3M™ Novec™
Engineered Fluid
HFE-7100

A new substitute carrier solvent for ninhydrin and DFO formulations

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3M™ Novec™ Engineered Fluid HFE-7100: A new substitute carrier solvent for Ninhydrin and DFO formulations

Abstract: *In 1974, CFC-113 became widely used as a carrier solvent for ninhydrin in the development of latent fingerprints. The 1987 Montreal Protocol mandated a phaseout of CFCs. Using their more than forty years of experience in the manufacturing of fluorinated materials, scientists at 3M set out to develop a new class of materials for applications where CFCs were previously used. This class of materials is now known as 3M™ Novec™ Engineered Fluids, the first of which is named 3M™ Novec™ Engineered Fluid HFE-7100. Evaluations performed by forensic labs in the UK and France have found Novec fluid HFE-7100 to be the optimum product for use with both ninhydrin and DFO formulations. It is low in toxicity, nonflammable, has demonstrated its compatibility with a wide range of inks and substrates, and, with the proper formulation, has been shown to develop a higher percentage of fingerprints in more cases than the alternative HFC formulations or the now restricted CFC-113 formulations. Recently, the Novec fluid HFE-7100-based formulation was found to develop fingerprints on U.S. currency, which was previously difficult at best using flammable carrier solvents. Additional work is in process to look at alternative uses for this product in Rhodamine and Luminol formulations as well as potential non-darkening formulations for obtaining fingerprints from thermal paper.*

Phaseout of CFC-113 creates need for new solvent

Latent fingerprint technology was greatly advanced in 1974 when CFC-113, a chlorofluorocarbon, was first used with ninhydrin to develop latent fingerprints on porous evidence after the process was recommended by Morris and Goode [1]. CFC-113 is nonflammable, low in toxicity, volatile and relatively non-polar. Because it is relatively non-polar, it did not cause significant running or bleeding of inks. Its volatility meant that processing was fast and clean. Its nonflammability and low toxicity meant that it could be used at the crime scene when necessary and in the lab without posing a significant safety risk. Because of these attributes, CFC-113 became the solvent of choice for forensic scientists around the world with ninhydrin, and later with DFO.

That changed in 1987, however, when the Montreal Protocol identified chlorofluorocarbons as ozone depleting substances. Subsequent amendments to the Montreal Protocol imposed restrictions on further manufacturing of CFCs until a production ban took effect in member nations effective January 1, 1996. While the use of existing supplies was possible—and some stockpiling did occur—many nations imposed taxes and restrictions on continued use of CFC-113 .

Searching for the replacement

In the wake of these restrictions, forensic scientists turned to a number of replacement solvents—none of which were as effective as CFC-113. The replacement carrier solvent formulation needed to provide good ridge detail with little or no ridge diffusion, be compatible with a wide range of inks and substrates, be nonflammable, low in toxicity and possess a favorable environmental profile.

At the beginning of the search for a replacement solvent, there was not a high degree of concern because many researchers assumed that the efficiency with which amino acid reagents like ninhydrin and DFO develop latent fingerprints on porous evidence was largely indifferent to its carrier solvent. At the time, the widespread belief was that the major factors affecting latent print quality were development temperatures and humidity levels. As the initial trials of replacement solvents showed, both the quality and quantity of latent prints developed were strongly dependent on the physical properties of the carrier solvent [2]. Consequently, the focus on the search for a replacement solvent shifted from a dependence on the fingerprint development parameters to more of a dependence on the chemistry of the developing formulation.

In 1996, during the initial evaluations at the Police Scientific Development Branch–United Kingdom, the available replacement solvent options were separated into two groups: nonflammable fluorinated solvents and flammable organic solvents. Initially, hydrochlorofluorocarbons (HCFCs) were thought to be excellent CFC replacements, but HCFCs are also ozone depleting, subjecting them to restrictions and legislation in Europe and the United States [2]. Additionally, because they are inherently more polar than CFC-113, HCFCs were found to cause running of some inks, and consequently destruction of some handwriting evidence. Additionally, HCFC-based formulations were found to develop fingerprints at a rather slow rate—sometimes taking as long as two weeks to fully develop prints [2].

Perfluorocarbons (PFCs)—although non-ozone-depleting—were considered but abandoned because of their immiscibility [2] with the polar solvents used to dissolve the ninhydrin.

