Binder performance and testing

Detection of the identity of bitumen and its modification based on visualization in spider charts

Martin Radenberg, Daniela Breddemann
Ruhr-Universität Bochum, Lehrstuhl Verkehrswegebau

Abstract

Bitumen shows variations in its material characteristics, depending on the crude oil, its type of production and treatment until use. Moreover, there are complex modifications of bitumen, which detection requires highly elaborated test procedures. In order to realize consistent performance properties of the asphalt, it is of high importance for an asphalt mixing plant to ensure bitumen deliveries with constant material characteristics. This might be possible using complex assay methods; however, the effort appears to be inappropriate for a fast verification of binder properties. So far, rheological characterization of bitumen and modified bitumen, using the dynamic shear rheometer, appears to provide the most comprehensive material description, while simultaneously offering an acceptable effort of quality assurance. The aim of the research project financed by the German Asphalt Association was to develop a three-stage investigation program and to evaluate the identification potential of these three methods. For this purpose, bitumen was examined in virgin and in a simulated long-term aging condition. In order to ensure the least possible effort and a sufficient high aging effect, an extended RTFOT-method (180 minutes at 175 ° C) was used. The tests were carried out on 54 bitumen. The “Simple Approach” examined the possibility of adequate characterization using conventional bitumen characteristics. The “Extended Approach” used the DSR (temperature: 30 to 90 ° C) to derive suitable characteristics of bitumen. Again, a simple and fast test system for this approach is sought. The “Complex Approach”, extended the test system in the DSR by realizing also characteristic values for the low-temperature in addition to the rheological characteristics in the higher temperature range (30 to 90 ° C). For this approach, only one test procedure was conducted to decrease time and effort. In order to evaluate the usefulness of the identification method binder characteristics were visualized in spider charts.

This paper was first published in 2020 in version 1.0 of the proceedings of the 7th E&E Congress. This second edition (version 1.1.) of the proceedings is published in June 2021. The content of this paper has not changed, but the author(s) may have provided additional information in the form of an annex.

1. INTRODUCTION AND OBJECTIVES

The binder is an essential determining factor in ensuring that asphalt performance properties are as uniformal as possible. For the quality assurance of asphalt production, therefore there must be a great deal of interest in deliveries of binder with material characteristics as constant as possible. With simple test methods (softening point ring and ball or needle penetration) alone, an incoming goods inspection may be considered questionable. With complex test methods, including chemical analysis if necessary, this is possible, but certainly unsuitable for a quick examination of the binder properties. The rheological characterization of bitumen with a DSR provides, according to the current state of knowledge, a more comprehensive material description, which possibly offers an approach to quality assurance that is still justifiable in terms of expenditure.
The investigation carried out on behalf of the DAI (Deutsches Asphalt Institut) had the aim of clearly detecting the identity of bitumen and visualizing the appropriate test results in such a way as to enable reliable quality monitoring. As a result, the expenditure had to be kept as low as possible (especially in terms of time).

2. METHODOLOGY, SAMPLE SELECTION AND SAMPLE PROCUREMENT

In terms of a variable design of the investigation program with regard to effort and significance of the results, a three-stage test program was chosen. For all three research approaches, a comparison of the characteristic data in both fresh and aged state was deemed necessary. In order to limit the effort and equipment requirements here as well, preliminary investigations were carried out to find a RTFOT ageing level that corresponds approximately to that of combined ageing with RTFOT and PAV. The study´s programme was thus divided into a simple approach, an extended approach and a complex approach to fresh and aged binders as well. The complex approach is not part of this article.
With the **simple approach,** the possibility of an adequate characterization was examined on the basis of the two characteristic binder properties softening point ring and ball and needle penetration [1, 2]. With the **extended approach**, the binders were characterized in the DSR using a temperature sweep at an oscillation frequency of 1.59 Hz [3].
In agreement with the client, it was decided to examine five types of binder (see Table 1).

Table 1: Selected binder types

bitumen normal paving grades (NPG)	**PmB**	**wax modified bitumen**
50/70 (9/3 delivery points)	**25/55-55 A** (12/4 delivery points)	**25/35 V** (9/3 delivery points)
160/220 (9/3 delivery points)	**45/80-50 A RC** (12/4 delivery points)	

Per binder type and per delivery point three samples were taken over an agreed period of time. The regional spread of the three/four delivery points ensured that different production sites/delivery points were taken into account, which means that different crude oil proveniences can also be assumed. A total of 51 binders in fresh and aged condition were tested using the three test approaches.

3. RESULTS

3.1 Laboratory aging method

In order to detect a difference in binder-performance of the same grade, it is advisable to carry out a supplementary analysis on the basis of simulated ageing regimes. Aging with the RTFOT [4] essentially represents the simulated stress during asphalt production (from hot mixing until the end of compaction). Further changes caused by ageing processes during the course of the service life are usually simulated in the laboratory by an additional ageing stage as with the PAV [5]. According to the current state of knowledge, this seems to result in a greater differentiability of the binder properties with increasing ageing, so that an expansion of the stress beyond the extent of RTFOT ageing was considered necessary for quality-monitoring investigations. In the sense of both limiting the necessary technical equipment and time expenditure, an extended RTFOT ageing was aimed at in analogy to the findings in [6], which corresponds as far as possible to the degree of ageing of a combined RTFOT+PAV ageing.
Although this prolonged RTFOT ageing is less suitable for scientific considerations due to the presumably higher proportion of distillative ageing, the aim of a suitable differentiability for quality monitoring is likely to be given. The extent to which a moderate increase in temperature (e.g. to 175 °C) would enable a reduction in the ageing time and at the same time a more reasonable temperature for higher-viscosity binders (PmB) was initially investigated experimentally in an upstream work step.
Comparisons between results from RTFOT+PAV ageing and variations in the RTFO regime led to an extended RTFOT ageing of 180 minutes at 175 °C .

3.2 Results with the simple approach

The possibility of a useful detection of the binder deliveries on the basis of the conventional test results softening point ring and ball and needle penetration was first investigated with the typical representation of these results on a semi-logarithmic scale (Fig. 1). The spread of data points (before and after ageing) was supplemented by the course of the penetration indices -1, 0 and 1.
Characteristics of the 51 binder samples before and after ageing show that the bitumen 160/220 shifts to a constant or even decreasing penetration index after ageing, while that of the bitumen 50/70 predominantly increase. Compared to normal paving grades unaged modified bitumen usually have a higher penetration index, which then usually increases as a result of aging.
In order to illustrate the individual changes caused by the ageing process, the relative changes in needle penetration and in softening point ring and ball were plotted (Fig. 2).

