

METHOD OF QUANTIFICATION OF HYDRATED LIME IN ASPHALT MIXTURES

Didier Lesueur¹, Virginie Mouillet², Didier Séjourné², Vincent Delmotte²

¹Lhoist R&D, Nivelles, Belgium

²LRPC Aix-en-Provence, CETE Méditerranée, France

ABSTRACT

Hydrated lime has been known as an additive for asphalt mixtures for a long time and is now considered as an additive that increases asphalt mixture durability. It has been extensively used in the past 40 years in the USA, and is being increasingly used in most European countries, in particular Austria, France, the Netherlands, the United Kingdom and Switzerland.

Given this context, it is necessary to have a fast and reliable quantification method of the hydrated lime content in an asphalt mixture.

A German method was used in order to do so. The testing protocol consisted in first extracting the filler from the mix using the usual solvent extraction method (EN 12697-1). Then, 1g of the extracted filler was titrated with a 0.5 M HCl solution using a method adapted from EN 459-2.

The protocol was validated on a AC 10 mixture manufactured in the laboratory. The nominal content was 2.0 % hydrated lime based on the dry aggregate. The measured content was found to be 1.7 %, in reasonable agreement with expected results.

As a result, the hydrated lime content in an asphalt mixture can be evaluated. An estimate of the precision of the method is also given.

Keywords: hydrated lime, asphalt mixture, titration, acid-base, test method

1 INTRODUCTION

Hydrated lime has been known as an additive for asphalt mixtures from their very beginning [1,2,3]. It experienced a strong interest during the 1970s in the USA, partly as a consequence of a general decrease in bitumen quality due to the petroleum crisis of 1973, when moisture damage and frost became some of the most pressing pavement failure modes of the time. Hydrated lime was observed to be the most effective additive [4] and as a consequence, it is now specified in many States and it is estimated that 10% of the asphalt mixtures produced in the USA now hold hydrated lime [5].

Given its extensive use in the past 40 years in the USA, hydrated lime has been seen to be more than a moisture damage additive [3,6,7,8,9]. Hydrated lime is known to reduce chemical ageing of the bitumen [3,6,7,8]. Furthermore, it stiffens the mastic more than normal mineral filler [3,6,7,8], an effect only observed above room temperature [3], that impacts the mechanical properties of the asphalt mixture.

Given that all the above mixture properties impact the durability of asphalt mixtures, the use of hydrated lime has a strong influence on asphalt mixtures durability:

- North American State agencies estimate that hydrated lime at 1-1.5% in the mixture increases the durability of asphalt mixtures by 2 to 10 years, that is by 20 to 50% [5],
- The French Northern motorway company, Sanef, currently specifies hydrated lime in the wearing courses of its network, because they observed that hydrated lime modified asphalt mixture have a 20-25% longer durability [10].
- Similar observations led the Netherlands to specify hydrated lime in porous asphalt [11], a type of mix that now covers 70% of the highways in the country.

As a result, hydrated lime is being increasingly used in asphalt mixtures in most European countries, in particular Austria, France, the Netherlands, the United Kingdom and Switzerland.

In this context, it is somewhat surprising that the problem of the quantification of hydrated lime in asphalt mixtures has only attracted interest in the recent years. As detailed below, only two methods can be found in the literature, both published less than 10 years ago: the first one coming from the USA and the second one from Germany. In this work, we chose to use the German method in order to quantify hydrated lime in an asphalt mixture, because the testing equipment needed to put it into practice is usually already found in most road laboratories. In addition, its cost is very limited and the method could therefore be easily made available to a large number of control laboratories.

Therefore, this article first describes the published methods to quantify hydrated lime in an asphalt mixture. Then, it details the German method which has been first validated and then tested for repeatability and reproducibility in a new European round-robin test.

2 AVAILABLE METHODS TO QUANTIFY HYDRATED LIME IN ASPHALT MIXTURES

2.1 US Method

The US method was developed by the Federal Highway Administration (FHWA – [12,13]). It consists in measuring the Fourier Transform Infra-Red (FTIR) spectrum of the filler and quantifying the hydrated lime content from the peak intensity at $3,640\text{ cm}^{-1}$ corresponding to calcium hydroxide (Figure 1). Calcium carbonate peaks at $1,390\text{ cm}^{-1}$ and can be unmistakably separated from the hydrate (Figure 1).

