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Quality Assurance Testing of an Explosives Trace Analysis Laboratory—Further Improvements

ABSTRACT: The Forensic Explosives Laboratory (FEL) operates within the Defence Science and Technology Laboratory (DSTL) which is part of the UK Government Ministry of Defence (MOD). The FEL provides support and advice to the Home Office and UK police forces on matters relating to the criminal misuse of explosives. During 1989 the FEL established a weekly quality assurance testing regime in its explosives trace analysis laboratory. The purpose of the regime is to prevent the accumulation of explosives traces within the laboratory at levels that could, if other precautions failed, result in the contamination of samples and controls. Designated areas within the laboratory are swabbed using cotton wool swabs moistened with ethanol water mixture, in equal amounts. The swabs are then extracted, cleaned up and analyzed using Gas Chromatographs with Thermal Energy Analyzer detectors. This paper follows on from a previous published paper describing the regime and summarizing subsequent results from approximately 6 years of tests. Lessons learned and improvements made over the period are also discussed. Monitoring samples taken from surfaces within the trace laboratories and trace vehicle examination bay have, with few exceptions, revealed only low levels of contamination, predominantly of RDX. Analysis of the control swabs, processed alongside the monitoring swabs, has demonstrated that in this environment the risk of forensic sample contamination, assuming all the relevant anti-contamination procedures have been followed, is so small that it is considered to be negligible. The monitoring regime has also been valuable in assessing the process of continuous improvement, allowing sources of contamination transfer into the trace areas to be identified and eliminated

KEYWORDS: forensic science, quality assurance, explosives, trace analysis, gas chromatography, chemiluminescence

In 1989 the Forensic Explosives Laboratory (FEL) adopted Gas Chromatography with a Thermal Energy Analyzer (commonly known as GC/TEA) as its principal technique for analysis of organic high explosives traces (1,2). These systems have been modified by FEL over the last 14 years to improve their performance, including their sensitivity. Where any forensic trace analysis is carried out, a rigorous system of contamination prevention procedures is essential. For this reason the instruments are located within a purpose built suite of laboratories referred to as the Trace Explosives Laboratory, in which forensic casework samples are processed and analyzed in order to detect any explosives present. The GC/TEA systems are routinely used to analyze for the presence of twelve compounds of explosives significance: ethylene glycol dinitrate (EGDN), nitrobenzene (NB), 2-nitrotoluene (2-NT), 3-nitrotoluene (3-NT), 4-nitrotoluene (4-NT), 1,2,3-propanetriol trinitrate (nitro-glycerine) (NG), 2,6-dinitrotoluene (2,6-DNT), 2,4-dinitrotoluene (2,4-DNT), 3,4-dinitrotoluene (3,4-DNT), 2,4,6-trinitrotoluene (TNT) 2,2-bis(dihydroxymethyl)-1,3-propanediol tetranitrate (pentaerythritol tetranitrate; PETN) and 1,3,5-trinitro-1,3,5-triazacyclohexane (cyclotrimethylene trinitramine; RDX). This paper will outline the contamination prevention procedures currently in use and present the data from the analysis of monitoring samples between January 1998 and January 2004. Since FEL operates a policy of continuous improvement, many amendments to the procedures and alterations to the facilities have been carried out over the period and some of these are also outlined. This paper should be read in conjunction with the previous paper (3) that presents the results of samples taken between November 1989 and February 1998.

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Main Trace Laboratory Contamination Prevention Procedures

A detailed discussion of explosives trace contamination control principles and practice has been given by Hiley (4). Additionally, a brief description explaining the main principles of the FEL prevention procedures was given in the previous paper (3).

Before entering the trace laboratory, there are several "external" precautions. The laboratory is held at positive air pressure to prevent the ingress of particulate contamination via any possible ingress points, including the main door. All personnel entering the laboratory must have showered and had a complete change of clothes since last handling bulk explosives or visiting areas where explosives are stored or processed. They must also be trained in an FEL Standard Operating Procedure "Work in Trace Areas" (FEL standard operating procedure SOP205) Access to the laboratory is restricted and is by swipe card only.

Once these precautions have been observed, the laboratory may be entered. Since November 1998 an extended arrangement of lobby areas has been in use (Fig. 1). The revised entry procedure into the trace laboratory is as follows:

(1) The operator enters the trace laboratory corridor, treading on a Micro-pure® clean room "sticky" mat with both feet.
(2) The air handling system monitoring panel is observed to ensure that the air filtration system is in working order, there is an adequate positive air pressure, and that entry is permitted.