Hydrofluoroethers (HFEs) and hydrofluorocarbons (HFCs) were not commercially available at the time of these initial trials.

Of the flammable organic solvents, *heptane* showed promising results in comparison to CFC-113 in a formulation developed by Watling and Smith and optimized by Hewlett and Sears [2]. However, with a flash point of -4°C (25°F), heptane is flammable making its use in a non-explosion-proof laboratory or an uncontrolled crime scene a potentially unsafe practice. In addition, depending on the heptane formulation used, it was found to cause running and smearing of some inks as well as inconsistent ridge detail in comparison to CFC-113-based formulations.

Acetone was also evaluated and was found to work well with the ninhydrin formulations. However, its drawbacks are that it is also flammable—with a flash point of -20°C (-4°F)—and thus should not be used without proper precautions with attention to possible ignition sources. Additionally, because it is a good all-purpose solvent, acetone has been shown to cause running and bleeding of almost all inks. Depending on the substrate, it can cause everything from slight running to total destruction of handwritten or printed evidence.

Petroleum ether, which is currently in widespread use, was found to be a better replacement solvent than either heptane or acetone. It causes minimal bleeding or running of inks, and is relatively inexpensive. But it is also flammable with a flash point of -18°C (0°F). Therefore, although petroleum ether-based formulations are effective, caution with respect to possible ignition sources must be taken when using them.

These initial replacement solvent evaluations clearly did not reveal a candidate that could offer the same balance of properties as CFC-113—namely performance, nonflammability, and safety in use. It was the right time for a carrier solvent designed with the right balance of properties.

The development of 3M™ Novec™ Engineered Fluids

Recognizing that CFCs and HCFCs would soon be obsolete after the Montreal Protocol of 1987, 3M scientists set out to develop a new class of materials—a technology platform—that would balance performance requirements of CFC and HCFC solvents with environmental, health and safety requirements. They were aware of the target properties of the fluid they were attempting to create, namely: zero ozone depletion potential, low global warming potential (GWP), non-VOC, low toxicity and nonflammable—basically a class of fluids that could replace chlorofluorocarbons in a variety of applications and industries. In developing a product with these targeted characteristics, they used a variety of

Comparison Guide of 3M™ Novec™ Engineered Fluid HFE-7100

Property	HFE-7100	HFC-43-10mee	CFC-113	Petroleum Ether (35-60°C Cut)	Acetone	Heptane
Flash Point (°C)	None	None	None	-18	-20	-4
Flammability in Air (Vol. %)	None	None	None	1.1	2	1.1
VOC	No	No	No	Yes	No	Yes
Exposure, 8 hr. avg. (ppm)	750	200	1000	400	500	400
Exposure Ceiling—STEL (ppm)	No	400	1250	No	750	500
Vapor Pressure (kPa)	28	30	44	5	24	6
Surface Tension (mN/m)	13.6	14.1	17.3	16.1	26.2	20.3
Density (g/cc)	1.52	1.58	1.56	0.74	0.79	0.68
Boiling Point (°C)	60	54	48	35-60	57	98

Sources: Novec fluid HFE-7100—AIHA; HFE-43-10mee—Manufacturer; CFC-113, acetone, heptane—1998 ACGIH; Petroleum ether—Sigma Aldrich MSDS

Table 1

product development techniques. One method they used is called "computational chemistry." Computational chemistry makes it possible to estimate desired properties, such as boiling point or atmospheric lifetime, based on the exact arrangement of atoms in a molecule.

The researchers modeled, synthesized and tested literally hundreds of potential materials before finally hitting upon the optimal answer: segregated hydrofluoroethers (HFEs). This new class of materials was introduced in 1996, the same year that CFC production was halted in developed nations. The products that grew from this HFE technology platform eventually gained the brand name 3M™ Novec™ Engineered Fluids.

Novec fluids are distinguished by a number of favorable environmental and performance properties:

1) Zero ozone depletion potential (ODP): No Novec fluid will cause depletion of the Earth's ozone layer.

2) Low global warming potential (GWP): The global warming potentials of Novec fluids range between 55 and 320, compared with 6000 for CFC-113.

3) Nonflammable and low in toxicity: Novec fluids exhibit an optimized balance of performance, health, safety and environmental properties.

