# ANNUAL REPORT

Sir Arthur Peterson KCB MVO
Permanent Under Secretary of State
The Home Office
London SW1

Sir

I have the honour to present the Annual Report of the Home Office Central Research Establishment for the period November 1972 to November 1973.

Yours faithfully

A S Curry, MA PhD FRIC FRCPath
DIRECTOR

November, 1973

(72536)

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# ANNUAL REPORT 1973

## 1. INTRODUCTION

The outstanding feature of this year's work has been the major changes in Senior Staff that have occurred. Mr J L Fish, Head of Biology Division was promoted to be the Director of the Cardiff Regional Laboratory; Dr A W Scaplehorn, Head of Chemistry Division and Dr E F Pearson, Head of Information and Contracts Division, were selected for the course at the Civil Service College and are now embarked on a two-year period of administration in the Home Office. In addition, Drs May, Fouweather and Renshaw left to gain experience in regional laboratory work as a result of deliberate career planning. Such changes emphasise the opportunities that a scientific career in the Home Office offers to its employees and indicate the close links that exist between The Central Research Establishment (CRE) and the Forensic Science Service. They also keep CRE as a dynamic Establishment as new brains are brought in to study the research problems. The changes brought an opportunity for reorganisation and now the Divisions number six – Biology, Chemistry, Toxicology, Information, Drugs of Abuse and External Contracts. All have Principal Scientific Officers in charge in post.

As far as scientific work is concerned one of the main features has been the increased use of computer facilities. The information retrieval service, interrogation of data banks, certain operations of the spark source mass spectrometer and automated liquid gas chromatographic injector are now computer controlled and it is hoped to interface both the inorganic and the newly installed organic mass spectrometer to the computer in the near future for data handling. Clearly, the use of computers in the regional laboratories following the accumulation of data at CRE is going to be a feature of the next few years. Already discussions are taking place on the use of computers by regional scientists in the assessment of more productive routes of analysis and in the statistical interpretation of evidence.

The gas chromatograph – linked to the organic mass spectrometer is proving to be a most useful analytical tool and an evaluation of its potential use in regional laboratories has been made thanks to the co-operation of our neighbours, the Director and Staff of the Home Counties Forensic Science Laboratory. Another technique which has been more extensively studied has been radioimmunoassay particularly in relation to the detection of drugs in body fluids. In 1973, 27 samples of post mortem blood were analysed for digoxin on behalf of regional laboratories.

It has always been the policy at CRE that the research carried out must bear a direct relationship to operational forensic science and one of the major themes in the last two years has been the development of automated devices to reduce tedious manual repetitive work done by forensic scientists. This has been demonstrated in relation to blood alcohol analyses where following an evaluation by CRE of a commercially available automated machine a total of 13 have been purchased by the regional laboratories and the Metropolitan Police Laboratory. It is fair to say that this work has enabled the regional laboratories to keep reasonably in pace with the increasing number of blood samples taken under the Road Traffic Act 1972. This work continues particularly in relation to the design of new containers for blood taken under the Road Traffic Act and into the automatic identification of the sample during analysis.

Investigations into low cost automated liquid injectors suitable for the smaller Home Office Laboratories have also been made and in addition a continuous flow blood alcohol analyser using the ADH system, developed under external contract, is being evaluated in the Chorley Laboratory. The automatic extractor of viscera for poisons is being tested at the Northern Forensic Science Laboratory at Newcastle and the automatic colour test analyser for drugs in tablets, capsules, etc, is being assessed in the Home Counties Laboratory. We are most grateful to the Directors and Staff of these laboratories for their help in the operational research phases of testing. Thanks are also due to those regional scientists who oversee external contracts initiated by CRE.

The positive and practical help given by the Home Office Scientific Advisory Council and its Forensic Committee have continued to be most valuable. It is a great pleasure to record our thanks to all members.

To summarise, despite staff upheavals, the year has been exciting, challenging and productive.

## 2. STAFF DETAILS

We welcome:

Mr R Barrett, a research fellow on secondment from the Department of Science, Commonwealth Customs Laboratory, Melbourne, Australia.

Mr P Burdett, (HSO) from the University of Leeds.

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

Mr V Emerson, (PSO) from the Cardiff Laboratory.

Dr L King, Senior Research Fellow from the University of Loughborough.

Dr Anne Kipps, (HSO) from the University of Reading.

Mr J Sutton, (SSO) from the Metropolitan Police Laboratory.

Mr G Walker, (PSO) from the Nottingham Laboratory.

Dr P Whitehead, (PSO) from the Metropolitan Police Laboratory.

Dr Janet Worthington, (SSO) from ECLP Limited, St Austell, Cornwall.

Departures:

Flt Lt D Blackmore, MBE, who was appointed Principal Biochemist at the Animal Health Trust, Newmarket.

Mrs V Bunker, (ASO) left to live in the Boscombe area.

Dr C Fouweather, (HSO) left to join the staff of the Nottingham Laboratory.

Miss R Hewitt, (SO) left to join the staff of the Cardiff Laboratory.

Mr P Jones, (HSO) left to join the staff of the Harrogate Laboratory.

Dr E Pearson, (PSO) was selected for the course at the Civil Service College.

Dr G Renshaw, (SSO) left to join the staff of the Home Counties Laboratory.

