To close the yellow note, click once to select it and then click the box in the upper left corner.
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#18 Determination of the Set Time for Epoxy Adhesive

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I. INTRODUCTION

Description

Most epoxy glues or adhesives, as well as epoxy resins, are produced when the epoxy group reacts with a difunctional backbone monomer producing a thermoset polymer. Thermoset polymers are mostly unchanged when heated after being cured while thermoplastic polymers soften and eventually flow when subjected to increased heat and pressure. The set or gel time of a specific thermoset polymer is important to know. It determines the length of time the material may be worked prior to hardening. Many epoxy glues are sold with specific set times for specific adhesive applications. In chemistry, the set time of an epoxy is similar to the time it takes a reaction to occur. Hence, a study of epoxy set times is very similar to a study of chemical kinetics. The set time may be determined by testing the gel every so often with a stick to observe its tackiness or, possibly more elegantly, by observing the disappearance of the epoxide IR band and the appearance of the IR O–H stretching frequency. The temperature profile of the reaction may also be monitored; however, it, as well as gel time, is dependent upon the volume of material mixed. Reproducible data may be obtained when the tests are performed reproducibly.

Student Audience

This lab is designed for students in a polymer chemistry or polymer engineering course as well as chemical technology or organic chemistry students.

Goals for the Experiment

Having completed this experiment,

- students should recognize epoxy glue formation as two part polymerization (usually the curing agents act as co-monomers).
- students should have monitored the reaction by the changes in viscosity of the reacting mass, the temperature rise and fall, and the appearance of product peaks in the IR spectrum with the concurrent disappearance of reactant peaks.
- if the IR study is done in %-transmittance mode, the students should get qualitative results. If they measure the IR in Absorbance mode, some kinetic results might be obtained.
- students will have had hands-on experience with a polymerization process with materials available from the hardware store.

II. STUDENT HANDOUT

Determination of the Set Time for Epoxy Adhesive

Scenario

A salesperson for an adhesive manufacturer has visited a customer who reports that the 30 minute set time epoxy glue that they utilize in a process seems to take almost an hour for it to set in their factory. The adhesive company's quality control laboratory points out that they have previously determined the set time may be longer at low temperatures. The customer says his factory is air conditioned but is not that much cooler. You agreed to determine the set time of the customer's epoxy glue and compare it to the adhesive company's quality control specifications for that product.

Safety, Handling, and Disposal

- Obey all safety rules.
- Goggles should be worn; gloves are recommended.
- Do not stir the epoxy mass with the (foil covered) thermometer.
- Read the Materials Safety Data Sheets or other safety precautions which accompany the epoxy glue. Components of resin might be irritants. Follow the manufacturer's safety directions.
- Materials should be disposed of according to your teacher's instructions.

Materials

- two part clear epoxy glue that sets in 30 minutes
- toothpicks
- aluminum boat
- flat bottomed wooden stirring paddle (made by sawing a craft stick in half across the short axis)
- 3-inch x 3-inch square of aluminum foil
- oil or grease
- small pieces of polyethylene film cut from a freezer or food storage bag
- scissors
- IR spectrometer
- timer or clock

Procedure

1. Prepare the thermometer by placing a little oil or grease in the center of the 3-inch x 3-inch square of aluminum foil. Wrap the bulb of the thermometer tightly with the oiled side of foil inside with the thermometer. This will allow the thermometer to be removed from the hardened epoxy mass after the test.

   Caution: Do not put an uncovered thermometer in the epoxy glue. If left while the epoxy hardens, the thermometer may break when you attempt to remove it.
2. Place about 15 mL of epoxy glue (6–8 mL of each component) in the aluminum boat. If the volume of glue is to be measured, be sure to use a **disposable** measuring device.
3. Stir the epoxy to mix the two components. Try to minimize bubble formation during stirring (hence the flat bottom stick). Begin timing.
4. Securely mount the thermometer such that its bulb is in the epoxy mass. Note room temperature as well as the initial temperature.

