lot or shipment, take separate samples of about I ib (0.5 kg) from each container. When more than one sample of a batch or shipment is taken because of lack of uniformity, send them to the laboratory as separate samples.

40.3 Handling Samples-If more than one portion is required to represent a lot or shipment of grease softer than 175 penetration (see ASTM Method D 217, Test for Cone Penetration of Lubricating Greases.² prepare a composite sample by mixing equal portions thoroughly. Use a large spoon or spatula in a clean container. Avoid vigorous mixing or working of air into the grease. As grease samples become partially "worked" in being removed from containers, the procedure is not suitable for obtaining samples of greases softer than 175 penetration on which unworked penetration is to be determined. For greases having a penetration less than 175, out samples from the container with a knife in the form of blocks about 6 by 6 by 2 m. (150 by 150 by 50 mm). If required, make unworked penetration tests on blocks is procured, and other inspection tests on grease out from the blocks.

SAMPLING INDUSTRIAL AROMATIC HYDROCARBONS

41. Application

41.1 For obtaining samples of industrial aromatic hydrocarbons (benzene, toluene, xylene, and solvent naphthas), proceed in accordance with Sections 4 to 6, particularly those pertaining to precautions, care, and cleanliness.

SAMPLING FOR SPECIFIC TESTS

42. Special Precautions

42.1 Special sampling precautions and instructions are required for some ASTM methods of test and specifications. Such instructions, Sections 43 to 48, supplement the general procedures of this method and supersede them if there is a conflict. ASTM methods in this category are as follows:

Sections

ASTM Methods of Test for:	
D 216-Distillation of Natural Gaso-	
line ⁴	43
D 323-Vapor Pressure of Petroleum	
Products (Reid Method)2	11
D 525-Oxidation Stability of Gaso-	
line (Induction Period Method) ³	45
D 873-Oxidation Stability of Avia-	-,
tion Fuels (Potential Residue	
Method) ⁴	45
D 268-Volatile Solvents for Use in	• 1
Paint, Varmish, Lucquer, and Re-	
lated Products*	46
D 1856-Recovery of Asphalt from	40
Solution oy Abson Method, * or. D	
2172-Quantitative Extraction of	
Bitumen from Situminous Paving	
Mixtures*	
D 244-Emulsified Asphalts*	47
	-48

43. Distillation of Natural Gasoline

43.1 When obtaining samples of natural gasoline which are to be tested using Method D 216, the bottle sampling procedure. Section 14 is preferred. Before obtaining the sample, precool the bottle by immersing it in the product, allow it to fill, and discard the first filling. If the bottle procedure cannot be used, obtain the sample by the tap procedure, and with the use of the cooling bath, as described in Section 17. Do not agitate the bottle while

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^{*} Annual Book LI ASTM Siandards. Part 1:

drawing the sample. After obtaining the sample, close the bottle immediately with a tightfitting stopper and store it in an ice bath or refingerator at a temperature of 32 to 40 F (0 to 4.5 C).

44. Reid Vapor Pressure

44.1 When sampling products that are to be tested using Method D 323, observe the following precautions and instructions:

44.1.1 Precautions-Vapor pressures are extremely sensitive to evaporation losses and to slight changes in composition. When obtaining, storing, or handling samples, observe the necessary precautions to ensure samples representative of the product and satisfactory for Reid vapor pressure tests. Official samples should be taken by, or under the immediate supervision of a person of judgment, skill, and sampling experience. If sampling or sample requirements for other tests differ from those described in 44.2 to 44.10, obtain a separate sample for the Reid vapor pressure test. Never prepare composite samples for this test. Make certain that containers which are to be shipped by common carrier conform to I.C.C., state, or local regulations. When flushing or purging lines or containers, observe the pertinent regulations and precautions against fire. explosion, and other hazards.

44.1.2 Cooling Bath—A bath (Fig. 10) of sufficient size to hold the sample container and a cooling coil of about 25 ft (8 m) of copper tubing ($\frac{3}{15}$ in. (9 mm) or less outside diameter) shall be required when using the procedure described in 44.1.7. One end of the coil is provided with a connection for attaching it to the tank sampling tap or valve. The other end is fitted with a suitable valve (outlet) of good quality. A removable copper tube of $\frac{3}{15}$ in. or less outside diameter and of sufficient length to reach the bottom of the sample container shall be connected to the open end of the outlet valve.

