Designation: D 5000 – 89 (Reapproved 2000)

Standard Practice for Evaluating Activity of Clay Elements Using a Side-Stream Sensor¹

This standard is issued under the fixed designation D 5000; 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. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Calcined clay is a widely used adsorptive medium for removing polar contaminants and additives from petroleum fluids. In refineries, clay is installed in large bed-type vessels to refine products such as lubricants or aviation turbine fuel. In the field, clay is usually used in canister or bag type elements installed as a bank of elements in a vessel to remove traces of contaminants, particularly from non-additive jet fuel immediately before delivery to an airport.

1. Scope

- 1.1 This practice describes a field procedure to determine whether the useful life of the clay has been exceeded in canister or bag-type clay elements that are installed in ground filtration units of aviation fuel handling systems.
- 1.2 The field procedure utilizes the apparatus of Test Methods D 3948 to periodically test a small clay capsule installed in a sidestream around a clay treatment vessel that receives a fixed ratio of the same fuel that flows through the clay elements in the vessel.
- 1.3 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses are for information only.
- 1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 2550 Test Method for Water Separation Characteristics of Aviation Turbine Fuels²
- D 3602 Test Method for Water-Separation Characteristics of Aviation Turbine Fuels (Field Test)³
- D 3948 Test Methods for Determining Water Separation

Characteristics of Aviation Turbine Fuels by Portable Separometer⁴

3. Terminology

- 3.1 Definitions:
- 3.1.1 *surfactants*—surface active molecular species that exhibit both water soluble and oil soluble properties, and affect the physical behavior at the interface between water and oil phases by forming emulsions or changing the wetting characteristics of solid surfaces exposed to water and oil.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *active limit*—the Micro-Separometer (MSEP) rating by Test Methods D 3948 of the effluent from a clay monitor that represents low surfactant content and therefore continued activity of the clay for absorption.
- 3.2.2 *clay*—a naturally occurring mineral, largely hydrous aluminum silicate, calcined at high temperature to remove water and volatile matter, used in granular form as an adsorptive media for removing polar compounds that are present in many hydrocarbon fluids.
- 3.2.3 *clay treatment*—a process for exposing fuels and blending components at ambient temperatures to granulated calcined clay in order to remove polar impurities such as surfactants.
- 3.2.4 deactivation of clay media—results when adsorptive surfaces are no longer capable of adsorbing polar species and improving the quality of the feed stream.
- 3.2.5 *deactive limit*—the MSEP rating by Test Methods D 3948 of the effluent from a clay monitor that represents high surfactant content and therefore the deactivation of the clay for adsorption.

¹ This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.J0 on Aviation Fuels.

Current edition approved Oct. 27, 1989. Published December 1989.

² Discontinued, See 1991 Annual Book of ASTM Standards, Vol 05.02.

³ Discontinued, See 1994 Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 05.02.

3.2.6 *sidestream*—flow system that parallels the main flow stream into and out of a unit, such as a vessel, holding filter elements but usually operating at a lower flow rate.

4. Summary of Practice

- 4.1 A sample of the clay from the element installed in a ground clay treatment vessel is placed into a clay holder that is then mounted in a sidestream cabinet that receives main fuel flow through an inlet probe at a rate proportional to main line flow. After preconditioning to ensure that flow is not bypassing the holder, it is placed on-stream to monitor the clay elements in the main vessel.
- 4.2 At intervals of two to eight weeks depending on surfactant levels experienced in the system, the clay holder is removed and tested twice in the Mark V deluxe or two-speed⁵ Micro-separometer with reference fuel containing a specified surfactant additive. The rating of the reference fuel from the clay holder by Test Methods D 3948 determines whether the clay is deactivated or still capable of adsorbing surfactants. In the latter case, the clay holder is returned to the sidestream cabinet and monitoring continues.
- 4.3 If the rating suggests that clay is spent, the elements are changed and a fresh clay holder is installed. A plot or record of successive periodic tests is desirable to anticipate changes of clay elements or to increase testing frequency. (Fig. 1 is a schematic of a sidestream sensor installation around a clay treating vessel).

5. Significance and Use

- 5.1 Clay elements are widely used in aviation fuel handling systems to adsorb polar contaminants that are picked up in shipments by tanker, barge, or pipeline from refineries to terminals, airports, or both. Some of these contaminants such as surfactants interfere with efficient operation of filter-separator units that remove water from fuel.
- 5.2 In order to determine whether the clay elements are spent, it is necessary to test fuel both into and out of clay treatment vessels frequently. Clay elements must be changed

⁵ The Mark V delux or two-speed Micro-Separometer is available from EMCEE

Electronics, Inc., 520 Cypress Ave., Venice, FL 34292.

when no improvement in quality is noted. Unless carried out frequently, such testing may not disclose a deactivated clay treatment vessel in time to prevent failure of downstream filter/separators.