Dr A Scaplehorn, (PSO) was selected for the course at the Civil Service College.

We record the attachments of Miss S Carlisle, from Liverpool Polytechnic, Miss M Dutton, Nottingham College of Technology, Mr T Hayes, PhD Student from Strathclyde University and Miss A Hayler from Surrey University. Mr B Clare from Christ's College, Cambridge worked with us as a vacation student.

Retirements:

Mr R H Fox, (SSO) was retired on medical grounds.



Promotions:

Mr C Brown to SSO

Dr M D G Dabbs to PSO

Mrs D Morgans to SO

Mr J Porter to HSO

Mr C A Pounds to SSO

Appointments and Qualifications:

Dr A S Curry was re-elected President of the International Association of Forensic Toxicologists.

P J Twitchett gained his PhD from the University of Reading.

A E Kipps gained her PhD from the University of Durham.

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

### Lectures given by Staff

The Director and members of staff have given lectures to the following bodies: The Police Colleges at Bramshill, Durham and Tulliallan Castle, Scotland; Wakefield Detective Training School; Hertfordshire Police Drugs Squad Officers Course; University College, London; Royal Aircraft Establishment, Farnborough; London Hospital Medical School; The London School of Hygiene and Tropical Medicine; Trinity College, Dublin; University of Surrey; The Association of Forensic Toxicologists, Ghent; SAG Meeting/Spectrometry Group, Nottingham; Joint BEA/BOAC Airways Medical Association and the London School of Pharmacy.

Dr Curry attended the Swedish Medical Research Council Meeting at Stockholm; the American Academy of Forensic Sciences, Las Vegas; and Bermuda to advise on the setting up of a forensic science laboratory; he headed the British delegation to a Conference on Narcotics and Dangerous Drugs in Paris, and attended the International Association of Forensic Toxicologists Symposium in Ghent.

### Lectures and Courses Attended by Members of Staff

Dr A Patterson attended a course on High Pressure Liquid Chromatography at the University of Sussex; Mr P Gomm, a conference on Future Trends in Automated Analysis at the University of Newcastle; Mr K Smalldon, 'A Systems Analysis for Information Workers' conference organised by the Institute of Information Scientists at Manchester University and the British Pharmaceutical Conference. Dr A Moffat also attended the British Pharmaceutical Conference, London School of Pharmacy and a conference on Cyclic AMP with Mr P Owen at the Middlesex Hospital;
Dr J Worthington attended the International Mass Spectrometry Conference in Edinburgh;
Mr V Emerson and Mr M Swain attended a Specialists' Meeting on New Developments in Storage, Retrieval and Dissemination of Aerospace Information organised by AGARD in London; Mrs A Brech and Dr A Kipps a Gel Filtration and Electrophoresis Course organised by the Loughborough University; Mr P Burdett and Mr J Sutton a symposium on Isoenzymes by the Royal Microscopical Society; Dr P Whitehead and Mr J Sutton a LKB Conference on Isoelectric Focussing in Glasgow, and Dr G Renshaw the 'Analysis 73' Conference in London.

A safety film and demonstration on Artificial Resuscitation techniques were given by AWRE Safety Division.

### Overseas Visitors

A total of 27 overseas visitors from 14 countries visited CRE and Superintendent Arne K Bergh, PhD from the Canadian Royal Military Police spent two weeks with us.



## 4. BIOLOGY DIVISION

This year there has been a disruption of work in the Division because of staff postings, and only those projects completed, or near completion, are described in detail below. However, with the influx of new staff, the opportunity is being taken to re-orientate the Division along new lines of enquiry described under the heading of future work.

### Sexing of Hairs

Previous reports have described the progress and problems encountered in indentifying the sex from which a human bloodstain originated by staining the Y chromosome in interphase leucocytes with quinacrine dihydrochloride. This work has been further continued in a study of sheath epithelial cells from human hairs. It has been found that, providing sheath cells can be recovered from the hair, good discrimination between male and female hairs can be achieved on the basis of Y chromosome staining alone.

However, unlike the situation with leucocytes from bloodstains, with hair epithelial cells there is the possibility of identifying Barr bodies as an independent complementary test of Y chromosome identification. It appears, therefore, that by a combination of chromosome and Barr body identification, hair sexing with a high degree of certainty could be performed in the forensic science laboratory. A day's demonstration of the Y chromosome technique was held at CRE for regional laboratory biologists during the year.

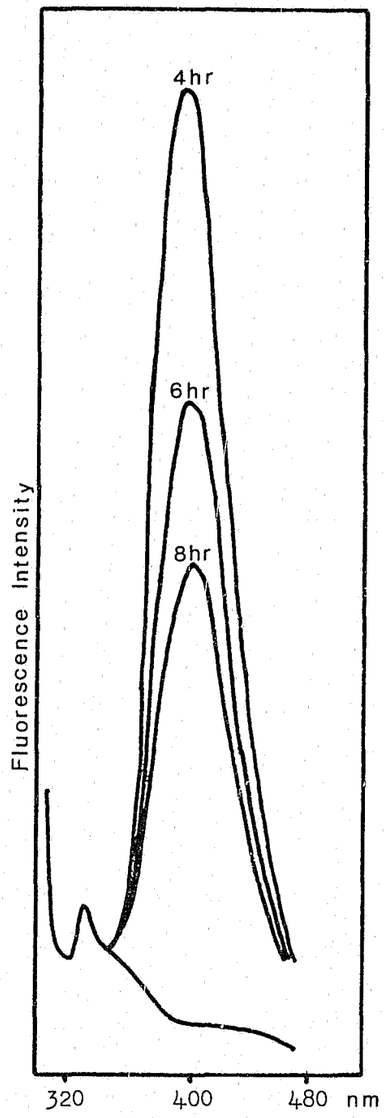
The problem of hair discrimination in forensic science is a very real one (CRE Report No. 98) and any move ahead in this direction is of particular value.

