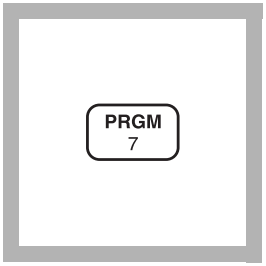
VOLATILE ACIDS (0 to 2800 as mg/L HOAc)

Esterification Method*



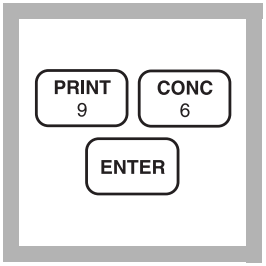
1. Enter the stored program number for Volatile Acids as acetic acid (HOAc).

Press: **PRGM**

The display will show:

PRGM ?

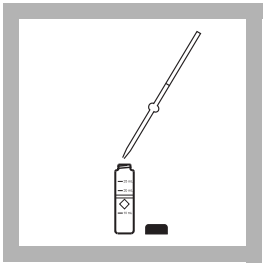
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **96 ENTER**

The display will show **mg/L, HOAc** and the **ZERO** icon**.**

Note: *If high levels of dissolved solids or mineral acids are present, distill as described in the Hach Distillation Apparatus manual.*



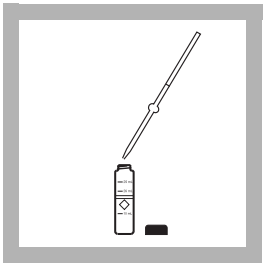
3. Pipet 0.5 mL of deionized water into a dry 25-mL sample cell (the blank).

Note: *Use a Class A or TenSette Pipet.*

Note: *Adjust the pH of stored samples before analysis.*

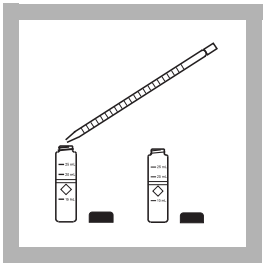
4. Filter or centrifuge 25 mL of the sample.

Note: *Centrifugation is faster than filtration.*

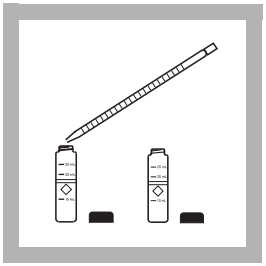


5. Pipet 0.5 mL of the filtrate or supernatant into another dry 25-mL sample cell (the prepared sample).

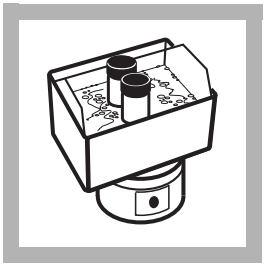
Note: *Use a Class A or TenSette Pipet.*



6. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



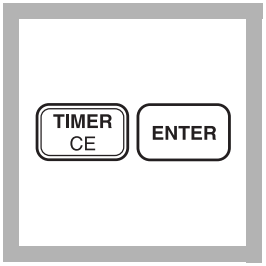
7. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.



8. Place both cells into a boiling water bath.

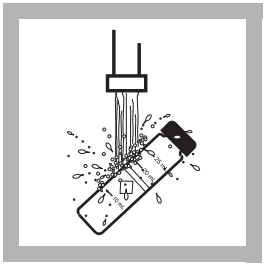
Note: *Samples may be boiled in a 600-mL beaker.*

* Adapted from *The Analyst*, 87, 949 (1962)

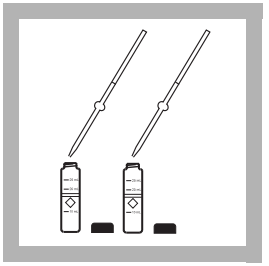


9. Press: **TIMER ENTER**

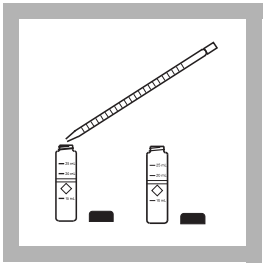
A 3-minute reaction period will begin.



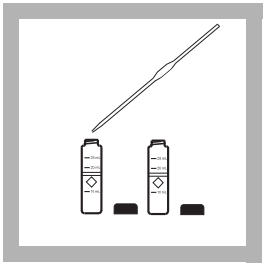
10. When the timer beeps, cool solutions to 25 °C (until cells feel cool) with running tap water. Then dry the cells with a soft cloth.



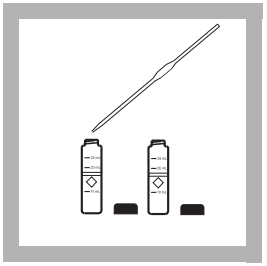
11. Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



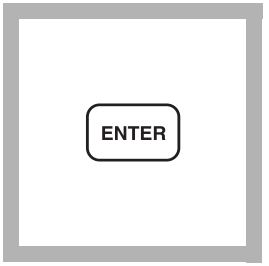
12. Pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Cap and invert to mix.



13. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Cap and invert to mix.



14. Add 10 mL of deionized water to each cell. Cap and invert to mix.

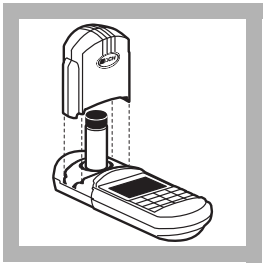


15. The display will show: **3:00 TIMER 2**

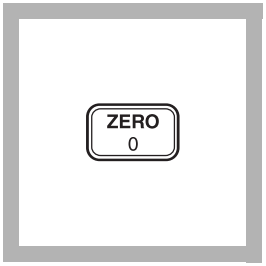
Press: **ENTER**

A 3-minute reaction period will begin.

Note: *After this three-minute reaction period, proceed immediately through steps 16-19.*



16. When the timer beeps, immediately place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

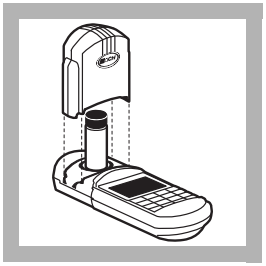


17. Press: **ZERO**

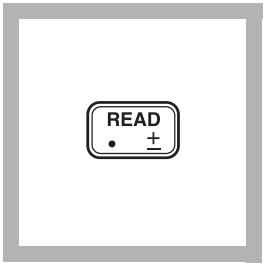
The cursor will move to the right, then the display will show:

0 mg/L HOAc

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



18. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L Volatile Acids as acetic acid will be displayed.

Sampling and Storage

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Volatile Acids PourRite Ampule Standard Solution, 62,500 mg/L as acetic acid.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL graduated mixing cylinders, each containing 25 mL of filtered sample. Stopper. Shake well to mix.

c) Remove a 0.5 mL aliquot of sample from each cylinder; add to three dry sample cells. Analyze all three samples along with the original test sample beginning with Step 5 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1.*

Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids PourRite Ampule Standard Solution (62,500 mg/L as acetic acid) to a 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix.

Method Performance

Precision

In a single laboratory, using a standard solution of 500 mg/L volatile acids as acetic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±8 mg/L.

Estimated Detection Limit

The estimated detection limit for program 96 is 17 mg/L HOAc. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The volatile acids test is designed specifically for the determination of volatile acids in digestor sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

REQUIRED REAGENTS

	Cat. No.
Volatile Acids Reagent Set (90 tests)	22447-00
Includes: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32	

Description	Quantity Required Per Test	Units	Cat. No.
Ethylene Glycol	3 mL	1000 mL	2039-53
Ferric Chloride-Sulfuric Acid Solution	20 mL	1000 mL	2042-53
Hydroxylamine Hydrochloride Solution, 100 g/L	1 mL	100 mL	818-42
Sodium Hydroxide Standard Solution, 4.5 N	4 mL	1000 mL	2040-53
Sulfuric Acid Standard Solution, 19.2 N	0.4 mL	100 mL	2038-32
Water, deionized	20.5 mL	4 L	272-56

REQUIRED APPARATUS

Description	Quantity Required Per Test	Units	Cat. No.
Cots, finger	2	2/pkg	14647-02
Cylinder, graduated, 10 mL	1	each	508-38
Filter Paper, folded, 12.5 cm	1	100/pkg	1894-57
Flask, erlenmeyer, 50 mL	1	each	505-41
Funnel, poly, 65 mm	1	each	1083-67
Hot Plate, circular, 3.5-inch diameter	1	each	12067-01
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 2 mL	2	each	532-36
Pipet, volumetric, Class A, 0.5 mL	3	each	14515-34
Pipet, volumetric, Class A, 10.00 mL	3	each	14515-38
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Water Bath and Rack	1	each	1955-55

OPTIONAL REAGENTS

Volatile Acids Standard Solution, PourRite ampule, 62,500 mg/L as acetic acid, 10 mL	16/pkg	14270-10

OPTIONAL APPARATUS

Ampule Breaker, PourRite	each	24846-00
Beaker, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Centrifuge, laboratory, 115 Vac	each	26765-00
Centrifuge, laboratory, 230 Vac	each	26765-02
Centrifuge Tubes, 15 mL	10/pkg	22787-39
Centrifuge Tube Caps	20/pkg	25852-20
Cylinder, graduated, mixing, 25 mL	each	1896-40
Cylinder, graduated, plastic, 250 mL	each	1081-46
Distillation Apparatus	each	22653-00
Distillation Heater and Support Apparatus	each	22744-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-10	50/pkg	21997-96

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.