## Collection Efficiency of Gunshot Residue (GSR) Particles from Hair and Hands Using Double-Side Adhesive Tape

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**ABSTRACT:** Experiments were carried out to sample GSR particles (GSRs) from hair using double-side adhesive coated aluminum stubs (the tape-lift method). Although it was claimed in the literature that double-side adhesive tape is not suitable for sampling GSRs from hair, we have not encountered problems with this method. In laboratory experiments it was possible to find GSRs even 24 h after shooting if the hair had not been washed. No significant difference in collection efficiency was found between this technique and the more complicated method of swabbing the hair using a comb with a solvent-damped cloth (the swabbing-and-comb method). It was found that 200 to 300 dabbings are necessary to achieve maximum collection efficiency from hair with the double-side adhesive. As assessed subjectively, it was noted that stickiness is lost after about 100 dabbings. This method has been used in casework in Israel for about a year and a half and, in some cases. GSRs were found in samples from hair but not in samples from hands. We have also studied collection efficiency from hands of the double-side adhesive. It was found that 50 to 100 dabbings are necessary to achieve maximum collection efficiency, while stickiness appeared lost after about 20 to 30 dabbings. No substantial danger of skin debris concealing GSRs was observed.

KEYWORDS: forensic science, criminalistics, gunshot residue collection techniques

Scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDX) is well-established for detection and identification of GSRs [1-5]. A major disadvantage of the SEM/EDX technique is the time needed for carrying out a test. The time required for scanning a single 13 mm diameter stub can be up to 8 hours [6]. One solution for this problem has been to automate the search and analysis procedures. Recently, a number of fully automatic methods, based on commercially available equipment, have been described [7-10]. An alternative and complementary approach to reduce the search time is to segregate and concentrate the GSRs onto a small area as described below.

GSR samples for SEM/EDX analyses are collected in a number of ways. They may be collected directly onto a specially treated sticky scanning electron microscope (SEM) stub kept previously uncontaminated [1, 6, 11-14]. These samples are then examined in the SEM. An alternative to direct sampling and examination is to collect the GSRs by using a swab or dabbing with a sticky surface to remove them from the subject's clothes, skin or both. After dissolution of the collecting medium or ultrasonic release, the GSRs

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are transferred to a filter which is, in turn, mounted on a SEM stub for examination [15,16]. This type of procedure also allows a pretreatment of the sample to remove interfering substances that hinder detection in the SEM. The particles may also be segregated from interfering substances by virtue of their greater density and then concentrated onto a small area [17,18]. However, it has already been shown that, in practice, there might be an intrinsic difficulty in concentrating GSRs on a small area due to pile-up and clogging of the filters used [19]. Concentration methods also involve considerable additional manipulation of the sample, which is time-consuming, requires skilled manpower and may well lead to loss or contamination. It has also been reported [20] that the tape-lift technique was considerably more efficient, with regard to the collection of particles, than the concentration methods [18]. A number of authors proposed vacuuming methods for collecting GSRs [21,22].

Although the probability of finding particles on hands more than three hours after firing is very low [23], most of the reported studies dealing with collection methods of GSRs recommend the hands of a suspect as the only sampling site. Commercial GSR sampling kits (for example, Lightning Powder Co. Inc., 1230 Host Street, S.E. Salem, Oregon, USA; Tri-Tech Inc., 5120 Revere Road, Durham, NC; Petersen-Bach, Enebaervej, Bjerringbro, Denmark) also deal only with the sampling of hands.

A much longer persistence of GSRs was found on hair and clothes [23]. However few studies deal with clothes sampling  $[1.6.22]^2$  and only two, as far as we know, describe hair sampling [16.23], using both swabbing and filtration method. Swabbing using a comb was described too.<sup>2</sup> It was also stated that the tape-lift method is not suitable for sampling hair [23]. The recommended collection procedure from hands by the tape-lift method is to press the double-side adhesive coated stub repeatedly against the hand until it has lost its stickiness [1.9.17]. However, as far as we know, there is no quantitative study that justifies stopping the dabbing when the stickiness appears to have been lost. Two studies measured the collection efficiency of the tape-lift method from hands by comparing number of particles recovered in two successive samplings [1.24].

The objective of this work was to study the application of the tape-lift method for sampling GSRs from hair and to evaluate its collection efficiency (as a function of the number of dabbings). Comparisons were carried out against the collection efficiency of swabbing and concentration technique. We have evaluated also the collection efficiency of the tape-lift from hands with regard to the number of dabbings.