### Species Identification

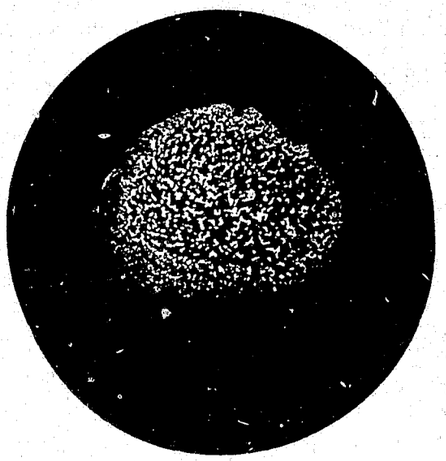
The identification of the species origin of a bloodstain or a piece of tissue remains one of the most basic tasks the forensic biologist has to carry out. Following work started at the Metropolitan Police Laboratory by Dr Whitehead and in collaboration with the Wellcome Research Laboratories a rapid means of identifying the species origin of blood has been developed and evaluated, the method being dependent on sensitised latex particules.

Latex particles sensitised with antibodies directed against a given antigen, will in the presence of that antigen form easily visible agglutinates within minutes of mixing. The reaction is carried out on a glass slide and forms the basis of a number of diagnostic aids in medicine, the rapid diagnosis of pregnancy is an example. The same principle has now been shown to be useful for identifying the species origin of blood or tissue. Latex reagents for identifying proteins from human, cow, sheep, pig, dog, cat, horse, mouse, chicken and guinea-pig have been prepared and evaluated. It is anticipated that this new approach to species identification may also find application in related fields such as meat identification in food and drug analyses as practised in Public Health Laboratories.

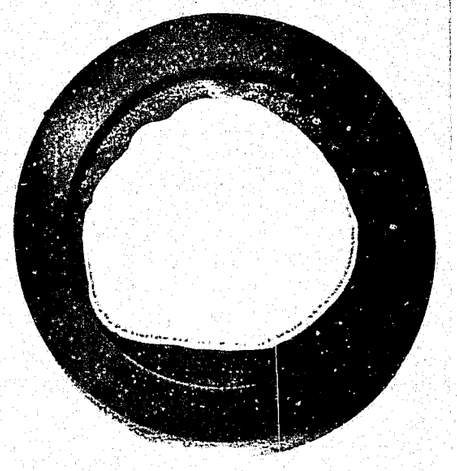
However, it must be borne in mind that the latex technique in no way resolves the problem of the identification of genetically closely related species. Further studies of this problem with respect of avian bloods have been made by using the technique of double diffusion in agar together with a commercially available antiserum of chicken serum proteins. Diffusing extracts of fresh blood and bloodstains from duck, goose, chicken, pheasant, turkey and pigeon against the antiserum from chicken, it was found possible to distinguish between duck and goose, pheasant and turkey as well as differentiating all the other listed avian species (CRE Report No. 78).




Fluorescence spectra of ethanol extracts of stains prepared from 25ul blood, showing times from ingestion of two aspirin tablets.
Excitation wavelength = 300nm.



Agglutination of Latex particles indicating a positive result.



Smooth suspension indicating a negative result.



### Bloodstain Identification Based on Non-Genetic markers

At present, the traditional means of bloodstain characterisation are almost entirely dependent on genetic markers in blood, eg, red cell antigens, but an alternative approach is to consider non-genetic or environmental factors; for example, the detection of therapeutic levels of drugs in bloodstain extracts. Using the specific fluorescence of salicylates in ethanol at 400 nm following excitation at 300 nm it has been shown possible to detect therapeutic levels of aspirin in ethanol extracts of very small bloodstains. As at any one time it is estimated that approximately 3% of the population of the United Kingdom will be taking aspirin, this factor can already act as a further means of bloodstain characterisation. Clearly if a combination of drugs can be detected this would increase the evidential value further, and work is continuing in this direction.

An extension of the above principle could involve the analysis of constituents normally found in blood, eg, urea, glucose, sodium, chloride, etc, and all of the other parameters routinely measured in hospital biochemistry laboratories. However, statistical appraisal of up to 18 such parameters suggests that this type of 'biochemical profiling' involving the measurement of continuous variables, provides less useful information than discrete variables and is considered to be of little value at the present time.

### Future Work

The aim of the Division is still to seek biochemical individuality either by introducing new concepts or developing and modifying traditional approaches.

Great advances have been made in forensic serology in the past decade and the staff of the Metropolitan Police Laboratory have pioneered this work.

In the immediate future it appears possible that the characterisation of a stain may be limited not so much by the number of groups capable of investigation but by the quantity of blood available. In anticipation of this, a programme of research has been started aimed at not only simplifying present electrophoretic techniques, but also making use of micro-fluorescent techniques for characterising different types of antibody.

