3.7 Powder suspensions

1. History

1.1 During the development of the small particle reagent in the late 1970s, many other particulates were investigated as constituents in the formulation, including amorphous carbon and graphite and the oxides of the magnetic elements cobalt and iron [1]. All of these gave good results, but none were as consistent as molybdenum disulphide and therefore were not pursued as systems for operational use.

1.2 In a significant development that appears to have been overlooked at the time, Haque and co-workers developed an alternative 'small particle suspension' based on iron oxide (Fe_3O_4) in 1989, and stated that this gave better results than the molybdenum disulphide-based small particle reagent in terms of sensitivity and contrast [2]. The new formulation was also noted to work on wetted surfaces, and to enhance further marks previously developed by powdering. This formulation does not appear to have entered widespread use for non-porous surfaces and was not developed further.

1.3 In the mid-1990s similar formulations were developed by researchers at the National Identification Centre, Tokyo Metropolitan Police, who were investigating simple methods for developing fingerprints deposited on the adhesive side of tapes [3]. This was noted by an American police officer on secondment in Japan and after experimentation with black powder suspensions he contacted the Lightning Powder Company, which developed the 'Sticky-Side Powder' product now sold commercially, consisting of a pre-mixed powder that was blended with Kodak Photoflo surfactant and distilled water. The resulting suspension was painted on to the adhesive side of tapes, then washed off using running water to reveal developed marks.

1.4 The new Sticky-Side Powder system was compared with techniques in general use for adhesive tapes in 1996, primarily basic violet 3 [4]. The powder suspension formulation was found to perform better than basic violet 3, in particular on marks known to be eccrine in nature. Several researchers began to investigate alternative powder suspension formulations, looking at the combination of commercial powders with surfactant/water mixtures. Bratton and Gregus [5,6] looked at Lightning Black Powder with Liquinox surfactant and reported it to give better results than Sticky-Side Powder, noting that the revised formulation reduced the occurrence of background staining that sometimes obscured marks with Sticky-Side Powder. Kimble [7] studied a wider range of powders, including grey and coloured systems, with Photoflo surfactant and water in different ratios. It was concluded that other powders could be used and a formulation incorporating a grey powder was proposed for black adhesive tapes. Other workers also investigated formulations for black adhesive tapes, Parisi [8] testing 'Pink Wop' fluorescent powder and a white fingerprint powder with Liquinox and Photoflo, and Martin [9]

looking at an ash grey powder with Photoflo. White powder in Liquinox and ash grey powder in Photoflo both gave suspensions that developed good quality fingerprints. Further testing of these revised powder suspension formulations against basic violet 3 continued to indicate that powder suspensions were the more effective single process for these surfaces [10].

1.5 The Police Scientific Development Branch (PSDB) began experimenting with powder suspensions for development of fingerprints on adhesive tapes in the late 1990s [11]. An initial assessment was carried out on the original Sticky-Side Powder formulation, characterising the base powder by electron microscopy and looking at optimised formulations. It was found that the base powder consisted of fine (~1μm) particles of iron oxide interspersed with larger (10–20μm diameter) flakes of aluminium. A range of other powder suspension formulations were investigated, with two ultimately being recommended for further research. A black powder suspension based on precipitated, magnetic iron oxide was proposed, together with a white powder suspension based on titanium dioxide powder. Both formulations utilised Photoflo as the surfactant. These formulations were trialled against Sticky-Side Powder, where the black formulation was shown to give superior results.

1.6 The black iron oxide-based formulation was then compared in effectiveness with two other treatments for the adhesive side of tapes, basic violet 3 and superglue followed by dyeing with basic yellow 40 [12]. In these trials powder suspension gave closely equivalent results to superglue, with the contrast of developed marks being slightly better. Basic Violet 3 was found less effective than either powder suspension or superglue, in accordance with previous observations.

1.7 Other researchers also concluded that titanium dioxide was the optimum particulate for white powder suspension formulations. Wade [13] used a commercially available white small particle reagent formulation based on titanium dioxide as a starting point, and demonstrated that improved performance was obtained by concentrating the solution and adding Photoflo. Alternative formulations based on different grades of titanium dioxide were investigated and it was observed that better results were obtained using the rutile, rather than anatase, form of titanium dioxide. Williams and Elliot [14] also looked at modifying white small particle reagent with Photoflo and studied different application methods including spraying, immersion, dipping, painting and pouring. It was concluded that the best development could be obtained by immersion and this method also reduced the risk of over-development but was also the most time-consuming.

1.8 Until the mid-2000s, development of fingerprints on adhesive surfaces was the sole application considered for powder suspensions. In 2004, Auld [15] carried out an investigation into the effectiveness of various fingerprint development techniques for detecting marks on motor vehicles, including vehicles that had been wetted. He compared

powdering, superglue, small particle reagent and Sticky-Side Powder, and found that Sticky-Side Powder was the most effective treatment for several scenarios, in particular where cars had been wetted at some stage prior to fingerprint development.

1.9 Around the same time, Strathclyde Police had begun to investigate the use of black and white powder suspensions for the treatment of articles recovered from arson scenes, the treatment both removing soot deposits and developing marks [16].

1.10 These observations resulted in further experimentation on non-porous surfaces, both by police forces on operational casework and by HOSDB in laboratory trials [17]. The HOSDB studies sought to establish the relative effectiveness of the black powder suspension technique on a range of different surfaces and the position of powder suspensions in sequential processing. It was soon apparent that superglue and powder suspensions are mutually exclusive processes and if one is applied the other cannot be used afterwards. An equivalent study was subsequently carried out for white powder suspensions on dark, non-porous surfaces, which came to similar conclusions, although white powder suspension was found to be less effective than black powder suspension overall [18]. Operationally, police forces applied powder suspensions either at crime scenes after powdering, or in the laboratory as a replacement for superglue on articles likely to have been wetted or contaminated (cowlings, number plates, drugs packaging). In both cases additional marks were found or recovery rates increased.

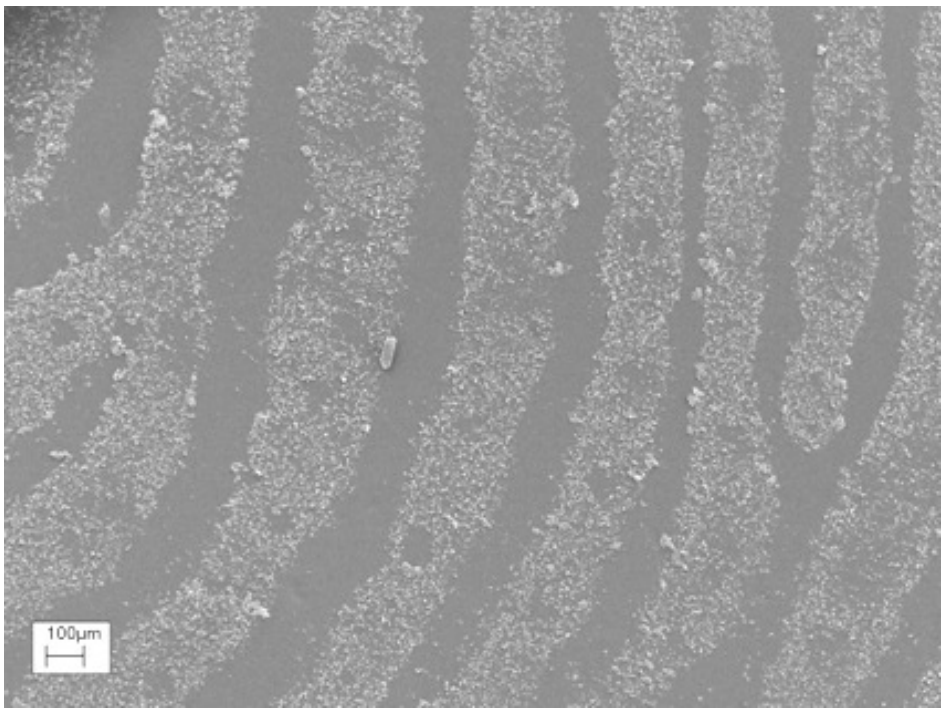
1.11 HOSDB also continued the assessment of powder suspensions for use on adhesive tapes, comparing the formulations developed in earlier work [11] with a range of commercially available powder suspensions. For the white powder suspension [19] it was found that the original HOSDB formulation gave marginally better results and this was used in a subsequent operational trial. For black powder suspensions it was discovered that commercial formulations based on carbon out-performed the HOSDB iron oxide-based formulation and work therefore focused on developing a non-proprietary carbon-based formulation [20]. It was not possible to identify a formulation giving equivalent or improved performance over the commercial systems and therefore commercial, carbon-based systems were included in operational trials. The results from these trials showed that carbon-based, black powder suspensions are the most effective process for the adhesive side of light coloured tapes, whereas for dark tapes superglue/basic yellow 40 is more effective, and white powder suspensions are only recommended for this application if it is known that the article has been wetted.

1.12 Subsequent work both at HOSDB and in operational police laboratories has continued to explore the range of surfaces for which powder suspensions can be used. Recent research has shown that they can be applied to plastic bags [21], semi-porous surfaces [22] and are one of the most effective processes for surfaces contaminated with drugs [23]. It is

anticipated that current (2011) advice regarding the treatment of such surfaces will be updated in due course.

2. Theory

2.1 The exact mechanism for development of marks using powder suspensions is unknown, and studies by CAST to establish which factors are most important are continuing. However, it is thought that the development process is very similar to that for small particle reagent, where the micelles are formed around the particles by the surfactant. Some component or property of the latent fingerprint destabilises these micelles, causing the particulates to deposit preferentially on the fingerprint ridges.



Scanning electron micrograph of fingerprint developed using black powder suspensions on clear adhesive tape, showing particles deposited on fingerprint ridges but not on background.

2.2 Powder suspension formulations contain far higher concentrations of powder than small particle reagent and this may account for some differences in behaviour noted between the two processes.

3. CAST processes

3.1 Powder suspensions have recently (December 2009) been incorporated into the CAST *Manual of Fingerprint Development Techniques* [24] in Charts 1, 2, 5 and 7, and it is likely that they will be incorporated into other charts as research progresses.

3.2 There are three slightly different powder suspensions recommended for operational use, these being outlined below.

3.3 Black powder suspension for use on the adhesive side of adhesive tapes (carbon-based): Commercially available, pre-mixed carbon-based powder suspensions, either Kjell Carlsson Wet Powder (Black) or Armor Forensics/Forensics Source WetWop™ (Black).

3.4 Black powder suspension for use on light, non-porous surfaces (iron oxide-based): Weigh 20g precipitated magnetic iron oxide (Fe_3O_4/Fe_2O_3) into a glass beaker, add 20mL of a pre-mixed 1:1 solution of Kodak Photoflo 200 and distilled water and stir with a brush to form a paste [25].

3.5 White powder suspension for use on dark, non-porous surfaces and wetted dark adhesive tapes (titanium dioxide-based): Commercially available, pre-mixed titanium dioxide-based powder suspensions, either Kjell Carlsson Wet Powder (White) or Armor Forensics WetWop™ (White).

3.6 The ratio of powder to surfactant/distilled water mixture recommended in the CAST formulations for application to adhesive tapes have been determined by laboratory tests [11]. If there is excess surfactant/water present, a thinner suspension is produced, which does develop marks although these are significantly fainter than those obtained with optimum formulations. If there is insufficient surfactant/water present, the suspensions do not flow and clumps of powder may be left behind on the tape.

3.7 For use on non-porous surfaces, it has been observed that the powder suspension can be diluted from the thicker paste applied to adhesive tapes and can still give effective results.

3.8 The role of the detergent in the formulation is to form micelles around the fine particulates and stabilise the suspension against indiscriminate precipitation over the entire surface. The CAST formulations utilise Photoflo, but the commercial formulations may contain other surfactant systems.

4. Critical issues

4.1 Performance of powder suspensions is often critically controlled by the particle size and the shape of the materials concerned which can vary widely with methods of preparation. Use of other generic sources of what is nominally the same chemical may result in very different results and batch testing is recommended.

5. Application

5.1 Suitable surfaces: The full range of application areas for powder suspensions are still being explored, but it is likely that they will be recommended for use in the following circumstances.

- On the adhesive side of light coloured, polymer backed adhesive tapes.
- On non-porous surfaces where it is thought that the surface has been wetted or exposed to high humidity environments.
- On non-porous surfaces where powder or particulate contamination (e.g. soot or drugs residues) is present on the surface.
- On non-porous surfaces where there is a surface layer of oily contamination present.
- On some 'semi-porous' surfaces.
- In a sequential treatment process after powders at a scene of crime and in laboratories.
- As a final treatment after blood dyes on non-porous surfaces.

5.2 Powder suspensions are applied to the surface of interest using a soft brush, ensuring that the brush is well loaded with the suspension mixture to avoid damage that could be caused to the fingerprint by a dry brush and to avoid 'streakiness' in background development. The suspension should be stirred to achieve a paint-like consistency and painted onto the surface, left in situ for 10–15 seconds and then washed off using running water (either from a tap, hose or wash bottle). The temperature of the wash water has not been found to be important.

5.3 Prints can become over-developed if the suspension is left on the surface too long because the suspension starts to dry and fills in ridges. Powder suspensions can also be reapplied, if necessary. There is also evidence to suggest that the different types of powder suspension can be applied in sequence and still develop additional marks.

5.4 The process is suited to application both in a laboratory and at scenes of crime, none of the constituents posing a significant health and safety issue. However, the process is messy to apply and the implications for cleaning of the scene should be considered before application.

6. Alternative formulations and processes

6.1 The initial formulation proposed for powder suspensions [2] was iron oxide mixed with Brij 35 and choline chloride surfactant, diluted with distilled water. This does not appear to have been widely adopted and has not been evaluated by CAST, although the current (2011) formulations are actually similar in nature.

6.2 The first formulation proposed for adhesive tapes was Sticky-Side Powder, consisting of Sticky-Side Powder (a mixture of iron oxide particles and aluminium flakes) mixed with a 1:1 blend of Photoflo and water, added until a thin paint consistency was achieved. Soon after this an alternative formulation was proposed that used 20g of Lightning Black Powder as the particulate, mixed with 20g Liquinox surfactant and 40mL of distilled water.

