ANNUAL REPORT

1974

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APPENDICES

Note: The references cited in the text of this report will be found in Appendix C.

ANNUAL REPORT 1974

1. INTRODUCTION

Due to printing difficulties the publication of the 1973 Annual Report was delayed, but this year it will be available for distribution by the end of 1974. The Report covers the work of the Central Research Establishment (CRE) from October 1973 to October 1974.

The Establishment's work is designed to increase the operational efficiency of the regional forensic science laboratories and it is therefore pertinent to draw the reader's attention at an early stage to what has been achieved in the year under review. Clearly it is impossible to do other here than pick out a few of the features from an intensive on-going research programme.

Analyses for alcohol in blood taken under the provisions of the Road Traffic Act form a major part of the case load of the regional forensic science laboratories. The Establishment has been involved in recent years in research into trying to reduce the time involved in doing these analyses without reducing the analyst's involvement and responsibility for obtaining an accurate and precise result. The fact that this has very largely been accomplished may be judged by a recent comment from the Director of a regional laboratory that his analysts only complaints now were that they could not keep up with the analytical rate - the speed of physically handling bottles, writing and checking results is now the major constraint on the system.

In the biological field results are beginning to appear which could have major time saving effects. Simple rapid methods for the determination of species are now in use and already the idea of doing several tests on the same sample at the same time is proving profitable. Automated devices for analysing large numbers of saliva, semen and blood samples are bringing accurate quantitation into general serology in forensic science and their use should result not only in time saving but also in increased reliability. The use of "antibody profiling" is another new promising concept in forensic science.

CRE has always had major interests in chemical analysis and as a result of research in the past few years the regional laboratories are now being equipped with organic mass spectrometers which should considerably shorten what up to now have been lengthy analyses, particularly in the drug field. The increasing use of computer and information data banks with the communication links to regional laboratories now being tested have occupied both the Chemistry and the Information Divisions during the last year. Continuing research on inorganic analyses are being conducted both at CRE and by external contracts.

Several operational services are provided by CRE for regional laboratories, a major one being radioimmunoassay. This technique is several thousand-fold more sensitive than the chemical techniques of only a few years ago and Drugs of Abuse Division is now investigating high pressure liquid chromatography with mass spectrometry in an effort to put this type of sensitivity within the range of regional laboratories.

CRE has very close links with the regional laboratories as any contractor must have with his customers. This year has seen these cemented by the formation of a Forensic Science Branch of the Home Office. In addition, a series of very successful Colloquia and attachments have meant that personal interchanges of information regularly occur. Special mention must be made of a week's attachment of senior chemists and biologists to CRE.

Finally, I would like to thank the Director of the Atomic Weapons Research Establishment and his staff for continuing to make us welcome at Aldermaston.

2. STAFF DETAILS

We welcome:

Dr R Ardrey, (HSO) from London University, King's College.

Dr M Davie, (SSO) from Wessex Regional Blood Transfusion Centre, Southampton.

Mrs M Dry, (CA) from ROF, Burghfield.

Dr S Fletcher, (HSO) from St Thomas's Hospital, London.

Mr D Gabb, (Laboratory Attendant) from MoD Police, Aldermaston.

Mrs A Golding, (Audio Typist) from ROF, Burghfield.

Miss T Holdstock, (ASO) from Harriet Costello School, Basingstoke.

Dr R Holleyhead, (SSO) from London University, Queen Mary College.

Mr C Howden, (SO) from Brooke Bond Liebig Research Centre, Reading.

Dr J Locke, (SSO) from Johnson Matthey & Co, London.

Mr D Loxley, (HSO) from Home Office Forensic Science Laboratory, Birmingham.

Miss M North, (EO) from Home Office Forensic Science Laboratory, Aldermaston.

Mr M Osselton, (HSO) from London University, Chelsea College.

Miss H Payne, (CA) from Newbury District Hospital Board.

Miss V Quarmby, (SO) from London University, Royal Holloway College.

Mrs P Ridout, (CO) from A & B Motors, Newbury.

Dr J Twibell, (SSO) from London University, Imperial College.

Mr D Werrett, (HSO) from University of Birmingham.

Departures:

Mrs A Brech, (SSO) left to have a baby.

Dr A Butterworth, (HSO) left to join the staff of the British Steel Corporation.

Mrs M Dry, (CA) left to join the staff of the Home Office Forensic Science Laboratory, Aldermaston.

Dr A Patterson (PSO) was appointed Staff Officer to the Controller of the Forensic Science Branch, Home Office, London.

Congratulations to:

Mrs A Brech on the birth of a daughter.

Dr J Drayton (née Worthington) on the occasion of her marriage.

We record the attachment of Mr Robin Barrett, from the Department of Science, Commonwealth Customs Laboratory, Melbourne, Australia; also the following Sandwich Course Students: Miss L Alexander, from Liverpool Polytechnic; Mr G Moore and Mr D Weaver from Nottingham College of Technology; Miss L Richards, from Surrey University; Mr D Fairhurst, from Hatfield Polytechnic and Miss C Rushton, Vacation Student, from Southampton University.

Retirements:

Mr C H Nicholson, our Mini-Bus Driver, retired in January.

Promotions:

Mrs A Brech to SSO

Dr A C Moffat to PSO

Mr P Owen to HSO

Mr E R Rutter to PSO

Mr K W Smalldon to PSO

Dr P J Twitchett to SSO

Mr G W Walker to SPSO and Deputy Director

Appointments and Qualifications:

Dr A C Moffat was designated as a Fellow of the Pharmaceutical Society.

Dr P Burdett gained his PhD from Leeds University.

Dr M D G Dabbs was admitted as a Corporate Member of the Institute of Electrical Engineers, and has been registered by the Council of Engineering Institutes as a Chartered Engineer.

Registered for Higher Degrees:

Mr P J Gomm, MPhil.

Mr P Owen, MPhil.

3. LECTURES GIVEN AND CONFERENCES ATTENDED BY STAFF; OVERSEAS VISITORS

Lectures given by Director and Staff

Dr Curry chaired the Ciba Symposium on "The Poisoned Patient - the Role of the Laboratory". He also lectured to the BEA/BOAC Joint Medical Service, Medical Society; the Institute of Petroleum Conference on Recent Analytical Developments in the Petroleum Industry; the Society of Analytical Chemistry Centenary Celebrations; the 87th MoD Weekend Course at the Royal Military College of Science, Shrivenham; the 6th International Conference on Alcohol, Drugs and Traffic Safety, Toronto, and the 10th International Symposium on Chromatography at Barcelona.

Members of staff have given lectures to the following bodies: Wakefield Detective Training School; the Society of Analytical Chemistry; the International Association of Forensic Toxicologists 1974 European Meeting; the University of Surrey; Glasgow University Dental School; the Scottish Police College and the Joint Meeting of the Forensic Science Society and Classification Society, Newcastle.

Conferences Attended by Staff

Mr G W Walker attended a Conference on the Science of Fingerprints in London and also the Applied Research Laboratories Ltd Analytical Symposium in York; Mr D J Nicholson attended the Electronics Conference at Brighton; Mr K W Smalldon and Mr V J Emerson attended a Home Office Statistics Course; Dr J V Drayton attended "Workshop in Gas Chromatography - Mass Spectrometry" at the Royal Postgraduate Medical School, Hammersmith; "Mass Spectrometry - Advances in Chemistry Series" at the University of Manchester Institute of Science and Technology; 7th Meeting of the British Mass Spectroscopy Group at the University of Warwick and also a Seminar on the "Mass Spectral Search System" with Mr C Brown at Honeywell Ltd; Dr P Burdett attended a course on "Gel Filtration and Electrophoresis" at Loughborough University of Technology; a Symposium on "Isoenzymes" by the Royal Microscopical Society at Southampton; Dr A E Kipps and Dr P H Whitehead attended a Symposium on "Molecular Variants in Disease" at the Royal College of Physicians; Dr L A King attended the "Micro '74" Symposium, London; Mr J G Sutton and Dr P H Whitehead attended an "LKB Conference" in Glasgow and Mr J G Sutton attended an "Isoenzyme Symposium" at Southampton University; Dr P H Whitehead, Mr V J Emerson and Mr M Swain also attended the Forensic Science Society National Meeting at Bangor; Mr E R Rutter attended a Biochemical Society Symposium on "Glycoproteins" in London; Dr A C Moffat and Mr P Owen attended a "Monoamines and the Clinician" Conference at the Institute of Child Health London; Dr A C Moffat with Mr P J Gomm attended the Pharmaceutical Conference in Nottingham; and Dr P J Twitchett attended a "High Pressure Liquid Chromatography Conference" at Stockport.

Overseas Visitors

A total of 51 visitors from 14 different countries visited CRE during the year.

4. BIOLOGY DIVISION

Following the staff changes reported last year, the opportunity has been taken to reorientate the Division along the new lines of enquiry as predicted in last year's Annual Report.

The work of the Division has fallen into two main areas:

A. Fundamental Studies

In recent years, experience in the Home Office Forensic Science Laboratories and the Metropolitan Police Laboratory, has suggested that our knowledge of the fundamental properties of blood group substances and red cell enzymes as they occur in body secretions is far from complete. Even the traditional distinction between "secretors" and "non-secretors" in the ABO system requires re-evaluation and little knowledge is available regarding the concentrations of blood group substances and red cell enzymes in saliva and semen either within, or between, individuals. Hence considerable effort is being devoted to this field of study. Apart from the results reported below, it is believed that the quantitative analysis of ABO blood group substances (by instrumentation) and enzyme variants in saliva and semen, will lead to a fuller appreciation of the problems of grouping secretion stains.

B. Work Simplification

At present our increasing ability to characterise blood, either using serological or biochemical means, is achieved by using additional reagents, anti-sera, apparatus, blood for examination, and of course, staff time and effort. While technically (finance permitting) one could investigate a bloodstain using numerous systems each with its own peculiar resource requirements, the blood available for study will always be limited by the circumstances of the crime, and the time available will be dependant on police requirements.

Hence, it is believed that there is a need for simplification of the means of characterising blood in order to save both time and materials - in particular the blood required for analysis.

A. Fundamental Studies

(i) Semen Enzymes

Much interest has been focussed in recent years on the possibility of using enzymes of the glycolytic cycle to type this body secretion in the same manner in which they are used to group blood.

One particular enzyme which has been used in this respect is phosphoglucomutase, (PGM) which has a number of genetic variants easily resolved by starch-gel electrophoresis. A study of the PGM variants in liquid semen samples from 200 normal and vasectomized men showed that 3% of them had "anomalous" patterns which could not be

equated with any known PGM phenotype. In addition 6% of them had very low PGM levels and were not detected by starch-gel electrophoresis[19]. Following up these observations a series of experiments was initiated to study the stability of PGM iso-enzymes in liquid semen and seminal stains. The results suggest two types of change may take place.

(a) An overall loss of enzyme activity in all iso-enzymes which may lead to difficulties in differentiating between types $PGM_1 2$ and $PGM_1 2\text{-}1$. The reasons for these changes are not yet clear but they are associated with bacterial contamination[27].

(b) A change in the mobility of the slowest iso-enzyme bands which results in a pattern quite different from any known phenotype[30]. As this change can be reversed by the addition of mercaptoethanol and semen is known to contain high levels of glutathione (GSH), we have postulated (following Hopkinson and Harris, 1969), a PGM-glutathione complex reversible by the addition of mercaptoethanol.

$$PGM - S - S - G + HS - C_2H_4OH \rightarrow PGM - SH + G - S - S - C_2H_4OH$$

It is anticipated that further work will involve a study of glutathione levels in semen and correlation with PGM genotypes both "normal" and "anomalous".

The work on PGM has emphasised how little is known of the properties of the enzyme in semen. One can only speculate on what new properties may be found when the environment is made even more complex by mixing the semen with vaginal secretion - as is often the case in crime work. Further studies will be pursued in this direction.

(ii) Saliva Enzymes

At the present time the extent of examination of saliva stains is restricted to identification, localisation and ABO grouping. The enzyme usually associated with saliva, ie, amylase, is involved in each of these steps as an aid to identification and localisation, and moreover may be taken as a measure of the quantity of saliva prior to ABO grouping. Studies have been made to improve the present means of identifying saliva stains and to question the fundamental association at present assumed to exist between amylase and ABO activity in saliva.

An amylase sensitive paper has been developed by precipitating onto filter paper a dye-amylopectin substrate which is susceptible to amylase. The substrate is pink and the presence of amylase action is indicated by a white area. The test paper is used in practice by laying the paper on top of a garment, moistening and incubating for 10 minutes at room temperature. Any saliva stains will be outlined in white on a pink background[10,79].

Although the paper was developed specifically for Forensic Science use the potential clinical use of this paper as a means of assaying levels of serum amylase has been recognized and a patent has been filed. Levels of amylase have been determined in a wide range of body fluids using the amylopectin substrate either on the paper or incorporated into agar and surprisingly high levels have been found in semen and sweat emphasising that amylase itself can only be taken as a guide to the presence of saliva[2,13,77]. Also, in lip-mucus secretion, a component of saliva, low levels of amylase were found[17]. It has been previously reported that high levels of blood group substances were present in lip-mucus and hence our results illustrate that low amylase levels may be associated with high levels of blood group substances. In practical terms it means that a stain with a low amylase activity may still have adequate saliva present to enable grouping to be done.

Amylase is potentially also an attractive genetic marker as polymorphic forms have been described following electrophoresis in various media. Using isoelectric focussing we have fractionated salivary amylase into at least 9 components. However, the genetic basis has yet to be established. Quantitative assay of salivary amylase using an automated assay on a Technicon Auto-Analyser suggests that the concentration of amylase itself may be constant within limits for a given individual over a period of months, (Fig 1).

(iii) Automated Studies

As was mentioned in last years report, the quantitation of blood grouping techniques which will result from automated systems of analysis is believed to hold the key to further investigation of quantitative differences that may exist between the levels of blood-group substances in body secretions either within or between individuals. Instruments are being developed "in house" and by External Contract. It is anticipated that results in this field will be forthcoming over the next 12 months.

B. Work Simplification of Techniques for Examining Blood

(i) Use of Latex

The species identification of blood found at a scene of crime is fundamental to further grouping studies. At present traditional immunochemical techniques include tube precipitation and cross-over electrophoresis. Alternative techniques of visualising antigen/antibody reactions include those dependant on latex particles coated with either antibody or antigen. In collaboration with the Wellcome Research Laboratories a range was prepared of latex preparations coated with antibodies directed against different animal species, eg cat, dog, horse, etc and these were evaluated for specificity and sensitivity at HOCRE[3,66]. In addition a blind trial was organised in collaboration with the Home Office Forensic Science Laboratories during which 10 stains were submitted "blind" to CRE for species identification using latex reagents.

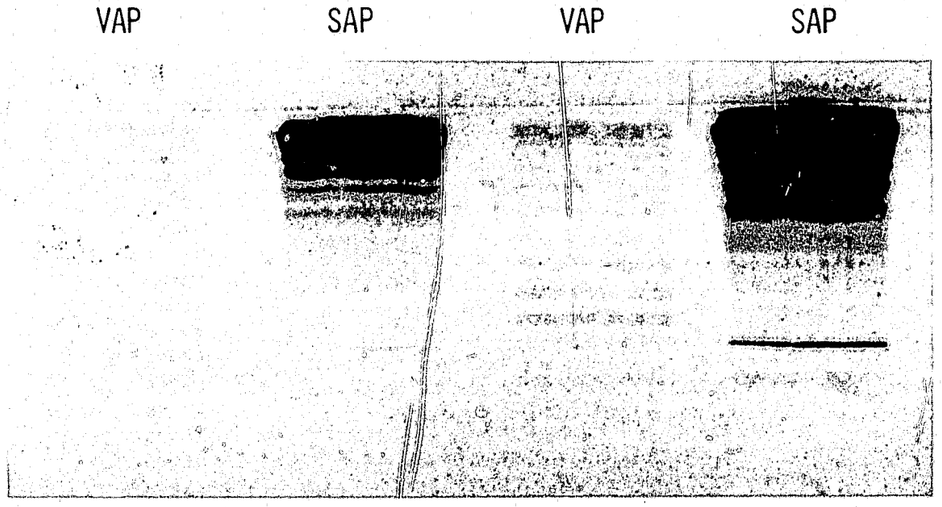


Fig. 1 Isoelectric Focussing of Semen (SAP) and Vaginal (VAP) Acid Phosphatase on Polyacrylamide using a pH 5 to 7 gradient.