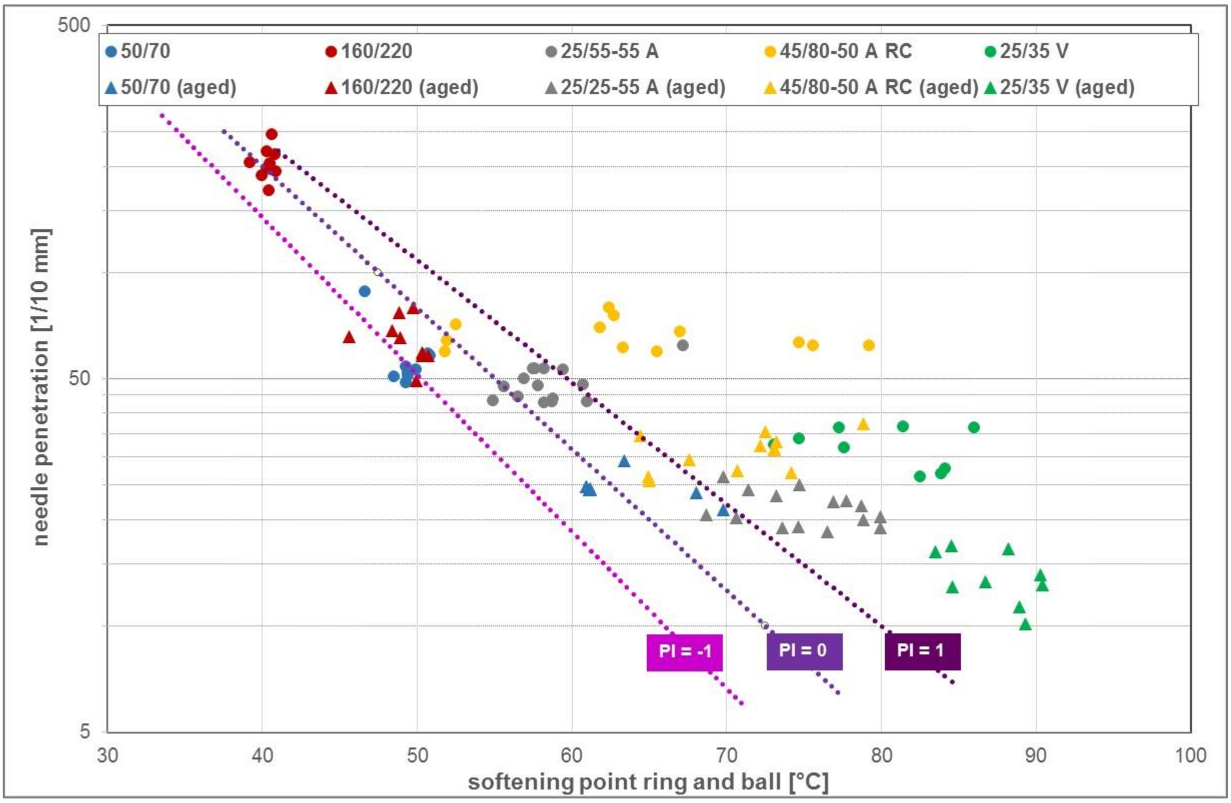

Figure 1: Softening point ring and ball and needle penetration, 51 samples (unaged and aged)

While the retained needle penetrations decrease to between 50 and 70 % for most of the binders investigated, the relative changes in the softening point ring and ball increase and are much more pronounced (from -10 % to +45 %). This results in groups with comparable changes, some of them allow separation into the points of delivery. Two points of delivery for PmB 25/55-55 A and one for both 50/70 and 45/80-50 A RC can therefore be separated from the rest of the data collective (identification).
For many delivery points, such as bitumen 160/220 and 50/70, sufficient differentiation hardly is possible. Even with taking the penetration index into account, which in the end is only a calculated value from the two test results, differentiability does not satisfy.

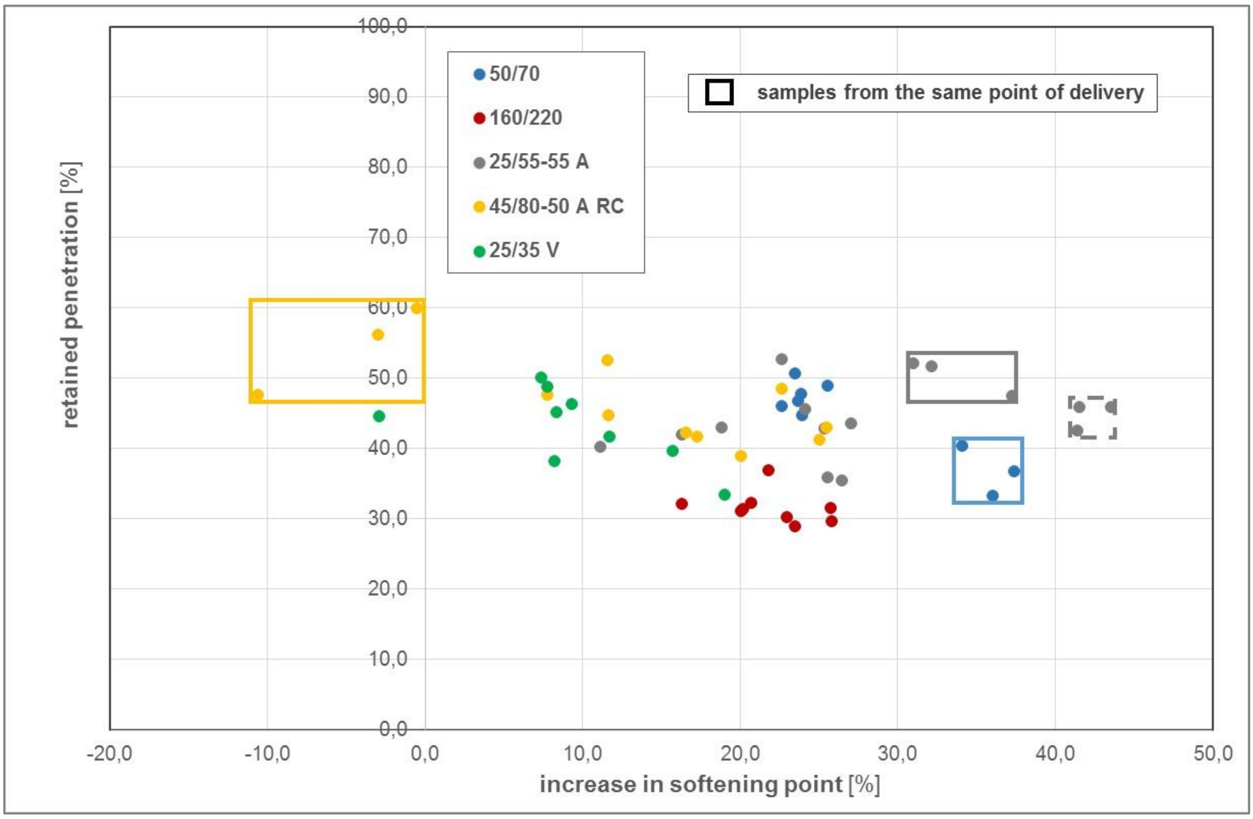

Figure 2: Change of penetration and softening point caused by aging

3.3 Results with the extended approach

The method for a rheological description of bitumen for the extended investigation approach is already widely used today [3]. The typical representation of the test results from this kind of investigations is the temperature-dependence of the complex shear modulus (semi-logarithmic) and the phase angle (linear). For the examined PmB 25/55-55 A, it can be seen in Fig. 3 and 4.

The differences between the complex shear moduli of unaged PmB 25/55-55 A are very small over the three different delivery points (range of equistiffness temperature at G*=15k Pa: 52.8 to 56.3 °C). After aging the complex shear moduli have a slightly larger span. Greater differences can be seen in the phase angle, especially when the change due to ageing is considered.

