Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T1005

Recording the infrared spectrum of materials

NOVEMBER 2012

Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added. Sections 10. and 11. Revised	D.Dash	July 2000
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T1005 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1005

Recording the infrared spectrum of materials

1. Scope

This test method sets out the procedure for recording the infrared spectrum of a material. Several methods of sample preparation are also described.

NOTE: Operation of the infrared spectrophotometer requires suitably qualified and experienced staff particularly with respect to sample preparation and the interpretation of results.

2. Apparatus

Infrared spectrophotometer

3. For Liquids and Films

Balance, accurate and readable to 0.0001g
Sodium chloride 25 mm diameter discs or demountable liquid cell
Suitable spacers for discs and liquid cell.
Holder for discs.

4. For Solids

Pressed disk:
Melt some AR potassium bromide in a platinum
crucible and cool in a dessicator. Grind to a fine powder in an agate mortar and pestle and store in desiccator.
Small agate mortar and pestle.
Small vibratory ball mill with agate capsule and balls
A 13 mm diameter evacuable pellet die.
Hydraulic press capable of pressures to 90 kN.
Rotary vacuum pump

5. Sample Preparation

For liquids and solutions, either of the following methods may be used:
Using demountable disc cell
For low viscosity liquids, select a round Teflon or metal foil spacer-ring that it is estimated will give nearly full scale absorbance with the strongest peak and place on a 25 mm sodium chloride disc.
Place a drop or two of liquid in the centre of the disc and immediately cover with another disc so that an interlayer is formed between the two disc. Make sure there are no entrapped air bubbles in the interlayer film.

NOTE: For viscous liquids, pastes or semi solids the spacer ring may not be necessary. The other disc is moved with a circular motion while applying downward pressure to squeeze sample from between the discs so as to obtain a suitable thickness of sample and ensuring there are no air bubbles trapped in the sample film.

6. Using Rectangular Liquid Cell

Select a matched pair of rectangular sodium chloride plates and using appropriate spacers assemble into a fixed path length cell in the demountable cell holder. Follow manufacturer's directions carefully

With a dropping pipette fill the cell with the sample solution, taking care to exclude air bubbles and plug the entry ports with the Teflon stoppers.

7. For Solid or Solutes One of the Following Methods is Used

7.1 Solid Samples Using the Pressed Disc Technique

Weigh accurately, approximately 300 mg potassium bromide (2.2a above) and place in agate capsule of vibratory mill, together with agate balls

Weigh accurately 1 to 1.5 mg of sample. Add sample to agate capsule, secure closure onto capsule, fasten capsule in holder on vibratory mill, switch mill on and allow to vibrate at maximum amplitude for 3 minutes

NOTE: For silicate samples a maximum of 1 mg of sample is usually sufficient.

Transfer sample from agate capsule to the evacuable pellet die, assemble top piston in die and evacuate for 5 minutes with a rotary vacuum pump.
While still evacuating, place the pellet die in the hydraulic press and compress to 70 kN for 30 seconds. Release the pressure, recompress to 70 kN for 30 seconds, release the pressure again and recompress once more to 70 kN for 2 minutes. Release the pressure.
Invert the pellet die and gently push the compressed KBr disc from the pellet die with the aid of the hydraulic press. The disc should be virtually transparent, if not discard and repeat.

NOTE: Thoroughly clean and dry the agate capsule, balls and pellet die after use.

7.2 Solutes

Spread some solution on a 25mm diameter NaCl disc, estimating the thickness to give nearly maximum absorption with the strongest peak. Allow the solvent to evaporate either in ambient conditions or in a 105°C oven until a uniform film free of solvent is obtained.

8. Procedure

Follow manufacturer's instructions for setting up the spectrophotometer to record a spectrum.

NOTE 1: For the Perkin-Elmer Model 297 spectrometer the following instrument parameters are normally used: Scan time - 10 min.; Slit program -2; Chart speed -x1.

NOTE 2: All window materials must always be optically clear and free from scratches etc. Consult an experienced chemist for the correct procedure for repolishing windows should they become defective.

NOTE 3: The window materials are hygroscopic and should not be handled with bare fingers; always use plastic gloves when handling windows. For the same reasons window materials are always stored in a desiccator or in a drying cabinet over silica gel.

If the spectrophotometer has not been used for 3 months or more perform the calibration as given in the document Australian Code of Good Manufacturing Practise for Therapeutic Goods, Guidelines for Laboratory Instrumentation, Appendix D, November 1992.

NOTE: The Code is attached to the front of the log book. Record the calibration in the log book and file the spectrum in the instruction manual.

Place the assembled discs from in a demountable disc holder, ensuring that rubber rings cushion the disc assembly, top and bottom, from the metal holder. Tighten the lock nuts gently so that the pressure is even and just sufficient to hold the top plate assembly in place. Place the whole assembly [whether with discs or liquid cell in the sample beam and record the spectrum or place the pressed KBr disc in the special disc holder, place this assembly in the sample beam of the spectrometer and record the spectrum.

NOTE: On completion, dismantle the disc holders and clean the disc with a suitable solvent. The liquid cell is drained, flushed several times with solvent and allowed to dry. Store all cells and discs in a dry environment.

9. To Obtain the Spectrum of a Solute in Solution, Proceed as Follows

Select another matched pair of rectangular NaCl plates and assemble into a cell using a slightly thinner cell spacer than for the sample cell. Alternatively a variable path length cell may be used filled with pure solvent.

Place the sample cell with sample prepared from above in the sample beam and the cell containing the solvent in the reference beam. If necessary adjust the path length of the variable path length cell to cancel out as best as possible the spectrum of the solvent in the sample beam and record the spectrum.

10. Recording

As soon as the infrared trace is complete record at the foot of the infrared spectrum, details of the sample including laboratory number, origin of the sample, name, mass of sample, procedure for sample preparation, spectrum number and instrument settings. Enter also the appropriate details into the instrument log book.

11. Interpretation

Refer to reference spectra and appropriate publications on infrared spectrophotometry for interpretation of the spectrum and identification of the sample (if required).