TECHNICAL NOTE

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Examination for Petrolatum Based Lubricants in Evidence from Rapes and Sodomies

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ABSTRACT: The detection of petrolatum based lubricants on evidence submissions from rapes and forcible sodomies and their extraction and comparison with standards can help to substantiate the victim's allegation. Sixteen commercial products having a petrolatum base were uniquely distinguished by the combination of gas-liquid chromatography and synchronous fluorescence spectroscopy, and as little as 0.5 mg of product could be identified.

KEYWORDS: criminalistics, petroleum products, criminal sex offenses, sodomy

During 1981 the U.S. Army Criminal Investigation Laboratory-Europe received evidence from nine cases of rape or forcible sodomy in which lubricants were allegedly used.

In a typical case the assailant and victim are known to each other and the assault may take place in the room of either. Therefore the question is not one of identifying a suspect, but of seeing if the physical evidence supports the victim's allegation. Lubricants for such acts are normally taken from what is readily available such as petrolatum based jellies and hair preparations.

Petrolatum was first introduced in 1872 when a U.S. patent was issued to Robert A. Chesebrough for his product Vaseline® which is still a registered trademark of Chesebrough-Ponds, Inc. [*1*]. Also known as petroleum jelly and cosmoline, petrolatum is a semi-solid mixture of hydrocarbons obtained from petroleum.

Listed in Table 1 are the 16 petrolatum based commercial products that were examined. These were either purchased in the U.S. Armed Forces Post Exchange in Frankfurt am Main, West Germany, or were taken from actual case submissions.

Experimental Procedure

Infrared spectroscopy was of little value for comparison purposes, as the various products showed few differences (Fig. 1).

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TABLE 1—*Petrolatum based commercial products examined.*

Sample	Product
1	Afro Sheen Conditioner & Hair Dress®
2	Dark & Lovely Protein Hair Dress/Conditioner®
3	Dixie Peach Pomade®
4	Grow Aid Medicated Hair Treatment®
5	Pameco Petroleum Jelly®
6	PermaStrate Hair Sheen®
7	Posner Bergamot Conditioner & Hair Groom®
8	Pro-Line Coconut Oil Conditioner®
9	Pro-Line Conditioner & Hair Dress®
10	Revlon Realistic Scalp Conditioner & Hairdress® (For Extra Dry Hair)
11	Sulfur-8 Medicated Hair & Scalp Conditioner®
12	Ultra Sheen Conditioner & Hair Dress®
13	Ultra Sheen Conditioner & Hair Dress® (For Extra Dry Hair)
14	Vaseline Pure Petroleum Jelly®
15	Vigorol 8 Peloderm Ointment®
16	Vi-Jon White Petroleum Jelly®

Proton magnetic resonance spectroscopy gave even less information, with only a peak in the area of 1.3 ppm corresponding to the methylene protons [$—CH_2—$], and a smaller peak around 1 ppm corresponding to the methyl protons [$—CH_3$].

Fluorescence Spectroscopy

Fluorescence spectroscopy is routinely used to help identify the source of an oil spill [*2–4*]. Since petrolatum is obtained from petroleum it should also contain fluorescent components. If the petrolatum in different sample lots was obtained from different sources of crude oil there should be differences in their fluorescence patterns.

The technique of synchronous fluorescence was first introduced by Lloyd [*5*] in 1971, and in 1976 was used by John and Soutar [*6*] for the identification of crude oils. In 1980 Lloyd et al [*7*] cited a case of "buggery" in which synchronous fluorescence was used to compare the residue on an anal swab from the victim with a sample of Vaseline® found in the possession of the suspect.

A scanning interval of 20 nm was used for all synchronous measurements. Spectra were recorded with a Perkin-Elmer MPF-3 fluorescence spectrometer and were uncorrected for the spectral sensitivity distribution of the instrument. Instrument conditions were the same as reported previously [*8*].

Samples were dissolved in spectroscopic grade cyclohexane (Merck) and transferred to a 10-mm square cuvette. After a scan by hand to find the maximum, samples were diluted with cyclohexane until a maximum fluorescence intensity of 70 to 100% full scale was obtained at an instrument sensitivity setting of 10. Samples must be compared at dilute concentrations since very concentrated solutions will produce distorted spectra because of internal quenching (Fig. 2). For dilute solutions these effects are negligible [*3*].

As with crude oils and other petroleum products [*3*], the fluorescence patterns obtained from different petrolatum samples were similar in general shape yet showed small but reproducible differences in the relative intensities of the various maxima and shoulders (Fig. 3).