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44.1.3 Sample Containers—Use containers of not less than 1 qt (1 liter) nor more than 2gal (7.5-liter) capacity, of sufficient strength to withstand the pressures to which they may be subjected, and of a type that will permit replacement of the cap or stopper with suitable connections for transferring the sample to the gasoline chamber of the vapor pressure apparatus. Open-type containers have a single opening which permits sampling by immersion. Closed-type containers have two openings, one in each end (or the equivalent thereof), fitted with valves suitable for sampling by water displacement or by purging.

44.1.4 Transfer Connections-The transfer connection for the open-type container consists of an air tube and a liquid delivery tube assembled in a cap or stopper. The air tube extends to the bottom of the container. One end of the liquid delivery tube is flush with the inside face of the cap or stopper and the tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred to the chamber. The transfer connection for the closed-type container consists of a single tube with a connection suitable for attaching it to one of the openings of the sample container. The tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred.

44.1.5 Sampling Open Tanks-Use clean containers of the open type when sampling open tanks and tank cars. An all-level sample obtained by the bottle procedure. Section 10, is recommended. Before taking the sample. fluish the container by immersing it in the product to be sampled. Then obtain the sample immediately. Pour off enough so that the container will be 70 to 80 percent full and close it promptly. Label the container and deliver it to the laboratory.

44.1.6 Sampling Closed Tanks—Containers of either the open or closed type may be used to obtain samples from closed or pressure tanks. If the open type is used, follow the cooling bath procedure described in 44.1.7. If the closed type is used, obtain the sample using the water displacement procedure, (44.1.8), or the purging procedure, (44.1.9). The water displacement procedure is preferable because the flow of product involved in the purging procedure may be hazardous.

44.1.7 Cooling Bath Procedure—When using a container of the open type, keep 11 at a temperature of 32 to 40 F (0 to 4.5 C) during the sampling operation by using the cooling bath (Fig. 10). Connect the coil to the tank sampling tap or valve and flush it with a

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samples from at least 5 percent of the number of containers in any shipment. The number of containers to be sampled may be increased at the discretion of the purchaser. In the case of expensive solvents which are purchased in small quantities, it is recommended that each container be sampled. Withdraw a portion from the center of each container to be sampled with a clean tube (Section 26) or weighted bottle 14.2. (A smaller bottle may be used.) Prepare a composite sample of at least 1 qt (1 liter) by mixing equal portions of not less than 1 pt (0.5 liter) from each container sampled.

47. Asphaltic Materials

47.1 When sampling asphaltic materials that are to be tested using Method D 1856. or. ASTM Method D 2172, obtain samples by the boring procedure (Section 32) or the grab procedure (Section 36). A sample of sufficient size to yield at least 100 g of recovered bitumen is required. About 1000 g of sheetasphalt mixtures usually will be sufficient. If the largest lumps in the sample are 1 in. (25 mm). 2000 g will usually be required, and still larger samples if the mixtures contain larger aggregates.

48. Emulsified Asphalts

48.1 It is frequently necessary to test samples in accordance with the requirements of ASTM Specifications D 977, for Emulsified Asphalt.' and Method D 244. Obtain samples from tanks, tank cars, and tank trucks by the bottle sampling procedure, Section 14, using a wide mouth (11/2-in. (38-mm) or greater) bottle. Use the dipper procedure, Section 23, to obtain samples from filling or discharge lines. Sample packages in accordance with Table 3. If the material is solid or semisolid, use the boring sampling procedure, Section 33. Obtain at least 1 gal (4 liters) or 10 lb (4.5 kg) from each lot or shipment. Store the samples in clean, airtight containers at a temperature of not less than 4 C until tested. Use glass or black iron containers for emulsified asphalts of the RS-I type.

