Comparison of three active 1,2-indanedione-zinc formulations on substrates commonly encountered and in the context of Seychelles limited resources and supply chain risks

Comparison of three active 1,2-indanedione-zinc formulations in the context of Seychelles limited resources and supply chain risks

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# Abstract

1,2-indanedione-zinc is recognised as one of the most single effective treatments for the detection of latent fingermark on paper surfaces and proliferation in smaller jurisdictions such as Seychelles is expected. However, within the fingermarks world detection techniques have been primarily developed in large jurisdictions with sizeable resources with a focus on performance. Whilst performance is important, in the context of small jurisdiction such as Seychelles, the challenges of limited resources and supply chain must also be considered. In this study the performance of three active IND-Zn formulations is investigated on substrates commonly encountered by the Seychelles Police, to explore whether the composition of the formulation can be modified to manage the inherent financial and supply chain risks. The results indicates that although the composition impact on the effectiveness of the formulation, overall, all three formulations were sensitive with similar performance characteristics providing the possibility to modify the formulation in consideration of supply chain issues, cost and safety. IND-Zn formulation containing petroleum ether 40-60bp, a multisource and cheaper carrier solvent was found to have comparable performance to formulations containing HFE-7100, a high-risk carrier solvent due to cost coupled with supply disruption associated with possible stringent environmental regulations and single-source solvent. The findings provide the basis for developing an IND-Zn formulation for the Seychelles police that may be applicable for other micro-jurisdictions.

**Keywords**: Latent fingermarks; paper surfaces; 1,2-indandione-zinc chloride; amino acids

# Introduction

Over the past two decades there has been significant research worldwide directed at improving 1,2-indanedione (IND-Zn) formulation as an operational latent fingermark detection technique on porous surfaces. 1,2-indanedione which reacts with amino acid to form a coloured and a luminescent product when viewed under green (500–550 nm) light and orange filter, was first proposed as an amino acid-sensitive fingermark detection technique in 1997 by Ramotowski et al. at the University of Pennsylvania [1]. Subsequently, studies conducted by separate research groups in various parts of the world including Australia [2-5], Israel [6, 7], USA [8, 9], and the UK [10-14] has led to its widespread use. Currently, IND-Zn is recognised as one of the most single effective treatment for the detection of latent fingermark on paper surfaces and proliferation in smaller jurisdictions such as Seychelles is anticipated [15].

Various IND-Zn formulations exist, that can be tested and validated by jurisdictions yet to introduce this technique. However, the IND-Zn formulation have been primarily developed in large jurisdictions with sizeable resources and the focus have been predominantly on performance. In the context of Seychelles, whilst performance is important, the inherent challenges of limited resources and supply chain issues [15] must also be considered. Techniques which is not only effective but economical are more likely to be implemented by the Seychelles Police in order to manage the scarcity of resources. Additionally, the Seychelles Police have to consistently manage chemical supply issues as a consequence of sitting at the end of the supply chain due to the geographical remote location of Seychelles. This have led to initiatives of large stock keeping, collaboration with other governmental laboratories and alternative grade or chemicals in order to maintain service continuity [15]. Therefore, introduction of new techniques in small jurisdictions such as Seychelles, should where possible and without compromising quality, be modified to be economical, safe and resilient to supply chain issues, to ensure a sustainable forensic provision - a concept coined as “frugal forensic” [16].

As a starting point three IND-Zn formulations in operational use by police forces in different parts of the world were identified; the Australian Federal Police (AFP) [17], the Bundeskriminalamt in Germany (BKA) [18] and the UK (CAST) [19]. In a recent comparative study [20] of the three formulations to develop fingermarks on old documents, we demonstrated that they are all effective and their performance appears to have similar characteristics. Hence, the key and the challenge for an IND-Zn formulation for the Seychelles Police lies in the main chemical components making up the composition of the formulations. A review of the composition of the working solution of the three formulation is shown in Figs.1. The carrier solvent forms the bulk of the working solution (86.7% to 90.0 % v/v) and consequently a major cost factor. The recommended formulation in the UK (CAST) and in Australia (AFP) uses HFE-7100, a carrier solvent that is provided by one supplier and may subject to future ban consideration within the EU [21]. The recommended formulation in Germany (BKA) uses Petroleum ether 40-60 bp, a carrier solvent readily available from multiple suppliers and at least three times cheaper than HFE-7100. Petroleum ether suffers from the drawback of being highly flammable, however this risk can be managed within a laboratory setting. Altogether, from a low-budgeted laboratory perspective, petroleum ether offers a better alternative in managing the financial and supply chain risks. Similar arguments in terms of cost and availability of 1,2-indanedione can be raised. However, 1,2-indanedione is present in minute amount in the working solution, ranging from 0.23 (CAST) to 0.99 (BKA) milligrams per litres (Figs. 2), resulting in a meagre cost factor compared to the carrier solvent. With regards to supply, a small jurisdiction can buy multiple years of 1,2-indanedione stock, whilst the same cannot be said for the carrier solvent. In summary, the cost and supply of 1,2-indanedione and zinc chloride, are unlikely to be of concerns but ensuring an effective concentration and ratio [22] would be the focus.