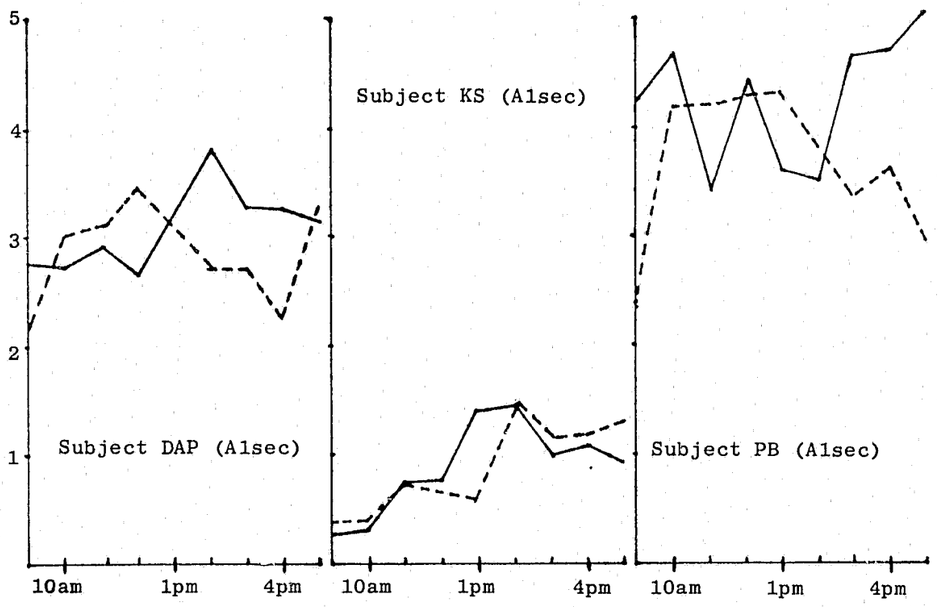


Fig. 2 Diurnal Variation of Amylase/Protein at Different Times of Year.

(The dashed lines represent amylase/protein ratios of salivas collected in February 1974 and the continuous lines those of saliva collected in July 1974.)

The result of these investigations was to show that the latex reagents for species identification were faster (taking only about 3 minutes), as sensitive and specific and at least as reliable as traditional techniques. This technique will enable the forensic scientist to give an answer to the question "Is this human blood?" immediately, at the scene of crime if necessary. Work is continuing on latex reagents with a view to using them ultimately for ABO grouping of stains.

One small, but potentially, important observation was that it is possible to group a stain for ABO grouping on the same fragment of blood following species identification using latex[6,56]. It is believed that this is made possible by the soluble nature of serum proteins (to react with latex) as distinct from the insoluble nature of some ABO blood group substances (necessary for the absorption elution technique of ABO typing).

(ii) Isoelectric Focussing

At the present time it is customary, when typing a bloodstain using genetic variants of red cell enzymes, to utilise electrophoresis at a given pH in a suitable medium, usually starch, followed by specific staining for a particular enzyme. As the number of red cell enzyme systems grow in number so it becomes progressively technically more difficult to accomodate in a busy laboratory, many different electrophoretic systems. In an effort to overcome this problem we have turned to iso-electric focussing in a flat bed polyacrylamide gel. This system is dependant on the formation of a stable pH gradient across the gel from anode to cathode. Proteins, under an applied potential, migrate through the gel until they reach their iso-electric point. The system achieves resolution of pro ein mixtures into a fine number of discrete bands.

It has been possible by using our particular pH range across a plate, to separate simultaneously the genetic variants of Hb, PGM and EAP from liquid blood, ie typing of blood in three systems has been achieved following one electrophoretic run. Work is in progress to extend the number of systems[26].

In addition we believe the technique of iso-electric focussing may prove valuable in studying reported genetic variants of seminal acid phosphatase. (See Fig 2.)

(iii) Stability of Enzyme Reagents

A time consuming feature of red-cell enzyme typing is that following electrophoresis, a complex mixture of biochemicals must be prepared in order to 'develop' the plate for a given enzyme. For example, the mixture for developing the PGM isoenzymes consists of:

Glucose-1-phosphate (30mg) including 1-6 diphosphate;
$MgCl_2$ $6H_2O$ (20mg); MTT tetrazolium (2mg);
Phenazine methosulphate (2mg);
Glucose-6-phosphate dehydrogenase (0.4IU); NADP (1mg);
dissolved in 10ml of 0.06M tris/HCl buffer pH 5.0.

It has been found possible to mix all these reagents, with the exception of the tris/HCl buffer components, in their dry form en masse, and then dispense into capsules (Eli Lilly drug capsules) such that one capsule contains sufficient material for 10ml of buffer. The mixtures were found to be stable at -20°C for at least 6 months. The capsules now provide an 'instant' developing mixture for PGM and save considerable time and effort for the scientist. Obviously, the principle could be extended to other enzyme mixtures[33].

(iv) Antibody Profiling

The present means of human bloodstain characterisation are all based on genetic factors associated with blood. However, in some instances other 'non-genetic' factors may provide information. For example, the presence of salicylate in a bloodstain extract may provide a discriminating feature, and the detection of therapeutic levels of aspirin in bloodstains (25µl blood) has been achieved by fluorimetric assay of an ethanol extract of the stain, (HOCRE Report 1973).

In contrast, a new means of discriminating blood has been investigated which is dependant on characterising antibodies in stains. In addition to offering a new approach to blood characterisation it may be carried out on very small bloodstains and using conventional microscopic techniques.

When an individual is infected with a parasitic organism, antigenic material from the parasite stimulates the immune system of the host to produce specific immunoglobulins which combine with the antigen. It is a feature of the host's immune system that specific antibodies may be found for many years following an original contact with an antigen. Using an indirect fluorescent antibody technique it is possible to detect a wide range of antibodies in bloodstains rapidly on a microscope slide[4,54].

On screening bloodstains from 74 adults for just 5 types of antibody (directed against M.tuberculosis; T.pallidum; T.vaginalis; M. albicans; V.cholera) a Discriminating Power of 0.71 was achieved which compares with a DP = 0.66 for the ABO system. Later work has shown that the blood from infants and adults may be readily distinguished using this approach[25]. In addition as a person's 'antibody profile' may be expected to reflect his health history over his lifetime, it may be possible to gain further information relating to his life style and country of origin, by simply extending the range of antigens studied.

C. Further Studies

One area which has not received attention in the Biology Division for the past year has been that of hair discrimination. The work of Dr A P Phillips (HOCRE Report 1973) showed the possibility of sexing hairs by studying the presence of Y chromosomes in human hair sheath cells. Pending staff availability this is an area which will be developed and extended over the coming year.

It should be recorded that the Biology Division has enjoyed the liaison established with the Body Fluids Committee and has been happy to assist on a number of projects initiated by them. Also it is a pleasure to record the Division's appreciation of comments, criticisms and suggestions made over the past year by biologists throughout the service.

Ref:

Hopkinson, D A and Harris, H, Ann Hum Genet, Lon, (1969), 33, 81.

5. CHEMISTRY DIVISION

The division has undergone considerable staff changes during the year and has been without a head of department for a major part of it. Some disruption of the research programme has resulted and therefore the opportunity has been taken to reorganise the division into three research groups. The progress of projects during the year and planned developments are detailed below.

A. Mass Spectrometry

(i) Organic

The Micromass 12 low resolution mass spectrometer has been fitted with a multiple ion monitor during the year and the Watson-Biemann separator for GC/MS has been replaced with a jet separator. Preliminary work with a Carrick computer interface has been completed and a cable link has now been installed so that normalised spectra can be printed from a visual display unit and unknown spectra automatically matched against a reference collection.

The emphasis during the year has continued to be placed on the identification of drugs and drug metabolites. A data collection which is specifically relevant to casework is being constructed for use in the forensic science service. Data, compiled from a number of external sources, is being complemented by spectra produced on the Micromass 12 at CRE.

A procedure for the rigorous identification of amphetamine using an isothiocyanate derivative has been developed during the year[11] and a casework evaluation of the MM12, conducted in conjunction with the Home Office Forensic Science Laboratory, Aldermaston, has been reported[14].

A service facility continues to be offered to both regional laboratories and the divisions of CRE. During the year 180 cases have been completed for regional laboratories. The detection of fluoroacetamide and fluoroacetic acid in tissues and body fluids, using gas chromatography followed by FCH_2 single ion monitoring, and the efficiency of drug removal from various TLC plates has been studied in conjunction with Toxicology Division. Various psychotropic drugs, which were separated using HPLC by Drugs of Abuse Division, have also been identified by mass spectrometry.

It is hoped that during the next year the instrument will be converted to allow chemical as well as electron impact ionisation. This will facilitate the examination of mixtures and those compounds which produce only weak molecular ions using electron ionisation and cannot be specifically identified from their fragmentation patterns.

(ii) Inorganic

The electrical detection accessory to the mass spectrometer has received further attention this year. The peak switching mode, which has already been linked to the Hewlett-Packard computer, still presents instability problems and these are under investigation with the manufacturers. Although the analytical results are more precise than those derived from the photoplate detection method, the electronic drift causes problems especially with small samples. The peak scanning mode provides a very fast method of analysis and is particularly useful in the trace element analysis of a large number of similar samples. The interface to the computer is now installed and only a little further programming is required before the output from peak scanning can be handled automatically.

The method previously developed for the quantitative trace element analysis of glass required a sample of at least 1mg[54]. However, experience has shown that glass fragments found on clothing can be as small as 100µg. A method is therefore under development which can analyse these small fragments. The problems still to be investigated include loss of precision and the possibility of interferences to certain elements caused by the large increase in the graphite content of the electrodes.

A project to develop a multi-element screening procedure for liver samples in cases of suspicious death has recently started. A low temperature asher, which uses atomic oxygen as oxidant at low pressure and low temperature, is being used to ash samples in order to minimise both contamination and losses of volatile elements during the ashing procedure. The low temperature asher is on loan from the Home Office Forensic Science Laboratory, Harrogate. The initial work is being concentrated on electrode preparation, sparking parameters and the identification of any molecular interferences so that only true elemental lines are measured.

The spark source mass spectrometer has also been used during the year to give assistance to regional laboratories in the analysis of glass fragments (3 cases), small metal fragments (1 case) and the analysis of toxic elements in tissue samples (3 cases).

B. Glass, Paint and Fibrous Materials

It is anticipated that a continuing research effort will be required for these evidential materials because they make up the bulk of the work in the chemistry sections of forensic science laboratories and are continuously undergoing industrial product development. A research group will therefore concentrate on the examination of these materials.

(i) Glass

Previous work has shown that "innocent" items of clothing may occasionally contain glass fragments which compare in physical properties with a control sample of window glass. It is likely that most of these "innocent" fragments do not originate from flat glass. If this is so then previous experience using inorganic mass spectrometry[54,58], has shown that trace element analysis would be an effective approach to such discrimination problems. Although forensic science laboratories are unlikely to have inorganic mass spectrometers available in the near future they do have access to other trace element techniques and for this reason a programme has been initiated to develop methods for the trace element analysis of glass using the technique of flameless atomic absorption. This work is still in the early stages but it is hoped that it will be possible to quantitate a number of useful elements for typical glass fragments received in casework. Other methods are also under consideration including emission spectrographic methods with various excitation sources. The microdensitometer is at present being modified so that rapid computer processing of photoplates can be achieved.

(ii) Paint

A limited amount of work has been done on this subject during the last year. The modified fibre optics colorimeter, which was developed specifically for CRE by the Paint Research Association, is at present being evaluated in Information Division.

The Laser Microspectral Analyser has shown considerable promise as a reproducible emission method for the analysis of paint flakes[67] and more especially for the analysis of paint smears, which presents difficulties using other techniques. In the case of smears the laser beam is de-focused so that only the smear is vapourised from the surface for emission analysis. It is hoped to evaluate the instrument fully for paint smear examination in the coming year. Also under consideration are further methods of identifying paint resins and the improved characterisation of intact multi-layer samples, with special reference to multi-layer white samples.

(iii) Fibrous Materials

The collection of data concerning the tracers and catalysts found in synthetic fibres has continued during the year although primarily from literature sources. A survey of the materials used in the processing of textiles has also been obtained by means of an external contract. Examination of tracers and catalysts would seem to present the only solution to the discrimination of synthetic fibres which are both physically and organically similar. The analytical problems with small samples of fibre seem formidable but if suitable background data can be obtained the approach could be used for more specific identification when larger samples are received in casework.

In the future it is hoped that the discrimination offered by various methods of dyestuff comparison will be determined. This project arose from an interlaboratory trial conducted by the Fibre Study Group and was suggested by this committee as a worthwhile research area.

Despite previous work in trace element analysis and in other fields the comparison of human head hairs continues to provide major problems. Although it is accepted that the probability of success is not high it is intended in the coming year to examine the profiles of elements, which are thought to originate mainly from diet, along single hairs.

(iv) Casework

During the year the atomic absorption instruments have been used to assist regional laboratories with the quantitative analysis of samples for particular toxic metals (3 cases) and the Laser Microspectral Analyser has been used in 10 cases involving paint and metal smears.

C. Other Evidential Materials

If glass, paint and fibres are excluded, a considerable number of different materials remain which are of interest to the forensic chemist and these provide a wide variety of problems. A research group will concentrate on this particular area. At the present time the group is concerned with the detection of organic gunshot residues on hands and the examination of soil, but it is anticipated that the materials of interest in this group will periodically change.

(i) Organic Gunshot Residues

Inorganic residues, such as lead, barium and antimony, may be detected on hands after a weapon has been fired but they may also contaminate the hands after a variety of other activities. If organic residues could be detected on hands then these would undoubtedly prove more specific.

The initiator or cap composition of a modern catridge is of the Van Hertz type, based on tetrazene and lead styphnate with an excess of barium nitrate. Appreciable amounts of the organic matter from such an oxidising system are unlikely to survive and therefore the major residues found after firing are likely to be traces of the propellant ingredients. All small arms propellants are based on nitrocellulose which may also contain plasticisers. Double based propellants use nitroglycerine as the plasticiser and contain diphenylamine, or one of its derivatives, as a stabiliser. About 1% of methyl or, more often, ethyl centrallite is added to the finished propellant granules as a burning modifier.

Preliminary work has begun to determine if organic residues can be detected on hands after a firing. Although traces of all the propellant ingredients have been found on penetrated cloth, only nitrocellulose has been found on hand swabs. Development of more sensitive methods is necessary for the remaining ingredients and a method is also required which distinguishes the nitrocellulose used in propellants from the less nitrated materials used in most nail varnishes and touch-up paints.

(ii) Soil

Both new and existing techniques for the characterisation of soils have been investigated during the last year. The emphasis has been mainly placed on fundamental aspects such as the comparison and recording of soil colour.

The Munsell Soil Colour Charts and the Methuen Handbook of Colour have been compared for the collection of colour data for soil populations[1]. Such compilations of data would enable the value of a particular colour match in casework samples to be assessed. The Munsell Soil Colour Charts were found to be the superior system and were recommended for all further work on soil colour. The use of moist and ashed soil colour was also examined[15] and the results indicated that, although moist colour yielded some discrimination, ashing at 850°C for 30 minutes was by far the superior additional technique and was recommended for routine use. The construction of apparatus for the examination of the cathodoluminescence of soils is now complete and an evaluation of the technique is in progress. A wide range of luminescence samples were found in a reference collection of minerals. Cathodoluminescence emission spectra of whole soils have been recorded at the University of Manchester Institute of Science and Technology on our behalf, but the technique was found to be of little value because the spectra were dominated by emissions from the common major minerals.

