Temperature Susceptibility of Modified Asphalt Binders

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Abstract. Temperature is considered here as an important measurement affecting the behaviour of asphalt cement. The relationships between temperature and viscosity for ten pure and modified asphalt cements were thus assessed using traditional methods (including penetration, and (R&B) softening point) and dynamic shear rheometer (DSR) and rotational viscometer (RV) tests. The temperature used for testing were (25°C), (120 to 195 °C), and (4 to 70 °C) for traditional, RV, and DSR tests, respectively. The effects of test type, asphalt cement type, modifier type, and content, on temperature-susceptibility were investigated. The results showed that using activation energy ($E_a$) for flow allowed discernment of asphalt cement’s susceptibility to temperature variation, and that the addition of modifiers (SBS, BR, and BG) to asphalt cement increased the $E_a$ indices. Higher increment rates in $E_a$ (23.57%) were caused by the addition of BR, while reduced VTS was seen with the addition 9% BR, and 5% and 7% SBS, with reduction rate of 44.138%, 15.994% and 15.241%, respectively, due to reductions in viscosity changes with variation in temperatures, while the addition of 0.075% BG Plus increased VTS by 2.496% compared with base asphalt binder.

1. Introduction

Asphalt binder properties are one of the key defining features of asphalt mixtures. Asphalt cements even those with similar physical properties such as viscosity, penetration, softening point and performance grade, can exhibit different rheological properties. Hence, it is necessary to characterise the behaviour of asphalt cement to capture their rheology over a wide range of temperatures and load frequencies, especially for modified asphalt binders [1]. The rheological behaviour of asphalt cement varies from completely elastic to highly viscous over various temperature ranges and loading times, and thus, a asphalt cement has a great impact on the properties of the whole mixture despite constituting only a small part of the mix volume [2]. In general, asphalt binder should have enough flexibility to resist thermal stresses without cracking at cold weather in winter, while retaining enough stiffness to resist hot temperatures in summer seasons [3]. Binder modification is a common way to enhance the properties of asphalt cement mixtures and their functionality and to reduce the effects of temperature on asphalt binder performance [4]. A variety of modifiers can be added to bitumen, including Styrene-Butadiene-Styrene Block Copolymer (SBS), Styrene-Butadiene-Rubber (SBR), Polyethylene (PE), Ethyl-Vinyl-Acetate (EVA) and other polymer modifiers; all of these have been used to modify the base bitumen, and they have become the main way to improve the performance of such bitumen. These also have become the main ways to improve the performance of bitumen [5]. Practical applications have
shown that SBS is superior to other polymer modifiers, and it is thus more popular due to its excellent properties, relatively good dispersion and appropriate solubility in bitumen and acceptable cost. It works by improving the low-temperature toughness and temperature sensitivity of the bitumen. As a commonly used modifier, SBS shows outstanding performance. Increasing the softening point and low-temperature ductility significantly. It can also greatly improve temperature sensitivity, and after modification, the elastic recovery rate of such bitumen was particularly large [6].

The use of asphalt binder in Iraq is neat asphalt cement supported by multiple different refineries in the country. The wide variety of climate conditions in the middle and southern areas of Iraq make flexible pavement subject to a considerable variation in surface temperature, from 5°C to more than 60°C, as well as them being subject to increasing stress levels from heavy vehicles moving at low speeds and increasing traffic congestions. Thus, studying the rheological properties of asphalt cement binders over a wide scope of loading times and temperatures is essential to preventing untimely permanent deformation on flexible pavements [7].

In terms of the viscoelastic behaviour of materials, rheological properties play a significant role in the evaluation and selection of paving materials and in the analysis and design processes for asphalt pavements because these properties reflect load-deformation relationships and can be considered crucial in terms of understanding the behaviours of these materials [8].

The rheological properties of asphalt binders at overlong loading times are measured using viscometers while the dynamic shear rheometer is more suitable for short loading times [9, 10].