To achieve the best possible visualization of difference in rheological properties, the complex shear moduli and the phase angles (absolute values and changes caused by ageing) were subjected to a comprehensive analysis in order to identify suitable characteristic values.

A characteristic value [3], already common in Germany, the equilibrium temperature at a complex shear modulus (G*) of 15 kPa (T(G*15 kPa)) was defined as a measure of the "hardness" of the binder (both unaged and aged). This property has the potential to replace the softening point ring and ball as well as the needle penetration in the future.

In the search for further characteristic values or identifying rheological parameters, the focus was on three values where the phase angle is of dominant importance and an individual temperature range is addressed.

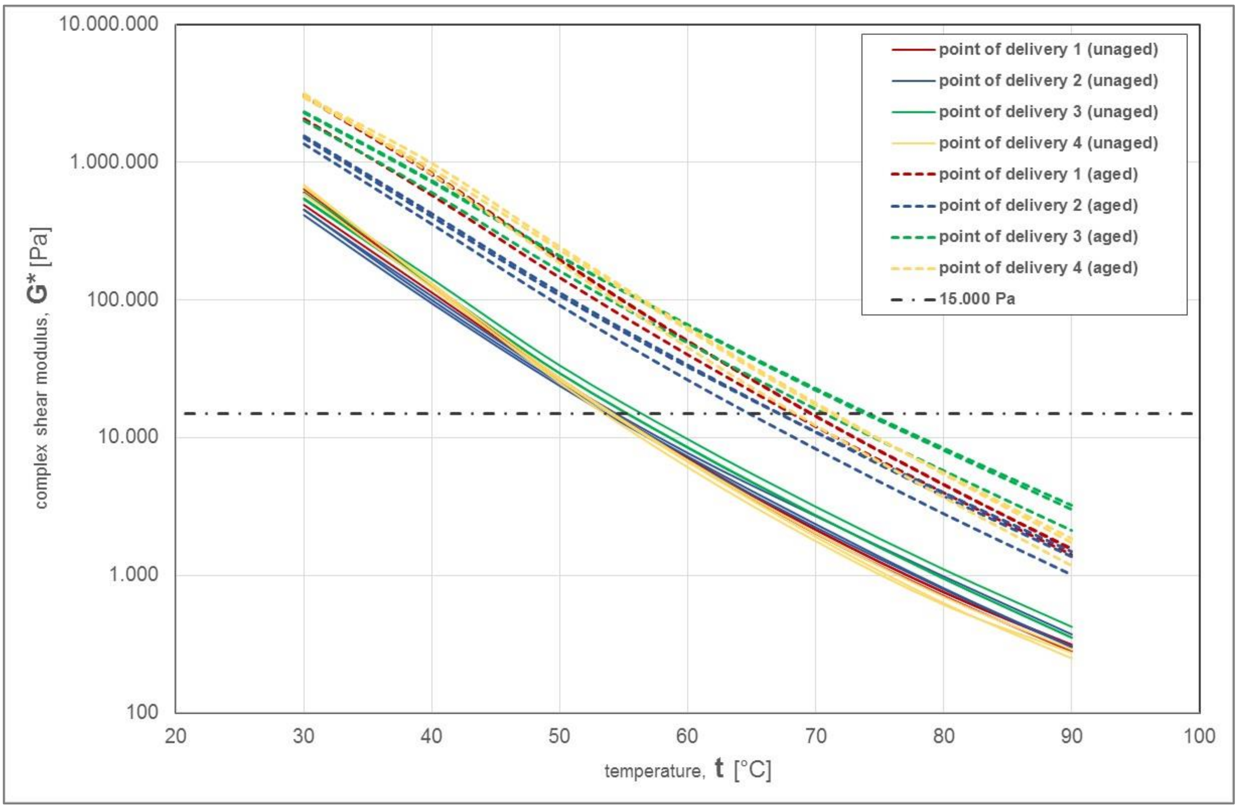

Figure 3: Complex shear modulus of PmB 25/55-55 A, unaged and aged

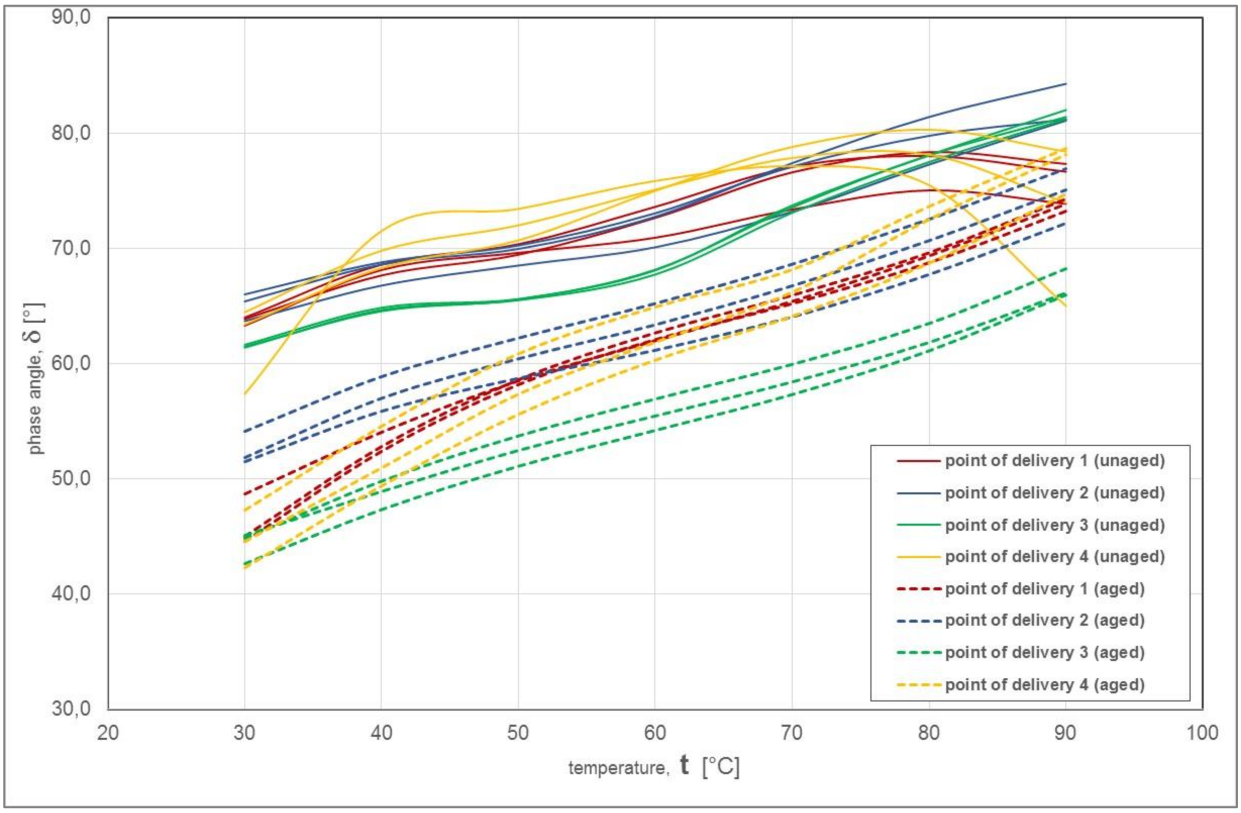

Figure 4: Phase angle of PmB 25/55-55 A, unaged and aged

Characteristic value 1: Relative storage modulus at equilibrium temperature, **relG'**(G*15 kPa)
The storage modulus is used to quantify the elastic stiffness fraction of the complex shear modulus. For the calculation, the complex shear modulus is multiplied by the cosine of the phase angle.

