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## Traces of polymethylsiloxane in case histories of rape: technique for detection

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## Abstract

A number of case histories were recently brought to our attention which were intriguing professionally, yet presented difficulties in proving positive results: reported rape by an agressor wearing a condom. We undertook an indepth analysis of the chemical components of condoms and selected the lubricant for further study. In chemical terms, the silicone oil used, polymethylsiloxane, is clear, colorless and odorless. In this paper, we describe a technique for the detection of polymethylsiloxane. Given that the quantities of lubricant available, in vivo, were likely to be extremely small, we opted to use <sup>1</sup>H-NMR. Our results confirm the range for identifying polymethylsiloxane as between 0.0426–0.0440 ppm and two individual case histories gave figures of 0.0424 ppm and 0.0420 ppm. Further studies are underway on the length of time polymethylsiloxane remains on the victim or clothing and on different detection techniques

Keywords: Polymethylsiloxane; Rape; Detection technique; Case histories

## 1. Introduction

Requests for examinations in order to investigate and confirm cases of rape normally reach the Institute of Forensic and Insurance-Related Medicine of the University of Rome's 'La Sapienza' campus through one of two channels: the first

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is from the victim, in the form of a direct request for an examination; the second, is from the health-care department of the Umberto I Polyclinic, which can request consulting and/or support services from forensic specialists for emergency units of the hospital such as the Admittance Area, Clinical Gynecology, Clinical Pediatrics, etc.

In the specific case of Italy, there has been a sharp rise in the number of sexual crimes: in 1980 there were 925 cases of rape reported, while in 1993 the figure had risen to 1758. Inevitably, therefore, the clinical facilities of our Institute have also recorded a rise in the number consulting and/or examination requests in cases of rape. Also, the official data provide only a relative picture of the phenomena, given that many episodes of rape do not lead to a request for action on the part of the authorities with responsibility for such matters.

A number of case histories were recently brought to our attention. Though they proved to be especially intriguing from a professional point of view, they presented extremely limited opportunities for positive findings: some victims reported having been raped by an aggressor using a condom and, in a number of these cases, general/specific medical examinations produced negative results.

As expected, the classic examinations designed to detect whether or not seminal fluid is present, performed on the slim hope that the condom had broken, were negative.

In our opinion there are two principal motives which lead the attacker, during a rape, to use a condom: the first regards the risk of contagious diseases; the second. which should not be underestimated, is the wish to eliminate evidence, in particular the seminal fluid, which could play a decisive role during an eventual criminal trial.

We have undertaken an in-depth analysis of the chemical components of condoms, attempting to identify those substances which are common to all such products, although in different quantities, and specimens/samples of which could be taken during the forensic/medical examination, as is done for the seminal fluid.

It is important to note that there exists in Italy a specific decree issued by the Ministry of Health (January 26, 1991-G.U. of March 15, 1991), stipulating that prophylactics for males are to be subject to the rules and regulations issued by the country's medical institutes.

More specifically, art. 2, paragraphs 1 and 2, states: '...prophylactics for males are produced using natural rubber. Synthetic rubber can also be used... the prophylactics can be lubricated with medical-quality silicone oil. Other lubricants can be used, assuming they do not lead to allergies, irritations or other undesired reactions, and that they do not damage the product. Their use will be subject to a specific evaluation during the phase involving authorization of the prophylactic for sale...'.

The 'Appendices' of the same decree also state that: '...The presence in the lubricating solution of aromatic substances meant to cover the odor of the vulcanized rubber is permitted. Documentation demonstrating that the use of such substances does not lead to irritation or other undesirable reactions must be presented..'.

In Italy, there are ten manufacturers of prophylactics who put 47 products on the market in no less than 135 different types of packaging. Each package is distinguished not only by the number of condoms it holds, but also by their different structural characteristics, e.g. transparent, translucent, opaque, anatomical ultrasensitive/sensitive, with or without reservoir ultra-thin. They can also come in different colors and/or flavors, such as strawberry, apple and banana. Also, on a number of types, substances are applied to the surface to perform the following functions: delay, stimulation, extended action, triple-action. Our attention, however, is focused on one substance in particular: the lubricant.

Based on the information contained in the decree issued by the Minister of Health, the lubricant in question consists of medical-quality silicone oil. In response to queries during the preparation of our study, it was learned that the different manufacturers commonly use 'Dow Corning 200 fluids'. In the course of processing the different information, it was discovered that a number of firms, though their names are different, are associated with the same group and, therefore adopt similar basic criteria and components in the manufacture of prophylactics.