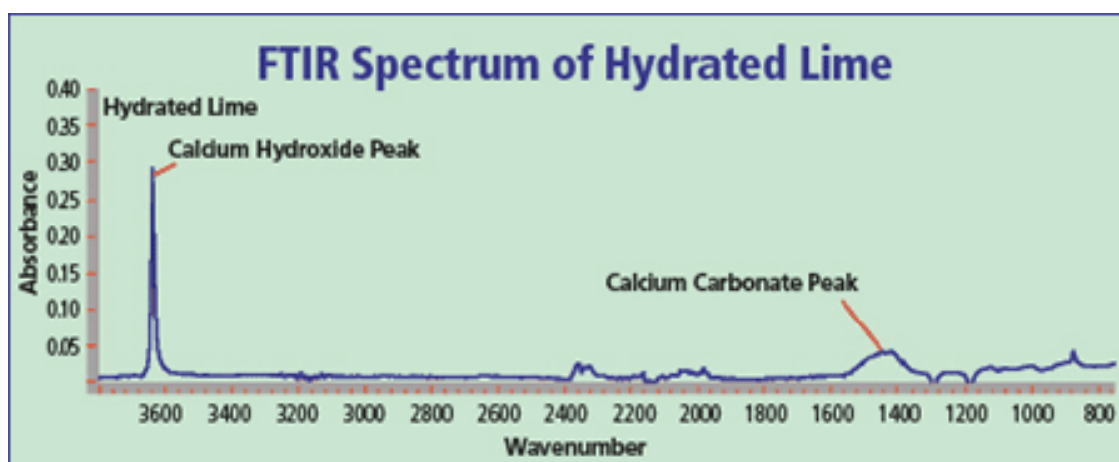


Figure 1: FTIR spectrum of hydrated lime (from [Error! Bookmark not defined.]).

The analysis was shown to be easily performed by using 15-20g of dust recovered by hammer drilling through an asphalt mixture with a 9.5mm tungsten carbide bit [12,13].

Interestingly, measurements on 10 years old materials from Nevada showed that no recarbonation or leaching had occurred in the corresponding time frame [12,13].

2.2 German Method

As explained in more details below, the German method [14,15] is very simple and derives from the lime characterization methods detailed in EN 459-2 [16]. In fact, the German method separates three different characterization sub-methods:

1. Hydrated lime purity,
2. Hydrated lime content in a mixed filler,
3. Hydrated lime content in the filler recovered from an asphalt mixture.

The test consists in a hydrochloric acid titration of a suspension of the product to be tested. The acid has to be weaker (0.5 M) when mixed or recovered fillers are concerned, in order to adapt for a lower basicity. The filler is recovered from an asphalt mixture using solvent extraction of the bitumen as described in EN 12697-1 (usually using trichloroethylene or tetrachloroethylene as a solvent - [17]). The suspension to be titrated is then obtained by blending 1g of recovered filler to 150ml of water, 10ml isobutanol and 5ml of a surfactant solution (1g Sodium DodecylSulfate and 1g polyethyleneglycol-dodecylether in 100ml water). The surfactant solution is needed only when recovered filler is tested, in order to wash out the filler from remaining bitumen or solvent from bitumen extraction. The coloured indicator is phenolphthalein (0.5g in 50ml ethanol, completed to 100ml by water). Titration rate is 12 ml/min initially, but decreases to 4 ml/min near the transition point. The method was shown to work with all types of fillers, including limestone filler [14].

A first national round-robin test was performed with 12 laboratories [14]. The repeatability (in terms of wt.% of hydrated lime in the filler) was 0.52wt.% and the reproducibility was 0.91wt.% for a mean value of 27.3wt.% .

The method was validated on samples taken out of cores 1.5 years after construction (Table 1 - [14]). The SMA 0/8 S mixes were made either with normal filler or with mixed filler containing 25wt.% hydrated lime and the results are given in Table 1 [14].

Table 1: Results of the validation of the German quantification method (after [14]).

Section	Nominal Hydrated Lime content	Measured Hydrated Lime Content in Recovered Filler
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	(wt.%)	(wt.%)
1	0	0.9
2	0	0.7
3	25	29.2
4	25	26.0

Note also that a study using different methods showed that the titration method was equivalent to the sugar method, which is the reference one in EN 459-2. Interestingly, the comparison based on asphalt mixtures made with different aggregates showed that part of the hydrated lime was not fully recovered, because of the hydrated lime – aggregate reactions (Figure 2). As a result, these reactions were more important for basalt aggregate (about 60% recovery), than moraine (about 80%) and limestone filler (about 90%).

