PROFICIENCY TESTING SCHEMES AND ROUND ROBIN TESTS IN DENMARK

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Abstract:

The Danish Road Institute, DRI, which is the national laboratory for road materials covering soils, aggregate, unbound materials, asphalt materials and bituminous binders is the Danish member of the FEHRL (Forum of European Highway Research Laboratories). DRI has for many years participated in the AASHTO Materials Reference Laboratory (AMRL) proficiency testing scheme in order to document its level as accredited laboratory for many of the international and Danish test methods available in the road sector. The quality section of the Danish Road Institute started in 1992 to offer the possibility to participate in yearly proficiency testing schemes (PTS) to facilitate to quality assurance activities in the Danish companies in the construction sector (geotechnic, gravel pits, quarries, asphalt plants, bitumen suppliers etc.)

The paper will describe the development of a very ambitious proficiency testing scheme which very soon involved the majority of the Danish companies engaged in unbound granular materials and virtual all the companies dealing with asphalt materials and bituminous binders. The yearly PTS involves three materials in each category being sent out in parallel with the possibility to apply all the test methods in the Danish Road Standards. For the asphalt materials and bituminous binders the PTS already in its second year developed into a full blown yearly round robin test (RR) with three materials in triplicate. This large effort in the Danish road sector has given a valuable input to the documentation of quality control and quality assurance in Denmark. The PTS and RR have also provided an important transition in the last years when European standards were introduced. The impact to adjusting the Danish Road Standards and the introduction of “Binder content by ignition” (EN 12697-39) will also be documented.

Key words:

Proficiency Testing Scheme, Round Robin, unbound granular materials, asphalt concrete, bitumen and bituminous binder
1 INTRODUCTION

As long as there have been measurements there has been a need for proficiency testing schemes and round robin tests. But the need has perhaps not been recognised. In historic perspective this need could lie dormant for many years. Goods were in the beginning only sold locally, so the items were all measured once by one person using one measuring tool. Problems could be revealed if that person for some reasons measured an item twice and recognised a difference in the two results. Even this could be put aside, as an error or peculiarity. But when both customer and supplier compared their own measuring results as the measuring tools improved in quality these deviations became apparent. People were forced to come up with procedures how to deal with this phenomenon that could have an impact on whether or not a product was acceptable. These issues are very tight connected to the development of the statistical awareness and insight – in reference to this presentation – in the Danish road sector.

Already in the early 1970s simple statistics was introduced in the Danish Road Standards for instance for control of compaction. The specified value was given as a confidence interval where the mean value of test results from samples was calculated; even with a factor from something called a t-Test to take into account the number of samples.

Later came the trends in EUROLAB with laboratory accreditation (EN 45001 which later transformed into ISO 17025), and quality assurance (ISO 9000) that slowly entered the Danish Road sector.

2 INITIATION AND INSPIRATION

Danish Road Institute is a part of the Danish national road administration and holds several sections dealing with accredited laboratory activities (both for control purposes and research and development and an independent quality section).

Density measurement by probes using radioactive isotopes has been known for decades, but in the 1980s a new generation of probes which were handy and easy to carry and use were introduced on the Danish market. Their potential for quick access to control data even when the unbound materials were compacted during road construction flagged a lot of interest. But to introduce this technique as an alternative in the Road Standards without thoroughly checking both the methodology and any changes in control criteria against the proven method was out of the question.

To provide this necessary background information and documentation for the Road Standards Organisation a large comparison exercise was planed by Danish Road Institute. This plan involved several jobsites and laboratories and therefore also a variety of personal and equipment. When the results were gathered some cases showed problems in getting coherent results. A small literature search was performed to see if similar experience was found elsewhere.