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TABLE 1	Summary of Sampling Procedures and Applicability

Application	* Type of Container	Procedure	Section 44	
Liquids of more than 16 lb (1.12 kgf/cm ⁴) and no more than 26 lb (1.83 kgf/cm ⁴) Ryo	storage tanks, ship and barge tanks, tank cars, tank trucks	precoaled bottle sam- pling		
Liquids of more than 16 lb and not more than 26 lb Ryp	storage tanks with taps	cooler tap sampling	45	
Liquids of more than 2 lb (0.14 kgf/cm ²) and not more than 18 lb Rvp	storage tanks, ship and barge tanks, tank cars, tank trucks	bottle sampling	12	
Liquids of more than 2 lb and not more than 16 lb Rvp	storage tanks with taps	tap sampling .	t 5	
Liquids of 16 lb or less Ryp	pipes or lines	continuous line sam- oling	18	
Liquids of 2 lb or less Ryp	storage tanks, ship and barge	F J		
Liquids of 2 lb or less Ryp	storage tanks with taps -	bottle sampling	12	
Liquids of 2 lb or less Ryp	free or open-discharge streams	tap sampling	15	
Liquids of 2 lb or less Rvp	drums, barrels, and cans	dipper sampling	21	
Bottom or thief sampling of liquids of 2		tube sampling	24	
Ib Ryp or less	tank cars, storage tanks	thief sampling	27	
Liquids and semiliquids of 2 lb Rvp or less	free or open-discharge streams. Open tanks or kettles or tanks or kettles with open heads: tank cars and tank trucks: drums	dipper sampling	21	
Crude petroleum	pipe lines	continuous line sam- pling	13	
Crude petroleum	storage tanks, ship and barge tanks,	thief sampling	27	
	tank cars, tank trucks	bottle sampling		
		tap sampling	12	
Industrial aromatic hydrocarbons	storage tanks, ship and barge tanks		15	
Waxes, solid bitumens, and other soft	barrels, cases, bags, caxes	bottle sampling	12	
solids	P	boring sampling	20	
Petroleum coke: lumpy solids	freight cars, conveyors, bags, barrels, boxes	grab sampling	J 4	
Greases, soft waxes, soft asphalts	kettles, drums, cans, tubes	grease sampling	37	
Asphaltic materials	storage tanks, tank cars, lines, pack-	Sterre stripping	47	
-	ages		47	
Emulsified asphalts	storage tanks, tank cars, lines, pack- ages	-	48	

TABLE 2 Sampling Instructions for Horizontal Cylindrical Tanks

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Liquid Depth, percent of di-	Sampling Level. percent of diameter above bottom			Composite Sample, proportionate parts of		
ameter	Upper	Middle	Lower	Upper	Middle	Lower
100	50	50	20	3	4	1
90	75	50	20	3	4	ĩ
30	70	50	20	2	ŝ	1
70		50	20	_	6	1
60	• • •	50	20		š	
50		40	20		Ĺ.	6
40			20		-	ĩõ
30			15		•••	10
20			10		••	10
10			5			10

Number of Packages in Lot	Number of Packages to be Sampled	Number of Packages in Lot	Number of Packages to be Sampled
1 to 3	all	1332 to 1728	12
4 to 64	4	1729 to 2197	17
65 to 125	5	2198 to 2744	13
126 to 216	6	2745 to 3375	14
217 to 343			15
344 to 512	,	3376 to 4096	16
513 to 729	8.	4097 to 4913	17
	9	4914 10 5832	18
730 to 1000	10	5833 to 6859	19
1001 to 1331	11	6860 or over	20

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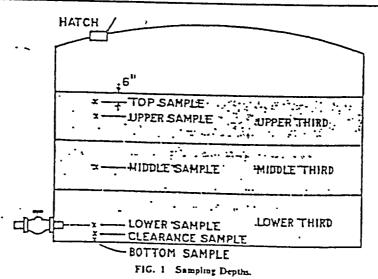
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TABLE 4 Weighted Sampling Bottle or Beaker

Matenal	Diameter of Opening, in. (mm)	
Light lubricating oils, kerosines, gaso- lines, transparent gas oils, diesel fuels, and distillates	3/4 (19 mm)	
Heavy lubricating oils, nontransparent	1½ (38 mm)	
Light crude oils (less than 200 s Say- bolt Universal Viscosity at 100 F (37.8 C))	¾ (19 mm)	
Heavy crude and fuel oils	(38 mm) دا	

