

Björn Schouenborg

Regulations for P-marking of Liming Products for Acidified Lakes and Watercourses

Abstract

Certification of liming products

Manufacturers may, after obtaining a permit from the Swedish National Testing and Research Institute (SP), use the P-symbol to mark their products. In order to obtain a marking permit, it must be verified that the product complies with the requirements contained in a standard recognized by SP, or an equivalent normative document. An agreement must also be entered into between SP and the manufacturer concerning continuous quality inspection of the products.

This report describes the requirements for P-marking of liming products for acidified lakes and watercourses.

The emphasis is on functional and safety requirements including neutralizing effect, grain size distribution, moisture content and heavy metal content.

The continuous in-process quality inspection is performed by the manufacturer (self inspection). The manufacturer's inspection is supervised through inspections by SP. These inspections are carried out in the plants and at locations for import. The main aim of the quality inspection is to ensure that the requirements placed on the liming products are fulfilled.

Key words: certification, liming products, P-marking, requirements, quality inspection



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Foreword

In collaboration with representatives of the Swedish Lime Association (SKF) and the Swedish Environmental Protection Agency (SNV), SP has developed a certification system referred to as "P-marking of liming products for acidified lakes and watercourses".

SP intends to adapt its inspection procedures to EEC policies for testing and certification wherever possible. The present inspection program has been drawn up in a flexible way, to facilitate the transition to future EEC norms and modules. The product specifications contained here have been established on the basis of the general guidelines for liming of lakes and watercourses ("Allmänna Råd 88:3") issued by SNV.

Primary responsibility for inspection rests with the manufacturer. This is also in agreement with the guidelines for EEC principles. The emphasis of the inspection procedure is on functional specifications and environmental safety. The main aim is to ensure that product specifications have been fulfilled. The requirements for the quality system have been drawn up pursuant to the applicable sections of EN 29 002.

The regulations were prepared by a project group including the following representatives of SP: Björn Schouenborg, project manager, Matz Sandström, Karl-Axel Olsson and the following representatives of SKF: Måns Öhman and Sten Jacobsson.

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1 Introduction

“ Regulations for P-marking of liming products for acidified lakes and water-courses” is the SP certification system for liming products whose primary use is to counteract acidification in lakes and watercourses.

The system certifies both the process by which the liming product is manufactured, as well as the product itself.

In order to be granted a P-marking permit, a manufacturer must be able to document the compliance of the product with the prevailing norm, see annex 1. The continuous quality of the product manufactured must also be ensured through the manufacturer's carrying out regular self inspections. SP supervises this self inspection through inspection visits. If the inspections indicates that the products fail to comply with the requirements, they may not be P-marked until the fault has been identified and remedied. The organization of and procedures for the in-process inspection are stipulated in the certification rules, and are confirmed in an agreement between the manufacturer and SP.

2 Conditions for issuing a P-marking permit

These certification rules regulate the manufacture of liming products for acidified water. "Manufacturing inspection" refers both to the self inspection procedures carried out by the manufacturer under expert guidance and supervision (items 3 and 4.1) as well as supervisory inspection visits from SP (item 4.2).

In order to be granted certification:

- a) The company shall have issued a manufacturer's description, see annex 4.
- b) The company shall produce documentation of its mapping and its quarrying plans.
- c) SP shall have inspected the facilities, equipment and manufacturing process.
- d) SP shall have completed a preliminary typetest of the product in order to determine whether the certification regulations have been fulfilled.
- e) The company and SP shall have drawn up an in-process inspection agreement.

The documents stipulated in items a-d above shall be submitted to SP with an application for a P-marking permit for products from a given plant.

SP shall maintain a list of approved companies which have been granted P-marking permits. SP shall confirm that the permit has been granted by issuing a separate certificate for each plant. When the permit has been granted, the plant may mark bills of sale and packages applying to the certified liming products with the SP P-symbol (see annex 2).

3 Quality assurance

The manufacturer shall have a quality system that ensures that the manufacture of a certified (P-marked) liming product continuously complies with the applicable requirements. The system shall be drawn up pursuant to the applicable sections of EN 29 002. The system shall be documented, and one employee at the company shall be designated as quality assurance manager. This individual shall have the authority and the obligation to see to it that the stipulated quality of the liming product is fulfilled and maintained.