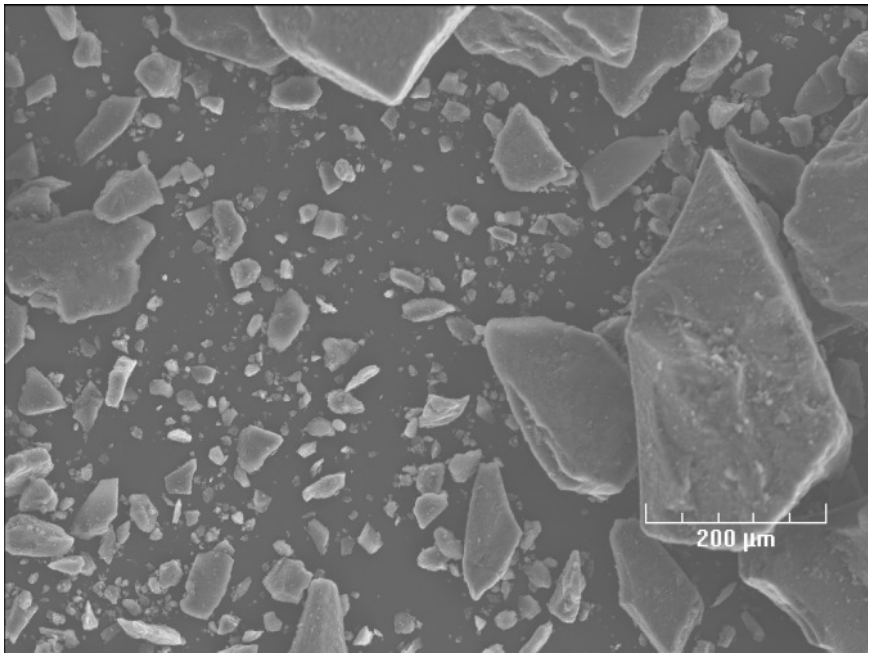
6.3 PSDB evaluated both of these formulations in comparative trials with many different types of particulate fillers in powder suspensions. In the initial investigation of an optimum formulation for the treatment of adhesive tapes [11], the range of powders below were tested in combination with Photoflo surfactant as candidate black powder suspensions.

Powder sample	Specific gravity	Particle size	Manufacturer/ supplier
Fe_3O_4	5.18	> 10μm	BDH Chemicals
Fe_3O_4	5.18	> 5μm	Sigma – Aldrich
Fe_3O_4 – magnetic/ precipitated	5.18	> 1μm	Fisher Chemicals Ltd
Fe_2O_3 – red, precipitated	5.24	> 5μm	BDH Chemicals
Fe powder	-	9 – 110μm	–
Lightning Black Powder	~1.8	> 1 μm (aggregates up to 40μm)	Lightning Powder Company
Lightning Magnetic Black Powder	-	Range from 1–30μm	Lightning Powder Company
Cobalt (II, III) oxide	6.11	> 1μm	Sigma – Aldrich
K9 – Black Fingerprint Powder	1.7–1.9	> 1μm (aggregates up to 150μm)	K9 Scene of Crime Ltd
K9 – Black Magnetic Powder	~1.8	> 1μm (aggregates up to 150μm)	K9 Scene of Crime Ltd
K9 – Jet Black Magnetic Powder	~5.18	> 1μm (aggregates up to 150μm)	K9 Scene of Crime Ltd
K9 – Magneta Flake	7.8	–	K9 Scene of Crime Ltd
K9 – Gold Powder	8.5	–	K9 Scene of Crime Ltd
K9 – Grey Magnetic Powder	~2.7	–	K9 Scene of Crime Ltd
Dactyl Black Fingerprint Powder	~2	> 1–24μm	Speciform
Copper (II) oxide	6.315	–	Sigma – Aldrich
Activated Charcoal	~2	–	BDH Chemicals
Graphite Powder	~2.09–2.23	–	Sigma – Aldrich
Graphite Powder (synthetic)	~2.09–2.23	–	Sigma – Aldrich
Molybdenum disulphide	4.80	–	-
Manganese disulphide	–	–	Sigma – Aldrich
Vanadium (III) oxide	4.87	–	Sigma – Aldrich

Particulates investigated by the Police Scientific Development Branch as the basis for black powder suspensions for adhesive tapes.

6.4 Of these, the precipitated magnetic Fe_3O_4 powder proved most effective (out-performing both formulations originally proposed for adhesive tapes in the literature) and was therefore used in the CAST formulation initially proposed for adhesive tapes. This formulation was subsequently found to give excellent results on non-porous surfaces.

6.5 Commercial, pre-mixed black powder suspensions have recently (post 2004) become available, including Wet Powder – Black (Kjell Carlsson) and WetWop™ – Black (Armor Forensics). An initial assessment of these formulations indicated that they were probably based on a powdered graphitic material.



Scanning electron micrograph of particulates from commercial carbon-based powder suspension.

6.6 It was established by comparative trials that carbon-based black powder suspensions were superior to iron oxide-based formulations on all types of adhesive tapes, and therefore a more in-depth assessment was carried out on carbon particulates. This focused on graphitic powders although several other forms of carbon were also investigated [20], as outlined in the table below.

Powder	Particle size	Manufacturer/supplier
Coke FC800	0.8mm	TIMREX
Graphite T800	0.71mm	TIMREX
Graphite	150μm	Fisher Chemicals Ltd
Activated charcoal	50–150μm	Sigma – Aldrich
Swedish black powder	95μm	BVDA
Natural graphite	75μm	GTC
Synthetic graphite	53μm	GTC
Graphite powder	50μm	VWR
Activated charcoal	40μm	Sigma – Aldrich
KS44	44μm	TIMREX
HSAG 300 AE-109	32μm	Timcal
Graphite	20μm	Sigma – Aldrich
Micronised graphite	10μm	GTC
Graphite KS6	7μm	TIMREX
Dispersion LB1300	7μm	TIMREX
Activated carbon	0.8μm	Sigma – Aldrich
Monarch 280 carbon black	0.41μm	Cabot Carbon
Carbon nanopowder	0.3μm	–
Vulcan VXC 72R	0.3μm	Cabot Carbon
Mogul L	0.24μm	Cabot Carbon

Carbon powders evaluated as constituents for non-proprietary carbon powder suspension formulation.

6.7 Several surfactants were also evaluated in this study, including:

- Photoflo;
- Aerosol OT;
- Liquinox.

6.8 For white powder suspensions, white powders with relatively high density and a spherical shape were researched [11]. Of these, initial trials indicated that zirconium oxide and titanium dioxide gave the best results, with titanium dioxide giving marks of higher contrast. Further studies therefore focused on optimising the titanium dioxide formulation.

6.9 A range of commercial white powder suspensions have also become available, including Wet Powder – White (Kjell Carlsson), WetWop™ – White (Armor Forensics/Forensics Source) and Adhesive Side Powder – Light (Sirchie). These have all been evaluated against the original CAST formulation on adhesive tapes [19] and found to give closely equivalent performance. A comparative trial on non-porous surfaces [18] found that for the limited range of surfaces evaluated there was little significant difference between any of the commercial formulations and the CAST

adhesive tapes formulation, and the white powder suspensions can be used interchangeably.

6.10 Various nanopowders were also been evaluated by HOSDB in 2007 (including aluminium, magnesium, titanium, tin, yttrium, iron, zirconium, copper, neodymium, tungsten, lanthanum, terbium, ytterbium, and bismuth oxides, carbon, and silicon carbide) [20]. Many of these failed to develop fingerprints when used in suspensions, but of those that did the best were found to be iron oxide, titanium dioxide and carbon (the same constituents as used in existing formulations), but none gave better results than the formulations outlined in the CAST processes section above.

7. Post-treatments

7.1 Marks developed using powder suspensions can be lifted once dry in the same way as marks developed using small particle reagent, using either adhesive tape or gelatine lifts.

8. Validation and operational experience

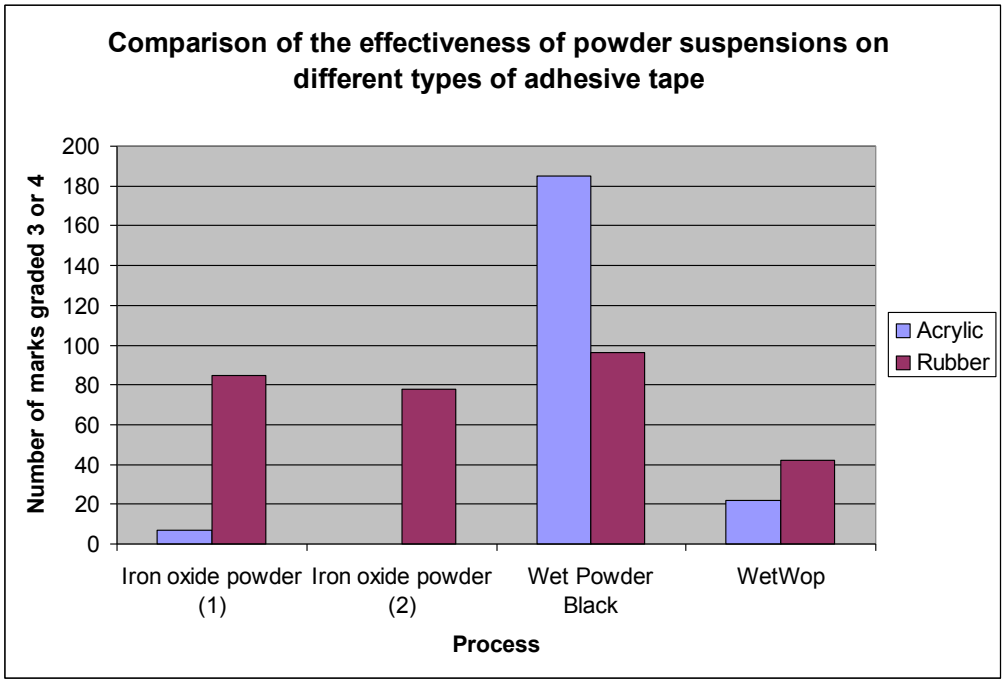
8.1 The operational experience of powder suspensions must take into account two primary applications – their use on adhesive tapes and their use on non-porous surfaces. There is a greater background knowledge regarding the effectiveness of powder suspensions on adhesive tapes, although the application of powder suspensions to other non-porous surfaces is becoming more widespread.

8.2 Laboratory trials

8.2.1 Initial laboratory comparisons on adhesive tapes were carried out at PSDB in 2000 between basic violet 3, iron oxide-based black powder suspension and superglue, with over 1,600 prints being evaluated for each process [12]. In these trials superglue and iron oxide-based black powder suspension gave the best results and were very similar in performance, but powder suspension marks had better contrast. An equivalent trial was carried out using titanium oxide-based white powder suspension, superglue and basic violet 3 (imaged via fluorescence and via the transfer technique). The results were closely equivalent to those observed for light tapes, with white powder suspension and superglue being closely equivalent in performance and both better than basic violet 3. The powder suspension again showed better contrast for developed marks.

8.2.2 During these trials it was observed that some tapes exhibited extensive background staining when treated with iron oxide-based powder suspensions whereas others did not. It was established by infrared (IR) spectroscopy that tapes using rubber-based adhesives did not

background stain while those with acrylic-based adhesives did. This resulted in the initial recommendation that a spot test be carried out to see whether background staining occurred prior to selecting a treatment [25]. However, it was subsequently noted that there were differences between powder suspensions, not all staining the background of acrylic tapes. It was established that the suspensions that did not stain the background contained carbon instead of iron oxide particulate, and a comparison of the relative effective of iron oxide- and carbon-based black powder suspension (WetWop™, Wet Powder Black) was carried out on both rubber and acrylic adhesive tapes. This trial looked at 300 half prints over a range of acrylic tapes and 480 half prints over a range of rubber tapes.



Results of comparative trials using different black powder suspensions on adhesive tapes.

8.2.3 These trials demonstrated that Wet Powder – Black gave the best overall performance, with both carbon powder formulations working on acrylic and rubber-based adhesives. Background staining of acrylic-based adhesive tapes by iron oxide powder suspension formulation was again observed.

a)

b)

Development of marks on adhesive tapes, a) acrylic-based adhesive tape showing background staining by iron oxide-based powder suspension applied to left half, no background staining from carbon-based powder suspension applied to right half b) rubber-based adhesive tape showing no background staining from iron oxide-based powder suspension applied to left half or carbon-based powder suspension applied to right half

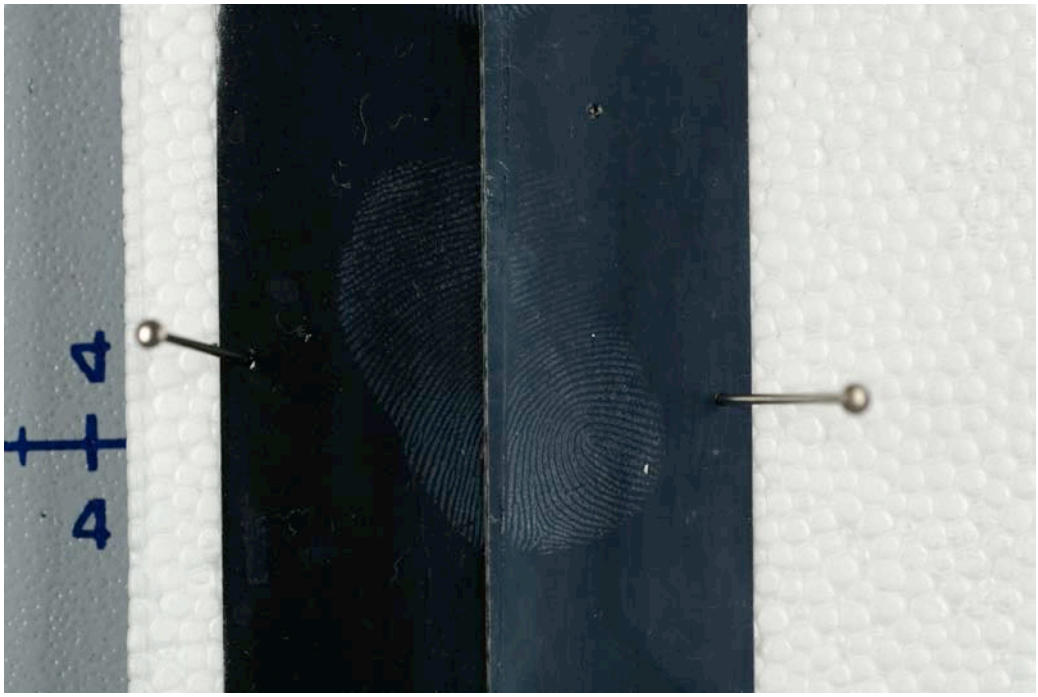
8.2.4 A similar comparison has been conducted for white powder suspensions on tapes. An initial investigation [19] compared the HOSDB formulation against the following commercially available products:

- Wet Powder – White (Kjell Carlsson);
- WetWop™ – White (Armor Forensics);
- Adhesive Side Powder – Light (Sirchie).

