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Short communication

Improved methodology for the measurement of vapor-liquid equilibria of binary mixtures with large boiling point differences via differential scanning calorimetry

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ABSTRACT

The transition from petrochemical to bio-based processes introduces a multitude of novel products and the need for corresponding thermodynamic phase equilibrium data. Differential scanning calorimetry (DSC) offers a small-scale and fast method to determine solid-liquid as well as isobaric vapor-liquid equilibrium data in a wide temperature range. This study aims to extend the use of DSC to measure boiling point data of binary mixtures, focusing on systems with large boiling point differences. By adjusting sample volume and heating rate, the accuracy of measured boiling points for systems with high boiling point differences was improved. The measuring method was applied to a high boiling binary system consisting of carvacrol and dimethylsulfoxide and the experimental data was successfully correlated using the NRTL model. This work broadens the applicability of DSC for future thermodynamic investigations in vapor-liquid equilibrium studies, particularly for high boiling systems with large boiling point differences.

1. Introduction

The transformation from established petrochemical-based processes to greener bio-based processes is giving rise to many novel separation processes, leading to a multitude of yet unknown thermodynamic phase equilibria of novel products with high relevance for the chemical industry [1]. Although qualitatively good results in predicting thermodynamic phase equilibria can be achieved with methods like COSMO-RS [2] and modern machine learning tools [3,4], experiments are still necessary to obtain reliable thermodynamic data. To derive thermodynamic data, differential scanning calorimetry (DSC) is a fast, small-scale measurement method applicable for a large temperature scale which matches the low availability and urgency of thermodynamic data of novel products [5].

Currently, DSC is used almost exclusively for the characterization of solid samples or suspensions, while its use for measuring vapour-liquid equilibria is scarce in literature. Despite its simplicity and suitability for high-temperature regimes, where conventional techniques using heating oil face limitations, only a few examples of successful determination of boiling point data in binary mixtures were recently published [6–11]. These studies mainly address process parameters influencing the accuracy of the measurement procedure, such as heating ramp, sample volume, and pin-hole size. However, these process parameters were only varied in a narrow range such as “typical” sample volume bewtween 1–15 μL [12] or heating rates of 5 - 20 K/min for pure compounds, while the heating rate of liquid mixtures should not exceed 10 K/min [5]. Further, only binary mixtures with low boiling point differences were successfully investigated due to inapplicability of the measuring procedure caused by pre-evaporation of the low-boiling compound [10]. Hence, the need for improved DSC procedures in the quantification of binary mixtures with large boiling point differences is raised [5].

To this end, we present and discuss limitations in DSC measurements of boiling point data and propose a strategy to extend its field of application. In the presented method the main process parameters (heating ramp and sample volume) were adjusted, broadening the window of applicability for DSC in measuring boiling point data of binary systems. Further, we apply the adjusted methodology of collecting boiling point data to a novel binary mixture consisting of the aromatic compound carvacrol and the polar solvent dimethylsulfoxide, which is a conventional solvent for the separation of the aromatic and aliphatic compounds [13].

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Table 1
Experimental boiling points of pure compounds investigated in binary systems.

Compound	Boiling point / °C	References
Water (H2O)	100	[14]
Ethanol (EtOH)	79	[15]
Ethylene Glycol (EG)	197	[16]
Dimethylsulfoxide (DMSO)	191	[17]
2,3-butanediol (2,3-BDO)	180	[18]

2. Materials and methods

2.1. Reagents

All experiments were conducted with the following chemicals: Ethanol (EtOH) (VWR, purity ≥ 99.5 %), Ethylene glycol (EG) (VWR, purity ≥ 99.5 %), 2,3-butanediol (98 %, VWR Chemicals), Dimethylsulfoxide (DMSO) (Sigma-Aldrich, purity ≥ 99.7 %), Carvacrol (VWR, purity ≥ 97.5 %).

