

# PHPA Polymer Concentration Test Kit Instruction Manual



Manual No. 209057, Revision D

Instrument No. 208769

**fann**<sup>®</sup>

Fann Instrument Company

## PHPA Polymer Concentration Test Kit Instruction Manual

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### Contact FANN



#### Phone

TELEPHONE: 281-871-4482  
TOLL FREE: 800-347-0450  
FAX: 281-871-4358



#### Mail

Fann Instrument Company  
P.O. Box 4350  
Houston, Texas, 77210 USA



#### Location

Fann Instrument Company  
15112 Morales Rd Gate 7  
Houston, Texas, 77032, USA



#### Online

[www.fann.com](http://www.fann.com)  
[fannmail@fann.com](mailto:fannmail@fann.com)

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## 1 Introduction

The PHPA Polymer Concentration Test Kit is a simple analytical field test that selectively measures the concentration of partially hydrolyzed polyacrylamide (PHPA). Polyacrylamide polymer is commonly used in drilling muds to prevent dispersion of shale cuttings, and it is also used as either a primary or secondary drilling fluid viscosifier. Although PHPA, as a viscosifier, helps the drilling fluid's carrying capacity to remove cuttings from the wellbore, it is not a very good or efficient suspension agent.

The PHPA Polymer Concentration Test Kit can measure PHPA in whole mud, supernatant solutions, and filtrates.

### 1.1 Modified Kjeldahl

The PHPA test is a modified Kjeldahl method to measure partially hydrolyzed polyacrylamide (PHPA) concentration in mud systems.

In this test, the sample is reacted with sodium hydroxide (caustic digestion) at 200 - 250°C (392 - 482°F). Ammonia is liberated as hydrolysis of the polymer occurs. The ammonia is distilled into a boric acid solution. After cooling, the mixture is titrated with sulfuric acid solution to determine the polyacrylamide content

### 1.2 Document Conventions

The following icons are used in this manual to distinguish elements of text.



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Notes emphasize additional information that may be useful to the reader.

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Caution messages give directions that, if not observed, could result in loss of data or in damage to equipment.

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**Describes an unsafe condition or practice that if not corrected, will result in personal injury, possibly death.**

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## 2 Safety

Chemicals used in this test may cause hazards to the user's health by direct contact, inhalation, ingestion, explosion or fire. Read all warnings, precautions and hazard classifications - fire, health and reactivity, on the container label.

For in depth information on handling, reactivity with other substances, storage and other safety information, refer to the Material Safety Data Sheet (MSDS) for each chemical.

If personal contact or an environmental accident occurs, use the corrective measures outlined on the label and on the MSDS.

For safe operation of the hot plate, make sure the electrical cord is in good condition and equipped with a grounding plug.

Read and follow the operating instructions for the hot plate. Do not leave it unattended while it is heating. Use caution when handling hot flasks and other laboratory containers.

### 3 Features and Specifications

The PHPA Polymer Concentration Test Kit consists of a hot plate with a flask bracket, a magnetic stirrer, and other laboratory equipment and reagents, all contained in a rugged, lightweight plastic carrying case.

Four Erlenmeyer flasks are included for containing the sample (reaction flask) and collecting the ammonia in the boric acid solution (collection flask).

The test kit arrangement is shown in Figure 3-1.

The chemicals and reagents along with their uses are as follows:

- Sulfuric Acid, 0.02N – to titrate the ammonia in receiving solution
- Sodium Hydroxide , 8N – to raise pH in sample (pH>11)
- Defoamer – to minimize or eliminate foaming
- Boric Acid, 2 volume% - to capture the ammonia gas and form an ammonium-borate complex
- Bromocresol Green-Methyl Red Indicator – for titration color change
- EZ-MUD<sup>®</sup>DP, a synthetic polymer containing PHPA – to create a calibration curve

**Table 3-1 PHPA Polymer Concentration Test Kit Specifications**

Category	Specification
Heat Source –Hot Plate	115VAC, 371°C (700°F) maximum
Carrying Case	26 x 21 x 10 inches
Dimensions (Width x Depth x Height)	66.04 x 53.3 x 25.4 centimeters
Weight	38 lb (17.2 kg)



**Figure 3-1 PHPA Polymer Concentration Test Kit**

## 4 Equipment Set-up

See Figure 3-1.