TABLE 5 Size of Grease Samples

Container	Lot or Shipment	Minimum Sample		
Tubes or packages, less than 1b 1-lb cans 5 or 10-lb cans Larger than 10 lb Larger than 10 lb Larger than 10 lb	ali ali less than 10 000 lb 10 000 to 50 000 lb more than 50 000 lb	enough units for a 2-lb sample three cans one can 2 to 3 lb from one or more containers 2 to 5 lb from two or more containers 2 to 5.lb from three or more containers		



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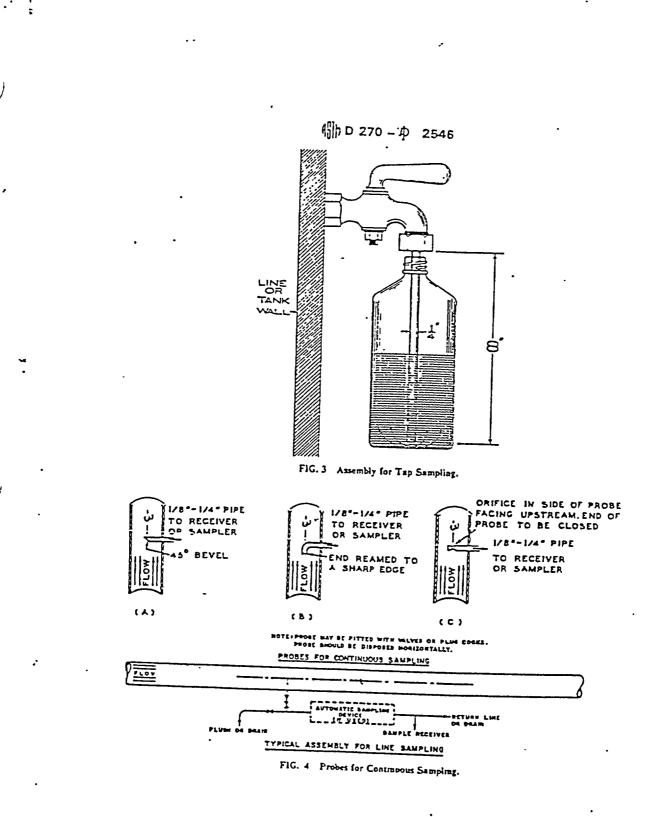
街D 270 - ₽ 2546 C CLOVE HITCH COR WASHER EYELET PIN THREADED AT BOTTOM CORK NUT WASHER Э -13/ 1/8"COPPER WIRE-HANDLE 12 ł 23/1 노요. LEAD WEIGHT I QUART WEIGHTED BOTTLE CATCHER COPPER⁻ WIRE LUGS (CAN BE FABRICATED TO FIT ANY SIZE BOTTLE) ALTERNATE RIG "WEI ---3" (a) 1-Qt Weighted Bottles 0. 1/8" SHEET 31/4" LEAD 10) I-Qt Weighted Beaker. Metric Equivalents

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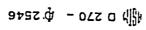
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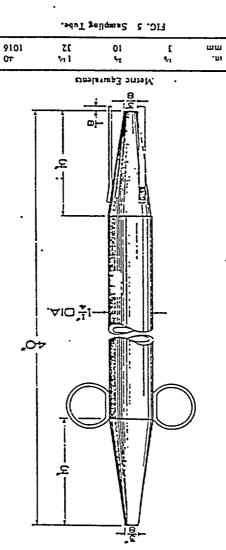
FIG. 2 Assembly for Bottle Samoling.