The last component are the polar solvents comprising ethyl acetate, acetic acid and alcohol in various amount, Figs.1. An interesting difference in the polar solvents, is the amount and use of methanol (4.1% v/v) in the CAST formulation compared to the use of ethanol at lower concentration in the BKA (1.0% v/v) and AFP (0.4%) formulations. In the early stage of IND-Zn formulation development, prior to the addition of zinc chloride, the use of alcohols as a solvent was considered to be detrimental to the formation of Joullie’s Pink due to hemiketal formation, which is less reactive to amino acid [23]. In contrast, recent studies [12] reported increase fluorescent intensity with formulation containing methanol in the presence of zinc chloride. Whilst the cost and supply of the polar solvents between the different formulations is trivial, the reported benefits of increased luminescent intensity with methanol and the use of ethanol as a safer option may be considered.

Fig. 1. Percentage by volume of carrier solvents and polar solvents in AFP, BKA and CAST IND-Zn working solution.

Fig. 2. Amount of 1,2-inadnedione and zinc chloride in milligrams per litres in AFP, BKA and CAST IND-Zn working solution.

Finally, although our previous study [20] indicated that the composition of the formulation did not seem to have a major effect on performance with paper surfaces, but it could with other substrates. This creates a need for broader studies using a range of porous substrates, particularly those likely to be encountered in casework for whichever jurisdiction is developing the formulation. In this study cardboard surfaces a commonly encountered surfaces by the Seychelles Police were also investigated.

This study investigates the effectiveness of three operational IND-Zn formulations on a variety of substrates commonly encountered by the Seychelles Police. To explore whether the composition of the formulation can be modified to develop an effective, safe and economical formulation resilient to the challenges of micro-jurisdictions such as Seychelles.

# Materials and methods

## Chemicals

1,2-Indanedione (Reddy Chemtech), Ethyl Acetate (≥99.7%, UNIVAR - APS), Acetic Acid (≥99.7%, Lab-Scan), Methanol (≥99.7%, Honeywell – Burdick & Jackson), Ethanol (≥99.5%, UNIVAR - Ajax Finechem), Zinc Chloride (≥98% Sigma Aldrich), HFE-7100 (3M Novec), Petroleum Ether (40-60ºC, VWR Chemicals). Reagents were used as received without further purification.

## Substrates

Five substrate that are commonly encountered by the Seychelles police were chosen and summarised in Table 1. This consist of two types of cardboards; Chipboard single walled general-purpose cardboard and fibreboard a heavy-duty cardboard. All substrates were sourced from Officeworks. Prior to fingermark collection a collection grid was printed on the substrates.

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| **Table 1** |  | | |  | |  | |
| Substrate types used in this study | | | |  | |  | |
| Substrate type | | Description | Colour | | Weight (gsm)/Thickness | |
| 1 | | Winc Copy paper A4, Carbon neutral | White | | 80 | |
| 2 | | PPS Envelope A4, 100% Recycled | White | | 80 | |
| 3 | | Notepad A4, 8mm ruled | Blue | | 70 | |
| 4 | | PPS Cardboard Box, Chipboard | Brown | | 3.5 mm | |
| 5 | | PPS Heavy duty Cardboard Box, Fireboard | Brown | | 6.5 mm | |

## Fingermark collection and storage

Natural, split depletion fingemarks were collected from five donors (3 males and 2 females). Donors were asked not to wear gloves or wash their hands 30 mins prior to deposition and to rub their hands together for homogenous deposition. All formulation were compared directly with each other and alternating the position of each head-to-head comparison (AFP|CAST, BKA|AFP, CAST|BKA) to reduce deposition pressure variability. To access the sensitivity of the techniques 1st, 5th and 10th depletion were collected on each substrate for each set of head-to-head comparison. The other depositions were collected on a separate piece of the same type of substrate. Samples were stored in a cupboard under office condition (~25 °C) for at least 3 days and cut into halves prior to IND-Zn treatment.

