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Determination of Purity by DSC

Practical 12 - Spectral interpretation practical

Aim

To assess a qualified DSC purity determination and its applicability.

Principle

Under suitable conditions an impurity can depress and broaden melting. DSC purity analysis based on melting behaviour requires assumptions about phase behaviour, calibration and absence of interfering decomposition or polymorphic transitions.

Materials and Requirements

- Recorded qualified spectra with acquisition settings
- Candidate formulae and structures
- Peak-assignment worksheet
- Approved method or teaching-data pack and a dated record sheet

Procedure

1. Define the preparation or dataset, study question and method version. Record identities, units and the criteria to be used before examining the outcome.
2. Check axis, units, calibration, solvent or reference and acquisition quality.
3. Check temperature/enthalpy calibration, sample mass, pan, atmosphere and scan programme.
4. Identify the melting event and assess whether the method assumptions and baseline treatment are justified; compare with orthogonal purity evidence.
5. Assign features with multiplicity, intensity or fragmentation context and list alternative explanations.
6. Compare all available evidence with the candidate structures, distinguishing a provisional assignment from a confirmed identity.
7. Retain the original observations and calculations. State deviations, missing information and the limits of the conclusion; do not replace measured values with expected results.

Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

Feature / region	Observed value	Proposed assignment	Supporting / conflicting evidence
Major feature	Enter	Assign	Assess
Diagnostic feature	Enter	Assign	Assess
Missing feature	State	Explain	Assess

Calculations

- For a dilute ideal impurity model: T0 - T = (R x T0^2 / deltaH_fusion) x impurity mole fraction in the liquid phase

Worked Example

Illustrative T0 = 400 K, deltaH_fusion = 20,000 J/mol and liquid-phase impurity fraction 0.010 predict a depression of about 0.665 K.

A real DSC purity estimate requires the full validated melting analysis; the onset temperature alone is insufficient.

Result and Interpretation

- Provide an annotated spectrum and a reasoned structural conclusion with limitations.

Precautions

- Do not read isolated peaks without context.
- Retain unprocessed data and processing choices.
- Use orthogonal evidence when an assignment is ambiguous.

Viva Questions and Answers

Why use kelvin in the model?

The thermodynamic equation uses absolute temperature.

Can decomposition invalidate the analysis?

Yes; it violates a simple equilibrium-melting interpretation.

Is a sharp melt proof of absolute purity?

Other compounds or phases may still be present.

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