Gas-Liquid Chromatography

The cyclohexane sample solutions used for fluorescence spectroscopy were evaporated to just a few drops and used for gas-liquid chromatography (GLC). Reproducibility of the chro-

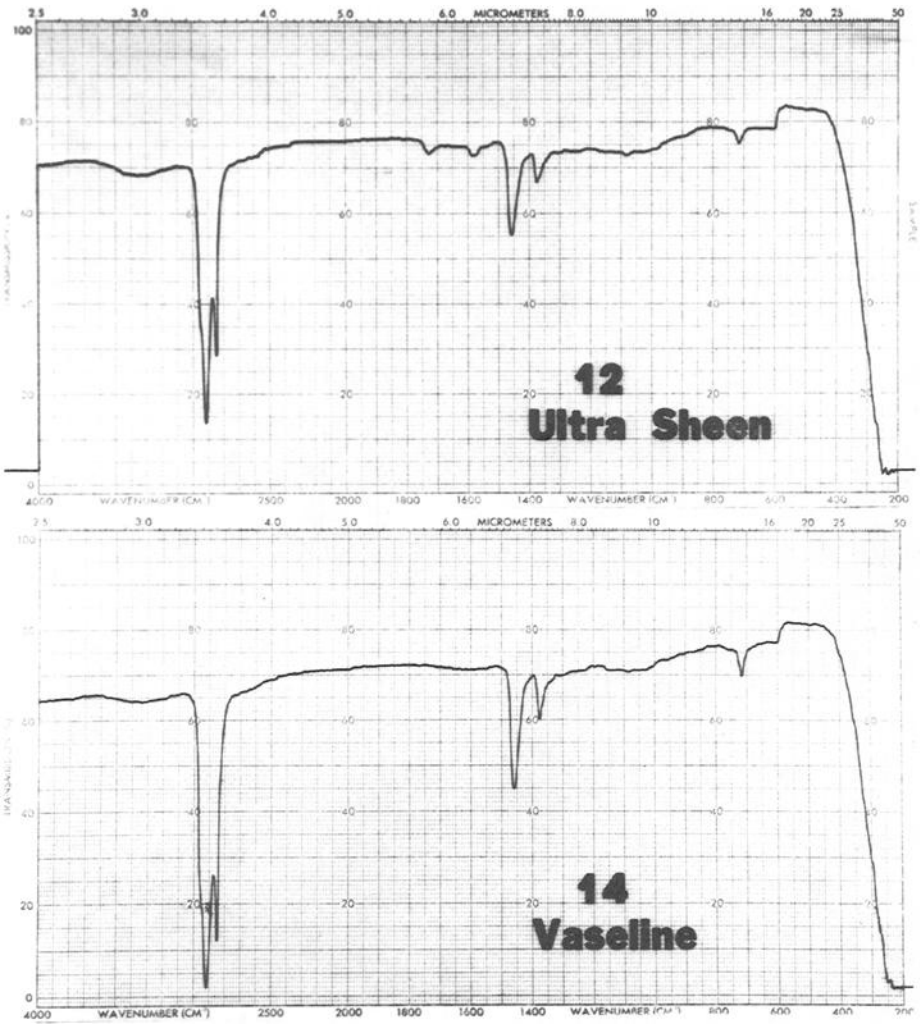

FIG. 1—*Infrared spectra of Ultra Sheen and Vaseline.*

matograms was checked daily by running a hydrocarbon test mixture (Polyscience Corp., Niles, IL, Kit No. 26A). An internal standard, *n*-nonadecane (n-C_{19}), was added to preliminary samples to calculate relative retention times. Table 2 lists the instrument conditions.

Figure 4 shows the GLC pattern obtained from Vigorol 8 Peloderm Ointment® (with n-C_{19} internal standard) compared to that obtained from the hydrocarbon test mixture. Comparison of the GLC patterns reveals that the majority of the hydrocarbons within the sample range from C_{15} to C_{35}.

Products of different manufacturers yielded chromatograms that were generally easily distinguishable. Figure 5 shows the GLC patterns obtained from Vi-Jon White Petroleum Jelly® (with n-C_{19} internal standard) and Pro-Line Conditioner & Hair Dress®. Different products from the same manufacturer gave very similar patterns. Samples 8 and 9 (Pro-Line) appeared identical whereas Samples 12 and 13 (Ultra Sheen®) could be distinguished by one large fraction found in 13 which was absent in 12.

