A RAPID METHOD FOR CHECKING THE BITUMEN CONTENT OF ASPHALT

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1. INTRODUCTION

In the manufacture of asphalt great emphasis is placed on the bitumen content of the material. Bitumen content tests are undertaken regularly at predetermined intervals and results are analysed statistically during the manufacture of the asphalt and the construction of the asphalt pavements. This is undertaken in order to ensure that the desired quality is obtained as well as to detect any problems in the asphalt manufacturing process timeously.

It is therefore necessary to ensure that bitumen content test results are accurate and reliable. In practice bitumen content tests are generally sufficiently accurate and reliable. However, this is not always the case and a rapid and efficient method of verifying the bitumen content is desirable. This applies especially in cases where conflicting test results are reported by two independent laboratories (process control and acceptance control laboratories), or when unexpected trends are observed in the test results.

2. THE IMPORTANCE OF RELIABLE AND ACCURATE BITUMEN CONTENT TEST RESULTS

Reliable and accurate bitumen content test results are important for the following reasons:

- The bitumen content of asphalt plays a vital role in the performance of asphalt - it has a pronounced influence on the permeability, durability and stability of the asphalt, the most important properties of asphalt. It follows that the control of the bitumen content is probably the most important element in the quality assurance of asphalt.
- Modern asphalt plants are equipped with very effective devices for metering the addition of bitumen to the hot, dry aggregate. However, as in any manufacturing process, things can go wrong and it is therefore necessary to monitor the bitumen content of the asphalt by means of laboratory testing.
- Every bitumen test result recorded is subject to an inherent error - like any other scientific test result. This inherent error is usually due to (a) sampling errors and (b) testing errors. This paper deals with the latter, i.e. testing errors. Standard test procedures take into consideration the magnitude of inherent errors and limits are given for repeatability (repeated tests by the same operator using the same equipment) and reproducibility (tests by different operators using different equipment - different laboratories). The standard test procedures should thus give a range (+ or -) within which a single test result should fall i.e. when the test is performed on a control sample with known bitumen content. Typical criteria in this regard are given in the ASTM (American Standard Testing Methods) testing specifications and are as follows for bitumen content (extraction method) and maximum theoretical density testing [1]:

Bitumen Content (BC) - (ASTM D2172)

- Repeatability : Single test difference, percent = ±0,295
- Repeatability : Standard deviation, percent = 0,21
- Reproducibility : Single test difference, percent = ±0,31
- Reproducibility : Standard deviation, percent = 0,22

Maximum Theoretical Density (MTD) – (ASTM D2172)

- Repeatability : Single test difference = ±0,0110
- Repeatability : Standard deviation = 0,0040
- Reproducibility : Single test difference = ±0,0190
- Reproducibility : Standard deviation = 0,0064

The above applies to the following test conditions: 2 replicates per sample, 1 samples and 112 laboratories. The above shows that the inherent errors in the bitumen content (BC) testing procedure is such that if two identical replicates are tested it would still be acceptable if the two measured bitumen content values are within 0,295 percent of each other. For instance if one result is say 5,0% the second test result can deviate by ±0,295% and still be acceptable, i.e. between 4,705% and 5,295%. This same reasoning applies to maximum theoretical density (MTD) testing procedure.

- In practice, errors in bitumen content tests, which are significant and which exceed the limits of reproducibility, occasionally occur. Such errors are not acceptable. This can result in the acceptance of substandard asphalt or the rejection of acceptable asphalt and such errors are a poor reflection on those responsible for the quality assurance of asphalt. Faulty equipment, incorrect procedures, poorly trained or careless operators or a combination of these or other causes could cause such a situation. This is often detected when there is a discrepancy between process control and acceptance control results. When there are two different sets of results which one is correct? A dispute often arises and then a third laboratory is commissioned. It is a time consuming and expensive exercise that may still be inconclusive.