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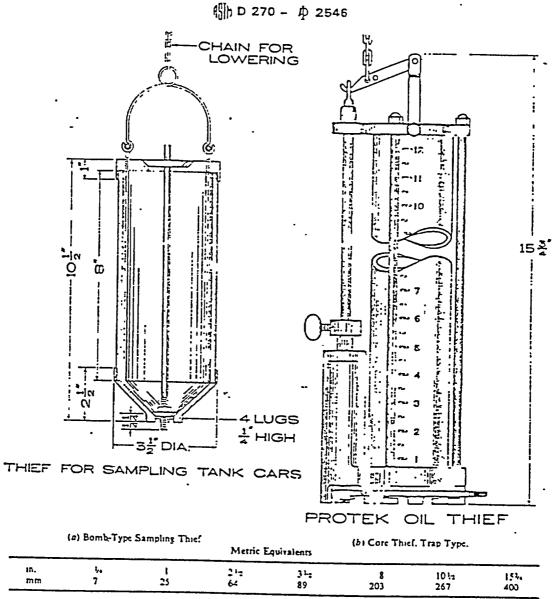
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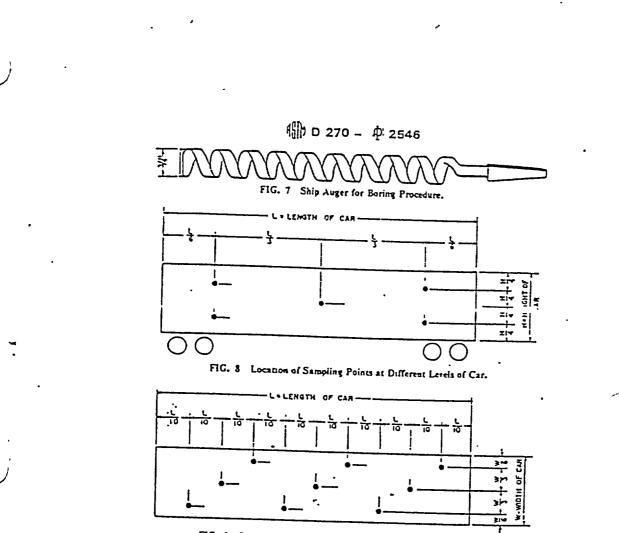
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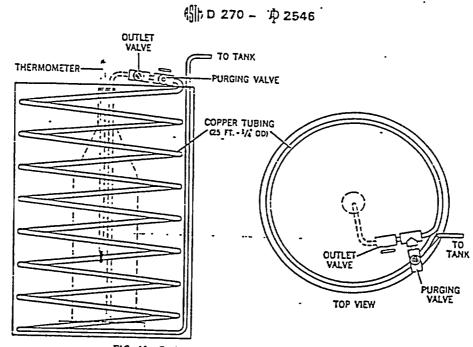
FIG. 6 Sampling Thiefs.



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FIG. 9 Location of Samoling Points from Exposed Surface of Car.

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FIG. 10 Cooling Bath for Reid Vapor Pressure Sampling.

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.

PREPARATION FOR SAMPLING

1. Freon Rinse

- A. Bombs
- B. Beakers
- C. Cylinder
- D. Carbouys
- E. Glass Bottles
- 2. Label Carbouy

7-day tank ID, 2BB, 1BB, 1AA, 2AA

3. Label Glass Beaker

Date/Sampling time Sample ID (2BB, 1BB, etc.)/Name (SQN/Gary L. Fiser/843-6714)

II. SAMPLING

1. For each saddle tank:

1.1 Top sample (Bomb fully submerged)

1.1.1 Fill and drain: Bomb → beaker → cylinder → return tank
1.1.2 Sample fill: A) Bomb → beaker (300 ml) → cylinder (300 ml) → carbouy
B) Return excess sample to tank

1.2 Middle sample (Bomb to 3 foot mark)

1.3 Lower sample (Bomb to bottom) .

1.3.1 Fill and drain: Bomb → beaker → cylinder → return tank
1.3.2 Sample fill: A) Bomb → beaker (300 ml) → cylinder (300 ml) → carbouy
B) Return excess to tank

- 2. Repeat step II.1 for each saddle tank and add composites to labeled carbouy.
- 3. Thoroughly mix carbouy and quickly fill labeled glass bottle.
- 4. Deliver glass bottle to the Central Lab for analysis.