Asphalt binders and asphalt mixtures properties are also studied by using activation energy (Ea) concepts in which the viscosity is used as measure of the shear resistance of the liquid to flow, which depends directly on the temperature. Increasing the temperature causes an increment in molecular thermal energy, decreasing the resistance to flow which in turn leads to a decrease in the viscosity of fluid. In flowing liquids, the molecules sliding over each other must overcome the intermolecular forces that resist the motion. Ea works as a barrier resisting the viscous flow and must be overstepped to allow flow to happen [9, 10]. The (Ea) concept can be applied to characterise the temperature susceptibility of asphalt binders and thus to differentiate between asphalt binders [1]. Equation (1), known as the Arrhenius equation, can thus be utilised to model the viscosity-temperature dependency in asphalt binders [11].

\[ \eta = A \times e^{-\frac{E_a}{RT}} \]

where, \( \eta \) is the asphalt binders dynamic shear viscosity (pa.s), T is the temperature of test in degrees Kelvin (K), A is a constant, \( (E_a) \) represents the activation energy and R which is the universal constant of gas (8.314 J. mol\(^{-1}\) K\(^{-1}\)).

Equation (1) linearized using the natural logarithm as follows:

\[ \ln \eta = \ln A + \frac{E_a}{RT} \times \ln e \]

\[ \ln e = 1 \]

\[ \ln \eta = \ln A + \frac{E_a}{R} \times \frac{1}{T} \]

\[ y = a + bx \]  

A plot of \( \ln \eta \) (y-axis) versus \((1/T)\) in x-axis) produces a straight line with a slope of \( E_a / R \). This slope and should thus be multiplied by the universal constant of gas in order to estimate\( E_a \).

In this study a number of tests were used to evaluate the thermal susceptibility of ten different types of asphalt cement binders (2 pure and 8 modified):

1- Dynamic Shear Rheometer (DSR) testing to determine the rheological properties of the asphalt binder;
2- Rotational Viscometer (RV) testing using a Brookfield device;
3- Ring and Ball (R&B) softening point testing; and
4- Penetration testing.

2. Research objectives
The main objectives of this research are:
1- Evaluate the thermal susceptibility to asphalt cement binders based on Activation Energy of flow ($E_a$), Penetration Index (PI), and Viscosity-Temperature Susceptibility (VTS).
2- Compare the results obtained from (DSR) and Rotational Viscometer (RV) testing at low, moderate and high degrees of temperature in term of calculating the $E_a$ of asphalt cement binder.
3- Predict various $E_a$ at compaction and mixing temperatures for modified binders to assess their temperature susceptibility.

3. Experimental work
3.1. Materials
Ten different pure and modified asphalt cement were gathered in order to conduct, DSR and RV tests and conventional tests (penetration test, R&B softening point test). The group of asphalt cement was utilised in the experimental work included two AC 40-50 pure asphalt cement samples from two refineries (Al-Basrah and Al-Durah) from crudes of different sources and origins in the middle and south of Iraq, their properties are shown in table (1) the rest of the group included radial styrene–butadiene styrene (SBS)-modified asphalt cement containing a two percentages (5 and 7% by weight of asphalt cement) of SBS mixed at a temperature of 180 °C for 2 hours one asphalt modified with Butyl rubber (BR) at 9% (by weight of asphalt cement) mixed for one hour at 165 °C and one modified asphalt cement with anti-stripping agent with the commercial name (BG plus) from Sika IRAQ L.L.C at 0.075% (by weight of asphalt cement) with mixed for 30 minutes at 150 °C. The properties of BG plus and SBS are presented in tables (2) and (3) respectively.

| Table 1. Physical properties of the pure asphalt cement |
|--------------------------------------------------------|
| **Property**                                             | **Asphalt type** | **Basrah** | **Durah** |
| Penetration (100g, 5sec, 25°C) ASTM- D5                |                 | 41         | 43        |
| Viscosity (c.p) at 135 °C , ASTM D4402                |                 | 527        | 479       |
| Ductility (5 cm/min, 25°C), ASTM -D 113              |                 | >100       | >100      |
| Softening point (˚C ) ASTM –D36                       |                 | 50         | 51        |
| Penetration Index (PI)                                |                 | -1.251     | -1.39     |

| Table 2. Properties of BG plus anti-stripping agent |
|-----------------------------------------------------|
| **Property**                                      | **Requirement** |
| Chemical Base                                     | Nano technology, Silane based |
| Form                                               | Liquid |
| Appearance                                        | Pale yellow |
| Density                                           | 1.0 Approximately @ 27° C |
| pH Value                                          | Approx. 9.00 |
| Consumption / Dosage                              | 0.02% to 0.1%. By weight of Binder |

| Table 3. Properties of SBS modifier |
|-------------------------------------|
| **Property**                        | **Unit** | **Requirement** |
| Density                             | Kg\ m³   | 1240           |
| Specific gravity                    | -------- | 0.95           |
| Tensile Strength (σt)               | MPa      | min.32         |
| Melting point                       | ˚C       | 170-190        |
| Elongation                          | %        | 880            |
| Molecular structure                 | -------- | Radial        |
3.2 Testing Methods