Designate one flask as the reaction flask and another flask as the collection flask.

The reaction flask will hold the sample. It will need a rubber stopper that has a glass tube inserted into it. This glass tube is also called the generator tube.

The collection flask will contain boric acid and will receive the distilled ammonia. It will also require a rubber stopper that has a glass tube inserted into it. This tube, also called the collection tube, will be a longer tube that will sit below the surface of the boric acid.

Use the Tygon<sup>®</sup> tubing to connect the reaction flask and collection flask. Place one end of this flexible tubing onto the glass tubing and stopper that goes into the collection flask. Attach the other end of the flexible tubing onto the glass tubing/stopper that goes into reaction flask.



## 5 PHPA Test Procedure



### NOTE

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You will need to prepare a standard curve by plotting known concentrations (lb/bbl) of PHPA for four solutions and corresponding amounts (ml) of sulfuric acid required to titrate to endpoint. See Sections 5.1 and 5.2 which outline steps for EZ-MUD® or another mud, respectively.

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1. Set the hot plate to 200- 250°C (392-482°F) and preheat 10 minutes.
2. Add 50 ml of deionized water to the reaction flask.
3. Add 10 ml of test sample (whole mud, supernatant, or filtrate) into the same flask.
4. Add 2 ml of defoamer and 6 - 10 boiling stones.



### WARNING

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**Boric acid solution is a weak acid. Avoid skin contact.**

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5. To the collection flask, add 30 ml of 2% boric acid. Also, add 4 - 6 drops of blended indicator which is composed of 1 part bromocresol green indicator with 2 parts methyl red indicator. The color should be lavender red.



### WARNING

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**Sodium hydroxide (NaOH) is a strong alkaline chemical. Avoid skin contact.**

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### WARNING

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**Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) is a corrosive and toxic acid. Avoid skin contact.**

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6. To the reaction flask, add 5 ml of 5N sodium hydroxide (NaOH) using a 5 ml syringe, and immediately place the stopper with glass tubing into the flask. Connect the flexible tubing to the glass tubing,
7. Attach the other end of the flexible tubing to the glass tubing in the stopper that goes in the collection flask. Make sure that tip of the glass tubing is submerged in the boric acid solution.

**CAUTION**

Keep the boiling under control or a vacuum might form and cause the fluid to flow from the collection flask to the reaction flask. If this backflow occurs, the titration will not be accurate.

8. Place the reaction flask on the hot plate and heat the solution to a constant boil.
9. The boiling rate should be such that approximately 25 ml is distilled within 45 to 55 minutes.

**WARNING**

**If you smell ammonia, stop the test and move to fresh air. An ammonia odor indicates a leak from the reaction flask. The test results will not be accurate.**

**NOTE**

If there is a leak, the test results will not be accurate.

10. Turn off the hotplate.

**CAUTION**

The flask and glass tubing will be hot. Wear thermally protective gloves or use tongs to handle the glass tubing or flask.

11. Remove glass tubing from the collection flask; allow the flask to cool.
12. Titrate the solution in the collection flask with 0.02N sulfuric acid to the indicator end point. The color will change from blue- green to lavender-red.
13. Record the volume (ml) of sulfuric acid used.
14. Find the volume of sulfuric acid used and the corresponding lb/bbl of PHPA, using a standard graph.

### 5.1 EZ-MUD® Calibration Graph

If you are using EZ-MUD®, you can use the graph in Figure 5-1 or create a standard graph for a known mud matrix that has been treated with various amounts of EZ-MUD®.