When SP makes an inspection visit, the company personnel responsible for quality shall be available to provide information and the necessary documentation. The manufacturer shall give the SP representatives free access to the manufacturing premises and storage facilities. Purchasing orders, internal requisitions, bills of sale, and manufacturing orders where such are used, shall be saved and made available to SP.

The self inspection shall be carried out by employees with the necessary competence and training. There shall be documentation of the job descriptions and authorities of these employees.

The manufacturer shall carry out comprehensive, planned in-house quality reviews, in order to verify that quality inspections conform to plans, and to determine that quality work is being done efficiently. Records of these reviews shall be kept and made known to the employees concerned.

4 Inspection regulations

Liming products ready for delivery shall be inspected both through self inspections pursuant to item 4.1 and through supervisory inspections made by SP as described in item 4.2.

4.1 Self inspection

Manufacture and regular self inspections shall be carried out under the leadership and supervision of a person with the necessary competence, see item 3.

Regular inspections shall be carried out at each plant at least to the extent described below. Inspection records shall be kept. Each entry shall include the results, the measure taken, and the date, and the log entry shall be signed by the inspector. Analysis results shall be submitted to SP on a monthly basis. Once every three months a metal content test shall be carried out as part of a composite sample. Increments for this composite sample shall be taken every second week throughout.

When importing, one composite sample per shipment and liming product shall be taken as described in item 5.3. SP may also perform random sampling tests of imports.

The purpose of inspection is to verify that the intended quality has been obtained, and includes the following testing, carried out at the laboratory of the plant/importer or any other laboratory contracted by the manufacturer for his self inspection under regulated conditions.

Type of liming product
Metal content
Sieve analysis
Acid-neutralizing effect
Moisture content

The individual at the company responsible for inspection shall notify SP immediately if the test results do not conform to the P-marking specifications.

Sampling shall be carried out as described in items 5.2 and 5.3.

4.2 Supervisory inspection

Supervisory inspection shall be carried out by SP and consists of three parts:

inspection
random sampling
interlaboratory test comparison

4.2.1 Inspection

Inspections are carried out in the form of visits to each plant twice a year without prior notification, the time being at the discretion of SP. No more than nine months shall pass between any two inspection visits of the same plant.

Inspection consists of:

- survey of the manufacturer's quality system*
- determination that sufficient self inspection has been carried out
- determination that sampling results satisfy the specifications for P-marking
- determination that SP has been notified if sampling results have not fulfilled the specifications for P-marking
- determination that the operating instructions were followed when the sampling results deviated from the set values or specifications
- determination that instruments and testing equipment used by the company for in-process inspection are calibrated**
- determination that the laboratory staff has the requisite knowledge of sampling and methods.**

No later than six weeks after the inspection, SP shall submit an inspection report to the quality assurance manager of the company. The inspection report shall contain a description of all inspection processes carried out and the results, as well as information regarding all observations and notations made during the inspection visit. If major unremedied defects have been found, a preliminary report shall be submitted to the manufacturer and the plant directly after the inspection visit.

4.2.2 Random sampling inspection

SP is responsible for taking two random samples per year, per brand and per type of lime. These random samples are normally taken on the occasions of SP's inspection visits. See also item 5.3.

The purpose of the random sampling is to determine that each liming product satisfies the requirements for P-marking.

* may be reduced if the quality system has been certified by a certification body, accredited as described in EN 45 012.

** may be reduced if the laboratory has been accredited for this product as described in EN 45 001.

The random samples shall be taken from liming products ready for delivery, preferably at one of the manufacturer's delivery points and, if possible, in conjunction with a lot being loaded for delivery. Two of the increments shall be submitted to SP. One of these is a reserve sample. One sample is submitted to the laboratory contracted by the manufacturer for regular self inspections.

Tests are taken with regard to:

Type of liming product
Metal content
Sieve analysis
Acid-neutralizing effect
Moisture content

No later than six weeks after the inspection and sampling, the manufacturer's quality assurance manager of the company shall notify SP of the test results from the random samples taken by SP.

SP evaluates the results and compiles an annual report on the random sampling inspection containing comparisons between the product statistics distributed by the manufacturer and the statistics drawn up by SP on the basis of their random sampling.