On a recent project the test results from two laboratories differed significantly. Whilst the mean bitumen contents were sufficiently close, the results from one laboratory appeared to be dubious as the measured bitumen content values remained fairly constant but consistently approaching the upper limit of the acceptable range. The other laboratory reported values that fluctuated within the acceptable range but generally remained lower than the target bitumen content. Instead of commissioning a third laboratory to settle the differences a method for verifying the test results was developed. The method is simple, rapid and can be carried out at virtually no cost. It is based on the indirect determination of the bitumen content using the MTD. No additional cost is involved since the MTD has to be determined in any case for the purpose of (compaction) density control. The method and its evaluation based on test results from an actual project are described in the following paragraphs.

3. THEORY

The maximum theoretical density (MTD) of an asphalt mixture is equal to the total weight of both the bitumen and the aggregate divided by the total volume occupied by both these two materials. The volume of air contained in the mixture is excluded. This is an important property of the material since the density specifications are related to this value.

This property is measured exclusively using the vacuum saturation test, better known as the Rice test. However the MTD of the asphalt mixture can also be calculated as follows :

$$MTD = (RD_{agg} + RD_{agg} \times BC') / (1 + (RD_{agg} \times BC') / RD_{bit}) \qquad (1)$$

Where :
- MTD – Maximum theoretical density (ton / m^3)
- RD_{agg} – Relative Density of the aggregate (ton / m^3)
- RD_{bit} – Relative Density of the bitumen (ton / m^3)
- BC' – Bitumen content (dimensionless, range : 0,0 to 1,0)

Therefore if the relative density of the aggregate, the relative density of the bitumen and the bitumen content are known the MTD of the material can easily be calculated.

It is however important to note that the relative density of the aggregate needs to take account of the pores in the aggregate into which a small portion of the bitumen can be absorbed (known as bitumen absorption).

By inspection of Formula 1 it can be seen that the bitumen content (BC') can readily be determined should the MTD and the relative densities of both the bitumen (RD_{bit}) and the aggregate (RD_{agg}) be known. Formula 2 is achieved by rearranging Formula 1 :

$$BC' = (MTD - RD_{agg}) / (RD_{agg} - (MTD \times RD_{agg} / RD_{bit}) \qquad (2)$$

Formula 2 can be used during acceptance control testing to calculate the bitumen content of various asphalt samples for which the MTD was determined. This procedure should provide fairly accurate results because the relative density of the aggregate should not vary much within the same quarry. This value should therefore only have to be determined on a few occasions during a particular project. The same holds true for the relative density of the bitumen.

The bitumen content value indicated above (BC') is based on the total mass of the aggregate alone and not based on the mass of the total mixture (BC). To convert the bitumen content value from BC' to BC (the normal manner in which binder content is specified) the following equation can be used :

$$BC = BC' / (1 + BC') \text{ ; where } BC' \text{ varies between 0,0 and 1,0} \qquad (3)$$

Note : To express BC as a percentage value subsequent to carrying out the calculations multiply value by 100, e.g. BC = 0,052 is 0,052x100 = 5,2 %

The relative density of bitumen could range between 1,000 to 1,060 ton/m^3 for penetration grades 20/30 to 150/200 [2]. The relative density of bitumen is approximately 1,025 ton / m^3 for 60/70 penetration grade bitumen at 20 °C. (60/70 penetration grade bitumen is most often used to manufacture asphalt for ordinary road applications in South Africa).

Before the application of this method is illustrated by means of test results obtained from an actual project it is important to briefly outline the general quality control process that is followed when asphalt material is evaluated. This is described in the following section.

4. QUALITY CONTROL DURING ACTUAL PROJECT

During a project the quality of the asphalt is controlled using the following properties:

- Bitumen content (BC)
- Air voids in the mix (VIM)
- Grading of the aggregate

The bitumen content is measured using the extraction method as described under Method C7(a) in the TMH1 document [3]. Six test results are generally obtained for each lot and the results are statistically analysed and evaluated against prescribed judgement schemes. A typical judgement scheme is given in COLTO's Standard Specifications for Road and Bridge Works for State Road Authorities [4].