## Fingermark development and grading

The IND-Zn working solutions were prepared as per the following protocols; AFP as outlined by Stoilovic et. al 2012 [17], BKA by Becker et al 2018 [18], and CAST by DSTL Fingermark Visualisation Newsletter 2019 [19], summarised in Table 2. All IND-Zn treated samples were processed in an ironing press at 160˚C for 10 s. Processed samples were re-combined, visualised and photographed in luminescent mode using Polilight PL500 (Rofin Australia Pty. Ltd., Australia) 505 nm light source and a Schott OG550 nm filter. Processed fingermark halves were graded according to the quality of the marks using the UK Home Office Centre for Applied Science and Technology (CAST) assessment scale [24] and the grades categories into 3 groups [25] for purposes of evaluating the trends, Table 3. Only fingermarks with a grade of 3 or 4 are considered to be suitable for comparison purposes.

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| **Table 2** |  | |  | |  | |  |
| IND-Zn formulations used in this study [17-19]. | | | |  |  | |  |
| Formulation # | | IND-Zn formulation | Country in-use | | | 1,2-indanedione formulation | Zinc stock composition |
| 1 | | Australian Federal Police (AFP) | Australia | | | *Indanedione stock solution:*  2.3 g 1,2-indanedione 480 mL ethyl acetate 20 mL acetic acid *IND-Zn working solution:* 130 mL indanedione stock solution 870 mL HFE-7100 4 mL zinc chloride stock solution | 8 g zinc chloride 200 mL ethanol |
| 2 | | Bundeskriminalamt (BKA) | Germany | | | 0.25 g 1,2-indanedione 22.5 mL ethyl acetate 2.5 mL acetic acid 225 mL petroleum ether bp 40-60 2.5 mL zinc chloride stock solution | 0.2 g zinc chloride 30 mL ethanol |
| 3 | | CAST (Presently Dstl) | UK | | | 0.25 g 1,2-indanedione 45 mL ethyl acetate 45 mL methanol 20 mL acetic acid 1 mL zinc chloride stock solution 1 L HFE-7100 | 0.1 g zinc chloride 4 mL ethyl acetate 1 mL acetic acid |

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| --- | --- | --- | --- | --- | --- |
| **Table 3** | |  | |  | |
| Fingermark grading scale and grading classification used in this study [24, 25]. | | | |  | |
| Grade | Friction ridge detail developed | | Classification | |
| 0 | No evidence of mark | | No fingermarks detected | |
| 1 | Weak development; evidence of contact but no ridge detail | | Detected, but not suitable for comparison | |
| 2 | Limited development, ridge details present; but probably cannot be used for identification purposes | | Detected, but not suitable for comparison | |
| 3 | Strong development more than 2/3 of fingermark continuous ridges | | Suitable for comparison | |
| 4 | Very strong development: full ridge details | | Suitable for comparison | |

## Supplementary experiments

Further experiments were carried out using a modified AFP formulation and a modified CAST formulation outlined in Table 4. AFP composition which contains the least amount of alcohol in formulation was found to consistently produce relatively lower fluorescent intensity. To investigate the role of alcohol in the formulation, a modified AFP formulation containing similar percentage by volume of alcohol in the working solution as the CAST formulation was compared with the original AFP formulation. The AFP formulation was modified by increasing the amount of ethanol to 4.0% v/v and a 1:1 volumetric reduction in the amount of ethyl acetate in order to keep the volume of the other components consistent. Another difference between the formulations is the type of alcohol use in the formulation. The CAST use methanol whilst AFP and BKA formulation use ethanol which safer than the former. To investigate the performance between the use of methanol or ethanol in the formulation, a modified CAST formulation by substituting methanol with ethanol in a 1:1 volumetric ratio, was compared with the original CAST formulation. Both experiments were performed on two substrates (Winc Copy paper A4 and notepad paper) and same methodology as the previous experiment was used.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Table 4** |  | |  |  |
| Modified AFP and CAST IND-Zn formulations used in this study. | | | |  |
| Formulation # | | IND-Zn formulation | 1,2-indanedione formulation | Zinc stock composition |
| 3 | | Modified AFP | *Indanedione stock solution:*  2.3 g 1,2-indanedione 340 mL ethyl acetate 20 mL acetic acid 140 mL ethanol *IND-Zn working solution:* 130 mL indanedione stock solution 870 mL HFE-7100 4 mL zinc chloride stock solution | 8 g zinc chloride 200 mL ethanol |
| 4 | | Modified CAST | 0.25 g 1,2-indanedione 45 mL ethyl acetate 45 mL ethanol 20 mL acetic acid 1 mL zinc chloride stock solution 1 L HFE-7100 | 0.1 g zinc chloride 4 mL ethyl acetate 1 mL acetic acid |