$$\mathbf{G'} = G^{*} \cdot \cos \delta \qquad [Pa]$$

This calculation is constant for all binders at G* = 15 kPa. The variable size is then the phase angle. The result, the storage modulus at this equilibrium temperature, is then divided by this temperature.

$$\mathbf{relG}'(G^{*}15\ \mathrm{kPa}) = \frac{\mathbf{G}'(G^{*}15\ \mathrm{kPa})}{\mathbf{T}(G^{*}15\ \mathrm{kPa})} \qquad [\mathrm{Pa/°C}]$$

The relative storage moduli at the equilibrium temperature were thus calculated for all the binders investigated before and after ageing. Fig. 5 shows the differences of the binders (different shapes marks for the type of binder, different colours for the points of delivery).
This figure clearly shows that the type of binder (modification) can be separated and detected. The bitumen, the polymer modified bitumen and the wax modified bitumen form clearly separated groups each. In addition, however, in many cases a very definite group formation of the individual delivery points can also be observed (by the colour of symbols).

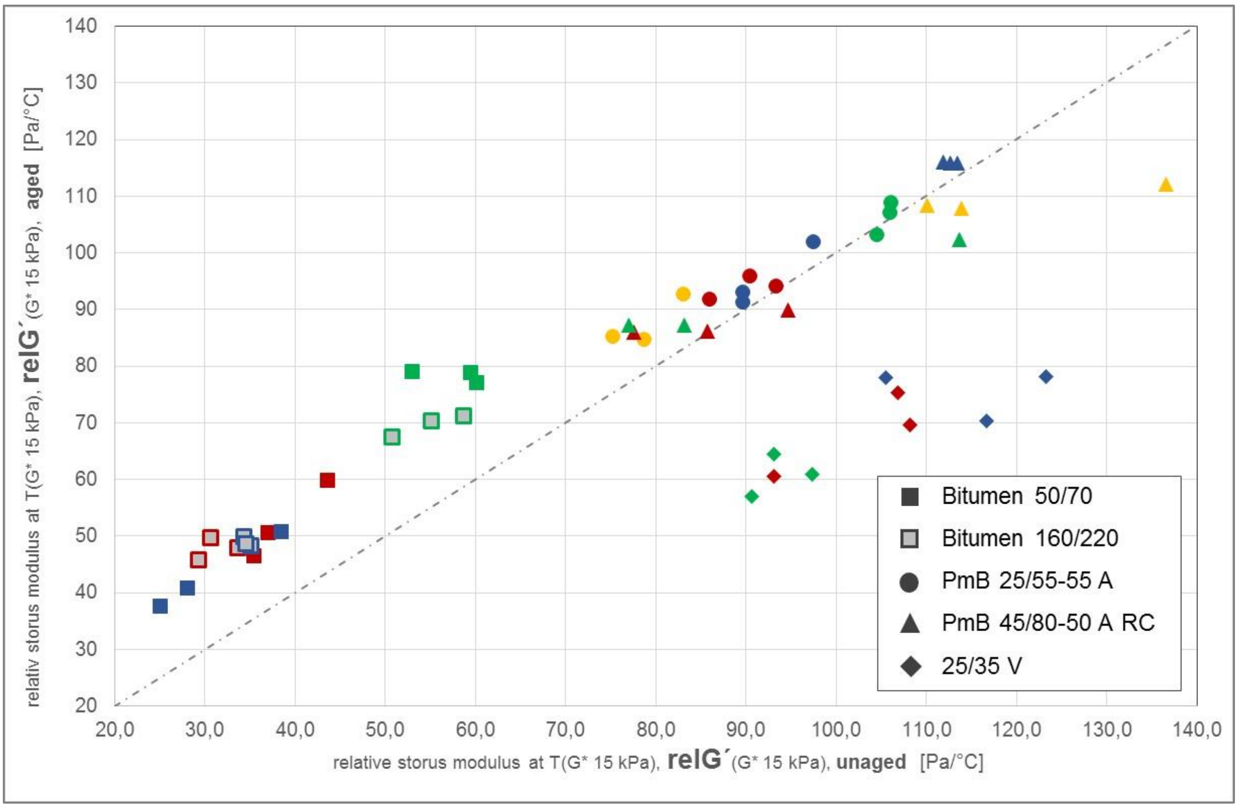

Figure 5: Relative storage modulus at T(G* = 15 kPa), unaged and aged

Characteristic value 2: Cross-over index at 30 °C, **COI30**
The mechanical properties of bitumen show a temperature-variable ratio between elastic and viscous stiffness (storage modulus/loss modulus). This temperature-dependent ratio is quantified by the phase angle. At a phase angle of 45°, the storage modulus (elastic stiffness component) and the loss modulus (viscous stiffness component) are identical (G' = G"). This marks the so called cross-over point and defines a temperature point. In the used temperature range (30 to 90 °C), some of the examined binders only showed a phase angle of less than 45° in the aged state. Against this background, a ratio was considered (loss modulus to storage modulus at 30 °C) to express the proximity to the cross-over point. This ratio is referred as the cross-over index at 30 °C (**COI30**).

$$\mathrm{COI30} = \frac{G''_{(30\,°C)}}{G'_{(30\,°C)}} \cdot 100 \qquad [-]$$

For a COI30 of 100 the phase angle at 30 °C is exactly 45°. A COI30 above 100 has a phase angle at 30 °C of more than 45°, while a COI30 below 100 has a phase angle at 30 °C of less than 45°.
The COI30 thus defines the viscous potential of the binder at the lowest temperature of the measured spectrum, which can be expected to provide a statement by trend on the behaviour at lower temperatures.
Fig. 6 shows the COI30 before and after ageing for all binders investigated. The associated binders (delivery points) are in turn identified by corresponding colours of the symbols.