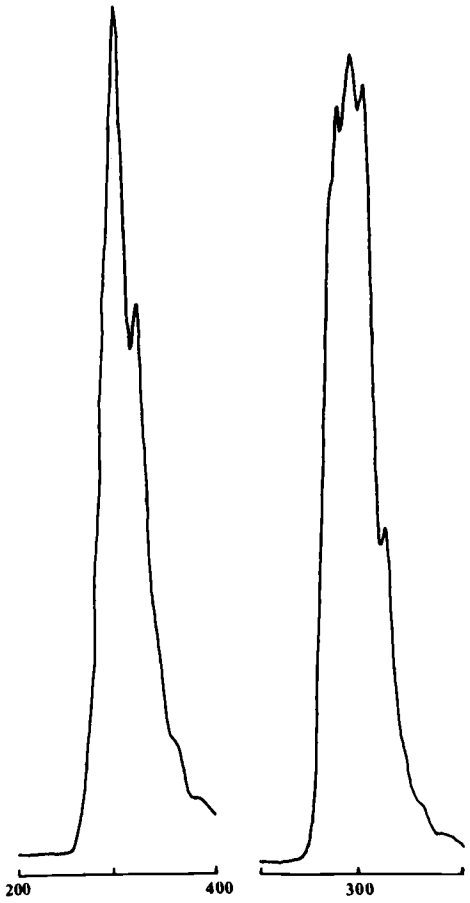

FIG. 2—*Synchronous fluorescence spectra of the same Vaseline sample in cyclohexane at different concentrations. Concentrated* (left), *sample sensitivity = 1, and dilute* (right) *sample sensitivity = 10. Both synchronously excited at an interval of 20 nm.*

Reproducibility

With both synchronous fluorescence and GLC no changes in patterns were observed for time periods up to one month. Small amounts of Vaseline Pure Petroleum Jelly® were rubbed into numerous cloth swatches which had been cut from a clean laboratory towel. Half of these swatches were stored in the dark and the remainder were kept in the open where they were exposed to fluorescent light and diffuse sunlight. At intervals of one day, two days, one week, two weeks, and one month swatches were extracted with cyclohexane and these solutions examined by synchronous fluorescence. No changes in fluorescence patterns were noted regardless of elapsed time or method of storage. These results are consistent with the work of Thruston and Knight [*3*] who found that "low-boiling volatiles in crude oils showed no fluorescing properties" and that "no analytical problems resulted when volatiles were removed by evaporation before analysis." No tests involving prolonged exposure to direct sunlight were attempted.

No significant GLC pattern changes were observed with several samples that were rerun after being left open to the air at room temperature for one month. Yip and Clair [*9*] showed

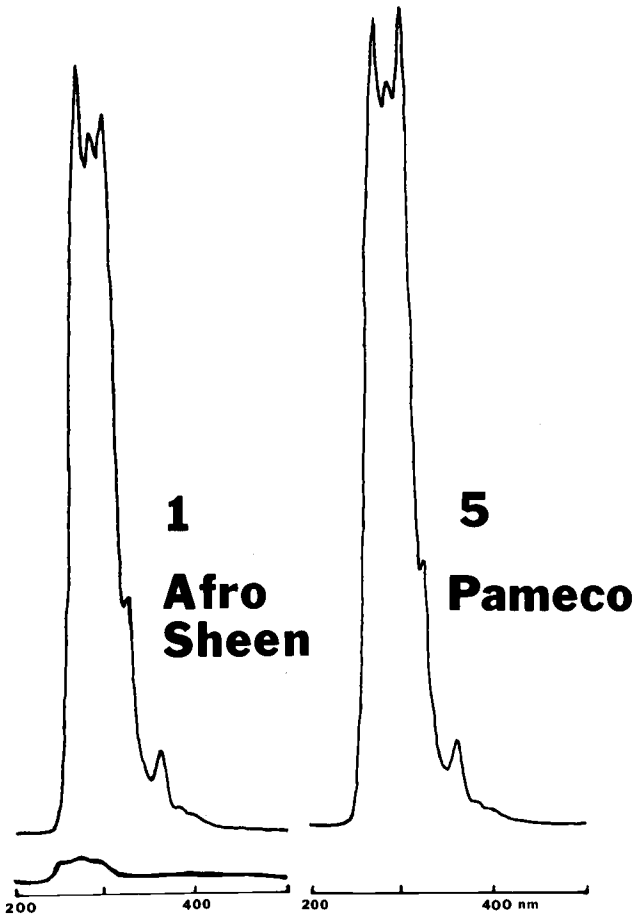

FIG. 3—*Examples of synchronous fluorescence spectra of petrolatum based products in cyclohexane. All at sample sensitivity = 10, and synchronously excited at an interval of 20 nm. Bottom left is neat cyclohexane.*