# Results and Discussion

## Overall performance

The relative performance of each IND-Zn formulations (AFP, BKA and CAST) was assessed by head-to-head comparison across five substrates, five donors with 3 depletion series. This generated a total of 450 fingermark halves, each graded and grouped according to Table 3. On average 47.3 % of the total marks developed with the three formulations were suitable for comparison (3-4 scores) with highest number 48.0% developed with the AFP formulation followed by 47.3% BKA and 46.7% CAST, Fig. 3. Interestingly 6.7% of the marks treated with AFP formulation were not detected (0 score) compared to 5.3% CAST and 3.3% BKA. Whilst 45.3 %, 48.0% and 49.3% of marks detected with AFP, CAST and BKA respectively were not suitable for comparison. The result is consistent with previous study [20] which shows that the three formulations were of comparable performance in spite of the differences in the formulation composition. Whilst this conclusion is made in the context of this study, other studies [22, 26] have also reported better reproducibility since the addition of zinc chloride to the formulation in contrast to earlier formulation. The result opens the possibility to modify the formulation in consideration of cost, supply chain issues faced by micro-jurisdictions.

Fig. 3. Summary of results per formulation for all donors and substrates categories into no marks detected (0), detected but not suitable for comparison (1-2) and suitable for comparison (3-4).

## Sensitivity

Depletion series 1, 5 and 10 were compared to assess the effectiveness of each formulation. Fig. 4 shows the results of each depletion series per formulation. As expected, there is a gradual decrease in the number of fingermarks detected down the depletion series. All first depletion marks were detected, out of which 82.0% of the marks treated with AFP formulation, 76.0% CAST and 78.0 % BKA were classified as suitable for comparison. The remaining detected marks were classified as not suitable for comparison. For the 10th depletion, the number of marks suitable for comparison decrease to 36.4% AFP, 37.7% BKA and 28.3% CAST. This represents a decrease from an average of 78.7% for the 1st depletion to 48.7% 5th depletion and 34.1% 10th depletion. The least number of marks not detected for the 10th depletion were 7.2% BKA followed by 11.7% CAST and 12.1% AFP. The result demonstrates that the three IND-Zn formulations to be an effective treatment for the detection of latent fingermark on the paper and cardboard surfaces used in this study. The relatively consistent decrease across the depletion series further underlines the comparable performance of the three IND-ZN formulations.

Fig. 4. Comparison of the 1st 5th and 10th depletion series results for all three sets of head-to-head comparison for all donors and substrates categories into no marks detected (0), detected but not suitable for comparison (1-2) and suitable for comparison (3-4).

Carrier solvents

The BKA formulation which contains petroleum ether bp 40-60, shows comparable overall performance with the least number of marks not detected when compared to AFP and CAST formulation which uses HFE-7100. Additionally, when compared to other formulations the BKA showed high sensitivity with the highest number of suitable fingermark (37.7%) detected and the least number of marks not detected (7.2%) for the 10th depletion. Previous spectroscopic studies [12] have reported that the carrier solvent appears to influence the properties of the formulation. In this study the result suggests that the difference in the impact of the two carrier solvents on the formulation properties, have minimal effect on the quality of fingermarks developed under the conditions of this study.