The VIM of the material is measured in the laboratory by means of compacting the material sampled in the field at 145 ºC in the laboratory using standard Marshall compaction. This generally requires reheating of the asphalt samples. After the material had been compacted into briquettes the bulk relative density of the material is measured by means of Method C3 described in the TMH1 document [3]. The MTD is measured using Method C4(a) described in the TMH1 document [3]. This test is known as the Rice test. The bulk relative density of the material is divided by the MTD of the material from which the VIM is calculated : $VIM = (1 - BRD/MTD) \times 100$.

The aggregate component of the sample from which the bitumen was extracted during the bitumen content determination is used for the particle size analysis. This information is generally reported with the bitumen content values but is only used for acceptance control when special mixtures are used, e.g. Stone Mastic Asphalt or Porous Asphalt (open graded asphalt). (For the standard continuously graded mixtures the aggregate grading should still be statistically controlled on a regular basis even though it is not a COLTO acceptance control requirement [4]).

From the above it can be seen that the MTD of the material has to be determined for each test for which the bulk relative density is required. The MTD of the asphalt material is therefore known for each sample of asphalt tested. This information can be used to back calculate the bitumen content of the material for the same lot. Assuming that the relative density of the aggregate and the relative density of the bitumen do not vary significantly and assuming that the MTD should be fairly accurately measured using the Rice test it should follow that the bitumen content calculated using the MTD should be similar to the measured bitumen content, taking into account inherent test errors.

For the remainder of the paper the focus will fall on the bitumen content determination and the use of the calculation technique described above.

5. RESULTS OBTAINED FROM AN ACTUAL PROJECT

On a particular project approximately 2000 tonnes of bitumen treated base (BTB) material was used for backfilling excavations during the pavement repair portion of the work. The design bitumen content of the BTB material was 4,7% by mass of the total mixture. The BTB material delivered to site was monitored on a daily basis by taking three random samples for testing. A private commercial laboratory undertook this testing. *This laboratory is hereinafter referred to as the independent laboratory*. Due to the quantity of material placed per day it was decided that two days work would be lumped together and treated as one lot for acceptance control purposes as six samples are required by the COLTO [4] acceptance control judgement scheme. The duration of this portion of the work was twelve days, which resulted in six lots to be evaluated. The average bitumen content values as obtained from the test results of the independent laboratory showed that the measured bitumen content for days 1 and 2 (Lot No. 1) conformed to the specification. However, for days 3 and 4 (Lot No. 2) the average measured bitumen content increased to 5,05%. This is just outside of the upper limit of the specification, namely 5,0% (the allowed tolerance for bitumen content is ±0,3%) and was therefore unacceptable.

As a general rule the independent laboratory's results are exclusively used for acceptance control, which effectively meant that the material had to be rejected.

Upon the rejection of Lot No. 2 the manufacturer presented the test results obtained from his process control laboratory, *hereinafter referred to as the manufacturer's laboratory*. The manufacturer has to test his product on a daily basis in order to do his own process control and therefore these test results are readily available. These results showed that the bitumen content values for days 1 to 4 conformed to the required specification.

After perusing the VIM values and the quantity of material passing the 0,075mm sieve, both values remaining fairly constant, it was concluded that the independent laboratory's bitumen content measurements might be in error. It was consequently decided that the available data would be analysed more closely and that additional third party testing will be undertaken if necessary in order to evaluate the quality of the asphalt objectively.

The analysis of the test data mainly revolved around the calculated bitumen content values of the material, as described above.

Table 1 shows a summary of the average test results obtained during the 12 days this portion of the work was in progress.