TABLE 2—*Gas-liquid chromatography conditions.*

Instrument:	Perkin-Elmer (P-E) 3920B with flame ionization detector, P-E 56 recorder, and P-E Sigma 10B Data Station.
Column:	Glass column 1.8-m by 2-mm inside diameter packed with 3% Dexsil 300 on 80-100 mesh Chromosorb W, acid washed.
Carrier gas:	Helium at 30 mL/min.
Chart speed:	10 mm/min.
Temperature:	Injector and detector both at 325°C and a temperature program of 140°C isothermal for 2 min and then increased at 16°C/min up to 300°C.

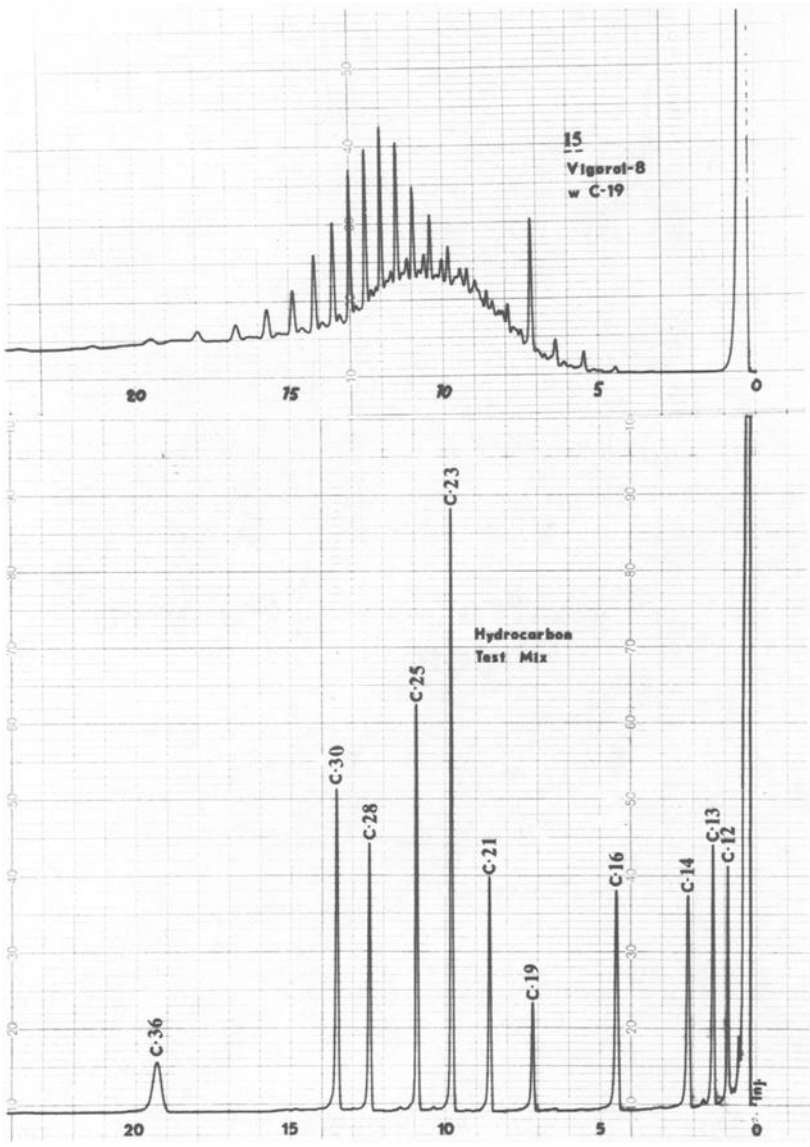

FIG. 4—*Gas chromatograms of hydrocarbon test mix and Vigorol 8 Peloderm Ointment.*

that the vapor pressure of any *n*-paraffin was a function of its temperature and that no appreciable sample loss would be expected of carbon numbers higher than dodecane (C_{12}) at room temperature (20°C) because of their low vapor pressure (less than 0.1 mm of mercury for C_{12}).