4.2.3 Interlaboratory test comparison

An interlaboratory test comparison is carried out once a year in conjunction with an inspection, and is made on a liming product ready for delivery. The participants in the test comparison are SP and the laboratories used by the manufacturer for self inspections.

The test comparison makes it possible to assess the magnitude of deviations in the test results between the various laboratories performing analyses, using tests made on the same liming product.

The test comparison is made on two lime samples, on each occasion, selected by SP. SP sends one increment of each type of lime (laboratory sample) to each participating laboratory, and retains three reserve samples. The test is made on double analysis samples.

Sampling is carried out as described in item 5.3.

The double samples are tested with regard to:

Type of liming product
Metal content
Sieve analysis
Acid-neutralizing effect
Moisture content

No later than six weeks after the inspection and sampling, the manufacturer's quality assurance manager of the company shall notify SP of the test results from the reference sample taken by SP.

SP compiles and evaluates the test results and reports to the participating laboratories.

If SWEDAC-accredited laboratories are used for self inspection, SP undertakes to coordinate the test comparisons described above with the test comparisons required by the SWEDAC accreditation regulations wherever possible.

4.2.4 Retesting

If unsatisfactory measurements are obtained from the supervisory inspection, retesting shall be carried out as soon as possible. SP may, under exceptional circumstances and for a limited time, permit products to continue being marked pending the results of retesting.

If unsatisfactory results are also obtained from the retesting, measures shall be implemented as described under 4.2.5.

4.2.5 Measures in case of defects or nonconformities

The manufacturer shall implement measures in consultation with SP to remedy defects and nonconformities found. Consultation and measures shall be recorded.

When defects and nonconformities are found, the manufacturer is under obligation to carry out testing and inspection beyond that which is ordinarily specified, as well as to perform the studies deemed necessary on the basis of the defects and nonconformities found.

When major defects or nonconformities are found or when repeated comments are made without resulting in a correction, withdrawal of the P-marking permit shall be considered (see item 6.3).

4.2.6 Reporting

SP shall compile an annual report of inspection activities. A copy shall be submitted to the manufacturer.

5 Sampling

5.1 Background

Pending the completion of the standardized sampling procedure described in CEN/TC 154, TG5 (sampling and precision) for aggregates, we refer to the recommendations given in the "General Guidelines" issued by SNV (88:3).

Samples are taken by a sampler from SP or appointed by the manufacturer (at self inspection), who issues the sampling certificate stating: the name of the commissioning party, place of work, sampling time, location and method, sampler's labelling of the sample, type of liming product, supplier, means of supply and the size of the batch or lot.

5.2 Sampling for self inspection

The frequency of sampling in regular production shall be one sample per 300 tonnes, but not less than one sample per production week. For imports, samples shall be taken as described below (item 5.3).

Testing frequency may be reduced in cases of continuous uninterrupted manufacturing if the test results obtained prove to satisfy the current requirements well. Such reductions are subject to the approval of SP.

5.3 Sampling for random sample inspections, inter-laboratory test comparisons and imports

It is the responsibility of the importer to ensure that imported liming products satisfy the specifications for P-marking. This may be done by SP or a controller appointed by the importer taking samples on every occasion of import. Alternately, the foreign producer may be affiliated with the current SP certification system.

A number, see below, of evenly-distributed increments of no less than 5 kg are taken for sampling. The increments, carefully protected against moisture, are mixed and reduced by repeated reduction (if necessary) so as to obtain a composite sample of no less than 15 kg. The composite sample is taken from liming products ready for delivery, preferably at one of the manufacturer's points of loading and, if possible, in conjunction with delivery.

The table below describes the required number of increments:

Size of lot in tonnes.....	<20	20-60	60-150
Number of increments	3	4	5

One increment per 30 tonnes shall be taken from lots or batches weighing more than 150 tonnes. The size and number of increments may also be set differently if the parties concerned so agree. This applies primarily to lots of over 1000 tonnes.

For random sample inspection, the composite sample is reduced through repeated reduction so as to obtain three laboratory samples of 5 kg each.