Environmental Properties

Property	HFE-7100	HFC-43-10mee	CFC-113
Ozone Depletion Potential (ODP)	0	0	0.8
Atmospheric Lifetime (years)	4.1	17.1	85
Global Warming Potential (GWP)	320	1700	6000

Table 2

3M™ Novec™ Engineered Fluid HFE-7100

In 1996, 3M introduced its first Novec fluid: 3M™ Novec™ Engineered Fluid HFE-7100. It was mainly targeted for use in precision electronics and industrial cleaning applications.

As early as 1995, however, 3M had sampled the new material to the Fingerprint Research Group, Police Scientific Development Branch, United Kingdom, as a candidate solvent for their CFC replacement evaluations. The PSDB–UK soon found that the fluid worked well in this application because: it is compatible with a wide range of materials, including porous substrates and inks; it dries quickly; and it is nonflammable and low in toxicity (Table 1). Additionally, it is non-ozone-depleting and has a low global warming potential (Table 2).

A ninhydrin carrier solvent

As a result of their favorable results obtained using the samples of Novec fluid HFE-7100, in 1997, the Fingerprint Research Group, Police Scientific Development Branch, United Kingdom, evaluated

Novec fluid HFE-7100		HFC-43-10mee		CFC-113	
formulation:		*formulation:*		*formulation:*	
Ninhydrin	5 g	Ninhydrin	5 g	Ninhydrin	5 g
Ethanol	45 ml	Ethanol	15 ml	Ethanol	20 ml
Ethyl acetate	2 ml	Ethyl acetate	5 ml	Acetic acid	10 ml
Acetic acid	5 ml	Acetic acid	10 ml	CFC-113	1 liter
HFE-7100	1 liter	HFC-43-10mee	1 liter		

Figure 1

both Novec fluid HFE-7100 and Vertrel XF®—a hydrofluorocarbon (HFC-43-10mee)—to determine if either could be used as a replacement for CFC-113 [3].

Their initial experiment was conducted by depositing split fingerprints on a variety of paper samples and evaluating the fingerprint quality versus the control (CFC-113) formulation. Both the 3M™ Novec™ Engineered Fluid HFE-7100 and HFC-43-10mee formulations produced fingerprints of similar quality to the CFC-113 formulation (Fig. 1) [3]. Additionally, the Novec fluid formulation was found to be stable for three months at temperatures between 0°C–35°C, while the HFC-43-10mee was stable for somewhat in excess of one month [3].

The second phase of their experiment employed a "pseudo-operational trial" in which they evaluated 75 checks taken from a range of cases [3]. They measured the number of latent fingerprints—containing eight or more Galton points—at 0, 3, 7 and 14 days after treatment to analyze the development efficiency of the two experimental formulations versus the control CFC-113 formulation [3]. Practically no handwriting diffusion was observed, and both experimental formulations were shown to develop a comparable number of good quality fingerprints to the CFC-113 formulation [3].

Although researchers were optimistic about the results obtained using both experimental CFC-113 replacement formulations, it was determined that large scale trials would be necessary in order to further validate the initial results obtained with both of the new experimental formulations.

In 1998, the Police Scientific Development Branch of the United Kingdom once again evaluated ninhydrin formulations using the two experimental carrier solvents under actual operating conditions [4]. Each week for two months the normal Essex County Police caseload was separated into three similar batches and treated using one of the three test formulations listed in Figure 1 [4].

Cumulative count of developed fingerprints

Week	Novec fluid HFE-7100		CFC-113		HFC-43-10mee	
	Cases	*Fingerprints*	*Cases*	*Fingerprints*	*Cases*	*Fingerprints*
1	15	89	14	50	12	70
2	32	146	29	140	29	102
3	42	204	39	156	42	139
4	54	243	54	196	55	220
5	66	309	69	238	68	263
6	83	384	80	242	79	347
7	102	427	102	280	102	393
8	111	468	110	331	110	430