2.2. Experimental determination of boiling point data

Differential scanning calorimetry (DSC) measurements were conducted to determine binary boiling points. Indium samples with a defined melting point of 156.6 °C were used for calibration. The DSC1 and DSC3 using the STARe System from Mettler Toledo was employed. Samples of the pure compounds and different molar ratios of water, ethanol (EtOH), ethylene glycol (EG), dimethylsulfoxide (DMSO), 2,3-butanediol (2,3-BDO), or carvacrol were prepared by inserting a defined volume between 15 µL and 40 µL into an aluminium crucible (maximum filling size 100 µL). The crucible was then hermetically sealed to prevent evaporation and equipped with a pinhole shortly before measurement to avoid pre-evporation of the mixture. A nitrogen flow of 80 mL/min was applied to avoid condensation in the furnace. All measurements were performed in triplicates. The samples were initially hold at 25 °C for 2 min and consecutively heated to 260 °C with different heating rates between 10 and 60 K/min. Analysis of the evaporation peak was conducted to derive the isobaric boiling point line at atmospheric pressure using the second derivative of the thermogram as presented in literature [8,11]. It is assumed that the liquid composition remains effectively constant during heating because the fast scanning rate establishes a quasi-equilibrium state in which any early vaporization is negligible [5]. The measured atmospheric pressure is 99.07 kPa, the uncertainty in pressure is $u(p) = 0.9$ kPa. The uncertainty in temperature measurement via DSC3 is given as $u(T) = 0.1$ K.

2.3. Experimental procedure

Boiling point data for four binary systems (H2O – EtOH, H2O – EG, H2O – DMSO, and 2,3-BDO – EG) is measured via DSC (see Table 1).

In all four binary systems, molar compositions of each compound between $x_i = 0.2$ and $x_i = 0.8$ are investigated in dublicates and the process parameters heating rate and sample volume are examined. The heating rates are 10, 25, 40, and 60 K/min, while the sample volume is either 15 or 40 µL. The H2O – EtOH system represents low boiling point differences (21 K). The H2O – EG and H2O – DMSO systems have large boiling point difference of 97 K and 91 K respectively, which is particularly challenging due to pre-evaporation resulting in two endothermic, flat peaks [10]. The 2,3-BDO – EG system has a low boiling point difference (17 K), however, consists of two high boiling compounds. Therefore, the applicability of DSC measurements at elevated temperatures is investigated. As a measure for deviation from literature values the relative boiling point is introduced and equals unity when the measured boiling point via DSC is equal to literature data. Using the resulting heating rate and filling volume (40 K/min and 40 µL), the high-boiling carvacrol–DMSO system was investigated at different compositions, with each composition prepared in triplicate. The uncertainty of the mole fractions of 2,3-BDO results from the weighing error and triplicates and is given as $u(x_{2,3-\mathrm{BDO}}) \leq 0.001$.

2.4. Thermodynamic modeling of phase equilibrium data

We use the well-known non-random two-liquid (NRTL) activity coefficient model to correlate experimental isobaric boiling point data (pTx-data) [19]. The following model equations are used to express the activity coefficient γ_i:

$$\ln\gamma_i = \frac{\sum_j x_j \tau_{ji} G_{ji}}{\sum_k x_k G_{kj}} + \sum_j \frac{x_j G_{ij}}{\sum_k x_k G_{kj}} \left(\tau_{ij} - \frac{\sum_m x_m \tau_{mj} G_{mj}}{\sum_k x_k G_{kj}} \right)$$