#### **Experimental Procedure**

All firing tests were carried out in an indoor shooting range, using a 9 mm FN semiautomatic pistol held in both hands and 9 mm Israeli TZZ ammunition. The ventilation in the shooting range was turned off during the experiments, except for three firing tests that will be pointed out.

SEM/EDX analyses for GSR were carried out using an automated search system attached to a CamScan IV SEM with a motorized stage drive and a four-samples holder, combined with a Tracor-Northern TN 5500 energy-dispersive X-ray (EDX) system [7,8,19]. In all cases the search area was about  $10 \times 8 \text{ mm}^2$ . If not otherwise stated, 25 mm diameter aluminum stubs, coated with double-side adhesive ("Scotch Tape" No. 465, 3M company), were used in all tape-lift samplings and the stubs were carbon-coated prior to the SEM examination. In order to reduce electron charging of the sample, especially when there were many particles on the stub, quite a thick (dark brown) carbon coating was applied.

-R. H. Keeley, Metropolitan Police Forensic Science Laboratory, London, England, personal communication, 1985.

Two individuals, one having straight hair and the other having curly hair. were chosen to study the applicability of the tape-lift method and its collection efficiency for sampling GSRs from hair. In all tests the two individuals were either one day or two days after washing their hair.

#### Persistence of GSRs on Hair

No technical problems were encountered in the experiments of sampling particles from hair by the tape-lift method. If too long hair shafts were collected on the sticky surface of the stub, they could have been removed by tweezers or cut by scissors along the edge of the stub. It appeared, as assessed subjectively, that the stub had lost its stickiness after about a hundred dabbings of hair. No significant difference was found between the two individuals in this respect, even though they had a difference in hair type.

To estimate the persistence of GSRs on hair, the front half of the subject's hair was sampled (applying 100 dabbings) at various times after firing; one sampling after every firing. Between firing and sampling the subject carried out his usual work in the laboratory (in an area not exposed to GSR contamination) and his usual activities at home.

The results of these experiments are presented in Table 1. It may be seen that GSRs may be found on the hair of the shooter even 24 h after firing, provided that he did not wash his hair. No substantial difference was found between the straight and the curly hair types. The results of experiment No. 3 indicate that ventilation in the shooting range greatly reduced the number of GSRs found on the hair.

# Collection Efficiency of GSRs from Hair by the Tape-Lift Method as Compared with the Swabbing-and-Comb Method

We compared the relative collection efficiency of the tape-lift and the swabbing-andcomb methods in the following manner. After firing, samplings from the shooter were carried out in pairs. Each pair was comprised of first sampling the hair by one method and then, immediately afterwards, sampling by the other one (for example, first by the tape-lift and then by swabbing). In the tape-lift method 100 dabbings were applied. The swabbing method was carried out similarly to the procedure used in the Metropolitan Police Forensic Science Laboratory, London, UK<sup>(2)</sup>: A comb, having a 4 × 6-cm<sup>2</sup> ethanol moistened pad (Litex 10, Lic Co., Sweden) on its teeth, was used to comb the front half of the hair. The pad was removed from the comb and placed in a beaker with 150 ml hexane. After ultrasonic agitation for 2 min, the liquid was filtered through a two-

Experiment No.	Type of Hair	Number of Rounds	Time after Firing	Number of 3-Elements GSRs on Search Area
1	curly	1	5 min	80
2	straight	1	5 min	40
3	straight	1	5 min	3ª
4	straight	2	6 h	26
5	curly	2	6 h	44
6	curly	1	12 h	8
7	straight	1	12 h	11
8	straight	1	23 h	3
9	curly	1	23 h	2

TABLE 1-Persistence of GSRs on hair.

"Ventilation was working in the shooting-range during firing.

Experiment No."	Type of Hair	Sampling Sequence	Number of 3-Elements GSRs on Search Area
10	Curly	Tape-Lift Swab	80 86
11	Curly	Swab Tape-Lift	57 34
12	Straight	Tape-Lift Swab	40 54
13	Straight	Swab Tape-Lift	97 30
14 <sup>b</sup>	Straight	Tape-Lift Swab	3 1
15 <sup>b</sup>	Straight	Swab Tape-Lift	2

TABLE 2—Comparison of tape-lift and swab collection of GSRs from hair.