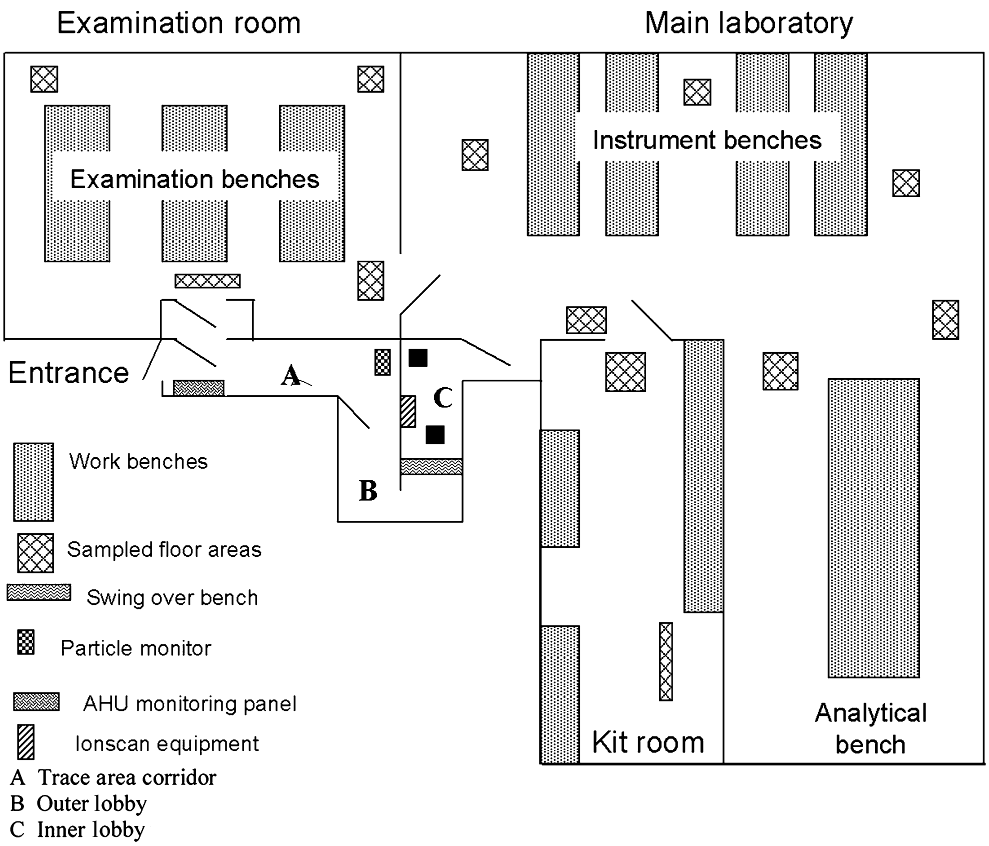

FIG. 1—*Outline map of main trace laboratory (not to scale) showing locations sampled.*

(3) The operator removes outdoor shoes and dons a pair of trace laboratory shoes, checks the particle monitor, signs the entry logbook and removes all wrist and finger jewellery.
(4) The operator enters the outer lobby area, stepping on another Micro-pure® clean room mat with both feet and immediately washes their hands and wrists thoroughly, drying them with disposable paper towel.
(5) The operator dons a pair of disposable gloves and while sitting on the narrow, low bench dividing the outer and inner lobbies, puts on one disposable overboot, swings the covered foot over the bench onto the inner lobby floor. An overboot is then placed on the other foot in the same manner. The operator can then stand up in the inner lobby.
(6) The operator dons a disposable oversuit and a disposable hat.
(7) An Ion Mobility Spectrometer (Smiths Detection Ionscan 400A) is then used to sample the outer surface of the disposable oversuit. If this indicates the presence of the explosives NG, TNT, PETN or RDX, entry is not permitted. If the Ionscan equipment is not operational, due to repair or maintenance work, entry is still permitted provided all the other detailed procedures have been carried out. The Ionscan equipment was formally commissioned on January 11, 1999, with the introduction of an FEL standard method (Standard method SM114). Since then, only 0.18% of entries to the trace laboratory have resulted in alarms for NG, TNT, PETN or RDX. Several of these alarms were caused by contamination within the laboratory, arising from casework, being transferred to the disposable suits of personnel within the inner lobby whilst they were donning them. Thus, it is unlikely that these persons ever posed a contamination risk to the laboratory. With rigorous monitoring, access control and contamination prevention procedures controlling all entry to the trace laboratory, the possibility of a contaminated user entering the laboratory is remote.
(8) On entering the main trace area, before touching any surface, the operator removes his or her disposable gloves, washes his or her hands and dons a clean pair of disposable gloves.

When all steps described in (1–8) have been followed, a work surface is prepared. The work surface is cleaned with proprietary cleaner, followed by ethanol, and covered with disposable glazed paper. This ensures that any possible small traces of explosives on the bench are either removed or isolated by the layer of disposable paper. If casework is to be carried out, control samples would be taken at this stage.

