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Quality Assurance Testing of an Explosive Trace Analysis Laboratory*

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ABSTRACT: During 1989, the Forensic Explosives Laboratory (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 which could, if other precautions failed, result in the contamination of samples and controls. This paper describes the regime and summarizes the results from approximately eight years of tests. Lessons learned and improvements made over the period are also discussed.

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

In 1989 the Forensic Explosives Laboratory (FEL), located near Sevenoaks, Kent, in the United Kingdom, adopted gas chromatography with a proprietary detector called the Thermal Energy Analyser (commonly known as GC/TEA) as its principle technique for explosives trace analysis (1,2). The detector is based upon the well known chemiluminescent reaction between nitric oxide and ozone, and is able to detect traces about one hundred times smaller than thin-layer chromatography, the method previously used at FEL (3,4). Thus, at the instigation of Dr. John Douse, a new and much broader system of contamination prevention procedures was introduced, and at about the same time regular monitoring samples began to be taken from surfaces in the laboratory. The improved prevention procedures were first implemented in a single room, but in early 1992 the FEL chemistry laboratories were transferred into a new building. The trace laboratory became a suite of rooms in this building and operations moved there in late April 1992. The rooms are isolated from the remainder of the building, and entry is through a single lobby in which protective clothing is donned. Entry to the suite is restricted to personnel trained in the contamination prevention procedures and visitors under escort of a trained person.

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Trace Laboratory Contamination Prevention Procedures

A detailed discussion of explosives trace contamination control principles and practice has been given by Hiley (5) and protocols used at the Forensic Science Agency of Northern Ireland have, in the same volume, been described by Murray (6). A brief description explaining the main principles of the FEL prevention procedures is given below.

The first and most important protection is to ensure that samples for trace explosives analysis do not come into direct contact with the laboratory, nor with the analyst, nor with anything else which may contaminate them. This can be regarded as the "inner" protection, and is of course simply an extension of the precautions which should be applied in all forensic examinations. It is achieved by using new disposable glassware and other items (such as disposable forceps to handle swabs) throughout. Samples only come into contact with these new disposable surfaces. On rare occasions, for example when unusual objects require the use of a particularly large piece of glassware which it would be wasteful to dispose of, the glassware is cleaned and tested for explosives traces immediately before use.

This paper concerns broader prevention procedures, called for clarity "outer," because they seek to ensure that explosives traces are minimized in the environment around the "inner" protection. For example, when a forensic sample is processed a number of procedures are used. First, the operator must have washed and had a complete change of clothes since last handling bulk explosives or visiting a magazine (an explosives store). Before entering the laboratory (in a transitional entrance lobby), the operator puts on a new disposable oversuit and overshoes, and once inside, washes hands and puts on new disposable plastic gloves. Separate hats with snoods which enclose long hair were added to the protective clothing in 1996. The operator makes a final clean of the bench work surface immediately before starting work, and then covers this cleaned work surface with disposable paper. Thus, even if the bench surface initially had a small amount of explosives contamination upon it, this would be largely removed by cleaning and any remainder isolated by a layer of paper. (The trace laboratory benches are never used to examine bulk explosives, but trace levels of explosives are sometimes detected on the bench surface during the quality assurance tests described below.)

A further "outer" protection is to control the entry of materials and air into the laboratory so as to minimize ingress of explosives traces from outside. Materials are covered in extra wrapping at the manufacturer's premises, and this wrapping is removed at the entrance to the laboratory. The air supply to the laboratory passes through large high efficiency (HEPA) filters which remove suspended particles, and the flow of air is controlled so as to maintain a slightly higher pressure within the laboratory than outside it. In

tandem with these various measures, a regular laboratory cleaning program prevents the accumulation of any traces which may enter.

The intention of the overall system is that contamination of samples can only take place if breaches of both outer and inner contamination prevention procedures occur—the passage of explosives traces somehow from outside the laboratory into the immediate environment of a forensic sample followed by their transfer through the inner protection into that sample. The discarding of clothing/gloves/paper and the laboratory cleaning regime ensures that a breach of outer protection does not persist to expose many sets of samples to a risk of contamination. In order to monitor the effectiveness of the outer prevention procedures, samples are regularly taken from surfaces within the laboratory.

