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Evaluation of improved methods for the recovery and detection of organic and inorganic cartridge discharge residues

S.J. Speers*, K. Doolan, J. McQuillan, J.S. Wallace

Northern Ireland Forensic Science Laboratory, 151 Belfast Road, Carrickfergus BT38 8PL, UK

Abstract

An efficient vacuuming system for the recovery of organic and inorganic cartridge discharge residues (CDRs) from clothing was developed. Sample extracts for organic CDR analysis were cleaned and concentrated by an automated solid-phase extraction system. Two systems were used for the analysis of organic CDRs, a sensitive gas chromatography–mass spectrometry method and a modified automated high-performance liquid chromatography pendant mercury drop electrode system. Inorganic cartridge discharge residues were analysed by scanning electron microscopy with energy dispersive analysis of X-rays. The combined systems have been applied to firearms casework.

1. Introduction

The analysis of cartridge discharge residues (CDRs) is important in determining if a person has been in contact with, or close to, the discharge of a firearm. At present the method of choice is scanning electron microscopy (SEM) with an energy dispersive analysis of X-rays (EDAX) for the investigation of the morphology and chemical composition of inorganic CDRs from the primer [1–3]. Automation of the system is necessary because the procedure for searching for residues is time consuming [4]. An alternative or complementary approach would be the detection of partial or unburnt propellant (organic CDRs) on hands or clothing.

Modern propellants are composed mainly of the explosive nitrocellulose (single-based). Other explosive ingredients may also be present for example, nitroglycerine (NG) (double-based) or NG and nitroguanidine (triple-based). Propellants also contain stabilizers such as diphenylamine (DPA), ethyl centralite (EC) or methyl centralite (MC), flash inhibitors such as 2,4-dinitrotoluene (2,4-DNT) and plasticisers.

Although there is extensive literature on the identification and detection of propellants [5–7], little work has been devoted to the combined analysis of organic and inorganic CDRs recovered from hands [8] and clothing [9] in forensic casework. The analysis of organic CDRs has concentrated on the detection of NG and 2,4-DNT by high-performance liquid chromatography (HPLC) with a pendant mercury drop electrode (PMDE) [10]. The HPLC–PMDE system requires a clean up and concentration of samples containing organic residues for optimum performance [8].

Initially this laboratory used the technique developed by Lloyd and King [8] for the clean-up and concentration of organic CDRs and explosive residues. A slurry mix of the sample

* Corresponding author.

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and Chromosorb 104 was sucked into a 1 mm I.D. PTFE tube containing Amberlite XAD-4. Organic explosive residues and CDRs were selectively retained on support material. The cleaned organic residues were then eluted from the tubing using acetonitrile–water (25:12, v/v). The technique was found to be time consuming and, because of the large number of samples processed by this laboratory, the extraction system has been adapted and optimised (Harvey and Speers [11]). In this study an efficient vacuuming system was investigated for the recovery of organic and inorganic CDRs from clothing. This was coupled to an automated system for the extraction and clean-up of the organic CDRs using a solid-phase extraction (SPE) system containing Chromosorb 104 and Amberlite XAD-4. A sensitive gas chromatography–mass spectrometry (GC–MS) method has been developed for the analysis of DPA, EC and MC. An existing HPLC–PMDE system [10] has been adapted such that the automated deoxygenation and injection of samples for the detection of NG and 2,4-DNT was achieved. GC–MS and HPLC–PMDE analysis were performed on fractions of the same extract. The system has been applied to routine firearms casework for a trial period to assess its evidential value.

2. Experimental

2.1. Standards

Pure samples of EC and MC were obtained from a munitions manufacturer. NG was extracted from a known composition of Super Dopex explosive (Explosives Chemical Products, UK). DPA, 1,3-dinitrobenzene (1,3-DNB) and 2,4-DNT were obtained from Aldrich (Gillingham, UK).

2.2. Materials

All solvents were HPLC grade unless stated otherwise. All reagents were analytical-reagent grade unless stated otherwise.

2.3. HPLC equipment

The equipment consisted of a 8810 isocratic HPLC pump (Spectra-Physics, Hemel Hempstead, UK) and a 7970/7980 series column block heater operating at 35°C (Jones Chromatography, Hengoed, UK).