A further protection is the control of all materials and air entering the laboratory, further reducing the likelihood of explosives contamination entering from outside. All materials used in the trace laboratory are carefully sourced so as to optimize their fitness for purpose. A list of approved suppliers is maintained and defined in the laboratory quality management system. All materials are covered in extra wrapping at the supplier's premises, which is removed in stages, during passage through the lobby areas, before entry. This ensures that any materials destined for the trace laboratory cannot become contaminated on their passage through the site. The air supply to the laboratory passes through bag filters, followed by large high efficiency particle arrestor (HEPA) filters that remove suspended particles. Since early 1999, the filtered air has been sampled, post-HEPA, by a particle monitoring system (Versaport 10, Multiport Particle Counter, OptiCal Sciences Ltd., Northampton, UK) via a port mounted above the fire damper before it enters the laboratory via a diffuser grille. The flow of air is controlled so as to maintain a slightly higher pressure within the laboratory than outside. At the entrance of the trace laboratory, there is a relay panel from the main control panel, allowing the air handling system to be monitored before entry. A formalized standard operating procedure for the use of the data produced by the relay panel and for system maintenance was introduced in April 2001 (FEL standard operating procedure SOP214).

A regular formalized laboratory cleaning rota (FEL standard operating procedure SOP203), introduced in October 1996, ensures that, in the unlikely event of small explosives traces being present in the laboratory, they are not permitted to persist or accumulate. This rota has been revised periodically since then to take into account various modifications to the laboratory, its procedures, and the introduction of new equipment.

The intention of the overall system is that contamination of samples could only take place if multiple breaches of contamination avoidance procedure occur. The routine use and discarding of disposable clothing, gloves and paper along with the laboratory cleaning regime is designed to ensure that a set of casework samples is not put at risk by any previous set. In order to monitor the effectiveness of the laboratory contamination prevention procedures, samples are regularly taken from surfaces within the laboratory.

The Laboratory Monitoring Regime

Apparatus, Materials and Analytical Procedure

Laboratory monitoring samples are taken using solvent-moistened cotton wool swabs. The solvent used since March 1996 is a mixture of ethanol and water in equal volumes. Sample processing and analysis has evolved slightly since 1989, but has been in essence that described by Crowson et al. (5). The moistened swabs are extracted using the same solvent, followed by solid phase extraction using specially prepared clean-up tubes containing Chromosorb 104 (Sigma-Aldrich, Dorset, UK, 100–120 mesh) and subsequent desorption using ethyl acetate (Standard method SM200). All monitoring samples are taken and processed using materials from the supplies also used in casework; this inherently provides weekly quality assurance of materials used in the processing of forensic samples. A single GC/TEA analysis is made of each sample and candidate explosives responses are confirmed by further analysis (see Action Criteria). A controlled standard, containing known quantities of NG, TNT, PETN and RDX, has been used to measure the limit of detection on the GC/TEA instruments on a formalized weekly basis since May 2000 (FEL standard operating procedure SOP112).

Locations Sampled—Main Trace Laboratory

Monitoring samples are taken from all of the laboratory bench surfaces upon which samples are processed. Figure 1 is an outline map showing the locations sampled in the main trace laboratory. In order to reduce the analytical burden, samples from three examination/sampling benches are united as one (referred to below as "examination benches," total swabbed area 11.25 m^2), and the relatively large bench upon which swab extractions and clean-ups are carried out, is not sub-divided ("analytical bench," total swabbed area 4.80 m^2). A series of benches upon which all of the analytical instruments stand are again sampled as one ("instrument benches," total area 16.20 m^2). The three benches in the room used only for the preparation of swabs, hand-test kits and trace explosives recovery kits are again sampled as one ("kit room," total swabbed area 2.55 m^2). It would be very time-consuming to swab the entire laboratory floor, therefore a series of 14 boxes, that cover a total area of approximately 4.25 m^2, has been marked in well-trodden sections and these are sampled ("floor"). This is approximately 3.8% of the total accessible floor area.

Monthly monitoring samples are taken from areas that are not normally covered during weekly sampling, for example the telephones, the clampstands, the windowsills, etc. These monthly monitoring samples are taken according to a rota introduced into the quality assurance standard operating procedure (FEL standard operating procedure SOP202) in December 1997.

The weekly monitoring samples have been taken (with few exceptions) since late 1989. Over the years, progressively more areas have been sampled and there was a major change when the laboratory moved to a new building. During the period under discussion the most significant change was the addition of two small floor areas of the inner lobby to the "Floor" sample in April 1999.

One swab is prepared alongside those used for sampling, but is retained unused as a control. It is then processed and the extract analyzed alongside the monitor samples. Two samples spiked at low levels with a known multi-component explosives standard are also processed and analyzed, the spiked swab sample and the spiked solution sample. The percentage of explosives recovered from the spiked samples shows the efficiency of the extraction and clean-up process.

Centrifuge

Monitoring samples from the centrifuge were taken weekly, during the period under consideration, until April 1999. With the introduction of redesigned sample clean-up tubes that contain a plug of fiber to trap particulates, the centrifuge had fallen into disuse and was removed from the weekly monitoring sample regime. The monitoring samples from the centrifuge were then added to the rota for monthly monitoring samples. If the centrifuge is to be used for any casework, the methods now state that monitoring samples must be taken before it is used and worked up and analyzed alongside the casework samples. The results obtained from the monitoring samples taken from the centrifuge during the period under discussion are not included in the data below. However, no explosives were detected in the samples.