Other fields demanding further attention are those involving saliva and semen; in contrast to the position with blood, the grouping of these fluids is usually limited to one system, ie, the ABO groups. One reason for this is almost certainly the fact that the investigation of genetic markers in blood has received considerably more attention from medical research workers. As a result a vast body of knowledge has been accumulated on which the forensic serologist, by a combination of skill and ingenuity, has been able to build and develop the specialised field of bloodstain grouping. The detailed study of the genetic markers in saliva and semen has received much less attention. The forensic serologist in order to answer the questions peculiar to his field must be prepared to carry out fundamental research work into the properties of salivary and seminal enzymes and associated blood group substances. It is believed that the automation and quantitation of grouping is a first step in this direction and this work has been initiated both in the Division and by two external contracts.

In addition work has started on a study of those enzymes in semen which may have a genetic origin.

## 5. CHEMISTRY DIVISION

The Division has worked essentially in accordance with the projects outlined in the last Annual Report. Progress was made in most of the projects and it was possible to begin others. This work is summarised below.

### Hair Discrimination

This work (CRE Report No. 98), was undertaken to evaluate certain techniques in discriminating between human hairs. Hair colour was found to be a valuable characteristic, although in many cases the variation in colour of single hairs over an individual head is such that results must frequently be interpreted with extreme caution, although of course the evidential value of colour agreement increases if the colour type is unusual, eg, red or genuine blonde.

Of the other parameters, diameters, medullary index, scale counts and patterns, amino acid composition, pyrolysis gas chromatography, refractive index, fatty acid composition in hair sebum, and low temperature luminescence were all found to be of little value in the discrimination of human hair.

Hair cosmetic treatment can provide useful discrimination and bleached or dyed hairs can normally be easily identified, while in the case of bleaching, it is possible to assess the degree of hair damage by means of a simple test. It was also found that certain constituents of some medicated shampoos tended to persist on hairs and could be readily detected.

### Pyrolysis — Gas Chromatography

Little work on this technique was done during the year in the Division, although rubbers and man-made fibres are the subject of external contracts. The relative discriminatory powers of Porapak Q and Carbowax 20M as stationary phases in polymer analysis were investigated. This confirmed that Carbowax 20M, was in general, superior to Porapak Q for differentiating polymers by their pyrolysis patterns, However, a considerable amount of data on the pyrolysis products of polymeric materials has been obtained using Porapak Q as the stationary phase and it is intended to continue to use this stable and reproducible material.

### Colour Measurement

Numerous advantages would ensue if forensic scientists were able to exchange data on colours, eg, of paint samples; for this to be effectively achieved it is necessary for the colour to be reduced to numerical terms. A number of colour systems were examined, and the Methuen system was finally suggested as a standard colour system for forensic science. Full details are described in Section 8.

During the year, a fibre optics colorimeter manufactured by the Paint Research Association came to our notice. This instrument, designed for use in the paint industry, gives a digital readout of tristimulus colour values. It is extremely sensitive, having a colour discriminatory power of the same order as the human eye. In its original form it is unsuitable for use in forensic science applications because it was designed for situations involving samples of large surface area. The Paint Research Association, at our request, have designed and built a special fibre optics head by means of which samples of the order of 1 $mm^2$ can be measured. A preliminary appraisal of the instrument fitted with this 'micro' head has yielded encouraging results and it is hoped to obtain one in order to test its possible application in forensic science. An instrument, capable of recording colour reproducibility in digital terms, would be of obvious value in enabling the easy interchange of colour data between laboratories.



### Examination of Glass

Work continued throughout the year on building a prototype instrument to measure automatically the refractive index of small particles of glass. It is unfortunate that deliveries of electronic components have been so long delayed that it has not yet been possible to complete this project and put the prototype into routine service in order to evaluate its performance under normal 'case-work' conditions. An external contract has been placed for the provision of a production prototype.

A number of glass samples have now been calibrated for use as secondary standards for measuring the density of glass samples in case-work and these are to be circulated to regional laboratories.

The discriminating powers of density and refractive index measurements for window glass have been studied, from which several points of practical interest have emerged. It has long been known that these properties have been correlated, but it has been shown that, where the errors of measurement are similar, density is approximately six times as discriminating as refractive index, a result that shows good agreement with theoretical prediction. However, it frequently happens that accurate density measurements are difficult if not impossible to achieve on many of the small fragments met with in case-work, whereas these fragments are amenable to refractive index determination.

The MS702 spark source mass spectrometer has continued to be used for the analysis of glass and small surveys have been made of glass from windscreens, vehicle lamps, spectacle lenses and containers. The results of the container glass survey showed that although if one has a full elemental analysis of the glass it is usually possible to decide if a piece of glass of unknown origin came from either a window or a clear glass container, extreme caution is required in the interpretation of the results when only values for a limited range of elements are available. Wide variations in composition were found in the lenses of vehicle lamps.

### Fibres

A small scale study of the trace element composition of acrylic fibres has been carried out using the MS702 mass spectrometer. This survey used large samples and was designed purely to obtain background data that could later be appraised with a view to developing specific methods for useful elements that would be applicable to small samples. This survey did not claim to be exhaustive, but many interesting results were obtained. For example, Courtelle could be differentiated from the other acrylonitrile/methyl acrylate fibres by its indium content, while all acrylic fibres of Japanese manufacturers were found to have a high niobium content.