3.2.1 Dynamic Shear Rheometer (DSR) Test

The modified and base asphalt binders from both sources were heated in an oven to temperatures not exceeding 163°C for a specific period of time to make them softer while preventing ageing. Samples were then stirred to remove air bubbles and ensure homogeneity before being poured into silicon moulds; the samples were then homogenous when poured into the moulds. The filled moulds were placed on a flat laboratory table surface to cool at room temperature without chilling [12] as shown in Figure 1. The RHEOTEST model RN4.3 dynamic shear rheometer (DSR Bitumen Rheometer RHEOTEST RN 4.3 including RHEOTEST Software meets the requirements of a DSR-Rheometer stated in various binding standards (DIN EN14770, ASTM D7175-08, ASTM D 4402 /D4402 M, ASTM D7405-10a, AASHTO T350-14, AASHTO M332-14, AASHTO T315-12, AASHTO M320-1), and this was utilised to obtain the rheological parameters of all asphalt binders, as shown in ‘figure 2’. Asphalt sample frequency sweep tests were thus performed with the 8 mm plate-plate setup at temperatures of 4, 16 and 28 °C and the 25 mm plate-plate set-up at temperatures of 40, 55 and 70 °C. Preparing and testing the samples at multiple temperatures was completed within two hours [13]. Each sample was loaded into the rheometer within 60 min of molding. Rheometer plates preheated during loading to assure adequate adhesion between the asphalt binder and the rheometer plates. For pure (unmodified) binders, the recommended temperature is 60°C and the samples were thus cooled to the test temperature before trimming it by using a heated spatula. The gap then reduced to exactly 2.0 mm for 8mm diameter and to 1mm gap for 25mm diameter. The samples then achieve thermal equilibrium before testing over period of 10 min.

The applied force on the sample in rotational rheometer testing is controlled shear stress, which allows measurement of properties of flow (such as dynamic shear viscosity from flow tests) and dynamic properties of material (as phase angle and viscoelastic modulus from oscillation tests). The loss modulus ($G''$), complex shear modulus ($G''$), storage modulus ($G''$), and phase angle ($\delta$) or loss tangent can also be determined.

3.2.2 Rotational Viscometer (RV) Test

A Brookfield viscometer was utilised to measure the rotational viscosity of the pure and modified asphalt cement binders. A SC4-28 spindle at 20 rpm was used, and measurements were made from 120°C to 200°C for all asphalt binders. For each binder sample, readings were taken at 120, 135, 150, 165, 180 and 195 °C; these readings were then averaged and used in calculating activation energy at high temperatures and at both compaction and mixing temperatures for asphalt cement.

4 Results and Discussion

The thermal susceptibility of asphalt binder is considered to be an important rheological property especially for modified asphalt, being related to its ability to resist permanent deformation [14]. Temperature susceptibility reflects how rapidly the properties of asphalt binders change with temperature in terms of indices such as activation energy, penetration index, and viscosity-temperature susceptibility [15].

4.1 Activation Energy ($E_a$) calculation

The Activation Energy ($E_a$) of flow can be determined from the slope between $\ln (\eta)$ and $1/T$ (Kelvin) based on a known R-value shown in Equation (3) where $\ln (\eta)$ is the dependent variable and $1/T$ (Kelvin) is the independent variable. This was calculated for each type of asphalt cement and for each test method. The intercept and slope acquired from the linear regression represent the Equation (3) parameters $\ln A$ and $E_a/R$ respectively. The ($E_a$) values were determined by multiplying the slope parameter by the universal gas constant (UGC) as presented in table (4).

Figure 3 shows an example of the linear relationship between $\ln \eta$ (y-axis) and $1/T$ (x-axis) for Basrah asphalt cement for both the DSR and RV methods. The range of test temperature depends on the type of test method, being between 4 to 70°C for the DSR test and 120 to 200°C for the RV test.

