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Preparing Samples for the Recording of Infrared Spectra from Synthetic Fibers

Infrared spectroscopy is widely used in forensic science laboratories to identify and compare synthetic textile fibers. It is particularly useful for differentiating between various types of acrylic or polyester and polyester/polyether fibers where copolymerization or the introduction of new polymer types has been used to modify physical properties or improve dyeing characteristics.

There are various well-established methods of preparing fiber samples for recording infrared spectra, but modifications of the technique are generally necessary for different varieties and amounts of fiber.

Probably the most common methods are to prepare either a potassium bromide (KBr) or a potassium chloride (KCl) pellet [1-4] of 13-mm size or to prepare a microdisk pellet of 1.5-mm diameter with an Ultra-micro KBr die assembly (Perkin-Elmer Ltd.) by holding the fibers over the die and cutting minute fragments of fiber into the KBr/KCl powder. The distribution of the chopped material in the halide powder cannot be controlled and may cause scattering; but this can be improved by grinding the sample and powder in a vibrating mill (Perkin-Elmer Ltd.). In forensic casework the amount of suspect material is often extremely limited, and therefore the preparative method must be optimally applicable to the smallest amounts of material, even a single fiber, a few millimeters in length. However, where such a sample is irreplaceable the handling, cutting, and transfer of a single fiber becomes a hazardous operation.

The preparation of mulls [3,4] is a more tedious process and requires greater experience to produce a mull that will yield a good spectrum. It is a difficult procedure to apply to very small samples, and the mulling medium may produce interfering absorptions.

Another widely used method of sample preparation is the solvent-film technique [3,4, pp. 135-136] where films are cast from a chosen solvent onto a glass surface, and the film can then be peeled off and inserted directly into the specimen holder of the recording machine. Generally, solvent films provide good spectra, but the method depends on selection of a suitable solvent, which in turn requires preliminary identification of the type of fiber being dealt with, introducing more handling problems. Different fiber types require different treatment for film preparation or solvent removal, which may be difficult and time consuming, and the analyst has to be familiar with all variations. The solvent must not react with the fibers, nor may it leave any residue on evaporation. With microfiber samples, a halide disk still has to be made from the chopped-up solvent film, making a two-stage process.

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Method

The suggested method is derived from the techniques described above. Previously published data [3, pp. 24–38, 5] reveals that many of the generic types of fiber [6] in common use are soluble in phenol (for example, acetate, triacetate, polyamide, vinyl alcohol) or chlorinated hydrocarbons (chlorofibers) or a mixture of both (polyesters, polyethylene); on this basis a mixture of phenol and chloroform, 10% w/v was selected for trial as a common solvent.

The fiber is dissolved conveniently in the well of a spot test plate, which allows observation under a low-power binocular microscope, a particular help in dealing with minute amounts. A drop of the phenol/chloroform mixture is added, followed by a small amount of spectroscopic grade KBr powder (Uvasol®, Merck, Sharp & Dohme). Some fibers dissolve readily, others require grinding and further addition of solvent mixture. Grinding is conveniently carried out with the sealed end of a small test tube used as a pestle. If necessary the spot plate can be gently warmed on a hot plate to aid dissolution. Partially dissolved or undissolved fibers generally form a mat with the KBr powder which can be cut or separated in the spot plate well by scalpel blade under the microscope.

The homogeneous powder-sample mixture should then be dried in an oven to evaporate all traces of solvent mixture. Any phenol residue is readily detectable by its odor. Heating at 100°C (212°F) for 1 h is more than sufficient to remove all traces. A blank KBr disk thus prepared showed complete absence of any absorption bands.