For interlaboratory test comparison, a 5 kg laboratory sample of each type of liming product is taken for each participating laboratory, plus three reserve samples for SP. Two types of liming products shall be analysed.

For imports, the composite sample is reduced through repeated reduction so as to obtain 2 laboratory samples of 5 kg each.

6 Guidelines for withdrawal of the right to use the P-symbol

6.1 Background

The general stipulations for issuing permits for P-marking are found in "General conditions for the right to use the SP certification marking, the P-symbol" (SP-INFO 1988:38).

These guidelines regulate in detail the general conditions for the routines SP intends to follow when issuing and withdrawing permits for P-marking of liming products.

A decision to withdraw the right to use the P-symbol is made by SP's manager of certification pursuant to the stipulations given in SP REPORT 1991:35. Decisions may be appealed to the SP certification committee as described in the general regulations for SP's certification procedures.

6.2 Changes in production conditions

The manufacturer is obligated to inform SP of changes in material or quarries. It is then up to SP to determine whether or to what extent a renewed typetest is required.

6.3 Withdrawal of the right to use the P-symbol

When serious problems are found, SP may withdraw the marking permit with immediate effect. Normally, the manufacturer is given the opportunity to rectify the faults found. In such cases, SP issues a written complaint to the manufacturer, stating a date by which the fault must be remedied.

When this period of time expires, SP makes an extra inspection. If serious shortcomings remain, the marking permit is withdrawn, formally on the same day as the inspection. If the marking permit is withdrawn, the manufacturer undertakes, upon penalty of a fine of SEK 200,000, to immediately cease both referring to the SP P-marking permit, and marking with the SP certification symbol.

If the marking permit is withdrawn, the manufacturer is obliged to comply with his obligations regarding P-marking pursuant to the agreement signed between purchaser and manufacturer through the date when the permit was withdrawn.

The right to use P-marking is also forfeited by a company which has not produced any P-marked liming products for one year.

6.4 Rejects

Material (lots) which are found not to conform to the quality requirements during in-process inspection pursuant to item 4.1 may not be used as P-marked products.

Lots which are found defective during self inspection pursuant to item 4.1 shall be reworked where possible, or rejected. The SP certification mark shall be destroyed on rejected lots.

6.5 Re-affiliation with the marking system

The same rules apply to re-affiliation with the marking system as when the permit was initially issued. If less than one year has passed since the marking permit was withdrawn, no new typetest is required, unless there have been changes in the regulations for P-marking or in the manufacturing conditions.

7 Fees charged

SP charges fees for the use of the P-symbol in accordance with the current rates.

The expenses associated with supervisory inspections are to be paid by the manufacturer/importer. Invoices shall be sent quarterly, in accordance with the ordinary SP rates. An annual fee is charged for affiliation with the P-marking system.

8 Revision of regulations

SP reserves the right to revise these regulations. One example of a reason for revision might be that the standards, stipulations, "General Guidelines", etc. referred to in this documents have been subject to revision.

Other reasons for revision might be new experience, or the development of new materials or products.

A copy of the revision of these certification regulations is appended to the agreement as a memorandum, and is confirmed in SPKB (the SP inspection regulations).

For extension of a marking permit granted under older regulations, the manufacturer must undertake to conform to the revised regulations.

However, the manufacturer shall be given reasonable time to make a transition to the revised requirements.

Annex 1 (page 1)

Testing methods and requirements for certification of liming products for acidified water by P-marking

Metal content (HNO₃ - decomposition)

Testing method: Swedish Standard (SS) 02 81 50 Metal content of water, sludge and sediment determined by atomic absorption spectroscopy, atomization in flame - General principles and guidelines.

Requirements: According to the recommendations in "General Advice 88:3" by SNV (see below).

Al	< 20 g/kg	Cu	< 20 mg/kg
Zn	< 50 mg/kg	V	< 20 mg/kg
Cr	< 50 mg/kg	Pb	< 20 mg/kg
Ni	< 20 mg/kg	Cd	< 0.5 mg/kg
Co	< 20 mg/kg	Hg	< 0.05 mg/kg

Acid neutralizing effect (Indicator titration method)

Testing method: ASTM C 602-90, reported as % CaCO_{3eq}

Requirements: A minimum content of 80 % CaCO_{3eq} is required for all liming products.