Table 3

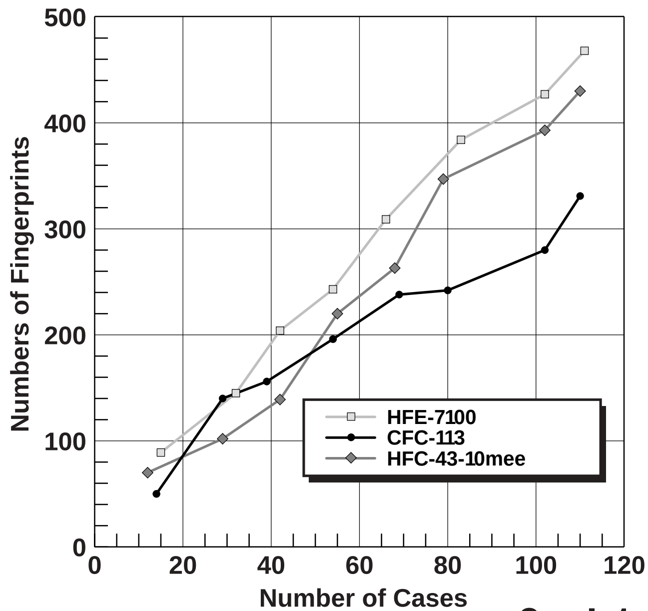
Articles were treated as described in the Manual of Fingerprint Development Techniques [5] whereby small flat items are drawn through a shallow trough filled with a solution and large, bulky items were painted with solution in a fume cupboard. Treated samples were dried in a fume cupboard before being heated and humidified in a Sanyo FDC® (Fingerprint Development Chamber) at 80°C and 62% relative humidity for 4 minutes [4]. The majority of the developed prints appeared during this four hour process, but enough prints developed slowly that the experimenters decided to measure and record prints with 8 or more Galton points twice: at two days after development and at two weeks [4].

Table 3 shows a week-by-week breakdown of the cumulative number of cases treated and the fingerprints developed by each formulation [4]:

As Graph 1 shows, their experiment was skewed by several sharp rises in the number of fingerprints in cases where unusually high numbers of fingerprints were developed—as was the case with the HFC-43-10mee formulation in which one-sixth of all the prints developed came from a single case [4].

To get a clearer picture of their performance, experimenters analyzed the formulations by the percentage of cases in which prints were developed (Table 4) [4]. Through 111 cases, the Novec fluid HFE-7100 formulations demonstrated good, consistent performance, developing prints in 67% of cases, better

Performance of Ninhydrin Formulations: Number of Fingerprints



Graph 1

than CFC-113's 60% and the 53% obtained with HFC-43-10mee. Additionally, ink running was minimal with the Novec fluid HFE-7100 formulation versus the CFC-113 solution because of the reduction in the acetic acid concentration [4].

The Police Scientific Development Branch concluded from this experiment that Novec fluid HFE-7100 is an effective alternative to the CFC-113 formulation [4]. Based on the results of this operational evaluation, the ninhydrin formulation using 3M™ Novec™

Percentage of cases with developed prints

Week	Novec fluid HFE-7100		CFC-113		HFC-43-10mee	
	Cases	*% with Fingerprints*	*Cases*	*% with Fingerprints*	*Cases*	*% with Fingerprints*
1	15	73	14	64	12	84
2	32	72	29	69	29	66
3	42	74	39	64	42	52
4	54	72	54	63	55	53
5	66	73	69	67	68	51
6	83	70	80	60	79	50
7	102	68	102	60	102	51
8	111	67	110	60	110	53

Table 4

Engineered Fluid HFE-7100 was introduced into Police Service use throughout the United Kingdom in 1998 [6].

A DFO carrier solvent

Encouraged by the success of Novec fluid HFE-7100 in the Police Scientific Development Branch trials, the Fingerprint Department, Institut de Recherche Criminelle de la Gendarmie Nationale, France, performed a preliminary study of the fluid as a replacement for CFC-113 in their DFO formulation [7]. Until that point, they had been using petroleum ether as a replacement solvent, but found that their petroleum ether formulation developed fingerprints less efficiently than the CFC-113 formulation, and noted its obvious inherent flammability drawback.

After some experimentation, the French team developed a formulation that was found to be very stable at room temperature several weeks after its preparation [7]:

DFO .... 0.25 g
Methanol .... 40 ml
Acetic acid .... 20 ml
Novec fluid HFE-7100 .... 940 ml

Their experiment consisted of testing split fingerprints on paper—one side tested with their standard CFC-113 formulation and the other using the Novec fluid HFE-7100 formulation. Treatment consisted of processing the items by soaking them for 5 seconds in the DFO solution, letting the solvent evaporate under a fume hood, redipping the item in the solution for another five seconds, allowing the solvent to evaporate, and drying the items for 10-20 minutes in an oven at a temperature ranging from 50°C to 100°C (depending upon the nature of the item) [7].