<sup>a</sup>In each experiment one round was fired and the subject was sampled 5 min after firing. <sup>b</sup>Ventilation was working in the shooting-range during firing.

stage filtration apparatus with a 13 mm final membrane filter (0.8 micron) of cellullose nitrate [15,16,19]. The effective filter area is approximately 9 mm in diameter. The pre-filter is made of 20 micron stainless steel mesh. After filtration, the beaker was swilled with another portion of 50 mL hexane that was filtered through the apparatus. The collecting membrane filter was then removed, dried, mounted on a stub, coated with carbon and examined in the SEM.

The results of these experiments are presented in Table 2. In all cases the search area was about 0.8 cm<sup>2</sup>. This is equivalent to the whole effective area of the filter but only about 20% of the tape-lift surface. The results show that neither of the two collecting techniques have a very high efficiency of GSRs recovery, since in most of the experiments the number of GSRs found in the second stage of sampling was similar to the one found in the first stage. Therefore, no clear advantage for either method was found for the same search area. However, in the tape-lift method only about 20% of the stub collecting area was searched, so that in all cases the over-all efficiency of the tape-lift method was considerably higher. Furthermore, since in the swabbing technique the preparation of the sampling kit is more complicated and there are many more handling stages involved in the sample preparation for examination in the SEM, it seems that from a practical point of view the tape-lift technique is preferable.

# The Effect of the Number of Dabbings on the Collecting Efficiency of GSRs from Hair by the Tape-Lift Method

In the experiments whose results are presented in Tables 1 and 2, from several hundreds up to several thousands different particles (besides GSRs) were detected and classified on every search area by the autosearch system. Yet, these particles are only those particles that provide a backscatter electron signal above a predefined threshold which was selected in order not to miss any GSR particle during the autosearch. It is obvious that many thousands of various particles may be collected from hair, using the tape-lift method and applying 100 dabbings. GSRs are only a small percentage of them, even if the hair is sampled shortly after firing. Therefore, it is reasonable that basically the collection efficiency of GSRs by tape-lift will depend on the number of dabbings, on the number of all particles and on the concentration of GSRs in relation to other particles found on the subject's hair.

It may be expected that if the quantity of all particles on the hair will be large enough, after a certain number of dabbings all the adhesive sites on the stub will be occupied and any additional dabbing will not be effective. If we could know the number of GSRs deposited on the hair after firing, we could simply calculate the collection efficiency, as a function of the number of dabbings, by counting the number of GSRs and dividing it by the number of such particles deposited on the hair. Since we do not have that information, the collection efficiency in the subsequent discussion will be defined as the number of GSRs detected in the sample.

The evaluation of the collection efficiency with regard to the number of dabbings was carried out indirectly. Namely, by counting the number of GSRs sampled by a stub after it was used to sample hair that was not contaminated by GSRs (preliminary sampling). The number of dabbings in the preliminary sampling (collecting of non-GSR particles from the uncontaminated individual's hair) was different from stub to stub while the number of dabbings for sampling GSRs was fixed. In such a way we could learn about the effect of the extent of the occupation of adhesive sites, which is correlated to the number of the preliminary dabbings, on the collection efficiency, and the probe for this is the second sampling. The time between the two samplings was a day or two, so it was assumed that the preliminary sampling did not effect the second sampling. In other words, we assumed that after about a day some kind of steady-state is achieved with regard to the number of particles on the hair. This assumption is reasonable if we consider individuals being at the same environment.

Forty dabbings were chosen for sampling GSRs, for most of the experiments, and multiples of this number were applied for the preliminary samplings. We also applied successive samplings by different stubs on the same individual after each firing experiment in order to gather more data. The results of these experiments are presented in Table 3.

We can see from the results of experiments Nos. 16 and 17 that there was no significant change in the number of GSRs obtained in three successive samplings of 40 dabbings from the same individual. This means that additional samplings, at least up to 120 dabbings, did not change significantly the GSRs population on the subject's hair in the above experiments. Based on these results, three successive samplings were carried out in experiments Nos. 18 and 19. We assumed that in each sampling there was a similar GSR population. In these experiments we have found that up to 160 preliminary dabbings did not change considerably the collection efficiency of the adhesive-coated stub. However a drastic decrease in the collection efficiency was observed in stubs that were dabbed 320 and 480 times prior to sampling GSRs (compare samplings 1 and 2 to samplings 3, 4 and 5 in experiments Nos. 20 and 21). This means that rather steep decrease in the collection efficiency oto 300 preliminary dabbings. About 2 min were needed for the 200 dabbings.