Using the example graph shown, find the total volume (ml) of sulfuric acid titrated on the y-axis and read the corresponding concentration (lb/bbl) of EZ-MUD® DP (dry powder) on the x-axis based on the standard curve.

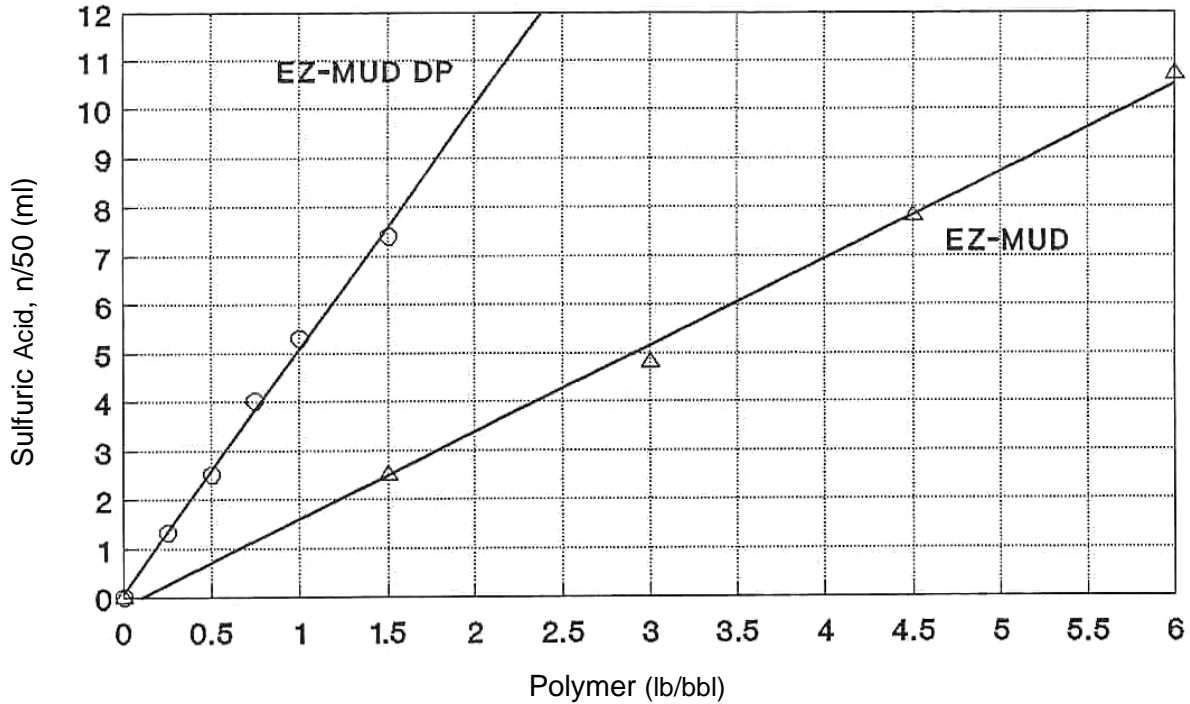


Figure 5-1 Example Calibration Curve: EZ-MUD® DP and EZ-MUD® in Field Muds

To create a standard curve for a given mud matrix, follow this procedure:

1. Add 0.5, 1.0, 1.5 and 2.0 grams of EZ-MUD® DP to 350 ml mud samples (i.e. 0.5, 1.0, 1.5, 2.0 lb/bbl). Stir vigorously with each addition.
2. Repeat steps in Section 5 using four reaction flasks and four collector flasks.
3. Heat all four flasks on hot plate into all four receiver flasks to create the curve with a minimum of heat ups.
4. Plot the graph and use it to determine the concentration of polymer (lb/bbl) in your mud sample.

