

CONSTRUCTION STANDARD
FOR
INSIDE PIPE CHEMICAL CLEANING

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1. SCOPE

This Standard specification covers the minimum requirements for Internal Chemical Cleaning of Piping Systems on upstream side of process machineries and lube oil/seal oil systems for which removal of rust, mill scale, grease and foreign matters is essential.

2. REFERENCES

Throughout this Standard the following standards and codes are referred to. The editions of these standards and codes that are in effect at the time of publication of this Standard shall, to the extent specified herein, form a part of this Standard. The applicability of changes in standards and codes that occur after the date of this Standard shall be mutually agreed upon by the Company and the Vendor.

ASTM (AMERICAN SOCIETY FOR TESTING AND MATERIALS)

D-887 "Practice for Sampling Water-Formed Deposits"

3. DEFINITIONS AND TERMINOLOGY

The following terms used hereinafter shall be understood to mean:

3.1 Executor

The Executor is the party which carries out all or part of construction and/or commissioning of the project.

3.2 Engineer

A person who shall be appointed from time to time by the Company to exercise the functions entrusted to him under the contract and whose appointment has been notified by the Company in writing to the executor.

4. GENERAL

4.1 The Executor shall furnish all equipment and materials such as pumps, filters, chemicals, piping, hoses etc. and provide technical personnel and labor to supervise and perform the job.

4.2 The Executor shall be responsible for the disconnection of any piping from equipment, if required, prior to cleaning.

4.3 The Executor shall be responsible for the removal of items such as valves with trim which would be attacked by the cleaning agents.

4.4 All work shall be coordinated with and executed under the direction of the Engineer. The Executor shall consult the Engineer prior to mobilization and seek his advice on adequacy of preparation activities before performing the work.

4.5 The Executor shall be responsible for the removal and proper and lawful disposal of all chemicals used for cleaning from working area and its surrounding without any cost incurring to the Company and to the satisfaction of the Engineer. The Executor shall not dispose acids, spent acids or other chemical cleaning agents into existing drainage systems.

5. CLEANING PROCEDURE

5.1 Normal Cleaning

Prior to chemical cleaning operation, normal cleaning shall be performed by flushing and/or mechanical means, as applicable.

5.2 Chemical Cleaning

All piping indicated on the drawing or otherwise specified shall be chemically cleaned in accordance with this Standard specification.

5.2.1 Preparation for chemical cleaning

a) Generally, installed piping shall be cleaned in place after hydrostatic testing has been completed, the systems shall be checked for leaks, prior to cleaning by chemicals.

Cleaning may be performed in a bath and/or by soaking or alternately portions of piping systems may be connected together in a predetermined location and cleaned using the circulation method unless field conditions dictate otherwise. However, after cleaning has been performed, no further welding work shall be carried out.

b) Piping to be cleaned shall be disconnected or blanked off from mechanical equipment. Chemicals shall not be circulated through bearings, cylinders, oil filter cartridges, instruments, screens and filters. Valves and equipment may be included in the circulation system provided that their materials are resistant to chemical solutions involved and prior approval of the Engineer is obtained. Otherwise, the Executor is responsible for any possible damage which may occur due to chemical reaction between the parts and the chemical, and shall replace the damaged parts at his own expense.

c) The Executor, in preparing his circulating system, shall ensure that circulation is complete and that there are no dead ends or branches that will create traps. The Executor shall make sure that there are adequate existing high point vents and low point drains in the circulation system. If additional high point vents and low point drains are required, they shall be installed by the Executor after obtaining Engineer's approval.

d) The portable pumping set provided by the Executor shall be capable of safe handling of the chemicals to be used. The pump design shall be such to have the desired capacity and shall be capable to circulate the chemical at velocity required for cleaning of piping system.

e) Prior to pickling, all heavy deposits of oils, greases, soils or foreign matter other than mill-scale, rust or rust-scale shall be removed by precleaning as cited in 5.1.

f) Exposed flange faces and pipe threads shall be protected from chemical solutions.

g) All solutions, during the recirculation sequence shall be filtered. Full flow filter or equipment shall be equipped with screen capable of removing particles that are 4 micron or more in diameter.

h) The Executor shall prepare chemical cleaning procedure.

This procedure shall be approved by the Engineer prior to implementation. The procedure must include but not limited to the following items.

- The result of the tests on composition of precipitates.
- Precleaning.
- Exact compositions (percentages) of cleaning solutions and inhibitors.
- Inhibitors to be used to prevent corrosion and hydrogen - brittleness.