Table 1: Measured and calculated bitumen content values (%) with the MTD values as obtained from the independent and the manufacturer's laboratories

Day	BC IL	C-BC IL	MTD - IL	BC Mnf	C-BC Mnf	MTD-Mnf
1	4,50	4,67	2,525	4,80	4,56	2,529
2	4,53	4,85	2,518	4,50	4,43	2,534
3	5,10	4,93	2,514	4,50	4,38	2,536
4	5,00	4,72	2,523	4,65	4,61	2,527
5	4,97	5,05	2,510	4,55	4,07	2,548
6	4,97	5,31	2,500	4,70	4,53	2,530
7	4,97	4,80	2,519	4,70	4,45	2,533
8	4,93	4,43	2,534	4,50	4,02	2,550
9	4,97	4,78	2,520	4,40	4,00	2,551
10	4,73	4,38	2,536	4,70	4,66	2,525
11	4,83	4,24	2,541	4,55	4,58	2,528
12	5,07	4,88	2,516	4,67	4,10	2,547
Ave	**4,88**	**4,74**	**2,521**	**4,60**	**4,37**	**2,537**
Std Dev	**0,20**	**0,30**	**0,012**	**0,12**	**0,25**	**0,010**

Note : BC IL : Bitumen content as measured by the independent laboratory
BC Mnf : Bitumen content as measured by the manufacturer's laboratory
C-BC IL : Calculated bitumen content using the independent laboratory's MTD values
C-BC Mnf : Calculated bitumen content using the manufacturer's MTD values
MTD – IL : Maximum theoretical density as measured by the independent laboratory
MTD – Mnf : Maximum theoretical density as measured by the manufacturer's laboratory

6. DISCUSSION OF THE TEST RESULTS

6.1 General details of reported test results

The calculated bitumen content values shown in Table 1 above were calculated using the following relative density values in Formula 2: RD_{agg} = 2,72 and RD_{bit} = 1,025. It has also been assumed that these two figures remain constant throughout the project as the source of the materials (bitumen and aggregate) remained constant. Since no specific testing has been undertaken in this regard it leaves the determination of the absolute bitumen content values in question but since these two values are constant throughout this exercise it leaves the calculated bitumen content values valid for comparative purposes. It is the evaluation of the trends between the measured and the calculated bitumen content values that are of particular interest.

By analysing the trends between the measured bitumen content and the calculated bitumen content values for both the laboratories an insight into the quality of a particular laboratory's testing is obtained. This is due to the fact that the bitumen content of the various samples is determined using two totally independent tests, namely the extraction test (TMH1 : Method C7a) [3] and the maximum theoretical density test (TMH1 : Method C4a) [3].

In order to draw comparisons in this regard the results for each laboratory are shown separately in Figure 1 and Figure 2 below.