As discussed previously, the difference in the carrier solvents between the formulations present the main challenge in terms of cost and supply chain issues for developing an IND-Zn formulation for the Seychelles Police. It is to be noted that the chemical supply issue has recently been reported by larger jurisdictions, such as inconsistency of supply due to COVID-19 and potential limited availability of chemical due to stringent environmental regulations [19, 27]. This includes the availability of HFE-7100 which is currently being monitored closely by DSTL (Defence Science and Technology Laboratory) in the UK [27], due to potential review under the EU Regulation, which may limit its supply. Another potential risk to the HFE-7100 supply, seldom discussed in open literature, is that it is a single source solvent (proprietary fluid of 3M™ Novec™) [28] compared to petroleum spirit which is multisource. Therefore, low budgeted laboratory where cost and supply chain are key factors in deciding the formulation of IND-Zn, petroleum ether may provide an alternative carrier solvent to HFE-7100 in managing these risks. However, this may also be of interest to other jurisdictions in finding an alternative to HFE-7100 potential supply disruption.

## Substrates

Overall, all formulations were efficient in developing fingermarks on all 5 substrates but the performance on cardboard was lower compared to other substrates, Fig. 5. The BKA formulation was found to be more effective on cardboard compared to the other formulations. Both the chipboard and fireboard used in this study are made from recycled paper which have lower content of cellulose and short fibres compared to normal paper. Fireboard which is corrugated to provide strength composed of three layers of paper; a fluting layer in the middle composed of low quality recycled paper, enclosed by two liners made of highly compact paper which has been recycled a limited number of times [29]. The lower cellulose content compared to normal paper may explain the lower performance as it has been reported [22] that the cellulose matrix in paper plays a number of important roles in the reaction between Ind-Zn reagent and amino acid in latent fingermark. Another consideration is the porosity or thickness of cardboard impacting on the absorption of the fingermark residue and the working solution into the matrix of the cardboard [30]. The concentration of 1,2-indanedione is more than 4 times higher in the BKA than the CAST formulation, Fig. 1. This high concentration in the BKA formulation may increase the availability of 1,2-indanedione in the cardboard matrix to react with amino acid in the latent fingermark residue and may explain its superiority on cardboard surfaces. The result indicates the need for further investigation in the optimal concentration of 1,2-indanedione for the detection of fingermarks on cardboard surfaces.

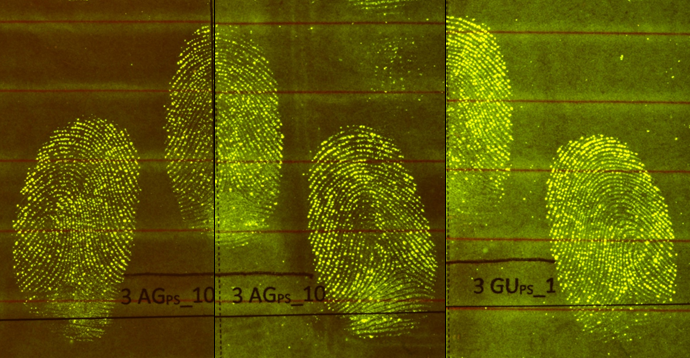
The greatest number of marks suitable for comparison (66.7%) were detected on notepad paper and the least number (30.0%) on chipboard, both with AFP formulation treatment. This may likely be that although the AFP formulation is relatively less sensitive on cardboards, it develops good quality fingermarks on substrate which is prone to background development and ink diffusion. This was evident on the coloured notepad with printed red redlines where the maximum number of suitable fingermarks were detected with the AFP formulation, whilst a yellowish background observed with the CAST formulation and BKA formulations (Fig. 6), impacted on the quality of the fingermarks. This was less apparent on clean surfaces such as copy paper where all fingermarks were detected by each formulation. This further underlines the need to balance the polar solvent composition between sensitivity and quality, and could also suggest that the effectiveness of the formulation composition to be substrate specific.

Fig. 5. Summary of results for all treated fingermarks per substrates for all formulation and donors categories into no marks detected (0), detected but not suitable for comparison (1-2) and suitable for comparison (3-4).

## Polar solvents

Examination of the marks shows higher fluorescent intensity of fingermarks developed with the CAST and BKA formulations compared to AFP formulation. In contrast the AFP produced higher quality fingermarks with clean background whilst bright fluorescent patches and marginal diffusion of ridge details were observed with CAST and BKA treatment, Fig. 6. The highest fluorescent intensity and marginal diffusion of ridge detail were more prominent in the UK formulation which is likely due to the high percentage of polar solvents in the CAST formulation. Other than ethyl acetate the CAST formulation contains more polar solvents, methanol (4.1% v/v) and acetic acid (1.8% v/v) compared to the Australian formulation containing the least amount ethanol (0.4% v/v) and acetic acid (0.5% v/v). The result is consistent with the UK study [12] were the combination of methanol and zinc chloride was found to produce higher fluorescent intensity, whilst excess methanol can caused diffusion of ridge detail. Additionally, the addition of acetic acid have been reported to cause diffused fingermark [7].