Figures 4 and 5 show the variations in the slopes that indicate that $E_a$ are affected by test method.
The experimental results in a table (4) and ‘figure4’ to ‘figure6’shown that:

1-Using different sources of asphalt binders (Al-Basrah and Al-Durah refineries) both pure and modified with different types of modifiers (SBS, BR and BG plus) gave different values of activation energy for flow for the same modifier and had different susceptibilities to temperature. This may be related to variations in the chemical nature of asphalt cement binders’ components and the nature of interaction between the components of asphalt binder and the modifiers used.

2-The values of $E_a$ for Durah asphalt cement modified using SBS were greater than for Basrah asphalt cement at all temperatures, which means its viscosity is better than Basrah asphalt cement, and it needs more energy to flow.

3- Basrah AB (both pure and modified with BR and BG Plus) was more sensitive to temperature than Durah AB, requiring higher $E_a$ to flow than Durah AB.

4-Regardless of asphalt type, the values of $E_a$ decreased as the percentage of the SBS modifier increased at low and moderate temperatures indicating that the modified asphalt cement binder has low sensitivity to changes in temperature as shown in ‘figure5’.

5-The effect of SBS and BG Plus modifiers at a low temperature appears to be the inverse of their behaviours at a high temperature, as per the experimental results from the RV and DSR tests while the effect of BR is the opposite that of the other two modifiers.

6-The $E_a$ values measured from the DSR test were consistently higher than the $E_a$ values calculated using RV test methods for all binders as shown in ‘figure 3’ mainly due to the modifiers enhancing the thermal susceptibility of asphalt cement at the service stage and decreasing the asphalt cements viscosity at the mixing stage. The thermal susceptibility of asphalt cement binders at high temperatures is thus much more sensitive.

Figure 1. RHEOTEST model RN4.3 dynamic shear rheometer (DSR)  
Figure 2. Cooling asphalt samples in silicon molds on lab table
Table 4. The $E_a$ values of different types of asphalt binder by using RV and DSR tests

| Modifier type and % | (RV) Test | (DSR) Test |
|---------------------|-----------|------------|
|                     | Durah     | Basrah    | Durah     | Basrah    |
| 0% (base)           | 62.308    | 64.682    | 102.312   | 101.855   |
| 5% SBS              | 73.099    | 69.730    | 119.115   | 113.403   |
| 7% SBS              | 73.745    | 69.999    | 97.074    | 75.753    |
| 9% BR               | 61.117    | 64.513    | 120.403   | 125.865   |
| 0.075% BG plus      | 65.962    | 69.096    | 100.982   | 98.604    |

Figure 3. Calculated $E_a$ by different test methods for pure Durah asphalt cement

Figure 4. Results of $E_a$ for local asphalt cement binders by using RV method for high temperatures (120 to 200 °C), (a) Basrah AB and (b) Durah AB
Figure 5. Results of $E_a$ for local asphalt cement binders by using the DSR method for low and moderated temperatures (4 to 70 °C), (a) Basrah AB and (b) Durah AB.

Figure 6. $E_a$ of Basrah and Durah asphalt binder at high temperatures.

4.2. Activation Energy ($E_a$) of flow at Mixing and Compaction Temperatures

Table 5. The $E_a$ of Asphalt cement at Mixing and Compaction Temperatures

| Asphalt type | Modifier type and % | (η) at 135°C(c.p) | $E_a$ (kJ/mol) | (η) at 165°C(c.p) | $E_a$ (kJ/mol) |
|--------------|----------------------|-------------------|----------------|------------------|----------------|
| Durah AC     | Base                 | 479               | 62.115         | 151              | 62.476         |
|              | 5% SBS               | 2859              | 73.339         | 617              | 73.145         |
|              | 7% SBS               | 3173              | 71.474         | 743              | 71.422         |
|              | 9% BR                | 4944              | 60.753         | 1411             | 60.646         |
|              | 0.075% BG            | 557               | 68.911         | 160              | 69.498         |
| Basrah AC    | Base                 | 527               | 64.408         | 154              | 64.660         |
|              | 5% SBS               | 2219              | 69.558         | 521              | 69.390         |
|              | 7% SBS               | 3113              | 69.726         | 758              | 69.700         |
|              | 9% BR                | 4597              | 64.479         | 1147             | 64.160         |
|              | 0.075% BG            | 601               | 68.938         | 163              | 71.515         |
The results in a table (5) represent the activation energy values calculated for different pure and modified asphalt cement at both compaction and mixing temperatures based on the experimental results gained from the RV test:

The values of dynamic viscosity and activation energy ($E_a$) at mixing and compaction temperatures were determined from the predicted model shown in ‘figure 4’ according to [16].