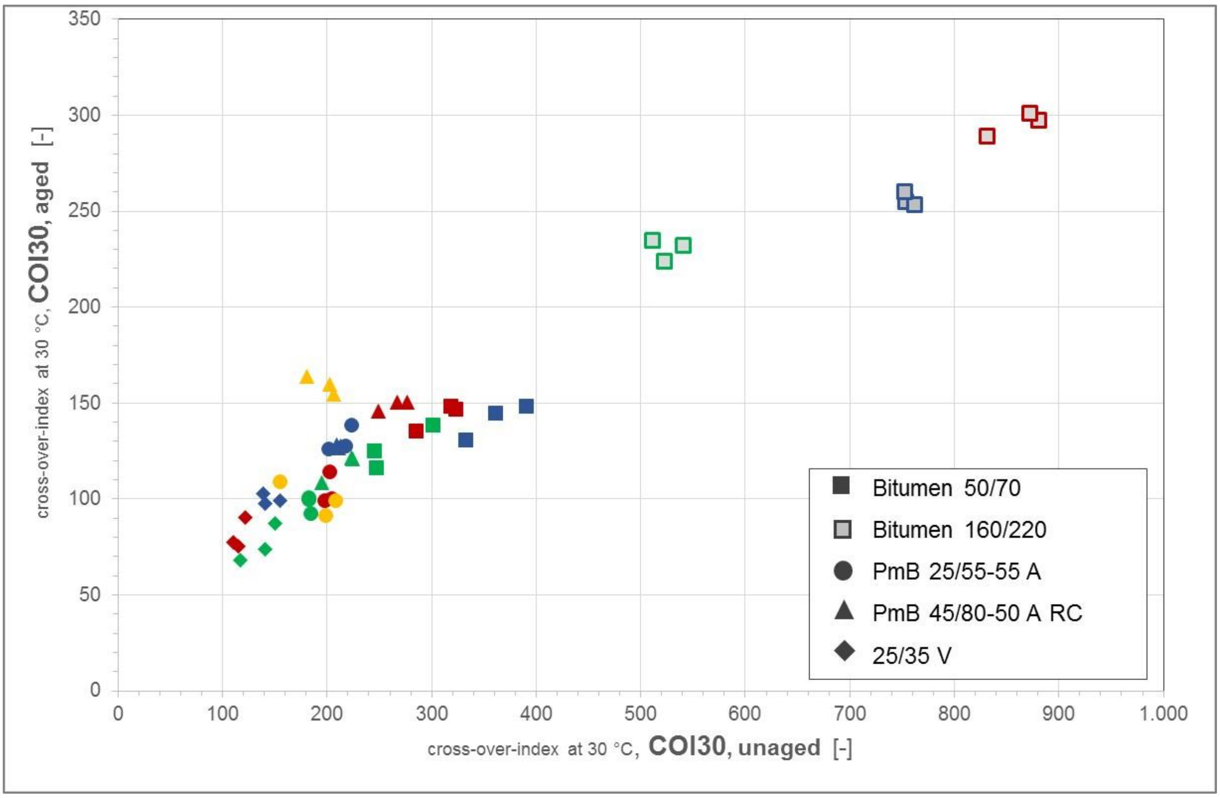

Figure 6: Cross-over-index at 30 °C, unaged and aged

While the relative storage modulus at equilibrium temperature essentially separates the type of modification and the change due to ageing, the COI30 allows additional differentiation of the "binder hardness". The 160/220 bitumen can thus be very clearly differentiated from the other binders as well as into the individual delivery points. For some other types of binder (45/80-50 A RC, 50/70 and 25/35 V), individual delivery points are also obvious.

Characteristic value 3: Change of phase angle between 50 and 40 °C, δ_{50}-$\Delta\delta_{40}$
The temperature-dependent development of the phase angle shows a partially characteristic gradient in visual response (see figure 5) between 50 and 40 °C, especially with modified binders. It often changes as a result of the ageing process. Against this background, the change of the phase angle gradient between 50 and 40 °C due to ageing was included in the evaluation as a further characteristic value.

$$\Delta\delta_{50}\text{-}\delta_{40} = (\delta_{50\,°C,aged} - \delta_{40\,°C,aged}) - (\delta_{50\,°C,fresh} - \delta_{40\,°C,fresh}) \quad [°]$$

With this characteristic value before and after ageing, a very clear classification of the source of supply of the binder can be detected (see Fig. 7). So there is a third characteristic value to be expected from the temperature range between the two other characteristic values.

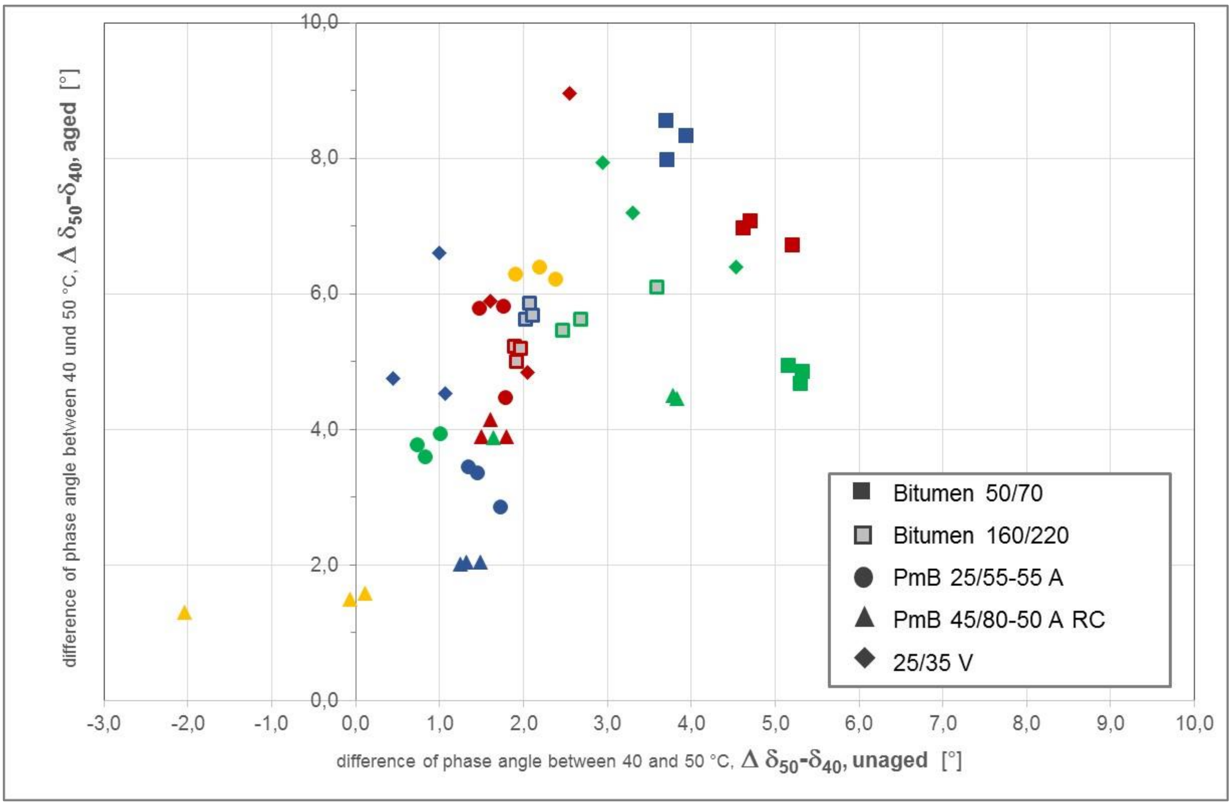

Figure 7: Difference of phase angle between 40 and 50 °C, unaged and aged

4. POSSIBILITIES OF IDENTIFICATION WITH SPIDER CHARTS

With the investigation for the identification of bituminous binders it could be shown that with the conventional investigation methods (simple approach) a viable, but not yet satisfactory approach is available. In principle, for such identification investigations of the binders before and after an extended RTFOT ageing (or RTFOT+PAV) are considered necessary.
The extended investigation approach, based on the results of a temperature sweep in the DSR following the AL DSR test (T-Sweep) [3], provides more comprehensive rheological characteristic values in the higher and high service temperature range. In Germany results from this test have to be provided as part of the additional binder testing according to [7, 8, 9, 10] since 2013, but so far only for unaged bitumen. In the future, it is likely that these regular examinations will also be carried out in the aged state, which would allow an immediate transfer of the visualization proposed here for identification.
For the best possible identification of the binders examined this way, a six-axis representation (hexagon) with the following characteristic values is proposed:

Table 2: Proposed six-axis representation (hexagon) for visualization

Characteristic values in virgin state	1. **T**(G*15 kPa), unaged 2. **relG'**(G*15 kPa), unaged 3. **COI30**, unaged	[°C] [Pa/°C] [-]
Characteristic value in aged state	4. **relG'**(G*15 kPa), aged	[Pa/°C]
Changes due to ageing	5. **ΔT**(G*15 kPa) 6. $\Delta\delta_{50}$-δ_{40}	[°C] [°]

4.1 Identification of origin

For an identification of origin for the same binder grade, a scaling adapted to this grade is recommended. The example given in Fig. 8 is adjusted to a bitumen 50/70 with regard to scaling. The typical and therefore proposed range of experience for the variety is highlighted in green, although this range could easily change due to future data additions. The axes have been scaled in a way that for each property the range of experience lies approximately in the middle of the axis. A conditional evaluation has been made for the direction of scaling. For example, the change in the equilibrium temperature due to ageing was reversed in scale, with external measured values probably documenting better quality.

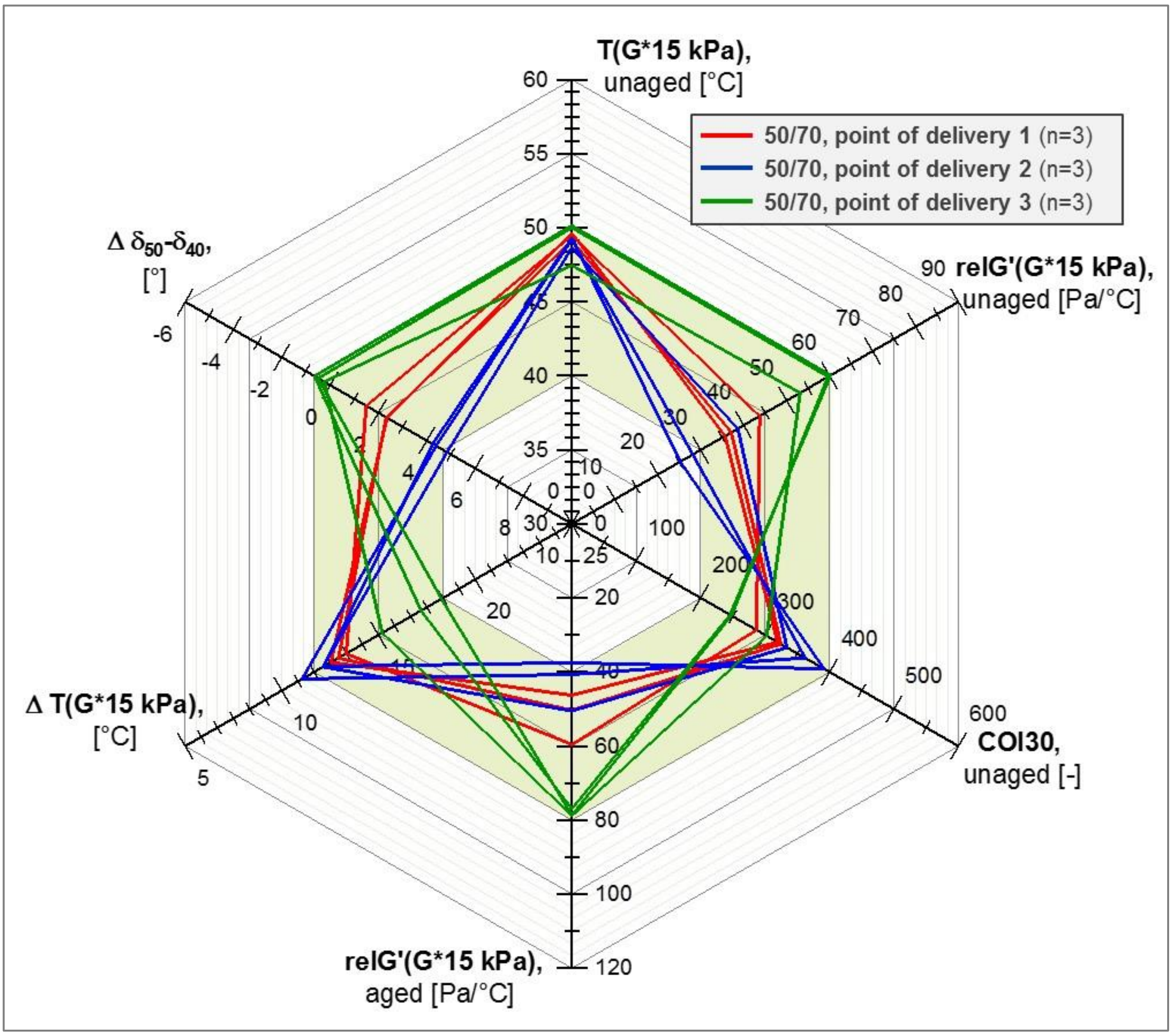

Figure 8: Hexagon for bitumen 50/70 (9 samples, 3 delivery points) and area of experience

The three delivery points, from which three samples were taken at different times, can easily be differentiated, whereby the rheological properties of the bitumen are quite similar for those of delivery points 1 and 2.
To illustrate the high identification potential of the Extended Approach, Fig. 9 compares the results of the bitumen 160/220 from the Extended Approach with the results of the Simple Approach using a four-axis diagram. Especially with the new, strongly differentiating parameters "relative storage modulus at equilibrium temperature" and "cross-over index at 30 °C", the rheological characteristics of the delivery points can now be identified very well.
In addition, it becomes clear that the rheological properties of the three investigated delivery points each exhibit a very high uniformity, although there was a period of several months between the first and the third sampling.

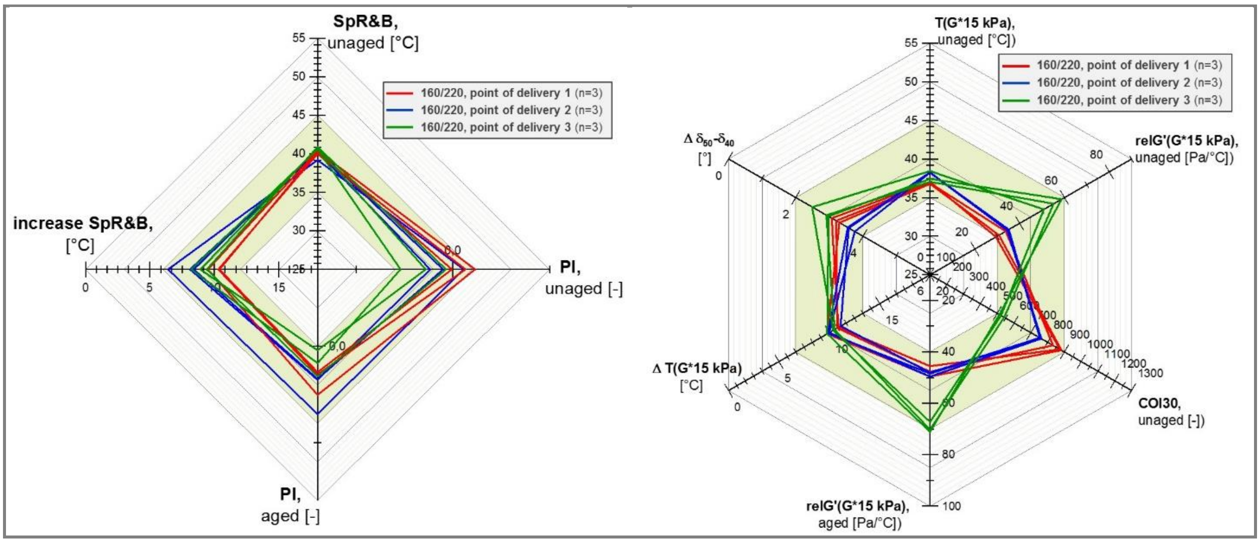

Figure 9: Comparison of the identification potential of the two research approaches (160/220 each) with areas of experience