### Laser Arc Emission Analysis

The evaluation of this instrument has been completed and a report published (CRE Report No. 81). It is possibly of interest to note that in a comparative study of methods of paint analysis, laser arc emission analysis was the most discriminating single technique for the coloured paints studied (CRE Report No. 82).

### Alcohol Analyses

Work on the refinement and automation of blood alcohol analyses is a continuing commitment. The completion of an external contract to develop an automated system of blood alcohol analysis by the ADH method resulted in the delivery of this apparatus to CRE. After an evaluation period and some minor modifications, the apparatus was installed in the North Western Forensic Science Laboratory at Chorley for field trials

Currently the Pye autoinjector is undergoing trials in the Division. This is an 'on-column' injection system with a 100 sample capability. To enable it to be used with blood samples it has been necessary to construct pre-columns to retain the deposited blood solids and with this obstacle now overcome, promising results are being obtained. The use of other column systems eg, capillary columns, to reduce analysis time are being investigated.

**Organic Mass Spectrometry**

Possibly the event of the year was the installation of a Micromass 12 low resolution organic mass spectrometer with a coupled GC system. The inevitable teething troubles associated with the installation of major equipment having been experienced and surmounted, the evaluation of the instrument proceeded smoothly. Initially, emphasis has been placed on drug analyses and there is no doubt that this technique could well become the method of choice for much of the drug identification analysis performed in forensic science laboratories.

In favourable cases, unambiguous identification can be obtained within minutes, whilst in unfavourable instances, the time involved is no longer than that required by alternative techniques.

A four week trial was conducted jointly with the Home Counties Forensic Science Laboratory, when all suitable drug cases were analysed by mass spectrometry. This trial showed the capability of the instrument to deal with this flow of work and produced good results for both laboratories. During the course of this trial, a case involving atropine in urine was encountered; the value of mass spectrometry in identifying the presence of a previously unsuspected drug was clearly demonstrated.

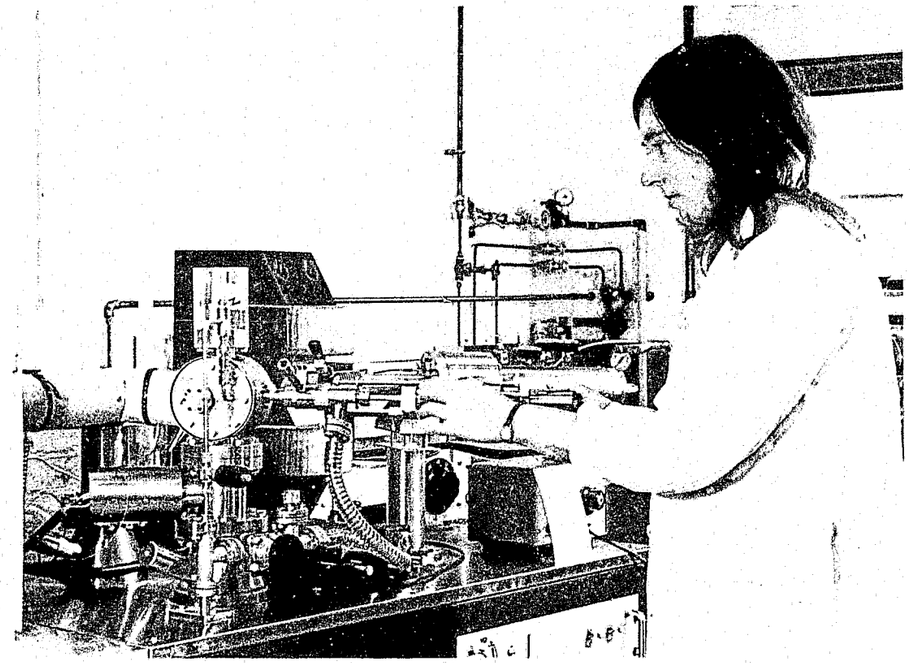
Although to date most the work on this instrument has been directed towards drug identification, minor investigations, such as the examination of the pyrolysis products of paints have shown its potential in other fields while a major investigation into the organic constitutents of gunshot residues is planned. The instrument has also been used to study the pyrolysis products of cannabinols.

**Soil**

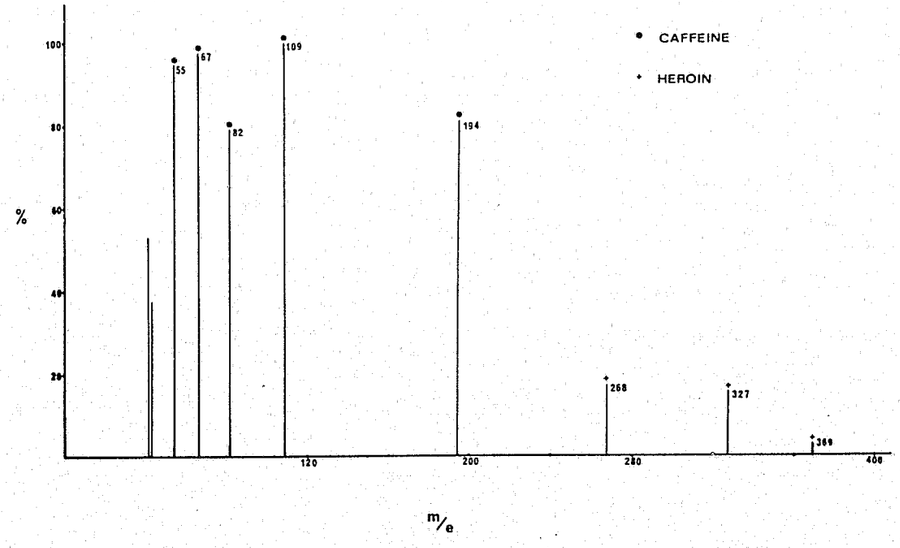
Work has just commenced on assessing the various methods, used or proposed, of examining soil in the context of forensic science.