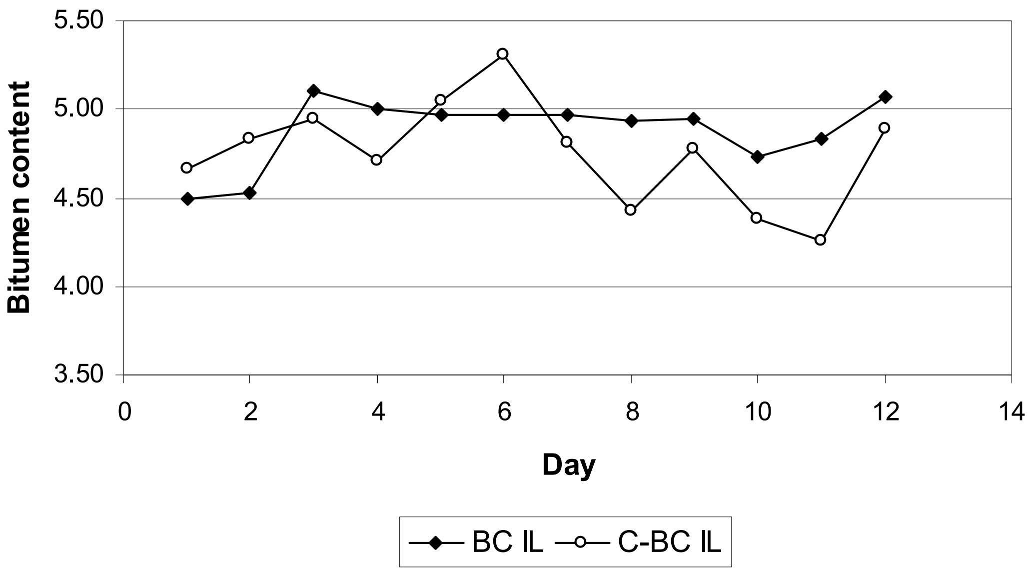

Figure 1: Independent laboratory – measured and calculated bitumen contents

From Figure 1 it can be seen that, in the case of the independent laboratory, the calculated bitumen content values and the measured bitumen content values don't follow any trend. The correlation coefficient (R^2) is 0,3196 which indicates poor correlation between these two data sets. It is particularly apparent between days 3 and 9 where the average measured bitumen content values remain fairly constant around the 5,0 % level and the calculated bitumen content values varied considerably between 4,43% and 5,31%.

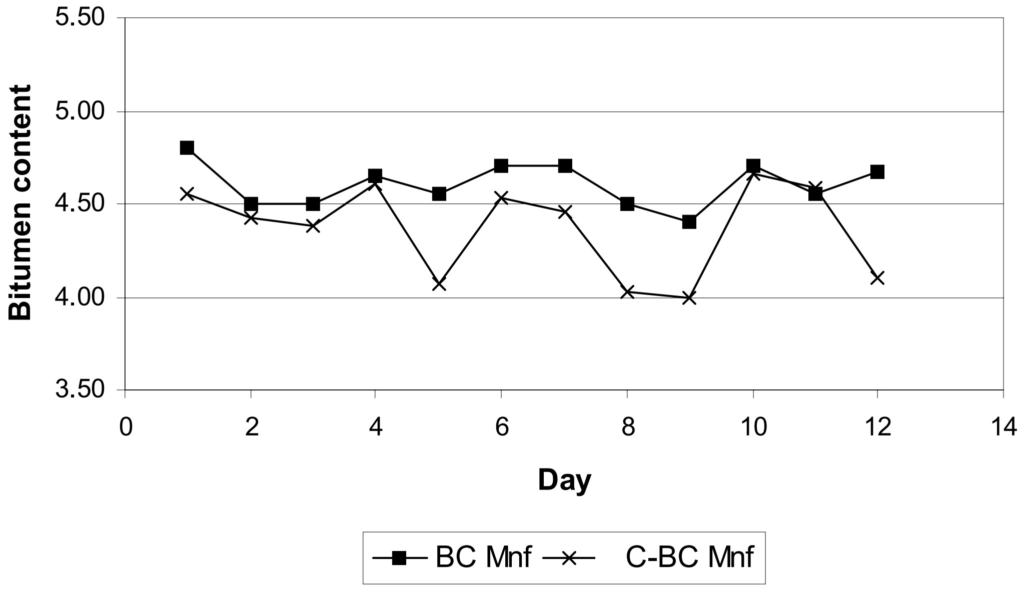

Figure 2: Manufacturer's laboratory – measured and calculated bitumen contents

From Figure 2 it can be seen that, in the case of the manufacturer's laboratory, the calculated bitumen content values and the measured bitumen content values follow a trend, i.e. if the measured bitumen content increases the calculated bitumen content also increases and conversely. In this case the correlation coefficient (R^2) is 0,5930 which is reasonable and nearly double the correlation coefficient for the independent laboratory. The calculated bitumen content values for days 5, 8 and 9 are significantly lower than the measured bitumen content although still following the same general trend. The calculated bitumen content for day 12 is the only obviously poor result. It is apparent that for days 1, 2, 3, 4, 6, 7, 10, and 11 there is good agreement between the measured and the calculated bitumen content values in addition to following the same trend.

It is also apparent from Figure 1 that some of the calculated bitumen content values are higher than the measured bitumen content values whereas in Figure 2 the calculated values are, with the exception of day 11, all lower than the measured bitumen content values. This can be explained by looking closely at Formula 2 and the influence of the MTD test as described in the following paragraph.

