

®

**Calcimeter and Recording
Calcimeter Kit
Instruction Manual**

Part No. E10110001EA

Rev. D

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SECTION 1 DESCRIPTION

The Fann Calcimeter and the Fann Recording Calcimeter are used to determine the amount of Calcium Carbonate and Magnesium Carbonate (Dolomite) in a sample of alkaline earth carbonates such as oil well cores or drilled cuttings. Calcite build up in drilling fluids and in water treatment processes causes scaling problems. Data from the Fann Calcimeter can help determine the proper chemical treatment.

These instruments comply with the ASTM D 4373 - 84 (Reapproved 1990) **STANDARD TEST METHOD FOR CALCIUM CARBONATE CONTENT IN SOILS.** This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D - 18.13 on Marine Geotechnics, published July 1984. The ASTM procedure is described in Section 4.

In the Fann Calcimeter the calcium carbonate and magnesium carbonate are reacted with 10 percent Hydrochloric Acid to form CO₂. This is done in a sealed reaction cell and the pressure build up due to the CO₂ is measured using either a pressure gauge or a pressure recorder. The use of a Calibration Curve, Fig. 4, determined through the use of pure Calcium Carbonate reagent allows the pressure developed to be related to the weight of calcium carbonate in the calibration sample. Several weights of sample are suggested to assure an accurate curve. These tests can be conducted using either the pressure gauge or recorder with the reaction cell. The sample can be weighed on a portable balance (10 mg Precision or better). Refer to Section 5 for calibration procedure.

The calcium carbonate content of soil [ASTM Procedure D 4373] is determined by treating a 1 gm dried soil specimen with hydrochloric acid (HCl) in an enclosed reactor vessel. Carbon dioxide gas is evolved during the reaction between the acid and carbonate fraction of the specimen. The resulting pressure generated in the closed reactor is directly proportional to the carbonate content of the specimen. This pressure is measured with a bourdon tube pressure gauge that can be pre-calibrated with reagent grade calcium carbonate. Refer to Section 5 for calibration procedure.

SECTION 2

SAFETY CONSIDERATIONS

The Calcimeter Test depends on reacting CaCO_3 with HCl. Hydrochloric Acid may be Corrosive and may cause Chemical Burns. Use care in handling the Hydrochloric Acid so that no acid is spilled on either skin or clothing or splashed into eyes. If acid contacts skin or eyes, immediately flush with large quantities of water for at least 15 minutes. Do not inhale vapors. Process Hydrochloric Acid beneath a laboratory hood or in a well ventilated area to reduce the inhalation of fumes. Wear appropriate safety equipment.

Do not take internally. Get medical attention immediately if accidentally contacted by the acid.

Always open the pressure bleed valve of the reaction cell following each test. Do not attempt to open the cell until all pressure has been dissipated.

The Fann Recording Calcimeter uses an electrically powered transducer and recorder to record the pressure in the Reaction Cell. The following Safety Considerations should be used with this Recorder as with any electrical appliance:

1. Make sure the Power switch is OFF before connecting power cable to electrical outlet.
2. Verify the power cable and the outlet receptacle are three wire grounding type plug and receptacle.
3. Always unplug the power cable before opening the Recorder for renewing the chart paper or other maintenance or repair work.

SECTION 3 FANN TEST PROCEDURE

Before starting the Test Procedure, make sure the equipment is clean and in good operating condition. Verify that a calibration curve is available for the particular equipment to be used. Should a calibration curve not be available, follow the procedures outlined in Section 5 to construct one. For an illustration of this curve Refer to Fig. 4.

- A. Obtain a sample of Core, Drilled Cuttings, or other solids that are to be analyzed. The sample should be dry and free of contaminants. Grind sample to 100 mesh or finer, using a mortar and pestle and a 100 mesh sieve. If it is unknown whether the sample has been dried, it is recommended to heat the sample in an oven at 220 °F (105 °C) for 12 to 24 hours.
- B. Weigh Sample for test, paragraph 1 or 2 depending on pressure capacity of equipment.
 - 1. Weigh approximately (.5 to .7 gms) of the ground sample and record the weight to nearest .001 gm (10 mg) if a calcimeter with a 15 psi gauge or 15 psi recorder and a small (10 ml) capacity acid cup is being used.
 - 2. Weigh approximately (1.0 to 1.4 gms) of the ground sample and record the weight to nearest .001 gm (10 mg) if a calcimeter with a 30 psi gauge or 30 psi recorder and a large (20 ml) capacity acid cup is being used.