- Temperatures of the solutions during cleaning.
- Circulation times.
- Indication when the solutions will be renewed; concerning: Iron content, acid concentration pollutions, etc.
- Corrosion to be expected (Degree of attack and uniformity).
- Flow velocities.
- Neutralization.
- Passivation.
- Flushing and drying.
- Preservation after cleaning.

5.2.2 Consideration to be given in chemical cleaning

5.2.2.1 Only clean fresh water (e.g. tap water) shall be used in preparing the acid concentration where pickling is performed in carbon steel or low alloy steel piping. Distilled or demineralized water shall be used in preparing the acid concentration where stainless steel piping is subject to chemical cleaning.

5.2.2.2 The pickling time shall be kept to a minimum.

5.2.2.3 The acid solution must not be allowed to stand undisturbed in any part of the system at any time.

5.2.2.4 Acid, inhibitors and neutralizers shall not harm the various materials of the system to be cleaned and shall be in accordance with accepted practice.

5.2.2.5 Before changing from one to another fluid, the piping must be allowed to drain sufficiently so that as little acid or water as possible is mixed with the subsequent fluid.

5.2.2.6 Process pumps shall not be used to circulate the acid solution.

5.2.2.7 All heating has to be external to the systems being cleaned. Live steam shall never be introduced into any part of the system.

5.2.2.8 Passivation shall be performed immediately after neutralization process.

5.2.2.9 In addition to all instruments (e.g. flow meters, thermowells, pressure gages, etc.), equipment, valves and other materials which may be attacked by the acid, corrosion coupons other than corrosion coupons which are required for pickling shall be removed from the system intended for cleaning.

5.2.2.10 The number and installation point of corrosion coupons required for the pickling shall be approved by the Engineer.

5.2.2.11 Suitable acid resistance gate valves shall be installed on the point of vent and drain by the Executor.

5.2.2.12 Installation point of instruments shall be tightly plugged after removal of the instruments.

5.2.2.13 Suitable spools shall be installed in place of removed valves and equipment.

5.2.2.14 During pickling with circulating system the velocity of the flushing, rinsing, acidizing, neutralizing and passivating shall be kept within the range of 90 to 150 cm/sec. (3 to 5 ft/sec.)

The power and capacity of the pump(s), size of connections and their set up shall be so that the above mentioned velocity range can be maintained in all parts of the system to be cleaned and there shall not exist dead ends.

5.2.2.15 The metal composition of piping to be cleaned should be provided to Executor prior to pickling for selection of suitable acid and pickling procedure.

5.2.2.16 The volume of the systems to be pickled shall be calculated by the Executor prior to commencement of the pickling.

5.2.2.17 The volume of the tank which shall be used for circulation process shall be at least 10% more than that of the system which has the largest volume and is subject to pickling.

5.2.2.18 For taking samples during pickling process suitable points near the inlet and outlet of cleaning system shall be selected. These points shall be approved by the Engineer and sampling facilities shall be installed at these points.

5.2.2.19 The Executor must include in his procedure the methods by which he intends to check acid solution in order to maintain the recommended concentrations (i.e. hydrometer, titration etc.).

5.2.3 Tests to be performed prior to pickling

Prior to pickling a sample shall be taken from deposit of each system to be pickled and analyzed by the Executor. The percentage of the following items in the deposit shall be determined using test methods (ASTM D-887).

- Iron content (including Fe⁺⁺ and total Iron)
- Silica
- Carbonates
- Phosphates
- Hydrocarbons

Based on result of the tests decision shall be made for the type and concentration of acid to be used and pickling procedure.

5.2.4 Cleaning of carbon steel piping by pickling

Proceeding outlined below gives requirements for various processes of pickling. The cleaning procedure proposed by the Executor shall include, but not limited to these requirements.

Circulation process shall be adopted for chemical cleaning of carbon steel piping unless, in special conditions where this process appears to be impractical; in such instances approval of the Engineer shall be obtained for using other process of pickling.

If equivalent chemicals are intended to be used it shall be clearly stated in the Executor's proposed procedure.

5.2.4.1 Pickling by circulation process

The following steps shall be taken in circulation process:

a) Flushing

Prior to pickling the system shall be flushed with clean fresh water (potable water is accepted). The system shall be checked for leaks. If any leak is observed the executor shall take remedial action to stop the leak and flushing shall be continued until the visual inspection of outlet water indicates it has same appearance of inlet water.