8.2.5 The results of these studies are summarised in the table below, but in general all formulations gave similar results, with the HOSDB formulation marginally better. Some differences were observed between the level of background staining, but in general all marks were clearly visible against the background. Approximately 14,400 marks were examined in this study.

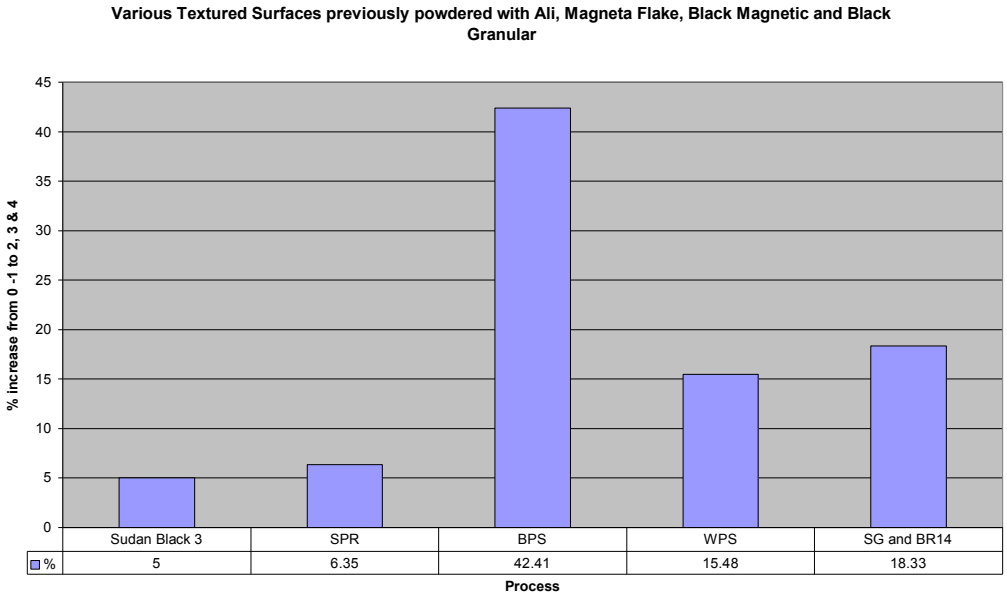
Mark grade	Sirchie	Wet Powder	WetWop	Stan Chem (HOSDB)
0	2.30%	1.63%	0.95%	1.51%
1	7.86%	6.83%	5.99%	4.09%
2	10.52%	13.63%	13.29%	11.88%
3	17.34%	21.79%	22.22%	20.79%
4	61.98%	56.13%	57.54%	61.73%
3s and 4s	**79.33%**	**77.92%**	**79.76%**	**82.53%**

Results of laboratory comparative trials for different white powder suspensions.



Comparison of fingerprint and background development using a) Home Office Scientific Development Branch formulation (left) and Sirchie Adhesive Side Powder (right) on black tapes.

8.2.6 Work on non-porous surfaces commenced with an initial assessment of the number of additional marks developed (or enhanced) by subsequent chemical processing after powdering. Several different processes were studied, including solvent black 3, small particle reagent, superglue and basic red 14 dye, and both white and black powder suspension (formulations, as published by HOSDB)[25]. The results of this exercise are illustrated below and clearly demonstrated that there were potential advantages in applying powder suspensions after powdering.



Results obtained by applying a secondary fingerprint development process (SPR = small particle reagent, BPS = black powder suspension, WPS = white powder suspension, SG and BR14 = superglue dyed with basic red 14) in sequence after powdering.

8.2.7 This prompted a further, in-depth study of the application of powder suspensions and alternative processes, both singly and in sequence [17]. It was soon established that carbon-based black powder suspensions were comparatively ineffective and studies therefore focused on the iron oxide-based black powder suspension formulation instead.

a)

b)

Black powder suspensions applied to a smooth, non-porous surface a) iron oxide-based formulations and b) commercial carbon-based formulation.

8.2.8 The study examined 37,560 marks deposited on 23 different smooth and textured non-porous (and in some cases semi-porous) surfaces representative of those that may be encountered at crime scenes, including ceramic tiles, melamine, painted metal, and uPVC, summarised below together with an outline of the number of marks deposited.

General surface classification	Specific description and designation
Smooth, non-porous	S1 Ceramic tile
	S2a Smooth wood effect laminate
	S2b Shiny, striped laminate
	S2c Beige laminate
	S3a White painted metal
	S3b Red painted metal
	S4 Glass
	S5 Perspex
	S6 Polyethylene
	S7 Polypropylene
Rough, non-porous	R1 Textured ceramic tile
	R2a Cream textured laminate
	R2b Wood effect laminate
	R2c Granite effect laminate
	R2d Grey textured laminate
	R2e Beige textured laminate
	R3 Textured painted metal
	R4 Fake leather texture laminated aluminium
	R5 Varnished wood
Other	O1 uPVC
	O2a Silk emulsion painted plasterboard
	O2b Kitchen/bathroom painted plasterboard
	O3 Textured vinyl wallpapered plasterboard

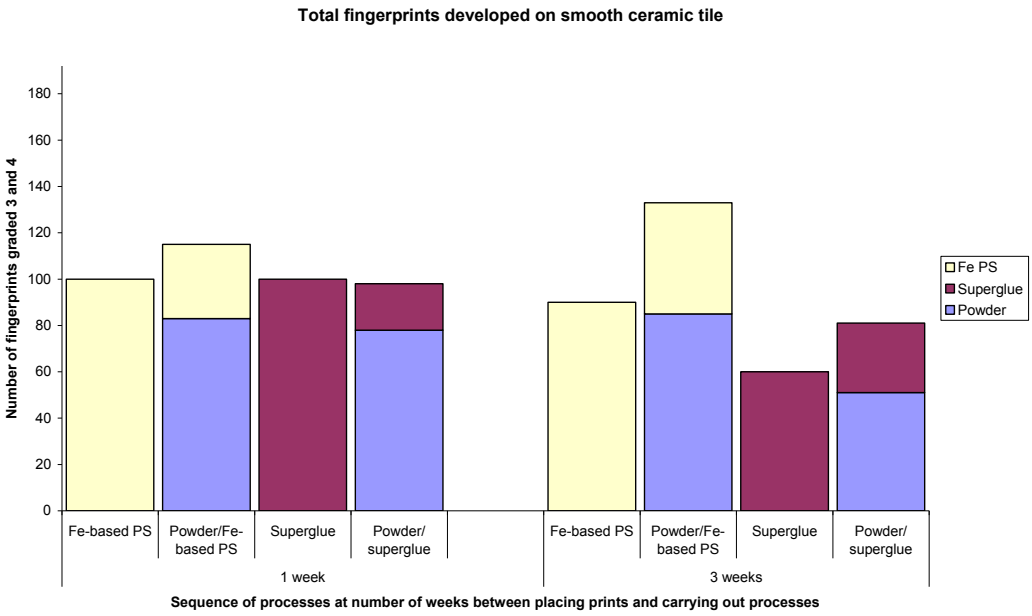
Description of the surfaces used in the comparative study between various sequences of superglue, powders and powder suspensions

Surface	Number of donors	Number in depletion	Number of repeats	Number of panels	Number of ages	Number of fingerprints
S1	40	12	5	50	3	4,800
S2a	35	10	5	50	3	3,500
S2b	14	10	2	20	2	1,400
S2c	7	10	1	8	2	560
S3a	14	10	2	20	2	1,400
S3b	28	10	4	44	3	3,080
S4	14	10	2	16	2	1,120
S5	14	10	2	20	2	1,400
S6	14	10	2	20	2	1,400
S7	14	10	2	20	2	1,400
R1	35	10	5	50	3	3,500
R2a	35	10	5	50	3	3,500
R2b	7	10	1	8	2	560
R2c	7	10	1	8	2	560
R2d	7	10	1	8	2	560
R2e	7	10	1	8	2	560

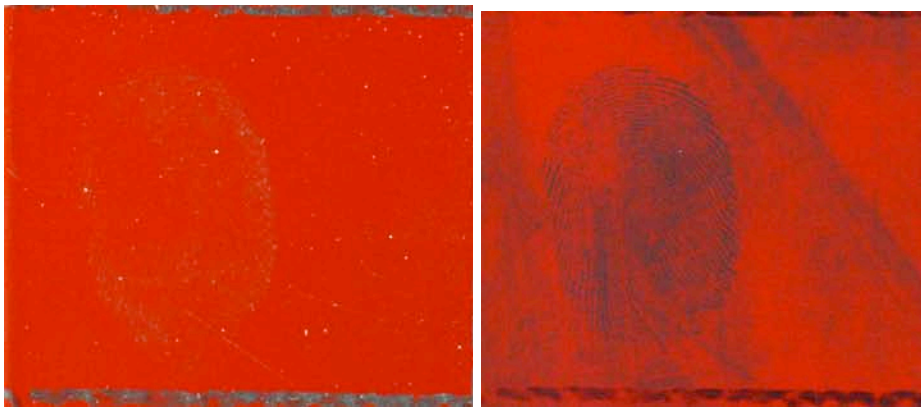
R3	21	10	3	32	3	2,240
R4	14	10	2	20	2	1,400
R5	7	10	1	10	2	700
O1	7	10	1	8	2	560
O2a	7	10	1	12	2	840
O2b	14	10	2	24	3	1,680
O3	7	10	1	12	2	840
					TOTAL	**37,560**

Summary of the experiments carried out in the comparative study and the total number of marks used.

8.2.9 The conclusions were that in many cases the powder-powder suspension sequence was more effective than superglue and dyeing and powder suspensions are clearly a highly effective process. Typical results from some of the surfaces used in the study are illustrated below.

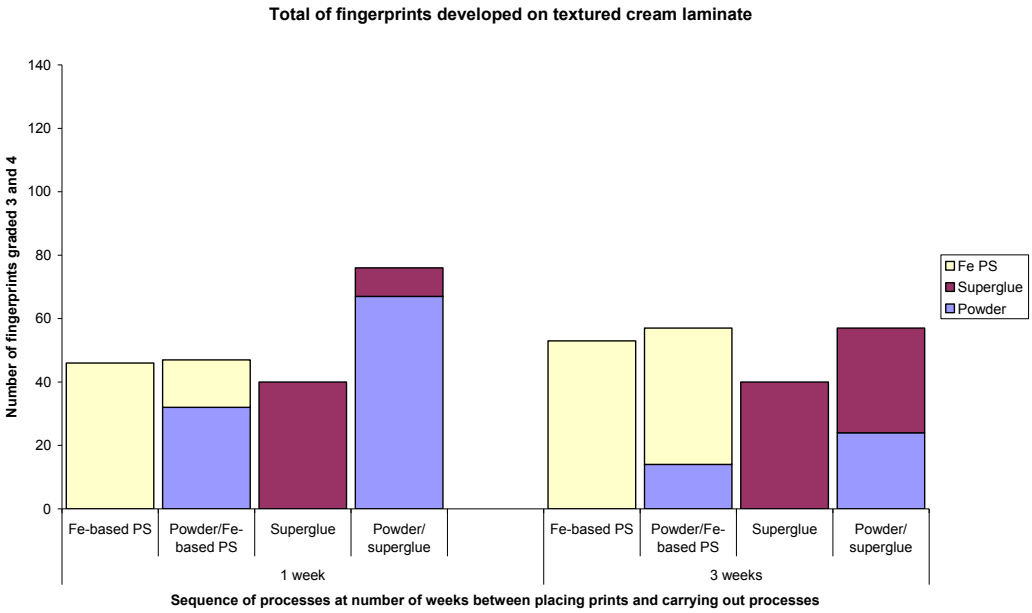


Typical results obtained comparing the effectiveness of powder suspensions, powders and superglue as single treatments and in sequence on a smooth surface.

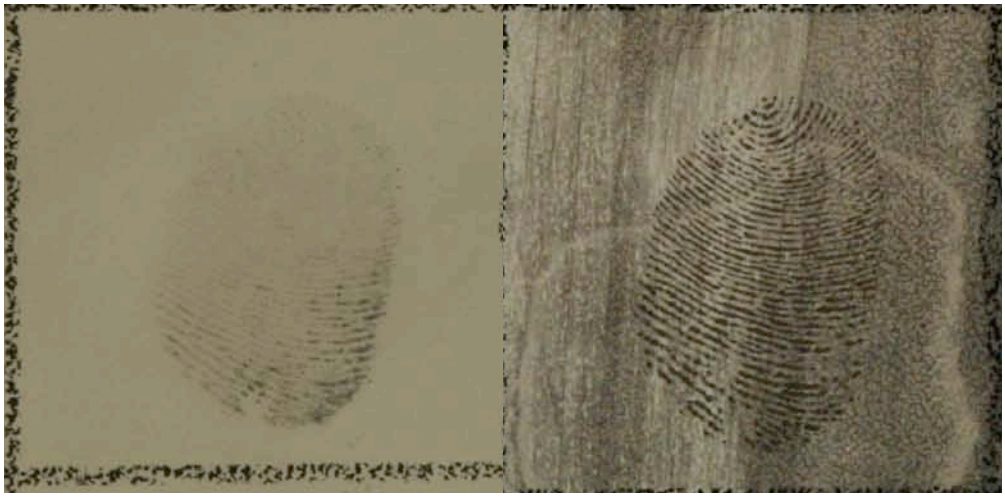


a) b)

Enhancement of a mark on a smooth painted surface a) after application of aluminium powder and b) improvement obtained by subsequent treatment with black powder suspension.