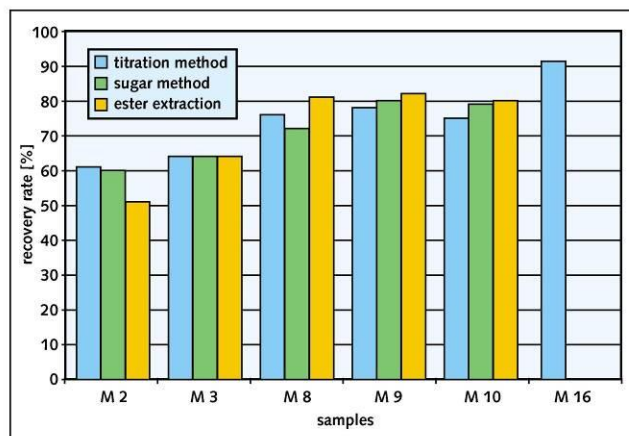


Figure 2: Percentage of hydrated lime eventually detected using three different chemical methods. (“Titration method” refers to the direct titration following the German method [15] described at length in the text. “Sugar method” refers to the titration of a saccharose extract of the filler to be tested and “Ester method”, to an ethyl-acetoacetate extract (from [14]). The materials were asphalt mixtures with different fillers mixed with hydrated lime: M2 and M3 with basalt filler (respectively 5 and 20% hydrated lime), M8 and M9 with moraine filler (respectively 5 and 20% hydrated lime), M10 with 67% moraine and 33% limestone filler (25% hydrated lime) and M16 with limestone filler (20% hydrated lime). The recovery rate is the ratio of dosificated lime over nominal lime content.)

3 EXPERIMENTAL

3.1 German Method

The German technical test method [15] is used for the determination of the calcium hydroxide content in hydrated lime and mixed fillers for hot mix asphalt. It can be used also for fillers extracted from asphalt mixture.

The testing protocol consists in first extracting the filler from the mix using the usual solvent extraction method (EN 12697-1). Then, two steps are performed to titrate the extracted filler:

- disperse the filler sample in a mixture of water, isobutanol and tenside solution in order to clean the filler (to remove the bitumen and/or the extraction solvent maybe still present)
- titrate in the alkaline range the calcium hydroxide content with hydrochloric acid using a method adapted from EN 459-2. But, it has to be noted that for mixed fillers and extracted fillers, it is necessary to determine the blank value of the used filler material.

This test method is very simple, easy to perform by all laboratories and unexperienced operators.

As 1 mole of Ca(OH)_2 reacts with 2 moles of HCl , the calcium hydroxide content expressed as (Ca(OH)_2) in mass fraction in %, is given by the following equation:

$$\% \text{Ca(OH)}_2 = 100 * 37.05 * (C1 * V_{eq}) / (1000 * m1)$$

where:

- C1 is the concentration of hydrochloric acid (mol/l) (note: as the concentration of solutions might deviate in time, a corrective factor has to be determined by titration with a base prepared by weighing)
- V_{eq} is the volume equivalent of hydrochloric acid (ml)
- m1 is the mass of taken filler sample (g)

3.2 Materials

In order to validate this method of quantification of hydrated lime (accuracy of the titration and repeatability), the first stage of this study has consisted in the titration of known concentration solutions of:

1. Pure components: two CL90 S (according to EN459-1) hydrated lime from Dugny and Flandersbach have been tested and a limestone filler coming from E.J.L. La Nerthe as well.
2. Mixed fillers manufactured in laboratory: preparation using a laboratory divider of mixed filler with known concentrations of hydrated lime: 25, 50 and 75% of lime. These percents of lime have been chosen in order to be representative of the mixed fillers available on the European market. For example, in Germany or the Netherlands, mixed fillers containing 20 or 25% of hydrated lime are mostly used, while, in France, they go up to 75% of hydrated lime. In general, the mineral filler used with hydrated lime is calcium carbonate but it could be another mineral filler. For this study, only one limestone filler has been selected in order to mimic using a mixed filler prior to introduction into the asphalt mix. Also, calcium carbonate being one of the most acid soluble rock that is commonly found in road aggregate, it serves as a good reference in order to check the risk of dosing some of the filler as hydrated lime.
3. Extracted filler from typical continuous asphalt mixture: preparation in laboratory of asphalt mix including filler with different known concentration of Dugny hydrated lime as a substitute for added filler followed by quantitative recovery of the extracted filler according to the European standard method EN 12697-1.