During flushing water velocity shall be kept within the range of 90 to 150 cm/sec. (3 to 5 ft/sec.); it is preferred to stick to about 150 cm/sec.

During flushing the system shall be packed with the water and no air shall be present in high points.

b) Degreasing

After completion of flushing, degreasing shall be commenced to eliminate presence of oil and/or paint stuck to the inside surface of pipe. A solution of Soda ash with concentration of 1 to 3 percent together with 0.05 percent penetrating agent and 0.5 to 1 percent three Sodium phosphate as additive should be used for degreasing. During degreasing operation, the solution temperature shall be maintained between 70°C and 80°C. The following solutions may also be used as alternative provided that approval of the Engineer is obtained.

1) 10% by weight solution of technical grade sodium hydroxide with 0.05% penetrating agent at temp. range of 65 to 80°C.

2) Alkaline degreasers

The degreasing solution shall be circulated in the system for 2 hours then it shall remain stagnant for 12 hours; thereafter the solution shall be circulated for one to two hours and finally drained.

During degreasing, tests shall be made for the followings at one hour intervals:

- Soda content of the outlet fluid.
- pH value of the outlet fluid.
- Oil content of the fluid.

Presence of sufficient active Soda in the system outlet is indicative of satisfactory completion of the degreasing operation.

c) Rinsing

After completion of degreasing, flushing with clean fresh water in ambient temperature with the same procedure as described in Section 5.2.4.1(a) shall be conducted. Rinsing shall be continued until a pH value of 7 to 8 is achieved as indicated on hydriion paper or equivalent.

d) Acid wash

After rinsing, acid wash shall be started, especially where system valves are included in the operation, it is necessary to utilize an inhibited phosphoric, hydrochloric, sulphuric or less aggressive organic acids. Type of the acid shall be chosen based on the result of test on deposit to be removed (see Clause 5.2.3).

The Executor shall exercise care and take necessary precautions against the followings:

- 1) The Executor shall ensure that there are no alloys other than carbon steel in the system which is subject to acid wash.
- 2) The Executor is responsible to make sure that all spent acid solutions are effectively inhibited.
- 3) The duration of acid wash operation must not be of such length to give greater than 5 microns (0.2 mil) general metal loss on the most vulnerable alloy, weld and heat affected zones, or galvanic couples in the composite metallurgical system. No pitting will be accepted. Metal loss may be determined by thickness measurements on pipe body or installation of corrosion test coupons.
- 4) The difference between Iron ions at the beginning and during acid wash shall not exceed 5000 ppm by weight. If this difference exceeds 5000 ppm, it means that no proper acid and/or inhibitor is used and the pipe metal is being solved in the acid. In this case the acid wash shall be immediately stopped and the acid be drained from the system followed by proper rinsing until the pH of 7 to 8 is achieved. Then the type and concentration of acid and inhibitor shall be changed. This change shall be approved by the Engineer prior to resumption of acid wash.

The concentration of the acid shall be chosen so that 80 to 95 percent of the precipitates on the pipe body can be dissolved in it within a period of 6 hours.

Based on the type of acid, suitable inhibitor shall be used. If the tests mentioned in 5.2.3 indicate presence of Silica in deposit, hydrofluoric acid or one of its salts with suitable concentration (as indicated earlier in this Standard) shall be added to the solution.

The acid shall be injected into the system and circulated at velocity of 90 to 150 cm/sec. (3 to 5 ft/sec.) for 6 to 8 hours. During acid wash following tests shall be conducted. Time intervals of the tests can be one hour but it shall be reduced to half an hour after 6 hours of circulation. The tests shall determine followings:

- pH value of acid
- Percent of acid
- ppm of Iron (Fe^{++} and total Iron)
- Percent of silica

At the end of the acid wash all rust, mill scale and foreign materials shall have been removed from the system. If visual inspection reveals presence of these matters the acid wash shall be repeated with the time interval which shall be approved by the Engineer.

e) Rinsing with fresh water or removal of acid by Nitrogen gas

After acid wash the system shall be rinsed using clean fresh water. Nitrogen gas may also be used for removal of acid. If fresh water is used for rinsing, it shall be circulated until pH value of 7 is achieved.