It can be deduced from results in a table (5) and result in ‘figure 7’ that:

1- The $E_a$ can be used as an indicator parameter to estimate relative temperature susceptibility for different modified asphalt binders

2- Binders with higher $E_a$ show a higher sensitivity to temperature change; the more difficult the compaction becomes, the higher compaction effort required to achieve the required density of the mix (increasing number of gyrations needed to achieve air voids and gain an optimum performance) [10, 17].

3- The lower the value of $E_a$, the lower asphalt cement sensitivity to temperature change reducing the required number of gyrations needed to achieve the required air voids decreasing the compaction effort.

4- The required effort in the compaction process is greater than in the mixing process because the dynamic viscosity and activation energy at compaction temperature are higher than at mixing temperature.

5- The viscosity and activation energy at mixing and compaction temperatures for Durah AB are higher than for Basrah AB which means the Durah AB is more sensitive to temperature changes than the Basrah AB.

6- Adding modifier (BR) increases asphalt viscosity and reduces the energy required to flow ($E_a$) such that decreased effort is required for compaction.

**Figure 7.** The viscosity-temperature relationship for Durah and Basrah asphalt cement.

### 4.3. Viscosity–Temperature Susceptibility (VTS)

Higher binder susceptibility to temperature means greater viscosity changes with temperature variation. Higher value of thermal susceptibility for asphalt cements is not appropriate because they are frequently exposed to ultraviolet (UV) and thermal oxidation [18]. In order to determine the viscosity-temperature relationship, the following equation is thus usually used:

$$VTS = \frac{\log \log \eta_1 - \log \log \eta_2}{\log T_2 - \log T_1} \quad (5)$$
where

T1 and T2 represent the temperatures of asphalt cement binders at known two points (in degrees Rankin (°R) where R is equal to (°K * 1.8)); and ℏ1 and ℏ2 are the asphalt cement viscosities at two known points (cP). The VTS has been demonstrated to be directly proportional to the binder’s temperature susceptibility: the higher the magnitude of the VTS, the more susceptible to change with temperature is the viscosity of asphalt cement [19].

In this research, the VTS values were calculated using Eq. (5) at various temperature ranges and for different asphalt cement types, as represented in ‘figures (8) and (9)’.

The addition of modifiers (SBS, BR, and BG plus) to asphalt binders generally reduced VTS by reducing viscosity changes due to the variation upwards in temperatures when compared with base asphalt binder for both types of asphalt binders.

BR modified asphalt cement binder has less susceptibility to change in viscosity with temperature than the other modified asphalt cement binders; it recorded the lowest values of VTS for both types of asphalt cement binders being followed by SBS then BG plus modified asphalt cement binders.

Figure 8. The values of (viscosity-temperature) susceptibility (VTS) of different sorts of modified Basrah asphalt cement binders.

Figure 9. The values of viscosity-temperature susceptibility (VTS) for different sorts of modified Durah asphalt cement binders.
4.4. Penetration Index (PI)
The penetration test is a common and simple way to evaluate asphalt cement binder’s thermal susceptibility [13]. The penetration index (PI) is a quantitative indicator that measures asphalt cement binder responses to change in temperature. As all asphalt binders have the same basic thermoplastic properties being harder when cooled and softer when heated).

The susceptibility of asphalt cement to temperature reduction is reflecting in increased values of PI. Normal asphalt cements have PI ranging between (-2 and +2). Increasing PI values to more than +2 means that asphalt cement binders have low susceptibility to temperature, while PI values less than -2 mean the binders are excessively susceptible to high-temperature.

The temperature susceptibility indicator values were measured based on the results gained from softening point and penetration tests.