One hit from UK was right on the spot and it also showed a way to deal with this problem of incoherent test results. The Road Research Laboratory had issued a report [1] which addressed the reproducibility of test results of soils classification and compaction test. P.T. Sherwood highlighted through a major round robin experiment the importance to identify outliers which could either be attributed to faulty equipment or wrong interpretation of the laboratory procedure.
P. Ahrentzen bridged the gap between the soil and granular material laboratory and the quality section of Danish Road Institute (DRI). He initiated in 1993 the first proficiency testing scheme (PTS) for unbound materials and aggregates in Denmark. The laboratory had at that time as an accredited laboratory and part of its quality assurance system taken part the AASHTO Materials Reference Laboratory (AMRL) proficiency testing scheme. By this activity and being the focal point of a national proficiency testing scheme Danish Road Institute could provide the Danish road sector traceability to an even larger laboratory community. Many of the methods were then either identical or quite similar to the American test methods.

3 PTS FOR SOIL, AGGREGATE AND UNBOUND MATERIAL

Since 1993 a very ambitious PTS has been running every year which just after a few years had a very high number of participants when you take into account the size of the country and the road sector. The Danish geology plays also a small role in this. We have virtual no mining industry and very little natural rock as the Danish underground predominantly consists of glacial deposits. For this reason we have only a few quarries but a lot of large and small gravel pits. The participants (approx. 75) come from different areas of the Danish road construction sector. This involves geotechnical laboratories and consultancies, road administrations, producers from gravel pits and quarries and customers like cement and asphalt companies. The PTS is set up in accordance with the ISO/IEC Guide 43 [2] which describes the proper steps for organizing a proficiency testing schemes or round robin tests. The identity of the participants is only known by two persons in the quality section and they hold the code by which the laboratories are mentioned by in the yearly report of the results of the PTS [3, 4]. Since 2000 the PTS has been recognised by EPTIS, a database for PTSs in Europe (http://www.eptis.bam.de).

The laboratories receive the necessary material to perform the test method once. In order to provide the laboratories an estimate of the “standard deviation within the laboratory” Danish Road Institute performs the five times (later reduced to three times) where the individual test results are given in the report. Like the AMRL proficiency testing scheme and [2] the Danish Road Institute uses the Z-score and class-division for evaluating the results. The Z-score is calculated from the following equation:

$$Z = \frac{x_i - x_{\text{global}}}{\sigma}$$  \hspace{1cm} (1)

where

- $x_i$ = individual laboratory result
- $x_{\text{global}}$ = global mean of all participating laboratories
- $\sigma$ = standard deviation for all the laboratories

Popular speaking the distance from the result of an individual laboratory to the global mean value obtained is evaluated against the standard deviation of all the participating laboratories. For simplicity in reporting the Z-score is converted into a class number.
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± 5 means that the laboratory is within ± 1.0 $\sigma$ from $x_{global}$

± 4 means that the laboratory is within ± 1.5 $\sigma$ from $x_{global}$

± 3 means that the laboratory is within ± 2.0 $\sigma$ from $x_{global}$

± 2 means that the laboratory is within ± 2.5 $\sigma$ from $x_{global}$

± 1 means that the laboratory is within ± 3.0 $\sigma$ from $x_{global}$

± 0 means that the laboratory is beyond ± 3.0 $\sigma$ from $x_{global}$

The sign shows if your result is above or below $x_{global}$.

Class number 5, 4 and 3 are acceptable; 2 and 1 is questionable and 0 is unacceptable. The simple score system identifies very clear to the participating laboratories their position which enables them to immediate action if necessary. It is our opinion that this together with a speedily reporting (within 2 weeks after deadline of test results) have contributed to the widespread recognition in the road sector of our PTS.

Apart from the high number of participants the word ambitious also covers the scope of the PTS. It is a rather long list of test methods covering EN 932-3, EN 933 part 1, 3, 4, 5 and 8, EN 13286-5, EN 1097 part 2 and 6 and eleven Danish test methods.