The Laboratory Monitoring Regime

Apparatus, Materials, and Analytical Procedure

Laboratory monitor samples are taken using solvent-moistened cotton wool swabs. The solvent was for many years methyl-*tert*-butyl ether (MTBE) but within the last two years has been changed to a mixture of ethanol and water in equal volumes. Sample processing and analysis has evolved slightly over the years since 1989, but has been in essence that described in Crowson et al. (7). The recent ethanol/water moistened swabs are extracted using the same solvent and the resulting extract cleaned-up by adsorption onto Chromosorb 104 (Sigma-Aldrich, Dorset, UK, 100-120 mesh). Samples are taken and processed using materials from the supplies also used in forensic casework. A single GC/TEA analysis is made of each sample, and since 1992, candidate explosive responses for which the mass (estimated by comparison of GC peak area with that produced by a single standard explosive solution) exceeds 5 ng have normally been confirmed by further analyses (see "Action Criteria" section below).

Locations Sampled

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 order to reduce the analytical burden samples from three, examination/sampling benches are united as one (referred to below as "examination benches"), and the relatively large bench upon which swab extractions and clean-ups are carried out is not sub-divided ("analytical bench"). A series of benches upon which all of the analytical instruments stand are again sampled as one ("instrument benches"). Because it would be very time-consuming to swab the entire laboratory floor, a series of 12 boxes, covering a total area of approximately 4 m^2, has been marked in well-trodden parts and these are sampled ("floor").

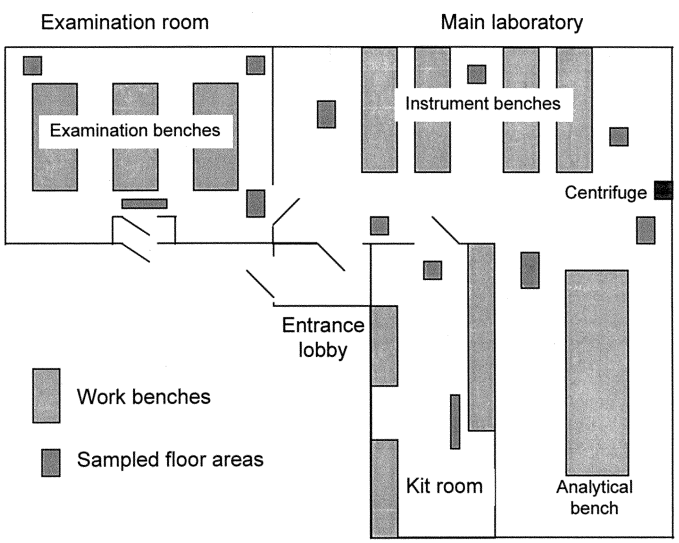

FIG. 1—*Outline map of trace laboratory (not to scale) showing locations sampled.*

The samples have been taken weekly (with very few exceptions) since late in 1989. Over the years, progressively more areas have been sampled, and there was of course a major change when the laboratory moved to the new building. Following the discovery of contamination in a centrifuge (see below), the replacement machine has been added to the sampling regime.

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 range of explosives are also processed and analyzed to confirm the efficiency of the recovery and analysis process.

Action Criteria

Since the main purpose of the weekly monitoring is to ensure the continuing cleanliness of the laboratory, actions are taken according to the results, as summarized in Table 1. The levels of explosives used to define the action criteria are based upon several factors, these being the real 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. The levels are kept under review.

Summary of Test Results and Discussion

Monitor Samples

The monitor sample results from November 1989 to February 1998 have been reviewed and assembled into a database. RDX (cyclotrimethylene trinitramine) has been detected in these samples far more frequently than any other explosive. Figure 2 shows the distribution of RDX findings by amount detected. Ninety-two percent of the samples were either confirmed as containing no RDX (65%), or gave results from a single GC/TEA analysis which indicated the possible presence of RDX up to an estimated mass of 5 ng (the identity of possible RDX at these levels is not normally con-

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

No More Than 5 ng of Explosive Detected in Any Sample	No Mandatory Action
More than 5 ng detected in a sample	Presence and identity of explosive confirmed by further analyses and clean the area.
More than 10 ng detected in a sample	Clean the area from which the sample was taken and re-test until shown to be negative.
More than 100 ng detected in a sample	Conduct local enquiry to establish the possible contamination source. Review casework which could have been affected by the contamination. Clean the area from which the sample was taken and re-test until shown to be negative.