A manufacturer of thermal equipment has examined a number of soil samples supplied by CRE. Thermogravimetry and derivative thermogravimetry did not yield very good discrimination, although the latter technique was marginally superior. Differential thermal analysis results are in preparation.

Particle size distribution will be investigated shortly using a Coulter Counter Model ZB.

Project development in the coming year will also involve the examination of soil organic matter, soil pH, a reappraisal of density gradient columns and the application of suitably proved techniques to the examination of soils in simulated cases.

D. Cases Involving Analysis of Arsenic in Hair

During the past year sectional analysis for arsenic in hair has been performed in 5 cases where it was suspected that there had been the injection of an arsenical poison.

The one positive case encountered involved a possible exposure to arsine. The patient in question was admitted to hospital with a sudden onset of vomiting, severe systemic upset and acute renal failure. On contacting the metal company where he worked it was suspected that he might be suffering from metal poisoning although the subsequent analysis of his blood showed only normal levels. To investigate further the possibility of arsenic poisoning a sample of hair was submitted to CRE for analysis.

The hair was cut into eight, one centimetre shaft sections and a root section of 2mm in length. Each section was then subjected to neutron activation analysis. The results showed that there were 66ppm of arsenic in the root section with 4ppm in the first centimetre and less than 1ppm in the remaining seven distal sections. This analysis confirmed the suspicion of arsine poisoning and indicated a recent exposure which agreed well with the clinical details.

When admitted to hospital the patient was sweating profusely and it is significant to note that arsenic was not deposited at distal portions of the hair as might be expected if sweat contained appreciable amounts of arsenic. It is hoped that samples of hair will be obtained regularly over the next year from the individual so that the movement of the arsenic with growth can be studied.

6. DRUGS OF ABUSE DIVISION

A. Drugs Intelligence

The collection of information from the 8 regional forensic science laboratories, 4 police laboratories, the Department of Industrial and Forensic Science at Belfast, and the Laboratory of the Government Chemist has been actively continued throughout the year. As in previous years in addition to the collection and dissemination of intelligence information, analytical services have been provided in order to examine illicit preparations of interest in greater detail. This work has been carried out with the following main objectives:

(i) To identify quickly the appearance of a new drug preparation.

(ii) To recognise similar preparations which have a common origin.

(iii) To investigate in depth any drug preparation which appears to have national significance.

Since the inception of the Central Drugs Intelligence Unit in March 1973 the close liaison which was quickly established has continued throughout the year to give an excellent working relationship. The contact with other Drug Abuse Agencies outside the United Kingdom, predominantly USA and Australia, has increased and the exchange of samples of current illicit preparations is now underway.

The intelligence information and results of the scientific examinations which have been made available to the Central Drugs Intelligence Unit during the year have been shown to assist in answering some of the questions which are posed during police enquiries into illicit drug traffic. For example such questions as:

(i) Is the source of manufacture within the UK or from a specific country abroad?

(ii) Is this the first seizure of its kind found in quantity in the UK?

(iii) Is the information gained from informants with regard to possible manufacture correct?

The number of Drug Abuse Trends Bulletins issued is now 10 and this year in addition to the intelligence background information, where possible, analytical details on newly encountered drugs have been included in order to assist the laboratories. The work of monitoring trends in drug abuse has resulted in the notification of 637 significant cases from which 247 samples have been received for further scientific examination. Cases involving cannabis plant and resin were excluded. An in-depth study was made into "Drinamyl type" amphetamine and amylobarbitone tablets which were seen in large quantities throughout the UK until the beginning of 1974. The results of this examination were reported in detail as an appendix to Drug Abuse Trends No 6. It was possible by monitoring the intelligence information from Customs seizures and regional laboratory cases to give an early warning of the abuse of two significant new trends

involving Hash Oil and Cocaine as early as six months before these appeared in quantity in the country. These two substances, Hash Oil and Cocaine, represent 12% and 10% respectively of the total number of cases notified this year, the main illicit drugs encountered being LSD 40% and amphetamine 20%. The total number of new tablet preparations appearing for the first time this year is 29 and two newly encountered drugs which have been shown to have national distribution are 2-5-Dimethoxy-4-Bromo-Amphetamine and Benactyzine.

The section was able to provide assistance to the regional laboratories in the initial cases involving 2-5-Dimethoxy-4-Bromo-Amphetamine by confirmatory analysis using the facilities of the Organic Mass Spectrometer at CRE. The compound is dispensed as spots on blotting paper.

As a result of the work carried out by the section and the recommendations of the Working Group on the Application of Scientific Aids to the Detection of Drug Offences a separate operational Drug Intelligence Laboratory is to be formed at CRE. The main objectives are to continue the work of the section and exploit the possibilities indicated during the past three years by the operational research programme.

B. High Pressure Liquid Chromatography

In December, 1973, a Water's Associates Liquid Chromatograph equipped with 6000 psi pumps and a gradient elution accessory was installed at CRE. A variable wavelength ultra-violet monitor has since been coupled to the instrument, and fluorometric detection is also currently being investigated. Although research projects to date have mainly comprised specific separation problems[22,23], a more general assessment of the value of HPLC in toxicological analysis is now in progress.

(i) Illicit Drug Preparations

HPLC has been used in attempts to compare and determine the origin of illicit drug seizures. Traces of chemical precursors, for example, may suggest the synthetic route by which the drug was made, while examination of the diluents and other components present may indicate the geographical origin of an illicit mixture.

Unfortunately, many of the LSD and cocaine samples analysed in this way have been notable only for their purity and so far traces of synthetic starting materials have not been positively identified. It is well-established, however, that illicit diamorphine preparations may contain a complex mixture of alkaloids, the composition of which varies according to the origin of the sample. In the United Kingdom, the diamorphine content is 30 - 60%, and the major diluent in Chinese Heroin is caffeine, whereas in America the diamorphine content is lower (5 - 10%) with a greater variety of diluents (quinine, procaine, etc). A HPLC separation has been devised which allows the qualitative and quantitative analysis of the alkaloids present in such mixtures to be performed in eleven minutes. Figure 4 illustrates the separation of a reference mixture of eight of the drugs and degradation products which have been found in illicit diamorphine seizures.

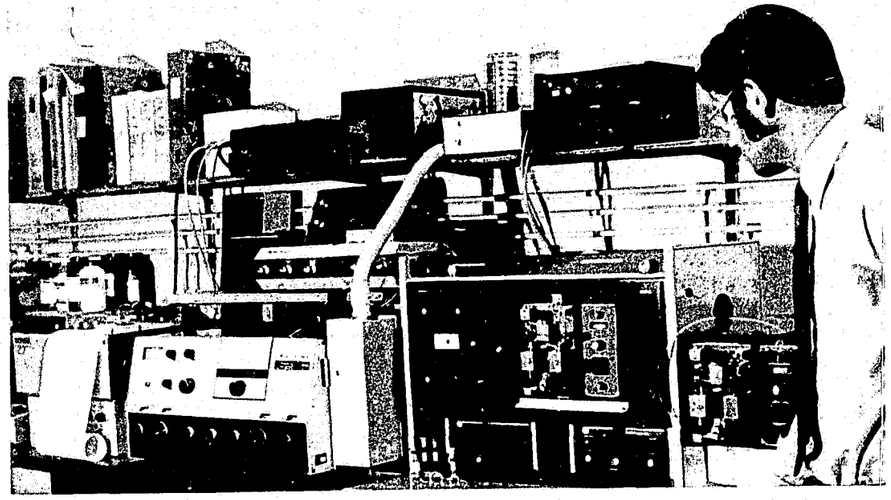
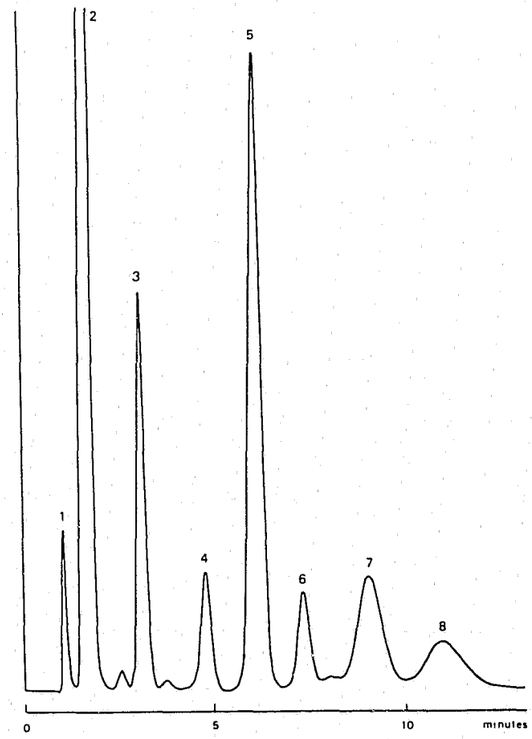


Fig. 3 The High Pressure Liquid Chromatograph in use in the Drugs Of Abuse Division.



Peaks

1. Barbitone
2. Caffeine
3. Morphine
4. Monoacetylmorphine
5. Strychnine
6. Diamorphine
7. Quinine
8. Cocaine

Fig. 4 High Pressure Liquid Chromatography of some Constituents of Illicit Diamorphine Seizures.

The method exemplifies the use and potential of HPLC in the separation and estimation of basic drugs. Not only are retention volumes highly reproducible (coefficient of variation for diamorphine retention : 1.2%) but the quantitative accuracy and reproducibility are also excellent (coefficient of variation for replicate diamorphine analysis : 2.3%)[22,23].

(ii) Drug Metabolites in Body Fluids

Attention has also been focussed on the detection of 11-hydroxy-tetrahydrocannabinol, which is present in the urine of cannabis smokers. It has been shown that mass spectrometry of 11-OH-THC requires 30ng of material applied to the probe, but although this quantity may be detected by HPLC, confirmation of identity at this level has not been possible because of the problems of handling and recovery of these very small quantities.

It is anticipated that the use of a new microparticulate column will enable greater resolution and sensitivity to be achieved and this may solve many of the problems associated with the handling of nanogram amounts of drugs.

C. Alcohol

A preliminary assessment of the FBG Trident blood vial which is being used by the police from October 1974 to contain blood samples taken for alcohol determinations under the Road Traffic Act 1972 has been made.

A new column packing material suitable for the GLC analysis of blood samples for alcohol has recently become available. The material is Carbopack A coated with 0.4% Carbowax 1500. Its properties are essentially those of the widely used Poropak Q in so far as the order of elution of low molecular weight solvents is practically identical on both of the columns. However, the Carbopack A column has far higher resolution and the analysis time can be reduced to 1 minute using this column. Two regional laboratories have been supplied with this material and so far the results are extremely promising.

7. EXTERNAL CONTRACTS DIVISION

The past year is the first full year that Contracts Division has existed as a separate division. The staff of the division was increased during the year so that closer supervision could be maintained on current contracts and a concerted effort could be put into the field of blood alcohol analysis. In addition it has been possible to expand the activities of the division to include some experimental work.

A. Completed Contracts

Five contracts have been completed during the year at the time of writing and two more are due for completion in October.

As a result of these contracts we have received reports on information on "Materials used in the Processing of Textiles" and "The Birefringence of Fibres". The former is a compilation of information on the many varied treatments that fibres may be subjected to during the course of the manufacturing processes. The second report was an investigation into the possibility of using the dispersion of birefringence as an aid in the discrimination of fibres. Unfortunately it appears that the technique will be of little value in Forensic Science. Both reports have been circulated to the regional Forensic Science Laboratories.

Photographs of a large number of softwoods available in this country prepared by The Building Research Establishment have now been delivered and, together with a collection of hardwood photographs collected a few years ago, form a library of wood species likely to be encountered in the UK. Information Division is currently examining the best way of circulating the softwood photographs to the regional Laboratories.

The examination of hair has long been a problem in Forensic Science and a contract has been completed during the year to provide additional background knowledge on the distribution of trace elements in hair. A technique known as Proton Induced X-Ray Analysis (PIXA) was used under contract by the Atomic Energy Research Establishment (Harwell) to examine the cross-sections of a number of different hairs. It was found that sulphur was distributed fairly evenly throughout the hair and a similar pattern was recorded for zinc. The surface/centre concentration ratio's for other elements ranged from 1 to 5 except for iron where it was as high as 11. In one case the copper concentration on the surface of a girls hair was 26 times that towards the centre; presumably due to a copper containing hair cosmetic. The surface/centre ratios for Arsenic ranged from 2.0 to 3.7 and for lead from 2.8 to 5.1 which shows that most of these elements are concentrated near to the surface of the hair. Such information will be invaluable when considering cleaning procedures to apply to strands of hair prior to analysis.

A device to assist in the evaporation of solvents without allowing them to boil dry has been developed under contract. The liquid to be evaporated may be in a beaker, flask or any other suitable container which gives it an advantage over some commercial level sensing devices. The container is placed on a hotplate or water bath and the probe placed in the liquid so that the tip is positioned at the point to which the liquid must be evaporated. As soon as the liquid has evaporated to a level where the tip of the probe is exposed an alarm will sound and, if required, the equipment will isolate the hotplate or water bath from the mains supply. The equipment functions satisfactorily for solvents boiling between 35° and 100°C. The equipment is completely solid state to eliminate any chance of fire caused by arcing contacts, and is capable of switching up to 3kW AC. The equipment is currently being assessed in the Nottingham Regional Laboratory.

Two further contracts are due for completion at about the time of writing. One is an updating of the library of pyrograms from fibres and the second is an automatic saliva serological grouping apparatus based on the Technicon system.

Three further contracts should have been completed but have been delayed for a variety of reasons. The contracts are for the production of a modified Perkin Elmer F40 gas chromatograph which will hold 200 samples, an apparatus for the automatic extraction of poisons from urine samples and an automatic saliva grouping machine designed specifically for small numbers of samples in a batch process.

B. Current Contracts

Only three new contracts have been awarded during the year because of a cut-back in expenditure. However, work has been initiated in the prevention of blood clotting in samples taken under the 1972 Road Traffic Act.

There are, currently 16 contracts being actively pursued plus two further ones which, although completed, require some modifications. Of the 16 contracts, 3 involve the collection of data, 4 have a relevance to toxicology, 3 to biology, 3 to chemistry and 3 are associated with various aspects of blood alcohol analysis. The three data collection contracts consist of two collections of pyrograms for rubber and fibres and a collection of tyre tread patterns from tyres currently in use in the UK. This latter contract also involves the development of a coding system so that tyres with certain tread characteristics may be retrieved from the collection. This coding system has proved to be somewhat difficult to devise and considerable effort has been put in by both the Contractor, the Tyre Committee and Contracts Division into devising a suitable system. The initial systems which were tried tended to be too complicated and difficult to use, but the latest coding system is much simpler and promises to have good discriminating power. The new system will shortly be circulated to tyre examiners in regional laboratories for their comments.

The four toxicological contracts cover very diverse fields including enzyme inhibition studies to find new ways of detecting drugs, and the development of techniques for the detection and estimation of therapeutic amounts of anti-histamines in blood. In addition under an external contract with a University the vast majority of all the compounds which fall within the restrictions of the Misuse of Drugs Act 1971 have been either collected or synthesised. The infra-red, ultra-violet and mass spectra of these compounds have been recorded together with TLC Rfs and have all been put onto a computer file. A program has been written which enables searches to be carried out on one or more of these characteristics. The contract should be completed and available for the Forensic Science Service towards the end of 1975. A further contract is the construction of an apparatus for the automatic extraction of drugs from urine. This equipment extracts drugs from 20ml aliquots of urine and collects the drugs in 5 fractions, strong acids, weak acids, neutrals, bases and morphine. Some problems have occurred with this contract largely due to long delivery delays on components, difficulties in obtaining suitable valves and difficulties in obtaining good extraction efficiences. However, much progress has been made in the last few months and delivery is expected at the end of the year.