4 PTS FOR ASPHALT AND BITUMINOUS MATERIALS

The positive response from the road sector towards the initial PTS for soil and unbound granular material in 1993 was quickly followed up by a similar scheme for asphalt contractors and bitumen suppliers. The set up was again a proficiency testing scheme covering the test methods which were used for control purposes in the Danish Road Standards for asphalt materials.

The PTS for asphalt materials involved from the start tests on two different asphalt materials:

- a dense graded asphalt concrete with 8 mm as maximum aggregate size which throughout the years has been used as reference material because it shows no problems in sampling (meaning if you have problems analysing this material you are really having problems!)
- The other material was initially selected as a material representing a more troublesome material with respect to handling.

The asphalt materials were laboratory produced samples with the intention to achieve good control of the composition.

Again the PTS only provided materials for one analysis per material with Danish Road Institute performing the analysis five times to estimate the “within laboratory standard deviation”. Example of test methods are EN 1426, EN 1427, EN 13702-2, EN 12607-1, EN 13398, EN 12697 part 2, 3, 5, 22, 33 and 34 and several Danish test methods.

5 The SET UP OF PTS IS CHANGED

The evaluation of the first PTS on asphalt materials gave some discussion on certain fundamental points. The asphalt contractors pointed out that the aim of producing the asphalt in the laboratory in order to control the homogeneity had shown an adverse effect because the numbers of batches needed gave some unwanted variation which
could be visible for the “difficult” mix type. At the same time nearly all the asphalt contractors were in the process of preparing for their ISO 9000 certification which meant that they had little use of the five repetitions that the Danish Road Institute performed. They wanted to perform multiple samples themselves so the “within laboratory standard deviation” also would be a parameter for the PTS (which effectively changed it into a Round Robin test, RR).

The following year in 1994 the PTS set up was changed for all the participants to have material for five repetitions on two different asphalt materials produced at during a running production at a full scale asphalt plant. This PTS/RR turned out to be a major exercise for the smaller asphalt contractors’ laboratories to be performed during the quiet winter season. But due to the ISO 9000 commitment of the asphalt industry the contractors volunteered to place the large effort in this program which became an integrated part of the individual companies’ policy for quality assurance program.

It was a firm decision that dense graded asphalt concrete, AC 8d, should be one of the tested materials each time as a kind of reference material. When the set up of the Round Robin analysis had been running for a few years, it was discussed to make some changes after the 1997 report after a proposal from the asphalt industry. It was suggested to change from

- two materials with five repetitions
- three materials with three repetitions.

The statistical argument was that it was only a minor loss in statistical interpretation of the result to reduce the number of repetitions from five to three, but the technical “statements” of the analysis would be greatly improved having three different materials each time for almost the same effort performed in all the laboratories. This new set up has then been used since 1998 for the analysis of asphalt materials.

### 6 BINDER CONTENT BY IGNITION

The members of the Danish asphalt contractors’ association, Asfaltindustrien, made a commitment in an environmental charter in 1999 to reduce the amount of chlorinated hydrocarbons used in the laboratories for binder content determination and binder recovery with 80 % within a five years period. This huge reduction could only be achieved by a shift in method from solvent extraction to something not involving solvents. A method for determining binder content by ignition had been developed and introduced in the laboratories.

The Danish road section was facing a problem if this method which had been developed over a few years wasn’t recognized as a European method alongside the already drafted EN 12697-3 Binder content by extraction. Due to the environmental legislation to substitute for less polluting processes and methods action had to be taken. It was foreseen that the resistance against the binder content by ignition would by three fold

1. a lack of comparison between binder content by ignition and by solvent extraction
2. a lack of knowledge with respect to comparison between extracted aggregate and aggregates after ignition for determining gradation and specific density
3. a lack of knowledge of the precision of the method (repeatability and reproducibility) in accordance with international standards, ISO 5725-2 [5] compared with the known methods it was going to substitute.