CAUTION

Do not attempt to run larger than
0.7 gm sample with the 10 ml acid cup.

- C. Load the Test Sample in the CO₂ reaction cell.
 - 1. Unscrew and remove the top with pressure gauge or pressure transducer from the Reaction Cell then remove the acid cup.
 - 2. Inspect to be sure CO₂ reaction cell and top are clean and dry.
 - 3. Be sure reaction cell "O" Ring seal on the top, and the

"O" Ring on the bleed valve are in good condition. Use a light coating of Vacuum Grease on "O" Ring seals. Make sure all pipe or tubing connections are tight and do not leak. Refer to Section 7-A.

4. Slide one paper and its sample to the bottom of the reaction cell by holding cell in horizontal position. Raise cell to vertical and dump sample onto cell bottom. Brush paper with small brush to remove traces of sample then remove the paper.
5. Pour 10 ml or 20 ml of 10% [1 Normal] Hydrochloric Acid (HCL) into the acid cup and cautiously lower the cup into the cell. Be careful not to spill or get any HCL on bottom of cup.
6. Hand tighten cell cap being careful not to splash acid onto sample.
7. Open bleed valve until a Zero pressure reading is obtained on the pressure gauge or on the recorder chart, then close the bleed valve tightly.

D. Tip the reaction cell and start timing the test. This will start the reaction between the HCl and the CaCO_3 . Observe the rapidly rising pressure and record it at 30 seconds after the reaction cell was tipped. Record this as CaCO_3 pressure. There should be a pause, then a slow second rise in pressure will be noted if dolomite is present. The dolomite reaction and pressure rise is much slower for dolomite. Swirl reaction cell and allow sufficient time for completion of the pressure build up. The reaction is assumed complete when the pressure stops increasing and remains constant. This should happen in 30 to 45 minutes. This final value of pressure is the total CaCO_3 plus dolomite pressure. To obtain the pressure due to the dolomite, subtract the calcium carbonate pressure (30 second reading) from the total reading (30-45 minute reading).

- E. For interpretation of the pressure readings. Refer to Fig. 1, 2, and 3. These are representative of pressure versus time graphs as they would appear on a Pressure Recorder Chart. Fig. 1 is representative of the CaCO_3 only. Fig. 2 is representative of dolomite only. Fig. 3 is representative of combined CaCO_3 and dolomite.
- F. Use equations [1] and [2] below for calculation of the percentages of CaCO_3 and dolomite. For values of "Slope" refer to Section 5.

$$\begin{aligned}
 [1] \quad & \% \text{CaCO}_3, \text{ as recorded} \\
 & \frac{(\text{Pressure Reading, PSI}) (100)}{(\text{Sample Weight}) (\text{Average Slope})} \quad 1
 \end{aligned}$$

$$\begin{aligned}
 [2] \quad & \% \text{ Dolomite, as recorded} \\
 & \frac{(\text{Total Press.} - \text{Press. CaCO}_3) (100) (.92)}{(\text{Sample Weight}) (\text{Average Slope})} \quad 2
 \end{aligned}$$

SECTION 4

ASTM STANDARD TEST METHOD FOR CALCIUM CARBONATE CONTENT OF SOILS

Before starting the test procedure for the ASTM Standard Test for Calcium Carbonate Content of Soils, make sure the equipment is clean and in good condition. Refer to Section 5 to verify that a calibration curve is available for the equipment to be used, and that the equipment uses a 20 ml acid cup. Should a calibration curve not be available, follow the procedures outlined in Section 5 to construct one using the 7 point plot. This ASTM test specifies the use of a 1.00 gm test specimen. For an illustration of the 7 point plot Refer to Fig. 4.

- A. Select a 20 to 30 gm. sample of Core, surface grab sample, or other specimen that is to be analyzed. The sample should be dried in an oven at 220 F (105 C) for 12 to 24 hours. Pulverize the sample with a mortar and pestle (or hammer) until all particles pass through a No. 10 (2mm) sieve or finer. The finer the sample the faster the reaction.
- B. Weigh out onto as small piece of glazed paper accurately a 1 gm. +/- 10 mg. sample of the specimen as prepared in Section A above.