The 1.5-mm KBr disks were prepared immediately after removal of the sample from the oven by using a Perkin-Elmer Ultra-Micro Die 186-0007. Sample disks were prepared under vacuum for 3 or 4 min by using a Carver hydraulic press and compressing the spring base of the microdie until the posts touched the die housing (about 400 lb or 180 kg pressure). This is sufficient pressure to give a satisfactory disk, and further compression may damage the die. Spectra were recorded using a Perkin-Elmer 621 Spectrophotometer with a 1 by 4 refracting beam condenser, scanning from 4000 to 600 cm^{-1} . The reference beam was usually set to between 60 and 70% transmission and scanning time was 16 min. Using the above method satisfactory spectra were recorded from the following types of fiber:

- acrylonitrile/methylacrylate,
- acrylonitrile/vinyl acetate,
- acrylonitrile/methylmethacrylate,
- cellulose acetate,
- cellulose triacetate,
- chlorofibers,
- modified acrylic,
- polyamide—nylon 6, 66, 11, Nomex®, and Qiana®,
- polyester—polyethylene terephthalate, poly-1, 4 cyclohexylene-dimethylene terephthalate, polyester/polyether co-polymers,
- polyethylene,
- polypropylene,
- viscose rayon, and
- vinyl alcohol.

No other examples of acrylic fiber types [2] were available.

The technique has definite advantages over other methods. First, the use of a “universal” solvent suitable for all the commonly encountered synthetic fiber types allows infrared spectra to be recorded directly without any preliminary identification work to decide on choice of solvent. Second, pellets are prepared directly from dried slurry of

solvent/fiber and KBr; the solvent causes total or partial dissolution of the fiber and gives better distribution onto the KBr particles. Third, the preparation of the slurry can be carried out under a low-power binocular microscope facilitating the handling of very small samples. Finally, the process is direct, standardized, and simple to operate, particularly for a small laboratory which only infrequently runs fiber spectra.

Spectra from over 70 different fibers were recorded. These were chosen as representative of the various groups. Figure 1 shows the recordings for ten of these fibers. They were recorded by using amounts of fiber comparable to those used by Fox and Schuetzman [1] with superior results. Reruns provided no reproducibility problems, which was a difficulty initially encountered using other pelleting methods.

Summary

An improved technique for recording infrared spectra from minute amounts of synthetic fibers by using KBr micropellets is described. The procedure is the same for all commonly encountered synthetic fiber varieties; hence, no preliminary identification is required.

References

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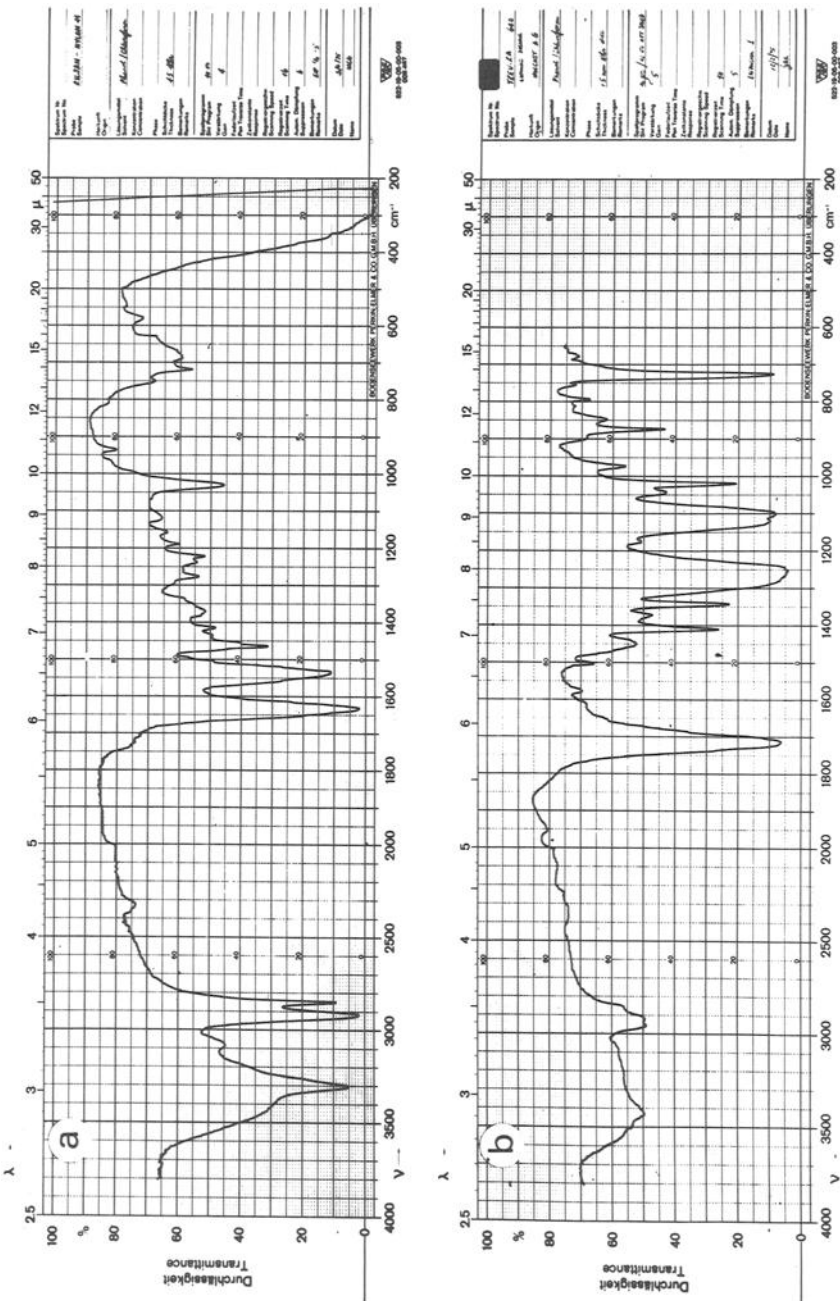