6.2 Influence of variation in MTD testing on calculated bitumen content values

From Formula 2 it can be seen that the bitumen content increases as the MTD of the asphalt mixture decreases and the converse also applies. Since the relative density of bitumen is lower than the relative density of the aggregate it follows that the MTD will decrease as the bitumen content of the asphalt increases. With this in mind it is clear that if some air is left in the asphalt sample during the execution of the MTD test it will lead to the measurement of a lower MTD, which in turn will result in the calculation of a higher bitumen content. Therefore it can be argued that the bitumen content calculated using the MTD of the material will have a lower limit, i.e. provided all the air is removed from the asphalt sample during the MTD test. The highest MTD will be measured which will result in the lowest calculated bitumen content. Should some air be trapped in the sample a lower MTD will be measured resulting in the calculation of a higher bitumen content, which could differ significantly, from the bitumen content measured by extraction.

The above argument can be used to explain why the calculated bitumen values shown in Figure 1 are criss-crossing the measured bitumen content values. This could be indicative of poorer quality testing on the part of the independent laboratory with regard to either the bitumen extraction test or the MTD test, or both.

6.3 Influence of relative density of bitumen and aggregate on calculated bitumen content values

From Table 1 it can be seen that the average calculated bitumen content values are lower than the average measured bitumen content values for both laboratories. Figure 2 further shows that the calculated bitumen content values are, except for one point, all lower than the measured bitumen content values. Therefore it is possible that the lower average calculated bitumen content value could be due to the use of too low relative density values for either the bitumen or the aggregate or both.

Note : On this project the relative density of the aggregate and the bitumen had not been measured and assumed values were used for the calculation of the theoretical bitumen contents. It is suggested that the relative density of the aggregate and the binder be measured at the start of every large asphalt paving contract so that these values can be used for assessing the reliability of the test results received from the different testing laboratories.

6.4 Statistical analysis of the differences between the bitumen content values measured by extraction and calculated from the MTD values

In addition to observing the trends between the two sets of bitumen content values the statistics of the differences between the measured and the calculated bitumen content values can be evaluated for each of the laboratories. The relevant statistics are shown in Table 2 below.

Table 2: Statistical evaluation of the differences between the measured and the calculated bitumen content values for each of the laboratories

Statistical value	Independent Laboratory	Manufacturer's Laboratory
Average	0,14	0,23
Standard Deviation	0,299	0,194
Minimum difference	-0,33	-0,03
Maximum difference	0,58	0,57

From Table 2 it can be seen that the range of the difference between the measured and the calculated bitumen content values is the greatest for the independent laboratory. In addition it can also be seen that the standard deviation of the difference between the measured and the calculated bitumen content values for the independent laboratory is the greatest. It is however important to notice that the standard deviation and the magnitude of the range of the data set, comprising the differences between the measured and the calculated bitumen content values, are independent of the offset between the measured and the calculated bitumen content values. Therefore should the relative density values of the aggregate and the bitumen be altered in order to render the average calculated bitumen content and the average measured bitumen content values the same it would not alter the standard deviation or the magnitude of the range of the abovementioned data set. Table 2 indicates that the standard deviation and the magnitude of the range of the data set for the manufacturer's laboratory are significantly lower than that of the independent laboratory and is indicative of better quality testing. Interestingly enough the standard deviation determined for the manufacturer's laboratory (0,194) is within the required limit for both the repeatability and reproducibility of the bitumen content test, as defined by ASTM precision criteria, and thereby indicates that it is sufficiently accurate to be used in this application.

From the information presented above it can be deduced that the quality of the testing by the manufacturer's laboratory is of a higher standard than the independent laboratory's testing.