NOTE: If pressure response is very low, as will be found in specimens of low calcium carbonate containment, the weight of the sample can be doubled, then dividing the pressure reading by 2 to obtain the correct calcium carbonate content. Conversely, if only a 10 ml acid cup or a 15 psi gauge or recorder is available, the size of the sample can be cut to 0.50 grams, then doubling the pressure reading.

- C. Load the Test Sample in the CO₂ reactor cell.
 - 1. Unscrew and remove the top with pressure gauge or pressure transducer from the Reactor Cell then remove the acid cup.
 - 2. Inspect to be sure CO₂ reactor cell and top are clean and dry.

3. Be sure cell "O" Ring seal on the top, and the "O" Ring on the bleed valve are in good condition. Use a light coating of Vacuum Grease on "O" Ring seals. Make sure all pipe or tubing connections are tight and do not leak.
4. Slide one paper and its 1.00 gm. test sample to the bottom of the cell while holding cell in horizontal position. Raise cell to vertical and dump sample onto cell bottom. Brush the paper with small brush to remove traces of sample then remove the paper.
5. Pour 20 ml of 10 percent HCL into acid cup (20 ml is the line on the side of the cup), and lower acid cup into cell. Be careful not to spill or get any HCL on bottom of cup.
6. Hand tighten cell cap being careful not to splash acid onto sample.
7. Open bleed valve until a Zero pressure reading is obtained on the pressure gauge or on the recorder chart, then close the bleed valve tightly.

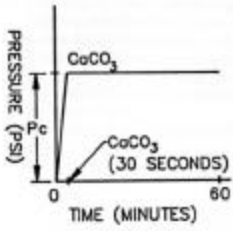


Fig. 1

CaCO₃ Pressure

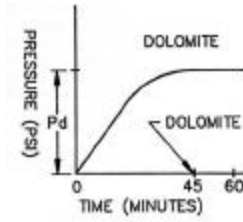


Fig. 2

Dolomite Pressure

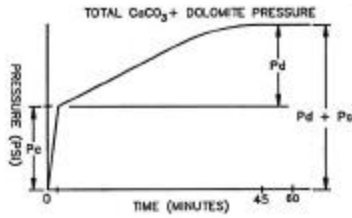


Fig. 3

Total Pressure - CaCO₃ + Dolomite

- D. Tip the reaction cell and start timing the test. This will start the reaction between the HCl and the CaCO₃. Observe the rapidly rising pressure and record it at 30 seconds after the reaction cell was tipped. Record this as CaCO₃ pressure. There should be a pause, then a slow second rise in pressure will be noted if dolomite is present. The dolomite reaction and pressure rise is much slower for dolomite. Swirl reaction cell and allow sufficient time for completion of the pressure build up. The reaction is assumed complete when the pressure stops increasing and remains constant. This should happen in 30 to 45 minutes. This final value of pressure is the total CaCO₃ plus dolomite pressure. To obtain the pressure due to the dolomite, subtract the calcium carbonate pressure (30 second reading) from the total reading (30-45 minute reading).
- E. For interpretation of the pressure readings. Refer to Fig. 1, 2, and 3. These are representative of pressure versus time graphs as they would appear on a Pressure Recorder Chart. Fig. 1 is representative of the CaCO₃ only. Fig. 2 is representative of dolomite only. Fig. 3 is representative of combined CaCO₃ and dolomite.
- F. Equations [1] and [2], Page 6, can be used for calculation of the percentages of CaCO₃ and dolomite, however since the sample size is specified as 1.00 gm these equations can be further simplified to equations [3] and [4] respectively.