Locations Sampled—Secondary Trace Laboratory

The secondary trace laboratory is used as a back up facility for the main trace laboratory. It is used mainly for research but is available for use with casework exhibits that are suspected of being heavily contaminated. On entry to the smaller secondary trace laboratory, similar precautions are taken as when entering the main trace laboratory, one difference being the lack of any Ionscan® equipment. Monitoring samples are taken from all benches in the laboratory upon which samples may be processed and from four marked floor areas. Figure 2 is an outline map showing the locations sampled. The instrument benches (total swabbed area, 9.0 m^2) are sampled using one swab and treated as one area. The analytical bench (total swabbed area, 6.7 m^2) is also sampled using one swab. The four floor areas, swabbed and treated as one monitoring sample, cover an area of approximately 1.8 m^2 and make up approximately 7.9% of the total accessible floor area.

One swab is prepared alongside those used for sampling, but is retained unused as a control sample. Monitoring samples from the secondary trace laboratory are taken monthly. The secondary trace laboratory was completely refurbished in late 1998. The first

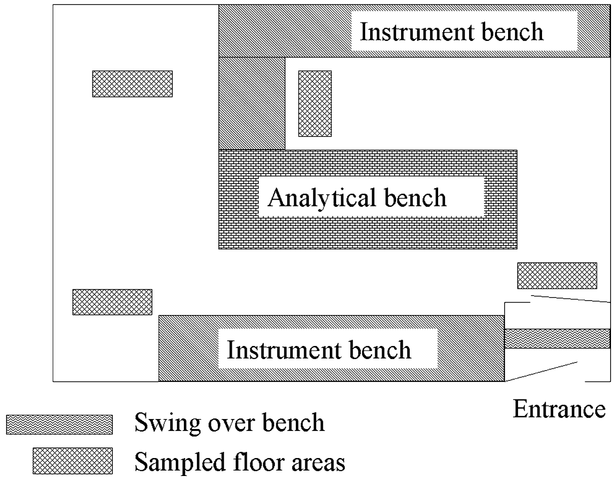

FIG. 2—*Outline map of secondary trace laboratory (not to scale) showing locations sampled.*

monthly quality assurance sampling after refurbishment was taken on 27 January 1999.

Locations Sampled–Trace Vehicle Examination Bay

The trace vehicle examination bay is used for trace sampling of vehicles and other exhibits too large to be taken into the other trace laboratories. Entry procedures for personnel and materials are identical to those described for the secondary trace laboratory. Entry and exit of vehicles and other large exhibits is through a roller shutter door to the main trace bay. Monitoring samples are taken from all benches (total swabbed area, 6.2 m^2) within the vehicle trace bay both before and after the cleaning which is carried out during the QA procedure. The benches are swabbed, cleaned with proprietary cleaner and ethanol, and swabbed again with a fresh swab. Figure 3 is an outline map showing the locations sampled. Floor area three is sampled each time monitoring samples are taken. One other floor area is also sampled on a rota basis. The two sampled floor areas, swabbed separately as two different monitoring samples, cover an area of approximately 2 m^2 and make up approximately 2% of the total accessible floor area.

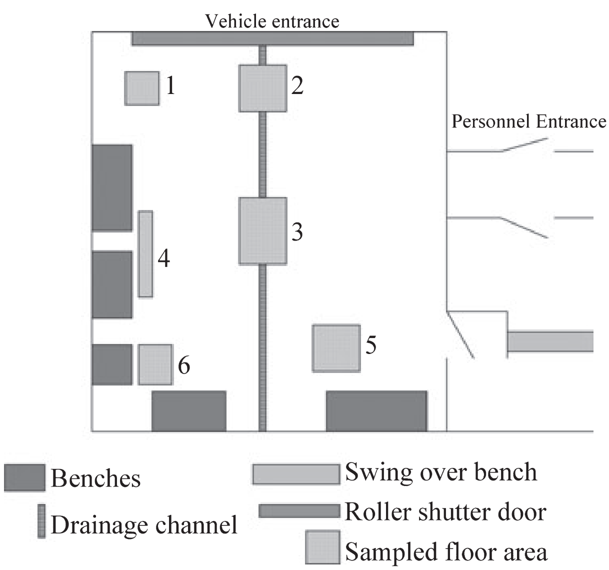

FIG. 3—*Outline map of trace vehicle sampling bay (not to scale) showing locations sampled.*

One swab is prepared along side those used for sampling, but is retained unused as a control sample. Another swab is used to sample the operator(s) and the glazed paper work surface. Monitoring samples are taken at least monthly and/or immediately before a vehicle is placed in the trace bay. Monitoring samples are also taken after cleaning has been carried out once a vehicle has been removed from the trace bay. The trace vehicle examination bay was completely refurbished in early 1997. The first monthly quality assurance samples, after refurbishment, were taken on 6 March 1997. A new lobby was built later that year and commissioned in March 1998.