As the first step of this study has been performed by a single research laboratory to determine the accuracy of the titration, it has been decided in a second phase to launch a European round-robin test to assess the repeatability of the titration method. In order to do so, the same extracted filler was sent to different lime-producer and road laboratories. In total, 27 laboratories from all over Europe participated in this round robin test, in which the extracted filler with known content of hydrated lime has been analyzed according to the German method. The exploitation of this round robin test is described below. It has contributed to the evaluation of method from a practical point of view and to the determination of the repeatability of the quantification of hydrated lime.

4 RESULTS

4.1 Titration of calcium hydroxide content of known concentration solutions

4.1.1 Purity of hydrated lime

The specifications on hydrated lime (EN 459-1) oblige to state the purity that can affect the titration. Indeed, the hydrated lime can come from a low purity calcium which would yield a low Ca(OH)_2 content. Also some of the Ca(OH)_2 could have recarbonated due to extended storage in wet conditions, which would also decrease the available Ca(OH)_2 content. Consequently, the purity of components has been determined using the German method (table 2). For each of the pure components, 5 repeatability trials have been performed to assess the standard deviation of the analysis. As expected, no

calcium hydroxide content has been found in the limestone filler and the available Ca(OH)_2 (purity) of the 2 hydrated limes was around 95%. The standard deviation of the analysis was very good, around 0.3%.

Table 2: Calcium hydroxide content of the pure components (hydrated lime and limestone filler).

PURE COMPONENTS	CALCIUM HYDROXIDE CONTENT
Limestone filler	0%
Dugny hydrated lime	94.7% (standard deviation of 0.2%)
Flandersbach hydrated lime	94.5% (standard deviation of 0.3%)

4.1.2 Titration of mixed filler with hydrated lime

As defined in paragraph 3.2, mixed fillers have been manufactured in laboratory using limestone filler with adding of known concentrations of hydrated lime: 25, 50 and 75%. The different mixed fillers have been prepared with the two kinds of hydrated lime:

- Mixed fillers with Dugny lime

The accuracy of the titration of mixed fillers with Dugny lime was quite good as the relative deviation in relation to theoretical content was less than 2% for all lime contents (Table 3). Note that the standard deviation of the analysis (performed on 5 repeatability trials) was very good, around 0.6%.

Table 3: Calcium hydroxide content of mixed fillers with Dugny lime

SAMPLES	NOMINAL LIME CONTENT	CALCIUM HYDROXIDE CONTENT		
<i>Mixed fillers with x% of Dugny lime</i>		Measured content	Theoretical content (*)	Relative deviation in relation to theoretical content
	25%	23.3% (standard deviation of 0.6%)	23.7%	1.7%
	50%	46.5% (standard deviation of 0.5%)	47.4%	1.9%
	75%	69.7% (standard deviation of 0.6%)	71.0%	1.8%

(*) expected value obtained by correcting the nominal content for the purity of lime shown in table 2

- Mixed fillers with Flandersbach lime

For the mixed fillers with Fandersbach lime, the accuracy of the titration was quite similar to the previous one, although with a somewhat higher deviation (3.0%) for the 25% lime content. The standard deviation was slightly higher at 1.3%. The relative deviation in relation to theoretical content was comprised between 1 and 3% (Table 4).

Table 4: Calcium hydroxide content of mixed fillers with Fandersbach lime

SAMPLES	NOMINAL LIME CONTENT	CALCIUM HYDROXIDE CONTENT		
		Measured content	Theoretical content (*)	Relative deviation in relation to theoretical content
<i>Mixed fillers with x% of Fandersbach lime</i>	25%	22.9% (standard deviation of 1.3%)	23.6%	3.0%
	50%	46.4% (standard deviation of 0.3%)	47.2%	1.7%
	75%	70.2% (standard deviation of 0.7%)	70.9%	1.0%

(*) expected value obtained by correcting the nominal content for the purity of lime shown in table 2

4.1.3 Titration of extracted filler from typical continuous asphalt cement

A typical continuous asphalt mixture has been prepared in laboratory including 3.8% of added limestone filler coming from Ejl La Nerthe (Table 5). The mixture was made of crushed river gravel from Durance Granulats.