As a start to this programme several hundred topsoil samples, taken from an area near Reading by the Soil Survey of England and Wales (Ministry of Agriculture), are to be examined for variation between samples. The aspects it is hoped to study initially are colour, particle size distribution and cathodic luminescence: apparatus is being constructed for examinations to be done by the last technique.





The Micromass 12 low resolution organic mass spectrometer, with a coupled GC system facility.




Normalised organic mass spectrum of a typical sample of adulterated Heroin.

## 6. DRUGS OF ABUSE DIVISION

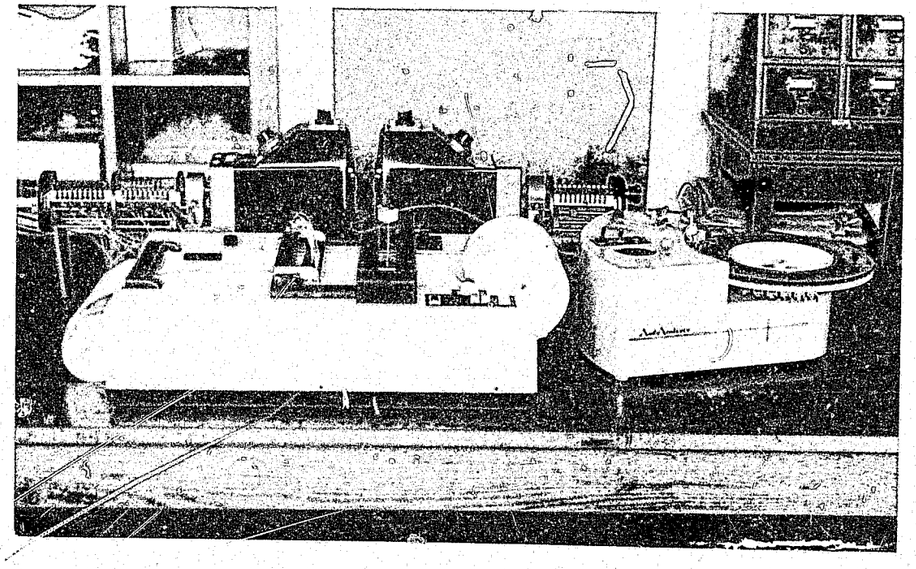
### Automatic Colour Reaction Device

In 1972, the construction of equipment for the automatic screening of drug samples, based upon chemical tests, was begun. A prototype device was assembled in which a drug extract was applied to absorbent paper in the form of a narrow band. This was then moved under a gantry of pens, each delivering a different test reagent and at the intersections of drug extract and reagents, colours were produced.

Further development has continued satisfactorily. Patents have been filed in the United Kingdom and the USA and the National Research and Development Corporation has undertaken its commercial exploitation. A paper describing the operation of the prototype is in press.

The construction of two professionally engineered versions was started under contract early in the year. One of the contractor's products has been delivered and offers a number of facilities not incorporated in the prototype. These include pre-selection of the number of spreads of drug sample extract, the spreading distance of the sample on the paper, and sequential numbering of reaction profiles. After a period of trial and adjustment, the instrument was tested operationally at the temporary laboratory set up by the Home Counties Forensic Science Laboratory for the Reading and Windsor Pop Festivals. When routine analyses were complete, samples were submitted for automatic analyses. Using thirteen chemical tests (Table 1) seventy tablet, powder and capsule samples were analysed during the four day period. Although working well below maximum capability, mechanical operation was faultless.

The device is now beginning a period of more prolonged operational trial in the environment of a regional forensic science laboratory and we are grateful to the Home Counties and Metropolitan Police Laboratories for agreeing to participate in this work. Without wishing to prejudge events, it is anticipated that the major difficulty likely to be encountered will be associated with the necessity to analyse drug samples of widely varying and unknown concentrations.