Two of the three biological contracts are for the quantitative grouping of saliva samples. The first contract is for the construction of a continuous flow Technicon type apparatus designed for the quantitative serological grouping of a large number of samples. The normal speed of analysis is about 25 samples per hour, but this can be increased to 40 samples per hour with an accompanying loss in precision. The apparatus is ideally suited for the analysis of large numbers of samples of liquid saliva on a continuous basis and is intended for fundamental studies on saliva at CRE. The other contract is for an instrument with a much slower throughput and is a batch process. The rateof analysis will be about 6 - 8 per hour and the apparatus is ideally suited to the analysis of small numbers of samples. Finally there is a research contract with a University to investigate blood protein levels with a view to increasing the discrimination of blood stains and to establish which proteins are likely to be of most use in Forensic Science.

A contract to manufacture an Atomic Absorption spectrometer for the simultaneous analysis of up to 12 elements has now reached an advanced stage. It is hoped that this apparatus will combine the advantages of atomic absorption spectrometry with the simultaneous analysis capabilities normally associated with arc emission spectroscopy, mass spectrometry and neutron activation analysis. A Massman furnace is used to atomise the sample to give greater sensitivity over the more commonly used air/acetylene flame. No figures are yet available for the precision and sensitivity of the apparatus. A further contract involving the use of atomic absorption spectrometry is for the analysis of toxic metals in blood, urine and tissue. The contract is only in the early stages and no results are yet available. The third chemistry contract is for the construction of a fully engineered prototype refractometer for the measurement of the refractive index of small fragments of glass. A laboratory prototype has been constructed at CRE which proved that the basic concept was feasible but that closer attention was required to the mechanical engineering aspect.

A high degree of interest has been maintained in the problems associated with blood alcohol analysis which is a perennial problem in the regional Forensic Science Laboratories. The construction of the modified F40 gas chromatograph to take 200 samples is nearing completion and it is planned to make the equipment available to a regional laboratory for assessment towards the end of 1974. The gas chromatograph itself is virtually unmodified but the 30 sample turntable has been replaced by a continuous belt system which immerses the samples into a thermostatted water bath about 30 minutes before analysis. Each of the 200 positions are numbered and this number will printed on the recorder chart near to the propanol peak. The machine has been designed to operate unattended.

The problem of clotted blood samples occurs from time to time which would cause great problems in any sort of automatic analysis system. Even in systems incorporating a manual dilution stage, problems can arise if the blood sample is badly clotted. The problem is being investigated under contract and it is hoped that a solution to the problem will be found during the early part of 1975.

Negotiations are taking place for the acquisition of two further pieces of equipment to assist in the blood alcohol analysis field. The first is the development of an automatic dilutor to take blood from the current blood container and transfer it to Multifract bottles together with a precisely metered quantity of the diluent containing the internal standard. Forty suppliers who market dilution equipment have been approached but it appears that only two are likely to be interested or capable of developing the sort of equipment which we require. Negotiations are well advanced and preliminary experiments have been carried out into the problems of sampling and injecting through septum caps. Results appear to be very encouraging and initial experiments with the unmodified dilutor indicate that precision will be better than the diluting systems currently used in many laboratories.

Another area which is being actively investigated is the problem of designing an 'ideal' blood container for use with an automated blood alcohol analysis system. Clearly the round containers in current use would be most difficult to adapt for this purpose, because of difficulty in labelling to make them compatable with an automatic system. A container has been designed of the 'Flat Pack' type and features a security cap which, when once fitted, cannot be removed. Access to the blood is obtained by pushing the cap into the container and resealing the container with a fresh cap when the blood sample has been removed. Hence, access to the container is indicated by the presence of a cap within the container. The security cap has been patented and negotiations are in hand to produce some prototype containers for assessment.

With the increase in the number of staff within the division it has been possible to pay more detailed attention to certain other contracts under consideration. A device for the detection of cannabis hairs in packing cases has been suggested and considerable design and development work has been undertaken within the Division. The basic principle appears sound and the possibility of getting a professionally engineered device built is being investigated. In addition some preliminary work has been done on the development of tubes, similar to blood-alcohol tubes, for the detection of certain drugs and explosive substances.

8. INFORMATION DIVISION

Following a period of expansion as reported in last years Annual Report, the division has been reorganised into four sections, each section having a group of staff working on specific areas of information. This has been done for several reasons, the two most important being (a) the responsibility for the establishments' computer has now moved to the division and (b) the obvious need that every aspect of the division should have at least one member of staff available at any one time. This thus enables the whole of Information Division to provide a continuous and effective service to all of its users throughout the British Isles. The reorganisation of the division can be seen in the Projects List.

A. Literature and Commercial Information

(i) Collection, Collation and Dissemination

The full literature collection and presentation scheme was fully described in last years report. However, since then additions have been made which are worthy of note. The Transport and Road Research Laboratory very kindly supply us with copies of all their reports, and whenever one of these is of specific interest to the service additional copies are obtained for circulation to the laboratories. Metals Abstracts are now regularly searched and this has enabled a collection of papers of metallurgical interest to be made and added to the computer. The rate of collection is small compared with other disciplines and so does not justify a monthly current awareness circulation, but it is planned to send copies of the title pages to all laboratories and should any paper be required for full reference then it will be available in the usual manner.

The rate of accession of papers fluctuated from month to month due to various reasons outside the control of the establishment. Journals were delayed at the early part of the year and then arrived in waves, but in general the rate of accession averaged about 220 per month. The number of papers accessed to date is of the order of 15,000. The Information room has been short staffed for about a year and was very pleased when a new member joined the division in April. With this extra member of staff working in the section great inroads have been made in the backlog of work which had accumulated.

The current situation is as follows: Journals are received, scanned by a member of the division, marked for the attention of various members of staff, checked by a senior member of staff in the appropriate division and returned to the Information room. The papers marked are copied, allocated accession numbers, indexed in full, and then passed to a keyworder. The keywords are put onto the computer together with the accession number and are therefore ready for interrogation. The time scale for this service is approximately 10 days from the time the journal is received to the time that the selected papers are available for interrogation.

Unfortunately it is not possible to be as up to date with papers from journals not taken in the establishment, a delay of months being often experienced before specific papers can be obtained. (These papers being alerted by means of any of the UKCIS computer profiles run on Chemical Abstract Condensates, Biological Abstracts, or the Metals Abstracts or alerted from Current Contents Life Sciences taken inhouse.) Under these circumatances a system has been devised in which the title alone is keyworded, and the full description can be keyworded as and when the paper is received. In this way the literature information stored on the computer is only a matter of weeks behind the original alert. Searches of the literature can be made quickly by interrogation of the computer file for a few specific keywords. Great efforts have also been made to get all the papers in the information room put on 16mm microfilm and out to the regional laboratories so that their collection equals that at CRE.

Apart from working through the backlog of accessed papers and arriving in the present happy state, the staff of the division have been working on the computer package. Permission has been granted for the whole of our literature collection stored on the computer to be made commercially available. Once this was received there was an immense amount of work to be carried out in the checking through the entire collection of papers to ensure that it was complete, and also to generate the index of author, title and journal relating to the accession number of each paper. This is now well in hand and the current state indicates that the whole collection will be on the computer, filmed and indexed by the end of this year.

(ii) Information Services

The number of enquiries dealt with by the information room has increased and now averages 60 per month. Some of these queries can be answered quickly, while others require a great deal of work. We are grateful to those in regional laboratories who have been kind enough to provide us with details of the success or otherwise of the information they received. We would like to make a plea to those who gained from an enquiry to inform us of the detail of their experience so that it can be used for the advantage of the service as a whole.

Following last years' annual report there were many requests for copies of past CRE reports from law enforcement agencies in various parts of the world. To facilitate the handling of such requests, reports up to number 122 have been now put on microfiche.

Although the pressure of work on the information room has been eased by the extra member of staff, the backlog of work has taken most of the extra manpower to date. Despite this, a new service of undertaking specific literature surveys has been started, and successfully carried out for two divisions at CRE. If surveys are required by others, a similar service can be provided.

(iii) Communication Links

Following the proposed ideas on communication experiments reported last year steps have been taken to implement these experiments.

Initially Nottingham, Harrogate and CRE were equipped with Rank Xerox Telecopier machines for a period of three months. At the end of this period it was felt that a more accurate evaluation of the usage could perhaps be achieved if the initial honeymoon period were extended for a further three months. This was agreed and at the end of the total six months period the usage at CRE has found to be of the order of 20 transmissions or receptions each month. There were a few occasions when the transmission of graphical data could have only been carried out by facsimile machine or by postal services but obviously the time factor has to be considered.

Further to this experiment we have been asked to assist the Directorate of Telecommunications to evaluate a Plessey facsimile machine, and to this we have gladly agreed. This will enable us to look at another manufacturer's instrument and also the usage of two other laboratories. Machines have been installed in Newcastle, Bristol and CRE and are in the initial stages of evaluation.

The second experiment, to place Telex into two other laboratories which would make a similar triangular arrangement to the Telecopier, has started. The Cardiff laboratory has had a Telex installed for several months now and it has been used regularly for the interchange of information. A Telex has been ordered for Birmingham laboratory but due to various factors it has yet to be installed, but we look forward to the results of this experiment.

The third experiment to link a laboratory direct to the CRE computer has run into difficulties, not however from the computer aspect but from the communication aspect. Acoustic couplers were purchased which enabled a remote teletype to be connected to the computer via a telephone line and this has been successfully tried over distances of a few hundred yards to a hundred miles. However, the portable teletype used for this experiment has refused to work successfully from the laboratory chosen for the experiment. The reason for this is not yet known, but may be due to the fact that there is a manual telephone exchange involved. This is being pursued at greater length.

(iv) Inter-Laboratory Liaison

The usual programme of attachment of staff from regional laboratories to CRE continued, although this last year it was confined to two separate weeks, to which each laboratory sent a representative. This new scheme provided a greater degree of awareness of the work of CRE and also created additional liaison between laboratories.

Apart from these two weeks an additional week was set aside when senior members of staff of Chemistry and Biology sections in several regional laboratories attended for a five day period. This was found to be particularly valuable especially to staff at CRE when a useful period of discussion concluding the meeting resulted in a greater appreciation of the problems and goals of all present.

As a result of discussion at that meeting it is hoped to hold a two day meeting of a member of staff of each regional laboratory who will act as the main link between the establishment and each laboratory. This will enable a greater degree of efficiency in the interchange of information, and the answering of specific problems.

B. Implementation of Data Banks and Crime Scene Studies

(i) Data Banks

The year has been a period of consolidation as far as data banks are concerned. Due to the amount of work in the division and the staff situation, no new data banks have been undertaken, and only a few have been updated. Those not mentioned therefore in this report are in the same position as last year.

The continually increasing number of pharmaceuticals on the market in the United Kingdom have presented a problem in the past and attempts to keep the collection of infra-red spectra up to date have not always been successful due to various reasons. This year the division has been fortunate in having the services of a sandwich course student who has spent most of his stay in the establishment working on this problem. Pharmaceuticals available were cross checked against the current IR file and all the omissions listed. Manufacturers were approached and samples of pure drug substances were obtained. A debt of gratitude is owed to all those manufacturers who took part in this service.

The newly acquired pharmaceuticals were then run on the Perkin Elmer 137 spectrophotometer and the results fed into the system bringing the total number of IR's on the file to 3,060. It is proposed to put all these new IR's together with the past fiche updates onto a fourth cartridge roll film for circulation to all the regional laboratories. Many manufacturers have kindly agreed to keep us informed of all of their new products which are to be launched onto the market, and in this way the IR collection and obviously associated with this the collection of pharmaceuticals will be continuously kept up to date.

As reported last year, efforts have been and are being made to attempt to achieve a collection of vehicle headlamp and auxiliary lamp lenses. The original collection of 178 is already in laboratories in the form of glossy photographs. A further 68 have been collected, photographed and are at present awaiting information as to the vehicles on which they are used. This extra collection of

photographs will be sent to laboratories as soon as this information is available. It is estimated that there is approximately a further 170 still to be obtained to make the collection complete and efforts are being made to achieve this.

The collection of photomicrographs of sections of softwoods has been received from Contracts Division and the possibility of providing laboratories with a set of glossy photographs has been investigated. The general consensus of opinion is that this form is preferable to microfilm, and it is hoped to send this to laboratories in the near future.

(ii) Crime Scene Studies

The Crime Scene studies on the transference and persistence of fibres and glass on clothing has progressed well this year and the initial stages of the fibre work have been completed. This was allowed to progress ahead of the glass studies while we were awaiting accommodation for the window smashing experiments.

The number of wool and acrylic fibres transferred to various articles of clothing during simulated contacts has been studied[16]. The variation of the number of fibres transferred from a new wool sweater with area of contact has been investigated and repeated contacts over the same area were found to cause the transfer of some fibres back to the garment of origin. The effects of pressure, type of recipient garment, number of repeated contact passes and fibre length were investigated for wool fibres using a balanced four way classification experiment. After the analysis of variance all four effects, the pressure-length interaction and the garment-length interaction were found to be significant. The number of fibres transferred increased considerably with pressure and despite their surface apearance more fibres were observed on the recipient jackets than on the sweaters. When the same area of material was used for repeated contact passes the number of fibres transferred at each pass progressively decreased. The transference of fibres from the new wool sweater was compared to that from an old sweater and a square of handknitted acrylic material in a three way classification experiment. The significance of recipient garment and the number of contact passes was confirmed but no significant difference was found in the number of fibres produced by the three different transferring materials. As high pressure and coarse recipient garments produced a greater proportion of short fibres than low pressure and smooth recipient garments, the fragmentation of fibres during contact may be an important mechanism in fibre transference.

The persistence of wool and acrylic fibres has been studied[18] on the surface of six representative articles of clothing during various periods of wear to a maximum of 34 hours. The initial rate of fibre decay was rapid for all the garments studied and the highest proportion of the original fibres which remained after 4 hours of wear was 18%. The rate of fibre decay was indistinguishable for wool and acrylic fibres on all garments studied. The three jackets and two

sweaters despite the considerable differences in their construction and appearance, produced very similar rates of fibre decay. Fibres were lost at a significantly higher rate from a finely textured sports jacket and very rapidly from a cotton laboratory coat. In some of the experiments the length of fibres was recorded throughout, and the size distribution was found to be relatively constant during wear.

The mechanism by which fibres are transferred and persist on the surface of clothing has been investigated[31] and found to be mainly by mechanical forces, and that electrostatic forces have little effect, certainly after the initial one or two hours.

An examination of five searching methods, for removing transferred fibres from five representative articles of men's clothing has been carried out[24]. Results are quoted for the searching efficiency of shaking, vacuuming, a nylon brush fabric and high and low adhesive tapes. The efficiency of the last three were significantly higher than the previous two.

The number of background fibres removed together with the transferred fibres from a recipient garment were compared for the best three searching techniques.

A study was also undertaken on the number of fibres lost during the packaging of the clothing exhibits. It was found that under the worst possible conditions tested, only 2 - 3% of fibres present on the surface of clothing, were transferred to the package in which they were contained during transit.

Having recently been able to move to a new venue for the window smashing experiments, the studies on measurement of number and size distribution of glass fragment produced by backward fragmentation has continued.

Experiments have been carried out to determine the number and size of fragments of glass found on the ground at various distances from three different size windows. The glass used was 4mm thick and the three sizes were approximately 1.5 x 1.5m; 1.0 x 0.5m and 0.5 x 0.2m. The effect of breaking the windows by either a brick or a hammer held in the hand are being considered and these experiments are at present continuing. From the initial experiments the number of small fragments (0.1 - 0.5mm) produced from smashing the large window have been found to be very much greater than that produced from the two smaller panes. In all cases the size distribution of fragments found on the clothing of individuals standing in front of the windows were similar to that found on the ground in front of the window.