The HPLC column was a Zorbax ODS 150 mm × 4.6 mm I.D., 5 μm particle size and 80 Å pore size (Jones Chromatography). The column was cleaned with 50 ml of methanol at the end of each analysis run.

The eluent consisted of methanol–phosphate buffer pH 3.0 (55:45). The flow-rate was 1.2 ml/min.

Phosphate buffer was prepared by adding 11.5 g of 85% (w/v) phosphoric acid to 4 l of deionised water. Anhydrous potassium carbonate was added to the mixture to increase the pH to 3.0.

The eluent was continuously refluxed in a 2-l flask, under an atmosphere of nitrogen, to remove oxygen.

The detector was a Model 420 pendant mercury drop LC electrode connected to a Model 400 electrochemical detector via an external cell cable and 407 module (EG&G Princeton Applied Research, Princeton, USA). The electrode settings were: standing mercury drop electrode (SMDE) and drop size small. The detector settings were: reductive d.c. mode, −1 V, reference electrode 5 *M* aqueous lithium chloride [10]. A new mercury drop was automatically dispensed at the start of each run via a signal from an autoinjector.

The standard contained 1 ng/μl of NG, 1,3-DNB and 2,4-DNT. The detection limits for NG and 2,4-DNT, based on a signal-to-noise ratio of 3, were 50 and 65 pg, respectively, per 10-μl injection.

The detector was connected to a Drew 3040 data capture unit linked to a 286–16 MHz personal computer operating the Drew Scientific Chromatography Roseate software (Drew Scientific, London, UK).

Samples were deoxygenated and injected using a programmable Model 231 autoinjector (Gilson Medical Electronics, France) fitted with a 10-μl sample loop, a Model 401 diluter and a universal

switching valve module with on-line nitrogen. Methanol was used for injector needle wash. The autoinjector was programmed to deoxygenate the sample for 3 min prior to injection, dispense a new mercury drop on the HPLC–PMDE system at the start of each run and commence the data collection on the Drew chromatography software system (a full description of the programme can be obtained from the author).

2.4. Gas chromatography–mass spectrometry

A Trio 2000 quadrupole mass spectrometer with VG Lab base data system (VG Biotech, Manchester, UK), Model HP5890 gas chromatograph and HP7673 autosampler (Hewlett-Packard, Wokingham, UK) was used. The capillary column was a Rtx-1, 15 m × 0.32 mm I.D., 1 μm film thickness (Thames Chromatography, Maidenhead, UK). The conditions of analysis were: helium carrier gas, inlet pressure 5 p.s.i. (1 p.s.i. = 6894.76 Pa); temperature settings 85°C initially then ramped to 250°C at 30°C/min, maintained at 250°C for 5 min; 1 μl splitless sample injection.

Further conditions were: GC–MS interface temperature 250°C; mass spectrometer source temperature 250°C; scan rate 0.9 s; interscan time 0.1 s; masses scanned 45 to 300 u full scan. The samples were analysed with the instrument in electron impact (EI) mode with a setting of 70 eV. The system was set up for selective ion recording (SIR). Samples were initially screened for single masses 169 (DPA), 120 (EC) and 134 (MC) and subsequently reinjected for confirmation based on the masses 77, 167, 168, 169 for DPA; 77, 120, 148 for EC and 77, 106, 134 for MC. The standard contained 1 ng/μl of DPA, EC and MC. Detection limits for all three analytes, based on signal-to-noise ratio of 3, was 10 pg per injection. Prior to injection the acetonitrile extract was blown down to a volume of 20 μl under an atmosphere of nitrogen.

2.5. SEM–EDAX

Inorganic CDRs were analysed using a Camscan series 2 scanning electron microscope (Cambridge, UK) connected to a Link AN 10000 analyser (High Wycombe, UK) and detected by an automatic residue detection system (ARDS) developed at this laboratory [12].