Research on the quantity of the substance originally applied to the inside and outside surface of the prophylactic has produced results which vary within a range of 200-400 mg as regards the total quantity of the substance.

In chemical terms, the silicone oil used is a polymethylsiloxane that is clear, colorless and odorless, having been synthesized through hydrolysis and polycondensation of dichlorodimethylsiloxane and chloromethylsiloxane, with a nominal kinetic viscosity, at 25°C, of 100 cS.

However, the most noteworthy characteristics of the substance is its insolubility in common liquids, together with the absence of significant modifications over time and the failure to react with other organic substances. Given that the lubricant possessed the parameters which we felt were important for the purposes of detection, it was chosen as the subject of the in vivo and in vitro experiments.

The first step was to select the best method of identifying the substance, which, normally, involves four possible approaches:

(a) kinematic viscosity at 25°C, in the form of an identification reaction obtained using a capillary and/or rotating viscometer;

(b) infra-red absorption spectrum, with the exception of the values from 850-750 cm<sup>-1</sup>, given that there can be differences traceable to the polymer length;

(c) 0.5 g of the substance heated over a flame, then combined in a second test tube with 1 ml of chromotropic acid sodium salt solution (1 g/l) in sulphuric acid and warmed in a water bath for 5 min until it takes on a violet colour;

(d) preparation of sulphuric ashes in a platinum crucible, using 50 mg of the substance; the residue obtained shows the reaction typical of silicates.

However, these methods were immediately rejected, because, although they are valid in the laboratory, they do not permit precise readings in real-life situations, given that the quantities of lubricating oil collected at the time of the examination could prove decidedly scarce.

Instead, we opted for the <sup>1</sup>H-NMR spectroscopy, using a Gemini Varian 200 MHz Fourier transformer spectrometer. We ran the initial trials on the pure

PPH

Fig. 1. Graph in which the main peak is due to an excell of polymethylsiloxane. Fig. 2. Double peaks—first corresponds to carbon tetrachloride; main peak corresponds to the silicone substance.

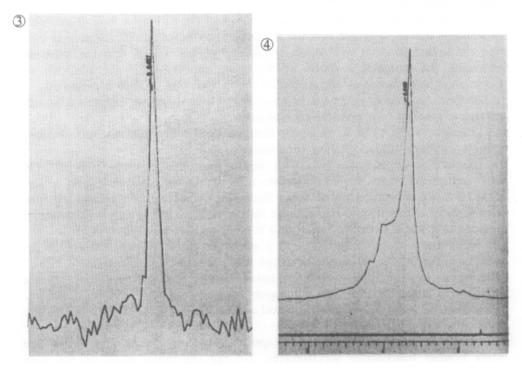


Fig. 3. Peak with dilution ratio equal 1 to 1000. Fig. 4. Peak of silicone substance obtained by rubbing surface of condom.

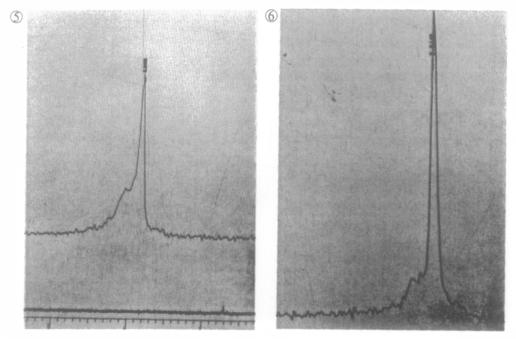


Fig. 5. Simple contact of 2s on the skin. Fig. 6. Contact of 2s on the skin followed by washing with running water.

chemical substance, the polymethylsiloxane which one of the manufacturers had kindly supplied us.

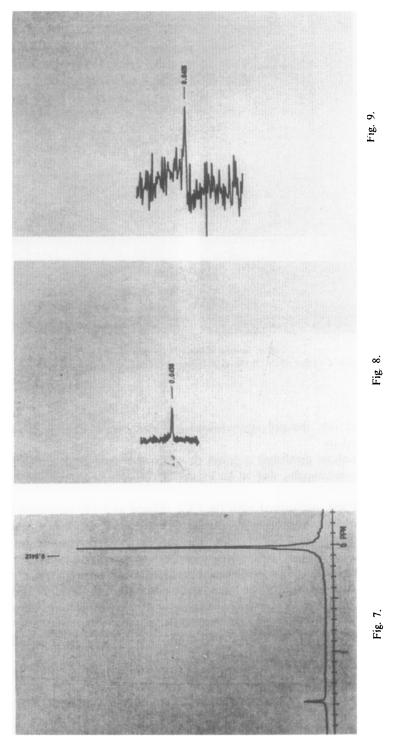
The first analysis produced a graph in which the main peak which fell outside the scale of measurement due to an excess of the substance, equaled 0.0552 ppm (Fig. 1).