A very efficient method of removing glass from clothing has been found to be by vigorously shaking onto a piece of clean paper, but this method does not enable the location of the glass on the clothing to be plotted. X-ray radiography has been investigated to overcome

this and a range of sizes of glass fragments were placed on a variety of clothing material and also shoes. It was found to be possible to locate a particle of glass down to 0.6mm in size on the clothing but shoes were found to be far more difficult due to the large number of extraneous high density patches normally found on shoes.

C. Systems Analysis and Quality Control

(i) Systems Analysis

This particular project as outlined in last years' annual report has progressed rapidly and the collection of casework data from regional laboratories has been continuously extended. Close liaison has been maintained with the specialist committees who meet regularly on such topics as tyres, fibres, paint, glass, body fluids, toxicology and drugs. These committees have kindly acted as a forum for the discussion on the design of their own specific data collection forms, and also their implementation on a trial or continuous basis. Computer retrieval systems have been designed and implemented in the case of tyre, fibre and blood group data. Forms have been designed and are at present subject to pilot trials for toxicology, drugs, paint and glass. These form the basic structure around which we plan to build a total information system which should provide CRE and the service as a whole with all the requirements as itemized last year.

The tyre sub-committee was anxious for its data to be collected continuously and therefore contributions to the tyre file are now routinely received from all laboratories and the number of records is rapidly approaching 2,000. Each record has a vehicle and tyre description together with details of the accident, followed by the results and conclusions of the laboratory examination. The data collected can be used in several ways. The tyre sub-committee uses the file to monitor the results and conclusions reached in the various laboratories. Comparisons are possible because tyres can generally be assumed to fail at similar rates and in similar ways throughout the country. The file can also be used to determine the approximate incidence of various vehicles and tyres for statistical purposes. The tyre file is rapidly reaching the stage where it represents the largest data bank of information concerning the role of tyres in road accidents, in which each tyre has been examined by an experienced tyre examiner. A report is made to the full tyre committee annually. This meeting has a representative from each laboratory present, together with representatives from the tyre industry and other interested bodies.

The fibre file was run for a six month period and it is hoped will be run continuously in the near future. The form design is typical of the other data collection forms in that it is divided into two halves. The left hand side is for recording the control data on a garment including its description, fibre type and colour coding with

the Methuen system. The right hand side of the form is used for recording details of the suspect samples and the analytical methods of comparison used. As far as this section of the form is concerned sufficient useful information particularly for management planning can be obtained quickly and therefore the need for it to be run on anything other than a sampling basis cannot be justified. However, the control side of the form should be run continuously so that details of the fibre population as a whole can be recorded and maintained fo statistical purposes. Consequently it is envisaged at present that management information will be processed manually to gain maximum insight into laboratory operations and that the large volumes of statistical incidence data will be handled by computer.

The computer program for fibres has six options available to the user, the most important being the option for laboratory, year, type of textile material, sex of wearer and fibre type and colour required. A printout can be obtained of the number of records searched and the number of 'hits'. If required, a full listing of the 'hits' can be obtained.

The data retrieval form for blood has been designed to collect blood grouping results for all liquid blood controls submitted to regional forensic science laboratories for examination. The blood file at present has 2,187 records on it and these are all from one laboratory. These records have been placed on computer and the various programs written as the answer to a specific request from the Director of that laboratory. However, it is anticipated that this will be used for all laboratories in the future and details of searching for any laboratory have been written into the program. Here again the user is able to obtain details of frequency of specific blood groups or combination of blood groups and printouts of the situation under the various options will be available.

The various components planned for the total information system should be completed in the coming year and it is anticipated that assistance with the design of the final system for data collection and retrieval will be obtained by means of an external contract.

(ii) Quality Control

The 'quality control' programmes in the various branches of forensic science have been monitored during the last year and a great deal of useful information has been obtained not only in collecting information as to the methods of analysis employed in all the laboratories but also by indicating various ways in which some analytical methods can be improved within the service. As always the quality control work has been carried out under the auspices of the various specialist committees and their assistance has been very much appreciated.

This last year has seen several tests carried out on a variety of paint samples not only on coloured layer identification but also on techniques. Biological stains have also been analysed by all

laboratories, and these were basically designed to gain experience in the identification of specific body fluids and different species.

Preparation of samples, collection and collation of results and reports to the Controller of Forensic Science on blood and aqueous alcohol samples have been carried out on a weekly basis for some nine months now and regional laboratories have found these of great value.

D. Computer Services

(i) Organisation and Equipment

The departure from the Establishment of the member of Chemistry Division staff working on the computer necessitated the move of the responsibility for the computer from the division to the Information Division. Although the expertise within the division was available the staff situation was inadequate to accomplish all the computer work that was required. The "Computer Services" section was created and a member of the division has undergone training and is now providing the back up to that section.

The Establishment's HP2100A computer has been equipped with an increase of core capacity and is now at 16K words. A visual display unit complete with hard copy facility was purchased and has already proved extremely useful. An optical card reader is also available in house for the rapid feeding of data into the computer. In these ways the section is better able to provide the service expected of it, and it is hoped in the new year to add further peripherals which will enable economies to be made in computer operation time.

(ii) Services to Information Division

The task of providing a computer service to the whole of the Establishment and the Forensic Science Service has been immense but it has now been achieved and provides an operational retrieval service on literature, data banks and systems.

There are occasions when existing programs need to be modified or in some cases rewritten to make the task of retrieval that much more efficient. In the case of the literature file some logic options have now been written into the program. A great deal of interest has been shown in the literature storage and retrieval system used, and visitors from various parts of the world as well as other governmental establishments have been enthusiastic to try out or create a similar system in their own establishments. As indicated above, it is hoped to provide this as a "computer package" on a commercial basis.

In all the "systems analysis" work, where the computer has been used, programs have had to be written to put the data on file to validate it at various stages and to reject any invalid terms. A project that relies on the data must have accurate data and validating programs are therefore essential. Each of the specialist committees who have assisted with the collection of data, is interested in a variety of answers from the files it has helped to generate. The most common ones are the requests for listings of specific categories, tables of the contents of the files, or statistical information on frequencies of specific categories. Examples of special options are:

(a) In the case of the tyre file, apart from the usual facilities X^2 tests can be carried out on the four conclusion categories.

(b) In the case of the blood file the users are interested in possible correlation studies, and so the facility has been provided which enables three dimensional arrays of blood group data to be produced.

All of these options and many more besides have been produced within the section and represent a great deal of time and expertise. With the amount of data being collected, the time is rapidly approaching when not only will a full time teletype operator be required but also additional automated data processing equipment will be essential if the project is to continue to its obvious conclusion.

(iii) Services to Chemistry Division

With the creation of the computer services section came the responsibility for the programming of the automated data processing of organic mass spectrometer results. Because of the lack of availability of materials this project has not progressed as well as had been hoped, however soon the first standard spectrum should be produced, normalised and printed out, directly from the mass spectrometer computer link.

Low resolution organic mass spectrometers are to be installed in two regional forensic science laboratories in the near future, and it has therefore been considered necessary to implement a computer retrieval system for mass spectra. Because of the problems encountered with the MS computer link, the eight peak index retrieval is not as far forward as was hoped. However when it is complete it is anticipated that it will contain a collection of compounds normally encountered in the Forensic Science Service. Complementary to this is an additional facility which has been added as an aid to the mass spectroscopist both in regional laboratories and at CRE. This will cover a wide range of compounds, the mass spectra of which are not necessarily available in the eight peak index. A major difficulty in the compilation of this file is the representation of complex chemical formulae in a form recognisable to a computer and which retains the structural information. The system to be used is the 'Wiswesser Line Notation'.

The use of WLN enables the spectroscopist to make use of the structural information obtained from sources other than the mass spectrum. A file containing data from approximately 1000 psychotropic drugs has been compiled and a trial is soon to be carried out with the aid of Chemistry Division.

(iv) Colour Measurement

CRE has recently purchased, from the Paint Research Association, a slightly modified version of their fibre optics colorimeter. Due to the staff situation and the need for the results to be computerised, not only for quick and easy evaluation but also for the calculation of the colour co-ordinates, this instrument is operated by this section of this division. In the standard version of the colorimeter the sample area is approximately 1sq cm and is illuminated normally and viewed at 45^{o}. In the modified version constructed under a Home Office contract, to enable samples of approximately 1mm diameter to be viewed, the arrangement has been reversed and $45^{o}/0^{o}$ geometry is employed. The illumination of the sample is at 45^{o} by four fibre optic bundles arranged around the viewing fibre at 90^{o} to each other. The sample is viewed normally so allowing a focussing lens to be employed to image the small surface area onto the face of the collecting fibre lightguide. The light then passes through the filter wheel unit and is detected by the photomultiplier. The output from the photomultiplier across a suitable load resistance is read on a digital voltmeter whose scale is adjusted to read directly the tristimulus values, X, Y and Z for the illuminant C, of the paint flake under test.

Assessment of the instrument under ideal conditions (ie using standard colour cards for samples) is very nearly complete and these initial experiments have demonstrated that the instrument is extremely sensitive to small colour differences and is highly reproducible. Any colour difference that can be detected by the human eye under favourable conditions can be easily and quickly measured by this instrument. In one experiment several determinations on six pairs of BS4800 colour cards exhibiting small colour differences were carried out, and results obtained from the repeated measurements of the colour difference between each of the above pairs of samples showed a spread of not more than 0.2%.

A limited amount of work has been carried out using actual casework samples and, apart from a possible modification to enable reproducible sample presentation, no great difficulties have so far arisen.

9. TOXICOLOGY DIVISION

The effort put into toxicological research during 1974 has been aimed at achieving a balance between potential long-term projects such as the biochemical toxicology, the medium/short term ones such as radioimmunoassay techniques, and the short ad hoc projects comprising those which test out methods in current use in the regional laboratories.

The increasing commitment to radioimmunoassay of drugs in body fluids, mentioned in the 1973 Annual Report, has been expanded by the appointment of another specialist member of staff.

The progress in the various topics of research in 1974 is described in more detail in the following pages.

A. Difficult Compounds

For the past two years this work has concentrated on water-soluble compounds which defeat orthodox methods of extraction into organic solvents, and which are somewhat inert chemically, although highly potent as poisons.

(i) Fluoroacetamide

The investigation reported in last year's Annual Report into the extraction and detection of this compound from water, urine and blood was continued. It was found that the colorimetric method of detection on thin-layer chromatography (TLC) plates was not sensitive enough to cater for the low levels expected in toxic cases, thought to be about 1µg per g.

Using gas chromatography (GC) on a Poropak Q column at 180°C, 1µg of the compound produced a peak of retention time 5.3 minutes, and recoveries of added fluoroacetamide from water or urine were 30 - 35%, and from blood (directly, or by extracting a deproteinized filtrate) about 15 - 20%.

The use of low resolution mass-spectrometry (MS) by means of probe analysis provided the most sensitive means of detection, by virtue of the production of the FCH_2 ion, which has a mass/charge ratio (m/e) of 33. This value is not a common one in visceral extracts. Probe analysis detected a peak at m/e 33 in a dichloromethane extract prepared from a tungstic acid deproteinized filtrate which was obtained from 5ml of blood to which 2µg of fluoroacetamide had been added. Combination of gas chromatography with mass spectrometry, and scanning the peak obtained at the appropriate retention time for an ion of m/e 33 by single ion monitoring (SIM) now offers a diagnostic method for detecting the presence of fluoroacetamide in tissue extracts.

Fluoroacetamide tended to be labile to hot mineral acid so the subsequent deproteination of samples was carried out at room temperature using a solution containing aluminium chloride (10% w/v) and citric acid (10% w/v) in 2N hydrochloric acid. The extracting solvent was changed to methyl iso-butyl ketone (MIBK) when the screening analysis was expanded to include fluoroacetic acid.

(ii) Fluoroacetic acid

The sodium salt of this acid is the extremely toxic rodenticide "Compound 1080", and the chemical inertness of fluoroacetic acid makes its detection exceedingly difficult. When a solution of the pure acid was run on silica TLC plates in tetrahydrofuran - water 9:1, it could only be detected by spraying the plates with selective pH indicators. As regards GC it would separate well from fluoroacetamide on the Poropak column at 180°C, and had a retention time of 3.6 minutes. The same retention time was also possessed by methyl fluoroacetate.

The acid, unlike the amide, was not soluble in dichloromethane but it was readily extracted by ether from water especially if the latter was saturated with salt. However, complete loss of the acid occurred if the ether was boiled.

In view of this volatility, deproteination of blood samples containing the acid, was carried out at room temperature (using aluminium chloride in 2N hydrochloric acid) and extracts were made by ether-extraction of the salt saturated filtrates. These ether extracts were concentrated at room temperature in a current of air.

The analyses of the extracts by GLC on a Poropak column and by TLC were masked by the presence of endogenous tissue acids, which were extracted with the fluoroacetic acid, but probe analysis on the mass spectrometer detected the FCH_2 ion of m/e 33 on an extract prepared from 25ml of blood containing 6µg of the acid.

The use of gas chromatography with single ion monitoring (SIM) on the mass spectrometer necessitated the exclusion of ether from the extracts as this solvent gave a small fragment of m/e 33 which was picked up under these conditions (this interference from ether was not encountered in probe analysis as all "blank" samples gave a negative result for the presence of the FCH_2 ion). The extracting solvent was later changed to methyl iso-butyl ketone (MIBK) which extracted both the acid and the amide from salt saturated filtrates produced by cold aluminium chloride deproteinisation of tissue samples. The MIBK extracts were concentrated at room temperature and the acid methylated by the addition of a few drops of a solution of diazomethane in dichloromethane before injection onto the gas chromatograph. With this technique SIM detected the FCH_2 ion at the appropriate retention time on an MIBK extract prepared from 10ml of blood containing 5µg of fluoroacetic acid.

Using the GC - MS - SIM combination in this way a distinction could be made as to whether the FCH_2 ion picked up by a probe-analysis screen on the MIBK extract was derived from the acid or amide, or a mixture of both.

The method described was also successfully used to extract the amide and the acid from liver samples after additions of these substances to liver-water macerates at a level of 2µg per g of wet liver tissue.

(iii) Analysis of Tissues from Animals Poisoned by Solutions of Fluoroacetamide and Sodium Fluoroacetate

Extractions with MIBK were made by the method described on aluminium chloride deproteinized filtrates prepared from a combined heart-lung -liver-blood mixture from the poisoned and control animals. The FCH_2 ion was detected in the extracts from all the poisoned animals both by probe analysis and by GC - MS - SIM analysis. Comparison with a standard fluoroacetamide solution gave a figure of about 3µg of this compound per g of tissue for both guinea pig and rabbit tissues.

The extracts from control animals yielded negative results for the presence of the FCH_2 ion.

The approximate levels of fluoroacetic acid in the animal tissues were 1µg per g for a rabbit and 2.5µg per g for a guinea pig.

B. Action of Solvents on Basic Drugs

(i) Pure Solvents

As a result of using chloroform in attempts to extract certain bases from aqueous alkaline filtrates in the work reported on protein precipitation techniques (HOCRE Report 1973), complete failure was experienced with five alkaloids, namely amitriptyline, caffeine, chlorpromazine, imipramine and thioridazine. These drugs were recovered in varying yields using ether as the extractant, but if ether was used to extract them after using chloroform, nil recoveries resulted. Chloroform had, therefore, caused a permanent "disappearance" of these alkaloids during its use for extraction purposes.