$$G_{ij} = \exp\left(-\alpha_{ij}\tau_{ij}\right)$$

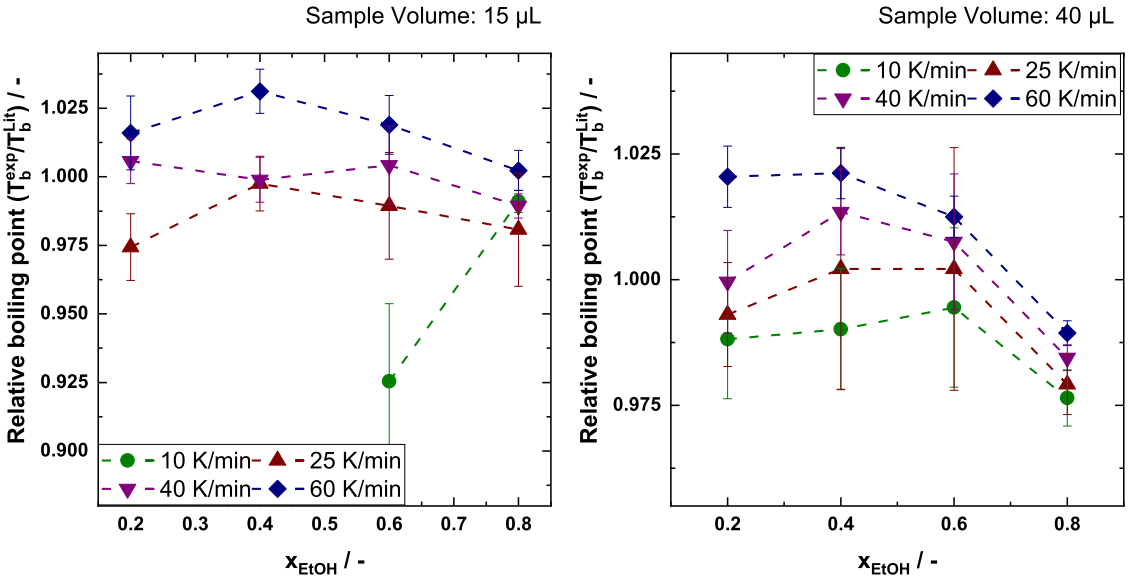

Fig. 1. The effect of heating rates (10, 25, 40, 60 K/min) on relative boiling points of the binary system H2O – EtOH. Fig. 1a and Fig. 1b present results with a sample volume of 15 µL and 40 µL.

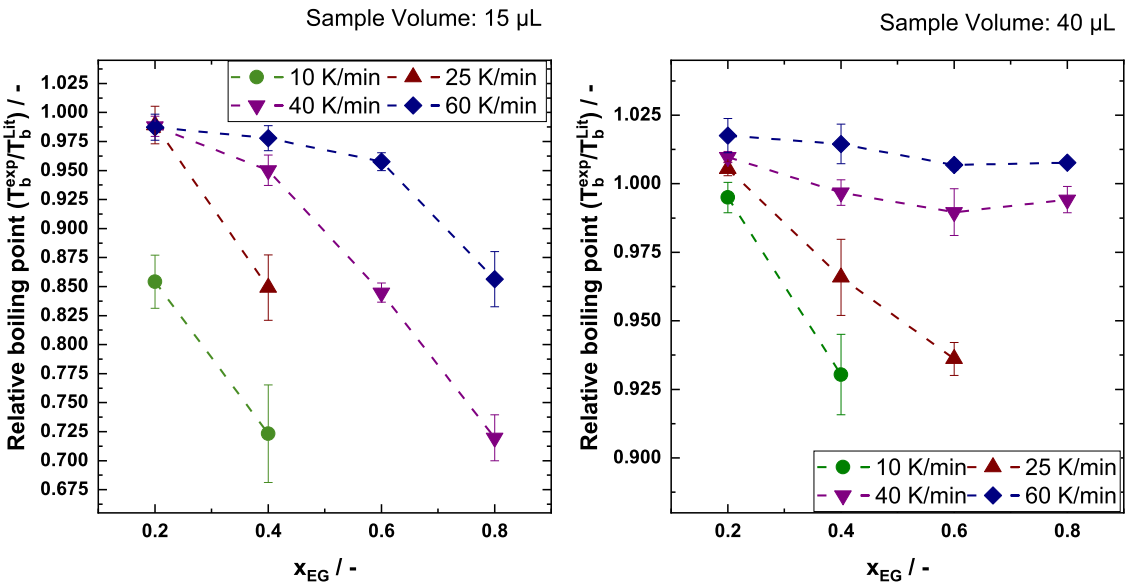

Fig. 2. The effect of heating rates (10, 25, 40, 60 K/min) on relative boiling points of the binary system H2O – EG. Fig. 2a and Fig. 2b present results with a sample volume of 15 µL and 40 µL.

$$\tau_{ij} = \frac{a_{ij}}{T}, \ (\tau_{ii} = 0$$

$$\alpha_{ij} = 0.3, \ (\alpha_{ij} = \alpha_{ji}); \ (\alpha_{ii} = 1)$$

The binary interaction parameters were regressed by adjusting a_{ij} and a_{ji} to minimize the objective function (OF):

$$OF(T) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}\left(\frac{T_n^{exp} - T_n^{NRTL}}{\sigma_n(T)}\right)^2}$$

where T_n^{exp} is the experimentally derived temperature of one of all N experimental points, σ_n is the standard uncertainty, and T_n^{NRTL} is the calculated temperature to fulfill the equilibrium condition of vapor-liquid equilibrium.

$$p\, x_i = x_i\, \gamma_i(x,T)\, p_i^s(T)$$

An ideal gas phase is assumed, while the calculation of the pure substance vapor pressure is performed by using antoine parameters adopted from literature [17,20].