2.6. Recovery of organic and inorganic CDRs from clothing

Suction sampling apparatus used for the recovery of organic and inorganic CDRs from clothing consisted of a 25 mm diameter in-line Deldrin filter holder unit (Gelman product No. 1109, Northampton, UK) with one of the nylon hose barb adapters removed. The filter used was a 25 mm diameter 1 μm fluoropore membrane filter FHLP 02500 (Millipore, Watford, UK). When in use the filter holder is attached to an Edwards E2 M12 vacuum pump (Crawley, UK). An autosampler cap is used to seal the holder before and after use. Wallace and McKeown [13] have described in detail the suction sampling and contamination avoidance procedures.

2.7. Extraction procedure on Millilab 1A workstation

The Millilab 1A workstation (Millipore) is a personal computer-controlled automated robotic system which performs sample extraction from filters and SPE according to user-defined programmes.

Organic residues were extracted from the Deldrin filter holders, then cleaned and concentrated by SPE on the Millilab workstation. The system is fully automated and incorporates wash procedures into its programme. (A full description of the programme can be obtained from the author.)

Deldrin filter holders were adapted for use on the Millilab workstation by the addition of a male and female PTFE luer adapter with 1/8 in. (1 in. = 2.54 cm) NPTF thread. (These were manufactured within the laboratory.)

2.8. Extraction of organic CDRs from Deldrin unit on Millilab 1A workstation

A 400-μl volume of methanol, containing internal standard 1,3-DNB (to monitor extraction efficiency, concentration 0.25 ng/μl), were

pipetted into each Deldrin filter holder unit to wet the filter. After 2 min 500 μl of acetonitrile were added and left for 5 min to dissolve any organic residues on the filter. The Deldrin unit was then purged with nitrogen for 20 s and the extract collected in a disposable glass tube 160 mm × 10 mm. This was repeated with a further 500 μl of acetonitrile. The total extract was cleaned and concentrated using SPE. The filter from the Deldrin holder was processed for inorganic residues (see below).

2.9. SPE of organic CDRs on Millilab 1A workstation

Chromosorb 104, 125–150 μm mesh size, was obtained from Phase Separations (Clwyd, UK) and Amberlite XAD-4 from Sigma (Poole, UK). Prior to use both materials were prepared and cleaned according to the procedure recommended by Lloyd [14]. Amberlite XAD-4 and Chromosorb 104 (10 mg:30 mg) were packed between frits into empty 1.5-ml size SPE tubes (Alltech, Carnforth, UK).

The packed SPE tubes were first rinsed with 2 ml of acetonitrile to remove possible contaminants and then conditioned with 2 ml of deionised water to activate the support material. The acetonitrile–methanol organic CDR extracts from the Deldrin units were diluted 1:9 with deionised water and applied to the SPE columns at a rate of no greater than 4 ml/min. The columns were then washed with 2 ml of acetonitrile–water (1:10) and purged to dryness in an atmosphere of nitrogen. Analytes were eluted from the columns with 300 μl of acetonitrile into tapered 1.1 ml glass vials (Chromacol, Welwyn Garden City, UK). The 300 μl acetonitrile samples were analysed by HPLC–PMDE for NG, 2,4-DNT and 1,3-DNB and by GC–MS for DPA, EC and MC.

2.10. Extraction of inorganic CDRs for SEM–EDAX analysis

After the extraction of organic CDRs by the Millilab workstation, the 1-μm fluoropore filter was removed from the Deldrin holder and placed in a 150-ml glass beaker. The filter holder interior and autosampler cap interior were rinsed with light petroleum (quality over 120°C) into the same beaker and the volume made up to 20 ml. The beaker was ultrasonicated for 10 min and allowed to settle. The suspension was filtered through a 13 mm diameter 25 μm wire-mesh coarse filter housed in a Swinnex holder No. SX 0001300 (Millipore) and then through a 13 mm diameter 1-μm fluoropore filter No. FALP 01300 also housed in a Swinnex holder. After filtration the final 1-μm filter was place on a 13 mm diameter aluminium stub (Agar Scientific, Stansted, UK) using double-sided adhesive tape. The stub was coated with carbon using an automatic vacuum controller E6430 (Bio-Rad Microscience Division, Hemel Hempstead, UK) and analysed by SEM–EDAX for the presence of inorganic CDRs. The Deldrin filter holders and glassware were reused after thorough cleaning according to the procedure published by Wallace and McKeown [13].

