The Benefits of Microscopic Methods in the Evaluation of Binary Systems

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The high potential of microscopic methods in the evaluation of binary systems is presented by an introduction to the Kofler's contact preparation method (KCPM). In combination with FTIR- and/or Raman-microscopy structural information of the involved phases assists the interpretation of the melting diagram.

The KCPM is a very fast and simple technique which enables the qualitative determination of organic two-component systems. It was first carried out and published by Otto Lehmann in 1888, however, forgotten until Adelheid Kofler discovered this method again fifty years later in Innsbruck [1], perfected [2], and applied it in hundreds of binary systems. The KCPM consists in allowing the melts of two different organic compounds to coalesce on an object slide beneath a cover slip [3]. The melts are caused to mix in their contact zone and to crystallize by cooling. During heating and recooling the microscopic observation of the contact zone offers the possibility to gain an insight into the behaviour of the mixed components with one single preparation. By this way it is possible to draw up the basic form of a melting diagram including the information whether the two substances form with each other a single eutectic, a molecular compound, mixed crystals or even a mixing gap between the liquid phases. Additionally, polymorphic transitions of the pure components or in any of their mixtures which may occur during temperature changes are detectable and provide with supplementary knowledge as to the kinetic stability of the different crystalline phases.

In combination with further analytical tools like differential scanning calorimetry, vibrational spectroscopy and X-ray powder diffractometry, the quantitative construction of even complex phase diagrams is enabled. This procedure is demonstrated in a step by step way on the example of the enantiomers of diprophylline [4].

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