Side-by-side comparisons of fingerprints developed with the HFE-based formulation to those obtained using the CFC-113 formulation revealed the following:

1) Ridges of the fingerprints obtained with the CFC-113 formulation are yellow and very luminescent. The Novec fluid HFE-7100 prints are darker orange under fluorescent excitation and slightly less luminescent than those developed with CFC-113 [7].

2) Prints developed with the Novec fluid HFE-7100 formulation are more regular. This is most likely because the prints are less luminescent, causing the small aggregates of DFO observed along the ridges to look less dotted. Minutiae, therefore, look clearer and better defined [7].

3) Fingerprints developed with the Novec fluid HFE-7100 formulation show better resolution of ridges than those developed with the CFC-113 formulation [7].

Because of its excellent performance, compatibility with many substrates and favorable environmental and safety profile, the Fingerprint Department, Institut de Recherche Criminelle de la Gendarmie Nationale, concluded that the DFO formulation using Novec fluid HFE-7100 shows acceptable performance compared to the CFC-113 formulation, and as such will be put into service as a replacement solvent for CFC-113 [7].

Compatibility of 3M™ Novec™ Engineered Fluid HFE-7100 with various inks and substrates

Mr. Jon Stimac, Forensic Services Division, Oregon State Police, United States, recently completed work on a validation study for the use of Novec fluid HFE-7100 in latent fingerprint development. Mr. Stimac has submitted a technical paper to the *Journal of Forensic Identification*, based in part on his findings during his validation study. The paper is entitled, "The Search for Safe, Non-Running Solvents: A Brief History." After peer review, Mr. Stimac's paper was published in Volume 50, Number 5, September/October 2000 of the *Journal of Forensic Identification*.

Inks, Mr. Stimac says, consist primarily of dyes, resins and medium components—typically glycol-based solvents or water. The medium components