To test the collection efficiency in this range, a different set of experiments was carried out. The paper back of the double-side adhesive was removed from only half of the stub area. Then the stub was used for preliminary sampling. To sample GSRs after firing, the second half of the paper backing on the stub was removed and the whole area of the stub was used to sample GSRs after firing. The search for GSRs was carried out in each half of the stub separately. Results are shown in Table 4. No significant difference was observed between 200 and 250 preliminary dabbings. However, as may be seen in comparison to the adhesive surface that was not used for preliminary sampling, the collection efficiency drops by about 50% after 200 or 250 preliminary samplings.

Based on the results of Tables 3 and 4, a plot was drawn showing the effect of the number of preliminary dabbings on the relative collection efficiency (Fig. 1). A value of 100 was assigned for an area that was not used for preliminary sampling in every exper-

Experiment No."	Type of Hair	Sampling Sequence for GSRs	Number of ''Clean Dabbings''	Number of Dabbings for GSRs	Number of 3-Elements GSRs on Search Area
		1		40	99
16	Curly	2		40	61
		2 3	—	40	78
		1	_	40	62
17	Straight	2	_	40	89
	Ū.	2 3		40	117
		1	160	40	53
18	Curly	2	80	40	42
	2	2 3		40	63
19		1	160	40	36
	Straight	2 3	80	40	65
	5	3		40	69
		1	480	40	_
		2	320	40	1
20	Curly	2 3	_	20	1 5
		4	_	200	60
		5	—	20	21
		1	480	40	1
			320	40	3
21	Straight	2 3 4 5	_	20	12
	U	4		200	59
		5	_	20	13

TABLE 3—The effect of the number of dabbings on the collection efficiency of GSRs from hair.

"In each experiment one round was fired and the subject was sampled 5 min after firing.

iment. Since there was no considerable difference between the results of the curly and the straight hair types, the ordinate values in the plot are the average between the results of these two types of hair. As was noted above, the 40 dabbings for GSRs were enough not to effect considerably the GSR population on the hair and the extent of the occupation of adhesive sites. Nevertheless, in order to take into account the effect of these dabbings

Experiment No. <sup>a</sup>	Type of Hair	Sampling Sequence for GSRs	Number of "Clean Dabbings"	Number of Dabbings for GSRs	Number of 3-Elements GSRs on Search Area
22 Curly		1	250	40 40	18 30
	Curly	2	200	40 40	26 60
23	~	1	250	40 40	19 35
	Straight	2	200	40 40	12 17

TABLE 4—The effect of the number of dabbings on the collection efficiency of GSRs from hair (half stub for preliminary sampling).

"In each experiment one round was fired and the subject was sampled 5 min after firing.

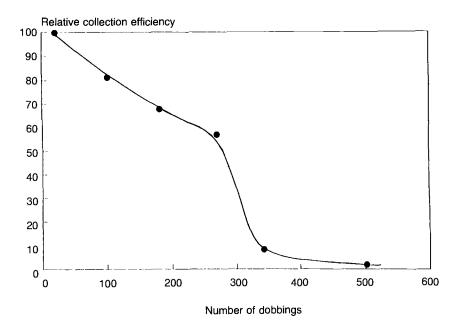


FIG. 1—The effect of the number of preliminary dabbings on the collection efficiency of GSRs from hair (see text).

on the collection efficiency, a value of 20 (half of the number of dabbings for GSRs) was added to the number of preliminary dabbings. These sum values are the abscissa values in the plot.

Several conclusions may be drawn from the experiments in this section:

1. The collection efficiency of particles drops practically to zero after a certain number of dabbings. In these experiments this number was about 300. However, this value may vary depending on how "clean" the hair of the individual may be.

The more objective criterion to assess when the drop in collection efficiency will occur is by examining the extent of coverage of the adhesive by various particles (Fig. 2). It is interesting to note that on stubs whose collection efficiency dropped to zero (Figs. 1c and 1d) a considerable part of the adhesive area is still not covered by particles, and additional dabbing from hair will not change the situation. In contrast to this, a total coverage of the stub usually occurs after certain number of dabbings in sampling clothes by the tape-lift method (Fig. 3). A possible explanation for this difference may be that a certain density of hair fragments on the adhesive surface prevents the effective contact between other particles and the unoccupied adhesive area. This explanation is also consistent with a rather sudden drop in the collection efficiency as a function of the number of dabbings instead of the expected linear decrease, that would be expected if all the adhesive area would be available for sampling.