3. Results and discussion

The effect of sample volume and heating rate on the relative boiling point of the system H2O – EtOH is presented in Fig. 1. The boiling point data at four different molar fractions was collected and compared to data from the work of Kurihara et al. [21]. In Fig. 1a boiling point data with a sample volume of 15 µL, which is in the upper range of typical sample volume that is used for DSC measurements [5,12], is shown. The variation of heating rate has significant effect on the measured boiling points. Slow heating rates (10 and 25 K/min) lead to inaccurate measurement of the boiling point, due to wide, flat peaks which can be attributed to pre-evaporation (see Figure S1 in the supplementary information). In agreement to Troni et al. [10], in particular, when small

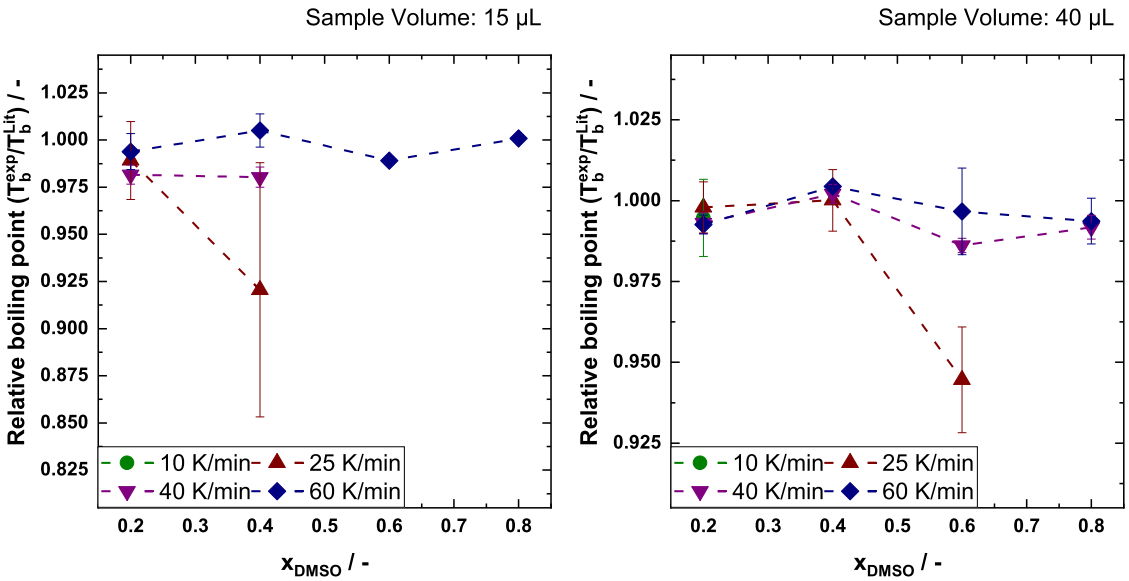

Fig. 3. The effect of heating rates (10, 25, 40, 60 K/min) on relative boiling points of the binary system H2O – DMSO. Fig. 3a and Fig. 3b present results with a sample volume of 15 µL and 40 µL.