5. Using the stirring stixck, place a little of the mixed epoxy glue between two polyethylene plastic sheets making an epoxy sandwich. Use only a little glue and spread it as thin as possible between the two sheets of polyethylene. IR spectra look best if the thinnest possible films are used (this minimizes peaks bottoming out). After placing the glue between the polyethylene sheets it may be necessary to cut the sandwich to size such that the center of it is in the beam path of the spectrometer. Record the spectrum of the glue and record the time. Repeat every 10 minutes.
6. At set time intervals (five minutes works well), poke a toothpick or other stick into the epoxy mass, noting the time and the viscosity of the mass and the extent the epoxy sticks to the toothpick. Record the temperature at set intervals as well.
7. When you are not taking temperature readings, testing tackiness (by probing with the toothpick), and making IR measurements, the background spectra of the polyethylene film, and each epoxy glue component (again recorded as a film between two polyethylene sheets) can be measured for reference. This will allow assignment of peaks to the polyethylene film, the colorless glue component, the yellow glue component, and the epoxy glue product.
8. If you have not already done so, present your data in table form.

Questions

1. How long does it take for the epoxy glue formation reaction to essentially go to completion? …for the tackiness test? …for the temperature profile? …and for completion as monitored by IR spectroscopy? How well do these tests agree?
2. Does the sample size involved seem to change the results? (The IR sample, once removed from the larger mass, is much smaller than the tackiness sample.)
3. Does this epoxy glue set in 30 minutes?
4. Why are the two parts of the glue packaged separately?
5. What happens if you heat the cured sample?

References

Dewprashad, B. and Eisenbraun, E. J. "Fundamentals of Epoxy Formulation" *J.Chem.Educ.* **1994**, *71*, 290-294.

Department of Polymer Science at the University of Southern Mississippi Web Site, the Macrogalleria, Epoxy Resins; http://www.psrc.usm.edu/macrog/epoxy.html (accessed 25 September 1997 and 7 May 1998).

1997 Annual Book of ASTM Standards, Volume 8.02 Plastics (II), Method D 2471-94, "Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins"

Mertzel, E.; Koenig, J.L. "Application of FT-IR and NMR to Epoxy Resins" Advances in Polymer Science 1986, (75), pp. 73-112.

III. INSTRUCTOR NOTES

Determination of the Set Time for Epoxy Adhesive

Purpose

In this experiment, the time an epoxy glue takes to set or gel is determined. The set time can be measured by probing the mass periodically with a stick, as suggested in ASTM D2471-71, observing and recording a temperature change during the reaction, and/or following the growth of the product O–H stretch in the IR. Parameters which determine set time may be explored.

Time required

The laboratory work for this investigation can be completed in a 2–hour period.

Group Size

Pairs are probably ideal but groups of 4 may be more feasible with respect to demand for the IR spectrometer. An FTIR will shorten the IR time requirement per sample.

Materials Needed

- two part clear epoxy glue that sets in 30 minutes [for example, DEVCON 2-Ton Crystal Clear Epoxy (available at Wal-Mart)]
- toothpicks
- aluminum boat
- flat bottomed wooden stirring paddle (cut off ice cream stick)
- 3-inch x 3-inch square piece of aluminum foil
- oil or grease
- small pieces of polyethylene film cut from a freezer or food storage bag
- scissors
- IR or FTIR spectrometer
- timer or clock

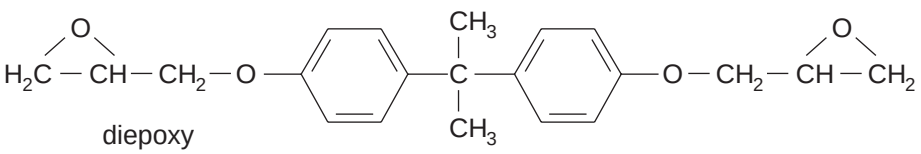
Safety, Handling, and Disposal

- Goggles should be worn, gloves are suggested.
- Be sure the students do not stir the epoxy mass with the thermometer.
- Materials should be disposed of according to local ordinances.