If Nitrogen gas is used for acid removal, it shall be injected from vent (highest point) and the spent acid be drained from the lowest point, until all fluids is drained.

f) Rinsing with organic acid

After rinsing with fresh water or acid removal by Nitrogen gas, the system shall be rinsed with citric acid with concentration of 1% for removal of free iron ion which is not removed by water rinsing (or Nitrogen gas). The duration of this step shall be 1 to 3 hours at ambient temperature.

g) Rinsing with fresh water or displacement with Nitrogen gas

After step "f" the system shall be rinsed once again with fresh water or displaced with Nitrogen gas as indicated in Paragraph "e" above.

h) Neutralization and passivation

After step "g" Neutralization and passivation shall be performed as one step. The solution shall be 1 to 3 percent by weight Soda ash with 0.5 to 1 percent three sodium phosphate or 1% NaOH or 0.6% Sodium Nitrate (Nitron). It should be noted that Nitron is preferred to other additives.

The solution shall be circulated for 2 to 4 hours with a temperature of 50 to 60°C.

i) Flushing and drying

After steps "a" to "h" are completed, the system shall be flushed with hot dry air until the system is completely dried. The dew point of spent air shall be lower than the minimum ambient temperature.

5.2.4.2 Pickling by soaking process

In chemical cleaning by soaking process the same concentrations and temperatures specified in circulation process shall be used and the same steps shall be followed except that the fluid(s) shall be injected from the lowest point of the system (drain) and air (or previous spent fluid) shall be vent from the highest point (vent). Since there is no movement of fluid(s), the duration of acid wash with soaking process shall be kept longer than that of the circulation process, but in any case the end of each step shall be determined as indicated in circulating process (steps a to i in Clause 5.2.4.1).

5.2.4.3 Pickling by dipping process

In dipping process a bath shall be prepared with a size that the biggest portion of piping system can be immersed into it. Then fluids with the same concentration and temperature as indicated in Paragraph 5.2.4.1 shall be used and same steps as those cited for circulation process shall be followed for dipping the piping system into the fluid baths.

5.2.5 Pickling stainless steel piping

Pickling of stainless steel piping should be avoided, but if project specification calls for it stainless steel piping may be chemically cleaned as per detail procedure cited in Clause 5.2.4 taking the following exceptions into account:

5.2.5.1 Only distilled and/or demineralized water shall be used in flushing, rinsing, preparation of acids, neutralizing, passivating and degreasing fluids.

5.2.5.2 Only phosphoric acid, sulphuric acid and/or organic acids shall be used for acid wash. Requirements given in Clause 5.2.4 in respect of concentration, temperature, and duration of pickling shall be adhered to.

5.2.5.3 Hydrochloric acid shall not be used in any circumstances.

5.2.5.4 Chloride concentration during pickling process shall never exceed 1 mg/kg (1 ppm by weight) before, during and after pickling.

5.2.6 Protection of cleaned piping

After completion of acid wash and subsequent drying of piping system, the hot air shall be completely displaced with Nitrogen gas. The system shall be tightened to obviate leakage of Nitrogen gas to open atmosphere and a positive pressure shall be maintained until the commissioning process gets started.

6. INSPECTION

6.1 The Engineer reserves the right to inspect and/or supervise cleaning at any step of the operation and to make any check deemed necessary to ensure that the desired degree of cleanness has been achieved.

6.2 During chemical cleaning in addition to the tests listed in Clause 5.2.4.1 the attack of the surfaces shall be checked by using pretreated steel plates. These tests shall be performed according to the Attachment A adopted to the field condition with approval of the Engineer.

7. DOCUMENTATION REQUIREMENTS

Chemical cleaning procedures, and test results shall be submitted to the Engineer on completion of chemical cleaning and prior to commissioning of the system. The documents shall include but not necessarily limited to the followings:

7.1 Approved Chemical cleaning procedure.

7.2 Flow diagram of the system(s) which have been chemically cleaned.

7.3 A certificate signed and approved by the Engineer showing that all piping system which require internal cleaning have been satisfactorily cleaned based on approved procedure.

7.4 The test results conducted before, during and after chemical cleaning.

7.5 The preservation system including the type of purging fluid and the pressure of the system(s).

Note:

The date of execution, the name and signature of the Executor shall appear on all documents.

ATTACHMENT A**1. TESTING OF PICKLING BATHS****1.1 Iron Content**

The method described below gives accurate results only if all the iron present is in the form of ferrous salts.