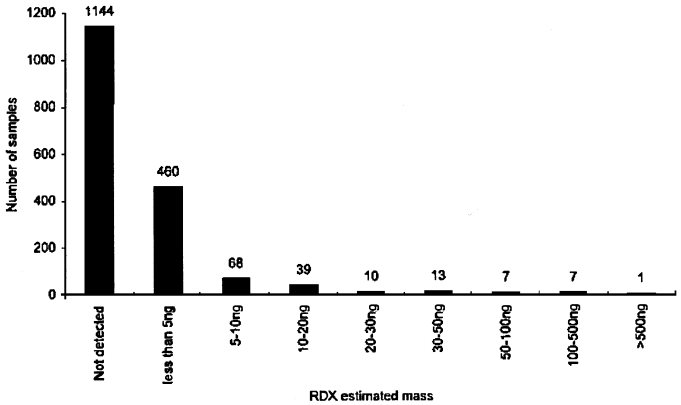

FIG. 2—*RDX in monitor samples—number of samples versus estimated mass.*

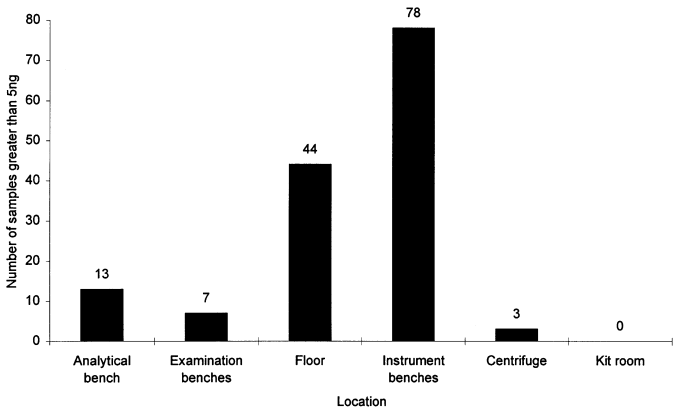

FIG. 3—*RDX in monitor samples—locations.*

firmed by further analyses; see Table 1 below). On eight occasions more than 100 ng has been detected and one of these was the March 1996 detection of contamination in the laboratory centrifuge (see further discussion of this incident below). Figure 3 shows for each sampling location the numbers of samples in which more than 5 ng of RDX was detected. The data for the examination benches include a few early results from a single bench in the old laboratory upon which examinations could be made or kits constructed. There is limited data from the centrifuge (104 samples tested) and kit room (88 samples tested) locations because these were added quite recently to the sampling regime. Two locations account for the large majority of these samples: the instrument benches and the floor. Higher levels of contamination were to be expected on the instrument benches before the move to the new laboratory since these were at that time outside the controlled trace area. However, the tendency for these to show greater numbers of samples above 5 ng has continued since the move. This can be ascribed to the fact that the instrument benches see a great deal of use and, although cleaned on a regular weekly basis, are not cleaned at every occasion of use (this is not considered necessary because samples are contained within septum vials during instrumental analysis). Analogous arguments apply to the floor areas which are sampled. These are in heavily trodden locations and, although regularly cleaned, the floor is not cleaned every time it has been trodden upon. The three findings in excess of 5 ng from the centrifuge were all associated with the major contamination incident dealt with below.

Figure 4 shows the amounts of explosives other than RDX in excess of 5 ng detected in the monitor samples, Fig. 5 shows which explosives have been detected, and Fig. 6 shows the locations. The numbers of detections are relatively small and the amounts have all been less than 100 ng. PETN has been detected most often, followed by TNT. The locations in which other explosives have been detected parallel those for RDX, for similar reasons.

Control Swabs

Possible explosives detections in the control swabs processed and analyzed alongside the monitor samples have been very uncommon and very small. Of 401 such samples analyzed, 397 have

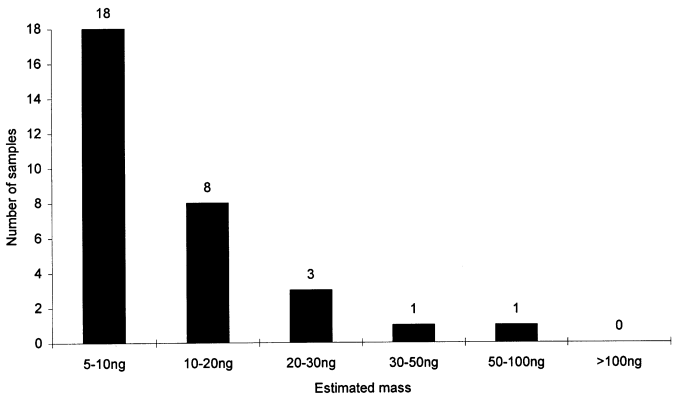

FIG. 4—*Other explosives in monitor samples—number of samples versus estimated mass.*