Action Criteria

The main purpose of taking the monitoring samples is to ensure and demonstrate the continuing cleanliness of the laboratory. Where any explosives contamination is detected, actions are taken in accordance with the graduated response protocols summarized in Table 1. The levels of explosives used to define the action criteria are based upon several factors. These are, the limit of detection of the entire procedure, the levels considered significant during casework, and experience of carrying out such work over a number of years. With regard to the limit of detection for the entire procedure, the aim is to achieve a detection of 1 ng in 100 μL ethyl acetate extract. Limits of detection are checked on a weekly basis by analyzing a standard solution containing 100 pg/μL NG, TNT, PETN, and RDX. The results obtained are used to calculate the limits of

TABLE 1—*Action criteria when explosives are detected.*

Nominal amount found (ng)	Action to be taken
<5	No action required. Levels are acceptable. The result may be confirmed on other TEA systems at the discretion of the laboratory manager.
Between 5 and 10	1 Confirm result on other TEA systems. 2 Clean the area.
>10	1 Confirm result on other TEA systems. 2 Suspend operations until area cleaned. 3 Reswab the area and check that the levels of explosives are acceptable before resuming operations.
>100	1 As for "Greater than 10" above. 2 A thorough inquiry is to be held. This inquiry must cover the following aspects: What were the possible sources of contamination? Could such an incident be avoided in future by changing any procedures? Could the incident have been dealt with more effectively? Is there any potential effect upon casework processed in the laboratory during the period under investigation? 3 The above inquiry may be carried out, at least in part, by the trace laboratory manager but must have the full approval of the head of chemistry and research who is ultimately responsible. 4 Recommendations from such inquiries must be considered at the following quality system review meeting and those which are accepted must be implemented as soon as practically possible but no later than 6 months after the meeting.

detection for these explosives for that week, these are then formulated into the control charts. The control charts are carefully maintained. The levels used to define the action criteria are kept under review.

Accreditation

It is a Home Office requirement that, in order for the laboratory to carry out forensic casework, it shall be externally accredited by the United Kingdom Accreditation Service (UKAS) to ISO 17025. ISO 17025 is an internationally recognized standard of competence for testing laboratories and accreditation is based on annual audit. UKAS appointed auditors visit the laboratory and observe demonstrations of specified methods and check the results obtained so they may evaluate whether the work carried out is of the appropriate standard.

Summary of Test Results and Discussion

Monitoring Samples—Main Trace Laboratory

The monitoring sample results have been reviewed and assembled into a database. The results from January 1998 to January 2004 are examined here. However, to set these results into the context of previous years, they have been added to the results since 1989 in Fig. 4. This shows that the number of RDX detections in monitoring samples has decreased markedly over recent years. RDX has been detected in these samples more frequently than other explosives, since it is the most common nitro-containing explosive detected in casework samples. It is the major constituent of Semtex, PE4 and C4 plastic explosives.

During the period under discussion 1919 samples were taken of which 320 were control samples, leaving a total of 1599 from the laboratory benches or the laboratory floor. There have been 24 positive samples, 21 containing RDX and three containing other explosives. This means that 1575 samples from the laboratory benches or the laboratory floor were negative, i.e., 98.5%. No samples contained two or more explosives, which demonstrates that low-level multi-component standards, used to calibrate the analytical instruments and for spiked samples, are not a source of laboratory contamination. Figure 5 shows the distribution of explosives by amount detected. Figure 6 shows the number of samples in which explosives were detected in each sampling location of the main trace laboratory.

There have been 21 detections of RDX in total during the period under discussion. As a result of one contamination incident, >100 ng of RDX was detected on three occasions. An exhibit, which subsequently proved to be heavily contaminated, was taken into the trace laboratory for examination and sampling. While the cleaning regime ensures that contamination does not remain in the laboratory, the levels of RDX present were relatively high, in trace terms, and the laboratory had to be cleaned several times, before monitoring samples demonstrated that the levels of RDX were <5 ng. The subsequent internal inquiry made several recommendations to avoid a similar incident occurring in the future. However, by its very nature, casework will always be the most likely source of contamination since the levels of explosives are unknown at the time of sampling. Thus all casework presents an inherent contamination risk. Two other detections at lower levels were due to the same incident. Nine of the RDX detections have been below the 5 ng threshold that is considered to be an acceptable background level, according to the laboratory quality assurance procedure (FEL standard operating procedure SOP202).

On one occasion approximately 128 ng of TNT was detected in the floor sample. This was directly attributable to a casework exhibit that proved to be heavily contaminated. The same incident resulted in the detection of approximately 3.2 ng of TNT in the examination bench sample. PETN has also been found on a single occasion in a floor sample at a level of approximately 6.3 ng. This was also directly attributable to the processing of casework samples. These three positive results represent the only detections of any explosives other than RDX.