4.2 Identification of modification

To illustrate the effect of additives (elastomers and waxes) on the selected characteristic values, Fig. 10 and 11 show the range of experience for a bitumen 50/70 and typical values for polymer-modified bitumen (Fig. 10) and wax-modified bitumen (Fig. 11).

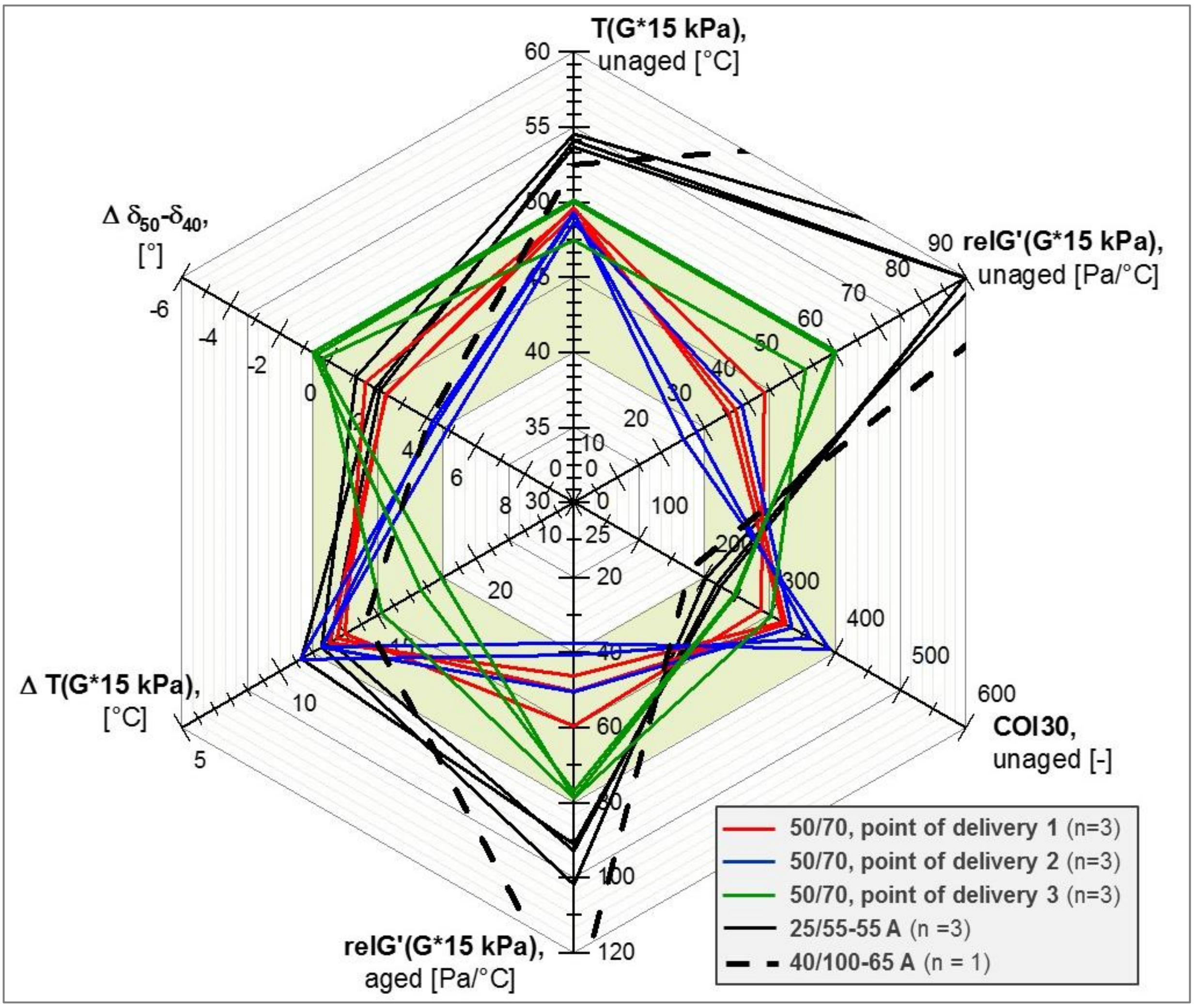

Figure 10: Example of the differentiation potential of bitumen 50/70 with polymer-modified bitumen

In the case of polymer-modified bitumen, different degrees of modification (standard: 25/55-55 A and higher modified: 40/100-65 A) have been considered. The needle penetration of all polymer modified bitumen considered here can all be found within the requirement range for a bitumen 50/70. The comparison of the binders in the hexagon clearly shows that modification with elastomers has a strong influence on the relative storage modulus at the equilibrium temperature (T(G*15 kPa)) before and after ageing. There are also indications that this value increases with the degree of modification, since the relative memory module of the higher modified bitumen (40/100-65 A) is even higher than that of the 25/55-55 A. The higher the degree of modification, the higher the memory module. Thus the relative memory module could be a parameter for the degree of modification.
In order to describe the effect of a wax modification, three test results of one wax-modified binder are integrated into the hexagram of bitumen 50/70. Here, however, there is no penetration equivalence compared to 50/70. The needle penetration of the wax-modified bitumen was 35 $^1/_{10}$ mm in the unaged state. Here, too, there is a strong effect (increase) on the relative storage modulus at the equilibrium temperature considered (T(G*15 kPa)), but only in the fresh state. After aging only the level of bitumen 50/70 is reached. Thus, the change in the relative storage modulus at the equilibrium temperature under consideration is obviously a specific property of the wax-modified bitumen. The equilibrium temperature at a G* of 15 kPa is also strongly influenced by the wax modification, even though a bitumen with lower needle penetration than 50 $^1/_{10}$ mm was presumably used as the base here.