Typical results obtained comparing the effectiveness of powder suspensions, powders and superglue as single treatments and in sequence on a textured surface.



a) b)

Enhancement of a mark on a textured surface a) after application of black magnetic powder and b) improvement obtained by subsequent treatment with black powder suspension.

8.2.10 The overall trends on all surfaces examined are summarised in the table below.

Surface	**Best process/sequence**			
	Fresh marks (1 day)		**Older marks (> 1 week)**	
	Superglue + dye	**Powders + powder suspensions**	**Superglue + dye**	**Powders + powder suspensions**
S1		X		X
S2a		X		X
S2b				X
S2c		X	X	
S3a			X	
S3b		X	X	
S4				X
S5				X
S6			=	=
S7			X	
R1		X		X
R2a	X			X
R2b		X		X
R2c	X		X	
R2d	X			X
R2e		X		X
R3		X	X	
R4				X
R5		X		X
O1		X		X
O2a				
O2b	X		X	

O3		X		X

Best optimum processing process/sequence for different ages of mark across all surfaces studied.

8.2.11 A similar study was conducted with white powder suspensions on dark surfaces to enable firm recommendations to be made [18]. In this study the following surfaces were examined.

General surface classification	**Specific description and designation**
Smooth, non-porous	S1 Grey PVC
	S2 Black polypropylene
	S3 Dark brown wood effect melamine laminate
	S4 Black gloss painted metal
	S5 Dark blue ceramic tile
	S6 Black compressed polystyrene
Rough, non-porous	R1 Mottled grey kitchen worktop melamine
	R2 Black 'fake leather' laminate on aluminium
	R3 Black polythene
	R4 Black matt painted metal
	R5 Black textured compressed polystyrene

Description of the surfaces used in the comparative study between various sequences of superglue, powders and powder suspensions on dark surfaces.

8.2.12 In this study, 21 donors placed depletion series of 10 marks on each of the 11 different surfaces studied. The experiment looked at marks that were 1 week and 3 weeks old, giving a total number of 4,620 graded marks. The purpose of the experiment was to determine the optimum processing sequence, assuming that powders would always be the first process used. White powder suspensions and superglue + basic yellow 40 were compared in terms of their effectiveness as secondary treatments. A summary of the trends observed in the data across all surfaces studied is given below.

Surface	**Best process/sequence**			
	1-week-old marks		**Older marks (> 1 week)**	
	Powders + superglue/dye	**Powders + powder suspensions**	**Powders + superglue/dye**	**Powders + powder suspensions**
S1		X	X	
S2		X		X
S3	X		X	
S4		X		X

S5		X	X	
S6		X		X
R1	X		X	
R2	X		X	
R3	X		X	
R4		X		X
R5		X		X

Best optimum processing process/sequence for different ages of marks across all surfaces studied.

8.2.13 It was observed that the white powder suspensions were less effective than black powder suspensions in developing marks on non-porous surfaces. On smooth, dark surfaces, powders followed by white powder suspensions give closely equivalent performance to powders followed by superglue and both sequences can be recommended with equal weighting. On rougher, dark surfaces the sequence of powders followed by superglue gives better results and would be the sequence of choice, unless it is known that the surface has been wetted.

8.2.14 Comparative work has also been carried out to establish the effectiveness of powder suspensions on wetted non-porous surfaces [26, 27]. The results indicated that on certain wetted surfaces powder suspensions may be more effective than vacuum metal deposition [27]. Slight differences were also observed between the effectiveness of different formulations of powder suspension [26].

8.3 Pseudo-operational trials and operational experience

8.3.1 Initial operational trials have been carried out to compare the effectiveness of basic violet 3 with black powder suspensions on the adhesive side of tapes. The results of these trials are summarised in Chapter 3.2 Basic violet 3, and demonstrate that powder suspensions are the more effective process. However, superglue is also known to be a highly effective treatment for adhesive tapes. A subsequent operational trial commenced comparing iron oxide-based black powder suspensions with the superglue process, looking at marks developed on both adhesive and non-adhesive sides of the tape.

8.3.2 Based on the results obtained using carbon-based black powder suspensions, this operational trial was modified to include a commercial carbon-based formulation in addition to the iron oxide-based formulation. The trial results are recorded below.

Process	Cases	Number of positive results (cases)				% positive
		Non-adhesive	Adhesive	Both	Total	
Superglue/basic yellow 40	59	9	13	1	23	39
Iron oxide powder suspension	45	1	15	1	17	38
Carbon powder suspension	33	1	14	1	16	48

Operational trial results for different processes on light coloured adhesive tapes.

8.3.3 It can be seen that carbon-based black powder suspensions were found to be the most effective process for the adhesive side of tapes, and were therefore recommended for operational use.

8.3.4 The HOSDB white powder formulation was then used in an operational trial, comparing results with those obtained using superglue and dyeing. The trial results are summarised below.

Process	Cases	Number of positive results (cases)				% positive
		Non-adhesive	Adhesive	Both	Total	
Superglue/basic yellow 40	33	1	11	1	13	40
White powder suspension	39	1	11	2	14	36

Operational trial results for different processes on dark coloured adhesive tapes.

8.3.5 Superglue was found to be the more effective process on operational casework and white powder suspensions were not ultimately recommended for use on adhesive tapes, except in circumstances where dark tapes had become wetted.

8.3.6 Most recently, CAST has conducted a repeat of the pseudo-operational trial on plastic bags last conducted in 1986. In this trial, 100 bags and plastic packaging materials from different sources (e.g. supermarket carrier bags, 'bags for life', black bin bags, clear magazine wrappings) were collected from as realistic environments as possible. Each bag was divided into quarters, with each quarter being examined using a different fluorescence examination regime followed by a separate sequence of chemical treatments [21]. The number of marks developed using each

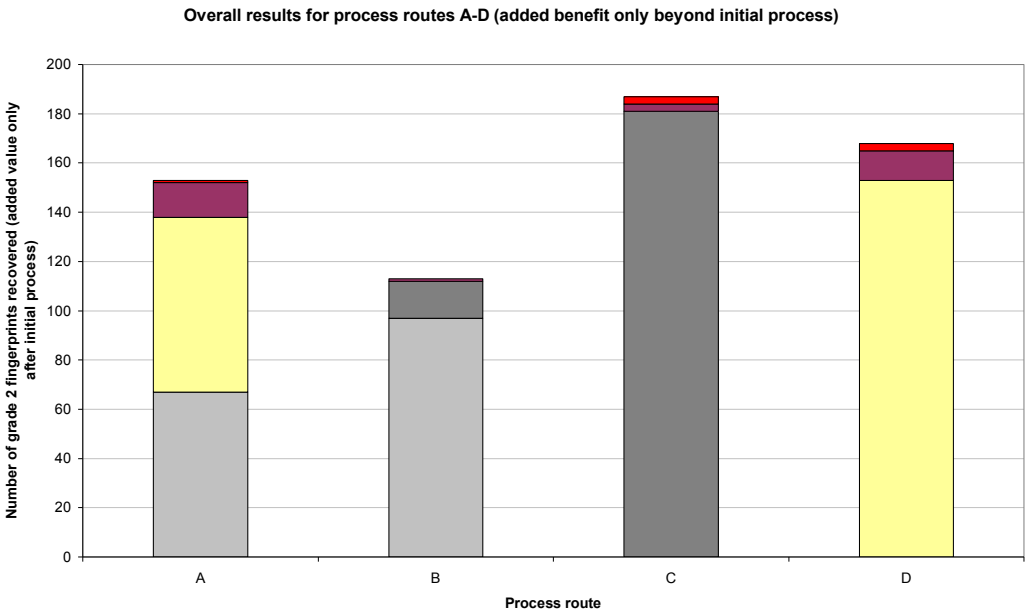
process was recorded. The results from the fluorescence examination stage in the trial have already been included in Chapter 2.2 Fluorescence examination. Iron oxide-based powder suspension was included as a chemical treatment in these trials, both as an initial process and as a secondary treatment subsequent to vacuum metal deposition (VMD).

8.3.7 The results of these trials for the first 50 bags are summarised below:

- Process route A = VMD –superglue – basic violet 3;
- Process route B = VMD –powder suspension – basic violet 3;
- Process route C = powder suspension – basic violet 3;
- Process route D = superglue – basic violet 3.

Process route	1st process	2nd process	Basic violet 3/ visible	Basic violet 3/ 577nm laser
A	67	71	14	1
B	97	15	1	0
C	181		3	3
D	153		12	3

Summary of the marks developed on plastic bags after each stage of sequential processing routes.

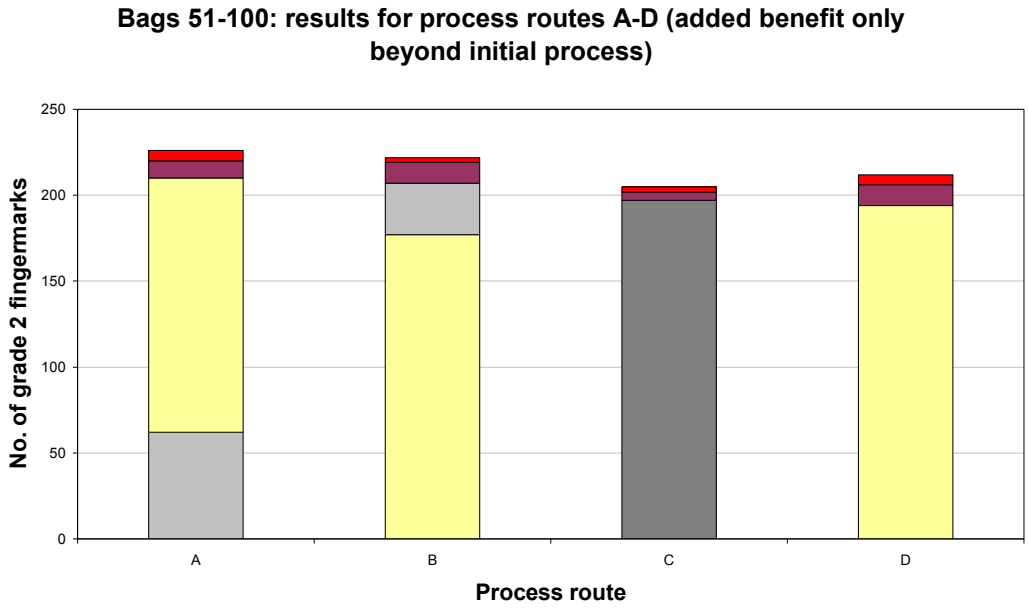


Graphical representation of the data summarised in the table above (light grey = VMD, dark grey = powder suspension, yellow = superglue + basic yellow 40, purple = basic violet 3 (visible), red = basic violet 3 (fluorescence with 577nm laser).

8.3.8 For the second 50 bags, the poorly performing process route B (VMD – powder suspensions – basic violet 3 route) was replaced by superglue – VMD – basic violet 3. The results from these studies are summarised below.

Process route	1st process	2nd process	Basic violet 3/ visible	Basic violet 3/ 577nm laser
A	62	148	10	6
B	177	30	12	3
C	197		5	3
D	194		12	6

Summary of the marks developed on plastic bags after each stage of sequential processing routes.



Graphical representation of the data summarised in the table above (light grey = VMD, dark grey = powder suspension, yellow = superglue + basic yellow 40, purple = basic violet 3 (visible), red = basic violet 3 (fluorescence with 577nm laser).

8.3.9 Powder suspensions performed well in these trials, giving equivalent, if not better, performance than any other single process. However, the superglue/VMD sequence gave the best results and it is this sequence that would be recommended, unless the bag is known to have been wetted. For wetted bags, the powder suspensions process is the main process recommended.

8.3.10 Many police forces have been using both black and white powder suspensions operationally in advance of the update to the *Manual of Fingerprint Development Techniques* [24], both in a laboratory as a replacement for superglue on items that may have been wetted (cowlings, car number plates) or contaminated (drugs wraps), and at

scenes after the application of powders. Results are still being collected but significant increases in the number of marks being developed are reported.

8.3.11 Powder suspensions have also been successfully used to develop marks on items recovered from arson scenes by more than one police force, in accordance with observations during CAST studies [28]. Laboratory tests have indicated that it may also be the best treatment for situations where cars have been sprayed with WD40 to destroy fingerprints [29].

8.3.12 It is clear that powder suspensions are a highly effective process for non-porous surfaces, give superior results to small particle reagent and may supersede superglue in some applications. CAST studies are continuing to establish the optimum position for the techniques within the full range of sequential processing charts.

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3.8 Small particle reagent

1. History

1.1 Small particle reagent (SPR) was first formulated in the mid-1970s by researchers at the Atomic Weapons Research Establishment (AWRE), Aldermaston, under a Police Scientific Development Branch (PSDB) contract. The objective of the contract was to devise a cheaper alternative to what was then termed surfactant 'stabilised physical developer (SPD)' (now known simply as physical developer) [1,2]. At the time, SPD was being investigated for the development of latent fingerprints on a range of surfaces, including plastics and paper, although it was recognised that the technique worked best on paper samples.

1.2 The SPD system was found to work by the deposition of silver particles, in the presence of a cationic surfactant, onto the surface being treated. Studies into this system showed that finely divided silver particles could also be used to develop latent fingerprints when prepared as a suspension, and that this behaviour was not exhibited when the suspension was dispersed in water alone, prompting studies into fine particle suspensions. This work indicated that the presence of the surfactant was essential if fingerprints were to be developed, and subsequent studies investigated a range of formulations incorporating different powders and surfactants. It was found that formulations containing powders with small particles of about 1μm suspended in a fluid at concentrations of between 1 and 10gl^{-1} were effective [2,3,4,5]. The generic name given to these systems was 'surfactant controlled SPR' and a provisional patent application covering such reagents was filed by Morris and Wells in 1976. A more comprehensive study of powders, surfactants and methods of application then followed [2,5].