Points to cover in Pre-Lab

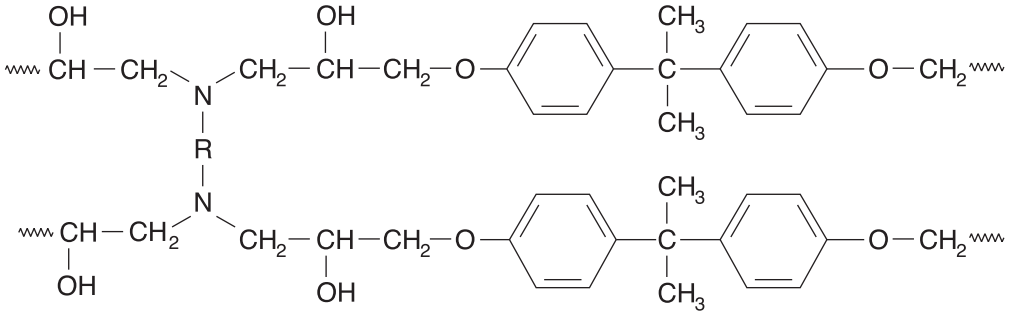
- Discuss epoxy formulations including the molecular structures, the chemistry which occurs when the two parts are mixed, and why the product is a thermoset plastic. Epoxy adhesives are polymers made when a molecule containing two epoxy groups (a diepoxy) and a molecule containing two amine groups (diamine) react. In addition to the formation of a linear chain, sometimes the diamine molecule will react with epoxy groups on different chains, causing them to bond together in a complex three-dimensional network. This three-dimensional network is what makes the epoxy adhesives become hard and rigid.

  One example of a small diepoxy is:



diepoxy

The diamine would be something like: $H_2N\text{-}R\text{-}NH_2$.
The basic structure of the epoxy resin is shown below. Note that it is not a linear polymer but rather a cross-linked network.



For more information, refer to the web site given in the references.

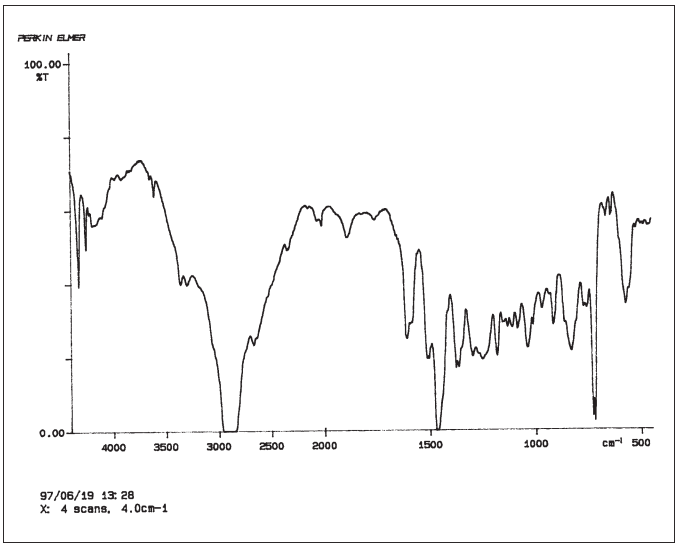
- If your students are unfamiliar with the IR spectrometer, explain how to use it and how to interpret the results.
- Review functional groups and the corresponding IR stretching frequencies including growth of O–H peak at 3450 cm^{-1} and disappearance of epoxy peak at 910 cm^{-1}.

Procedural Tips and Suggestions

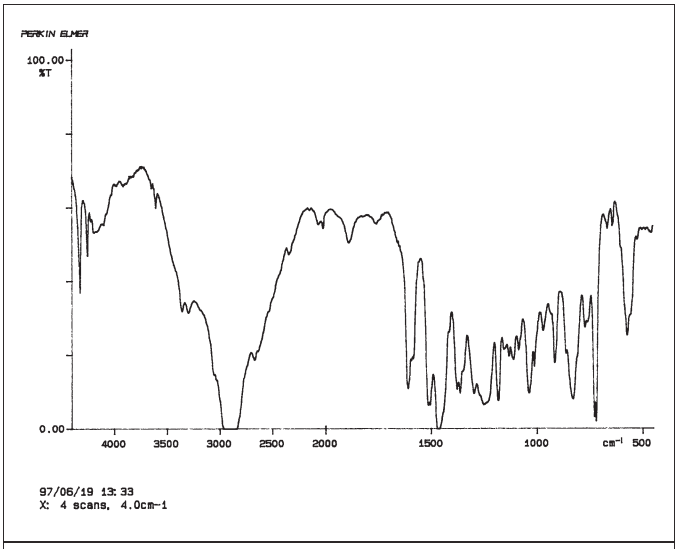
- If the class size is too large for each group to repeat the IR analysis at intervals, one group might do 5 minutes, another 15 minutes, another 30 minutes, etc. and the data pooled.
- Students will probably find tackiness tests every 3 to 5 minutes very boring. Monitoring the reaction by IR adds interest to this experiment and also focuses on the actual chemistry involved—bond making and bond breaking.