Moisture content

Testing method: ASTM C 25-91, Chemical analysis of limestone, quick lime and hydrated lime.

Requirements: There are no limit values. The manufacturer or importer shall declare the maximum moisture content of the product.

Sieve analysis (wet screening)

Testing method: SS 02 71 23 Geotechnical tests - Particle size distribution - Sieving.

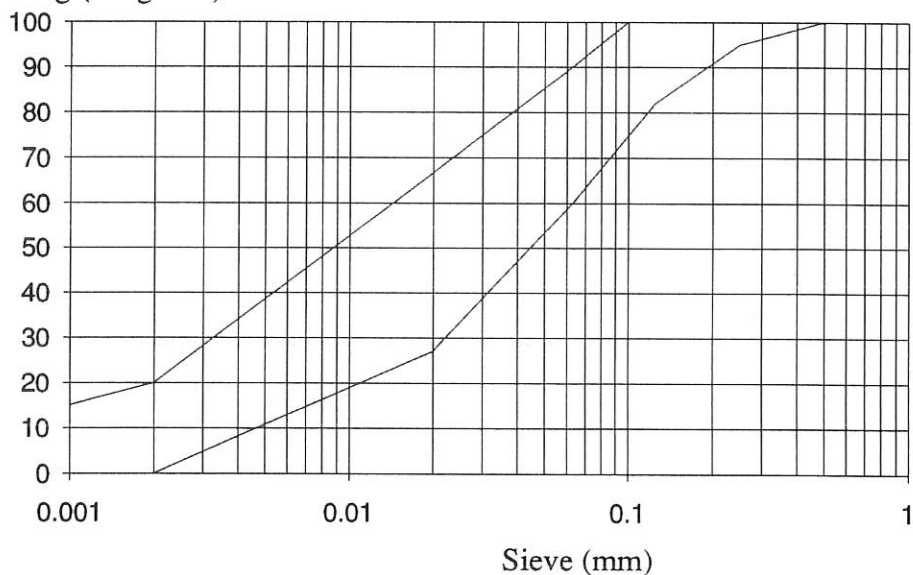
Analysis of size-fractions smaller than 63 μm is included in the supervisory inspection and shall be performed using a sedigraph or a laser granulometer.

Requirements: For all powdered limestone products used in lakes and watercourses, the limit values are shown in the diagram and the table below. These limits are partly based on the recommendations in "General Advice 88:3" by SNV. Permitted maximum deviation from the sieve analysis certificate stated by the manufacturer is 10 %.

For dolomitic liming products: Dolomitic liming products used in lakes and watercourses: 90 % < 63 μm . When a doser is used: 90 % < 30 μm is recommended according to "General Advice 88:3" by SNV.

Limit values for size-fractions of powdered limestone products

Passing (weight %)



Limit coordinates for the upper curve:

Limit coordinates for the lower curve:

Sieve (mm)	Passing (weight %)	Sieve (mm)	Passing (weight %)
0.001	15	0.002	0
0.002	20	0.02	27
0.063	90	0.063	60
0.1	100	0.125	82
		0.25	95
		0.5	100

Marking

All packaging material and bills of sale shall be marked so that the products are identifiable.

Bulk deliveries of liming products inspected at manufacture shall include a bill of sale, issued upon delivery, which contains the following information:

Manufacturer, place of manufacture, place of delivery
Type of liming product (powdered limestone, powdered dolomite or olivine)
CaCO_{3eq} content*
Moisture content*
Size fraction (sieve analysis)*
Packing date or other marking which enables the lot to be identified
P-symbol
Type of delivery and lot size

Only packages containing liming products approved through in-process inspection and satisfying the requirements for P-marking may be accompanied by a bill of sale carrying the P-symbol.

When the product is delivered in bags, every bill of sale per lot/package shall be marked as below:

Manufacturer, place of manufacture, place of delivery
Type of liming product (powdered limestone, powdered dolomite or olivine)
CaCO_{3eq} content*
Moisture content*
Size fraction (sieve analysis)*
Packing date or other marking which enables the lot to be identified
P-symbol
Weight per package

Only packages containing liming products approved through in-process inspection and satisfying the requirements for P-marking may be labelled with the P-symbol.