2. A subjective assessment in loss of stickiness of the double-side adhesive is not a good criterion to estimate when a substantial decrease in the collection efficiency will occur. As was described previously, we found, by subjective assessment, that the stub had lost its stickiness after about 100 dabbings, while a considerable decrease in the collection efficiency occurred only after 200-300 dabbings.

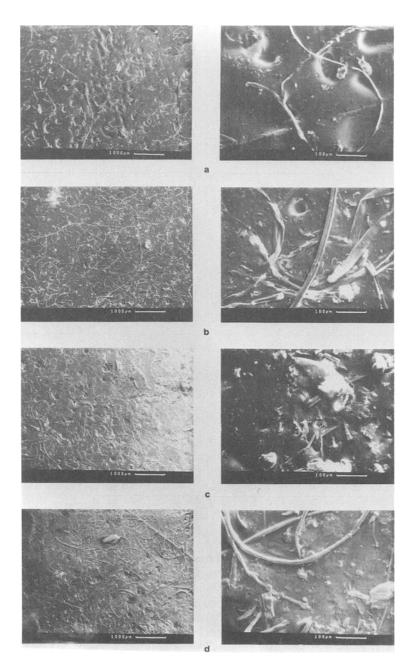


FIG. 2—The coverage of the double-side adhesive by various particles as a function of the number of dabbings from hair: a: 40 dabbings, b: 200 dabbings, c: 360 dabbings, d: 520 dabbings.

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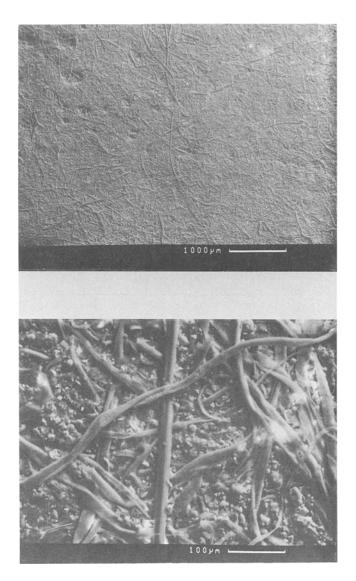


FIG. 3—The coverage of the double-side adhesive by various particles after sampling of a laboratory coat.

# The Effect of the Number of Dabbings on the Collecting Efficiency of GSRs by the Tape-Lift Method from Hands

As was described above, the usual recommended collection procedure from hands by the tape-lift method is to dab the stub against the hand until it has lost its stickiness [1.9,17]. As far as we know, no quantitative study has been conducted to support this recommendation. The concern from continuing dabbing is that skin debris may conceal GSR particles from view [15,21,23].

Since we found, and described above, that the subjective assessment of stickiness may be erroneous with regard to collection efficiency from hair, it was interesting to examine this aspect as pertains to the sampling of GSRs from hands.

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The two individuals from the previous experiments participated also in the experiments concerning the sampling from hands. It was found that stickiness is lost after 10 to 30 dabbing, as assessed subjectively.

A first series of experiment was carried out applying preliminary sampling using the half stub, as in the case of the sampling from hair. The time between the preliminary sampling and the sampling after firing was at least two hours. During this time, the individuals carried out their usual activities in the laboratory, except for contact with firearms. The individuals fired the weapon by holding the gun with both hands, and the back area of both hands, especially the web, thumb and forefinger, was sampled by the stub [1]. The results are shown in Table 5.

We can see that, as in the case of sampling from hair, loss of stickiness, as assessed subjectively, does not eliminate the sampling capability of adhesive, although there is a decrease in the collection efficiency of about 50% after 20 to 40 preliminary dabbings. Here also, the GSRs are a minority among other assorted particles.

Another set of experiments was carried out to examine the possibility that skin debris will cover the sampled GSRs as follows. An array of four small stubs (1.5 cm diameter each) was mounted on a rectangle rubber pad (Fig. 4). These stubs were covered with the same double-side adhesive as in the previous experiments. After firing, the array was used to sample the hands of the shooter so that after 30 dabbings one stub was removed from the pad, after additional 30 dabbings the second stub was removed, then the third stub removed after additional 30 dabbings and finally the last stub was used for 30 additional dabbings. In this way we obtained 4 stubs that were used for 30, 60, 90 and 120 dabbings respectively. Before coating with carbon the stubs were shaken for several seconds to remove particles, if any, that were loosely held on the surface. The results of this set of experiments are presented in Table 6.