FIG. 1—Infrared spectra data for commonly encountered synthetic fibers. (a) Rilsan® (nylon 11) and (b) Trevira® 640 (polyester).

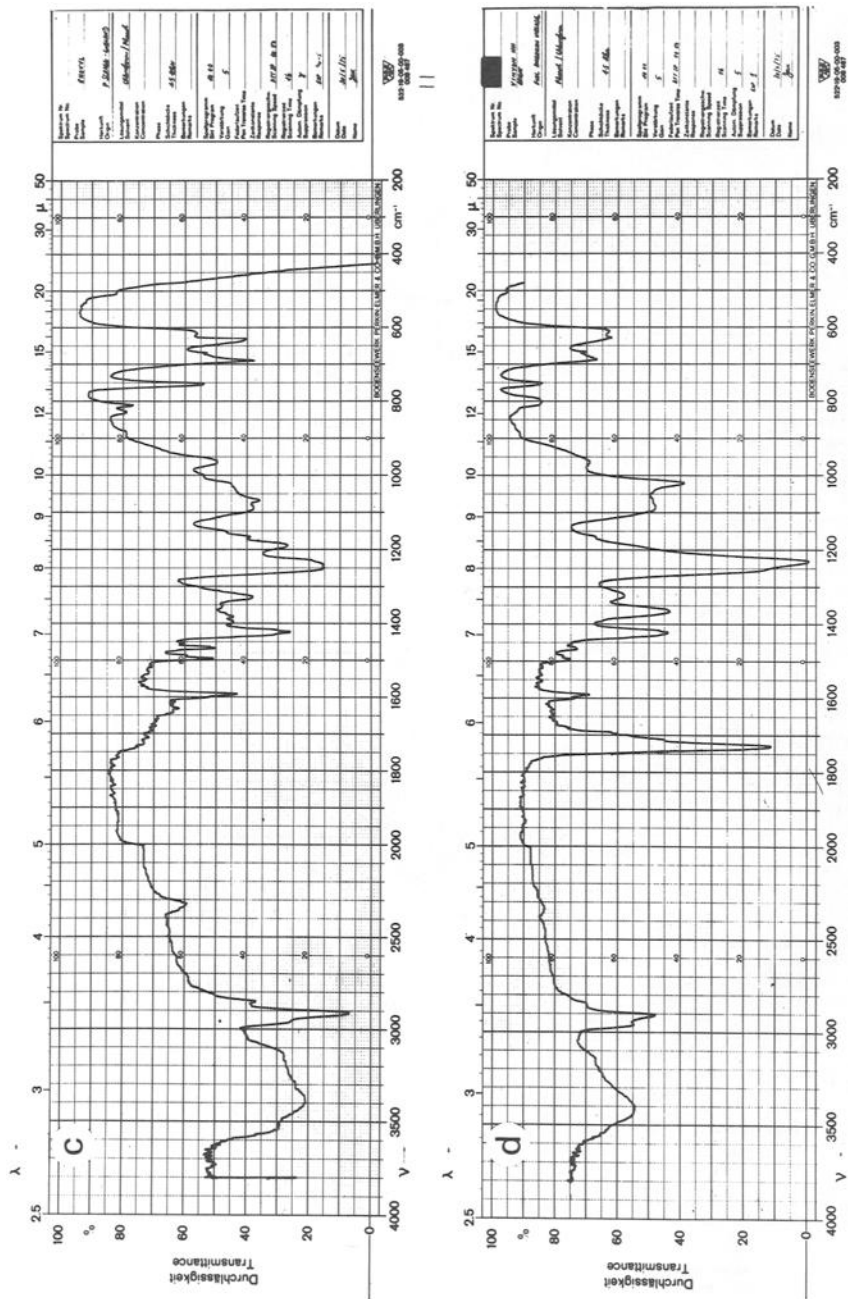


FIG. 1—(c) Rhovyl® and (d) Vinyon® HH (both chlorofibers).