To investigate the role of alcohol in the formulation, a modified AFP formulation containing same percentage by volume of alcohol as in the CAST formulation was compared with the original AFP formulation. Two substrates which shows the least (copy paper) and the most (notepad paper) background development were used. Similar observation of higher fluorescent intensity including brighter background and marginal diffusion seen in the UK formulation was observed with the modified AFP formulation when compared with the original AFP formulation, Fig. 7. However, these differences did not have significant impact on the number and quality of fingermarks developed. It was also observed that on thin paper (notepad) the high fluorescent fingermarks developed with CAST, BKA and modified CAST were apparent from the reverse side. Whilst this shows good sensitivity, this may hinder examination of marks on the reverse side. Altogether, the current results reinforce the findings [12] of the benefit of alcohol in combination with zinc chloride towards luminescent intensity and reiterate the need to strike a balance between sensitivity and quality of the developed fingermarks.



c)

b)

a)

Fig. 6. Fingermarks treated with a) AFP formulation, b) BKA, c) CAST on notepad paper showing the fluorescent intensity and background development.

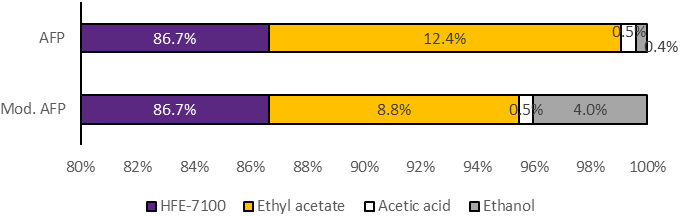


Fig. 7. Fingermarks treated with AFP formulation left and modified AFP formulation right, on copy paper showing the composition and fluorescent intensity and background development.

Another difference between the formulation compositions is the type of alcohol used in the formulation. The CAST formulation uses methanol whilst AFP and BKA formulation uses ethanol which is safer than the former. To investigate the performance between the use of methanol or ethanol in the formulation, a modified CAST formulation by directly substituting methanol with ethanol was compared with the original CAST formulation on two substrates copy paper and notepad paper. Assessment of the developed marks indicates the use of methanol or ethanol in the formulation to be of comparable performance with marginal difference observed with the original CAST formulation developing producing higher fluorescent intensity fingermarks, Fig.8. However due to the limited sample size of this study further investigation are required to assess the performance between methanol and ethanol and the role of alcohol in combination with zinc chloride in formulation.