1.3 Initial experiments showed that good results could be obtained using dish development with molybdenum disulphide (MoS_2) particulate and this was used as a control against which formulations based on alternative powders could be assessed [2,7]. These experiments identified the best performing powders as cobalt oxide(Co_2O_3), lead oxide (PbO_2), MoS_2, graphite and the pigment Monastral Blue (copper phthalocyanine), although for some of these there was a large batch-to-batch and supplier-to-supplier variation. In this respect MoS_2 was found to be the most consistent in performance across all batches tested. It was also found that all 'ionic' types of surfactant evaluated gave good results, but that poor results were obtained when the surfactant molecule has a 'tail' of fewer than eight carbon atoms (C_8) [2]. On the basis of these studies, a combination of Tergitol 7 and choline chloride was selected as the surfactant solution, although it was subsequently found that the latter constituent was unnecessary and it was omitted from the SPR formulation initially recommended for operational use. Subsequently, Tergitol 7 (3,9 diethyl-6-tridecanol hydrogen sulphate sodium salt) became unavailable because of the harmful impact it could

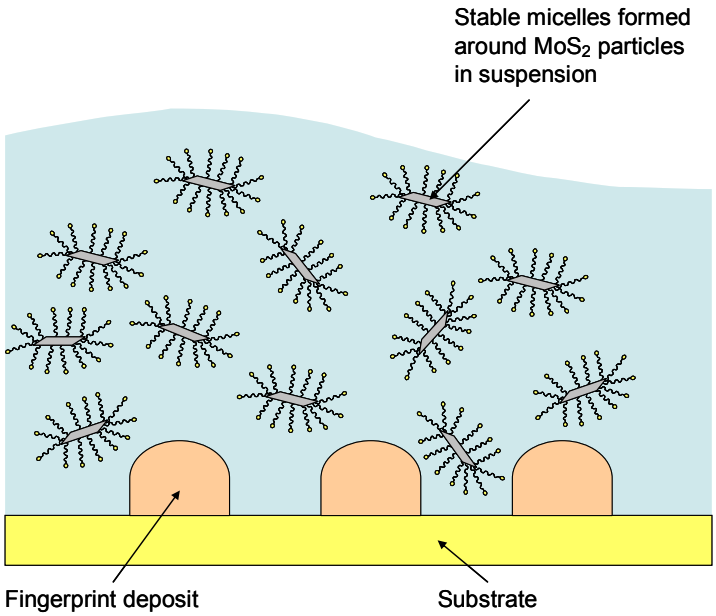
have on the environment, and a revised formulation was developed by the Home Office Scientific Research and Development Branch (HO SRDB) based on Aerosol OT (AOT) surfactant. It is this formulation that is recommended for use in the UK to the present day (2011).

1.4 SPR dish development was trialled operationally against vacuum metal deposition (VMD) for the development of marks on polythene bags [8]. This trial indicated that although SPR was not as effective as VMD for this type of surface, it was far more effective than powdering and the technique was recommended for operational use on non-porous surfaces and wetted items because it was recognised that few police forces had access to VMD.

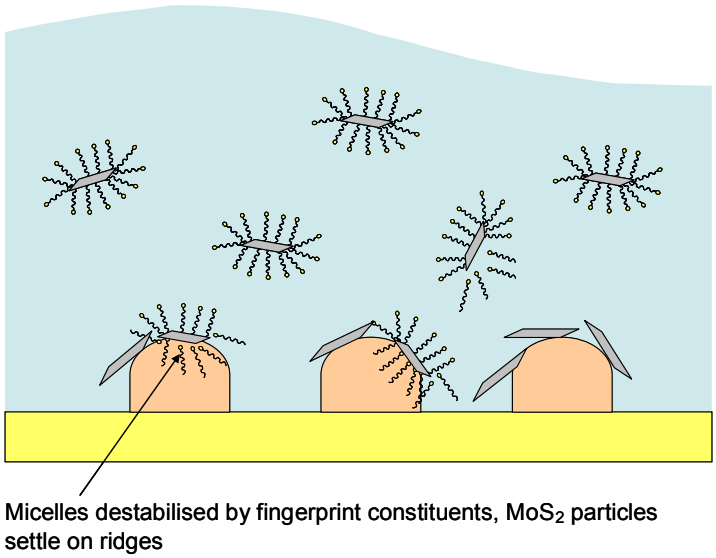
1.5 Work by the Home Office Central Research Establishment (HO CRE) in the early 1980 suggested that spraying of SPR was an effective method for cars which were wet and could not be dried, and for other exterior wetted surfaces such as windows and window frames. SPR was found to be capable of developing marks on surfaces exposed to the outside environment for prolonged periods of time, e.g. window glass. SPR was found to detect marks that had not been developed during aluminium powdering [9], although the presence of excess quantities of aluminium powder on the surface were found to inhibit SPR [10]. Operational trials using SPR alone on wetted surfaces, and surfaces that were still wet, demonstrated that the technique was effective in such circumstances [10] and it was subsequently recommended for operational use. However, it is recognised that spray application of SPR is less effective than dish development, and that use at crime scenes should be restricted to surfaces that cannot be recovered to a laboratory. It remained the principal treatment for fixed outdoor surfaces that are known to have been wetted until the recent development of powder suspensions in 2009.

2. Theory

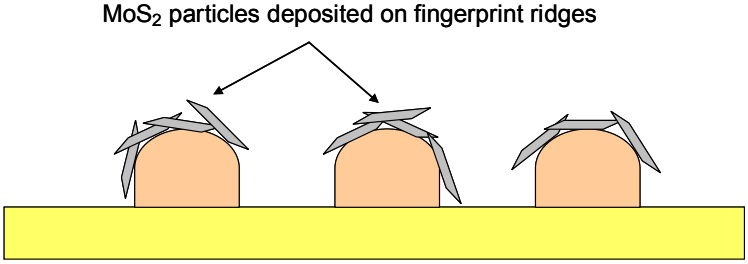
2.1 The mechanism by which SPR is thought to develop fingerprints is shown schematically in the illustrations below.


Stable micelles formed around MoS_2 particles in suspension
Fingerprint deposit
Substrate

a)


Micelles destabilised by fingerprint constituents, MoS_2 particles settle on ridges

b)


MoS_2 particles deposited on fingerprint ridges

c)

Schematic illustration of the small particle reagent process a) stable micelles formed around particles of molybdenum disulphide b) destabilisation of micelles by fingerprint constituents leading to particles settling on ridges and c) dried mark, leaving particles adhering to ridges.

2.2 The fine MoS_2 particles detect high molecular weight constituents and so adhere to the oily and fatty components of latent fingerprints by reaction between the fatty components present and the hydrophobic tails of the surfactant forming micelles around the particles. These tails are linked to a hydrophilic head, which reacts with metal salt to give a black precipitate, hence making the fingerprint visible.

3. CAST processes

3.1 The process recommended by CAST is first to prepare a concentrated solution by mixing 7.5mL of 10% AOT (also known by its chemical name dioctyl sulfosuccinate, sodium salt) solution with 500mL of tap water, then add 50g of MoS_2 powder. It may be difficult practically to prepare a 10% solution of AOT, and therefore the 10% solution should be attempted as the starting point and small quantities of water added until all solids are dissolved. This concentrated solution is then further diluted according to the development process being used. If the dish development SPR process is required, 4.5 litres of water are added to the concentrate and if the SPR is to be used for spray development 3 litres of water should be added.

3.2 The role of the AOT surfactant is to control the deposition of suspended particles onto fingerprint ridges in preference to the background surface. The surfactant will form micelles around the suspended particles and although the nature (anionic, cationic, non-ionic) of the surfactant is not critical there are properties that were found to be favourable in surfactant selection:

- it must be suitably soluble to achieve the optimum working concentration;
- the ‘tail’ of the surfactant should have an open carbon atom chain with no fewer than C_8, with the optimum number of carbon atoms in the chain being between12 and 17.

AOT meets both these criteria.

3.3 The concentration of AOT used is again not critical but must be controlled to be below the critical micelle concentration (CMC), the optimum being between one-third and one times of the CMC. The concentration used in both CAST formulations falls within these limits. If AOT concentration is below this limit, deposition of MoS_2 on the background surface increases and the definition of ridge detail is reduced, while at higher concentrations the clarity of the print diminishes and at best only a very faint outline of the print is observed. At these high

concentrations little general deposition takes place, signifying that micelle formation blocks the process of deposition, perhaps by providing a more attractive species for adsorption on the fingerprint deposit.

3.4 The role of the MoS_2 is to deposit preferentially on the fingerprint ridges and aid the visualisation of the mark. Several different materials can be used in this role, but in general the best results were obtained with materials with a density of ~4 gcm^{-3} and a layer lattice structure, both of which apply to MoS_2. There must be a sufficient quantity of MoS_2 in suspension for the particles to adhere to the fingerprint ridges and give a clear print. However, if the quantity is too great the powder also adheres to the background, giving background staining and smudging the developed ridges. The quantity used in the CAST formulation is sufficient to give good development without background staining.

3.5 Uniform wetting of the powder by the AOT surfactant is difficult to achieve if the powder is directly added to the working concentration solution of surfactant, so the MoS_2 should be added to a concentration greater than the CMC and after dispersion, diluted to the working concentration.

4. Critical issues

4.1 There are no critical issues relating to the application of SPR. The formulation is tolerant of changes in water content and made up solutions will keep indefinitely. In very cold weather additions of ethanol may be required for the spray application method to work effectively.

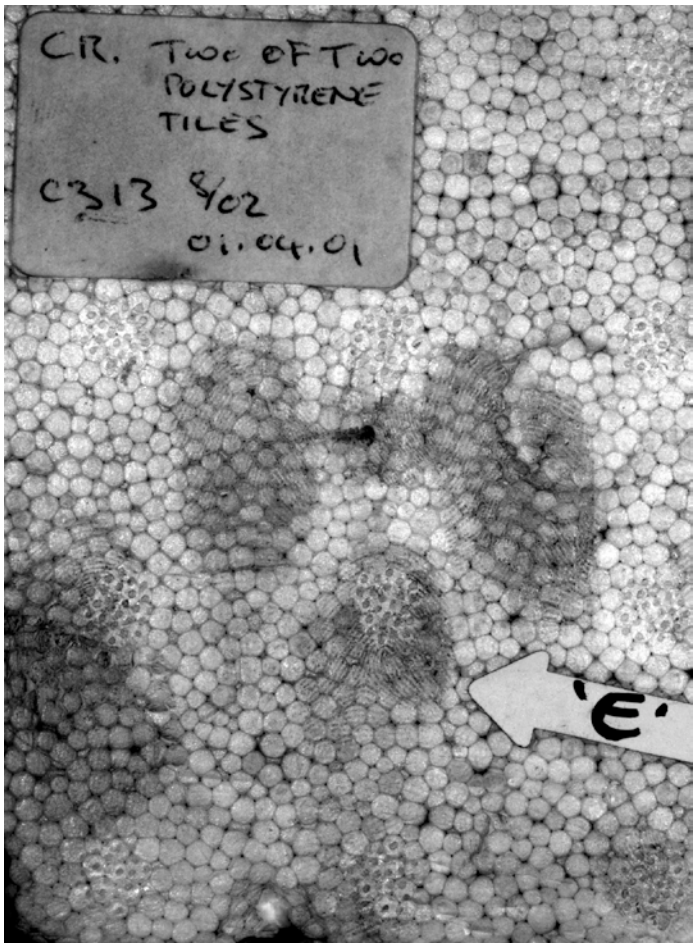
5. Application

5.1 Suitable surfaces: SPR is suitable for use on non-porous surfaces, such as plastic bags, glass bottles, waxed paper and other waxy items, such as candles. It can be used on expanded polystyrene items such as drinking cups. It will still develop marks on surfaces that have been wet, but is not suitable for heavily contaminated surfaces.

5.2 SPR is a process recommended for use on non-porous articles that have been wetted. Because the process targets the insoluble lipid components of fingerprint residues, immersion in water or exposure to rain will in many cases leave sufficient deposits for SPR to continue to develop marks. It is not as sensitive as VMD for this type of exhibit, but for the majority of police forces that do not have VMD equipment, SPR was until 2009 the only option for non-porous articles known to have been wetted. Tests have indicated that SPR may still develop additional marks if used in sequence before powder suspensions on wetted non-porous surfaces.

5.3 The two application techniques recommended for operational use are dish development and spray application. The dish development

technique can be applied to non-porous surfaces, such as plastic bags and packaging materials, waxed and plastic-coated paper, small gloss painted or glass articles and expanded polystyrene articles, such as drinking cups and ceiling tiles. Such items are difficult to treat with superglue, where uptake of the fluorescent dye by the expanded polymer makes any marks developed very difficult to visualise.



Fingerprints developed on expanded polystyrene tile using small particle reagent.

5.4 A tray or tank of sufficient size for the article being processed should be filled with sufficient working solution to enable the article to be submerged 50mm below the surface. The working solution is then stirred to ensure all powder is in suspension before submerging the article with the surface of interest facing upwards. The article is then kept submerged and stationary for 30 seconds while the MoS_2 particles come out of suspension and settle evenly over the object. For small, complex shaped articles the article may be placed in a dish and the working solution poured over it from a beaker. The article is then removed carefully from the dish and the uniform grey deposit carefully washed off by placing the surface of interest face downwards into a second dish of water and agitating it gently. The article should then be dried at room temperature. The dish development technique limits the size of the

article that can be treated in the laboratory, but for use at scenes a formulation for spray application has been developed.

5.5 Spray application may be carried out on all non-porous surfaces, but it is recommended for objects that are outside, awkwardly shaped, large or immovable. Although wet or damp articles can be processed, when treating articles outside, the area being treated needs to be sheltered from direct rainfall.