*) Alternately, this information may be given in the current product sheet or a certificate of analysis (no older than six months) supplied with the offer.



Agreement

as to the right to use the SP
certification mark

Annex 3 (page 1)

*EXAMPLE*

Liming products for liming of acidified lakes and watercourses.

The following agreement has been entered into between SP and

..... (manufacturer)

The "general conditions for the right to use the SP P-symbol" are applicable to this agreement. This agreement gives the party the right to use the P-symbol for products on the condition that a marking permit has been issued by SP. The marking permit shall state the products and variants for which the P-symbol may be used.

1 Product

Liming products as described in the issued marking permit (annex 2 to this agreement)

2 Purpose

The purpose of the P-marking is to ensure the quality of the liming products. The requirements are stipulated in SPKB (the SP inspection regulations) with reference to SP REPORT 1991:35 (Regulations for P-marking of liming products for acidified lakes and watercourses) which corresponds to applicable portions of the SNV General Guidelines 88:3.

3 Manufacturer's commitments

The manufacturer undertakes to comply with the regulations and instructions given in SP REPORT 1991:35 (Regulations for P-marking of liming products for acidified lakes and watercourses) and to perform self inspection in accordance with an established inspection plan, approved by SP.

4 SP's commitments

SP undertakes to perform the supervisory inspections as described in the instructions given in SP REPORT 1991:35 (Regulations for P-marking of liming products for acidified lakes and watercourses).

5 Special conditions for the validity of the marking permit

If the marking permit is withdrawn, the manufacturer undertakes, upon penalty of a fine of SEK 200,000, to immediately cease both referring to the SP P-marking permit, and marking with the SP certification symbol.

6 Expenses

All expenses for manufacturing inspection as described in this agreement and all testing required pursuant to it shall be borne by the manufacturer.

7 Period of validity of this agreement

This agreement shall be effective as of and to remain in force until further notice. Notice shall be given in writing no less than three months prior to the date when the agreement is to be annulled.

This agreement has been produced and signed in duplicate, and one copy has been deposited with SP and the other with the manufacturer.

The agreement shall be dated on the date of its signature by the manufacturer/importer.

Contact person for the manufacturer/importer

Contact person for SP

Each contact person has the right to sign a binding agreement for his principal. Additions or other changes of the agreement shall be made in writing to be legal.

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(manufacturer/importer)

Annexes

1 Place of manufacture and contact person

2 Marking permit

Manufacturing place and contact person

Manufacturer/importer:

Address and telephone:

Place of manufacture:

Storage facilities:

Management and quality assurance manager

Name:

Telephone:

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(manufacturer/importer)

Marking permit

SP hereby grants

the right to mark the following products with the certification symbol below

Products

Design of the Symbol

The liming product ready for delivery shall be marked with SP's certification symbol:



The mark shall be clearly visible on each inspected delivery (bag and/or bill of sale) and the marking shall be carried out according to the directions given in annex 2.

The products satisfy the requirements given in annex 1. An agreement has been entered into with the aim of ensuring that the products continuously comply with the requirements. The agreement stipulates that self inspections shall be carried out. Moreover, supervisory inspections shall be made by SP.

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Guidelines for establishment of a quality system

1 Manufacturer

- 1.1 State the manufacturer's name and address and the current manufacturing sites

2 Expertise and production management

- 2.1 State the name of the special expert responsible for manufacturing and inspection (SP's contact person).
- 2.2 Give the organizational chart and the distribution of responsibility at the company.
- 2.3 Documentation of the training and qualifications of the special expert and his or her experience in the field.

3 Facilities

- 3.1 Document the handling facilities and space.
- 3.2 Document the storage space.
- 3.3 Describe the packaging methods, if any.
- 3.4 Document the equipment for self inspection/testing.

4 Description of manufacturing

Give a brief description of the procedure used for manufacturing the liming product. In addition to describing the process, the description shall contain information as to the number of people employed in manufacturing, their functions and job descriptions, and the average daily production volume,.

5 In-house instructions

The in-house instructions shall contain:

- Routines for operational testing
- Regulations for calibration
- Regulations for measures to be taken when the requirements are not complied with.

Date

Manufacturer's name

.....
(signature of individual authorized to sign for company)

