
**Petroleum and natural gas industries —
Completion fluids and materials —**

**Part 4:
Procedure for measuring stimulation and
gravel-pack fluid leakoff under static
conditions**

*Industries du pétrole et du gaz naturel — Fluides de complétion et
matériaux —*

*Partie 4: Mode opératoire pour mesurer la stimulation et la fuite du
fluide filtrant dans des conditions statiques*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13503-4 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

ISO 13503 consists of the following parts, under the general title *Petroleum and natural gas industries — Completion fluids and materials*:

- *Part 1: Measurement of viscous properties of completion fluids*
- *Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations*
- *Part 3: Testing of heavy brines*
- *Part 4: Procedure for measuring stimulation and gravel-pack fluid leakoff under static conditions*
- *Part 5: Procedures for measuring the long-term conductivity of proppants*

Introduction

The objective of this part of ISO 13503 is to provide a standard procedure for measuring fluid loss under static conditions. This standard procedure was compiled on the basis of several years of comparative testing, debate, discussion and continued research by the industry¹⁾.

In this part of ISO 13503, where practical, US Customary (USC) units are included in parentheses for information.

1) PENNY, G.S. and CONWAY, M.W. Fluid Leakoff, *Recent Advances in Hydraulic Fracturing*, J.L. Gidley, S.A. Holditch, D.E. Nierode and R.W. Veatch Jr. (eds), SPE Monograph 1989.

Petroleum and natural gas industries — Completion fluids and materials —

Part 4: Procedure for measuring stimulation and gravel-pack fluid leakoff under static conditions

1 Scope

This part of ISO 13503 provides for consistent methodology to measure fluid loss of stimulation and gravel-pack fluid under static conditions. However, the procedure in this part of ISO 13503 excludes fluids that react with porous media.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1

base fluid

solution media used to prepare completion fluid

2.2

filtrate

fluid that permeates into the porous medium

2.3

filter cake

build-up of materials on the face or within the matrix of porous medium due to fluid leakoff

2.4

fluid loss

fluid loss is a measure of fluid volume that leaks into a porous medium over time

2.5

gravel-pack fluids

fluids used to place filtration media to control formation sand production from oil and gas wells

2.6

leakoff

entry of fluid into a porous media

2.7

pH

negative of the logarithm (base 10) of the hydrogen ion concentration

2.8

spurt time

time between the initial entry of fluid into porous medium and the onset of square-root-of-time leakoff behaviour

2.9
shut-in time

time from loading the cell to the initiation of leakoff test

2.10
spurt loss

theoretical loss of fluid/filtrate at first exposure of that fluid into a porous medium

2.11
stimulation fluids

fluids used to enhance production from oil and gas wells by fracturing or acidizing

2.12
viscosity-controlled fluid-loss coefficient

measure of the leakoff rate controlled by the viscosity of filtrate

2.13
viscosity of fluid

measure of the internal friction of a fluid whenever it is caused to move by an external force

2.14
wall-building coefficient

measure of the leakoff rate due to filter cake formation

3 Measurement and precision

Temperature shall be measured to a precision of ± 1 °C (± 2 °F). All other quantitative measurements shall be made to a precision of ± 2 %, unless specified otherwise.

4 Fluid preparation

Certain aspects of sample preparation and handling can affect properties of a fluid. During all procedures, steps shall be taken to minimize air entrainment into the fluid.

The procedure used to prepare the fluid sample shall be documented as follows:

- a) description and/or composition of the base fluid;
- b) base fluid pre-treatment such as filtration;
- c) preparation of the fluid, which shall be described, starting with the base fluid, such as deionized water, tap water source, seawater (location) or type of organic fluids;
- d) identification of mixing apparatus, container volume and total volume of fluid prepared;
- e) time of mixing [should include mixing time(s) at one or more mixer speed(s)];
- f) identification of each component and amount added;
- g) order and method of addition of each component;
- h) aging or holding time at temperature, if required, prior to tests;
- i) test temperature;
- j) pH (for aqueous fluids, where applicable);
- k) all other aspects of the fluid preparation that are known to affect the outcome of measurement.

5 Instrument calibration

The instruments associated with these procedures shall be calibrated according to each manufacturer's recommended method.

