Testing regulations and procedures for environmental auditing of recycled aggregates

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1. Introduction

In the Netherlands, quality certification of the environmental aspects of recycled aggregates is based on the Building Materials Decree [1], which contain specifications for leaching and composition of stony materials that are applied and used outdoors. In this annex an overview of some key procedures is provided. For the full text on the Building Materials Decree the reader is referred to references [1; 2]; the current quality control document may be found as reference [3]. An English summary on the Building Materials Decree is available through the Dutch Ministry of Housing, Spatial Planning and the Environment [4]. In this article the procedures are presented to be used in quality certification of recycled aggregates in order to assure that these materials meet the legislation.

2. Testing regulations

In order to undertake an audit the bulk production of a material is subdivided into (fictitious) units that are considered as 'lots' (or samples). The actual size of these lots depends on a prior determination of their properties. As a rule the testing consists of determining one or more properties on one sub-sample per lot.

The frequency at which these audits should be carried out depends on the level and consistency of the quality of the product. When judging the environmental properties, a distinction is being made between a random control and a lot-oriented control.
In the case of the random control, lots are tested at random on composition and emission; successive analytical results are used for the determination of the testing frequency. In case of lot-oriented testing every single lot will have to be inspected individually.
When judging the remaining properties a lot-testing procedure is applied with a fixed testing frequency.

When auditing, the bulk production of a material is divided into lots with a fixed size. The producer is required to specify – in advance – [the terms, under which the producer considers this case as a 'lot',] to the certifying institution. In case of determination of the composition and emission it is required that for each product the size may amount to 1/10 of the annual production at most. For the determination of the remaining properties the size of the 'lots' depends on the testing-frequency.

Individual sub-samples should be of an approximately similar size (with a margin of + or - 25%) and meet the requirements for the minimal size, as presented in table 1.

The minimum number of sub-samples per sample and the number of samples - per lot - that are required to be tested are presented in table 2.

Table 1. Minimal size of sub-samples

Product	Property	Minimal size of sub-samples
Bound recycled aggregates	Composition, emission and permanent dimensional stability Other properties	1 sample (7,0 kg) 3,9 kg
Hydraulically bound recycled mixed aggregates (judged as if composed out of one piece)	All properties	3,9 kg
All recycled aggregates 0/4 and crusher sand	Composition, emission Other properties	180 g 2,0 kg
Hydraulically bound recycled mixed aggregates (judged as if **not** composed out of one piece)	All properties	3,9 kg

Table 2. Minimum number of sub-samples per sample/number of samples per lot

Product	Property	Minimum number of sub-samples	Number of samples per lot
Bound recycled aggregates	Composition, emission and permanent dimensional stability Other properties	4 16	1 1
Hydraulically bound recycled mixed aggregates (judged as if composed out of one piece)	Composition, emission and permanent dimensional stability Other properties	1 sample 16	1 1
All recycled aggregates 0/4 and crusher sand	Composition and emission Other properties	32 16	1 1
Hydraulically bound recycled mixed aggregates (judged as if **not** composed out of one piece)	Composition and emission Other properties	32 16	1 1

If in testing the emission or composition, within either the framework of the admission-examination or within the framework of the continuation of the certificate, it is established that the product does not meet the requirements for one of the components, the product is required to be tested, where that particular component is concerned, in the production audit under the lot-testing protocol.
In this case, contrary to the above-mentioned, a minimum of 2 samples per lot is required to be tested, where that particular component is concerned.

3. Components to be defined (emission and composition)

All the organic and inorganic components of building materials - other than those for which specifications for composition, respectively immission, are drawn up in the decree on building materials - should in principle be defined separately.

3.1 *Testing on variables - principles*

The frequency, with which lots are to be tested on emission and composition, is determined by means of the quantity k.

$$k = \frac{\text{In}\,(T) - \overline{y}}{S_y}$$

in which

T = testing requirements

$\overline{y}$ = progressive average of lognormal transformed observations

S_y = progressive standard-deviation of log-normal transformed observations

The quantity k is to be determined for each component tested.

The above formula is based on the assumption that observations are divided log-normally. If in reality the observations are divided normally, it may be more favorable to express this when calculating k.

A component within a certain product is considered as being non-critical, if with a reliability of 90%, at least 99% of the lots of that product, have an average that is less than the testing-value for the product concerned. In this case one should start with 'testing on variables'.
Non-critical components are to be determined at least once a year. For this, per annum and per product, of at least one lot that is representative, all organic components (composition) and inorganic components (emission) are to be determined. In such cases it is not allowed to use any abbreviated testing-procedures.

When determining the frequency of testing for the critical components under a random control-regime, one can start from testing on variables, or testing on attributes. It is allowed to use both methods. An exception is the determination of the polyaromatic hydrocarbons (PAH)-content in bituminous aggregates for the application as a semi-manufactured product for the production of bituminous aggregates for asphalt mixes. In this case the standard procedure is 'testing on variables'. Details will be outlined in the paragraphs below.