5.6 For spray application, a simple, commercially available garden spray unit is used. The nozzle of the unit should be set to give a conical, fine spray and the filter unit removed to prevent it clogging. The working solution should be shaken to give an even particulate distribution and the area to be processed should be sprayed liberally, starting at the top edge and working down towards the bottom. As the liquid runs down the surface fingerprints may begin to become visible and spraying should be continued just above the relevant area until there is no more build up of the grey deposit. A second spray unit filled with water is then sprayed above the developed fingerprints before they have dried, allowing the flowing water to carry away excess particles. Prints should not be directly sprayed with water as this may damage them. In cold weather, 200ml of ethanol may be added per 1 litre of suspension to prevent freezing on the surface.

Spray application of small particle reagent to a car.

5.7 The spray formulation is much less effective than the dish formulation and should only be used where dish development is not possible.

5.8 Studies have shown that SPR has potential for developing fingerprints in specialist applications, such as on wetted firearms [11] and on incendiary bottles soaked in accelerant [12].

6. Alternative formulations and processes

6.1 Several other particles have been investigated as the basis of SPR. Some of those investigated in early studies [2] are summarised in the table below.

Material type	**Compound**	**SPR performance**
Metals	Silver powder	Good
	Zinc powder	Fair
	Aluminium powder	Fair
	Aluminium fingerprint powder	Fair
	Lead powder	Poor
	Copper powder	Poor
	Iron powder	Poor
	Manganese powder	Poor
Metal oxides	Iron (Fe_2O_3)	Good
	Cobalt (Co_2O_3)	Excellent
	Chromium (Cr_2O_3)	Good
	Uranium (UO_2)	Good
	Lead (Pb_3O_4)	Poor
	Lead (PbO_2)	Excellent
	Manganese (MnO_2)	Good
	Silver (Ag_2O)	Fair
	Copper (CuO)	Good
Metal sulphides	Zinc (ZnS) Batch 1	Excellent
	Zinc (ZnS) Batch 2	Poor
	Molybdenum (MoS_2) Batch 1	Excellent
	Molybdenum (MoS_2) Batch 2	Good
Other	Tungsten carbide (WC)	Good
	Silicon carbide (SiC)	Good
	Titanium boride (TiB_2)	Poor
	Carbon (amorphous)	Good
	Carbon (graphite)	Excellent
	Monasterol Blue	Good

Summary of compounds investigated as the basis of small particle reagent.

6.2 As described above, MoS_2 was ultimately selected because it gave good performance and was more consistent in performance across different batches and different manufacturers. A further series of powders

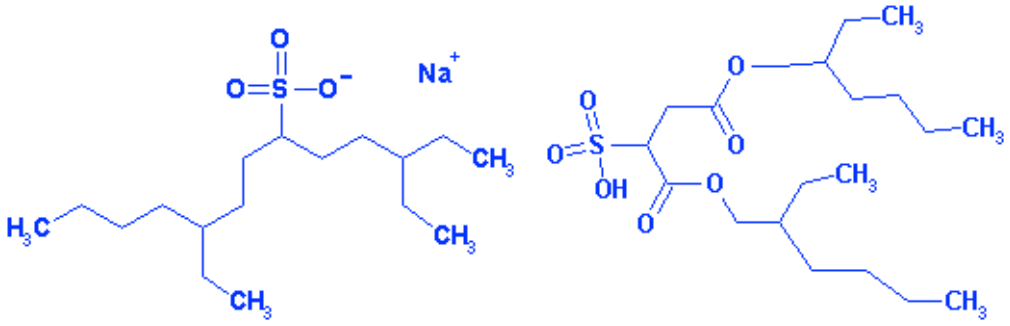
including boron nitride, cadmium sulphide, cadmium selenide, kaolin, molybdenum carbide, silicon nitride and tungsten sulphide were subsequently investigated [13] but none were found to give better performance.

6.3 In addition to the particulate component, a range of different surfactants were investigated [2]. These are also summarised below.

Surfactant			Performance
Name	**Chemical**	**Ionic type**	
Teepol 610	Sodium lauryl sulphate	Anionic	Good
Teepol 514	Sodium lauryl sulphate	Anionic	Good
–	Sodium lauryl sulphate	Anionic	Good
Teepol Green	Sodium lauryl sulphate	Anionic	Good
Tergitol	Heptadecyl sulphate	Anionic	Excellent
Manoxal 1B	Dibutyl sodium sulfosuccinic acid	Anionic	Very poor
Manoxal OT	Diacetyl sodium sulfosuccinic acid	Anionic	Good
Armac 12D	Lauramine acetate	Cationic	Fair
-	Lauramine acetate	Cationic	Fair
Choline citrate	Trimethyl 2 hydroxy ethyl amine citrate	Cationic	Poor
Choline chloride	Trimethyl 2 hydroxy ethyl amine chloride	Cationic	Poor
Hyamine 2389	Methyl, dodecyl benzyl trimethyl amine chloride	Cationic	Good
Hyamine 1622	Di isobutyl phenoxy ethoxy benzyl amine chloride monohydrate	Cationic	Excellent
Brij 35	Phenoxy ethylated lauryl alcohol	Non-ionic	Excellent
Lissapol NDB			Fair
Lissapol D	Sodium acetorley sulphate		Fair
Lissapol LS	Sodium N octyl amino sulphonic acid		Fair
Flow 7X	Unknown		Good
Photoflo	Unknown		Good

Summary of surfactant systems considered for use in small particle reagent.

6.4 A further range of surfactants were subsequently studied [13] including Nonidet P40, Triton GR-5, Triton X405, and the series of Tween surfactants 85, 80, 40, 20. Manoxol OT (another trade name for AOT) gave the best performance and ultimately replaced Tergitol 7 in the operational formulation when the latter surfactant became unavailable.



The structures of Tergitol 7 ($C_{17}H_{35}NaO_3S$) and Aerosol OT ($C_{20}H_{37}NaO_7S$).

6.5 More recently, there has been much interest in the use of powder suspensions for the development of fingerprints on the adhesive side of tapes. These have similarities to some of the formulations evaluated for SPR, albeit with far higher solids content, and are available in black (with carbon or iron oxide particulates) and white (with titanium dioxide particulates) forms. It was found that some of these formulations work very well on non-porous surfaces and recent results indicate that they will supersede SPR in this application on most types of surface. A detailed description of these formulations is given in Chapter 3.7 Powder suspensions.

6.6 SPRs based on other particulates have been reported, including light coloured zinc carbonate [14] and fluorescent particles [15]. Commercial, pre-mixed formulations are also available in various colours. The relative effectiveness of these formulations has not been tested by CAST against the recommended process, and in the case of the commercial pre-mixed products the nature of the filler particles is not known.

7. Post-treatments

7.1 Once entirely dry, marks developed using SPR are essentially the same as a mark developed by a regular powdering technique and can therefore be lifted in the same way by low-tack, clear adhesive tapes [3,11]. On occasions the lifting tape may not adhere to the surface very well, so care must be taken not to let the tape slip when lifting the developed mark. Lifting fingerprint marks is especially useful when dealing with highly patterned and/or coloured surfaces, however damage may be caused to the mark during lifting and the priority should be to photograph the mark in situ first.

8. Validation and operational experience

8.1 Laboratory trials

8.1.1 CAST has carried out few laboratory trials of SPR because the formulation was developed by the Atomic Weapons Research Establishment AWRE and HO CRE, and until recently there has been no other process for treating fixed, outdoor surfaces that have been wetted, to carry out a comparison with. A small-scale study using split depletion series was carried out in 1992 when the surfactant was changed from the discontinued Tergitol 7 to AOT [16]. This test used five different donors, each depositing five prints on three different plastics. These results, and the grading scheme used, are summarised below.

1 = no obvious development
2 = print area visible but poorly defined ridge structure
3 = some clear ridge structure
4 = useful mark

Grade	**John Lewis white plastic bag**		**Sainsbury's white plastic bag**		**Clear plastic**	
	Tergitol 7	AOT	Tergitol 7	AOT	Tergitol 7	AOT
1	4	4	2	9	0	2
2	5	4	7	2	2	7
3	9	12	7	5	8	6
4	7	5	9	9	15	10

Results of comparative studies on plastic bags using different small particle reagent formulations.

8.1.2 It can be seen that the AOT formulation is slightly less effective than the Tergitol 7-based formulation, but a range of equivalent tests carried out using different surfactants showed that AOT was the best performing Tergitol 7 replacement and it was therefore incorporated into the revised formulation for operational use.

8.2 Pseudo-operational trials and operational experience

8.2.1 HO CRE carried out several trials before implementing SPR. In the initial investigation, SPR was compared with powders and VMD on paper, polythene and window glass surfaces [9]. On paper SPR gave reasonable results, but it affected subsequent ninhydrin treatment and therefore could not be used in sequence. On polythene, SPR was shown to be capable of developing marks on polythene that had been exposed to the environment (including rain), but VMD gave better results. This observation was confirmed in a full operational trial [8], the results of which are summarised in Chapter 3.11 Vacuum metal deposition. On window glass SPR gave similar performance to aluminium powder on the inside surface. However, on the outside, which had been exposed to

autumnal weather conditions for two weeks, SPR gave significantly improved performance and could be used in sequence after powders.

8.2.2 An operational trial was then conducted over two winter months using three police forces, spray applying SPR after powdering [10]. These initial trials gave poor results, which were attributed to excessive application of aluminium powder inhibiting SPR, and therefore sequential processing was not recommended at scenes. A second phase of the operational trial was carried out over two months using four police forces, applying SPR to surfaces that had not been previously powdered. During this trial 106 outside surfaces were examined and 55 useful prints recovered from 24 of the surfaces. Of these surfaces, five were examined while still wet (not possible with powders) and seven useful marks were recovered. SPR was therefore recommended for use on wet or damp surfaces and at scenes of crime on articles where powdering is not feasible.

8.2.3 With the development of the superglue process (see Chapter 3.10 Superglue), the effectiveness of SPR was compared with that of superglue and VMD in a pseudo-operational trial on polythene bags. The results are reported in detail in Chapter 3.11 Vacuum metal deposition and indicated that SPR was less effective than superglue and dyeing and VMD on this type of surface, in accordance with earlier studies.

8.2.4 However, until recently (2009) SPR remained the process of choice where non-porous exhibits had been wetted and were either not portable or could not be treated with VMD. In the last two years it has become apparent that powder suspensions give superior performance to SPR on most surfaces studied, and advice is in the process of being updated to reflect this change in recommendations.

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3.9 Solvent black 3 (Sudan Black)

1. History

1.1 Solvent black 3, alternatively known as Sudan Black B, is one of a class of azo dyes. Although some related compounds such as Sudan III (solvent red 23) and Sudan IV (solvent red 24) were available in the late 1800s and early 1900s, solvent black 3 was not introduced until the mid-1930s. Industrially, the dye is used for the coloration of organic solvents, printing inks, lacquers and a range of fats and wax substances [1].

1.2 Soon after its introduction the dye was proposed as a stain for fats and various other microbiological applications and has been successfully utilised in this role to this date (2011). The first published use of solvent black 3 for the development of latent fingerprints was by Mitsui *et al.* in 1980 [2]. They used a solution of solvent black 3 in a mixture of ethylene glycol, ethanol and water to develop prints on water-soaked paper items, the performance of Solvent Black 3 being shown to be superior to ninhydrin on this type of exhibit. This was soon followed by a further study by Stone and Metzger [3], comparing solvent black 3 with black magnetic powder on wetted porous items. In this comparison magnetic powder was found to give the best results.

1.3 In the early 1980s the Home Office Central Research Establishment (HO CRE) conducted an evaluation of over 60 biological dyes for their ability to develop latent fingerprints on both paper and polythene surfaces [4]. These studies also identified solvent black 3 as having particular potential for the development of fingerprints, in this case the best results being obtained on polythene. It was decided to proceed with an operational trial comparing the effectiveness of solvent black 3 with the two existing techniques recommended for polythene at the time, vacuum metal deposition (VMD) and small particle reagent (SPR) [5]. An initial phase of the work suggested that solvent black 3 gave superior results to VMD on polythene bags and the study was extended to a full operational trial. In these more detailed studies both VMD and SPR were found to be more effective than solvent black 3 and the reagent was not considered further for these applications.

1.4 The Police Scientific Development Branch (PSDB) subsequently re-evaluated the reagent and found that it had potential for developing fingerprints in cases where surfaces were contaminated and powdering was not possible. Examples of this type of surface included takeaway food containers or fizzy drinks cans. The process was subsequently included in the CAST *Manual of Fingerprint Development Techniques* [6] and recommended for these applications.

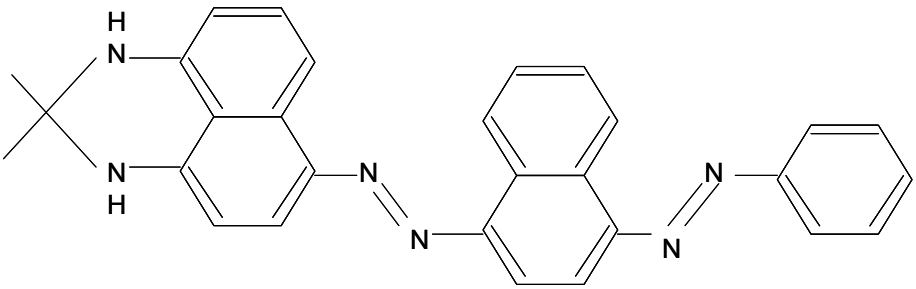
1.5 PSDB carried out a re-evaluation of a range of lipid reagents in 1999–2000 [7] and investigated several other lysochromes including Oil Red O (solvent red 27) and Sudan III (solvent red 23). These studies confirmed solvent black 3 to be the best performing of this type of lipid dye and it

was not considered worthwhile initiating development of formulations based on other dyes. Instead, research was initiated to develop a formulation based on a less flammable solvent than ethanol that gave potential for the reagent to be used at crime scenes. As a consequence of this research 1-methoxy-2-propanol was identified as a suitable solvent and laboratory trials indicated that there was no discernible difference between this and the ethanol-based formulation. This formulation was subsequently published for operational use [8]. The studies did raise the issue of how best to test reagents for contaminated surfaces, because a method for consistently contaminating test surfaces needs to be devised. Several alternative techniques were investigated during the course of the experiments [9] but none of these were regarded as being truly satisfactory.