5.3 To avoid such failures, a small sample of clay in a clay holder contained in a sidestream installation that receives flow proportional to the main stream flow is evaluated periodically using a reference fuel containing a known surfactant. When the rating of the reference fuel by Test Methods D 3948 indicates that the capsule clay is becoming spent, the elements in the main filter vessel are ready for change.

6. Apparatus

- 6.1 *Sidestream Sensor*, consisting of the following components as illustrated in Fig. 2:
 - 6.1.1 *Inlet Probe*, metal line and valve to the cabinet,⁶
 - 6.1.2 Metal Line and Valve, from the cabinet,⁶
 - 6.1.3 Cabinet, Flow Meter, accurate to \pm 5 %, ⁶
- 6.1.4 Clay Holder and Air Bleed, contained in the sensor cabinet, 6 and
 - 6.1.5 Clay Cone Insert, (test capsule).6
 - 6.2 Micro-Separometer, Mark V⁵ deluxe or two-speed.
 - 6.3 Clay Holder Bracket, and tubing assembly.

Note 1—The sensor cabinet and probes are installed across the inlet and effluent of the clay treating vessel being monitored. The inlet probe may consist of a short 6.4 mm ($\frac{1}{4} \text{ in.}$) diameter tube that protrudes into the moving stream of fluid.⁶

7. Reagents and Materials

7.1 Reference Fluid Base, is fuel from the fuel supply system under evaluation. In the event the fuel contains additives or has a WSIM less than 96 the fuel should be clay treated as described in Test Methods D 3948.

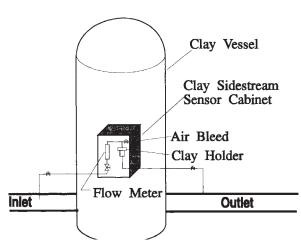


FIG. 1 Schematic of Clay Sidestream Sensor Installation

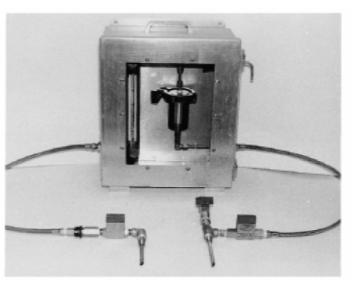


FIG. 2 Clay Side-Stream Sensor

⁶ Available from Gammon Technical Products, 235 Parker Avenue, Manasquan, NJ 08736.

- 7.2 Dispersing Agent, is a toluene solution containing 1 mg of solid (100 % dry) bis 2 ethyl hexyl sodium sulfosuccinate per mL of toluene.
- 7.3 Reference Fuel—consists of dispersing agent in reference fluid base. To produce a MSEP rating by Test Methods D 3948 of 40 to 60 about 1 mL/L of dispersing agent is required in the base.

8. Preparation of Clay Holder

- 8.1 Clay to fill the clay holder is obtained from a new clay element (canister or bag type) at the time all elements in a clay treatment vessel are changed. About 100 mL of clay should be placed in a clean jar. The cover of the jar should be screwed on tightly. A portion of resin treated filter paper from the center tube of the element or media migration barrier of the bag type element should also be removed and saved.
- 8.2 Insert components in the clay holder in the following order as illustrated in Fig. 3:
- 8.2.1 *Gasket*—(An additional gasket, as shown, may be added to ensure a leak-tight fit. The gasket hole should be of approximately the same size as the bottom of the cone.)
- 8.2.2 Filter paper, or media migration barrier from the clay element.
- 8.2.3 *Cone Spacer*—Insert cone spacer into holder (large end on top) and pour clay into cavity, (See Fig. 4). Tap holder to settle clay. Alternately add make-up clay and tap until clay does not settle below the top of cone section. Place section of filter paper (media migration barrier) on top of clay in cone. Insert gasket(s) to give firm compression of all components when top is clamped in place. Install holder lid, o-ring seal, lid, and clamp. Tighten clamp firmly.