Sample Results

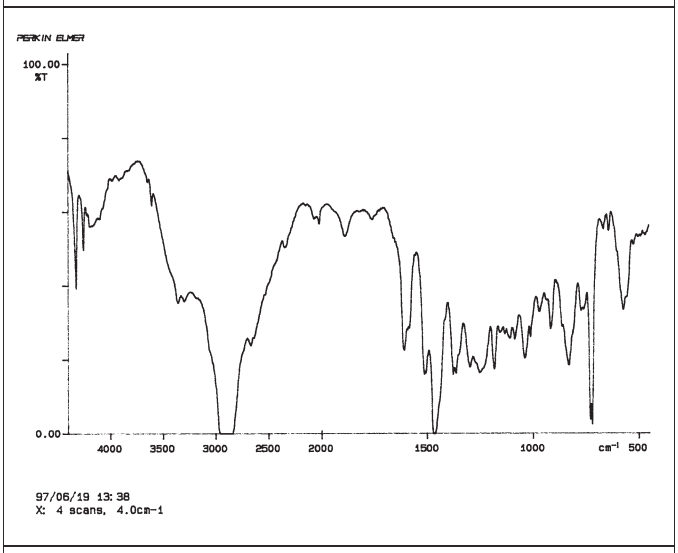
Perkin Elmer series 1600 FT-IR spectrometer was used.



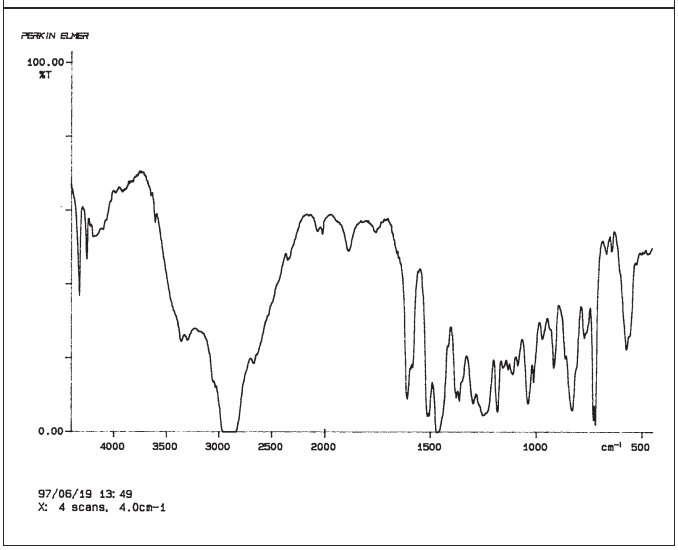
epoxy glue after 4 minutes drying time in polyethylene sandwich