1.6 In the interim, there has been very little published work on the use of solvent black 3. One recent study assesses the effectiveness of solvent black 3 in both powder and solution form, with the solution treatment found to be more effective. Marks up to 75 days old were successfully detected on porous surfaces using this approach [10]. The authors also recommend the reagent for development of lipstick marks.

2. Theory

2.1 Solvent black 3 is a lysochrome, more commonly known as a fat stain. Most lysochromes are azo dyes, which because of their structure have undergone molecular rearrangement making them incapable of ionising.



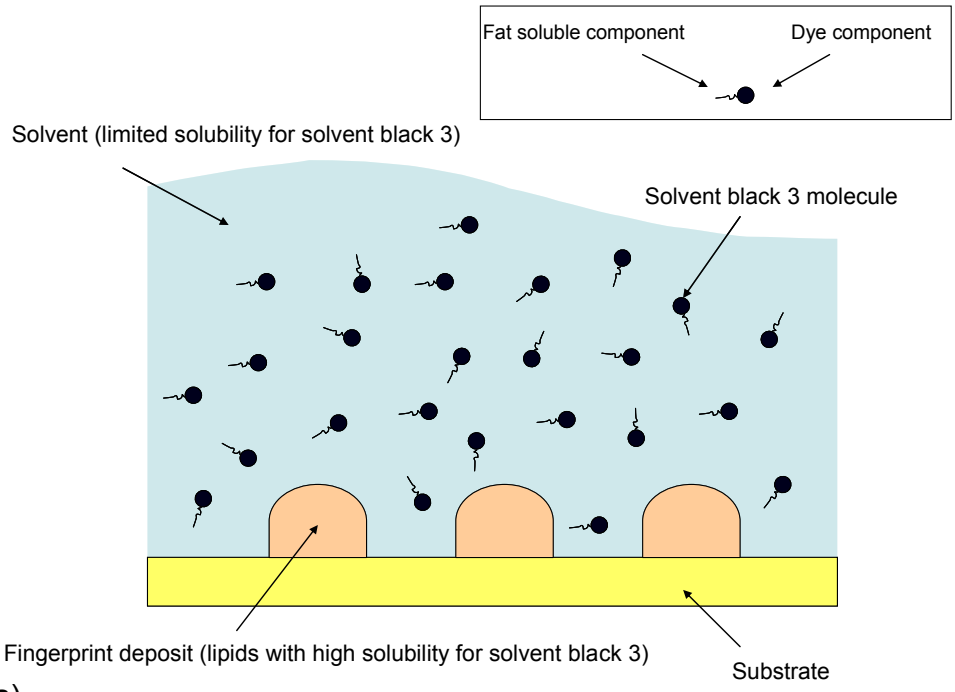
Structure of solvent black 3.

2.2 The basis for these dyes colouring fats is that they dissolve into them. From another perspective, the fat is the solvent for the dye. Lysochromes are mostly insoluble in strongly polar solvents, such as water, and somewhat more so in less polar solvents, such as ethanol. They are quite strongly soluble in non-polar solvents, such as xylene. Triglycerides, being non-polar compounds, dissolve them quite well. Other lipids, having fatty components, may also dissolve them.

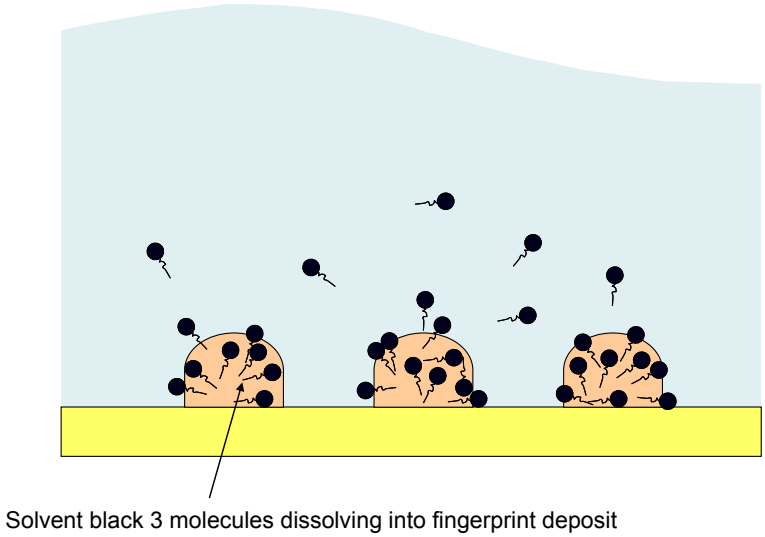
2.3 Lysochromes such as solvent black 3 are applied from solvents in which they are sparingly soluble. As they come into contact with materials in which they are strongly soluble, they transfer to them significantly, often colouring them more strongly than the original solvent. This process is known as preferential solubility.

2.4 Although the primary action of solvent black 3 is to stain lipids by dissolving in them, it can also stain materials ionically. This may result in some background staining.

2.5 The dyeing process of solvent black 3 is illustrated schematically below.



a)



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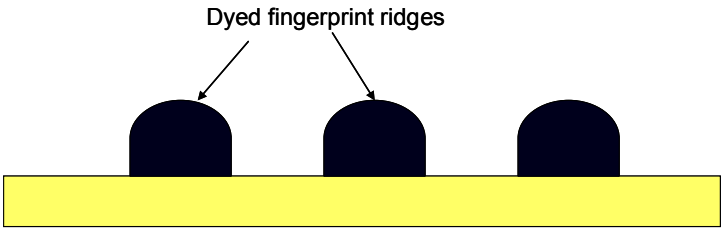


c)

Schematic illustration of the solvent black 3 process a) solvent black 3 molecules in solvent with limited solubility b) lipophilic component of solvent black 3 molecule preferentially dissolving into lipids in fingerprint ridges and c) fingerprint after drying, leaving dyed ridges.

3. CAST processes

3.1 The process recommended by CAST does not differ significantly from that originally proposed by the HO CRE. The solution consists of 15g of solvent black 3 dissolved in 1 litre of ethanol, to which is subsequently added 500ml of distilled water.

3.2 The role of solvent black 3 in the formulation is to act as the dye for the fingerprint ridges. The concentration used is such that the limit of solubility in the ethanol/water solvent is almost exceeded, and some precipitation of solvent black 3 is occurring. It was proposed by HO CRE that these precipitating particles may preferentially settle on fingerprint ridges in addition to the dyeing action of solvent black 3 dissolving into the lipids.

3.3 The role of ethanol is to act as the initial solvent for solvent black 3 and it is capable of dissolving the quantity of solvent black 3 outlined above.

3.4 Solvent black 3 is insoluble in water, and the addition of water reduces the solubility of solvent black 3 to the point where precipitation is beginning to occur.

3.5 In the more recently developed, less flammable formulation of solvent black 3, 1-methoxy-2-propanol fulfils the same role as ethanol while having a reduced flammability.

4. Critical issues

4.1 Any metallic films forming on the surface of the working solution should be removed using tissue or blotting paper prior to use. This is because these films will otherwise cause excessive staining of the background and may obscure marks.

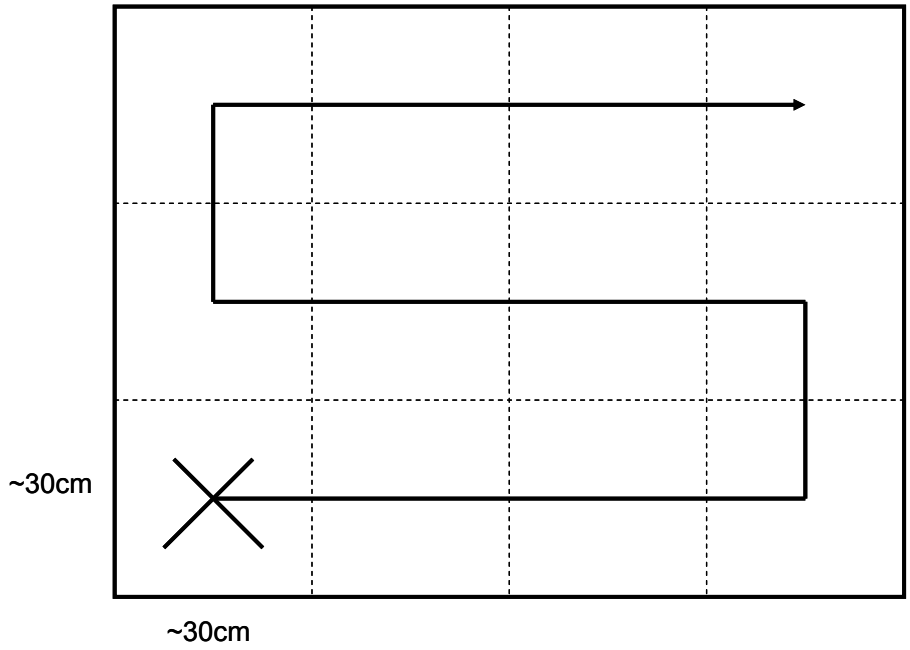
5. Application

5.1 Suitable surfaces: Solvent black 3 is suitable for use on all types of non-porous surface where particular types of contamination are present. The two types of contamination for which solvent black 3 is known to be effective are: fatty deposits (similar in nature to sebaceous fingerprints), and drinks' residues, where chemical discrimination may be of value.

5.2 Solvent black 3 is not recommended as a primary treatment for any particular surface, but appears in several of the processing charts for non-porous surfaces as a treatment for surfaces that have been contaminated. In these situations, the lipid specific nature of solvent black 3 may enable it to selectively stain fingerprint ridges without causing background staining of the contaminant. Basic violet 3 can be considered as an alternative treatment for contaminated surfaces and although laboratory trials indicate that solvent black 3 may be more effective than basic violet 3 on latent prints, the most effective treatment on contaminated surfaces has not been conclusively identified. Examples of the types of exhibit that can be effectively treated with solvent black 3 include fast food containers and drinks cans.

Photograph of beer can treated with solvent black 3, showing developed ridge detail.

5.3 A formulation of solvent black 3 with reduced flammability has recently been developed [11] with the potential for use at scenes. The types of scenes where this formulation could be used include potentially contaminated areas such as kitchens and bathrooms. Guidelines for application are given [7,11], starting application at the bottom of the surface and then working up. This minimises dye running down over unprocessed areas and affecting subsequent development of marks.



a)

b)

Application of solvent black 3 at scenes of crime a) suggested application sequence for vertical surfaces, and b) solvent black 3 being applied to a cupboard.

6. Alternative formulations and processes

6.1 HOSDB carried out an evaluation of a range of alternative solvents with the objective of providing a less flammable solvent black 3 formulation with the potential for use at scenes of crime. These solvents were tested individually, and in some cases diluted with water or heptane. A summary of the systems evaluated is given in the table below.

Solvent	**Formulations examined**	**Flammability**	**Results**
Dichloromethane/ Heptane	Various	Not studied	Only faint staining of prints
Ethyl acetate/ Heptane	Various	Not studied	Only faint staining of prints
Acetone	25, 50, 75, 100%	Similar to ethanol formulation	Similar level of fingerprint development with existing ethanol formulation
Propan-2-ol	25, 50, 75, 100%	Slightly lower than ethanol formulation	Similar level of fingerprint development with existing ethanol formulation
Propylene carbonate	100%	Not studied	Immiscible with water – poor results
Propylene glycol methyl ether acetate (PGMEA)	100%	Not studied	Immiscible with water – poor results
Dipropylene glycol dimethyl ether (DPGDME)	100%	Not studied	Immiscible with water – poor results
2,2-Dimethoxy Propane (2,2-DMP)	100%	Not studied	Immiscible with water – poor results
Propan-1,2,3-triol	Various	Ethanol had to be added to dissolve Solvent black 3, similar	Solvent black 3 not soluble in glycerol or water/glycerol mix
Propan-1,2-diol	25, 50, 75, 100%	Lower than ethanol formulation	Poor performance in staining marks
Propylene glycol methyl ether (PGME)	Various, including 40, 50, 55, 60, 75%	Much lower than ethanol formulation	Equivalent level of fingerprint development to existing ethanol formulation

Dipropylene glycol methyl ether (DPGME)	Various, including 30, 40, 50, 60%	Much lower than ethanol formulation	Equivalent level of fingerprint development to existing ethanol formulation

Solvents investigated as alternatives to ethanol in the solvent black 3 formulation.

6.2 The results indicated that solvent black 3 was soluble in most polar organic solvents and that formulations based on diluted solvents worked better in the development of fingerprints. Water was found to be essential to give good fingerprint development.

6.3 Of the range of solvents investigated, propylene-based glycol ethers were identified as best performing group in terms of reduced formulation flammability and good fingerprint development. Optimised formulations were subsequently developed based on propylene glycol methyl ether (PGME) and dipropylene glycol methyl ether (DPGME). Further detail on both of these solvents is provided below.

6.4 Propylene glycol methyl ether (PGME, 1-methoxypropan-2-ol, dowanol PM)
Molecular formula: $C_4H_{10}O_2$
CAS number: 107-98-2
Boiling point: 118–119°C
Flash point: 33.88°C
Lower Flammability Limit: 1.8%
Upper Flammability Limit: 16.0%
Purity: 99.5+%
Main contaminant: 2-methoxypropan-2-ol

6.5 Dipropylene glycol methyl ether (DPGME, dowanol DPM)
Molecular formula: $C_7H_{16}O_3$
CAS number: 34590-94-8
Boiling point: 90–91°C
Flash point: 74°C
Purity: 97% (mixture of isomers)

6.6 The two best performing systems of those optimised were:

10g solvent black 3, 500mL PGME, 500mL water (50%);
10g solvent black 3, 400mL DPGME, 600mL water (40%).

6.7 The flash points of both PGME and DPGME-based solvent black 3 formulations were also assessed, and found to be:

PGME = 55ºC;
DPGME >87ºC.