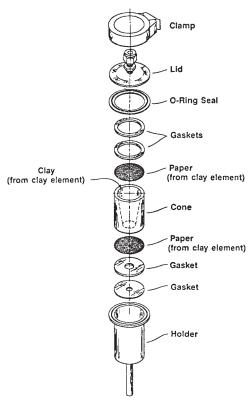


FIG. 3 Clay Holder (Exploded View)



 ${
m Note}\ 1$ —Insert cone spacer, (large end of cone on top). Pour clay into cavity. Repeatedly, tap holder and add clay until level with top of cone.

FIG. 4 Clay Cone Spacer

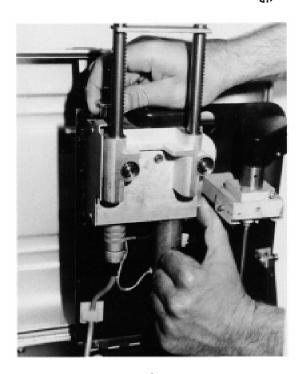
Note 2—Use of a bench vise will aid in compressing gaskets, positioning the lid to holder body and installing the lid clamp.

- 8.2.4 Filter Paper or Media Migration Barrier from Clay Element.
- 8.2.5 *Gasket*—(An additional gasket, as shown, may be added to assure a leak tight fit. The gasket hole should be of approximately the same size as the top of the cone insert.)

8.2.6 *O-Ring Seal*.

9. Pre-Conditioning the Clay Holder

- 9.1 The clay holder is tested to ensure that flow will not by-pass the clay or components. The Test Methods D 3948 Mark V^5 Micro-Separometer is used to perform this test as follows:
 - 9.1.1 Using the two-speed Mark V Micro-Separometer:
- 9.1.1.1 In order to operate at the 15 s drive-speed required to test the clay holder, it is necessary to change the syringe drive gear selector to HIGH by lifting the release knob on the top left of the syringe drive casting. The moveable gear carriage located on the lower right of the housing can then be pushed to the left as illustrated in Fig. 5. While holding the gear carriage in, lower the release knob to lock it in place.
- 9.1.1.2 Turn power switch to ON and preset UP/AUTO/DOWN switch to the UP position. The syringe drive will rise to the top limit.
- 9.1.1.3 Remove plunger from the plastic syringe and pour 50 mL of reference fuel into the syringe barrel. Replace plunger using the wire aid.
- 9.1.1.4 Insert the holder bracket on the microseparometer in the slot above the stirrer. See Fig. 6.
- 9.1.1.5 Place the clay holder in the bracket and connect the tubing from the holder top to the syringe.
- 9.1.1.6 Move the UP/AUTO/DOWN switch to the DOWN position. The syringe will immediately start down.
 - 9.1.1.7 Collect the effluent from the clay holder and discard.
- 9.1.1.8 Immediately repeat 9.1.1.3-9.1.1.6 through passing two additional 50 mL samples of reference fuel through the holder in succession.



Note 1—When using MK V^A deluxe version of the Micro-Separometer, there is no need to make gear changes.

Note 2—Gear carriage is at lower right under thumb. Release knob is at upper left.

FIG. 5 Syringe Drive Gear Selector



Note 1—Bracket is held in slot above stirrer. The bracket fits in the same way for all versions of the Micro-Separometer, but there is no gear change mechanism in Mark V^A Deluxe because the drive is preprogrammed.

FIG. 6 Holder Bracket

9.1.1.9 Collect the entire effluent from each pass separately into marked 50 mL flasks.

Note 3—Because of minor hold-up of fuel, less than 50 mL of effluent

may be collected. Do not add reference fuel to effluents.

- 9.1.1.10 Reset the syringe drive to the normal gear ratio and reset the UP/AUTO/DOWN switch to the AUTO position. The instrument is now ready for a normal Test Methods D 3948 test.
- 9.1.1.11 Run the standard Test Methods D 3948 test on each of the two test method effluent samples collected.
- 9.1.1.12 Record results on the data sheet described in Table 1.
 - 9.1.2 Using the Deluxe Mark V Micro-Separometer:
- 9.1.2.1 It is not necessary to change the syringe drive speed. After turning the apparatus ON, the switch marked "F" initiates the automatic program for clay holder testing. The annunciator lamps in the *SYRINGE* section will indicate that manual control of the drive mechanism can be initiated.
- 9.1.2.2 Depress the UP switch and the drive mechanism will move to the upper limit. The annunciator lamp in the PRO-GRAM section will indicate that the turbidimeter can be manually activated.
- 9.1.2.3 The steps described in 9.1.1.3-9.1.1.12 should be followed.
- 9.1.3 If either of the two MSEP results in less than 96, flow through the clay holder may be by-passing the clay. The holder should be dismantled, additional clay or gaskets, or both, installed, the holder reassembled and retested. A MSEP reading of 97 or better must be obtained before the clay holder can be installed in the sidestream cabinet for field operation.