Automatic Colour Reaction Device



TABLE 1

Colour Tests in Use

| Colour Test | Colour Produced | Example of Compounds Detected |
|---|---|---|
| (1) Ferric Chloride | Various | Phenolics — Salicylates |
| (2) p-Dimethylamino-benzaldehyde | Blue-Violet | Ergot Alkaloids — LSD |
| (3) Iodoplatinate | Dark Blue | Bases — Quinine |
| (4) Cobalt Thiocyanate | Turquoise | Amines — Diphenhydramine |
| (5) Dragendorff | Orange | Bases — Nicotine |
| (6) Palladium Chloride | Dark Red | Sulphur — Compounds — Phenothiazines |
| (7) Perchloric Acid/ Ferric Chloride | Red Brown | Indole Derivatives — Tryptamines |
| (8) Vanillin | Yellow | Sulphonamides |
| (9) Fast Blue Salt B/ Sodium Hydroxide | Various | Phenolics — Cannabis Compounds |
| (10) Cobalt Nitrate/ Isopropylamine | Violet | Barbiturates |
| (11) Potassium Ferricyanide/ Ferric Chloride | Dark Blue | Reducing Compounds, Amines, Tryptamines |
| (12) Iodic Acid/Ammonia | Red | Morphine |
| (13) 2,4,6-Trinitrobenzene Sulphonic Acid/ Sodium Hydroxide | Orange | Primary Amines — Amphetamine |

### Drug Abuse Trends

Work has continued in monitoring the trends in drug abuse in the United Kingdom. Regular weekly contact has been maintained with the regional forensic science and police laboratories and with the Laboratory of the Government Chemist. This resulted in the notification of 566 cases considered to be of significance and in 203 instances samples were submitted for comparative analysis.

Procedures have been established for both physical and chemical comparisons. Low power microscopy, the use of the Projectina comparison microscope, the observation of tablet punch marks, the identification of capsule locking types and the identification of starches, sugars and dye-stuffs provide the basis for this work. In some cases quantitation of constituents has been carried out and in January, for example, the results of analyses for 50 diamorphine samples seized during the years 1969/71/72 were reported.

Of the samples examined during the year, 28 were designated as new drug abuse preparations with at least some element of national distribution. Examples of less usual drugs of abuse encountered were 4-bromo-STP, α-methyl tryptamine, methylene-dioxyamphetamine, phencyclidine and 'hashish oil'.

During March, the Central Drugs Intelligence Unit was constituted and close co-operation has quickly developed. Through their auspices, fruitful communication with Drug Abuse Agencies in other countries, notably Australia and North America, has developed.

Five issues of the new Divisional Newsletter 'Drugs Abuse Trends' have been produced during the year.

**Identification of Stereo-Isomers**

Unlike their dextro-isomers, levorphanol and levomethorphan are controlled under drug abuse legislation, but, except where substantial amounts of material are available, an unequivocal method for differentiating between the pairs of optical isomers has not previously been reported. During the year, possible solutions to the problem, such as the use of polarimetry, microcrystal tests, chromatography, electrophoresis and circular dichroism have been investigated.

Polarimetry is inherently insensitive and microcrystal tests, though highly sensitive, suffer the disadvantage that much experience is needed by the analyst if reliable and unambiguous results are to be obtained. Some success has been achieved by the use of low pressure liquid chromatography using starch columns but the method is slow and we look forward to the availability of high pressure equipment.

Circular dichroism offers the best solution so far. The phenomenon is observed when a compound exhibits different extinction coefficients in light which is circularly polarised in opposite directions. Fortunately, the two pairs of isomers of interest exhibit the phenomenon to an extent which allows differentiation with less than 100 ug of material using standard sample cells. The use of microcells could of course further enhance the sensitivity.

We are extremely grateful to Westfield College, London, for useful discussions and the use of their Circular Dichroism Spectrometer.

**Pyrolysis of Cannabinols**

Cannabidiol (CBD), the two major isomers of tetrahydro-cannabinol ($\Delta^8$–THC and $\Delta^9$–THC) and cannabinol (CBN) have been pyrolysed by use of Curie Point and hot wire devices and also by heating to 500°C in a sealed, evacuated, Pyrex tube. Separation of the products has been achieved by temperature programmed gas chromatography on 2½% OV17, and identification is proceeding using the GC-linked mass spectrometer. It is anticipated that further separations using a Carbowax 20M column will be necessary to allow study of the lower molecular weight products.

This work has been done by Mr R M Barret, a research fellow on secondment from the Department of Science, Commonwealth Customs Laboratory, Melbourne.

**Reference Standards**

The collection of reference standards and their associated analytical data has continued, the immediate objective being to obtain samples of all substances scheduled in the Misuse of Drugs Act which came into force during July. By means of an external contract with the University of Aston and the co-operation of the pharmaceutical industry we are now in possession of 84% of these and it is hoped to complete the task during the forthcoming year.

Ultra-violet, infra-red and mass spectra, together with thin-layer chromatographic data are being compiled and a computer retrieval programme is being assembled.



## 7. EXTERNAL CONTRACTS DIVISION

External Contracts Division was first formed as a separate entity in June 1973. Previously external contracts were supervised by Information Division but because of the increasing work load in the Division it was decided that the creation of a separate External Contracts Division was desirable.

External contracts are necessary because in certain areas of research CRE does not have the staff or facilities to undertake all the research work that is required in the Forensic Science Service. This is particularly true when it involves data collection or research in highly specialised fields.

Contracts are generally placed for three different types of work.

### 1. DATA COLLECTION

The collection of data forms an important part of forensic scientific investigation in determining the significance of a piece of evidence. The following projects are being undertaken.

(a) A system for the retrieval of analytical data on drugs covered by the Misuse of Drugs Act 1971.

(b) Collection of examples of men's patterned sole units from all UK manufacturers in the Shoe Trade Directory. A coding system is also being devised.

(c) Pyrograms of a range of natural and synthetic rubbers and rubber mixes.