Since sample weight = 1.00 gm,

$$[3] \quad \%CaCO_3 = \frac{\text{Pressure PSI} \times 100}{\text{Slope}} \quad 3$$

Since sample weight = 1.00 gm,

$$[4] \quad \%Dolomite = \frac{\text{Pressure PSI} \times 92}{\text{Slope}} \quad 4$$

SECTION 5 CONSTRUCTION OF CALIBRATION CURVE

The volume of the reaction cell of a Calcimeter of the type used for the Fann Calcimeter and described in the ASTM D 4373 controls the relationship of the pressure observed to the amount of CO₂ released by a given weight of CaCO₃ when reacted with HCl. This relationship is a constant for a given reaction cell. The calibration curve σ calculated calibration factor (the reciprocal of the slope x 100 percent of the calibration curve x 100 percent) are used to translate the observed pressure resulting from the CO₂ released by a given weight of reagent grade CaCO₃ to percent calcium carbonate. All points on the calibration curve represent 100 percent CaCO₃ for that sample weight. Any number of samples can be used to construct the calibration curve. The following are recommended to give good accuracy.

A. Weigh Out Calibration Samples

1. For 15 psi full scale gauge, 15 psi full scale recorder and a 10 ml acid cup, weigh exactly .200, .400, .600, and .700 gms of Reagent Grade CaCO₃ onto small pieces of glazed paper for calibration.
2. For 30 psi full scale gauge, 30 psi full scale recorder and a 20 ml acid cup. weigh out additional samples of .800, 1.000 , and 1.200 gms CaCO₃.

NOTE: Do not use samples of CaCO₃
larger than .700 gms unless a 20
ml acid cup is available.

B. Load a Calibration Sample.

Perform the procedure outlined in Section 3-C for Fann Test Procedure, Steps C-1 through C-7.

- ### C.
- Tip the cell and allow acid to run out of cup onto the sample. Swirl gently and continuously until a constant pressure is obtained. This will usually take about 30 seconds. Keep reactants in lower part of cell to avoid getting acid into pressure gauge or pressure transducer. As soon as the reaction is started observe the rapidly rising pressure. Record this pressure at its peak. Record this as the CaCO₃ pressure for the sample weight used.

NOTE: If a mechanical shaker is available, it may be used to

agitate the cell rather than swirling the reactants as described above. Agitate the sample for 10 minutes.

- D. Repeat the procedure outlined in Section B and C above for each of the samples prepared in Section A above. This will be a total of four samples if a 15 psi gauge or Recorder and a 10 ml acid sample cup are being used or a total of seven samples if a 30 psi gauge or recorder and a 20 ml acid cup are being used.
- E. Plot grams of CaCO_3 for each sample versus final pressure reading for each sample on linear graph paper. Draw a straight line averaged through these points. A sample graph for the four point plot and the addition for the 7 point graph is shown in Fig. 4. Larger samples of CaCO_3 cannot be reacted using the 10 ml acid cup.
- F. Note the linear relation between the pressure and sample size, therefore this curve may be assumed to be a straight line, and its slope will be a constant. As illustrated in Fig. 4 the slope of the curve is 2 psi / .1 gm CaCO_3 , or 20 psi / 1.0 gm sample, resulting in a slope of 20. Therefore the Calibration Factor in this case is $1/20 = .05 \times 100$ or 5. This number is the slope or average slope for the particular equipment calibrated. It is a function of the volume of the reaction cell.

As shown by equations [5] and [6] below the slope can be written as a "Cell Factor" to multiply the pressure reading by to directly obtain percentages of Calcium Carbonate and Dolomite.

As described above Slope = 20 psi for a 1 gm sample therefore

$$[5] \quad \%CaCO_3 = \frac{Pressure \times 100}{1 \text{ gm} \times 20} \quad 5$$

or

$$\%CaCO_3 = 5 \times Pressure \quad 6$$

$$[6] \quad \%Dolomite = \frac{(Total \text{ Press} - CaCO_3 \text{ Press})(.92)(100)}{1 \text{ gm} \times 120} \quad 7$$

or

$$= 4.6 \times (Total \text{ Press} - CaCO_3 \text{ Press}) \quad 8$$

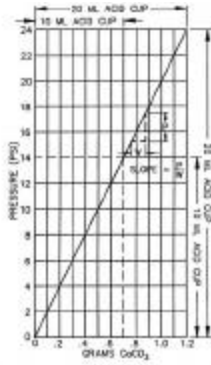


Fig. 4
CaCO₃ Calibration Curve
[10 & 20 ml Acid Cups]

SECTION 6 FANN RECORDING CALCIMETER

The Fann Recording Calcimeter differs from the pressure gauge model only in that a pressure transducer and small strip chart recorder is provided to replace the pressure gauge. This section will primarily describe the set up and operation of the transducer and recorder.