6 Measurement procedure

6.1 Introduction

6.1.1 General considerations

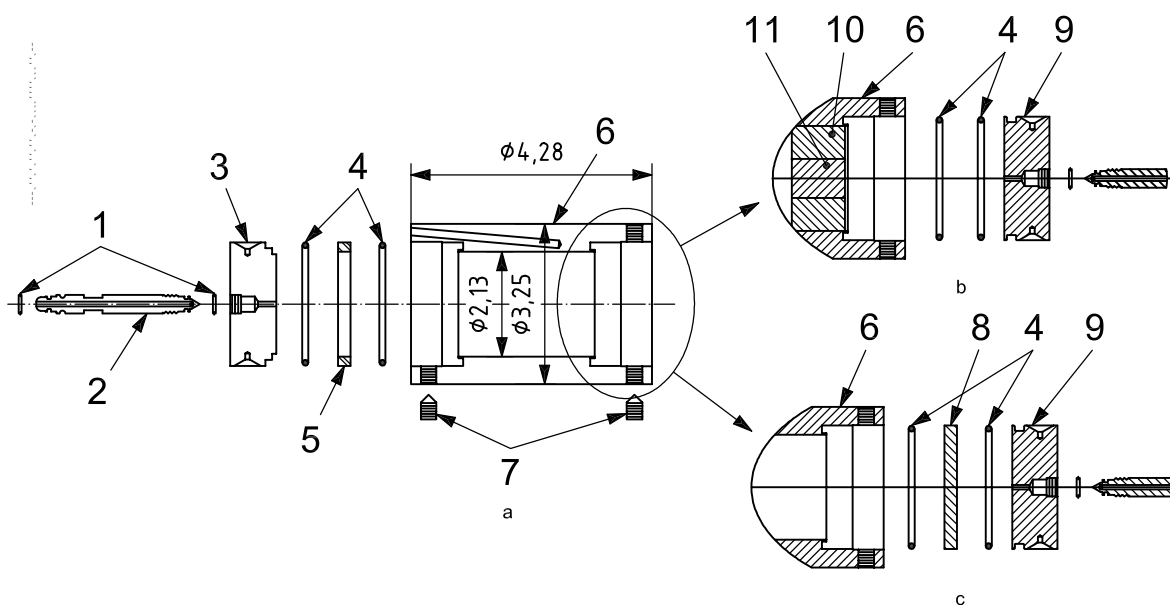
Fluid-loss tests are conducted to simulate leakoff into a formation. Fluid-loss tests measure the rate of leakoff into a porous medium to calculate fluid-loss coefficients to guide engineering design of well completion operations.

This part of ISO 13503 provides guidelines on known limitations to the testing procedure. Where data are reported as being obtained using this procedure, the procedure shall be followed exactly. The fluid shall not react with instrument surfaces to generate contaminants, change critical measurement dimensions or impair proper mechanical operation.

6.1.2 Apparatus

Figures 1 and 2 present drawings of two types of typical static fluid-loss apparatus²⁾ with 175 ml and 500 ml capacities, respectively.

2) Examples of suitable fluid-loss cells are Baroid HPHT Filter Press Part Number 38700 and Chandler Engineering Model 4214. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of these products.



Key

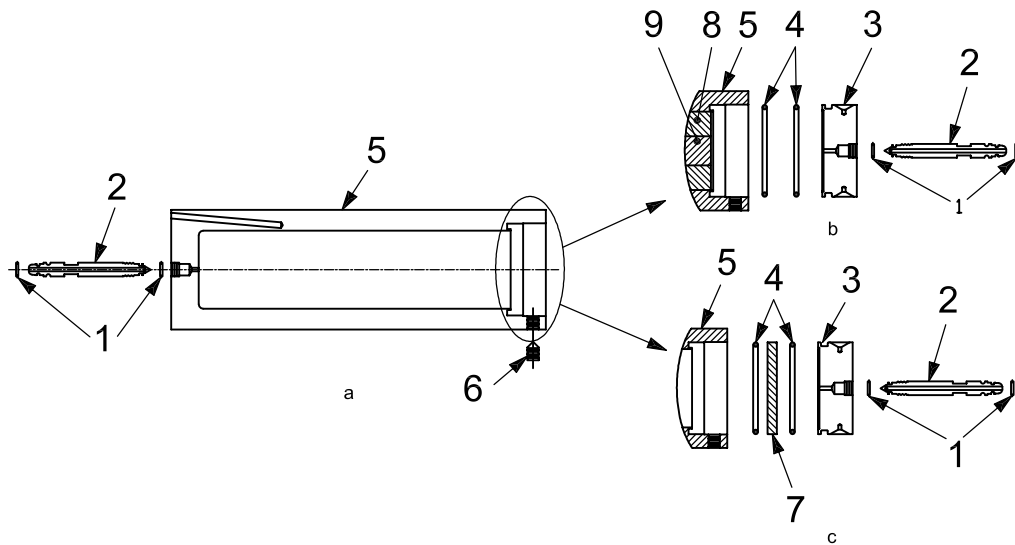
- 1 O-ring seal
- 2 stem/valve
- 3 top cap
- 4 O-ring seal
- 5 backup ring
- 6 cell body
- 7 set screw
- 8 filter-paper assembly or synthetic core
- 9 bottom cap
- 10 seal mechanism
- 11 natural core