Although after about 60 dabbings the stubs appear to be practically covered completely by skin debris (Fig. 5), the results show that sampling capability of GSRs by a stub covered with skin debris is not significantly decreased, if at all, up to 120 dabbings. The maximum number of GSRs were sampled after 60 to 90 dabbings in the first experiment and 90-120 dabbings in the second one. In fact most of the GSRs on the fourth stub (in the array) in both experiments were on skin debris. Examples are shown in Fig. 6. Consequently, the danger of skin debris concealing GSRs is not substantial, at least up to 120 dabbings, while stickiness is lost after about 30 dabbings.

Experiment No."	Individual	Sampling Sequence for GSRs	Number of ''Clean Dabbings''	Number of Dabbings for GSRs	Number of 3-Elements GSRs on Search Area
24	1	1	40	20	38 58
		2	20	20	23 49
25	2	1	40	20	21 62
		2	20	20	30 51

TABLE 5—The effect of the number of dabbings on the collecting efficiency of GSRs from hands.

"In each experiment three rounds were fired and the subject was sampled 5 min after firing.

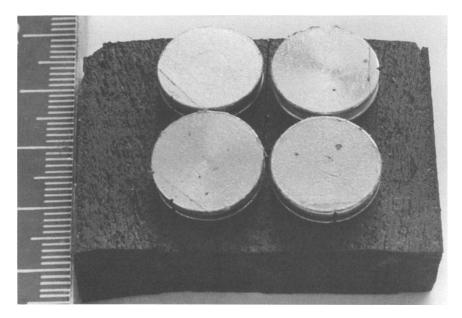


FIG. 4—An array of four stubs used for sampling GSRs from hands (see text).

### Conclusions

1. Tape-lift was found to be a suitable method for collecting GSRs from hair.

2. No significant difference in collecting efficiency was found between this method and the more complicated method of swabbing the hair using a comb with a solvent damped cloth. From a practical point of view, the former technique is advantageous since it involves much less sample handling.

3. 200 to 300 dabbings, taking about 2 to 3 minutes, are necessary to achieve maximum collection efficiency from hair, while the subjectively assessed loss of stickiness is achieved after only about 100 dabbings.

4. The method has been used in casework in Israel for about a year and a half, and in some cases GSRs were found in samples from hair but not in samples from hands.

Experiment No."	Individual	Stub Sequence in Array	Number of Dabbings	Number of 3-Elements GSRs of Search Area
		1	30	59
26	1	2	60	185
20 1	1	3	90	136
		4	120	110
27		1	30	46
	2	2	60	53
	2	3	90	110
		4	120	131

 TABLE 6—The effect of the number of dabbings on the collecting efficiency of GSRs from hands (sampling by an array of stubs).

"In each experiment three rounds were fired and the subject was sampled 5 min after firing.

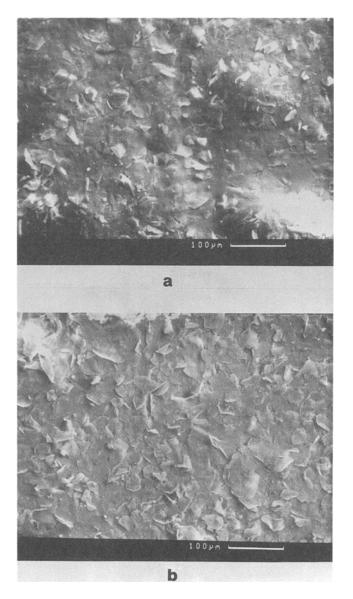


FIG. 5—The coverage of the double-side adhesive by skin debris after sampling hands: a: 60 dabbings, b: 120 dabbings.

5. 50 to 100 dabbings are necessary to achieve maximum collection efficiency of GSRs from hands, while stickiness is lost after about 20 to 30 dabbings. No substantial danger of skin debris concealing GSRs was observed.

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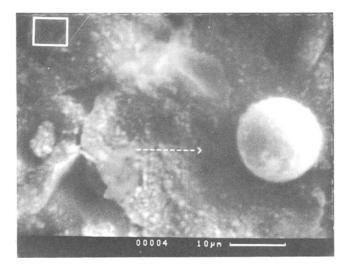


FIG. 6—Examples of GSR particles on skin debris sampled on the double-side adhesive. An arrow points towards the GSR particle in each SEM micrograph.

tification and Forensic Science, for their assistance in the experimental work and for fruitful discussion.

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