A. Description of Recorder.

The recorder is a RUSTRAK with a 2 inch strip chart and 30 inches per hour chart speed. It has been adapted for use with the Calcimeter by connecting a pressure transducer and power supply for the transducer to enable recording of pressure changes in the reactor cell as they occur. The recorder chart will read the pressure in the reaction cell. By applying the calibration factor to the chart reading the percent calcium carbonate and dolomite can be read from the recorder chart.

B. System Specifications

Power	Source	115 Volt AC, 60 Hz.
	Connection	3 wire grounded receptacle
Transducer	Pressure Range	0 to 15 psi (103.4 kPa)
	Output Signal	4 to 20 Milliampers
	Connection	3 - 18 AWG. W/ PLUG & RECEPTICAL
Recorder	Signal input	4 to 20- Milliampers
	Connection	6 terminal Plug and receptacle.
	Chart Speed	30 in/hr.
	Chart length	63 feet.
	Chart Width	2-5/16 inches
	Chart Cal.	2 inches = 15 psi.

C. Chart Installation

A warning to "RENEW CHART" appears on the last three feet of each roll of paper. Refer to item numbers [] in Fig. 5. For use with the Calcimeter, the chart paper is loaded in the "Tear Off Mode".

1. Unplug Recorder from power source before loading the paper.
2. Open the recorder by loosening thumbscrew [1].
3. Unlatch paper retaining clips [2].
4. Open panel to chassis latch [3] Right Hand side plate.
5. Slide drive belts [9] from chamfered grooves to center of top roller to release pressure on paper.
6. Remove supply roll [4]. If paper is still attached to supply roll, carefully slide the paper from between the front panel and chart drive. Do not pull the paper backward through the recorder.
7. Insert the supply roller into the new roll of chart paper. The perforated end of the paper nearest the roller shoulder.
8. Unroll about a foot of paper. Slide the paper between the panel and side plate, sprocket holes first. Keep the paper taut and close to the drive drum to prevent snagging the pointer.
9. Engage the supply roller shaft in both seating notches [6] and check to be sure that the paper sprocket holes engage the time drum sprockets.
10. Pull the drive belts [9] back into the grooves [10].
11. Close clips [2], latch [3], and recorder front panel. Tighten thumbscrew [1].
12. Advance paper with the chart advance thumb wheel [8] by pushing inward and turning downward, to assure that

paper drives through the recorder. Set the time using the chart advance thumb wheel.

13. Reconnect power to the recorder.

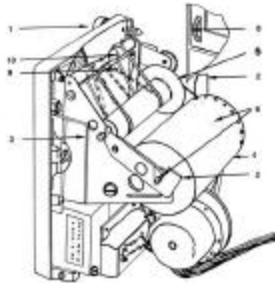


Fig. 5

**CHART INSTALLATION - RUSTRAK RECORDER
TEAR OF MODE**

SECTION 7 MAINTENANCE AND REPAIR

A. Testing For Leaks

Leaks in the pressure system are probably the greatest source of potential trouble.

1. Periodically inspect Reaction Cell and replace O-Rings if necessary.
2. Check the pipe thread connection between the reaction cell top and the pressure Gauge or transducer with a brush and soap suds. Repair by disassembling the pipe threaded connection between the reaction cell and the gauge or transducer. Use Loctite pipe sealant with teflon or similar product.
3. Check for plugging in the connection between reaction cell and gauge or transducer, also in the gauge entrance or the transducer barrel and diaphragm for build-up of calcium deposit over long periods of time. A warm Chlorox wash should clean these parts.

Note: Some units have a filter in the connection. Replace this filter if plugged.

4. On recorder models check that the paper travel is 1/2 " per minute. Refer to Section 6-C.
5. To check for leaks, pressurize the instrument as described in Section 5 -A1, B, and C. using a .6 gm. sample and let stand at least one hour. Pressure should not decrease unless leaks are present.