a Assembly fluid-loss cell, 175 ml, 12 400 kPa (1 800 psi), 303 SS.

b Natural core.

c Synthetic core or filter-paper assembly.

Figure 1 — Typical 175 ml fluid-loss cell

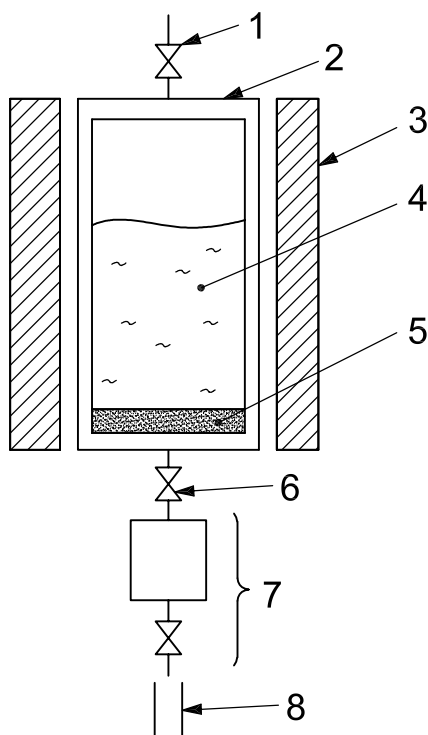


Key

- 1 O-ring seal
 - 2 stem/valve
 - 3 bottom cap
 - 4 O-ring seal
 - 5 cell body
 - 6 set screw
 - 7 filter-paper assembly or synthetic core
 - 8 seal mechanism
 - 9 natural core
- a Assembly fluid-loss cell, 500 ml, 12 400 kPa (1 800 psi), 303 SS.
 b Natural core.
 c Synthetic core or filter-paper assembly.

Figure 2 — Typical 500 ml fluid-loss cell

The type of fluid-loss cell is not specified. However, the fluid-loss cell should permit use of filter paper, natural- or synthetic-core samples as the filter medium. It shall be further equipped with a back-pressure receiver to be used when the test temperature exceeds the boiling point of the filtrate. Both the fluid-loss cell and back-pressure receiver shall have operating limits of at least 10 342 kPa (1 500 psi) and 121° C (250 °F). The test core or filter medium shall be mounted within the cell in such a way that fluid cannot bypass the core or filter medium. A schematic diagram of fluid-loss apparatus is shown in Figure 3.



Key

- 1 pressurizing valve
- 2 fluid-loss cell
- 3 heating source
- 4 sample fluid
- 5 porous medium
- 6 filtrate valve
- 7 back-pressure receiver, optional
- 8 filtrate collector

Figure 3 — Static fluid loss schematic

6.2 Core

6.2.1 Selection

A core sample with permeability and porosity similar to that of the formation to be treated is preferred (formation core may be used). The core shall be 2,54 cm (1,0 in) long and 2,54 cm (1,0 in) in diameter. Permeability of the core to air shall be determined. A synthetic, porous filter medium with physical properties similar to natural rock may also be used.

6.2.2 Preparation

The core shall be saturated with the base fluid or synthetic formation fluid (examples are 2 % by mass KCl or 4 % by mass NH₄Cl). In case of unknown formation fluid, the core shall be saturated with a non-sensitive brine solution that doesn't react with the matrix mineralogy.

7 Operational procedure

7.1 Assembly

There are three procedures to assemble fluid-loss cells depending on the porous medium. These procedures are described below.