Table 5: Calcium hydroxide content of extracted fillers with Dugny lime

COMPONENTS	INTERNAL PERCENT	PERCENT OF FILLER OF EACH GRANULAR FRACTION (*)	PERCENT OF RECOVERED FILLER
0/2 mm granular fraction	22.7%	15% maximum	3.41%
2/6 mm granular fraction	34.1%	0.5% maximum	0.17%
6/10 mm granular fraction	34.1%	0.5% maximum	0.17%
Added filler	3.8%	99% minimum	3.76%
Bituminous binder	5.3%	/	/
Total filler			7.51%

(*) as requested in the French standard NF P 18-545

Note that mixed fillers with different known concentrations of Dugny lime (0, 50 and 100%) were used as a substitute for the added filler. Then, the total filler composed of a mix of hydrated lime, limestone filler and filler coming from the sandy fraction of the river gravel (almost 8%) has been quantitatively recovered according to European standard method EN 12697-1 (Asphalt Analysator method) and titrated according to German method (table 6).

Table 6: Calcium hydroxide content of extracted fillers with Dugny lime.

SAMPLES	NOMINAL LIME CONTENT IN THE ADDED FILLER	NOMINAL LIME CONTENT IN THE RECOVERED FILLER	CALCIUM HYDROXIDE CONTENT		
			Measured content	Theoretical content (*)	Relative deviation in relation to theoretical content
<i>Asphalt Cement including mixed fillers with x% of Dugny lime</i>					
	0%	0%	0%	0%	0%
	50%	23.5%	19.7%	22.3%	11.7%
	100%	47.0%	42.1%	44.5%	5.4%

(*) expected value obtained by correcting the nominal content for the purity of lime shown in table 2

For the extracted filler, the accuracy of the titration was higher than for laboratory mixed filler: the relative deviation in relation to theoretical content lied between 5 and 12% (Table 6). This could come in part from variations in the percent of filler coming from the sand (0/2 aggregate), as the French standard NF P 18-545 set only a maximum of 15% in the granular fraction 0/2 mm.

4.2 International Round-Robin test

The round-robin test was performed in the summer of 2011. 36 laboratories from all over Europe participated (Table 7), 20 being control or research laboratories of lime producers and 16 being road laboratories involved in the formulation and control of asphalt mixtures.

Table 7: List of laboratories involved in the round-robin.

Country	Number of laboratories	Lime Producer	Road Laboratory
Austria	7	1	6
Belgium	5	3	2
France	1	0	1
Germany	12	10	2
Italy	4	1	3
Netherlands	1	0	1
Norway	2	1	1
Spain	3	3	0
Total	36	20	16

The round-robin consisted in first manufacturing in the laboratory a new asphalt mixture containing a given hydrated lime content, namely 30.86%. This figure was obtained by adding 6% filler holding 70% hydrated lime with 94% purity, in a mixture having in total 12.76% filler (i.e. added filler plus filler from the aggregate fractions). This nominal lime content was not disclosed to the laboratories. All the filler was extracted from the mixture at LRPC Aix-en-Provence following EN 12697-1 and was then sent to the laboratory of the Bundesverband der Deutschen Kalkindustrie (BVK) in Köln (Germany). There, the filler was prepared into 25-g specimen sent to each participating laboratory. Along with the filler, the needed tensides (sodium dodecylsulfate and polyethyleneglycol) were added. This was done so because the local availability of the surfactants was not insured in all countries.

Each laboratory received the test method in three languages (English, French and German). It was asked that each laboratory would duplicate the quantification of calcium hydroxide in the received filler specimen, and report both results. Together with the final result expressed in calcium hydroxide content in the filler, we requested that the laboratory also report the mass of filler used for the trial and the volume of acid used. This way, potential deviations coming from the use of stronger / weaker acids could be easily detected.

At the time of writing of this paper, 27 laboratories had replied and the results are therefore still preliminary.