3.2. *Testing on variables*

The progressive average and the progressive standard deviation are determined on the basis of the most recent five observations. An observation in this case is the average emission or composition per lot. Valid for the testing frequency is the classification outlined below in table 3:

Table 3. Testing frequencies classification (testing on variables)	
Value of k for 5 observations	minimum frequency
$k > 2{,}74$	1 x per 20.000 tonnes, yet at least 5 x per annum
$1{,}46 < k \leq 2{,}74$	1 x per 10.000 tonnes, yet at least $5+0.25\ x \frac{\text{annual productions (tonnes)}}{\text{lot - size (tonnes)}}$ *per annum*
$0{,}69 < k \leq 1{,}46$	1 x per 5.000 tonnes, yet at least $5+0.50\ x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ *per annum*
$k \leq 0{,}69$	In conformity with the lot testing protocol

3.3. Attribute testing

On the basis of the number of transgressions in the progressive series of the most recent observations the following classification per quality-specified product produced is used (table 4):

Table 4. Testing frequencies classification (testing on attributes)		
Number of permitted results from the total number of observations, which are allowed to exceed the requirement	Total number in the series of most recent observations	Frequency
≤1	38	1 x per 20.000 tonne, yet at least 5 x per annum
≤1	12	1 x per 10.000 tonne, yet at least $5 + 0.25\ x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ *per annum*
≤2	9	1 x per 5.000 tonne, yet at least $5 + 0.50\ x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ *per annum*
≤2	9	In conformity with the lot testing protocol

Note: an observation in this case is the average emission or composition per lot.

3.5 Determining the PAH-content in bituminous aggregate as a semi-manufactured product

For the determination of the PAH-content in bituminous aggregates for the application as a semi-manufactured product for the production of bituminous aggregates for asphalt mixes the testing frequency is as follows (table 5).

Table 5. Testing frequencies classification (testing on PAH-content)	
Value for k in 5 observations	Minimum frequency
$k > 4{,}67$	1 x per 10.000 tonne, yet at least 10 x per annum
$2{,}74 < k \leq 4{,}67$	1 x per 2.500 tonne, yet at least $10 + 0.50\,x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ per annum
$k \leq 2{,}74$	In conformity with the lot testing protocol

The way in which the PAK(10)-content of the fine fractional part of recycled aggregate 0/40 is determined proceeds analogous, albeit that the initial testing-frequency is determined on the basis of the five most recent observations and that the criteria for the various quality classifications are different.

3.5.1. *Testing on variables*

The progressive average and the progressive standard deviation are determined on the basis of the 5 most recent observations. For this the following classification is valid for the testing-frequency (table 6):

Table 6. Testing frequencies classification (testing on variables; PAH-content)	
Value for k in 5	Minimum frequency
$k > 0{,}69$	1x per 20,000 tons, yet at least 5x per annum
$0{,}05 < k \leq 0{,}69$	1x per 10,000 tons, yet at least $5 + 0.25\,x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ per annum
$-0{,}68 < k \leq 0{,}05$	1x 5,000 tons, yet at least $5 + 0.50\,x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ per annum
$k \leq -0{,}68$	Advance sifting compulsory; check of the crusher-sieve sand quality in conformity with the lot-testing control

3.5.2. *Testing on attributes*

On the basis of the number of transgressions in the progressive series of the most recent observations the following classification per quality produced is used (table 7):

Table 7. Testing frequencies classification (testing on attributes; PAH-content)		
Number of permitted results from the tota number of observations, which are allowed to exceed the requirement	Total number in the series of most recentl observations	Minimum frequency
≤ 1	7	1x per 20,000 tons, yet at least 5x per annum
≤ 2	6	1x per 10,000 tons, yet at least $5 + 0.25\,x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ per annum
≤ 3	5	1x 5,000 tons, yet at least $5 + 0.50\,x \frac{\text{annual production (tonnes)}}{\text{lot - size (tonnes)}}$ per annum
≤ 3	5	advance sifting compulsory; check of the crusher-sieve sand quality in conformity with the lot-testing control

4. Control

4.1. *General*

For every product there is a control-check for each component that is examined to see if the following criteria are met:

$\bar{y} + A \times S_y \leq \ln(T)$.

in which:

T = testing-value

$\bar{y}$ = progressive average of lognormal transformed observations

$y_i = \ln(x_i)$ with x_i = average per annum per producer i

S_y = progressive standard-deviation of log-normal transformed observations

A = testing-factor, dependent on the number of observations

Using this criterion, the chance that the real values of the product investigated will exceed the testing value can be neglected.

4.2 Acceptance of lots in the lot-testing protocol

In the lot-testing control lots are actually approved or disapproved. Lots are approved if:

$\bar{x} = \frac{T}{ZF}$, to be sure that the real values are in agreement with the requirements

in which:

T = testing value

$\bar{x}$ = average of observations per lot

ZF = certainty-factor. This certainty-factor corresponds to the overview of lot-sizes in table 8.