The second test, performed with the  $CCl_4$  diluted at 1:100 produced two peaks; the first represented carbon tetrachloride (1.5167 ppm), while the second represented the silicone substance, which registered a value corresponding to 0.0462 ppm (Fig. 2).

When the dilution ratio was increased to 1:1000, the detection peak remained at 0.0467 ppm (Fig. 3). Additional trials, performed up to a set limit of 1:1 000 000 continued to give the same results. We purposely stopped at the above dilution because we felt the range to be exhaustive enough for the purposes of the research. Therefore, the figure in question was taken as the reference value for the identification of the substance.

All of the trials, both in vitro and in vivo, were performed using the standard technique, which always involved the following procedures:

- (a) taking of a sample using a wad of pure cotton (non-cellulose);
- (b) immersion of the cotton in 2  $\text{cm}^3$  of carbon tetrachloride (CCl<sub>4</sub>);
- (c) 0.1 ml of the resulting solution is mixed with 0.5 ml of CDCl<sub>3</sub>.



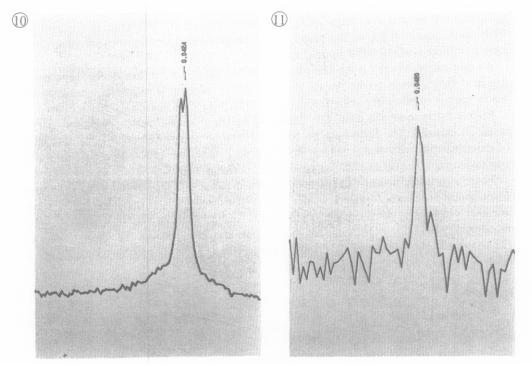


Fig. 10. Sampling immediately following the attack. Fig. 11. Sampling after a number of hours later, following these washings.

The next phase, again performed on an experimental basis involved a further series of tests on a variety of prophylactics, carried out using the following techniques: (a) prophylactic rubbed dry on a strip of skin approximately 5 cm long and 1 cm wide;

(b) simple contact of 2 s on the skin, having a surface area of  $\sim 2$  cm in diameter; with subsequent drying;

(c) contact of 2 s on skin, followed by washing with running water and drying. The results (Figs. 4-6) confirm the range for identifying polymethylsiloxane as falling between 0.0428 and 0.0440 ppm, with a typical wave shape.

Further tests, all of which produced positive results, were performed in order to detect possible interference by specific substances which, at times, are added for the purpose of delay stimulation, spermicidal effect or their aromatic favors (Figs. 7-9).

In moving from the experimental to the practical phase, the samples were always taken with a wad of pure cotton, such as those normally sold to the public; the use of classic pads on support backings was avoided, given that the adhesive could interfere with the results.

By way of example, we list two of the cases brought to our attention for detection research.

(1) sampling performed in situ immediately following the attack and showing a peak of 0.0424 ppm (Fig. 10);

(2) sampling in situ a number of hours after the assault, following two washings with water and a third with a detergent substance used for feminine hygiene; the figure obtained was 0.0420 ppm (Fig. 11).

As can easily be seen from the plotted curves, there are further peaks, representing not only carbon tetrachloride but also other organic substances, which in no way overlap or interfere with the proper performance of the analysis.

In our estimation the data can definitely be used to confirm the validity of the research method. The follow-up on this study, to be performed on a larger number of case histories, will also permit a more precise calculation of the length of time the substance remains on the victim or on the clothing worn.

There is no doubt that this identification technique does not fully cover the issue of polymethylsiloxane. Indeed, at the Rome Institute of Forensic Medicine, we are currently extending this type of detection effort to include the use of gas chromatography (GLC), experimenting with a specific HT5 model 'capillary column' ( $12 \text{ m} \times 0.32 \text{ mm}$  I.D.) set for high temperatures ( $360^{\circ}$ C). Further contributions on this subject are expected shortly.

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