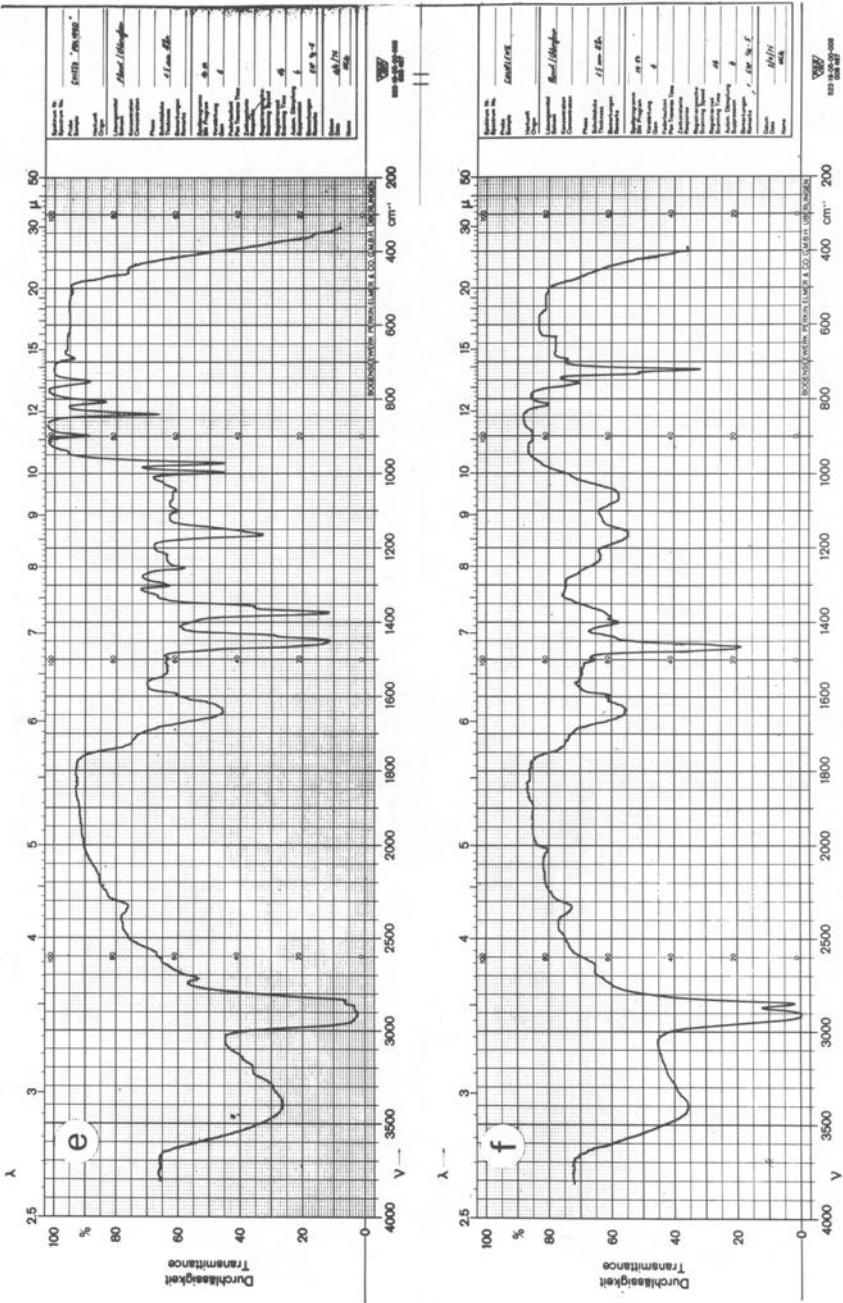


FIG. 1—(c) Chisso Polypyro® (polypropylene) and (f) Courlene® (polyethylene).