To investigate this phenomenon further the action of four representative solvents, commonly in use in forensic toxicology, namely ether, ethyl acetate, chloroform and dichloromethane were tried upon 16 different alkaloids representing a cross-section of the different chemical classes of basic drugs encountered in regional laboratories. The interactions were studied first of all on each individual alkaloid in the pure dry solvents for up to 144 hours. Each drug was then extracted from alkaline aqueous solution by the 4 solvents in turn. Finally, the same amounts of each alkaloid were added in turn to blood samples (10ml) and extractions, using each of the 4 solvents individually, were carried out after protein had been removed by employing 5N hydrochloric acid at 90°C and by aluminium chloride in 2N hydrochloric acid at 60°C.

The assay of the drugs from the pure solvents, and of those extracted by these solvents from the deproteinised filtrates were carried out by the UV spectrophotometry of an 0.5N sulphuric acid extract of the solvents using known volumes of acid, and comparing the absorbancies obtained with those produced from standard solutions

of the alkaloids. This provided approximate recovery figures for the chromophoric part of the drug structures. In order to ascertain whether any change in species had occurred, the alkaloids were re-extracted from the sulphuric acid extract after UV assay, and run on TLC plates against control spots of the original drugs. Any significant changes in the Rf values of the spots, or the appearance of new ones were noted.

The UV assay for the alkaloids at different periods showed that in pure solvents they were stable over 144 hours, the exceptions being loss of a peak (λmax) for ephedrine at 262nm after 72 hours in chloroform and total loss of spectrum with all solvents except ethyl acetate at 144 hours. Dextropropoxyphene lost its λmax at 251nm after 144 hours in dichloromethane, and pethidine its λmax at 250nm after 72 hours in ether and dichloromethane, and after 120 hours in ethyl acetate. When methadone remained in contact with dichloromethane for 72 hours no appreciable change occurred in its UV spectrum, but if aliquot portions of the solution were analysed periodically by GC an extract peak at half the retention time of methadone gradually built up with time. Analysis of the same methadone solution on TLC plates furnished an extra spot of low Rf value, not originally present in the starting material. When this spot was extracted from the plate, and the extract analysed by GC it gave a peak of the same retention time as the previously observed extra peak. It is hoped to elucidate the nature of the substance responsible by mass spectrometry.

(ii) Extraction of the Alkaloids from Water

Extraction of the alkaloids from water with halogenated solvents under alkaline conditions distorted or destroyed the UV spectra of amitriptyline, caffeine, methaqualone and thioridazine, while the use of ethyl acetate as extractant was found to be unsuitable for drugs possessing λmax values of less than 255nm. Strong absorption by this solvent was experienced below this wavelength, even when the solvent had been cleaned up and redistilled.

(iii) Extraction from Blood

The use of the halogenated solvents, chloroform and dichloromethane, caused gross distortion, or destruction, of the UV spectra of tricyclic compounds (such as amitriptyline and methaqualone) and this effect was especially marked when hot 5N hydrochloric acid had been used as the deproteinizing agent before extraction with these solvents. No appreciable effect was noted if ether had been used to extract the tricyclic alkaloids. The halogenated solvents did <u>not</u> affect monocyclic compounds (eg amphetamine and ephedrine) or the complex "vegetable" alkaloids (eg strychnine) if used to extract them from deproteinated digests or filtrates.

Ethyl acetate caused distortion of the UV spectra of caffeine (λmax shift 272 → 263nm), 2-phenylethylamine (λmax shift 256 → 261nm with loss of other peaks), and methaqualone (spectrum masked by ethyl acetate).

(iv) Other Effects on Alkaloids

Extractions were carried out for methylamphetamine and ephedrine after addition to samples of very old transfusion blood which was showing signs of putrefaction. The spectra of the recovered drugs were grossly distorted, but this distortion was not cancelled out by using an extract prepared from a "blank" sample of the blood as a reference solution. This indicated that the distortions were due to interaction of a blood component with the drugs, and were not simply additive to the spectra.

If old blood was replaced by fresh blood these distortions did not occur.

(v) Kinetics of the Distortion of the UV Spectra of Tricyclic-Type Alkaloids

Amitriptyline was chosen as a typical tricyclic alkaloid whose UV spectrum was greatly distorted by alkaline chloroform extraction after hot 5N hydrochloric acid deproteination of blood samples.

Distortion of the spectrum was observed if the method was carried out on a solution of the drug in 5N acid in the absence of blood, and occurred also if the acid solution of the drug had not been heated.

Reduction in the normality of the hydrochloric acid in the aqueous solution before chloroform extraction caused a reduction in the distortion of the spectrum of the extracted drug.

The spectrum of amitriptyline was not altered by simply heating the drug in 5N hydrochloric acid to 90°C, without chloroform extraction.

Some progressive distortion of the amitriptyline spectrum was also caused by increasing the strength of alkali in an aqueous solution of the drug before immediate chloroform extraction.

The above observations indicate that distortion of the UV spectrum is not due to hot acid per se, or to any blood constituent, but appears to be caused by a combined effect of acid, chloroform and alkali during extraction. It is hoped that further investigation will elucidate the nature of the compound(s) formed during extraction under the above conditions.

The work carried out to date on solvent-alkaloid interactions illustrates the necessity for

(a) Testing for any interaction under laboratory extraction conditions, and not to rely solely on the reactions of alkaloids dissolved in pure solvents;

(b) Generally using ether for the extraction of all alkaloids and in any case prior to any extraction with halogenated hydrocarbons. Ethyl acetate is also not advocated for general use, only perhaps if the drug to be extracted is known, and has a $E^{1\%}_{1cm}$ value of at least 100 at a λmax greater than 255nm.

(c) Recognizing that pitfalls may attend the extraction of alkaloids from putrefying blood samples.

C. Recovery of Compounds from TLC Plates

(i) Extraction of Alkaloidal Drug-Spots from TLC Plates

It is standard practice in toxicology to run extracts of viscera containing basic drugs on TLC plates, primarily to clean up the extract and to ascertain whether or not a single alkaloid or a mixture of alkaloids is present. After the plate has been run, detection is usually done by spraying the plate with potassium iodoplatinate solution which converts alkaloids to their bluish-purple iodoplatinate complexes. Owing to the high potency of certain drugs very small quantities are present in the viscera and the quantity of extract placed on the TLC plate sometimes represents the majority of the drug present in the visceral sample. In these cases it is vital to be able to re-extract the original alkaloid from the plate spot for further examination, as the spot may represent the entire sample at the disposal of the analyst.

It was found that the most satisfactory method of doing this was to scrape off the plate material from the spot area and treat the powder with warm 0.5N sulphuric acid containing sodium sulphite. This dissociated the complex, and the freed alkaloid was extracted from the mixture with ether at an alkaline pH. For UV assay the whole of the ether extract was shaken with a small known volume of 0.5N sulphuric acid which was then scanned at the appropriate wavelength. For MS the ether extract was concentrated at room temperature to about 50µl of which 1 - 3µl were used for each probe analysis on the mass spectrometer.

The method was carried out on spots of 7 standard alkaloid solutions, each containing about 10µg of drug base, on cellulose, silica and alumina TLC plates. Recoveries were, in general, most satisfactory from silica plates (using an eluant of methanol-ammonia 100:1.5) and were of the order of 50 - 60% of the amount of alkaloid placed on them. Recoveries from the cellulose system (using a butanol-aqueous citric acid eluant) were inferior to a small degree (about 45 - 55%) but this system also suffered from the long duration of run on the TLC plate ($3\frac{1}{2}$ hours as against 15 minutes for silica). Alumina plates were found to be very unsatisfactory, both because of inferior recoveries and for the amount of interfering material extracted from the plates.

It was calculated that in general the amount of an alkaloid from a TLC spot required to give a reasonable mass spectrum was of the order of 200ng.

The presence of extraneous substances, which are co-extracted with alkaloids from blood samples, reduced the amount of alkaloids extracted from TLC plates to not less than 70% of the amounts recovered using pure alkaloids. On the cellulose plates, recoveries were 92 - 100% of those obtained using the 7 alkaloids amitriptyline, chlorpromazine, imipramine, morphine, nicotine, quinine and strychnine alone, and on the silica plates 72 - 100%.

Extractions from TLC plates using other methods, such as the direct extraction of damp alkali-sulphite treated spots with methanol or chloroform yielded inferior recoveries to those already described.

The method of alkaloid extraction from TLC plates was applied to 7 case-samples received from regional laboratories during 1974, where it was necessary to recover alkaloids from spots obtained by running extracts of human and animal tissues.

(ii) Ageing of Spots on the Plate

Occasionally, in casework alkaloid spots are left on TLC plates for a time (days) before removing them for further examination.

To ascertain whether the drugs would undergo change, especially as regards their UV spectra, when allowed to remain on the plates in this way, standard spots of 19 alkaloids, including the 7 already used in the extraction studies, were run on the cellulose and silica TLC plates. Two µl spots containing 10µg of each alkaloid were used for this. Spots of each alkaloid were removed at daily intervals over 7 days for extraction and UV assay as described previously. Primary and secondary amines (eg amphetamine, methylamphetamine) which possessed low $E^{1\%}_{1cm}$ values were assayed by first converting them in situ on the plate to their dithiocarbamates (by exposure to carbon disulphide vapour) and then extracting the spots with water. The aqueous extracts were scanned for maxima in the regions of 254nm and 284nm.

Spots of tertiary amine drugs with low $E^{1\%}_{1cm}$ values (eg codeine, methadone, orphenadrine and dextropropoxyphene) were assayed by gas chromatography of the ether extract produced by the procedure described above.

Results obtained so far have shown that in general, alkaloids are stable for up to 7 days on the TLC plates. Exceptions which have been observed included the gradual conversion of chlorpromazine to its sulphoxide (3 days) and spectral distortions with pentazocine (1 day) and tranylcypromine (2 days). Thioridazine could not be extracted from silica plates even from a freshly run spot of the drug.

The work on ageing is continuing with a further 12 alkaloids.

D. Radioimmunoassay

The radioimmunoassay service for cardiac glycosides in post mortem blood samples has continued as a service to the regional laboratories and 15 samples have so far been analysed in 1974. Two cases of lanatoside C poisoning were included, but all the others were digoxin overdose cases. The highest level of digoxin found was 90ng digoxin per ml of serum. Our experience with digoxin overdose cases led to the writing of publication 82 as an aid to the interpretation of blood levels of digoxin in cases of suspected overdosage.

Antibodies to LSD-protein conjugates have been raised in rabbits and guinea pigs at the Microbiological Research Establishment. The conjugates were formed by conjugating bovine serum albumen to the LSD molecule at the 1 position by the Mannich reaction and also by conjugation at the amide nitrogen of lysergic acid ethyl amide. A six monthly immunisation schedule was used and the antibodies produced are now being tested with tritium labelled LSD obtained from the Radiochemical Centre (Amersham). Commercially available reagents for the radioimmunoassay of LSD were found to be non-specific for LSD and also suffered from interference from normal constituents present in urine[7]. Expected levels of LSD (or its closely related metabolite) in the urine are in the region 1 - 50ng per ml. However, the equivalent of 1ng LSD per ml urine was encountered with some samples and drugs such as ergometrine, chlorpromazine and amitriptyline, at concentrations of 100µg per ml gave equivalent concentrations of up to 2ng per ml of LSD. It is hoped that the new antibodies, or those that have recently become available from other commercial sources will allow a specific radioimmunoassay for LSD in urine to be developed.

Due to requests for a simple introduction to the technique of radioimmuno assay as applied to drugs, an article was written explaining methodology of the technique as well as listing the instruments and facilities needed (83, 88).

E. Assessment of Analytical Techniques for the Identification of Drugs

The concept of discriminating power[45] which is defined as "the probability that two drugs selected at random from a large population would be discriminated by the technique" has continued to be used for the evaluation of chromatographic methods of analysis[68]. Its application to paper and thin layer chromatography[69] enabled the most important features of these systems to be recognized, namely speed of analysis, separation, sensitivity, reproducibility and lack of correlation. Thirty-seven paper and thin layer chromatographic systems in general use for the analysis of basic drugs were examined and their discriminating powers were measured, both individually and when used in combination. The better systems were comprised of thin layer plates of silica gel which had been sprayed with 0.1N NaOH, dried and run using one of the following solvents:

(a) Chloroform-methanol (90:10); (b) cyclohexane-toluene-diethylamine (75:15:10); and (c) acetone.

A thin layer cellulose system using n-butanol - water - citric acid (87:13:0.48) was suitable if speed was not a requirement and a reversed phase paper system run with an aqueous buffer solution (pH 4.6) at 95° was the fastest system examined[5,75]. The highest individual discriminating power obtained was 0.78. Combinations of two and three systems gave discriminating powers of 0.93 and 0.98 respectively.

A similar evaluation of gas-liquid chromatography[34,63,70] showed that many columns had similar properties and the best choice was undoubtedly an SE-30 one. With the co-operation and help of the regional forensic science laboratories, the inter-laboratory variation of measurement of the retention indices of drugs chromatographed using an SE-30 column was found to be between 15 and 20 retention index units. A compilation of the retention indices of 480 drugs and commonly occurring chemicals such as plasticisers has been made[20] and this should enable regional laboratories to standardise on SE-30 as the stationary phase of choice for the GC analysis of drugs, now that a comprehensive retention data bank has been formed.

The concept of discriminating power has also been applied to infra-red and ultra-violet spectroscopy with the result that a probability of identification can now be given to a technique whether it is a chromatographic or spectroscopic method[75].

F. Biochemical Toxicology

(i) Cyclic AMP

From a study of the effects of therapeutic doses of drugs on the cyclic AMP levels in urine it was found that neither imipramine nor trifluoperazine altered the cyclic AMP/creatinine ratio significantly.

Normally the cyclic AMP concentration in urine samples stored at room temperature was stable for only two days and so a number of compounds were in turn added to the samples as preservatives. Of these, sodium fluoride, phenyl mercuric nitrate, chlorpromazine and sodium azide all maintained the cyclic AMP concentration for at least three weeks. When it was necessary to store the urine samples at room temperature a tablet containing phenyl mercuric nitrate (50mg) and sodium fluoride (100mg) was added to each sample. This tablet is commercially available and by its use a consistent quantity of preservative could be conveniently added to any urine sample.

Cyclic AMP/creatinine ratios have been determined in urine samples from a number of non-drug deaths. The majority of the values obtained were within the normal cyclic AMP/creatinine ratio range (1.3 - 4.5nmol per mg). In the absence of preservative several of these urines showed a progressive decrease in cyclic AMP concentration with time even though the urines were deep-frozen. This is probably due to enzymatic degradation caused by bacteria or blood. The latter was found to be present in small quantity in all the post mortem urines analysed. The enzymatic action

probably occurs before freezing and during thaw out for analysis. This indicates the necessity of adding a preservative immediately after sampling has been completed.

Thirty-nine urine samples from drug overdose deaths have been analysed. Preservative had been added to all of these samples and no decrease of cyclic AMP level with time was observed. Four samples showed a significantly increased cyclic AMP/creatinine ratio (5.3 - 7.6nmol/mg and fourteen samples showed a significant decrease (nil to a maximum of 0.78nmol/mg) with respect to the normal range.

There are indications that certain types of drug overdosage may be reflected in a significant change in the urinary cyclic AMP/creatinine ratio. Three of the four urines in which increased ratios were observed came from deaths in which antidepressants (amitriptyline, nortriptyline) were involved. The fourth urine was derived from a case of fatal barbiturate poisoning which indicates that a high ratio is not unique to antidepressant drug deaths.

Thirteen of the fourteen low-ratio urines came from deaths involving the tranquillising or hypnotic drugs (chloral, methaqualone, barbiturates, phenytoin and diazepam). The fourteenth death involved an analgesic drug (aspirin). These results suggest that this type of drug death could be associated with a low cyclic AMP/creatinine ratio. The remaining twenty-one urine samples had "normal range" cyclic AMP/creatinine ratios.