7.1.1 Filter-paper medium

Place the spacer at the bottom of the cup and a 38 μm (400 US mesh) screen on the spacer. Making sure the bottom valve is closed, introduce the base fluid into the cell to assure all the dead volume is filled. Then, place three 8 μm pore-size cellulosic filter papers³⁾ on top of the screen. Assemble the top and close the upper valves. Place the cell into a heat jacket and connect the back-pressure receiver if the test temperature is above the boiling point of the fluid. Connect the pressure line to the top valve. The back-pressure receiver and heat jacket should be operated according to the manufacturer's procedure.

7.1.2 Natural core

Place the spacer, if applicable, at the bottom of the cup. Making sure the bottom valve is closed, introduce the base fluid into the cell to assure all the dead volume is filled. Then, place the pre-saturated core plug of 2,54 cm (1,0 in) diameter and 2,54 cm (1,0 in) length in a core holder and place it inside the cell according to the manufacturer's procedure. Assemble the top and close the upper valves. Place the cell into a heat jacket and connect the back-pressure receiver if the test temperature is above the boiling point of the fluid. Connect the pressure line to the top valve. The back-pressure receiver and heat jacket should be operated according to the manufacturer's procedure.

7.1.3 Synthetic core

Place the spacer at the bottom of the cup and a pre-saturated ceramic disk, 6,35 cm (2,5 in) in diameter and 0,635 cm (0,25 in) thick or a pre-saturated synthetic core of similar size on top of the spacer. Making sure the bottom valve is closed, introduce the base fluid into the cell to assure all the dead volume is filled and assemble the porous medium. Assemble the top and close the upper valves. Place the cell into a heat jacket and connect the back-pressure receiver if the test temperature is above the boiling point of the fluid. Connect the pressure line to the top valve. The back-pressure receiver and heat jacket should be operated according to the manufacturer's procedure.

7.2 Test procedure

Apply a constant pressure to the cell, typically 6 895 kPa (1 000 psi) above the intended back-pressure, by opening the top valve. Allow the fluid to reach test temperature. Optionally, a shut-in time may be applied. Once at test temperature (or completion shut-in time), open the bottom valve and collect the filtrate into a graduated cylinder and record the collected volume as a function of time. Typically time intervals of 1 min, 2 min, 4 min, 9 min, 16 min, 25 min and 36 min are used. The volume may be collected in a container, making sure the evaporation is minimized (the volume may be calculated from fluid mass by collecting the fluid in a tared container). These data are used for calculating spurt loss, the fluid-loss coefficient or the completion fluid's filtrate viscosity.

3) Example: Whatman 40.

8 Calculations

8.1 Fluid-loss graph

A fluid-loss graph is constructed by plotting the filtrate volume versus time in minutes using rectilinear coordinates. A plot of the data is linear if the fluid loss is viscosity-controlled (see 8.2). If the fluid loss is wall-building, the plot will be non-linear with respect to time and it will follow the square root of time (see 8.3).

8.2 Viscosity-controlled leakoff coefficient

If the plot is linear through the origin (see 8.4.1 for example), the filtrate viscosity, μ , at test temperature, expressed in centipoise, is calculated according to Equation (1):

$$\mu = \frac{kA\Delta P}{QL} \quad (1)$$

where

- k is the permeability to liquid, expressed in darcies;
- A is the cross-sectional area of porous medium surface exposed to liquid, expressed in square centimetres;
- ΔP is the differential pressure across the filtration medium, expressed in atmospheres;
- Q is the flow rate, expressed in cubic centimetres per second;
- L is the length of filtration medium, expressed in centimetres.

Using the calculated filtrate viscosity, the fluid-loss control coefficient, C_v , expressed in $\text{m/s}^{1/2}$ ($\text{ft/min}^{1/2}$), due to fluid viscosity, can be determined using the general Equation (2), which can be rearranged as shown in Equations (3) and (4):

$$C_v = \sqrt{\frac{k\Delta P\phi}{2\mu}} \quad (2)$$

$$C_v = 0,707 \sqrt{\frac{k\phi\Delta P}{\mu}} \quad (\text{expressed in SI units}) \quad (3)$$

$$C_v = 0,0469 \sqrt{\frac{k\phi\Delta P}{\mu}} \quad (\text{expressed in USC units}) \quad (4)$$

where

- k is the permeability to liquid, expressed in square metres (darcies);
- ϕ is the effective porosity of the filtration medium, dimensionless fraction;
- ΔP is the differential pressure across the filtration medium, expressed in pascals (pounds per square inch);
- μ is the viscosity of the filtrate at test temperature, expressed in pascal-seconds (centipoise).