## **5.2 Calibration Graph for Other Additives**

If you are using another polyacrylamide-based additive, you will need to create a standard curve as follows:

1. Add 0.5, 1.0, 1.5 and 2.0 grams of your product to 350 ml mud samples (i.e. 0.5, 1.0, 1.5, 2.0 lbs/bbl). Stir vigorously with each addition.
2. Repeat steps in Section 5 using four reaction flasks and four collector flasks.
3. Heat all four flasks on hot plate into all four receiver flasks, minimizing heat usage.
4. Plot the graph and use it to determine the concentration of polymer (lb/bbl) in your mud sample.

## 6 Parts List

**Table 6-1 PHPA Polymer Concentration Test Kit**

Item No.	Part No.	Quantity	Description
0001	206555	4	ERLENMEYER FLASK, GLASS, 125ml
0002	206666	1	SODIUM HYDROXIDE, 8N, 4 oz
0003	207560	1	SAMPLE CUP f/MODEL 35 VISCOMETER, 350ml
0004			
0005	209043	1	FLASK BRACKET
0006	209044	5	GENERATOR TUBE
0007	209045	5	COLLECTION TUBE
0008	209057	1	INSTRUCTION MANUAL
0009	209835	1	DEFOAMER, 8oz
0010	209863	1	SULFURIC ACID, N/50, 8oz
0011	209878	1	BORIC ACID, 2% BY VOLUME, 16oz
0012	209891	1	BROMCRESOL GREEN METHYL RED INDICATOR, 4oz
0014	209945	2	DISTILLED WATER, 16oz
0015	203624	1	BLANK CASE, PLASTIC
0016	203631	1	FOAM INSERT
0017	204541	1	PHPA LABEL
0018	205235	6	DISPOSABLE SYRINGE, 5 ml, w/o NEEDLE
0019	205245	1	BOILING STONES, (PLAIN) 250G
0020	205247	1	HOT PLATE, 115V AC, MAX TEMP 371°C
0021	205248	5	No. 5 RUBBER STOPPER w/2 HOLES
0022	205249	5	No. 5 RUBBER STOPPER w/1-5 mm HOLE
0023	205623	2	TYGON TUBING, 1/4 in. ID x 1/16 in.
0024	205868	1	GRADUATED GLASS CYLINDER, 25ml TC
0025	205898	2	PLASTIC SYRINGE, 10 ml
0026	205997	1	PORTABLE MAGNETIC STIRRER, BATTERY OPERATED
0027	206000	2	MAGNETIC STIRRING BAR, 3/8in. x 1in.
0028	206028	1	PIPETTE, 5 ml, SEROLOGICAL
0029	206029	1	PIPETTE, 10ml, SEROLOGICAL