3. Results and discussion

3.1. Efficiency of SPE of organic CDRs

Samples containing organic CDRs extracted from Deldrin filter units used for the suction sampling of clothing need to cleaned and concentrated using SPE to maximise the performance of the HPLC–PMDE and GC–MS detection systems. The previous SPE system, using a mixture of Chromosorb 104–Amberlite XAD-4 (10 mg:3.5 mg) in 1 mm I.D. PTFE tubing developed by Lloyd and King [8], was laborious and time consuming in its preparation and execution. An alternative clean-up and concentration system capable of being automated using 1.5-ml SPE columns was investigated.

Commercial reversed-phase (C_{18}) and aminopropyl (NH_2) 100-mg 1.5-ml SPE columns were compared to a 1.5-ml SPE column prepared in the laboratory containing 40 mg Chromosorb 104–Amberlite XAD-4 (30:10). The ability of the different SPE materials to extract and re-

cover organic CDRs from acetonitrile using the Millilab workstation was assessed.

An acetonitrile standard containing 10 ng each of NG, 1,3-DNB, 2,4-DNT, DPA, EC and MC was used to simulate organic CDRs extracted from a Deldrin filter unit. The acetonitrile standard was added to the reversed-phase C_{18}, aminopropyl and Chromosorb 104–Amberlite XAD-4 SPE columns according to the procedure recommended in the Experimental section. To improve the binding of the organic residues to the aminopropyl support the mixed standard was diluted 1:19 with hexane.

The organic CDRs were recovered from the SPE columns in 300 μl of acetonitrile, analysed by HPLC–PMDE and GC–MS and the recovery calculated. The experiments were performed twice to obtain an average recovery. The results are listed in Table 1.

It was demonstrated that the recovery of residues from the Chromosorb 104–Amberlite XAD-4 SPE column prepared in the laboratory was more efficient (greater than 95%) compared to the commercial C_{18} (32–47%) and aminopropyl SPE columns (2–9%). This confirms the work of Lloyd [15] who found that Chromosorb 104 and Amberlite XAD-4 were the most efficient supports for the recovery of organic explosive residues from relatively polar solvents. To reduce the minimum volume required to elute the organic residues from the 1.5-ml SPE columns prepared in the laboratory, 40 mg of support material was used. Using these columns a 1.4-ml extract from the Deldrin unit is cleaned and concentrated to 300 μl.

Table 1

Average recovery of 10-ng standard containing organic CDRs by SPE on the Millilab workstation

Organic CDR	Recovery (%)		
	SPE support material		
	Chromosorb–Amberlite	C_{18}	Aminopropyl
NG	95	47	5
1,3-DNB	96	36	9
2,4-DNT	96	35	9
DPA	98	42	7
EC	95	39	2
MC	96	32	5

The relative standard deviation of the percentage recovery of the organic residues from the Chromosorb 104–Amberlite XAD-4 SPE columns ranged from 3.5 for 1,3-DNB to 5.5% for DPA.

The Chromosorb 104–Amberlite XAD-4 SPE column prepared in the laboratory allows full automation of the extraction process on the Millilab workstation. Subsequent experiments were performed using these SPE columns.

3.2. Assessment of Millilab 1A workstation extraction of organic CDRs

CDRs are recovered from clothing by suction sampling using a Deldrin filter holder (1-μm fluoropore filter) connected to an Edwards vacuum pump. The efficiency of extraction of organic CDRs from the Deldrin unit and subsequent SPE clean-up and concentration using the Millilab workstation was assessed. Three new cotton laboratory coats were vacuumed for a period of 5 min each using the procedure described in the Experimental section. A 400-μl volume of methanol containing 10 ng of NG, 1,3-DNB, 2,4-DNT, DPA, EC and MC were added to each Deldrin holder to simulate the recovery of organic CDRs from clothing. Organic CDRs were extracted from the three Deldrin units and cleaned-up and concentrated by SPE using the Millilab workstation. To test the system for carryover, clean Deldrin units and SPE tubes (blanks) were extracted after each sample. The extracted organic CDRs and blanks were analysed by HPLC–PMDE and GC–MS. The results are listed in Table 2. All blanks were negative.