Ink compatibility in Ninhydrin formulations

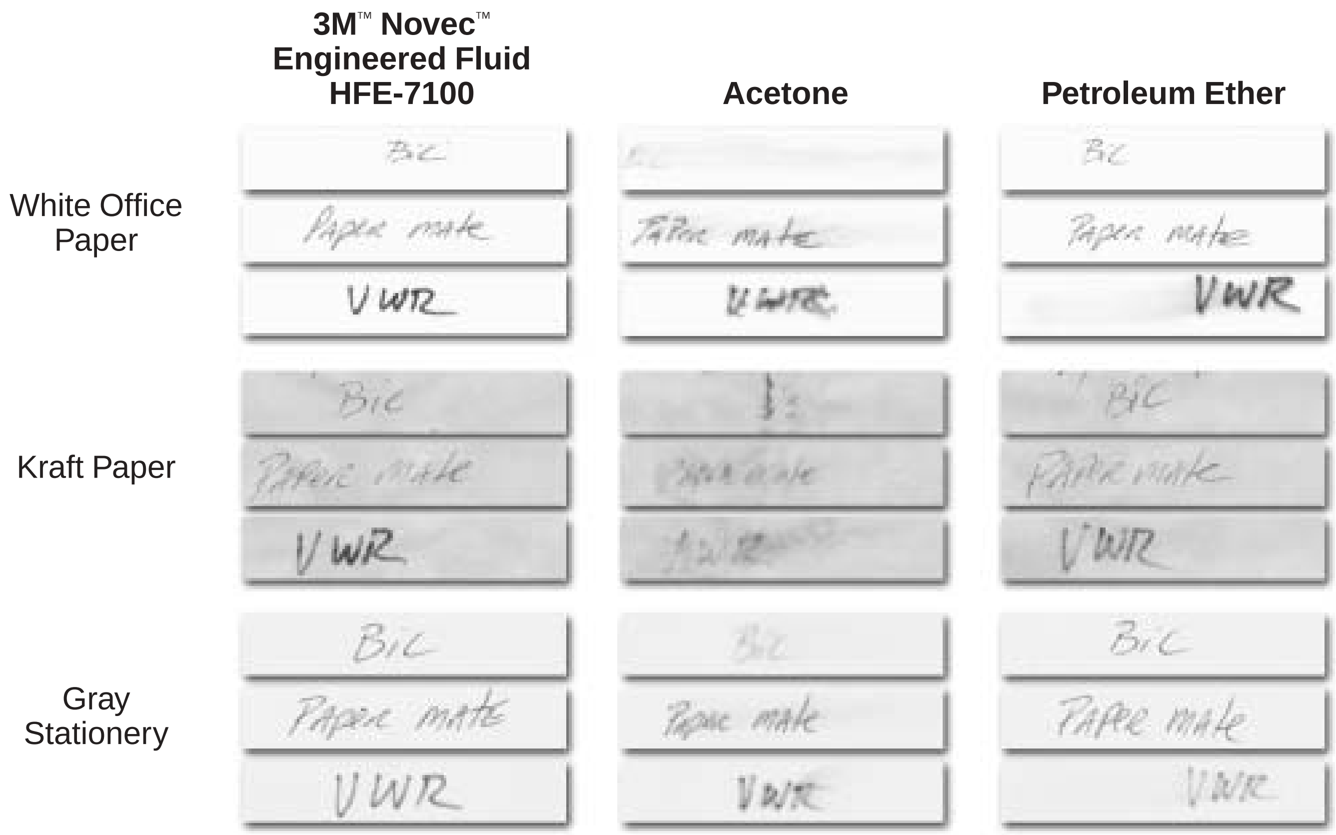


Figure 2

are used to suspend these dyes and resins, and provide a smooth flow of ink onto the paper [8]. The danger when developing latent prints on porous surfaces is loss of evidence. Highly-polar solvents can cause dyes and resins on the porous surface to run and bleed, permanently damaging important documents. It is not enough to be compatible with a few inks and substrates, however. Compatibility must be wide and comprehensive. In order to determine compatibility, Mr. Stimac tested Novec fluid HFE-7100 with several types of inks and papers.

He first conducted an ink running/substrate evaluation using the Novec fluid HFE-7100 ninhydrin formulation. For this evaluation, fingerprints were placed on white stationery, brown paper bag, cover paper and cardboard substrates, each having fifteen different ink sources applied to them (Figure 2) [8]. Using the Novec fluid HFE-7100 ninhydrin formulation put forth by the UK's Home office [3], the samples were processed and compared to samples processed with acetone-, methanol- and petroleum ether-based ninhydrin formulations. None of the inks ran or bled with the Novec fluid HFE-7100 formulation, but 10 of 15 inks processed with the acetone formulation had minimal to significant bleeding and 10 of 15 inks processed with the methanol formulation had minimal to significant bleeding [8]. Petroleum ether caused only one brand of ink to run, but the contrast (color intensity) of the developed latent prints was far weaker than those developed with the Novec fluid HFE-7100 solution [8].

Mr. Stimac then conducted experiments with the Novec fluid HFE-7100 DFO formulation recommended by the Institut de Recherche Criminelle de la Gendarmie Nationale [7] versus a commercially available methanol-based DFO spray developer. Once again the Novec fluid HFE-7100 formulation proved itself to be the better option, as 8 of the 15 inks processed with the methanol-based DFO spray showed consistent bleeding or running, ranging from minimal to significant, while the HFE-7100 fluid formulation had none [8].

The Oregon State Police concluded that the use of Novec fluid HFE-7100 in both ninhydrin and DFO solutions yields developed fingerprints of equal or better quality than those produced with acetone or petroleum ether [9]. Additionally, they discovered that whether the reagent was ninhydrin or DFO, the 3M™ Novec™ Engineered Fluid HFE-7100 formulation dried faster than any other tested [9]. Finally, because of its favorable environmental and safety profiles—non-ozone-depleting, low global warming potential, nonflammable and low toxicity—they recommended Novec fluid HFE-7100 as a replacement for petroleum ether to help reduce the possible hazards to examiners in the laboratory [9].

3M's continuing research

There are many reasons for consideration of 3M™ Novec™ Engineered Fluid HFE-7100 as a carrier solvent in latent fingerprint formulations for ninhydrin and DFO. Certainly its environmental and safety profiles make it attractive as a long-term replacement, but if the solvent were not an effective carrier—i.e., if formulations using Novec fluid HFE-7100 could not develop prints—it would not be under consideration by these agencies.

There are a number of factors involved in developing a clear, distinct latent print, but 3M scientists believe that a combination of three key physical properties is responsible for the developing ability of the HFE-7100 fluid-based formulation: density, surface tension and viscosity.