The raw test results are presented in a graphical way in Figure 3. Note that each lab were asked to perform two replicates, but some labs performed four, so they then appear a second time (e.g., lab 7 was in this case, so it appears as 7a and 7b). Clearly, the raw data showed 2 anomalies:

1. labs 15 and 16 had very high lime contents. They were asked to redo the testing. The new set of data has not arrived yet and they are therefore considered as anomalous at the present time and were taken out of the final statistical analysis,
2. labs 3, 24, 32, 33, 34 and 35 had very low lime contents, about half of the mean value. After checking with them, it appeared that the strength of the acid was not correctly reported and the data were then corrected for this.

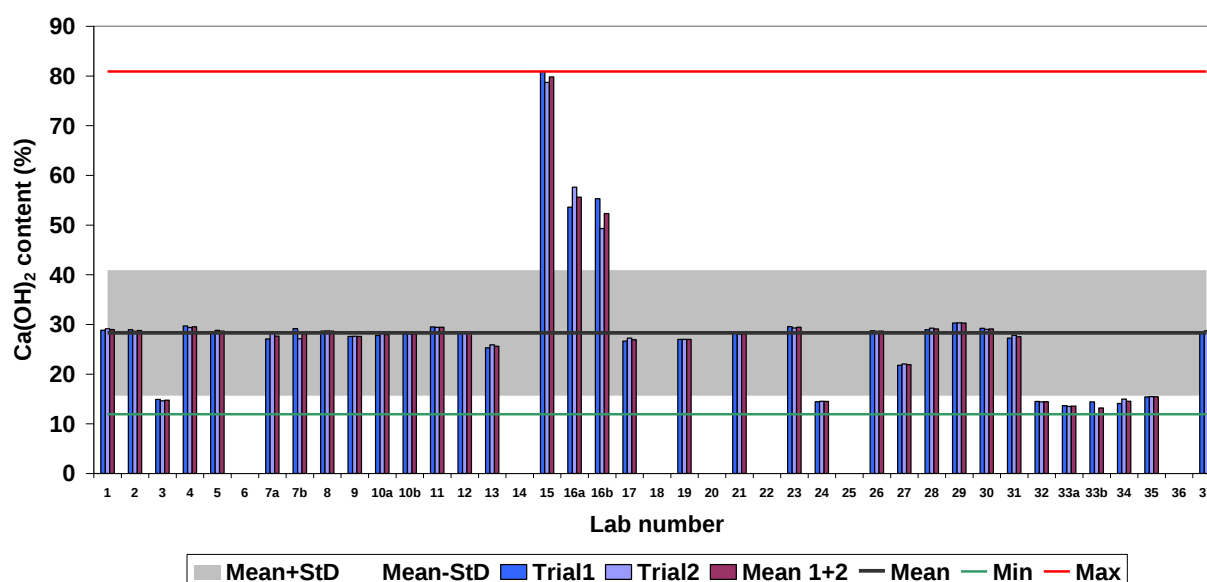


Figure 3: Uncorrected raw results from the round-robin test. Note that the nominal lime content was 30.86% in the filler and was not known to the laboratories. Each lab was asked to perform two replicates, but some labs performed 4 so they then appear a second time (e.g., lab 7 was in this case, so it appears as 7a and 7b).