Comparison of Different Sample Lots

Members of the laboratory staff contributed six jars of Vaseline Pure Petroleum Jelly that had been purchased at various locations over a period of several years and should therefore

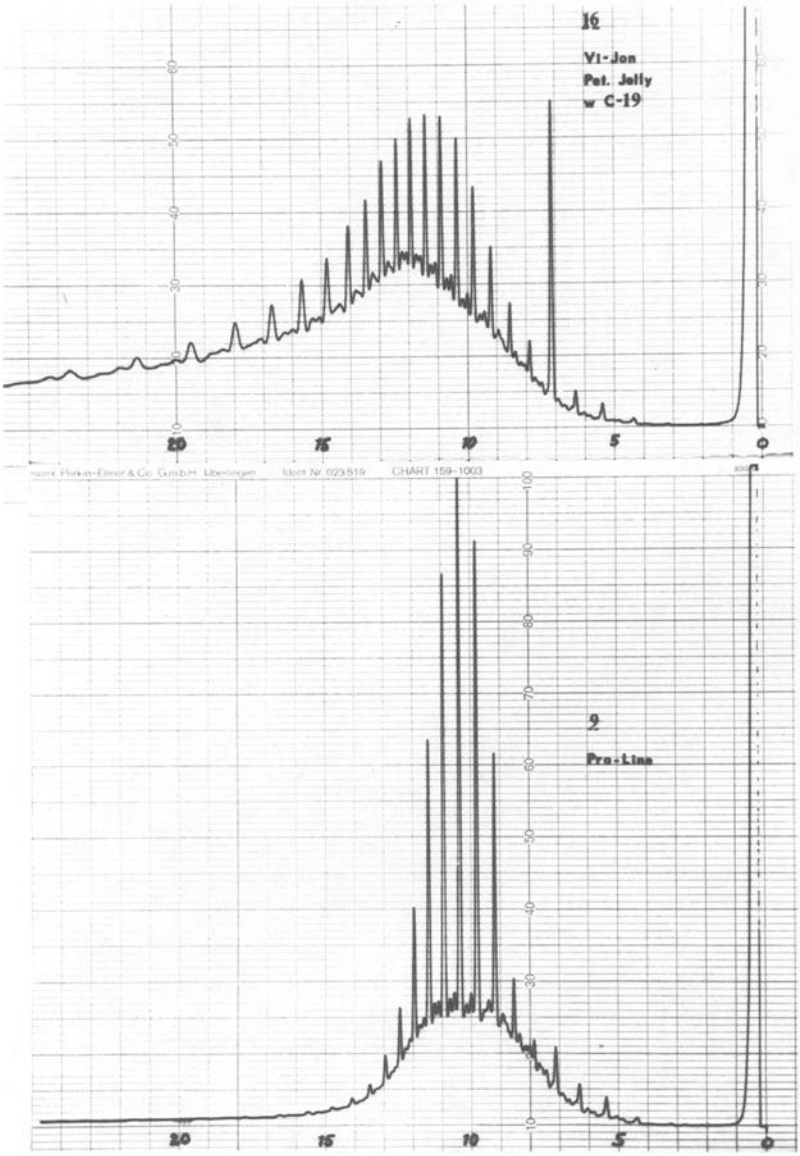

FIG. 5—*Gas chromatograms of Vi-Jon White Petroleum Jelly and Pro-Line Conditioner & Hair Dress.*

represent at least several different sample lots. Vaseline was used for sample lot testing because of its availability and because of its frequent appearance in actual case work. The six samples produced four different synchronous fluoresence patterns, two of which are shown in Fig. 6. GLC patterns tended to be very similar for all six samples (Fig. 7).

Interferences

With synchronous fluorescence no interference was encountered from semen, blood, urine, saliva, perspiration, mucous, or fecal materials, but sizing and detergent residues in