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WORK REQUEST SUBMI If Tag Not Placed Leave Attached To Card W.R. SUBMITTED W.R. SUBMITTED NO. **B**762535 NO. B 7,62535 DATE DATE: PRIORITY. NO. Im. Arcn. PRIORITY: Routine 2 NO. Routine **B**762535 Atten Emer. Ιm 02 В 271930 į. П ASSIGNED TO: DATE: Honch 8 Day/4Year 89 ASSIGNED TO: NPRDS NOED ID Month & Day/Syear 8 NPRDS MAINT С MAINT HIST. 🗆 ıka: C i C MAINT MAINT. HIST. K MODIF MOEOSO OPERATOR AUTHORIZATION YES ONO MODIF MOEOSO cssc 10CFR50.49 OPERATOR AUTHORIZATION OTHER 6 cssc 10CFR50.49 Non-CSSC 🛛 YES NOD OTHER Non-CSSC YES NOD YES 🖸 NO 🗖 EQUIPMENT IDENTIFIER: EQUIPMENT IDENTIFIER: 2 8 RII ν A 0 Ų U FUNCTION SYSTEM ADDRESS S U FUNCTION SYSTEM ADDRESS NAME NAME CLASE TAW EOUIP EQUIP シト COLUMN COLUMN LOCATION 724 ХR 2AA + 2*BB* 16 Caus 2 YES D NO LOC EXPIRES: HRS. 🗆 DAYS ວິ NOK LOC EXPIRES: YES 🔲 HRS. 🗆 DAYS [REQUESTED FOUF UORK ACCNHOANICS WORK COm הס In 20 EXT SECT SUPV. INIT Jup Las ORIC SECT SUPV. HAR . Cíc, her 7430 6930 SHOULD SHOULD 8-15 SHOULD WORK J व्राप्छ let 1= - 14. SHOULD 2011-2 ---

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08/16/1939 14:47 TVA CLSB 615 697 4137 P.02 Page InF2 CHATTANOOGA POWER SERVICE CENTER DIESEL FUEL ANALYSIS DATE <u>8-15-89</u>. SEQUOYAH _ NUCLEAR PLANT 89-3935 Lale Ha. *89-3937 89-3936 89-3938 8-15-84 . 8-15-89 8-15-89 8-15-89 . 521 IAA - 7- day IBB. 7-dzy JAA . 7-day 2BB J-day ianzification. Liziting TZLK T2nK Tank . TanK Requirement. SNP-4005 SNP=4006 5NP . 4007 5NP-4008 lash Foint. ^OF 123/=in. loud Point, "F 23/<u>==</u>. ate: and sediment, .05 .05 .25 .0.2 oluze Percant 0.05/22%. aroca residue on . بسنسید 0 Percant Residua 0.33/=zx. سيم . ` sh, Weight Percent 0.01/===. istiliation, Temp. of 540/===. O Percent Point 640/=zx. 'iscesity at 40C/100F .2,8/34.8 1.9-4.1 CET 2.8 /34.8 Z.8/34K8 2.8 134.8 instruc/Savbolt . · <u>-</u> julfur. Persent by Wc. 0.5/=zr. /Strip Corresion 3/2ex. ٩ latane Number (Calc.) 40/=1=. Accelerated Oxid. Test 41 **K** I 1.1 2/=<u>2x</u>. cz. pe= 100 ml. Particulate contamina-· >10/=2x. · • • • • • cion, Method A mg/liter Clear and Bright · Pass/Fail ----27-39 API Gravity COMMENTS: •.• .* 8-15-89,20:00 DATE PECEIVED: BLH 8-15-89', 21:30 by phune -DATE H-FOLIED: Ben L. Hartman, A.S. Gar PERFORMED EY : Jonn LL Rose, Jr., Sugersisor Chamical Laboratory Section DISTRIBUTION: RIMS, MR 4N72A-C. Don Amos, Of \$5-4, SNR Leonard Bush, POB-2; UNS, SNP Danny Cross POB-2, OPS, SNP F1000312 W.L. Fison Of PS-4 SNF Ted Gatewood, OPS-E.718, SNP TOTAL P.02

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1	TO : <u>G.h.Fiser</u> Revision 12
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La man	FROM : Supervisor, R egulatory Licensing Section (RLS) NER DATE : 8/11/87
~ *	SUBJECT: NUCLEAR EXPERIENCE REVIEW - ITEM
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त्र	PRIORITY:IMMEDIATE ATTENTIONPROMPT ATTENTIONNORMAL ATTENTION DUE DATE 8/23/89
-	Please review and evaluate the attached information and answer the appropriate disposition statement below. Please coordinate your response with the other sections listed above, if necessary, and return this form to RLS by the above due date. Please coordinate any necessary due disposition statement
ſ	prior to them becoming overdue.
	Disposition Statements part attached recommendations
	1. Upon review of the subject NER material/do you recommend any action be taken? <u>Check One</u>
4	() Yes Provide recommended action including implementing organization and due dates for each action below. Provide all CAQR numbers below which have been initiated as a result of your review.
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¥	() No Because:
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Tanta	2. If part 1 above was checked yes, please answer the following questions:
¥	a. Do these recommendations affect the 10 CFR 50.49 program? () Yes () No If yes, coordinate with the EQ coordinator (EQ coordinator initials)
	b. Do these recommendations affect the SQN FSAR? () Yes () No If yes, specify which section(s) of the FSAR:
·. ¶	c. Do these recommendations require a revision to a vendor manual? () Yes () No
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ATTACHMENT A TO SQA 26 ATTACHMENT 1