(ii) Brain Amines

Fluorometric analyses of extracts of brain tissue for alterations in endogenous noradrenaline, dopamine and 5-hydroxytryptamine levels, as an indicator of a fatal dose of a monoamine oxidase inhibitor, have been discontinued owing to the instability of these compounds in post mortem tissue. Even though the relevant brain areas were rapidly deep frozen after removal from the cadaver (within 2 minutes of extraction of the brain from the skull), it was found that after storage in the deep freeze for approximately 3 weeks (a time period necessary for the collection of a reasonable number of suitable brain samples) brain amine fluorescence was indistinguishable from background fluorescence.

(iii) Anti-asthmatic Drugs

Anti-asthmatic drugs are usually difficult to extract and identify, and hence quantify, in viscera owing to

(a) the low tissue levels encountered, and

(b) their water solubility.

Initially, procedures were evaluated which might identify and quantify low concentrations of the commonly used anti-asthmatic drugs isoprenaline, orciprenaline and salbutamol.

Using fluorometric methods, only isoprenaline could be detected at the sub-microgram level (1ng per ml in aqueous solution). Both the trihydroxyindole derivative, and the ethylene diamine condensation product could be detected at this level.

The trimethyl silyl derivative of salbutamol has been separated from those of isoprenaline and orciprenaline by gas chromatography. Isoprenaline and orciprenaline have very similar retention indices but should be distinguishable from one another by their mass spectra.

It is hoped that using the above techniques, combined with a study of various methods for extracting these drugs from tissues, a method for the analysis of viscera or body fluids can be devised.

APPENDIX A

LIST OF STAFF MEMBERS

Name and Division	Rank	Telephone Extensions
DIRECTOR		
Dr A S Curry	DCSO	5853, 5856
(Mrs I L White, Personal Secretary, 5853, 4212)		4212
DEPUTY DIRECTOR		
Mr G W Walker	SPSO	5853, 5856
(Mrs I L White, Personal Secretary, 5853, 4212)		4212
BIOLOGY DIVISION		
Dr P H Whitehead	PSO	5947
Mr E R Rutter	PSO	5938
Mr J G Sutton	SSO	5937
Mr M J Davie	SSO	5937
Dr A E Kipps	HSO	5937
Dr P E Burdett	HSO	5937
Mr D J Werrett	HSO	5937
Miss V E Quarmby	SO	5937
Dr L A King	SRF	4289
CHEMISTRY DIVISION		
Mr K W Smalldon	PSO	5947
Mr B German	SSO	7574
Dr J V Drayton	SSO	5505
Dr J D Twibell	SSO	5505
Dr J Locke	SSO	7574
Dr R J Dudley	HSO	5505
Mrs D Morgans	SO	7574
Mr C R Howden	SO	7574
Mrs I B Beattie	ASO	5505
DRUGS OF ABUSE DIVISION		
Dr A C Moffat	PSO	5947
Mr P J Gomm	SSO	5948
Dr P J Twitchett	SSO	5948
Dr S Fletcher	HSO	5938

Name and Division	Rank	Telephone Extensions
EXTERNAL CONTRACTS DIVISION		
Dr M D G Dabbs	PSO	5930
Dr R Holleyhead	SSO	5930
Mr D S Loxley	HSO	5930
INFORMATION DIVISION		
Mr V J Emerson	PSO	6585
Mr M Swain	SSO	6996
Mr C Brown	SSO	5952
Mr C A Pounds	SSO	4289
Mr G W Owen	HSO	4289
Mr J Porter	HSO	5505
Dr R E Ardrey	HSO	6996
Mr M C D Harold	SO	6996
Mr S Brandish	CA	6996
TOXICOLOGY DIVISION		
Dr H M Stevens	PSO	5947
Mr P Owen	HSO	5938
Mr M D Osselton	HSO	5938
Miss T M Holdstock	ASO	5938
TECHNICAL STAFF		
Mr D J Nicholson	P & TO III	5783
CLERICAL STAFF		
Miss M North	EO	5502
Mr S Jones	CO	5942
Mrs P Ridout	CO	5560
Mrs S M Webb	Typist	5560
Mrs M A Golding	Typist	5853
Miss H K Payne	CA	5942
NON-TECHNICAL STAFF		
Mr L Rowbottom	Driver	
Mr D M Gabb	Laboratory Attendant	

APPENDIX B

CURRENT PROJECTS

BIOLOGY DIVISION

Head of Division - Dr P H Whitehead

Subject	Staff	State of Progress
Discrimination Studies Using Non-Genetic Parameters		
(a) Blood		
(i) Drugs		Therapeutic salicylate levels in micro blood stains can be detected. Difficulties encountered with other drugs, but research continuing.
(ii) Parasitic antibodies	L King D Werrett V Quarmby	Technique shows potential new means of discriminating between blood, especially of children and adults. Other areas under investigation.
(iii) Clinical biochemical parameters		Appraisal complete. Limited value in biochemical profiling at present.
(b) Hair		Tryptophan fluorescence studies complete and technique shown to have no value for discrimination of human hairs. Sexing of hair shown viable. Work continuing.
Discrimination Studies Using Genetic Parameters	J Sutton A Kipps P Burdett	
(a) Semen		
(i) Acid phosphatase		Work continuing. A report of genetic variants not confirmed. Possibility of separating seminal AP from vaginal AP by isoelectric focussing.

Subject	Staff	State of Progress
(ii) Other semen enzymes	J Sutton A Kipps P Burdett	Abnormal PGM variants found in semen. PGM isoenzymes found to be labile in liquid semen. Chemistry of changes being investigated. G6PD found in semen.
(b) Saliva		
(i) Amylase genetic variants		New means of detection and quantitation of amylase in body fluids described. Patent filed.
(ii) Other salivary enzymes		Studies on the genetic variants of immunoglobulins found in saliva initiated. Other parameters in saliva being considered, eg, Thiocyanate.
(c) Blood		
(i) Studies on established enzyme variants		Work on quantitation and simplification started PGM, EAP, HB separated simultaneously by isoelectric focussing. Other media considered.
Immunology Studies		
(a) Use of Latex		
(i) Species identification of blood and tissues	M Davie P Burdett	Technique shown to be viable. Successful blind trial held. Batches evaluated for use by other forensic science laboratories.
(ii) Serology		ABO grouping by latex being considered.
(iii) Other antigens		To be started.
(b) General studies		Development studies and appraisal of new techniques continue.
Correlation Studies	E Rutter A Kipps	Studies into possible correlations between genetic enzymes and serological groups started. Original observations made on amylase levels in lip secretions.

Subject	Staff	State of Progress
Automation		
Blood	E Rutter M Davie	Continuous flow and batch processing machines for ABO grouping being developed "in house" and by external contract. Amylase automated. Differentiation of saliva on basis of amylase concentrations a possibility.
Botanical Studies		
Pollen	A Kipps	Cannabis hairs found on outside of containers by microscopy; further work in Contracts Division.

CHEMISTRY DIVISION

Head of Division - Mr K W Smalldon

Mass Spectrometry		
(a) Organic MM12		Micromass 12 with GC link and 8-Channel multiple ion monitor.
(i) Data collection		Hard copy and computer retrieval systems for drugs being constructed. Automatic data handling to be operational shortly.
(ii) Service facility for other divisions	J Drayton J Locke D Morgans B Beattie	Project co-operation with Toxicology and Drugs of Abuse Divisions.
(iii) Service facility for regional laboratories		Samples examined which cannot be identified satisfactorily with existing techniques.
(b) Inorganic MS702		Output from peak scanning mode to be handled by computer. Peak switching undergoing investigation with manufacturer.

Subject	Staff	State of Progress
(i) Trace elements in liver tissue	J Drayton J Locke D Morgans B Beattie	Method for the detection and quantitation of a large number of elements simultaneously being developed. Low temperature asher on loan from Harrogate Laboratory.
(ii) Glass		Discrimination obtainable with reduced sample size being investigated.
(iii) Service facility for regional laboratories		Analysis of glass samples and small fragments of metal, especially ferrous metals.
Glass, Paint and Fibrous Materials		
(a) Glass		Surveys of trace elements in glass from various sources completed by inorganic MS. Development of method for routine casework now required. Atomic Absorption studies begun. Other methods under consideration including emission methods with computerised plate reader and alternative excitation sources.
(b) Paint	B German A Bond C Howden	
(i) Smears		Characterisation methods for paint smears to be investigated. Full evaluation of Laser Spark Emission to be made.
(ii) Single layer systems		Further methods of organic analysis to be studied.
(iii) Multi-layer systems		Improved characterisation of samples to be attempted using intact flakes. Approach to include staining tests, luminescence methods and etching of binder to expose pigment particles for improved microscopy.

Subject	Staff	State of Progress
(c) Fibrous materials		
(i) Tracers and catalysts in synthetic fibres		Survey of trace elements in acrylic fibres completed on MS702. Data collection on tracer elements and catalysts in polyester fibres in progress from literature sources.
(ii) Fibre dyestuffs	B German A Bond C Howden	To be considered after further discussions with the "Fibre Study Group".
(iii) Hair		Possible approaches being considered including elemental profiles along single hairs.
(d) Service facility for regional laboratories		Service facility for Atomic Absorption and Laser Spark Emission in appropriate cases.
Other Evidential Materials		
(a) Firearms' blow-back residues on hands	J Twibell R Dudley	Preliminary study. Experimental work has just begun to determine if organic residues can be detected.
(b) Soil		Wet colour, dry colour and ash colour investigated. Several other techniques being evaluated at the present time. Simulated crimes to be studied within the next year.

DRUGS OF ABUSE DIVISION

Head of Division - Dr A C Moffat

Drugs Intelligence	P Gomm	Operational research into the distribution of "target drugs" (LSD, amphetamine, heroin, cocaine, and hash oil) and characterisation of illicitly manufacturered drug preparations successfully continuing.

Subject	Staff	State of Progress
High Pressure Liquid Chromatography		
(a) Evaluation	P Twitchett	Development of optimum conditions for detecting common tranquilisers and sedatives in small volumes of blood.
(b) LSD and cannabinoids		Analysis of drugs and metabolites at the nanogram level from body fluids in conjunction with MM12.
(c) Illicit preparations		Separation of precursors, drug, excipients and adulterants. Chinese heroin nearly finished.
Blood Alcohol		
(a) Evaluation of containers for blood and urine	R Holleyhead	Evaluation of containers used in Road Traffic Act cases nearing completion.
(b) Increase of throughput of cases		Improvement of column and instrument technology to allow analysis times of one minute, continuing.
(c) Breath testing devices		Periodic testing of devices submitted by manufacturers to be examined.
Radioimmunoassay		
(a) LSD	S Fletcher P Owen	Antibodies prepared at MRE and ^{3}H-LSD from Radiochemical Centre, to be used in an attempt to develop an assay that is applicable for urine and blood.
(b) Insulin		Method of assay to be evaluated for human insulin and pharmaceutical preparations in diabetic and non-diabetic conditions. Extraction of insulin from tissue and body fluids. Degradation of insulin after death.
(c) Cardiac glycosides		Distribution ratio between serum and erythrocytes to be determined.
(d) THC		To be started.

Subject	Staff	State of Progress
EXTERNAL CONTRACTS DIVISION		
Head of Division - Dr M D G Dabbs		
Initiation and Supervision of all External Contracts	R Holleyhead D Loxley	
INFORMATION DIVISION		
Head of Division - Mr V J Emerson		
Literature and Commercial Information Collection Collation and Dissemination		
(a) In house searching		15,000 reprints on file HP2100A used for searching.
(b) Use of UKCIS		
(c) Literature presentation to regional laboratories	M Swain R Ardrey M Harold S Brandish	Selected papers to all laboratories monthly.
(d) Microfilm and microfiche		Full collection to all laboratories on film
(e) Preparation of literature package for commercial exploitation		Author, Title, Journal index 80% completed. Hoped to be completed by end of year.
(f) Communication links		Facsimile still under trial. Telex under trial. Direct computer link under trial.
(g) Collecting firearm data		To be started.

Subject	Staff	State of Progress
Implementation of Data Banks and Crime Scene Studies		
(a) IR collection		Up to 3,060 spectra in current update.
(b) UV collection		1,000 spectra on file.
(c) Collection of pharmaceuticals		In excess of 2,000 samples available.
(d) Collection of agricultural chemicals		200 plus UV and IR available.
(e) Register of human toxicology		Details of unusual poisonings in regional laboratories.
(f) Collection of head lamps		Now about 246 in total: to be completed.
(g) Boot and shoe patterns		Subject of an external contract. British makes in laboratories.
(h) Tyre patterns	C Pounds	Subject of an external contract, expect 400 by end of year.
(i) Car paints		All details of manufacturers and vehicle details coded and in the laboratories.
(j) House paints		Details of new products being collected.
(k) Wood sections		Collection of soft wood now received from contracts division.
(l) Collection of PGC rubbers		Subject of an external contract.
(m) Collection of side lamps		To be started.
(n) The analysis of glass on clothing		In the final stages with small windows.
(o) The study of fibre transference under real life conditions		To be started.

Subject	Staff	State of Progress
Computer Services		
(a) Computer services for all Information division		Data banks, literature and systems work, all in progress.
(b) Computer services for Chemistry division	C Brown J Porter	Mass spectrometry and microdensitometer to be started.
(c) Computer services for Administration section		Accounts starting.
(d) Evaluation of the new colorimeter and compute results		Has started.
Systems Analysis and Quality Control		
(a) Supervision on systems running		A detailed study of work carried out in regional laboratories and analysis made to help plan research.
(b) Analysis of system data		
(c) Co-ordination with External Contracts on systems	G Owen	Agreed to be started.
(d) Run all quality control trials		Continuing regularly.
(e) Analysis of quality control results		Samples for analysis sent to all laboratories

Subject	Staff	State of Progress
TOXICOLOGY DIVISION		
Head of Division - Dr H M Stevens		
Difficult Compounds		
(a) Quaternary Ammonium compounds		Completed, except for assistance with odd cases.
(b) Fluoroacetamide/ fluoroacetate		Animal extraction experiments only left to do - probe MS on animal extracts shows FCH_2 on poisoned animals.
(c) Protein precipitation		Blood studies with added drugs completed - limited study using liver tissue to be done, including chlorimipramine.
(d) Extraction of benzodiazepin drugs from tissues		Preliminary experiments suggest extraction from tissue only feasible as benzophenones.
(e) Extraction procedures and assays for catecholamine drugs in tissues	H Stevens P Owen M Osselton T Holdstock	Fluorescence assay applicable to some members. GC applicable to all compounds tried. Future work will involve use of GC/MS.
(f) Extraction of alkaloids from TLC plates with a study of ageing on plates		Best method to extract bases from Pt1$\overline{G}$ - treated plates determined. Ageing experiments in progress for about 20 alkaloids.
(g) Action of solvents on basic drugs		Preliminary studies on the stability of 16 alkaloids in pure solvents, and when extracted from water and blood nearing completion. Further studies on mechanism of interaction with different chemical classes to continue, and will include chlorimipramine.
(h) Action of formal-saline on barbiturate levels in case liver		Not started.

Subject	Staff	State of Progress
Putrefaction of Human Viscera	T Holdstock	Not started. Atlas of analytical data to be compiled for acid and neutral compounds.
Biochemical Toxicology		
(a) Cyclic AMP in urine		Last 30 urines to be assayed for cyclic AMP to complete picture of levels in cases of intoxication with hypnotic and anti-depressant drugs
(b) Natural catecholamines in brain tissues as an indication of drug intoxication	P Owen	Assays rendered useless by post-mortem decomposition of brain tissues.
(c) Action of drugs on iso-enzymes of phosphodiesterase and monoamine oxidase		Not started.
(d) Use of enzymes for the detection of drugs		Not started.
Assessment of Analytical Techniques		
(a) GLC systems	A C Moffat	Choice of optimum system, measurement of inter-laboratory reproducibility of retention indices and collection of data for 480 drugs completed.
(b) TLC systems		Three systems have been chosen and their measurements of reproducibility are now in progress. Data collections to follow.