This is generally the case if the sample is taken from a pickling bath which is in operation (or in which pickling has taken place only a short while previously). The sample must then be tested immediately after having been taken. In all other cases part of the sample (approx. 25 ml) must first be reduced (e.g. with zinc amalgam) until all the iron is present in the form of ferrous salts, after which the sample is tested according to the following method.

Chemicals

4 n sulphuric acid.

85% wt phosphoric acid

Manganese sulphate solution, obtained by dissolving 70 g $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ in 500 ml distilled water and adding 125 ml 85% phosphoric acid and 125 ml concentrated sulphuric acid, and then making the solution up to 1 litre with distilled water.

0.1 n potassium permanganate.

Procedure

Dilute a suitable quantity, V ml, bath fluid with distilled water to about 100 ml. If the bath fluid does not contain any hydrochloric acid, add 10 ml sulphuric acid and 0.5 ml phosphoric acid. If it does, add 25 ml manganese sulphate solution. Titrate with potassium permanganate until the pale pink color remains for 15 seconds (a ml).

Calculate the iron content of the bath fluid in g/l from $\frac{\alpha}{V} \times b \times 56$, in which b represents the titre of the potassium permanganate.

1.2 Acid Content

The titration method with methyl orange as indicator described below is simple to carry out, but has the drawback that the correct change of color is not clearly perceptible to everyone.

Therefore a method with phenolphthalein as indicator is described as an alternative, but this is less accurate.

The titration can also be carried out potentiometrically (not further described here). This requires a relatively expensive apparatus, but the method is more suitable, particularly for conducting larger series of tests.

Chemicals

1 n caustic soda solution and 0.1 n caustic soda solution.

Methyl orange.

Phenolphthalein.

(to be continued)

ATTACHMENT A (continued)

Procedure

a) Acid content of pickling baths

a.1) Methyl orange indicator

Dilute a suitable quantity, V ml, bath fluid with distilled water to about 100 ml. Add a few drops of methyl orange and titrate with 1 n caustic soda solution until the color changes from red to yellow (a ml).

Calculate the acid content of the pickling bath in g/l from:

$$\begin{aligned} \frac{\alpha}{V} \times b \times 36.5 \text{ g/l} & \quad \text{if the acid is hydrochloric acid.} \\ \frac{\alpha}{V} \times b \times 49 \text{ g/l} & \quad \text{if the acid is sulphuric acid.} \\ \frac{\alpha}{V} \times b \times 98 \text{ g/l} & \quad \text{if the acid is phosphoric acid.} \end{aligned}$$

in which b represents the titre of the caustic soda solution.

a.2) Phenolphthalein indicator

Dilute a suitable quantity, V ml, bath fluid with distilled water to approx. 100 ml. Add a few drops of phenolphthalein and titrate with 0.1 n caustic soda solution until the color changes to red (a ml). If the liquid contains a lot of iron, it is practically impossible to detect the change in color, so that the following steps must be taken.

First carry out a preliminary determination to ascertain how much caustic soda solution is roughly required for the color change.

For accurate titration add in one portion enough caustic soda solution to bring the liquid very close to the expected end point and then titrate further with small quantities of caustic soda solution. Shake the Erlenmeyer flask well after each addition of caustic soda solution and add a few drops of phenolphthalein.

Keep the flask tilted, allow the precipitate to settle and observe whether the clear layer that separates out is pink or red.

Calculate the acid content of the pickling bath in g/l from:

$$\begin{aligned} \left(\frac{\alpha}{V} \times b - 2 \times \frac{c}{56} \right) \times 36.5 \text{ g/l} & \quad \text{if the acid is hydrochloric acid.} \\ \left(\frac{\alpha}{V} \times b - 2 \times \frac{c}{56} \right) \times 49 \text{ g/l} & \quad \text{if the acid is sulphuric acid.} \\ \left(\frac{\alpha}{V} \times b - 2 \times \frac{c}{56} \right) \times 98 \text{ g/l} & \quad \text{if the acid is phosphoric acid.} \end{aligned}$$

in which b represents the titre of the caustic soda solution and c the iron content of the bath fluid in g/l (test 1.1 of this attachment).

(to be continued)

ATTACHMENT A (continued)

b) Acid content of rinsing baths

Dilute a suitable quantity, V ml, bath fluid with distilled water to about 100 ml. Add a few drops of methyl orange and titrate with 0.1 n caustic soda solution until the color changes from red to yellow (a ml).