B. Calibration Data Does Not Give a Straight Line

If there are no leaks in the system, but results are not giving a straight line calibration curve or data is otherwise questionable check the following:

1. Check for sticking pressure gauge or recorder malfunction. Refer to Section 7-C below for possible malfunction of Rustrak recorder or transducer circuit.
2. Make sure the scale or balance is clean and free of corrosion on weights and pans. Shield balance from air currents and vibration as much as possible when weighing sample or CaCO_3 for calibration.
3. Check Reaction Cell cell for contaminants. Be sure cell is clean and dry.
4. Check for impurities in reagents. Moisture in CaCO_3 standard will result in low pressure readings.

C. Recorder Not Responding To Pressure Changes In Reaction Cell.

1. Make sure the power cable is plugged into the electrical out and that power is available at the outlet.
2. Verify the Power switch in the recorder is ON.
3. Check the plug and receptacle going into the recorder to make sure they are making a good connection.
4. Check the plug and receptacle connecting the transducer to the Recorder. Make sure this plug and receptacle are making good connection

D. Checking the Transducer Circuit

This test sequence requires a qualified electrical technician familiar with electrical test instruments and the potential hazards of electrical circuits.

1. Check AC terminals of power supply mounted on top of recorder for 115 volt AC. If No, check wiring to power supply Refer to Transducer and Power Cord Assembly and Wiring Diagram - Recording Calcimeter, 43210-W.
2. Check + to - terminals on power supply for 12 volt DC. If 115 volts is found but no 12 Volt DC, power supply is defective.

3. Test 12 Volt circuit from + on power supply to [orange wire] through recorder connector pin 2, then [red wire] to transducer. Then back from transducer [black wire] to pin 1 of connector, then to the + side of the recorder galvanometer, then from the - of the galvanometer [w/black wire] to the - of the power supply.
4. Disconnect either the [red] wire or the [W/black] wire from the power supply. Connect a suitable VOM (volt-ohm-meter) set on a milliampere scale to measure up to 20 milliamperes DC in series between the terminal and the removed wire. Pressure the Reactor Cell and the transducer up to 15 psi. Measure the current. Zero pressure should read 4 Ma, 15 psi should read 20 Ma. If not the transducer is either defective or connected backward.
5. If the Galvanometer does not follow the signal, check to see that when the striker is in its maximum open position the distance between the striker and the paper should be 1/8 inch. The pointer should be midway. If the pointer is not centered, the galvanometer could have a bent pointer, broken taut band, or cracked jewel. Defects not so visible such as open or shorted coil or debris in the air gap will also cause the problem.
6. The chart speed used on this instrument is 30 inches per hour or 1/2 inch per minute. If the chart is not moving at this rate, the chart drive motor is not getting power, is defective, or the gear train is defective.
7. If tests in 3 and 4 above show good, and the checks in 5 or 6 show problems, the recorder is defective and must be repaired.

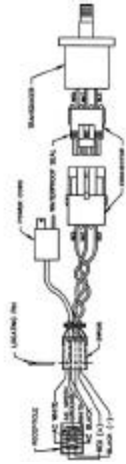


FIG. 6

TRANSDUCER AND POWER CORD ASSEMBLY

SECTION 8 PARTS LIST

PART NO.	DESCRIPTION
31141	ELL, STREET, BRASS, 1/4 FNPT X 1/4 MNPT
43201	REACTION CELL
43221	ACID CUP W/HANDLE
43211	CASE SS FOR CALCIMETER W/ RECORDER
E1011	CALCIMETER INSTRUCTIONS
E3111	POWER SUPPLY FOR TRANSDUCER
L3705	GAUGE, 30 PSI, 3.5 INCH
L4026	LUBRICANT GREASE
L4505	"O" RING, 1/4 ID X 1/16, BLEED VALVE
L4525	"O" RING, 1-3/8 ID X 1/16, CELL TOP
L6214	SWITCH, RECORDER POWER
L8152	PAPER, CROSS SECTION, F/CAL. CHART
L8480	MORTAR & PESTLE
L8810	RECORDER, RUSTRAK
L8862	PAPER, RUSTRAK, WB
N0000	BALANCE, PORTABLE
N3000	CYLINDER, 25 ML, GRADUATED
P1009	TRANSDUCER
REAGENTS	
52473	10 % HYDROCHLORIC ACID SOLUTION, 8 OZ.
52501	CALCIUM CARBONATE, REAGENT GRADE
52600	WETTING AGENT

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