1. Introduction

An inherent characteristic of a pure liquid substance is its boiling point. By definition, the boiling point refers to the temperature at which the vapor pressure equals the ambient pressure. For comparability reasons, the boiling temperature is usually corrected to the temperature at which the vapor pressure of the liquid is equal to 101.3 kPa. [1]

In addition to the ambient pressure, the boiling point depends on the molecular weight, the molecular structure, intermolecular forces and impurities of the liquid analyzed.

Here, two established boiling point determination methods are compared.

The first method, already described by Siwoloboff in 1886, is preferentially applied to small amounts of liquid. [2] For the Siwoloboff method a boiling point capillary is immersed in a drop of liquid placed in a boiling point tube. Upon heating, bubbles rise in the liquid and ultimately form a constant rising bubble-chain at the boiling point (Figure 1). At the boiling temperature the bubbles are released from the boiling point capillary at a frequency of 0.6 Hz.

The second method is for larger sample quantities, i.e. 20 ml or more, and described in detail in Ph. Eur. 2.2.12. [1]

2. Experimental

All experiments were conducted by TECHPharm GmbH Germany. For the Siwoloboff boiling point determination the BUCHI M-565 was employed. Common laboratory distillation glassware was used for the Ph. Eur. method. For details regarding the apparatus we refer to Ph. Eur. 2.2.12 method.

The boiling point of ethanol, N,N-dimethylformamide (DMF) and glycerine was determined. For each substance either 250 µl for the Siwoloboff method and 20 ml for the Ph. Eur. procedure were needed.

3. Results and discussion

Obtained boiling points are shown in Table 1. Comparing the boiling point detected using both, the Melting Point M-565 and a distillation apparatus according Ph. Eur. 2.2.12, revealed that the results obtained with Melting Point M-565 device confirmed those obtained with the distillation apparatus.

4. Conclusion

Boiling point determinations according to Siwoloboff confirmed the results obtained by measuring the boiling point according Ph. Eur. 2.2.12 and the specified boiling point temperature.

Compared to the Ph. Eur. method the boiling point determinations according to Siwoloboff require only small sample volumes of 250 µl. Further important advantages of the Siwoloboff method are due to the use of a commercial device, the M-565 which

i) accelerates the boiling point determination compared to the Ph. Eur. method

ii) automates the process due to gradient programming

iii) improves reproducibility

iv) allows post-experimental analysis due to recording function

v) enables print-out of results for GMP compliance

vi) relies on a temperature calibration traceable to primary standards

5. Acknowledgement

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6. References

[1] Ph. Eur. 2.2.12, European Pharmacopeia 7.0.