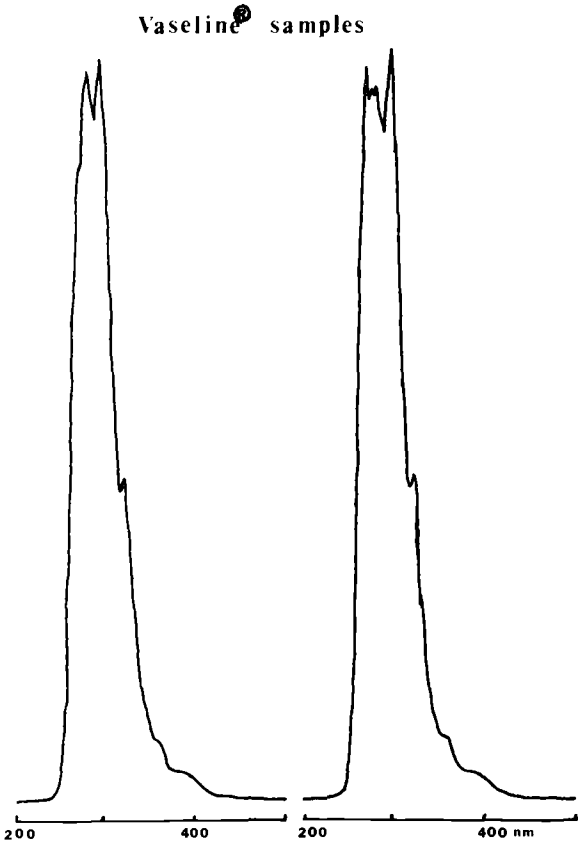

FIG. 6—*Synchronous fluorescence spectra from two different lots of Vaseline Pure Petroleum Jelly. Synchronously excited at an interval of 20 nm and at sample sensitivity = 10.*

fabrics may produce interfering fluorescence. Because of sizing, more interference may be encountered in new garments than those that have been laundered numerous times; however, interference is not encountered in every case. Figure 8 shows the synchronous fluorescence results from four separate cases.

With GLC any substance that is soluble in cyclohexane and volatilized in the injection port can produce additional peaks, but we seldom see enough additional or interfering peaks to obliterate the entire pattern. Use of cyclohexane as the extracting solvent eliminated most water soluble substances, and gave suitable extraction of the petrolatum based lubricants. A cyclohexane extraction from a control area of the exhibit usually eliminates any extraneous peaks. Figure 9 shows the GLC results from the sodomy case referred to in Fig. 8*a*. The volatile components are unreliable for comparison, but from about 6 min on there is good agreement between the two samples except for the major peak eluting just before 10 min. From examination of control samples taken from other areas on the garment it was apparent that an unidentified material present throughout the garment contributed to this peak.

Results and Discussion

Together, synchronous fluorescence and GLC were uniquely able to characterize the 16 commercial samples and identify as little as 0.5 mg of sample.