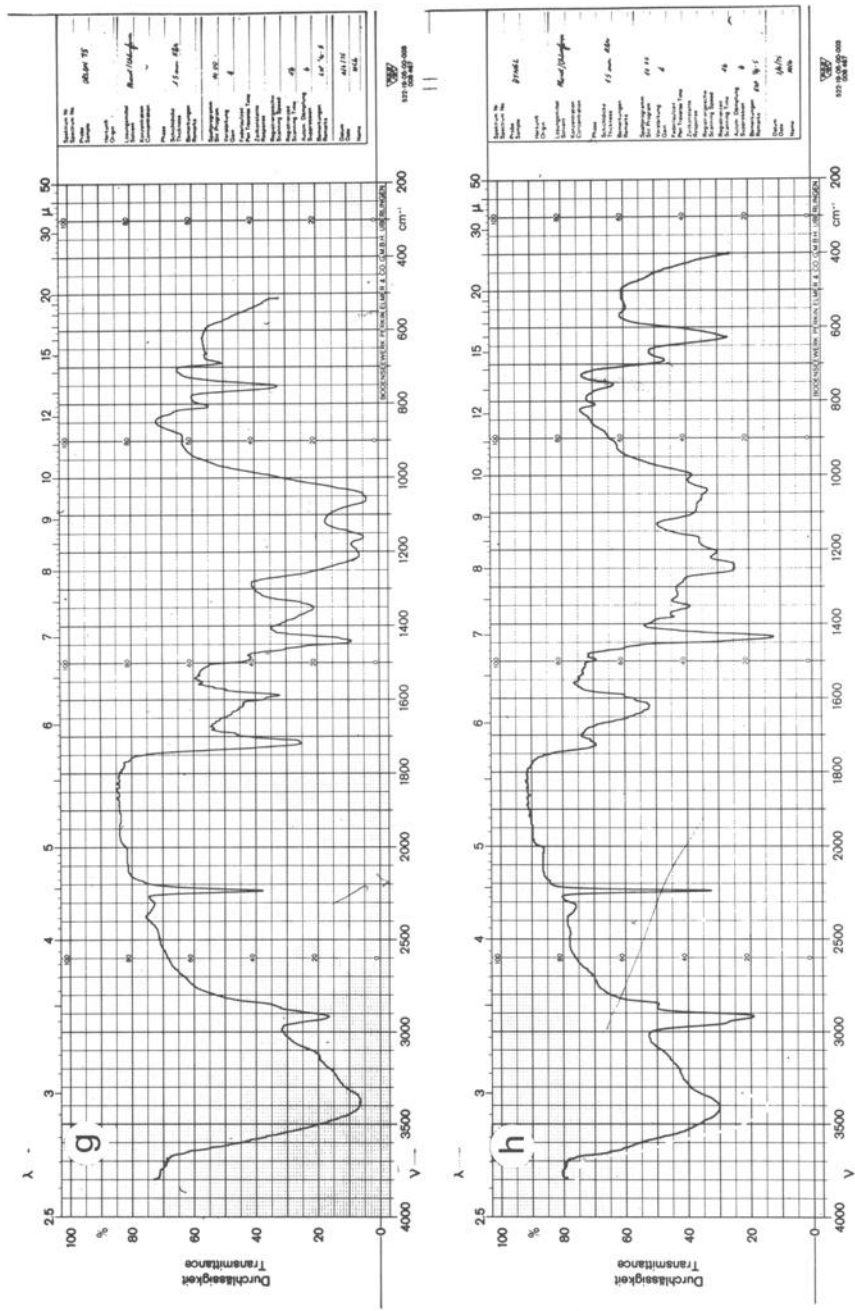


FIG. 1—(g) Orlon® 75 (acrylonitrile/methyl acrylate) and (h) Dynel® (acrylonitrile/vinyl chloride).