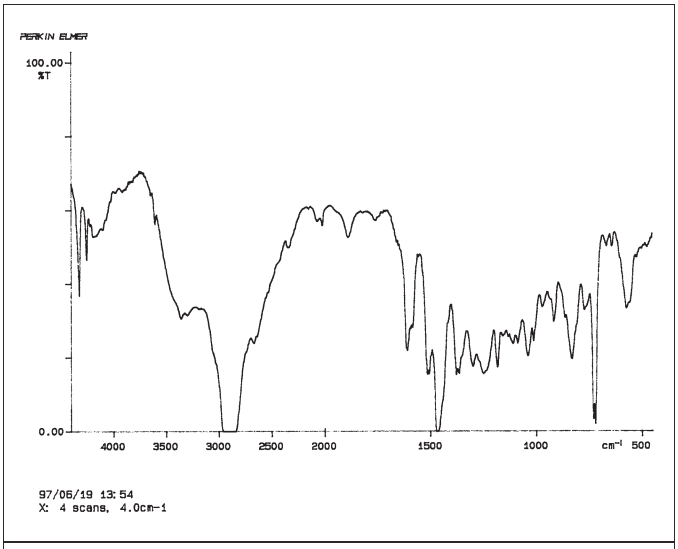
after 9 minutes



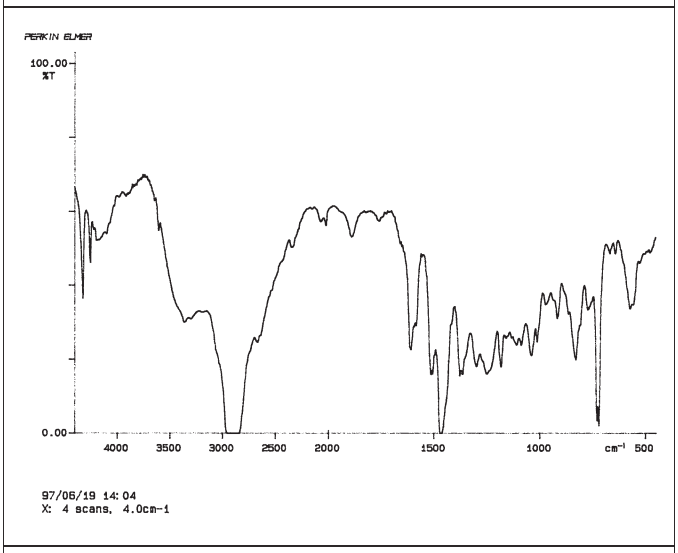
after 18 minutes



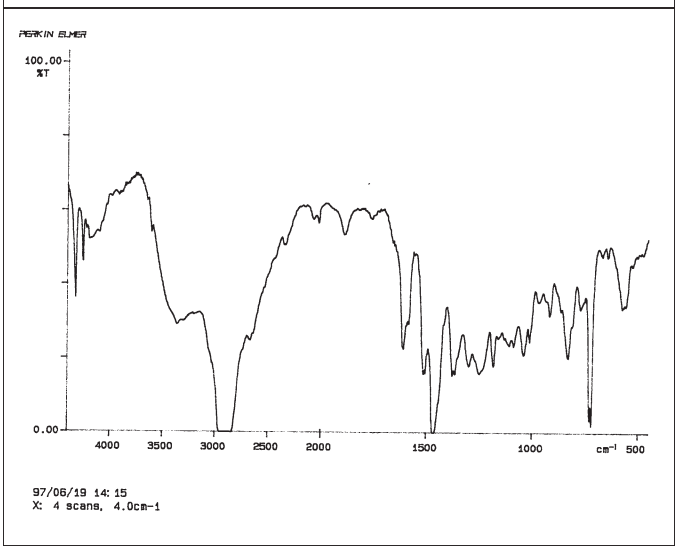
after 25 minutes



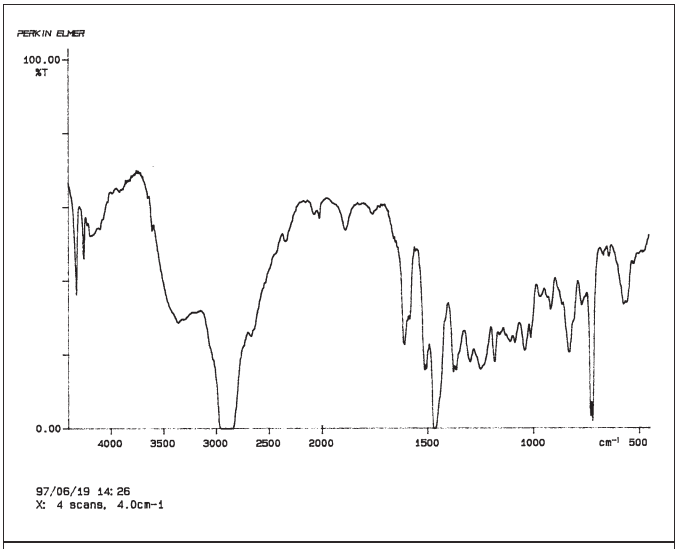
after 30 minutes



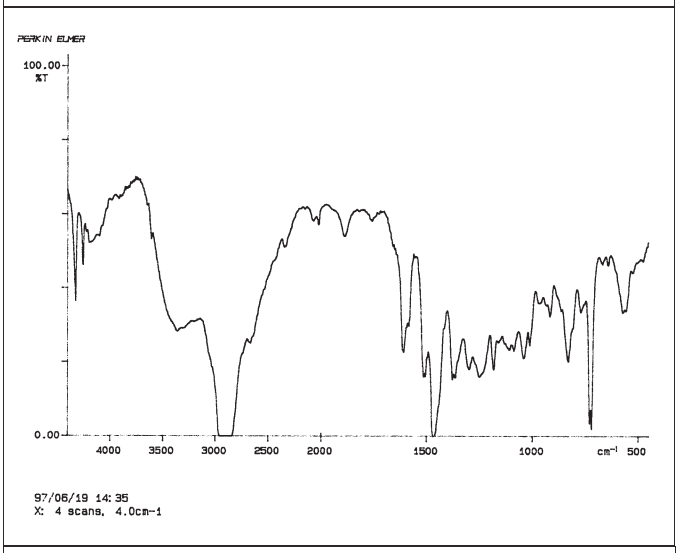
after 40 minutes



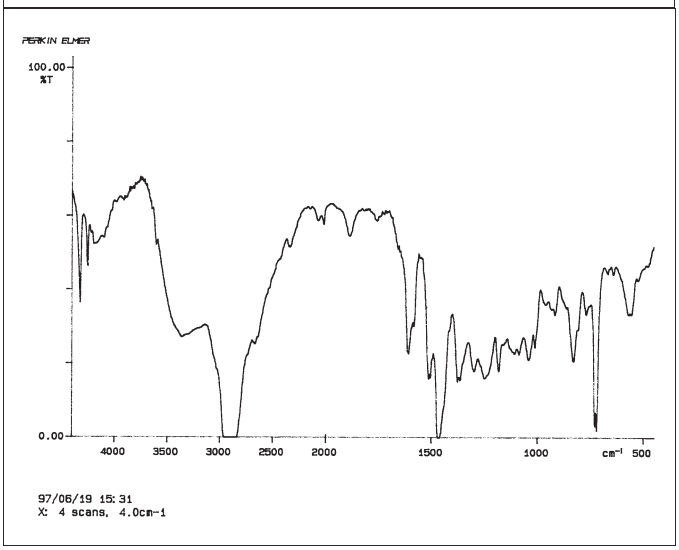
after 51 minutes



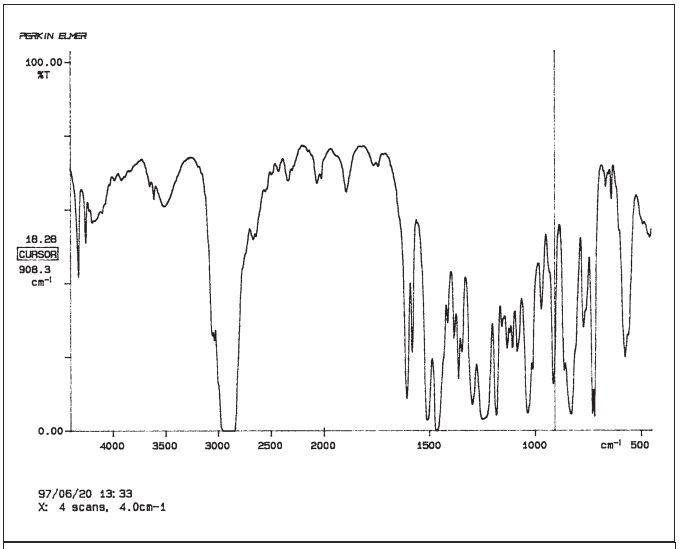
after 62 minutes



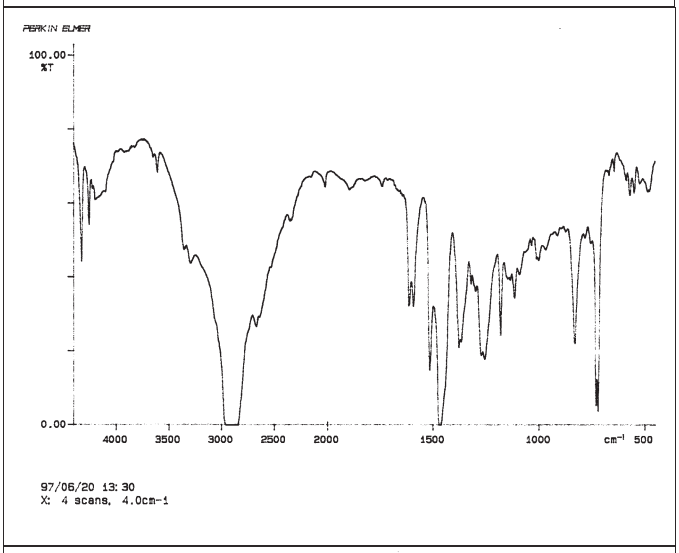
after 71 minutes



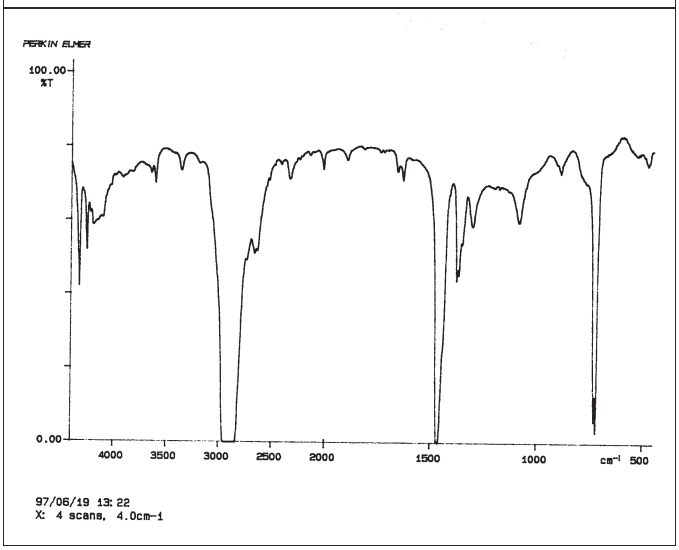
after 127 minutes



colorless componenet of two-part epoxy adhesive in polyethylene sandwich



yellow component of two-part epoxy adhesive in polyethylene sandwich



polyethylene sandwich without epoxy filling

Plausible Answers to Questions