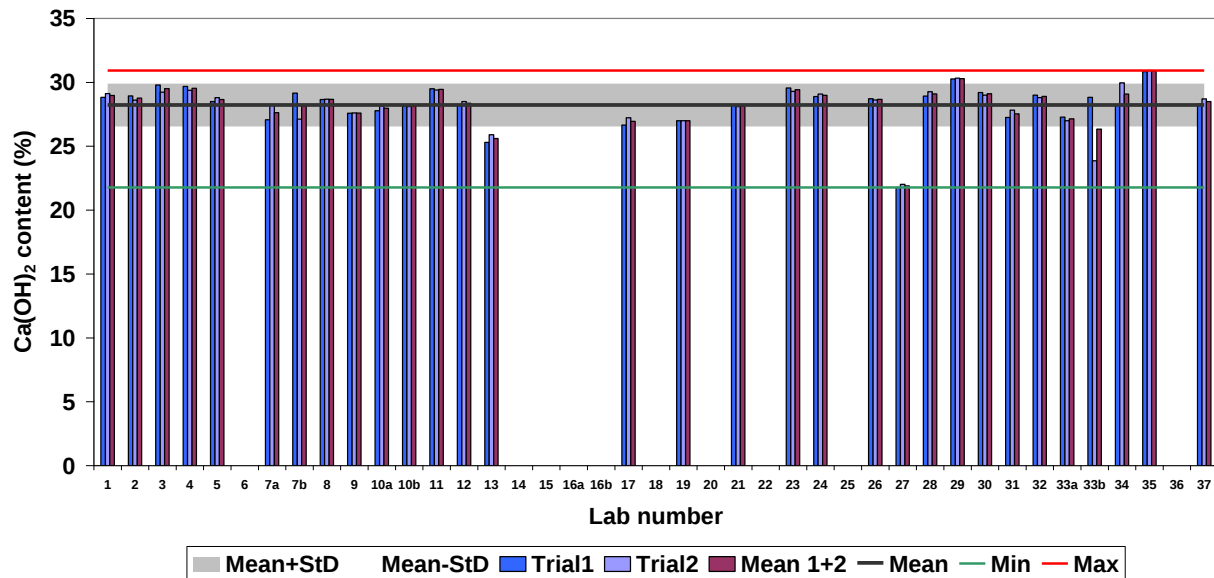


Figure 4: Corrected results from the round-robin test. Note that the nominal lime content was 30.86% in the filler and was not known to the laboratories. Each lab was asked to perform two replicates, but some labs performed 4 so they then appear a second time (e.g., lab 7 was in this case, so it appears as 7a and 7b). Anomalous data were corrected as described in the text.

After correction for anomalous data as explained below, the new set of results became the one shown in Figure 4. Now, the range of measured values was a lot smaller than without corrections. The minimum value was 21.97 % and the maximum, 30.92 %. The mean was 28.22% with a standard deviation of 1.68% in absolute terms or 5.95% in relative terms.

The full statistical analysis is still to be performed since some of the results have not been received yet. In all cases, it is quite clear from these preliminary results that the method has a good repeatability and reproducibility with a standard deviation below 2% in terms of absolute Ca(OH)_2 content.

In this sense, the mean value would be significantly below the theoretical value. Apart from manufacturing issues (less lime or more filler than wanted could have finally ended up in the mixture), this could also be a consequence of some hydrated lime consumption due to chemical interactions with the aggregate, as already observed in former work (see Figure 2 – [14]). This suggests that the use of this method for production control would be better suited if a first calibration is made in order to assess the lime consumption factor for the mixture, and then use this calibration factor to express the measured Ca(OH)_2 content in terms of initial Ca(OH)_2 content, i.e., the quantity of hydrated lime that was present in the mixer / drum before reacting with the aggregate.

5 CONCLUSIONS

This article first presented the two existing methods found in the literature, one from the USA and the other from Germany, in order to quantify the hydrated lime content in an asphalt mixture. The US method is based on Infra-Red spectroscopy while the German method is based on acid-base titration.

This last one seems to be the easiest to install in control laboratories given that the test set-up, i.e., a titrator, is cheap and simple and already found in most road laboratories. Therefore, it was carefully evaluated, first in one laboratory, then in a European round-robin gathering more than 27 laboratories from 8 different countries. The round-robin consisted in quantifying the calcium hydroxide content in the sample of extracted filler sent to each lab.

The German method consists in first extracting the filler from the mixture using the standard method EN 12697-1 already used daily in asphalt laboratories. The extracted filler is then titrated with a hydrochloric acid solution and the calcium hydroxide content of the tested filler is then obtained. Knowing the filler content in the mix, it is easy to calculate the calcium hydroxide content which is almost similar to the hydrated lime content in the mix.

Although the full analysis of the round-robin test remains to be done (because of some missing results), the method was seen to be quite robust, with a mean value of 28.22% and a standard deviation of 1.68% in absolute terms. Given that the real content was 30.86%, a slight but significant deviation was observed.

This deviation could come from manufacturing issues (less lime or more filler) but could also come from hydrated lime consumption by the aggregate. This was already observed in former work. Therefore, it would be better to evaluate the lime consumption factor of the mixture to be controlled prior to using this method on a daily basis as a tool to control the hydrated lime content in asphalt mixtures. In all cases, the robustness of the method makes it a valuable method for the industry in order to assess the hydrated lime content in an asphalt mixture.

6 ACKNOWLEDGEMENT

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