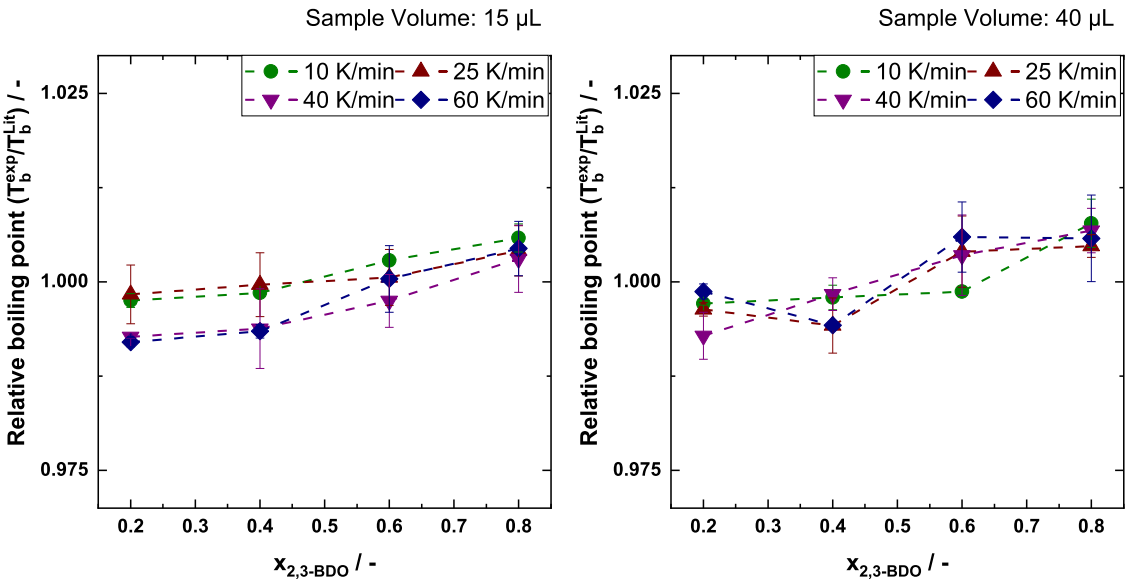

Fig. 4. The effect of heating rates (10, 25, 40, 60 K/min) on relative boiling points of the binary system 2,3-BDO – EG. Fig. 4a and Fig. 4b present results with a sample volume of 15 µL and 40 µL.

molar fractions of the low-boiling compound are present ($x_{EtOH} = 0.2 - 0.4$) this effect is emphasized, leading to non-processable peaks. As expected, fast heating rates (60 K/min) lead to boil retardation and consequently to increased measured boiling temperatures. A heating rate of 40 K/min leads to the most accurate results for the H2O – EtOH system when using 15 µL as sample volume, where the largest deviation from a literature value was 0.83 K for a mole fraction of $x_{EtOH} = 0.8$.

In Fig. 1b, results with increased sample volume of 40 µL are presented. Contrary to suggestions in literature [5] and ASTM E1782–14, it is hypothesized that large absolute sample mass reduces the impact of pre-evaporation on the measured boiling point while boiling retardation is minimal. The measured boiling points using 40 µL sample volume follow a similar trend as in Fig. 1a: The slowest heating rate (10 K/min) leads to flat peaks and, therefore, to lower measured temperatures (maximum deviation from literature 1.8 K). The fastetst heating rate (60 K/min) leads to boiling retardation and, hence, increased boiling temperatures are measured (maximum deviation from literature 1.7 K). Again, a heating rate of 40 K/min leads to the most accurate results (maximum deviation from literature 1.1 K). In summary, when facing systems with low boiling point difference, fast heating rates are favorable and large sample volume leads to applicability of DSC measurements, even with slow heating rates, due to reduction of pre-evaporation impact.

In Fig. 2, the boiling point data of the system with large boiling point difference (H2O – EG) is given and compared to the data from Kamihama et al. [22]. In Fig. 2a, the problem of large boiling differences is evident: With standard sample volume (15 µL), regardless of the heating rate, large deviation from boiling points measured in literature were observed. For the highest heating rate (60 K/min), boiling points at least in the correct range can be determined over the widest range in comparison to the other heating rates, but these also fail as soon as only small fractions of low boiling component are present.

The large deviation of the experimental boiling points can be attributed to pre-evaporation, which can be reduced via very fast heating and large sample volume. In Fig. 2b, large sample volume (40 µL) was tested.

The combination of large sample volume and fast heating rates lead to satisfactory results over the whole concentration range, while slow heating rates lead to the problem of pre-evaporation. Analoguously to the results in Fig. 1, the fastest heating rate (60 K/min) leads to boiling retardation. The largest deviation from literature values was 1.9 K, while a heaing rate of 40 K/min again leads to the most accurate results with the largest deviation from literature values of 1.3 K, which is in the acceptable range for boiling point measurements via DSC [23].