Over 146 monthly monitoring samples have been taken during the period January 1998 to January 2004. No explosives were detected in all but 2 of these samples. These samples contained <5 ng of RDX and the detections were directly attributable to the heavily contaminated casework sample mentioned above.

No explosives were detected in any of the 320 control swabs analyzed during this period. Unpublished research work, carried out by the FEL, indicates that the site background contamination at Fort Halstead consists primarily of low levels of TNT, PETN, and RDX. Significantly, there has not been a single occasion in the main trace laboratory when RDX has been detected along with either TNT or PETN, in the same monitoring sample, during the period under consideration. This indicates that the contamination prevention procedures work effectively against the background contamination. The presence of such background contamination could be viewed as advantageous in that it provides a constant challenge

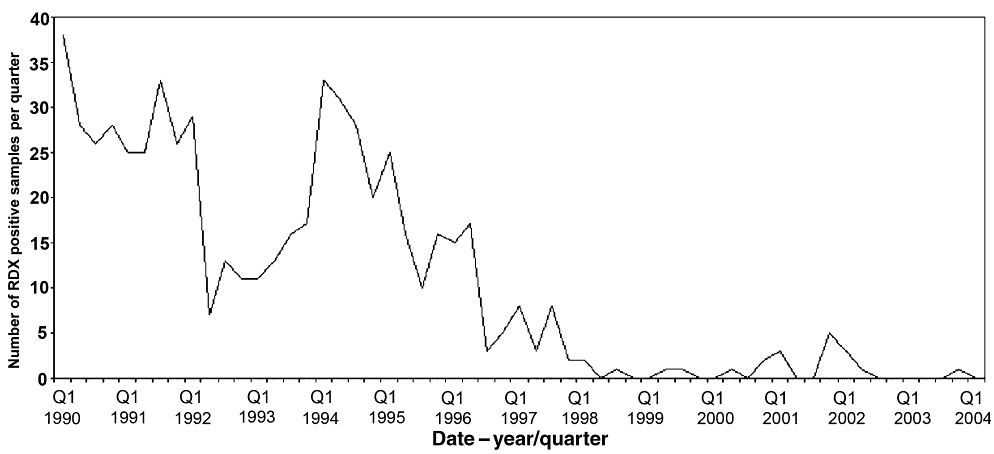

FIG. 4—*RDX in monitoring samples of main trace laboratory—November 1989 to January 2004—number of positive samples versus time.*

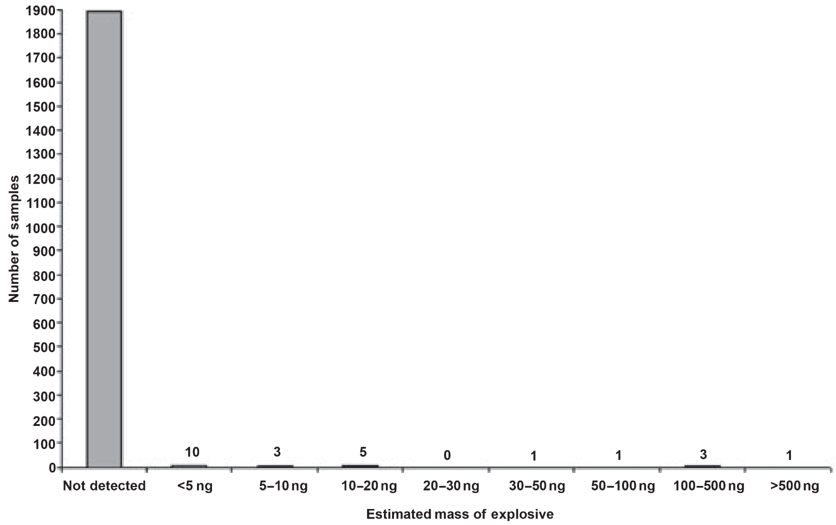

FIG. 5—*Explosives in monitoring samples of main trace laboratory—January 1998 to January 2004—number of samples versus estimated mass.*