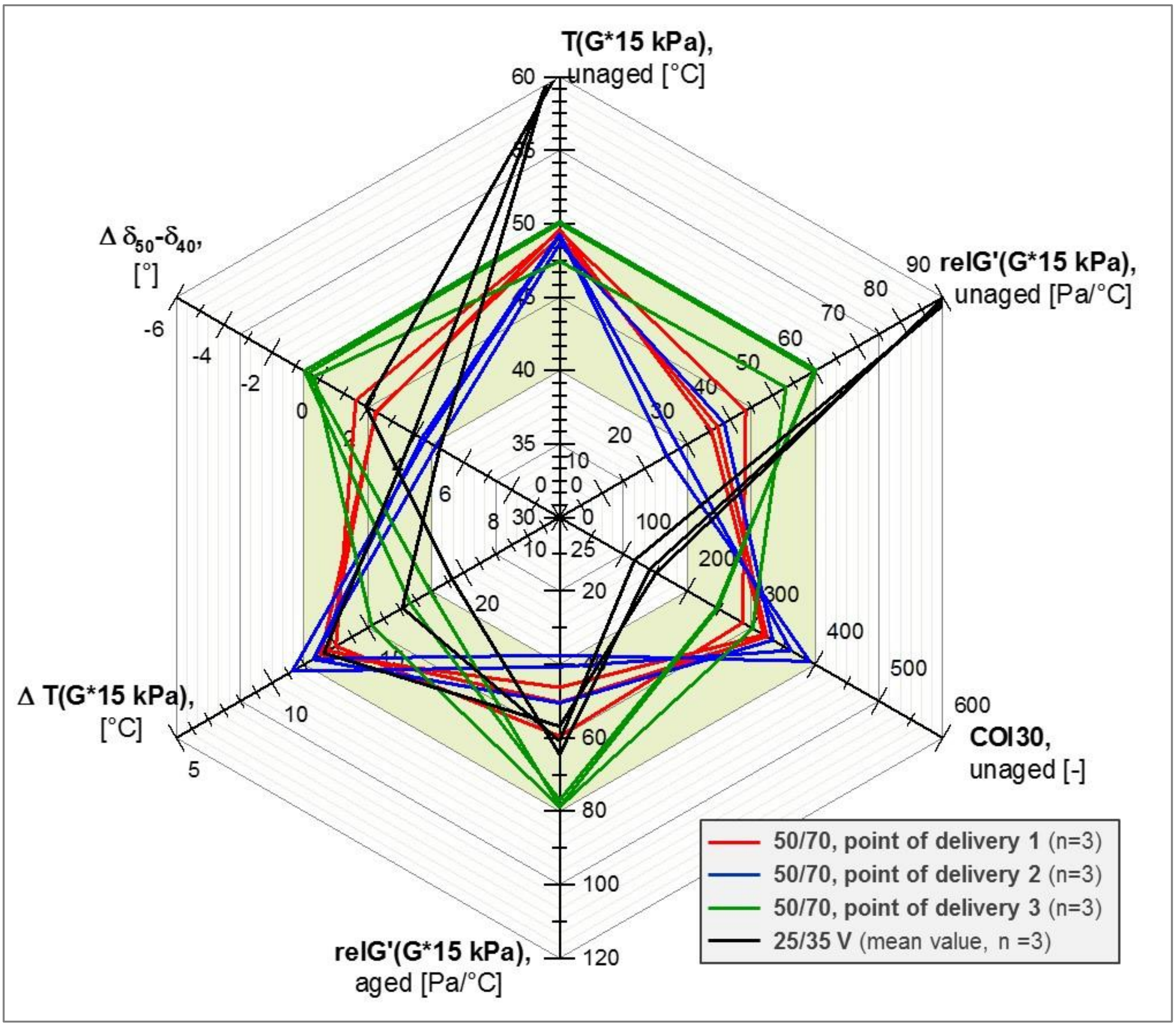

Figure 11: Example of the differentiation potential of bitumen 50/70 compared to a wax-modified bitumen

5. CONCLUSION AND OUTLOOK

The investigations on bitumen and bitumen with different modifications carried out on behalf of the DAI have shown that the identity of bitumen and modified bitumen can clearly be detected by DSR temperature sweeps from 30 °C to 90 °C before and after ageing. It is helpful to visualize the proposed binder parameters in a six-axis diagram (hexagon). The effort required for this investigation is still moderate, so that regular implementation within the framework of quality monitoring is justifiable. Since in Germany such investigations may presumably become relevant for building contracts in 2019, large quantities of data can be expected in the medium term to validate this evaluation approach. In the next step, asphalt performance tests on selected binder samples would then be useful in order to establish a reference to the performance of the binders. Until then, measured values should only be used to identify the binder properties. Additionally it is planned to perform chemical analysis with the FTIR spectroscope in order to identify a correlation between the different rheological properties and material compositions.

6. ACKNOWLEDGEMENTS

This paper is based on the research project carried out at the request of the DAI (Deutsches Asphalt Institut). The authors are solely responsible for the content.

7. BIBLIOGRAPHY & REFERENCES

8. REFERENCES

[1] **DIN EN 1427. 2007:** Bitumen und bitumenhaltige Bindemittel - Bestimmung des Erweichungspunktes - Ring- und Kugel-Verfahren. Berlin : Beuth Verlag, 2007.

[2] **DIN EN 1426. 2007:** Bitumen und bitumenhaltige Bindemittel - Bestimmung der Nadelpenetration. Berlin : Beuth Verlag, 2007

[3] **AL DSR-Prüfung (T-Sweep):** Arbeitsanleitung zur Bestimmung des Verformungsver-haltens von Bitumen und bitumenhaltigen Bindemitteln im Dynamischem Scher-rheometer (DSR) – Durchführung im Temperatursweep, Forschungsgesellschaft für Straßen- und Verkehrswesen e.V. (FGSV) und Arbeitsgruppe „Asphaltbauweisen“, Köln, Ausgabe 2014

[4] **DIN EN 12607-1. 2007:** Bitumen und bitumenhaltige Bindemittel - Bestimmung der Beständigkeit gegen Verhärtung unter Einfluss von Wärme und Luft - Teil 1: RTFOT-Verfahren. Fassung 2014. Berlin : Beuth Verlag, 2007

[5] **DIN EN 14769. 2012:** Bitumen und bitumenhaltige Bindemittel - Beschleunigte Langzeit-Alterung mit einem Druckalterungsbehälter (PAV). Berlin : Beuth Verlag, 2012

[6] **Spiegl, M.**; **Steidl, H.:** Entwicklungen von gebrauchsverhaltensorientierten Bitumenspezifikationen, GESTRATA Bauseminar 2009

[7] **ARS 11/2012:** Allgemeines Rundschreiben Straßenbau Nr. 11/2012 (ARS 11/2012)- Änderungen und Ergänzungen des Technischen Regelwerkes Asphaltstraßen. s.l. : Bundesministerium für Verkehr, Bau und Stadtentwicklung, 2012

[8] **TL Asphalt-StB 07/13**: Technische Lieferbedingungen für Asphaltmischgut für den Bau von Verkehrsflächenbefestigungen, Forschungsgesellschaft für Straßen- und Verkehrswesen e. V., Köln, Ausgabe 2007, Fassung 2013

[9] **TL Bitumen-StB 07/13**: Technische Lieferbedingungen für Asphaltmischgut für den Bau von Verkehrsflächenbefestigungen, Forschungsgesellschaft für Straßen- und Verkehrswesen e. V., Köln, Ausgabe 2007, Fassung 2013

[10] **ZTV Asphalt-StB 07/13**: Zusätzliche Technische Vertragsbedingungen und Richtlinien für den Bau von Fahrbahndecken aus Asphalt, Forschungsgesellschaft für Straßen- und Verkehrswesen e. V., Köln, Ausgabe 2007, Fassung 2013