In Fig. 3, the second system (H2O – DMSO) with large boiling point difference is given and compared to data Zhang *et al.* have published [24]. Analogous to previous systems, the measurement of boiling points does not work well when the fill volume or heating rate is low (see Fig. 3a) and the low boilers are present only in very small amounts. When applying a heating rate of 10 K/min, a boiling point can only be derived for small fractions of the high-boiling compound ($x_{DMSO} = 0.2$) while for higher fractions of DMSO the thermograms are not processable. The peaks to be evaluated then become increasingly flatter until they are no longer distinguishable. Also with a high filling volume (40 µL), the boiling points are measurable only when using the fast heating rates of 40 K/min or 60 K/min (see Fig. 3b). At 40 K/min the maximum deviation is 1.3 K and at 60 K/min it is 1 K, meaning that both heating rates perform similarly well and remain within an acceptable range [23].

Finally, Fig. 4 presents the system of the high-boiling diols 2,3-BDO – EG. These components are high-boiling components with a boiling point difference of 17 K. This system is used to verify the DSC procedure for high-boiling components and compared to the work of Zhong et al. [25].

Essentially, the measurement works well with both, the standard fill volume of 15 µL (see Fig. 4a) and the higher filling volume of 40 µL (Fig. 4b). The measurement results are very good regardless of the heating rate. The largest deviation is again observed with the slowest heating rate of 10 K/min, showing a deviation of 1.4 K at $x_{2,3-BDO} = 0.2$.

Due to their high boiling points, the system does not tend to undergo pre-evaporation in the lower temperature region, resulting in excellent reproducibility. Moreover, because of the small boiling point difference, the fill volume and heating rate do not seem to be decisive factors, which aligns with the initial hypothesis and findings from the previous systems. Further, it demonstrates that the DSC method is suitable for analyzing components with high boiling points.

In conclusion, the boiling point data was most accurately measured using a sample volume of 40 µL and a heating rate of 40 K/min, resulting in an average deviation of 0.9 K from literature values for the four systems. Therefore, it was demonstrated that the adjustment of the processing parameters allows for the determination of boiling point data for a broad range of systems, including systems with large boiling point

Table 2
Experimental boiling point data for the binary systems consisting of carvacrol and DSMO at atmospheric pressure. Standard deviations are representing the 95 % confidence intervall.

	T_b /°C	σ /°C
0	191.05	0.41
0.1	195.04	1.83
0.2	200.98	1.93
0.3	212.28	0.93
0.4	222.75	3.43
0.5	230.56	1.79
0.6	235.54	1.45
0.7	237.58	1.03
0.8	239.08	0.77
0.9	238.95	1.00
1	236.88	0.39

Table 3
Antoine parameters to calculate the pure component vapor pressure of carvacrol and DMSO are taken from literature.

Component	A	B	C	Source
Carvacrol	5.34179	2549.857	−32.705	[20]
DMSO	4.49107	1807.002	−60.995	[17]

Table 4
NRTL paramters for correlation of binary boiling point data of the binary system carvacrol – DMSO.

Component i	Component j	a_{ij}	a_{ji}	α_{ij}
Carvacrol	DMSO	2321.9351	−1354.0337	0.3

difference.

The adjusted methodology for the collection of boiling point data (sample volume 40 µL and a heating rate of 40 K/min) was applied to the binary system consisting of carvacrol (boiling point = 237 °C, [20]) and DMSO (boiling point = 191 °C, [17]). The difference in boiling points of this system is approximately 46 K, which is therefore considered as a challenging system to measure via DSC [5,10]. To best of our knowledge, no boiling point data of the system carvacrol – DMSO was presented so far. In Table 2, the molar composition of the binary system carvacrol – DMSO, the corresponding mean boiling point resulting from triplicates and the standard deviation representing the 95 % confidence intervall are presented. The boiling points of the pure substances are in good agreement with boiling points found in literature [22,20].

The boiling point data was used to correlate NRTL parameters as described in Section 2.4. To calculate the pure component vapor pressures, antoine parameters taken from literature are used (see Table 3). The correlated NRTL parameters of the binary pair are documented in Table 4.