8.3 Wall-building coefficient

When the plot of filtrate volume versus time is non-linear, then plot the filtrate volume, expressed in millilitres, against the square root of time. Using the last three data points collected (typically 16 min, 25 min, 36 min), project a straight line back to the ordinate axis to obtain a zero-time intercept visually and to calculate the slope of the line (see 8.4.2 for example). Alternatively, one may use the least square error to calculate the intercept and slope. The slope, m , for the three data points, is calculated as given in Equation (5):

$$m = \frac{3 \sum_{i=1}^3 t_i v_i - \sum_{i=1}^3 t_i \sum_{i=1}^3 v_i}{3 \sum_{i=1}^3 t_i^2 - \left(\sum_{i=1}^3 t_i \right)^2} \quad (5)$$

where

- t_i is the square root of time;
- v_i is the filtrate volume eluted at time t_i ;
- i is the number of the data point, 1 to 3.

The intercept, b , is calculated as follows:

$$b = \bar{v} - m \bar{t}_i \quad (6)$$

where

- \bar{t}_i is the average of the square root of time readings;
- \bar{v} is the average of the volume eluted readings.

Using these two values, calculate the wall-building leakoff coefficient, C_w , expressed in $\text{m/s}^{1/2}$ ($\text{ft}/\text{min}^{1/2}$), as given in Equations (7) and (8) and spurt loss, S_L , expressed in m^3/m^2 (gal/ft^2), as given in Equations (9) and (10):

$$C_w = \frac{m}{2A} \quad (\text{expressed in SI units}) \quad (7)$$

$$C_w = 0,0164 \frac{m}{A} \quad (\text{expressed in USC units}) \quad (8)$$

$$S_L = \frac{b}{A} \quad (\text{expressed in SI units}) \quad (9)$$

$$S_L = 0,246 \frac{b}{A} \quad (\text{expressed in USC units}) \quad (10)$$

where

- m is the slope of the fluid-loss curve, $\text{m}^3/\text{s}^{1/2}$ ($\text{cm}^3/\text{min}^{1/2}$);
- A is the cross sectional area of the filter medium, square metres (square centimetres);
- b is the value of filtrate volume at $t_i = 0$ from the fluid-loss curve, cubic metres (cubic centimetres).

8.4 Examples

8.4.1 Example of viscosity-controlled leakoff

The data from Table 1 are used.

$$\mu = [(0,1 \times 5,07) \times 68,04] / (0,013 \times 2,54) = 1\,045 \text{ cP (expressed in USC units)}$$

$$= 1,045 \text{ Pa}\cdot\text{s (expressed in SI units)}$$

$$C_v = 0,0469 \sqrt{\frac{0,1 \times 0,23 \times 1\,000}{1\,045}} = 0,007 \text{ ft/min}^{1/2}$$

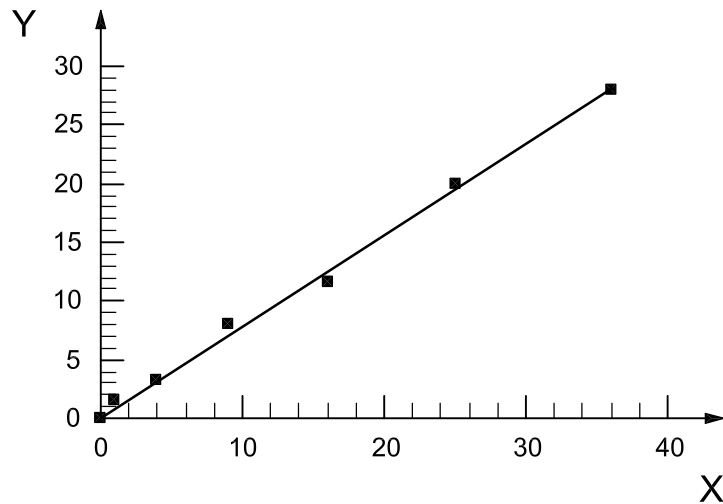
$$C_v = 0,707 \sqrt{\frac{0,0987 \times 10^{-12} \times 0,23 \times 6\,894\,757}{1,045}} = 0,000\,274 \text{ m/s}^{1/2}$$

Table 1 — Example of viscosity-controlled leakoff

HTHP filter press parameters		
Natural core material		Unit
Length	2,54	cm
Diameter	2,54	cm
Area	5,07	cm ²
Porosity	23	%
ΔP_{core}	1 000	psi
	68,04	atm
	6 894 757	Pa
k	100	mD
	0,987E- 12	m ²
Temperature	125	°F
	51,6	°C