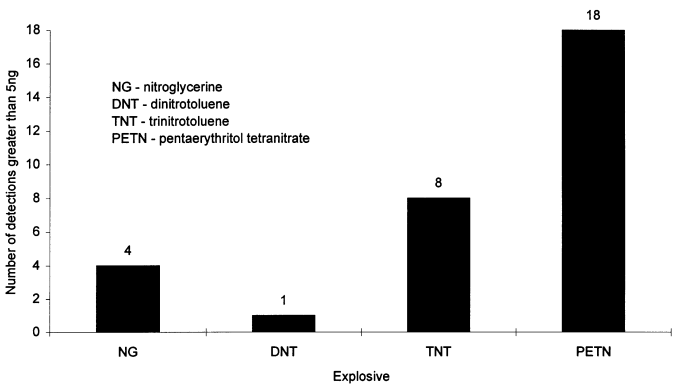

FIG. 5—*Other explosives in monitor samples—explosives detected.*

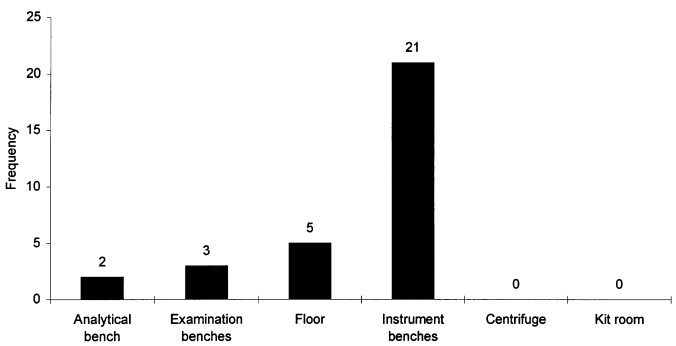

FIG. 6—*Other explosives in monitor samples—locations.*

been negative. On one occasion a response indicating the possible presence of RDX was observed, and on two occasions responses indicating other possible explosives have been indicated by single (i.e., unconfirmed) analyses. On all three occasions the estimated levels were less than 1 ng. On one further occasion possible TNT was indicated by a single analysis at an estimated level of 1.2 ng. 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 monitor samples taken at the same time shows that some contamination was present in the laboratory. The reason for this observation is of course that the "inner" prevention procedures have effectively isolated the samples from contamination.

Lessons Learned from Particular Incidents

Contaminated Photographic Equipment

In December 1989, soon after the establishment of the new contamination prevention procedures, approximately 140 ng of RDX was detected on the examination/kit preparation bench. Enquiries revealed that photographs had been taken of kits laid out on this bench during the previous week, and that the camera employed was probably contaminated with RDX. Since that time, care has been taken to ensure that photographic, video, and similar equipment which enters the laboratory on exceptional occasions is both clean and isolated as far as possible from contact with laboratory surfaces. Disposable paper and other protections are used to minimize contact.

Watches and Wrist Jewelry

In the first week of July 1992, the routine monitor samples were found to contain abnormal levels of RDX (approximately 160 ng in the instrument benches sample and 30 ng in the analytical bench sample) and, in accordance with the action criteria given in Table 1 a local inquiry was held. No casework had been affected, but the inquiry revealed that one member of staff had handled plastic explosive (British PE4 explosive which consists mainly of RDX) the previous week. Although this person had conscientiously followed all of the procedures then in force (bathed, washed hair and complete change of clothes), a swab of her wristwatch and watch strap revealed approximately 300 ng of RDX. An immediate ban upon the wearing of wristwatches and other wrist jewelry in the trace laboratory was introduced and has remained in force ever since.

Contaminated Centrifuge

In March 1996, substantial RDX contamination, estimated at 43 μg, was discovered in the trace laboratory centrifuge and the facts reported to the Home Office. This was such an exceptional event that the Home Secretary appointed Professor Brian Caddy of Strathclyde University to make an independent assessment of the matter. Professor Caddy reported his findings and conclusions later that year (8). Professor Caddy included a number of recommendations for improvement of the trace laboratory regime, all of which have been acted upon.

Maintenance of Laboratory Air Filtration Units

In June 1996, approximately 120 ng of RDX was detected on the instrument bench. An investigation revealed that failure of one of the air filters, leading to a release of accumulated contamination, was the likely cause. Since that time the filters have been subject to a more rigorous planned maintenance program, and additional laboratory monitoring samples are taken at the time of maintenance.

Conclusions

A system of contamination prevention procedures incorporating both inner and outer protective measures has been implemented, with progressive improvements, for about eight years. Over this time, monitor samples taken weekly from surfaces within the laboratory have, with few exceptions, revealed only low levels of contamination, predominantly of RDX. Analysis of 401 control swabs, processed alongside the monitor swabs, has demonstrated that in this environment the risk of forensic sample contamination is extremely small. The monitoring regime has also been valuable in a process of continuous improvement, allowing sources of contamination transfer into the laboratory to be identified and eliminated.

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