In Fig. 5a, the experimental boiling points, the corresponding standard deviations as well as the correlation of boiling point data and calculated dew point data are given. The corresponding activity coefficients of carvacrol and DMSO are given in Fig. 5b

The presented experimental boiling point data of the binary system consisting of carvacrol and DMSO was successfully correlated with the NRTL model. It should be noted, however, that only (pTx)-data are available, so a more comprehensive assessment of thermodynamic consistency based on energy-related properties was not possible. One potential approach would be the Olson test [26], which is based on the Gibbs-Helmholtz relationship, but its application requires reliable excess enthalpy data at the relevant temperatures. Such data are experimentally difficult to obtain and would require dedicated calorimetric measurements. The system is defined by a temperature maximum azeotrope and corresponding activity coefficents below unity, indicating strong interactions of the two compounds in the liquid phase. Since carvacrol is a strong assymetric hydrogen bond donor [27], and DMSO is a conventionally used hydrogen bond acceptor [13], the strongly non-ideal behaviour of the mixture can be well explained.

4. Conclusion

This study successfully demonstrates the extension of differential scanning calorimetry (DSC) to measure the boiling point data of binary systems with significant boiling point differences. By optimizing the process parameters sample volume and heating rate, DSC measurements could be improved, especially for challenging systems with high boiling point differences, such as H2O – EG or H2O – DMSO. A sample volume of 40 µL and a heating rate of 40 K/min gave the most accurate results for systems with both low and high boiling point differences. The method was also applied to the binary system of carvacrol and DMSO, which has not previously been described in the literature. The experimental boiling

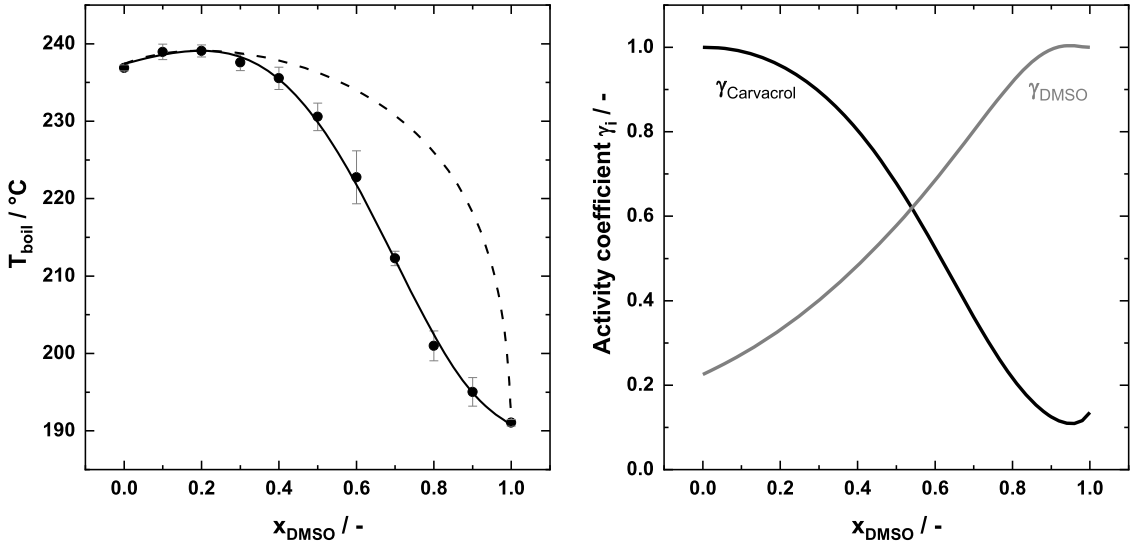

Fig. 5. Binary vapor-liquid equilibria data of carvacrol – DMSO. The experimental boiling points (black circles) and standard deviations are presented (left). The experimental boiling points are correlated using the NRTL model. The corresponding boiling point line is presented as full line and the calculated dew point line is presented as dashed line. The calculated activity coefficients are given (right).

point data was correlated using the NRTL model, revealing a temperature maximum azeotrope, indicating strong hydrogen bonding interactions between the two compounds. In conclusion, it is demonstrated that DSC provides a reliable, small-scale and fast method for the determination of boiling point data, even for high boiling compounds with complex thermodynamic behavior.

CRediT authorship contribution statement

William Graf von Westarp: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Andreas Jupke:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.tca.2026.180222.

Data availability

Data will be made available on request.

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