It was found that the recovery of organic CDRs was reduced when the Deldrin filter unit was used (57–78% recovery compared to 95% recovery from the SPE columns). This may be explained by the presence of garment fibres recovered with the CDRs. The more material present within the Deldrin unit, the more difficult it is to extract the CDRs with a given

Table 2

Recovery of organic CDRs from Deldrin units and subsequent SPE on Millilab workstation

Organic CDR	Extraction efficiency (%)
NG	78
1,3-DNB	72
2,4-DNT	74
DPA	57
EC	60
MC	67

volume of acetonitrile (total extract 1.4 ml). Using a greater volume of acetonitrile poses problems with the subsequent 1:9 dilution of extracts for SPE. The Millilab workstation is limited to using 160 mm × 10 mm tubes for dilution (a total workable volume of 14 ml).

When examining "dirty" garments a number of Deldrin units may be required to cover the entire surface as a result of the fluoropore filter becoming clogged with material, hence reducing the vacuuming efficiency. An attempt to use a 20-μm pre-filter to prevent clogging was abandoned because this resulted in reduced recovery of inorganic CDRs.

3.3. Recovery and analysis of CDRs from clothing (six shots)

The efficiency of the technique to recover and detect CDRs from different types of clothing worn during the firing of six rounds of ammunition from a revolver was assessed.

Three different items of clothing with varying retentive properties for CDRs (laboratory coat, woollen jumper and sweatshirt) were doped with residues from a Colt python .357 Magnum revolver using double-based Winchester .357 ammunition by wearing each garment and firing six shots in still air conditions. The garments were suction sampled for residues within 1 h of the shooting using recommended procedures. The recovered residues were extracted and analysed for organic and inorganic CDRs. Results are listed in Table 3. Precautions were taken to ensure that no contamination of garments with CDRs from other sources occurred. Samples of air within the room where the shooting took place and the hands and clothing of the person prior to performing the shootings were analysed and found to be negative.

CDRs were recovered from all garments, with the sweatshirt and laboratory coat giving better recovery than the woollen jumper. In all cases organic and inorganic CDRs were easily identified. It was assumed at the start of the experiment that the woollen jumper would have the best retention of CDRs but this was not reflected in the results. It is suggested that a reason for this could be the vacuum suction sampling procedure which works best on flat/tight weave garments such as the laboratory coat and sweat shirt.

3.4. Recovery and analysis of CDRs from clothing (one shot)

The suction sampling and analysis techniques were repeated to determine if organic and inorganic CDRs could be detected on clothing worn

Table 3

Analysis of CDRs recovered from clothing (six shots)

Garment	Organic CDR (ng)				Inorganic CDR (No. of particles)			
	NG	2,4-DNT	DPA	EC	Pb, Sb, Ba	Sb, Ba	Pb, Sb	Pb, Ba
Laboratory coat	976	39	4.6	1	3	2	71	3
Sweatshirt	1273	39	7.4	2.2	30	5	175	13
Woollen jumper	730	10	1.7	0.5	1	–	34	6

during the firing of one round of ammunition from a revolver.

Two new laboratory coats were each doped with residues from a single shot using the same revolver and ammunition already described. Precautions were taken to avoid contamination from extraneous sources. The samples were extracted and analysed for CDRs. The results are listed in Table 4.

Detectable quantities of organic and inorganic residues were recovered from the laboratory coats (although no 2,4-DNT or MC was detected). The amounts of NG, DPA and EC detected were well above the detection limits of the systems. A smaller number of inorganic CDR particles were recovered from the laboratory coats compared to the garments doped with six shots. Examples of HLPC–PMDE and GC–MS chromatograms of organic residues recovered from a laboratory coat worn during the firing of a single shot are shown in Figs. 1 and 2.