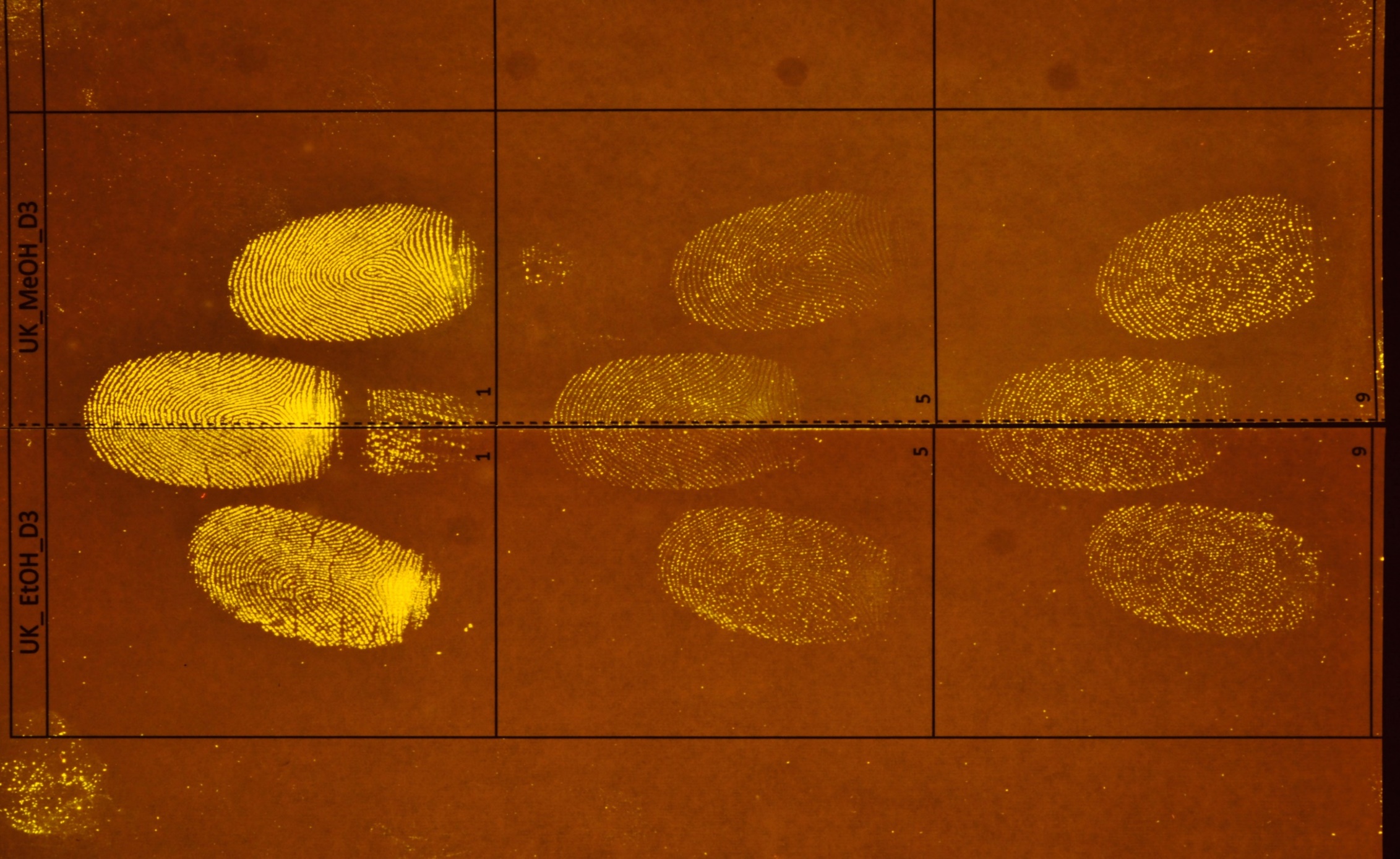


Fig. 8. Fingermarks treated with modified CAST formulation (ethanol) left and CAST formulation (methanol) right, on copy paper showing the composition and fluorescent intensity and background development.

# Conclusion

In this study the performance of three active IND-Zn formulations is investigated on substrate commonly encountered by the Seychelles Police. To explore whether the composition of the formulation can be modified to develop an effective and resilient to the challenges of a micro-jurisdictions such as Seychelles.

The evaluation of the formulation indicated that although the composition has an impact on the effectiveness of the formulation, overall, all three formulations were highly sensitive and of comparable performance. The high reproducibility in contrast to earlier formulation provide greater ability to modify the formulation in consideration of cost, supply chain issues faced by micro-jurisdictions.

The difference in the carrier solvents between the formulations present the main challenge in terms of cost and supply chain issues for developing an IND-Zn formulation for the Seychelles Police. In this study two carrier solvent; HFE-7100 and petroleum ether bp 40-60 was used. The result suggests that the two carrier solvents have similar performance characteristics. For low budgeted laboratory where cost and supply chain are key factors in deciding the formulation composition. Petroleum ether which is cheaper and multisource provides a better alternative in managing the associated risks compared to HFE-7100 which have higher risk of supply disruption due to possible stringent environmental regulations and being a single-source solvent.

The differential results between the different surfaces particularly cardboard suggest that the effectiveness of the formulation composition to be substrate specific. This indicates the need for further investigation in the optimal concentration of 1,2-indanedione for the detection of fingermarks on cardboard and other surfaces.

The use of ethanol or methanol in the IND-Zn formulation was found to be of comparable performance, although marginally higher fluorescent intensity was observed with methanol-containing formulation. The result reinforces the benefit of alcohol in combination with zinc chloride towards luminescent intensity and reiterate the need to strike a balance between sensitivity and quality. However due to the limited sample size of this study further investigation are required to assess the performance between methanol and ethanol and the role of alcohol in combination with zinc chloride in formulation.

In summary, the above findings provide the basis for developing an IND-Zn formulation for the Seychelles police that may be applicable for other micro-jurisdictions.

# CRediT authorship contribution statement:

T.B.A

# Declarations of interest:

T.B.A

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