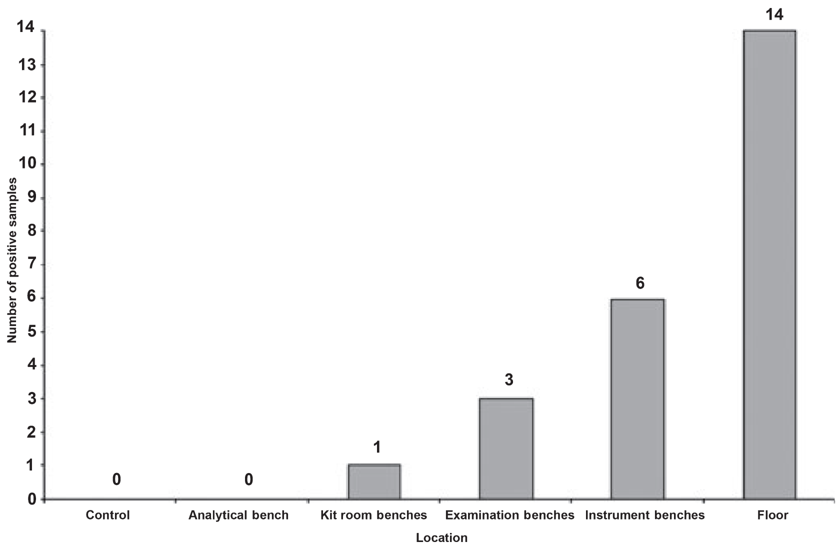

FIG. 6—*Explosives in monitoring samples of main trace laboratory—January 1998 to January 2004—locations.*

to the procedures and validates their effectiveness. Furthermore, quality assurance procedures similar to those outlined here would still be required if there were no site background contamination since casework containing explosives traces would still be examined within the laboratory.

Monitoring Samples—Secondary Trace Laboratory

The monitoring samples from January 1999 to January 2004 have been assembled into a database and reviewed. As in the case of the main trace laboratory, RDX is the most commonly detected explosive in the monitoring samples. In fact, no other explosives have been detected in these monitoring samples. The database contains 61 sets of results making a total of 244 samples of which 61 are control samples. Of the 183 laboratory bench and floor samples, 10 contained RDX of which only four were above 5 ng. Therefore 94.5% of all the samples taken have been confirmed as containing no explosives and 97.8% contain <5 ng. Figure 7 shows the distribution by amount detected in the secondary trace laboratory and Fig. 8 shows the locations of these detections. Of the 61 control samples that have been taken between January 1999 and January 2004, none have been found to contain any explosives.

Monitoring Samples—Trace Vehicle Examination Bay

The monitoring samples taken from the Trace Vehicle Examination Bay between March 1997 and January 2004 have been collated into a database and reviewed. There are 106 sets of data making a total of 588 samples of which 211 are control samples. In common with the other trace facilities, RDX is the most commonly detected explosive in the monitoring samples.

Twenty-nine samples have been found to contain RDX, 13 samples contained TNT and one sample contained NG. Thirty-two samples contained one or more explosives, of which 11 samples contained both RDX and TNT. Only one of these 11 samples contained >5 ng of either explosive; 7.9 ng of TNT. Of the 406 bench and floor samples 7.9% contained one or more explosives, 98.5% contained <5 ng of explosives and no explosives were detected in 92.1%. The three "benches after cleaning" samples

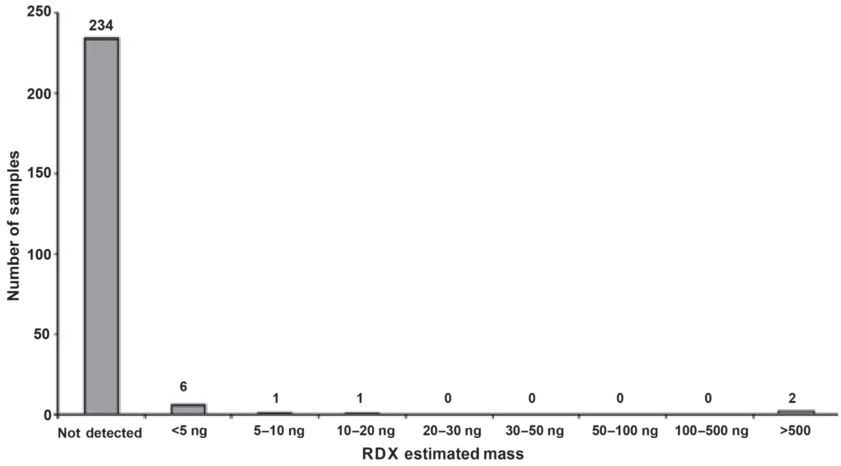

FIG. 7—*Explosives in monitoring samples of secondary trace laboratory—January 1999 to January 2004—number of samples versus estimated mass.*

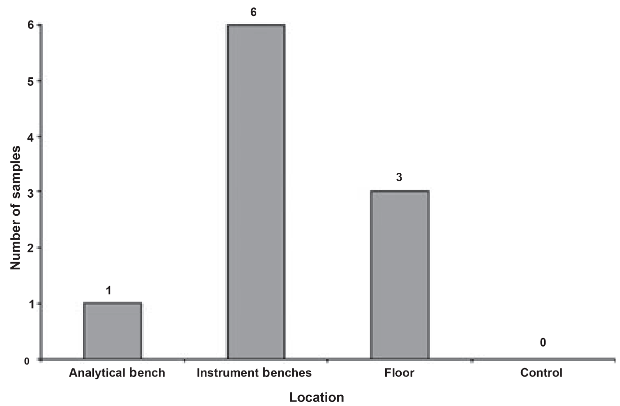

FIG. 8—*Explosives in monitoring samples of secondary trace laboratory—January 1999 to January 2004—locations.*

that contained explosives all contained <5 ng. Figure 9 shows the distribution by amount detected and Fig. 10 shows the locations of the detections.