6.5 Influence of the required precision of the MTD test results on the precision of the calculated bitumen content values

It is also important to look at the effect of the inherent variability of the MTD test on the calculated bitumen content values. It is noted in the introduction that the repeatability of the MTD test should satisfy the following criteria: single test difference = ±0,011; standard deviation = 0,0040. Assuming that the specific gravities of the aggregate and the bitumen are 2,72 and 1,025 respectively a single test difference of ±0,011 in the MTD test will result in a ±0,28% difference in the calculated bitumen content of the asphalt. A standard deviation of 0,0040 will result in a standard deviation of 0,11% in the calculated bitumen content of the asphalt. These two figures (0,28% and 0,11%) conform to the precision required by the ASTM criteria for the bitumen extraction test which is 0,295% and 0,21% respectively. Therefore if the variation in the specific gravities of the aggregate and the bitumen, originating from the same sources, is negligible it will render the calculation technique sufficiently sensitive to calculate the bitumen content values for comparative purposes. It is apparent from the lower limits noted above that the calculation method should be able to more accurately determine the bitumen content of the asphalt than the extraction method provided the correct relative density values for both the aggregate and the bitumen are used. Since this introduces additional variability into the calculation procedure it is necessary that the variation in the relative density of the aggregate and the bitumen be investigated and its influence on the calculated bitumen content values determined. This will require a separate investigation.

An appealing aspect of the procedure described above is that it provides a method by which the laboratory's testing quality can be assessed and since it is difficult to fudge the test results of the Rice test this procedure should prove to be sufficiently robust.

7. CONCLUSIONS

From the theory presented and the example given the following conclusions can be drawn:

- The calculation technique is a quick and easy calculation that can be used to determine the bitumen content of asphalt indirectly. It affords the opportunity to determine the bitumen content of the asphalt using two totally independent tests, namely the bitumen extraction test and the maximum density test.
- It has been shown that there should be correlation between the measured and the calculated bitumen content values regardless of the inherent test errors. This affords the opportunity to assess the quality of the testing of different laboratories. The better the correlation between the bitumen content measured by extraction and by the calculation method the better the quality of the testing and consequently the more reliable the test results.

- It has also been shown that the standard deviation of the differences between the measured and the calculated bitumen content values are within the precision required for the reproducibility of the bitumen extraction test as defined by the ASTM. This indicates that calculation technique should yield reliable bitumen content values provided the relative density of the aggregate and the relative density of the bitumen is correctly determined and the variation thereof is negligible for a particular project. These values should be measured at the start of the project and checked at appropriate intervals during the construction period.
- The calculation technique for determining binder content is dependent on the relative density of the binder and the aggregate, the absorption of the aggregate and the accuracy of the Rice test results i.e. four factors. It is important to measure all of these properties and their variation very accurately in order to ensure reliably calculated binder contents.
- This technique is powerful in the sense that a better evaluation of the acceptability of the test results can be made quickly without resorting to additional testing.

8. RECOMMENDATIONS

- In theory, there should be no difference between the binder content obtained by extraction and by calculation. Therefore research should be undertaken to determine the cause of the small differences found in this investigation.
- It is recommended that the magnitude and the variation of the relative density of the aggregate and the bitumen be investigated and its influence on the calculated bitumen content values determined.
- Since variation in the test results is unavoidable it is recommended that this method be evaluated under controlled laboratory conditions and that a correlation between the measured and the calculated bitumen content values be determined for various asphalt samples.
- Statistical precision criteria also need to the determined for this calculation technique. This can be achieved by the evaluation of the test results obtained from the controlled laboratory study mentioned above.
- It is recommended that this technique be used as part of the quality control process on projects where asphalt work is being undertaken. The output of these projects could then be used to develop acceptance criteria by which the quality of the testing can be measured. The departure point should not be to replace the MTD test but rather to enhance the reliability of the binder content assessment through a check method.
- The technique can be used together with historical data available at commercial laboratories in order to determine the long-term correlation between the measured and calculated bitumen content values. This should provide an indication of the quality of the asphalt testing undertaken at a particular laboratory.

REFERENCES:

1) American Standard Test Methods (ASTM), Annual Book of Standards, Volume 04.03, USA, 1998
2) Green, G., South African Shell Bitumen Handbook, Shell, 1991
3) Committee for State Road Authorities (CSRA), TMH1: Standard Test Methods for Road Building Materials, South Africa, 1990
4) Committee of Land Transport Officials (COLTO), Standard Specifications for Road and Bridge Works, South Africa, 1998

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