Calculate the acid content of the rinsing bath in g/l from:

$$\frac{\alpha}{V} \times b \times 36.5 \text{ g/l} \quad \text{if the acid is hydrochloric acid.}$$

$$\frac{\alpha}{V} \times b \times 49 \text{ g/l} \quad \text{if the acid is sulphuric acid.}$$

$$\frac{\alpha}{V} \times b \times 98 \text{ g/l} \quad \text{if the acid is phosphoric acid.}$$

in which b represents the titre of the caustic soda solution.

1.3 Reduction in Weight and Attack of the Surface During the Pickling of Steel (Draft specification of the "Vereniging Metaalbeits") (Metal Pickling Association).

The test is intended as a check on the proper functioning of the pickling bath, i.e. that the attack of the steel is minimal and that it occurs uniformly.

The rate of corrosion of the steel is ascertained by pickling pretreated steel plates for a certain length of time and determining the resultant reduction in weight; the plates are then examined visually in order to establish whether uniform attack has taken place.

Apparatus

Plates, bright steel Qmc 37, with a hole for suspension, dimensions 50 mm × 50 mm × 5 mm.

Balance, weighing capacity 200 g, sensitivity 2 mg.

Magnifying glass, magnification at least 5 × and at most 8 ×.

Two glass beakers, each with a capacity of 1000 ml.

One pair of crucible tongs, for inserting and withdrawing the test plates.

Sulphuric acid, chemically pure, diluted (50 g H₂SO₄/l).

Alcohol, denaturated (methylated spirits).

Procedure

a) Pretreatment of the test plates

At least three test plates are required for the test; however, it is recommended to pretreat a few extra plates.

a.1) Remove grease from the plates with an organic de-greasing agent, e.g., tri- or perchloroethylene.

a.2) Pickle the plates for 2 minutes in diluted sulphuric acid at a temperature of 60°C.

If a fresh quantity of sulphuric acid is not used for each test, the fluid must be renewed as soon as the iron content exceeds 5 g/l (Test 2.1).

a.3) Rinse the plates in clean, cold and fresh running water (e.g. tap water).

a.4) Dip the plates in alcohol.

a.5) Dry the plates in air.

a.6) Weigh each plate to the nearest 2 mg.

a.7) Inspect the surface of the plates for corrosion with the aid of the magnifying glass. Only non-corroded plates may be used for carrying out the test (b).

(to be continued)

ATTACHMENT A (continued)**b) Execution of the test**

Use at least three pretreated plates which the check in accordance with a(7) has shown to be uncorroded.

b.1) By means of a nylon thread or steel wire with a suitable plastic covering suspend the plates in the pickling bath, together with a charge of the material to be pickled.

Suspend the plates at about half the depth of the pickling fluid in such a way that they are evenly distributed over the length of the charge. Ensure that the pickling fluid has free access everywhere.

Pickle the plates for the same length of time as the charge, but no longer than 30 minutes.

b.2) Then rinse the plates one by one in clean, cold and fresh running water (e.g. tap water).

b.3) Dip the plates in alcohol.

b.4) Dry the plates in air.

b.5) Weigh each plate to the nearest 2 mg.

b.6) Calculate from the reduction in weight and the area the loss of weight in mg / dm² for each plate as follows:

$$\text{loss of weight in mg / dm}^2 = \frac{\text{loss of weight in mg}}{0.6}$$

b.7) Inspect the surface of the plates for irregular corrosion with the aid of the magnifying glass.

In practice it will not always be possible to conduct the test on the spot in the manner described above, for instance, because of the lack of the necessary apparatus. The Engineer considers that in those cases the test can be conducted at a laboratory elsewhere.

For this purpose one litre samples must be taken at those places in the pickling bath where the plates would have been suspended if the test had been carried out normally: the temperature at those places should be measured at the same time. The samples must be clearly labelled with the data on the temperature and the pickling time of the charge. The samples should then be dispatched to the laboratory for testing.

Procedure for the laboratory test

Pour the samples of pickling fluid into wide 1500 ml glass beakers. Heat the contents of these beakers to the temperature indicated on the samples concerned and keep the fluid at this temperature for the whole test. In each beaker suspend a plate, pretreated and weighed as described above [see a(1). to a(7). inclusive] in such a way that it hangs roughly in the middle of the fluid. Cover the beaker well to prevent evaporation losses. Pickle each plate for the pickling time of the charge marked on the samples, but not longer than 30 minutes. Then treat the plates further as described above under b(2) to b(7) inclusive.