The fact that some of the Trace Vehicle Examination Bay monitoring samples contain both RDX and TNT indicates that these explosives are constituents of the site background and highlights the difficulty of keeping an area trace clean when a vehicle has to enter via a large roller shutter door. It is important to note that when casework exhibits are sampled further controls are taken once the exhibit has been brought into the bay. No explosives were detected in any of the 211 control swabs taken.

Control Samples

There have been no explosives detected in any of the control swabs taken, processed and analyzed alongside monitoring samples during the period under question. The control swab results, taken together, are most significant because they demonstrate that the risk of forensic sample contamination, arising either from contaminated sampling materials or from contamination during processing, is extremely small, even when one or other of the monitoring samples taken at the same time shows that some contamination was present in the laboratory. The reason for this observation is that the prevention procedures have effectively isolated the samples from contamination.

Conclusions

A system of contamination prevention procedures incorporating both inner and outer protective measures has been implemented,

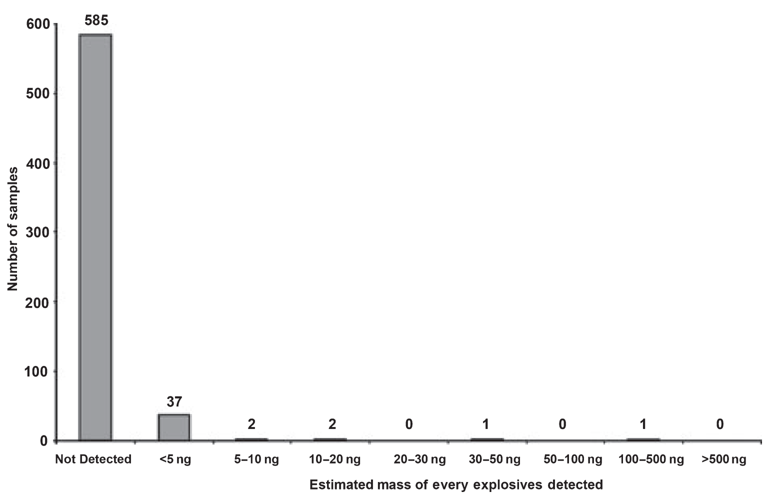

FIG. 9—*Explosives in monitoring samples in trace vehicle examination bay—March 1997 to January 2004—number of samples versus estimated mass.*

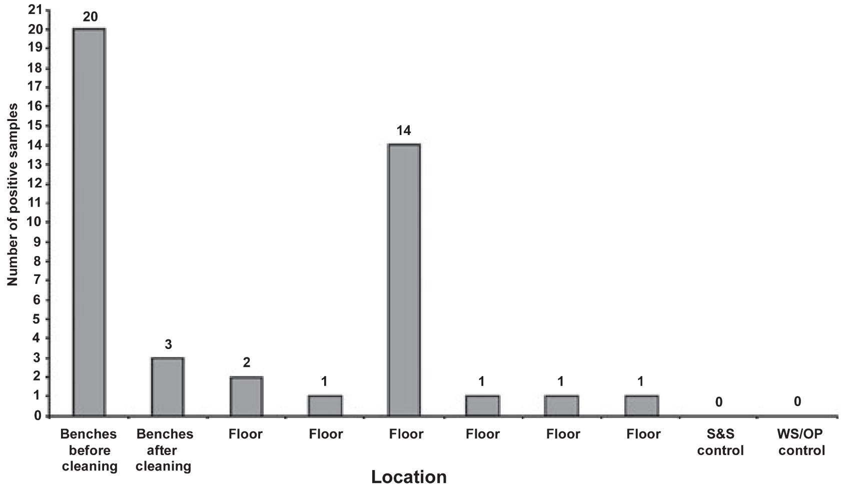

FIG. 10—*Explosives in monitoring samples in trace vehicle examination bay—March 1997 to January 2004—locations.*

with progressive improvements over the last 14 years. Monitoring samples taken from surfaces within the trace laboratories and trace vehicle examination bay have, with few exceptions, revealed only low levels of contamination, predominantly of RDX. Analysis of the control swabs, processed alongside the monitoring swabs, has demonstrated that in this environment the risk of forensic sample contamination, assuming all the relevant anti-contamination procedures have been followed, is so small that it is considered to be negligible. The monitoring regime has also been valuable in assessing the process of continuous improvement, allowing sources of contamination transfer into the trace areas to be identified and eliminated. The main risk of contamination comes from casework samples themselves. The complete lack of positive monitoring samples for the main laboratory containing RDX along with either TNT or PETN shows that the contamination prevention procedures in place are effective against the background explosives levels at Fort Halstead.

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Disclaimer

Any views expressed are those of the authors and do not necessarily represent those of the Department/HM Government.

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