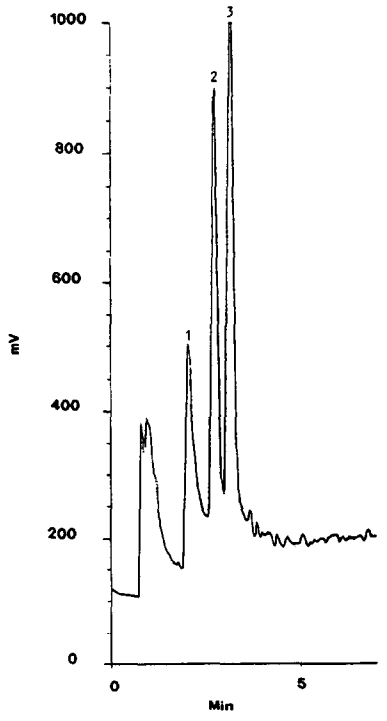

Fig. 1. HPLC–PMDE chromatogram (50 nA full scale) of organic residues recovered from a laboratory coat worn during the firing of one shot. Chromatographic conditions: Zorbax ODS column, 150 × 4.6 mm I.D.; eluent, methanol–phosphate buffer pH 3.0 (55:45), flow-rate 1.2 ml/min. Detector setting −1 V. Peaks: 1 = oxygen; 2 = 1,3-dinitrobenzene; 3 = nitroglycerine.

3.5. Survey of clothing submitted to the laboratory for CDR examination

For a trial period of three months clothing submitted to the laboratory for inorganic CDR analysis were also examined for organic residues. Organic residues detected during the trail period were not used as evidence in criminal proceedings. Thirteen different firearm-related incidents (cases F1–F13) with a total of 186 exhibits were examined. One case F1 accounted for 100 exhibits. The positive results are listed in Table 5.

Only one exhibit, mask (a) in case F13, was positive for inorganic CDRs although no organic CDRs were detected for this item. The indicative inorganic particle Pb, Ba was detected in 17 exhibits from four cases but, in the absence of any unique inorganic particles (Pb, Sb, Ba/Sb, Ba), they were reported as negative.

Five exhibit extracts from three different cases were positive for organic CDRs with two of these exhibits also having a single indicative (Pb,

Table 4

Analysis of CDRs recovered from clothing (one shot)

	Organic CDR (ng)			Inorganic CDR (No. of particles)			
	NG	DPA	EC	Pb, Sb, Ba	Sb, Ba	Pb, Sb	Pb, Ba
Laboratory coat 1	775	3.5	2.6	2	1	1	–
Laboratory coat 2	910	8.7	4.3	3	–	7	14