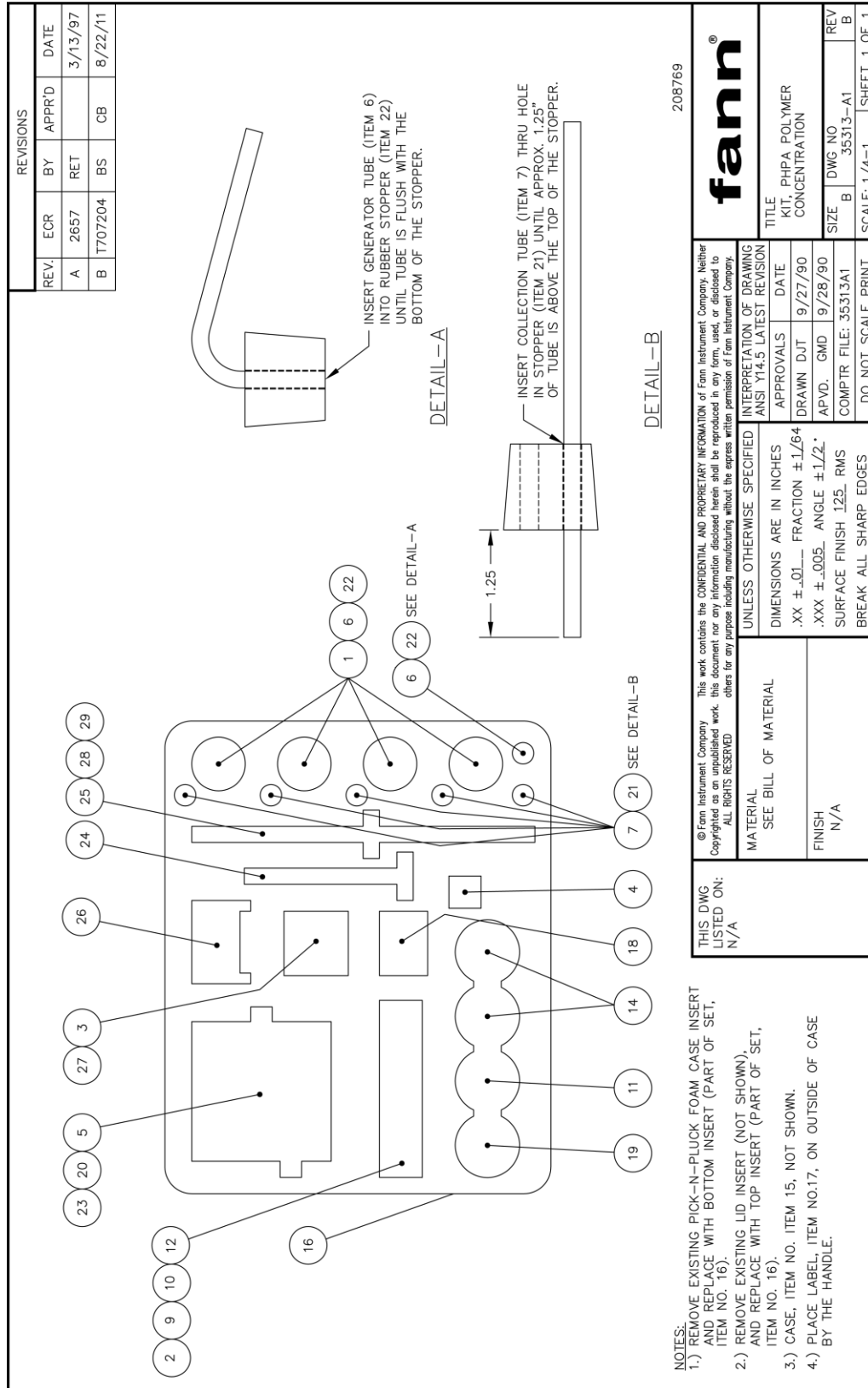


Figure 6-1 PHPA Polymer Concentration Test Kit

## **7 Warranty and Returns**

### **7.1 Warranty**

Fann Instrument Company warrants its products to be free from defects in material and workmanship for a period of 12 months from the time of shipment. If repair or adjustment is necessary, and has not been the result of abuse or misuse within the twelve-month period, please return, freight prepaid, and correction of the defect will be made without charge.

Out of warranty products will be repaired for a nominal charge.

Please refer to the accompanying warranty statement enclosed with the product.

### **7.2 Returns**

For your protection, items being returned must be carefully packed to prevent damage in shipment and insured against possible damage or loss. Fann will not be responsible for damage resulting from careless or insufficient packing.

Before returning items for any reason, authorization must be obtained from Fann Instrument Company. When applying for authorization, please include information regarding the reason the items are to be returned.

Our correspondence address is:

**Fann Instrument Company**

P.O. Box 4350

Houston, Texas USA 77210

Telephone: 281-871-4482

Toll Free: 800-347-0450

FAX: 281-871-4446

Email [fannmail@fann.com](mailto:fannmail@fann.com)

Our shipping address is:

**Fann Instrument Company**

15112 Morales Road

Gate 7, Houston, Texas USA 77032