Table 2 — Leakoff data (See Figure 4)

Time	Volume
min	cm ³
0,00	0,00
1,00	1,50
4,00	3,30
9,00	8,00
16,00	11,60
25,00	20,00
36,00	28,00
Q (cm ³ /s)	0,0130

**Key**

X time, expressed in minutes

Y volume, expressed in cubic centimetres

Figure 4 — Linear-leakoff chart (See Table 2)**8.4.2 Example calculation for a wall-building coefficient**

The data from Table 3 are used.

$$m = 1,75 \text{ cm}^3/\text{min}^{1/2}$$

$$b = 9,3 \text{ cm}^3$$

$$A = 31,67 \text{ cm}^2$$

$$C_w = 0,0164 \frac{1,75}{31,67} = 0,0009 \text{ ft}/\text{min}^{1/2}$$

$$S_L = 0,264 \frac{9,3}{31,67} = 0,0725 \text{ gal}/\text{ft}^2$$

$$m = 2,25 \times 10^{-7} \text{ m}^3/\text{s}^{1/2}$$

$$b = 9,3 \times 10^{-6} \text{ m}^3$$

$$C_w = \frac{2,25 \times 10^{-7}}{2 \times 31,67 \times 10^{-4}} = 3,55 \times 10^{-5} \text{ m}/\text{s}^{1/2}$$

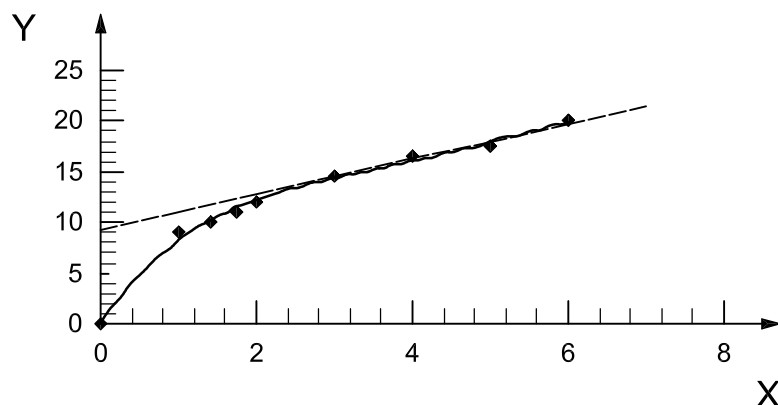
$$S_L = \frac{9,3 \times 10^{-6}}{31,67 \times 10^{-4}} = 2,94 \times 10^{-3} \text{ m}^3/\text{m}^2$$

Table 3 — Example of wall-building coefficient

HTHP filter press parameters		
Synthetic core material		Unit
Length	0,635	cm
Diameter	6,35	cm
Area	31,67	cm ²
	3,17E-03	m ²
Porosity	43	%
ΔP_{core}	1 000	psi
	6 894 757	Pa
k	750	mD
	0,740E-12	m ²
Temperature	150	°F
	65,6	°C

Table 4 — Leakoff data (See Figure 5)

Time	Volume
min	cm ³
0,00	0,00
1,00	9,00
2,00	10,00
3,00	11,00
4,00	12,00
9,00	14,50
16,00	16,50
25,00	17,50
36,00	20,00

**Key**

- X time, expressed as the square root of the minutes
 Y filtrate volume, expressed in cubic centimetres

NOTE The dashed line is extrapolated.

Figure 5 — Static-leakoff chart (See Table 4)

9 Report

A report should include as a minimum the following information.

9.1 General data

- a) date;
- b) name of person(s) performing the test;
- c) name of organization/laboratory.

9.2 Fluid data

Fluid description shall include source, base-fluid identifications and additive concentrations.

9.3 Fluid-loss apparatus data

- a) manufacturer;
- b) model number;
- c) container volume.

9.4 Test parameters

- a) filter medium properties:
 - type,
 - diameter,

- length/thickness,
 - porosity,
 - permeability to liquid;
- b) pressure;
- c) temperature;
- d) fluid volume tested;
- e) shut-in time.

9.5 Results

Tabulate fluid data according to the outline given in Clause 4. Tabulate the leakoff data and the calculated results as shown in the examples in Clause 8.

10 Procedure modifications

If a modification of this procedure is used it shall be described in sufficient detail in the report to allow others to reproduce the data.

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