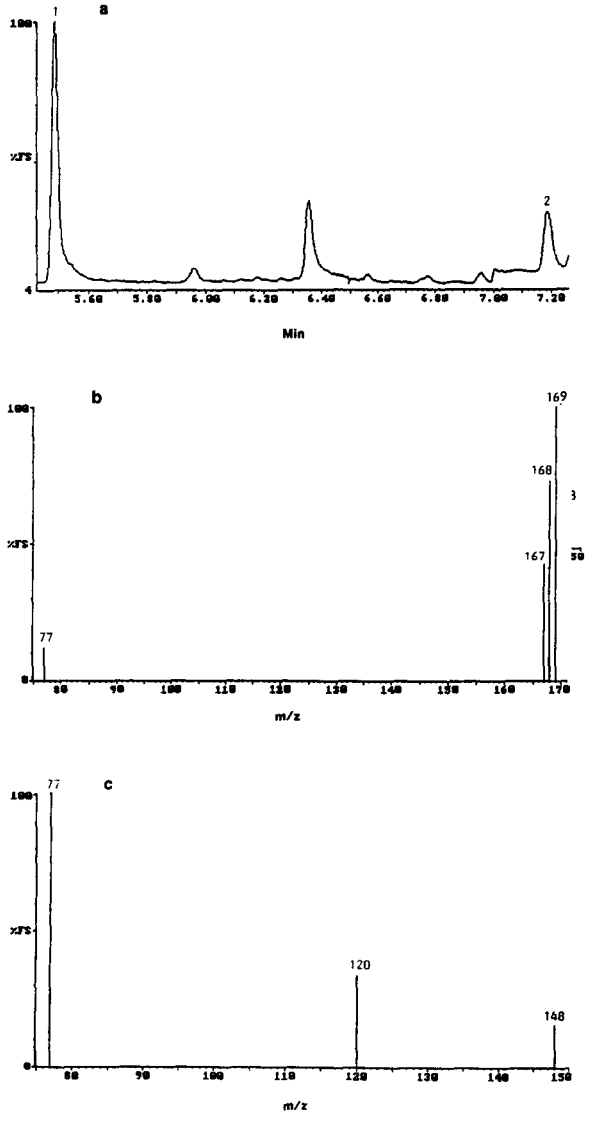

Fig. 2 (a) GC–MS total ion chromatogram of organic residues recovered from a laboratory coat worn during the firing of one shot. Chromatographic conditions: capillary column, Rtx-1, 15 m × 0.32 mm I.D., 1 μm film thickness; helium carrier gas, inlet pressure 5 p.s.i.; temperature 85°C initially then ramped to 250°C at 30°C/min, maintained at 250°C for 5 min. GC–MS interface and source temperature 250°C; scan rate 0.9 s, interscan time 0.1 s; masses scanned 45–300 u full scan; electron impact mode 70 eV. Peaks: 1 = diphenylamine; 2 = ethyl centralite. (b) GC–MS selective ion recording confirmation of diphenylamine recovered from a laboratory coat worn during the firing of one shot. GC–MS conditions as for (a). (c) GC–MS selective ion recording confirmation of ethyl centralite recovered from a laboratory coat worn during the firing of one shot. GC–MS conditions as for (a).

Ba) inorganic particle detected. All three cases would have been reported as negative based on the inorganic CDR results. Unfortunately it was not possible to confirm DPA, EC and MC in the extracts from the 186 items by GC–MS due to a terrorist explosion at the laboratory which resulted in the loss of the samples prior to analysis.

Only 5 out of 186 exhibits were positive for organic CDRs and only 1 exhibit positive for unique inorganic CDR particles. This may be the result of a number of factors:

(1) The suspects may not have fired a weapon.

(2) The suspects in the 13 firearm incidents were not arrested at the scene of the crime. It was therefore a period of time before their clothing was collected and submitted to the laboratory.

(3) Terrorists in Northern Ireland take considerable precautions to avoid the deposition and recovery of CDRs from their clothing (such as the wearing of boiler suits and rubber gloves).

On the basis of this trial the analysis of organic CDRs has greater sensitivity than inorganic CDR analysis. The detection of a unique inorganic CDR particle in the absence of organic residues from case F13 exhibit (a), may be the result of the composition of the ammunition used. A survey of propellants encountered in the British Forensic Science Laboratories found that 2 out of 5 propellants did not contain NG [16]. Using GC–MS analysis increases the range and specificity of organic CDRs that can be detected. Further work analysing unburnt or partially burnt propellant removed from gunshot entrance holes on clothing submitted to the laboratory over period 1991–1992 found that a combination of organic CDRs were identified in 60 out of 61 samples. DPA was identified in 57 propellant samples. More importantly DPA only was found in 34 samples, emphasising the need for all systems —HPLC–PMDE, GC–MS and SEM–EDAX.

4. Conclusions

An efficient vacuuming system for the recovery of organic and inorganic CDRs from

Table 5

Survey of clothing submitted to the laboratory for CDR analysis

Case	Exhibit	Organic CDR (ng)		Inorganic CDR (No. of particles)			
		NG	2,4-DNT	Pb, Sb, Ba	Sb, Ba	Pb, Sb	Pb, Ba
F1	(a) Upper front body/cuffs	124	93	–	–	–	–
F1	(b) Pockets	300	4	–	–	1	–
F3	(a) General outer/body	124	–	–	–	–	–
F3	(b) General outer/body	2068	4	–	–	–	–
F8	(a) Front pocket	1685	–	–	–	1	–
F13	(a) Mask	–	–	1	1	1	1

clothing and an automated system for the clean-up and concentration of organic residues has been developed. Using this system CDRs from a single shot fired under laboratory conditions can be detected on clothing by HPLC–PMDE, GC–MS and SEM–EDAX.

A survey of clothing submitted to the firearms laboratory for examination suggests that the systems used for the detection of organic CDRs are more sensitive than the SEM–EDAX system for the detection of inorganic CDRs.

In the survey five items of clothing were positive for organic CDRs and only one item of clothing positive for inorganic CDRs, emphasising the need to analyse for both types of residue. The automated clean-up technique is to be applied to the recovery of CDRs from hand swabs.

5. Acknowledgements

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