6.8 Considering that both these flash points were well in excess of temperatures experienced at scenes and that effectiveness in developing fingerprints was equivalent to the existing ethanol-based formulation, the PGME-based formulation was ultimately recommended for operational use both at scenes and in the laboratory.

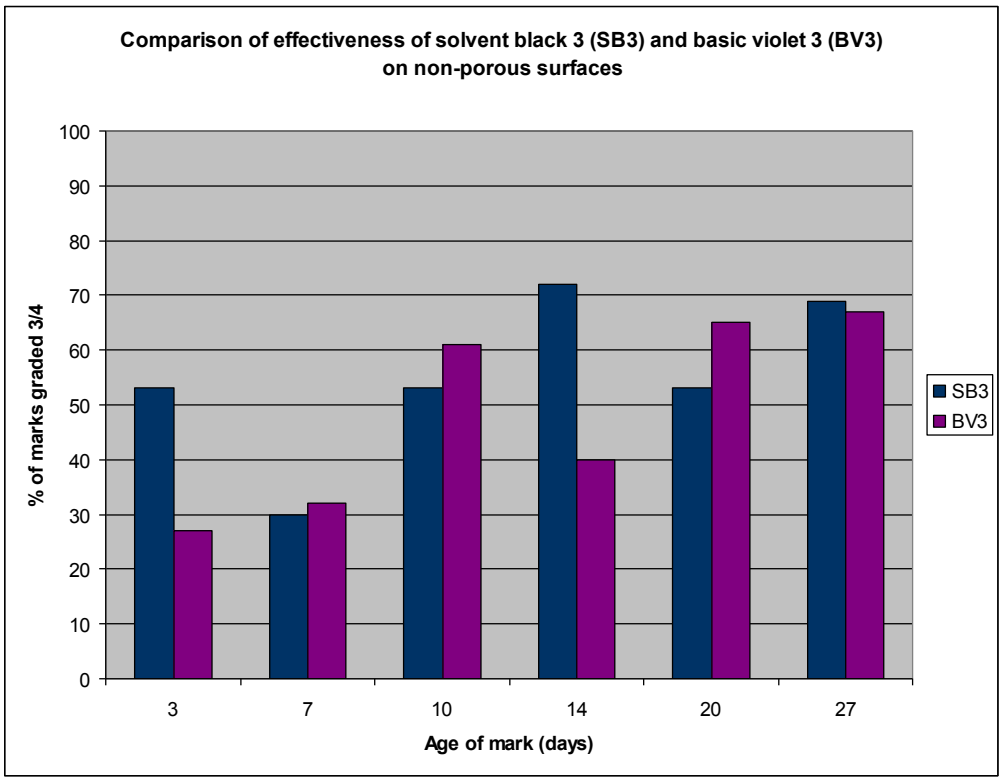
7. Post-treatments

7.1 No post-treatments are used after solvent black 3.

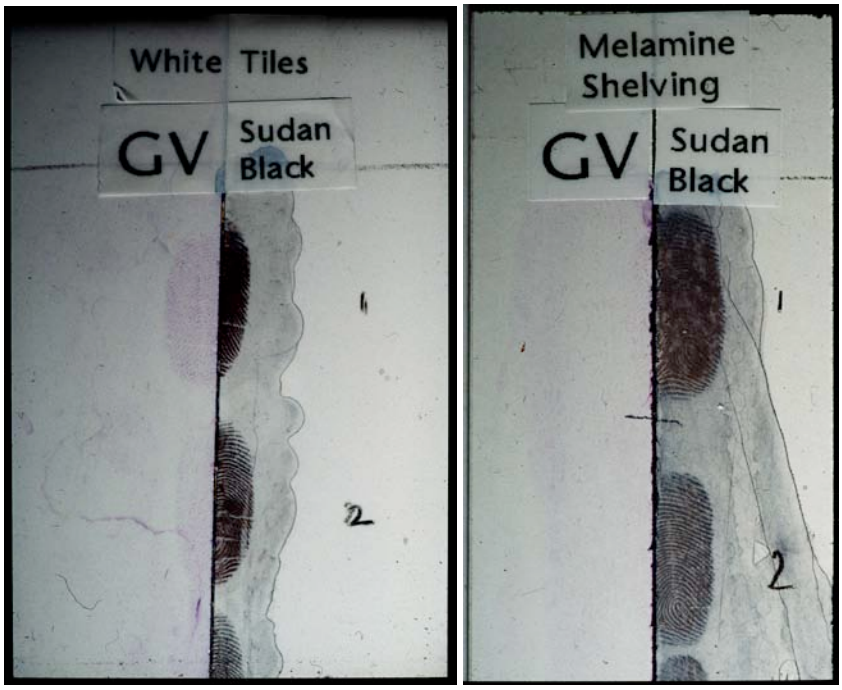
8. Validation and operational experience

8.1 Laboratory trials

8.1.1 The effectiveness of solvent black 3 on non-porous surfaces has more recently been evaluated in a laboratory trial, comparing it with the other reagent recommended for contaminated surfaces, basic violet 3. The results of this trial, carried out on 2,592 half prints, are illustrated below.



a)



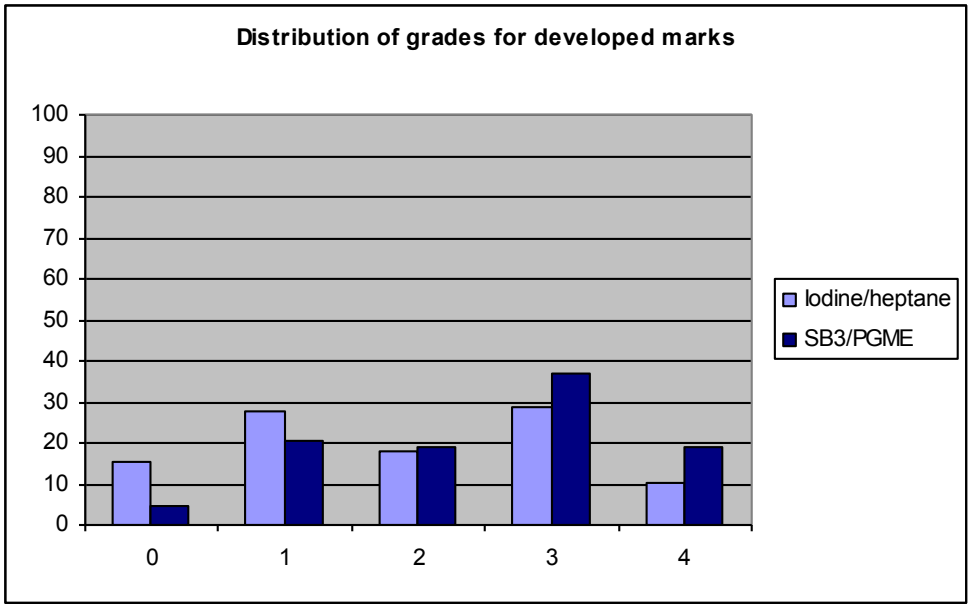
b)

Results from a comparison of the effectiveness of solvent black 3 (Sudan Black) and basic violet 3 (GV) on non-porous surfaces a) results of grading marks of different ages and b) photographs of marks developed on different surfaces.

8.1.2 These results indicate that solvent black 3 may be more effective, but are not conclusive. The trials were conducted on clean non-porous surfaces and are therefore not fully representative of the contaminated surfaces that the techniques are proposed for. However, there are reduced health and safety issues associated with solvent black 3, which may make it preferable to basic violet 3 for operational use on contaminated exhibits.

8.1.3 Comparisons were also carried out between solvent black 3 and a heptane-based iodine solution. This involved grading 2,592 half prints, the results of numbers of mark at each grade being summarised below:

Grade	Technique		
	Iodine/heptane	**SB3 after iodine**	**SB3/PGME**
0	297	63	83
1	361	152	304
2	212	95	245
3	341	326	465
4	85	84	199
Total	**1,296**	**720**	**1,296**

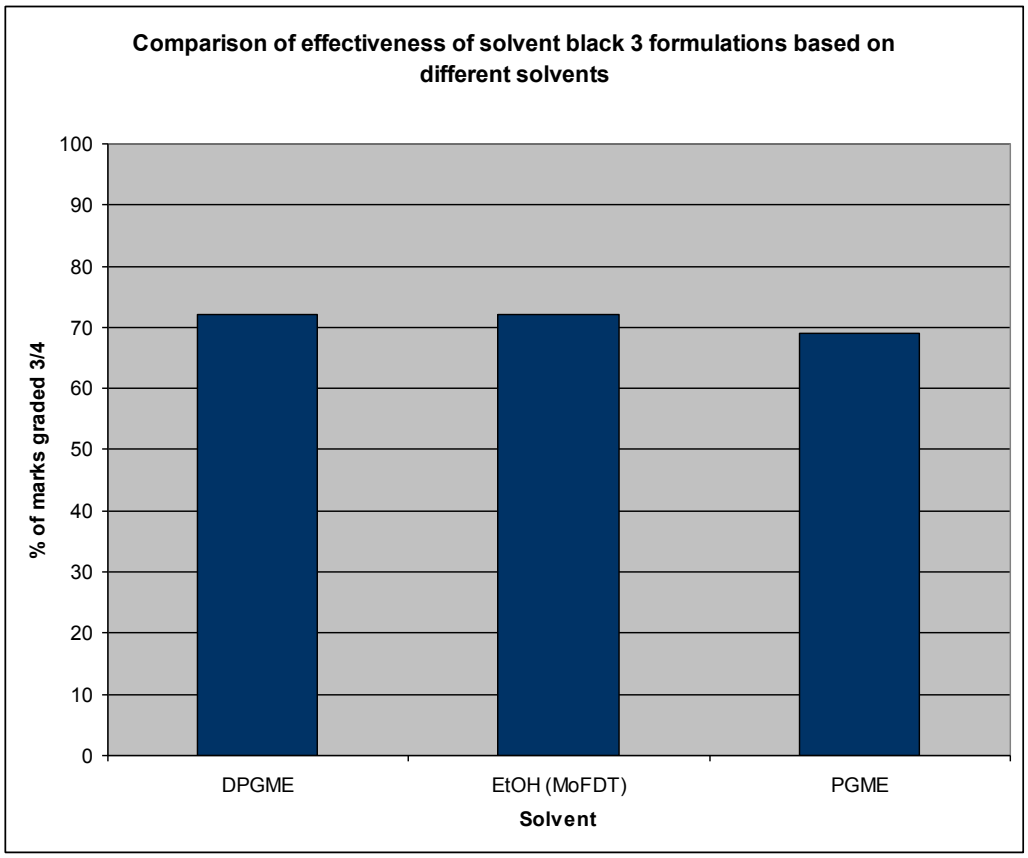


Results of comparative tests between Solvent Black 3 and iodine solution.

8.1.4 In general, the results show that solvent black 3 is a more effective treatment than iodine for latent fingerprints. However, an in-depth analysis of the results across all the surfaces examined (which included various laminates, uPVC, ceramic tile and gloss painted wood) showed that there were certain surfaces (e.g. gloss painted wood) where iodine did out-perform solvent black 3. However, the overall better performance of solvent black 3 combined with the flammability issues of iodine, meant that solvent black 3 continued to be the technique recommended for operational use.

8.1.5 Prior to the publication of the current reduced flammability solvent black 3 formulation in 2005 [8], a three-way trial was carried out comparing PGME- and DPGME-based formulations with the ethanol-based formulation recommended in the *Manual of Fingerprint Development Techniques* [6].

8.1.6 During the course of this three-way trial, 5,040 half prints were graded. The results of this study are illustrated below.



Results of three-way comparison between solvent black 3 formulations based on different solvents

8.1.7 The results demonstrate closely equivalent performance between all three formulations, and it was considered that they could be used interchangeably according to circumstances.

8.1.8 It should be noted that all trials outlined above utilise latent fingerprints. These results are therefore not truly representative of the operational use because solvent black 3 is recommended for use on greasy, contaminated surfaces and fingerprints. However, there are difficulties in producing a model 'contaminant' for such studies in the same way that horse blood is used as a contaminant for studies into blood dyes, and further research is required in this area.

8.2 Pseudo-operational trials and operational experience

8.2.1 Initial operational trials were carried out in 1986 to determine the relative effectiveness of the technique in developing fingerprints on polythene bags. In these trials solvent black 3 was compared with VMD and SPR [5]. The results of this comparison are reproduced below.

	Characteristics				Number of marks	
	>16		8–16*			
	SB3	VMD	SB3	VMD	SB3	VMD
Number of cases	11	18	13	6	24	24
Number of fingerprints	56	81	80	102	–	–

** Number of prints of 8–16 characteristics recorded only when no prints of >16 characteristics were revealed.*

	Characteristics				Number of marks	
	>16		8–16*			
	SB3	SPR	SB3	SPR	SB3	SPR
Number of cases	4	10	5	10	39	28
Number of fingerprints	8	24	21	72	–	–

** Number of prints of 8–16 characteristics recorded only when no prints of >16 characteristics were revealed.*

Results of comparative trials between solvent black 3, small particle reagent and vacuum metal deposition.

8.2.2 These trials indicated that solvent black 3 was not as effective as either VMD or SPR for developing fingerprints on polythene bags and it was not subsequently recommended for this application. However, the potential of the technique to develop marks on greasy, contaminated surfaces was later recognised and the technique was developed for this purpose.

8.2.3 A full operational trial has not been conducted on the use of solvent black 3 on contaminated surfaces, nor has a side-by-side comparison been conducted between ethanol and PGME-based solutions. This is because there are so few cases where the use of solvent black 3 is necessary and to build up statistically meaningful operational data would take several years. Because nature of the contaminant is known, unlike 'real' fingerprints that are variable in composition, the performance in operational use will be the same as that in laboratory tests. In the case of the solvent black 3 formulation, the decision was taken to issue the less flammable formulation because this provided a scene of crime capability where none was previously available. Laboratory results suggest the two formulations are very similar in performance and there is no reason to assume that this would significantly change when applied at a scene.

9. References

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