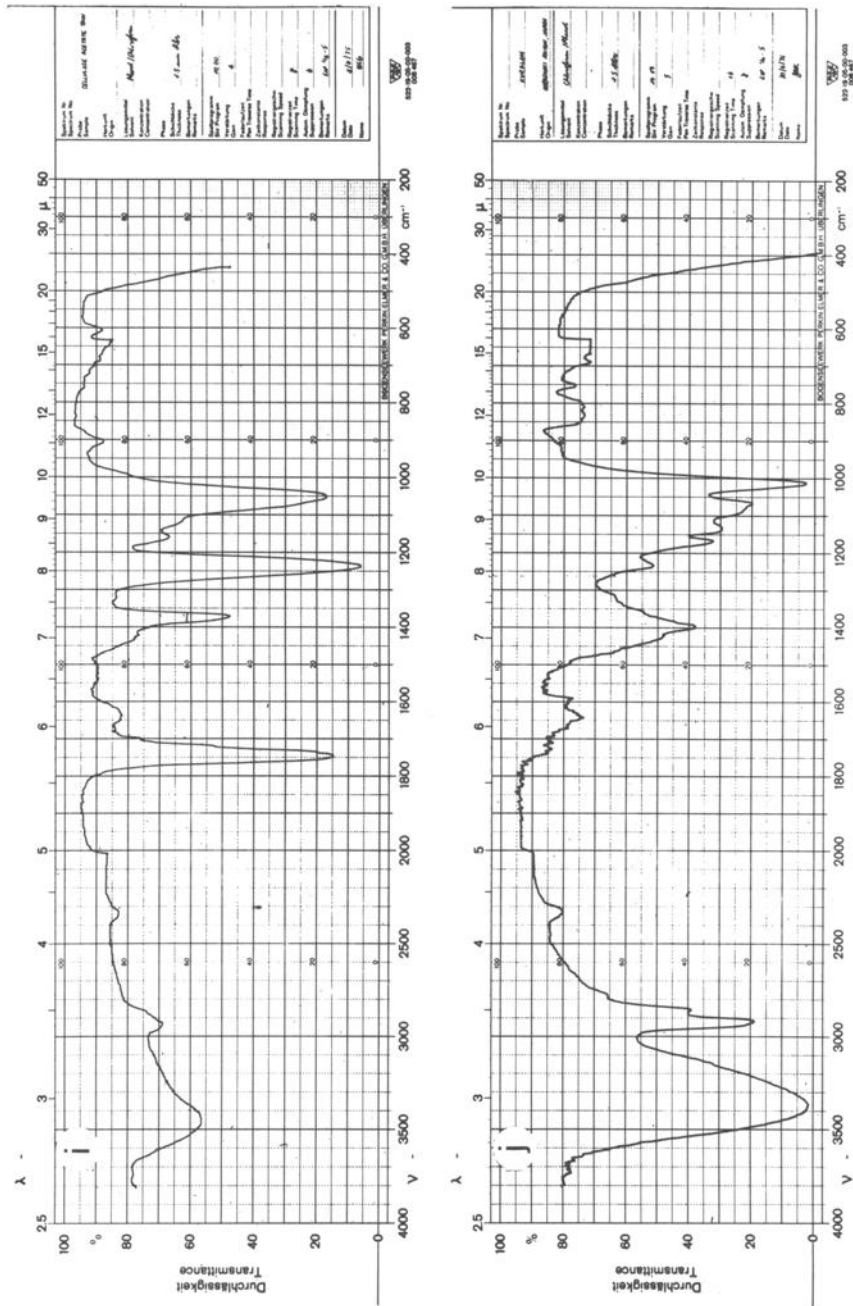


FIG. 1—(i) Cellulose acetate and (j) Kuralon® (vinyl alcohol).