1. How long does it take for the epoxy glue formation reaction to essentially go to completion? …for the tackiness test? …for the temperature profile? …and for completion as monitored by IR spectroscopy? How well do these tests agree?
2. Does the sample size involved seem to change the results? (The IR sample, once removed from the larger mass, is much smaller than the tackiness sample.)
3. Does this epoxy glue set in 30 minutes?

   The answers to questions 1–3 will depend on the epoxy used in the investigation as well as thetemperature. Students answers should reflect their data. See "Sample Results" above.
4. Why are the two parts of the glue packaged separately?

A: They are packaged separately so that the user can control when the reaction between the parts occurs.

5. What happens if you heat the cured sample?

A: When the sample is heated in boiling water or over a flame, it does not soften as a thermoplastic would. If heated for long over an open flame, the sample will char.

Extensions and Variations

- Heat the cured sample in boiling water or a small piece in a flame in the fume hood. (This could be a demonstration to cut down on the fumes.)
- The normal mode (%-transmittance) of IR spectroscopy is not quantitative. If the IR is run on Absorbance mode (like visible Beer's Law spectra), quantitative results for use on a graph of amount of epoxide vs. time (or alcohol vs. time, etc.) will be obtained.

References

Dewprashad, B. and Eisenbraun, E. J. "Fundamentals of Epoxy Formulation" *J.Chem.Educ.* **1994**, *71*, 290-294.

Department of Polymer Science at the University of Southern Mississippi Web Site, the Macrogalleria, Epoxy Resins; http://www.psrc.usm.edu/macrog/epoxy.html (accessed 25 September 1997 and 7 May 1998).

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