A parameter called the "wetting index" brings these critical properties together and may further explain why density, surface tension and viscosity play such important roles. Introduced more than 20 years ago, the wetting index was developed to evaluate the potential of different solvents to penetrate or "wet" tight spaces. Not a true dimensionless number—like a Reynolds number—the wetting index is based more on the empirical observations which indicate that a fluid with high density, low viscosity and low surface tension is better able to penetrate a porous surface. 3M has found that the performance of various solvents does tend to scale with the wetting index in situations where the flow or wetting characteristics of the solvent are important.

The wetting index is calculated as:
*1000*density/(surface tension*viscosity)*

Organic liquids like petroleum ether, heptane, methanol and acetone have density values ranging from 0.64 to 0.79. Fluorinated liquids, however, all measure greater than 1.

The second and third properties—surface tension and viscosity—are responsible in part for the number of fingerprints developed. The lower the surface tension of the carrier solvent, the more invasive the fluid is. That is, it can more effectively penetrate a porous surface than a solvent with higher surface tension, allowing it to develop older fingerprints—fingerprints whose amino acid signatures have gone deep into the porous surface. The surface tension and viscosity of Novec fluid HFE-7100 (13.6 dynes/cm at 25°C and 0.61 cp) are lower than both CFC-113 (17.3 dynes/cm at 25°C and 0.68 cp) and HFC-43-10mee (14.1 dynes/cm at 25°C and 0.67 cp). The

Solvent	Density (g/ml)	Viscosity (cp)	Surface Tension (dynes/cm)	Wetting Index
Novec fluid HFE-7100	1.52	0.61	13.6	183
HFC-43-10mee	1.58	0.67	14.1	167
CFC-113	1.56	0.68	17.3	133
Petroleum ether	0.74	0.38	16.1	121
Acetone	0.79	0.32	26.2	94
Heptane	0.68	0.41	20.3	82

Table 5

combination of these three properties gives Novec fluid HFE-7100 the highest wetting index of all solvents considered for fingerprint formulations.

The detailed experiments performed by the Police Scientific Development Branch, United Kingdom, in which the number of prints and then the percentage of cases with developed prints was compared, lend further credence to this hypothesis [4].

Continuing testing of 3M™ Novec™ Engineered Fluid HFE-7100 in 3M laboratories is largely confirming the research conducted by the aforementioned law enforcement agencies. 3M scientists have used the same formulations as these agencies to produce latent fingerprints of excellent quality on several types of papers and cardboards. Recently 3M researchers developed a full handprint off a Kleenex® tissue—using a Novec fluid HFE-7100 ninhydrin formulation—without ruining the tissue. Additionally, the HFE-7100 based ninhydrin formulation was found to effectively develop fingerprints on U.S. currency. Formulations are under development to effectively develop fingerprints on thermal paper without blackening the paper itself.

3M is also investigating the use of Novec fluid HFE-7100 and other 3M™ Novec™ Engineered Fluids in additional forensics applications, including use as a carrier solvent for Rhodamine for non-porous substrates, THC/Marijuana identification field test, blood analysis and blood spattering formulations.

Conclusions

Research conducted by the Fingerprint Research Group, Police Scientific Development Branch, United Kingdom; Fingerprint Department, Institut de Recherche Criminelle de la Gendarmie Nationale, France; and Forensic Services Division, Oregon State Police, United States, and validated by 3M Company, shows that ninhydrin and DFO formulations using 3M™ Novec™ Engineered Fluid HFE-7100 have demonstrated their ability to successfully and repeatedly develop high-quality latent fingerprints with crisp, clear, distinct ridge detail [4,7,8]. Low in toxicity and nonflammable, these formulations can be safely used at crime scenes. Additionally, these Novec fluid HFE-7100-based formulations have repeatedly demonstrated their reliability and performance in development of latent fingerprints on some previously un-processable porous evidence. Clearly, 3M™ Novec™ Engineered Fluid HFE-7100 has demonstrated excellent performance in all criteria as a replacement solvent for CFC-113 in latent fingerprint development formulations.

For more information on 3M™ Novec™ Engineered Fluid HFE-7100 for use in latent fingerprint development on porous surfaces, please contact your local 3M office or 3M in St. Paul, MN U.S.A., at 800-810-8513.

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