10. Field Installation of Clay Holder

- 10.1 After preconditioning and testing the clay holder is installed in the cabinet and air from the tubing is bled through the vent valve at the top of the cabinet.
- 10.2 The flow rate through the clay holder in mL/minute is established by reference to Fig. 7 in order to adjust the flow meter. The clay filter vessel must be operated at its normal flow rate when the sidestream sensor flow meter is adjusted.

Note 4—Normal flow rate is the maximum rate that will occur in the system. It may not be the rated flow of the vessel itself. Both the sensor and main flow rates should be checked and recorded weekly, readjusting if necessary.

11. Periodic Clay Holder Testing

11.1 At periodic intervals the clay holder is removed from the sidestream cabinet and evaluated in the assembled condition with the Mark V micro-separometer. Testing intervals are a function of estimated clay life. If clay life is less than 6 months, two week testing intervals may be appropriate. For longer clay lives, monthly testing may be sufficient.

Note 5—When testing of fuel for surfactants by the Test Methods D 2550, D 3602, or D 3948 test method results indicate that higher than normal levels of surfactants are present, the evaluation period of the clay holder should be twice as frequent as suggested in 11.1.

11.2 To remove the clay holder for evaluation, close valves in the inlet and outlet sampling probe lines. Do not touch the valve that controls the flow meter.

TABLE 1 Clay Sidestream Sensor Data Log

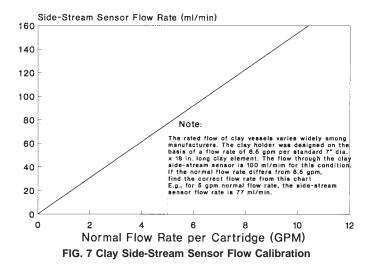
E 1 Clay Sidestream Sensor Data Log

Normal Vessel Flow:	USGPM	
Clay Holder (CSS) Flow Rate:		MI/min ^A
Start Date:		

Location:	
Vessel Model:	
Element Model:	_
Quantity Elements:	
Reference Fuel MSEP:	

INFORMATION

	Sensor Information				Supplementary System Date, (if available)					
Date	Cumulative Throughput USGPM	Sensor MSEP Ratings (Refer 9.1)	Remarks	MSEP		Aqua-Glo	Pressure Drop		Corrected Pressure	
		Sample #1	Sample #2		in	out	(ppm) —	psi	gpm	Drop for Normal Flow Rate



- 11.3 Remove the clay holder from the cabinet by loosening the tubing fittings on top and bottom.
- 11.4 Evaluate the clay holder by following the procedure of 9.1.1 or 9.1.2. Two separate samples of reference fuel should be used for the two evaluations.
- 11.5 Clay holder evaluation results and clay vessel cumulative through-put data should be noted in Table 1.

12. Evaluation of Results

12.1 If MSEP readings from both evaluations are greater than the active limit the clay in the vessel is considered still active and the clay holder should be reinstalled.

12.2 If either MSEP reading is below the deactive limit the clay in the vessel has become deactivated. Elements should be changed immediately and a fresh clay holder prepared and installed.

Note 6—Field evaluation of clay systems cited in the CRC Report No. 552^7 suggests that the active limit is 93 MSEP or higher while the deactive limit is 90 MSEP or lower. If one of the two evaluations is between 90 and 93 MSEP deactivation is considered imminent and evaluation frequency should be increased three-fold. Users of this practice may select different MSEP criteria for clay element change-out depending on experience with the particular system being monitored.

13. Reinstallation of the Clay Holder

- 13.1 The syringe adapter should be removed from the clay holder that should be returned to the cabinet. Tube fittings should be tightened and valves opened to the sampling probes.
- 13.2 Air should be bled from the tubing using the vent valve and the flow setting of the flow meter should be checked.

14. Report

14.1 A data log, such as Table 1, is the best method for reporting both clay holder evaluations and other significant data. Tests of the fuel itself as well as system data on flow rate and pressure drop should also be recorded.

15. Keywords

15.1 aviation turbine fuel; filtration; jet fuel; side-stream sensor; surfactants; water separation

 $^{^7\,\}mathrm{CRC}$ Report No. 552 "Field Evaluation of the Sidestream Sensors for Filter/Separators and Clay Filters," (February 1987).

D 5000 – 89 (2000)

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).