Table 8. Classification of lot-size and category with corresponding certainty-factors	
Lots of ≤ 2.000 tonnes	ZF = 1.00
Lots of > 2.000 tonnes	
● Emission and composition of recycled aggregates **not** composed out of one piece	
● 1 Sample per lot	ZF = 1.18
● 2 Samples per lot	ZF = 1.09
● 3 Samples per lot	ZF = 1.07
● Emission and composition of bound recycled aggregates	
● 1 Sample per lot	ZF = 1.26
● 2 Samples per lot	ZF = 1.14
● 3 Samples per lot	ZF = 1.10
● Hydraulically bound recycled aggregates	
● Composition and emission (judged as if **not** composed out of one piece)	
● 1 Sample per lot	ZF = 1.18
● 2 Samples per lot	ZF = 1.09
● 3 Samples per lot	ZF = 1.07
● emission (judged as if composed out of one piece)	
● 1 Sample per lot	ZF = 1.19
● 2 Samples per lot	ZF = 1.10
● 3 Samples per lot	ZF = 1.07

Note:

The permanence-factor has been derived, starting from a fixed variation-coefficient VC for the distribution within a lot and the measuring fault (VC= 0.25). Should the suppositions not agree with the real situation, then a different permanence-factor doesn't agree with the real situation, a different permanence-factor can be used. Also in the case that more than 3 samples per lot are tested, the permanence-factor should be determined again.

4.3. Joining together and/or splitting-up of lots

It is permitted to join together lots that are classified in the same category.

For recycled aggregates it is operative that lots may only be split into smaller units, when tested in a random control procedure or after approval in a lot-testing procedure, in which the minimum size of such a unit, or a portion of a lot, is decisive for the testing criterion.

When splitting up the flow of material into units smaller than the amount relative to the maximum testing-frequency of the moment (determined by the most critical component), then in calculating k, instead of starting from the testing requirement T, one starts from T multiplied by a factor P, in which $P \leq 1$.

$$\bar{k} = \frac{\ln (P \times T) - \bar{y}}{S_y}$$

in which:

T = testing requirement

y = progressive average of log-normal transformed observations $y_i = \ln (x_i)$ with x_i = observation i

S_y = progressive standard deviation of *ln* transformed observations.

P = correction factor.

Should the variation-coefficient within lots of analyses be unknown, one should start from a variation-coefficient of 0.65. An overview of the relation between lot-size and variation coefficients is provided in table 9.

Table 9. Relation between lot-size and variation coefficients			
Minimum size of lots after splitting up	variation-coefficient in lots after splitting up		
	0.30	0.65	1.00
≤ 1x testing-frequency *	1.00	1.00	1.00
$^1/_2$ x up to 1 x testing-frequency	0.99	0.98	0.96
$^1/_4$ x up to $^1/_2$ x testing-frequency	0.98	0.93	0.89
$^1/_8$ x up to $^1/_4$ x testing-frequency	0.95	0.85	0.78
$^1/_{16}$ x up to $^1/_8$ x testing-frequency	0.91	0.73	0.62
< $^1/_{16}$ x testing-frequency	0.82	0.57	0.45

Note : the lot-size in case of ≥ 1 x testing-frequency at least equals the annual production divided by the number of analyses per annum, as determined on the basis of k established for that particular case.*

The value of *k*, as obtained in this manner, determines the testing-frequency in the random control.

In the lot-testing procedure, lots can be split up to a certain minimum size, if for each of the components is applicable:

$$\bar{x} = \frac{P \times T}{ZF}$$

in which:

T = testing value

$\bar{x}$ = average of observations per lot

ZF = permanence factor

P = correction factor.

Should the variation-coefficient within lots be unknown, one should start from a variation-coefficient of 0.65, and using the overview provided in table 10.

Table 10. Relation between lot-size and variation coefficients			
Minimum size of lots after splitting up	variation-coefficient of analyses within lots		
	0.30	0.65	1.00
$^1/_2$ x up to 1 x lot-size	0.99	0.98	0.96
$^1/_4$ x up to $^1/_2$ x lot-size	0.98	0.93	0.89
$^1/_8$ x up to $^1/_4$ x lot-size	0.95	0.85	0.78
$^1/_{16}$ x up to $^1/_8$ x lot-size	0.91	0.73	0.62
< $^1/_{16}$ x lot-size	0.82	0.57	0.45

5. References

[1] Building Materials Decree (soil and surface water protection). Bulletin of acts, orders and decrees. No. 12689/165; idem: published by the Netherlands Government Gazette no. 247, 22 December 1995.

[2] Hendriks, Ch.F. and Schreurs, J., The Building products Decree, Æneas (in Dutch) 1999.

[3] BRL 2506 (National quality control document for recycled aggregates applied in concrete and for road constructions, in Dutch), CROW 1999.

[4] The Building Materials Decree, Ministry of Housing, Spatial Planning and the Environment, Directory of Information and External Relations, The Hague, The Netherlands 1996.