A traditional method of PI calculation mentioned in the Shell bitumen handbook [20] is represented by the equation below:

\[
PI = \frac{1952 - 500 \times \log(\text{Pen}_{25}) - 20 \times \text{SP}}{50 \times \log(\text{Pen}_{25}) - \text{SP} - 120}
\]

where

- \(\text{Pen}_{25}\) represent the value of penetration of asphalt cement binder at 25°C while \(\text{SP}\) is the softening point temperature of the specimen.

The penetration index (PI) results for pure and modified asphalt cement binders are presented in table (5); these suggest that asphalt cement binder modified with 7% SBS have the maximum value of PI and asphalt cement binder modified with 0.075% BG plus have the minimum values respectively, meaning the modified asphalt cement binders with SBS have lower sensitivity to temperature compared with 0.075% BG plus modified asphalt cement binders.

| Modifier type | Penetration @ 25°C | Softening point (Ring& Ball, °C) | PI |
|---------------|-------------------|----------------------------------|----|
|               | Durah | Basrah | Durah | Basrah | Durah | Basrah |
| Base          | 41    | 49     | 51    | 50     | -1.39 | -1.250 |
| 5%SBS         | 33    | 38     | 68.3  | 69.5   | 1.542 | 2.062 |
| 7%SBS         | 29    | 35     | 71.5  | 72.6   | 1.767 | 2.357 |
| 9%BR          | 28    | 30     | 70.1  | 69.1   | 1.474 | 1.463 |
| 0.075%BG      | 45    | 52     | 50.3  | 49.5   | -1.362| -1.243 |

5- Conclusions
Analysis of experimental results for the two types of asphalt binders from different sources with three types of additives showed that:

1. The addition of modifiers (SBS, BR, and BG) to asphalt binders increased their \(E_a\) and PI indices and decreased VTS by reducing viscosity changes due to variation in temperatures as compared with both types of asphalt cement binder.

2. Using varied asphalt sources from the middle and south of Iraq with a variety of modifier types produced different activation energy \((E_a)\) values, which may be related to the nature of interaction between the component of asphalt cement binders and different types of modifiers and to variations in the chemical nature of asphalt cement binders.

3. The type of test method used has important impacts on the calculation of \(E_a\) for asphalt cement binders employing different temperatures ranges (4 to 70°C and 120 to 200°C) to characterise the pure and modified asphalt cement binders.
4-The $E_a$ values at high-temperatures increased with the addition of modifiers by 7.315 and 17.072% for 5% SBS, 7.795 and 14.319% for 7% SBS and 10.602 and 11.139% for anti-stripping BG plus with the addition of BR reducing the $E_a$ values by 0.77 and 2.929% for Basrah and Durah asphalt binders respectively compared with base binders. Using these modifiers improves the rheological properties of both types of asphalt binder and binders with higher $E_a$ are more viscous at higher temperatures. The lower $E_a$ to flow value, the lower asphalt binder sensitivity to temperature changes, while higher values of $E_a$ indicate higher sensitivity to changes in temperature.

6-Using SBS as a modifier for asphalt binder improves the viscosity better than using the other tested modifiers, increasing the energy required to flow by 18.08% and 17.077% for Durah asphalt and by 7.99% and 7.315% for Basrah asphalt at moderate and high temperatures respectively compared with base asphalt binders.

7- The addition of BR modifier causes a higher increment rate in $E_a$ values at low temperatures by 23.573% for Basrah asphalt binder and 17.682% for Durah asphalt binder compared with base asphalt binder.

8- The addition of modifiers (SBS, BR and BG Plus) to asphalt binders generally reduces VTS by reducing viscosity changes due to variation in temperatures when compared with base asphalt binder for both types of asphalt binders.

9- The reduction rates of VTS for the two types of asphalt binders were recorded for BR modified binder as 40.563% and 44.138%, with 18.696% and 15.994% for 5% SBS-modified binder 16.933% and 15.241% for 7% SBS – modified binder 4.876% and 2.496% with the addition of BR plus for Basrah and Durah asphalt binder respectively compared with base asphalt binder.

10-Increasing the values of penetration index (PI) with the addition of modifiers and improving the plastic range, means the temperature susceptibility is improved. SBS and BR can thus improve the properties of asphalt matrices; increase the mixture stability and enhancing the anti-rut ability of mixture at high temperature while the effect of BG plus is negligible.

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