With the experience gained through the yearly PTS and RR analysis from the Danish Road Institute and the commitment to the environmental charter it was easy to get the road section to sign up to a series of additional Round Robin tests to provided the necessary documentation as quickly as possible in order to influence the European scene on new test methods. The asphalt materials chosen were selectively materials which would be seen as troublesome materials due to

1. gravel aggregate that might crack and deteriorate during the high temperature and therefore influence the gradation curve
2. polymer modified binders which some solvent extraction methods would see as problematic
3. content of cellulose fibre (in Stone Mastic Asphalt)
4. hydrated lime which could gain weight during ignition due to CO₂ absorption

The results of these tests were documented the Danish method for binder content by ignition in a paper at the ISAP conference in Copenhagen 2002 [6]. It showed the same or even sometimes better precision than binder content by solvent extraction. This documentation and additional input from the annual RRs together with a draft method went to the CEN TC227 WG1 TG2 which was the drafting group for new test methods. It is believed in Denmark that this input due to the skill that the Danish road sector had gained throughout the years and the high focus on quality assurance was a major factor for the development of EN 12697-39 Binder content by ignition [7] and that it can be used in quality control and as documentation between producer and client of asphalt materials.

The commitment made by the asphalt contractors was indeed reached. Already after four years a reduction in chlorinated hydrocarbons of 77 % was a fact [8].

7 THE IMPACT FROM CEN

In the late 1990s the yearly PTS/RR analysis coordinated by the Danish Road Institute became instrumental in the introduction of new European test methods for all materials concerned. Each year an assessment was made into which new test equipments were purchased in the Danish laboratories, so the next PTS/RR could incorporate the new methods in parallel to the “old”, known method which was bound to be squeezed out eventually. In this way the laboratories could use the exercise as a documentation of the new laboratory procedures and check if the same precision level as written in the standards could be reached. At the same time the laboratories could provide input to the mirror committee commenting on the European proposals for test methods.

The assessment of the results of the yearly RR test for asphalt and bituminous materials was still done by Z-score and Class number divisions, but from time to time the huge amount of data was statistically evaluated in accordance with ISO 5725-2 to demonstrate the level of achievement in Denmark.

The yearly RR analysis on bituminous materials was only incorporating penetration and softening point ring and ball. For this reason S 293, the Danish mirror committee on bitumen and bituminous binders, coordinated several RR analysis focusing specific on introduction of the new European test methods on paving grade bitumen
and polymer modified bitumen. For these RRs the assessment was done in accordance with ISO 5725-2. There were some use in Denmark for polymer modified binders (PMBs) with high softening point (> 75 °C) and S 293 recognised the fact that the European test method for softening point did not contain a precision statement for this kind of bituminous binders. The committee coordinated in two successive years RR analysis where five different binders of this nature. It is of course difficult to embrace all kinds of PMBs and to have a proper weight between results from different materials in a RR to arrive at a sound precision statement, but we are proud that the result is mentioned in a foot note in the precision paragraph of the revised EN1427 for softening point ring and ball.

8 IMPACT OF THE PTS / RRs

A report [9] is right now being drafted that will try to address whether or not the yearly PTS and the RR have had a influence and positive effect on the variability in the Danish laboratories. The initial conclusion from this report is in general a positive impact.

9 CLOSING REMARKS

The Danish Road Institute has as earlier mentioned participated in the American AMRL Proficiency Testing Scheme in order to document its own laboratory level of competence but also to provide the Danish road sector a focal point for traceability of certain key test methods. With the forth coming European test methods the status of AMRL’s PTS is challenged in Europe, as some deviations are evident between the American and European variant of analogous methods. For that reasons Danish Road Institute has reached out to some institutes in neighbouring countries where we more or less share the same language in order to establish common ground for PTS/RR on regional level based on our long tradition and experience. We hope to broaden our yearly PTS/RR without endangering the national support, but on some specific European methods the number of participating laboratories in Denmark is small, which is a good reason to broaden the horizon for our PTS.

REFERENCES

[5] ISO 5725-2: Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method