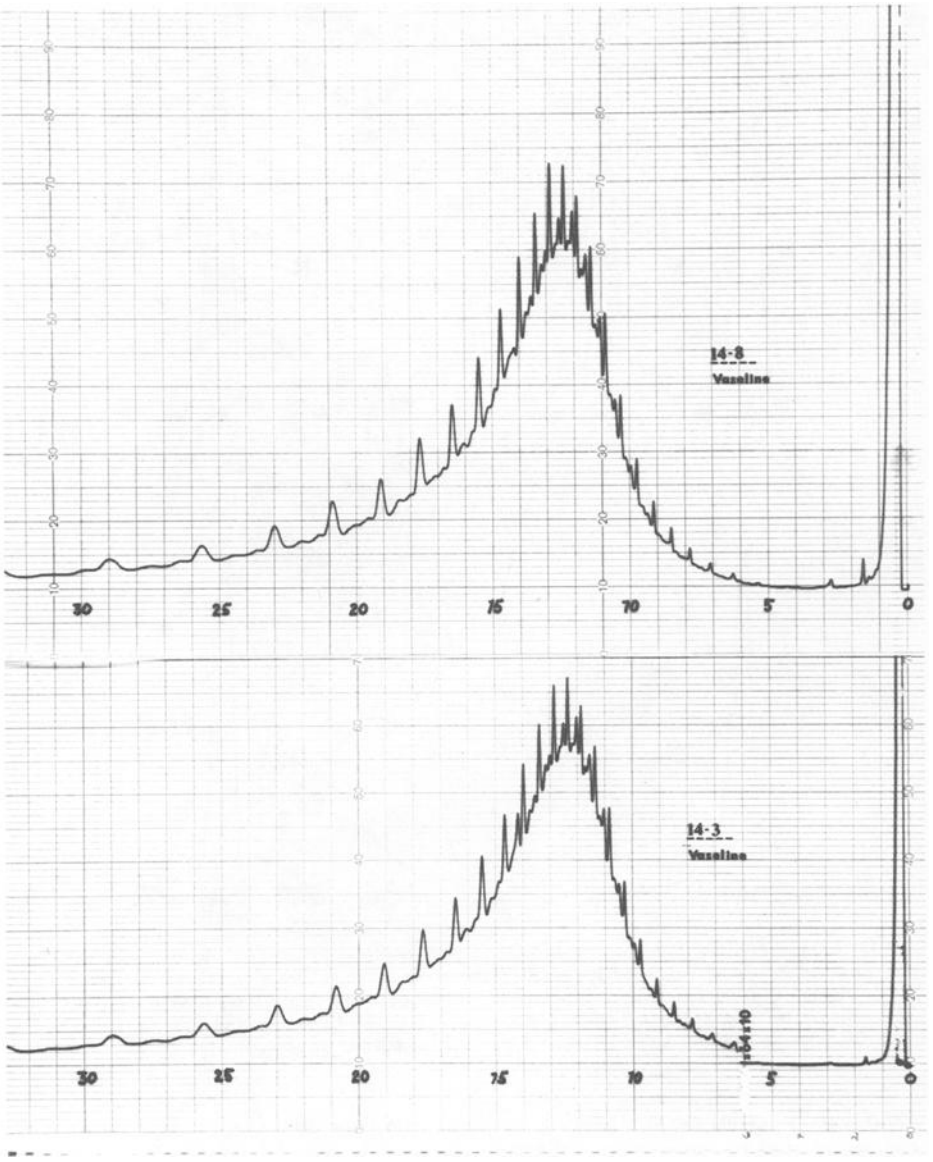

FIG. 7—*Gas chromatograms of two different sample lots of Vaseline Pure Petroleum Jelly.*

Evidence submissions in actual cases may include various swabs, assorted clothing, bedding, and the suspected lubricant containers with contents. The examination for lubricants is begun only after all the usual serological examinations have been completed.

Suspected lubricant stains are extracted with spectroscopic grade cyclohexane and these extractions are used for all subsequent tests. As a control, some areas on garments and bedding where stains appear to be absent are also extracted. Extractions are performed by placing the garment so that the suspected area forms a depression over the mouth of a beaker. The cyclohexane is then dripped over this area and collected in the beaker. This efficiently extracts any petrolatum and fewer interfering fluorescent materials are removed from the

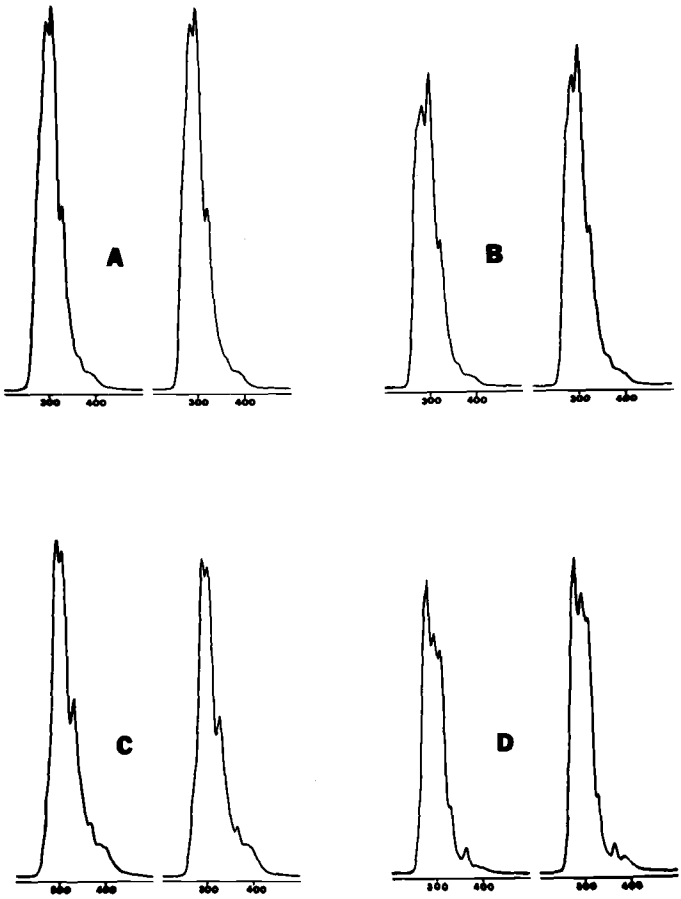

FIG. 8—*Synchronous fluorescence spectra (20-nm scanning interval) from four different rape/sodomy cases:* (a) *from Vaseline jar from suspect's room* (left) *and from anus area of victim's undershorts* (right); (b) *from Vaseline jar from suspect's room* (left) *and from crotch area of suspect's undershorts* (right); (c) *from Sulfur-8 jar from suspect's room* (left) *and from a bed sheet stain* (right); *and* (d) *from Ultra Sheen jar from suspect's room* (left), *and from a bed sheet stain* (right).

cloth than by cutting out swatches and soaking them. For cotton swabs having plastic shafts, the cotton must be removed from the shaft before soaking in cyclohexane.