SUBJECT: ATTACHMENT 1 TO SQA 26; DISPOSITION STATEMENTS, number 1. states "Upon review of the subject NER material do you recommend any action be taken?" Under the Check One "NO" category "Because:", "Check one:", "Already covered by instruction(s)".

In regard to the subject question on SQA26's attachment 1, we request that when the response to a Nuclear Experience Review (NER) item is that the item is already covered by an instruction:

- 1. The applicable section, paragraph, etc. of the instruction(s) which pertains to the NER item be referenced.
- 2. A photostatic copy of the cover page of the applicable procedure, the applicable page(s) of the procedure, with applicable portion(s) highlighted that resolve the NER item be included with the SQA26 Attachment 1.

The reason for this request is that the Procedures Staff will be annotating the applicable portion of the procedure so that in the future the procedure will not be revised and the portion(s) that are satisfying an NER item will not inadvertently be omitted.

If the entire procedure is deemed as required to satisfy the NER item then the NER item will be referenced against the procedure. Future changes to the procedure will require a review of the NER item and a statement being signed stating that no changes have been made to the procedure that invalidate the compliance with the NER item. OE 3491 I DAVIE (LP&L) 08-AUG-89 10:48 EST Subject: Faulty Chemistry Sampling Technique

Unit: Docket No: NSSS/AE: Rating: Date Of Event: Date Of Commercial Operation:

Waterford 3 SES 50-382 CE/EBASCO 1104 NMWe 26 April 1989 September 1985

EVENT DESCRIPTION

A recent assessment of chemistry activities identified a discrepancy between the method used to sample oil stored in the Emergency Diesel Generator Fuel Oil Storage Tanks (FOST) and the methods specified by Technical Specifications.

EVENT ANALYSIS

Technical Specifications require utilization of methods specified in ASTM-D270-1975. The ASTM specifies using the bottle sampling or tap sampling methods when sampling the subject tanks. Bottle sampling requires taking multiple samples from different levels within the tank. Tap sampling requires drawing samples from taps located in at least 3 different levels on the tank.

Sampling of the FOST has been accomplished in the past by drawing a sample from the recirc header of the fuel oil transfer pump.

The bottle sampling method is not preferred due to the potential for introducing foreign material into the tank interior. The tap method is not possible because no taps are installed on the tanks. Additionally, chemistry supervision feels that the ASTM is written for use by a buyer/seller to standardize the process of sampling and to provide a means of ensuring a representative product during transport and receipt. The Tech Spec is written for use by a storer/user to confirm oil quality has not deteriorated during storage to the point where the generator will not operate.

Although sampling from the recirc line is more likely to detect the presence of water, biological growth and sedimentation, the intent of the ASTM method is to obtain a representative sample. Sampling from the bottom does not meet this intent.

CORRECTIVE ACTION

An engineering evaluation was performed where a sample drawn from the recirc line was compared with a sample drawn by the bottle method collected using a sampling thief analyzed by an independent laboratory. Results of this comparison indicated that the samples were similar.

A tech spec change is being sought to allow samples to be collected from the recirc line of the fuel oil transfer pump.

XECILLXX AUG 10 1089 Advance Copy firet G.L. Fiser / D. Advas for evaluation. Pelerens I-116 (recire 2 volumes) (recire 387) (CM -5387)

Until a determination is made on the tech spec change, samples of the FOST are collected in accordance with the ASTM bottle method using a sampling thief.

INFORMATION CONTACT: Jerrol Taylor, 504-739-6280

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