APPENDIX C

REPORTS PUBLISHED SINCE NOVEMBER 1973

Report & Ref No	Title	Date
111 (1)	The Evaluation and Application of Two Standard Colour Systems in the Forensic Science Examination of Soil.	December 1973
112 (2)	Studies on Saliva. I - The Detection of Saliva Stains Using a Soluble Starch Derivative - Procion Red-Dyed Amylopectin.	December 1973
113 (3)	The Identification of the Species Origin of Bloodstains Using Sensitised Latex.	January 1974
114 (4)	The Identification of Anti-Parasitic Antibodies in Bloodstains Using an Indirect Fluorescent Antibody Technique. I - Preliminary Report.	January 1974
115 (5)	The Choice of Paper and Thin-Layer Chromatographic Systems for the Analysis of Basic Drugs.	January 1974
116 (6)	A Micro-Technique Involving Species Identification and ABO Grouping on the same Fragment of Blood.	February 1974
117 (7)	Evaluation of the Radioimmunoassay for Lysergide.	February 1974
118 (8)	The Discriminating Power of Discrete Attributes which Produce a Proportion of Ambiguous Results.	March 1974
119 (9)	The Use of 'Cellogel' in the Determination of PGM Isoenzymes.	March 1974
120 (10)	Studies on Saliva. II - A Test Paper for Detecting Saliva Stains.	March 1974
121 (11)	Organic Mass Spectrometry. I - Use of the Isothiocyanate Derivatives of Amphetamine for Rigorous Identification in Routine Casework.	March 1974
122 (12)	Microsampling Techniques in Visible and Ultra-Violet Spectroscopy and Related Problems in Forensic Science.	March 1974

Report & Ref No	Title	Date
123 (13)	Studies on Saliva. III - A Method for Quantitating Amylase and its use in the Investigation of Various Body Fluids.	June 1974
124 (14)	Organic Mass Spectrometry. II - Use of Micromass 12 Mass Spectrometer for Routine Casework.	June 1974
125 (15)	Further Discrimination Between Soils by Examining Their Moist and Ashed Colours.	June 1974
126 (16)	The Transfer of Fibres Between Clothing Materials During Simulated Contacts and Their Persistence During Wear. I - Fibre Transference.	June 1974
127 (17)	Studies on Saliva. IV - Amylase in Lip Mucus Secretion.	June 1974
128 (18)	The Transfer of Fibres Between Clothing Materials During Simulated Contacts and Their Persistence During Wear. II - Fibre Persistence	June 1974
129 (19)	Studies on Semen Enzymes. I - Anomalies in the Typing of Phosphoglucomutase Isoenzymes in Semen.	June 1974
130 (20)	The Use of SE-30 as a Stationary Phase for the Gas-Liquid Chromatography of Drugs.	July 1974
131 (21)	Studies on Semen Enzymes. II - 6 Phosphogluconate Dehydrogenase in Vasectomized and Non-Vasectomized Semen.	July 1974
132 (22)	High Pressure Liquid Chromatography.	August 1974
133 (23)	The Analysis of Illicit Diamorphine Preparations by High Pressure Liquid Chromatography.	August 1974
134 (24)	The Recovery of Fibres from the Surface of Clothing for Forensic Examinations.	September 1974
135 (25)	The Identification of Anti-Parasitic Antibodies in Bloodstains Using an Indirect Fluorescent Antibody Technique. II - The Discrimination Achieved by a Comparative Method.	September 1974

Report & Ref No	Title	Date
136 (26)	A Potential Means of Simplifying the Phenotype of Blood.	September 1974
137 (27)	Studies on Semen Enzymes. III - The Stability of PGM Isoenzymes in Liquid Semen and Seminal Stains.	October 1974
138 (28)	The Age Relationship of Antibody Levels to M.tuberculosis and V.cholera in Human Blood.	October 1974
139 (29)	Review of Information Queries Received 1973 - 1974.	October 1974
140 (30)	Studies on Semen Enzymes. IV - Anomalies in the Typing of PGM Isoenzymes in Semen from Vasectomized Men - The Effect of Mercaptoethanol.	October 1974
141 (31)	The Transfer of Fibres Between Clothing Materials During Simulated Contacts and Their Persistence During Wear. III - Mechanisms.	October 1974
142 (32)	Graphitized Carbon as a GC Column Packing for Blood-Alcohol Analyses.	October 1974
143 (33)	Pre-Packaged Overlay Reagents for PGM Isoenzyme Visualisation.	October 1974

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(43) Laser-Arc Emission Spectroscopy.
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(50) The Radioimmunoassay of Digoxin in Post Mortem Blood.
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(51) The Sexing of Human Bloodstains.
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(52) The Use of Organic Mass Spectrometry in Forensic Science.
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Phillips, A P; J For Sci.

(79) A Test Paper for Detecting Saliva Stains.
Whitehead, P H and Kipps, A E; J For Sci Soc.

(80) Blood and Semen Stains on Outer Clothing and Shoes not Related to Crime - Report of a Survey Using Presumptive Tests.
Owen, G W and Smalldon, K W; J For Sci.

(81) Interpretation of Post Mortem Serum Levels of Cardiac Glycosides After Suspected Overdosage.
Moffat, A C. Acta Pharm et Toxicol.

(82) Structure-Luminescence Correlations in the Thiobarbiturates.
King, L A and Gifford, L A; Anal Chem.

(83) The Radioimmunoassay of Drugs.
Landon, J and Moffat, A C; Analyst.

(84) The Value of Biochemical Profiling for the Discrimination of Bloodstains.
King, L A; J For Sci Soc.

(85) Evaluation of Chromatographic and Spectroscopic Procedures.
Moffat, A C; In "The Poisoned Patient: The Role of the Laboratory", Ed. M O'Connor, Ciba Symposium 1974, Elsevier.

(86) The Poisoned Patient.
Curry, A S; In "The Poisoned Patient: The Role of the Laboratory", Ed. M O'Connor, Ciba Symposium 1974, Elsevier.

(87) Mass Spectrometry.
Scaplehorn, A W; In "Isolation and Identification of Drugs", Ed. E G Clarke, Pharmaceutical Press.

(88) The Radioimmunoassay of Drugs.
Moffat, A C; In "Isolation and Identification of Drugs", Ed. E G Clarke, Pharmaceutical Press.

Ref No

(89) Pyrolysis Gas Chromatography.
May, R W, Pearson, E F and Scothern, D; Analyst.

(90) Poison Detection in Human Organs.
Curry, A S; 3rd Edition, Pub. Charles C Thomas.

(91) Reliability and Significance of the Numbers Resulting from Alcohol and Drug Analyses.
Curry, A S; 6th International Conference on Alcohol, Drugs and Traffic Safety, Toronto, September 1974.

APPENDIX D

COLLOQUIA

During last winter the customary series of colloquia were held by CRE. However, this time we deviated from our usual programme of speakers from within the service, and also moved from our usual venue.

The series last winter were of a high academic level with the majority of speakers coming from Educational and Research Establishments up and down the country. We were also privileged to have several eminent people to Chair these colloquia. Due to a variety of reasons we decided that the Sir William Penney Theatre at AWRE would provide the best accommodation and buffet lunches were provided for those that required them. In this way we were not only able to invite interested people from outside the service and in fact did have 150 people attending one meeting, but were also able to carry on valuable discussions throughout the lunch recess. The whole series benefited from these two factors.

We are indebted to all the Chairmen and speakers who contributed to the success of our colloquia, the programmes of which are on the following pages.

'Colloquium on

Drug Metabolism'

Friday, 28 September, 1973

Chairman: Dr F L Rose, OBE, FRS
Pharmaceutical Division,
ICI, Macclesfield.

The Metabolism of Cannabinols
Dr L King, Department of Biochemistry, University of Surrey.

Discussion

The Metabolism of Amines
Professor A H Beckett, Department of Pharmacy, Chelsea College, University of London.

Discussion

The Metabolism of Barbiturates
Dr B Millard, School of Pharmacy, London.

Discussion

'Colloquium on

The Chemistry of Blood Group Substances'

Friday, 9 November, 1973

Chairman: Dr R D Marshall
Dept of Chemical Pathology,
St Mary's Hospital, London W2

The In-Vivo Biosynthesis and Degradation of Glycoproteins
Dr G Robinson, University of Oxford.

Discussion

The Structure of Blood Group Specific Glycoproteins
Dr A S R Donald, Lister Institute of Preventive Medicine.

Discussion

The Distribution of ABO, P, I and MN antigens Within the Red Cell Membrane
Dr D Anstee, National Blood Transfusion Service, Bristol.

Discussion

'Colloquium on

New Developments in Analytical Chemistry'

Friday, 25 January, 1974

Chairman: Professor R Belcher
Dept of Chemistry,
University of Birmingham

MECA Spectroscopy
Dr A Townshend, University of Birmingham.

Discussion

Low Temperature Luminescence
Dr D Thorburn-Burns and Dr J N Miller, University of Technology, Loughborough.

Discussion

Plasma Spectroscopy
Mr S Greenfield, Albright & Wilson Limited, Warley.

Discussion

'Colloquium on

The Interpretation of Scientific Evidence'

Friday, 8 March, 1974

Chairman: Sir Douglas Osmond, CBE, QPM
Chief Constable,
Hampshire Constabulary.

The Lawyer's View
Mr C J I Bourke, Office of the Director of Public Prosecutions, London.

Discussion

The Adelaide Murder
Mr J L Fish and Mr C F Tippett, Home Office Forensic Science Laboratory, Cardiff.

Discussion

Faulty Inference
Professor K Simpson, University Professor of Forensic Medicine, London.

Discussion

'Colloquium on

Genetics'

Friday, 26 April, 1974

Chairman: Dr C E Ford, FRS
Sir William Dunn School of Pathology,
University of Oxford

Mammalian Genetics with Special Reference to Pigmentation
Dr A G Searle, MRC Radiobiology Unit, Harwell.

Discussion

Recent Developments on Isoenzyme Variants
Dr D A Hopkinson, MRC Human Biochemical Genetics Unit,
University College, London.

Discussion

Genetics of HL-A Antigens
Dr S J Starkie, National Tissue Typing Reference
Laboratory, Bristol.

Discussion

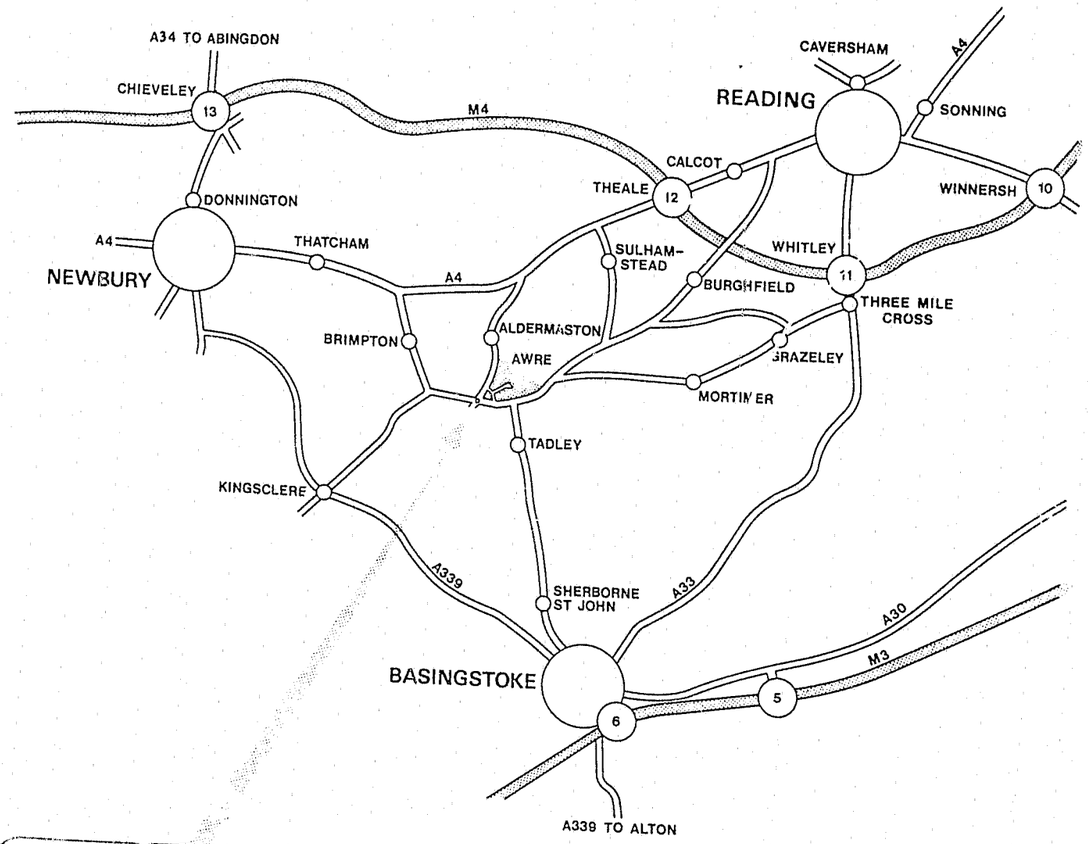
APPENDIX E

THE GEOGRAPHY OF ALDERMASTON

For UK visitors, Motorways M3 and M4 are adjacent and are shown on the map. The telephone is manned by the Director's Secretary on Tadley (07356) 3833 ext 5853. It is essential to telephone beforehand so that the necessary pass can be lodged with the police.

Trains from London (Paddington to Reading or Waterloo to Basingstoke) are advised for overseas visitors. Services are frequent and fast (30 to 50 minutes). There is also a British Railways bus link direct from London (Heathrow) Airport to Reading (50 minutes). Transport will be arranged from these railway stations which are about 10 miles away from CRE, provided that 24 hours' notice is given to the Director's Secretary.

Visitors not used to British Railways are advised that the method of opening carriage doors is first open the window and use the handle on the outside of the carriage!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CHIEVELEY
13
M4
DONNINGTON
A4
NEWBURY
THATCHAM
A4
BRIMPTON
ALDERMASTON
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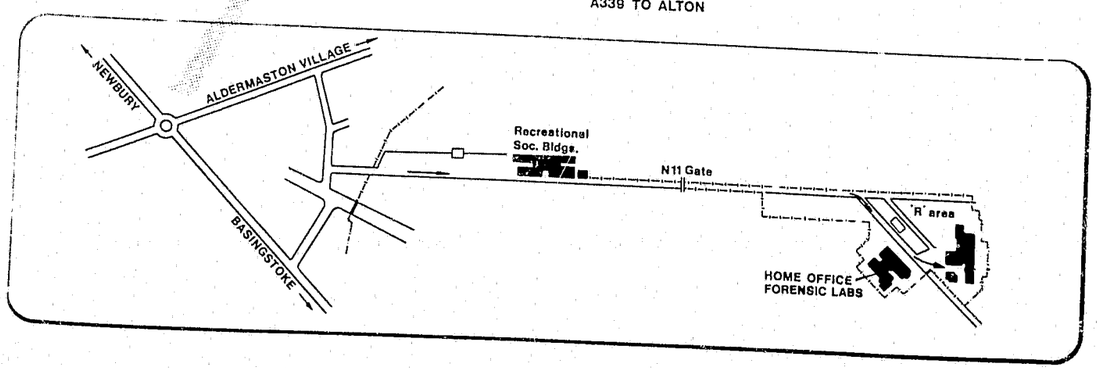
SULHAM-
STEAD
THEALE
12
CALCOT
READING
CAVERSHAM
A4
SONNING
WINNERSH
10
WHITLEY
11
BURGHFIELD
THREE MILE
CROSS
GRAZELEY
MORTIMER
TADLEY
KINGSCLERE
A339
SHERBORNE
ST JOHN
A33
BASINGSTOKE
6
5
A30
M3
A339 TO ALTON


NEWBURY
ALDERMASTON VILLAGE
BASINGSTOKE
Recreational
Soc. Bldgs.
N11 Gate
'A' area
HOME OFFICE
FORENSIC LABS

END