Locating stains may be difficult. Cotton swabs containing petrolatum will fluoresce when examined under short wave (254-nm) ultraviolet light, but on fabrics the results are uncertain. Many light colored fabrics will also fluoresce and petrolatum stains may even appear as shadowy areas. If stains cannot be located visually then extractions are conducted at the most logical areas on undergarments and also any areas that gave positive results for acid phosphatase.

Summary

Petrolatum based lubricants may be used in cases of rape of forcible sodomy. Their location on evidence materials, extraction, and comparison with standards can help to substantiate the victim's allegation.

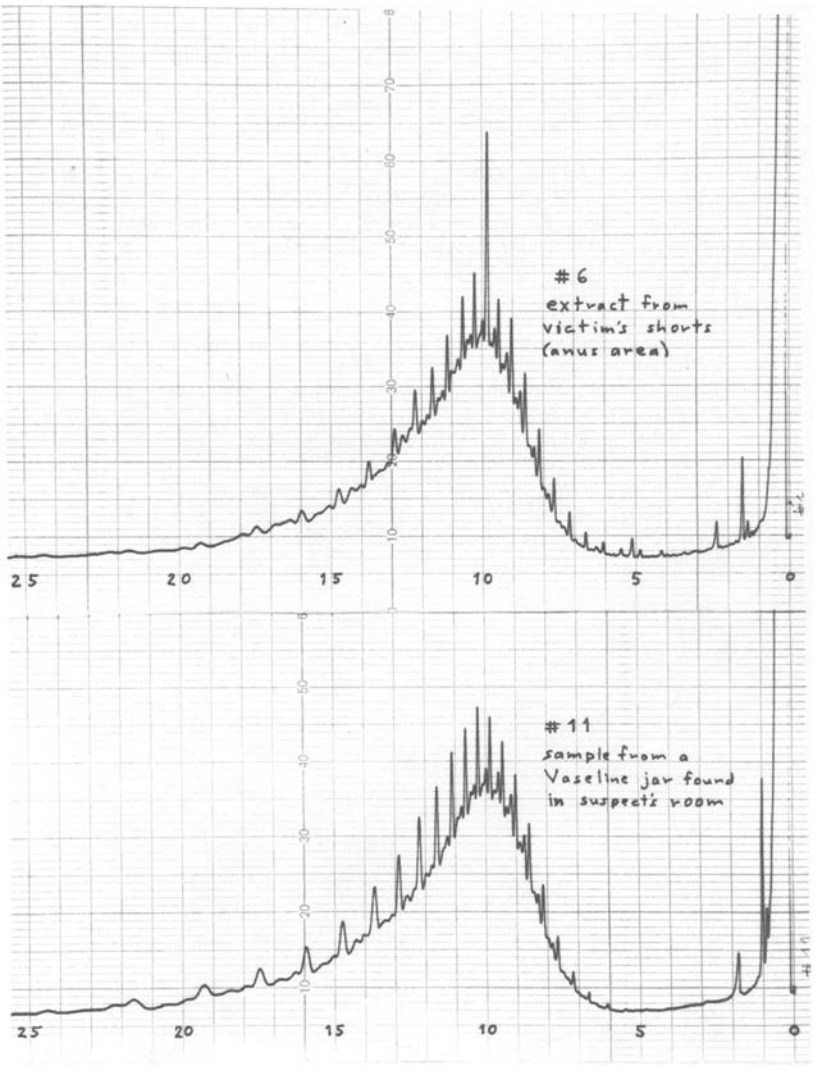

FIG. 9—*Gas chromatograms of cyclohexane extracts of sodomy victim's shorts and from a jar of Vaseline Pure Petroleum Jelly which was found in the suspect's room.*

Using a cyclohexane solvent 16 commerical products having a petrolatum base were examined by synchronous fluorescence spectroscopy and GLC.

GLC was generally characteristic of specific commerical products and showed little variation with different sample lots. Synchronous fluorescence was characteristic of the source of crude oil from which the petrolatum was obtained, and was therefore characteristic of the sample lot rather than of specific commercial products. The two methods together were able to uniquely characterize the 16 samples, and as little as 0.5 mg of sample could be identified.

The two methods have been used in the examination of evidence materials, and although interferences may be encountered, they are usually insufficient to prevent a comparison.

References

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