(d) Pyrograms of a wide range of fibres.

(e) Patterns of all car and truck tyres readily available in the UK.

(f) The compilation of data on all the various treatments used on fibres during manufacture of an article.

(g) Photomicrographs of wood sections for softwood identification.

### 2. CONSTRUCTION PROJECTS

As a result of research in the regional forensic science laboratories and CRE, it is sometimes necessary to construct a particular piece of apparatus. Prototypes of different degrees of sophistication are sometimes constructed at CRE but a fully engineered model needs to be made by an outside contractor. The following projects are being undertaken.

(h) A multi-element Atomic Absorption Spectrometer for the analysis of 12 elements simultaneously.

(i) An automatic colour reaction machine for the preliminary screening of tablet extracts for a variety of drugs.

(j) An automatic saliva grouping apparatus based on the Technicon system able to group a very large number of samples on a continuous basis.

(k) An apparatus to automatically give warning when the level of solvent in a beaker has been evaporated down to a pre-determined level.

(l) An apparatus for the automatic extraction of drugs from urine. The drugs are separated into strong and weak acids, neutrals, bases and morphine.

(m) The construction of a prototype production automatic refractometer based on the laboratory prototype constructed at CRE. This apparatus will automatically measure the refractive index of small glass fragments.

(n) (2 contracts) The construction of the necessary interfaces to link the organic and inorganic mass spectrometers to the HP2100A computer.

(o) An apparatus for the automatic grouping of saliva stains using radio-active labelling techniques. The apparatus is being designed to deal with small batches of samples rather than the very large numbers envisaged for the apparatus described in (j).

(p) Prototype apparatus to increase the capacity of the Perkin-Elmer F40 Gas Chromatograph from 30 samples to 200 samples.

## 3. FUNDAMENTAL RESEARCH

The forensic scientist is constantly searching for new ideas and techniques to solve his varied problems. This must involve some investment in long term speculative research. The following contracts are aimed in this direction.

(q) An investigation of the effect of certain drugs of addiction on various enzyme systems with a view to finding new methods for the detection and estimation of drugs in biological materials.

(r) The development of an analytical procedure to detect, identify and quantitate anti histamines in blood after the adminstration of therapeutic doses.

(s) The discrimination of blood stains by the analysis of protein levels.

(t) The use of birefringence in the examination of fibres.

At the end of September 1973, twenty-one contracts (seven with universities, fourteen with companies) were in force. Six contracts have been completed during the year, a further three have been negotiated and are awaiting Home Office approval and a further one is being negotiated. All contracts are supervised by regular visits to the contractor.

As a result of the work that has been carried out both at CRE and under contract it has been found worthwhile to apply for several patents. In addition some projects are being commercially exploited by NRDC.



## 8. INFORMATION DIVISION

The Division's work, considerably extended as reported in the last Annual Report, continued along similar lines until June of this year when the work of external contracts was transferred to a new Division and the section of Chemistry Division dealing with the collection and collation of data on paint and glass was transferred to Information. This formed a larger but more compact section dealing with data banks. The Division can now be split into five separate sections each dealing with various aspects of the broad based information theme.

### Information Collection and Presentation

The Establishment takes 60 journals which are scanned by the staff and relevant papers are extracted. A chemistry profile is run biweekly on the UKCIS Chemical Condensates and Chemical Biological Abstracts. A biology profile is also run at similar intervals on the Biological Abstract previews. These profiles each produce approximately 100 titles a month which are also scanned by CRE staff and the extracted papers are examined and decisions taken as to their relevance. In addition to this, Current Contents (Life Sciences) and other governmental establishments' publication lists are searched.

Those papers that are considered of immediate importance are sent to all the regional forensic science laboratories in the monthly 'Current Awareness' circulation. Others which may be of interest to the regional scientists and to the researcher both present and future are stored in the Division. In this way, approximately 200 papers are collected each month to which a computer accession number is allocated. Every paper is then 'keyworded' and the accession number together with its relevant keywords are stored on the computer. This was achieved on a time-shared Burroughs Computer in Brussels by means of a Telex link until July of this year. At that time, 10,660 stored records were transferred to the Hewlett Packard 2100 mini-computer at CRE. This has enabled modifications and extensions to be made to the program, the literature information bank now contains 12,020 records, and a reduction in the overall running costs has been achieved.

All these records have been microfilmed and copies sent to the regional laboratories. Regular updating is undertaken. A scientist in a regional laboratory with an enquiry, has access to the computer indexing system by means of a telephone call to CRE Information Room. Within minutes he can be referred to the relevant microfilmed papers and in this way has ready access to all the Information processed at CRE.

Use has been made of the Information Room and its staff by many people from establishments outside the Service. Enquiries and requests for papers have been received from Hong Kong, Israel, South Australia, Canada, Bermuda and the FBI as well as other law enforcement agencies in the United States. In view of this it is hoped that a package may be made available commercially which will include a magnetic tape of data and interrogation program together with a microfilm which will relate computer accession numbers to author, title and journal from which the papers were extracted.

The Library collection continues to grow and we now have 450 books together with a great many bound volumes of journals, some kindly donated by regional laboratories. In addition the Sadtler Collections of ultra-violet, infra-red prism and infra-red prism commercial spectra are a common service. The library is now very short of space.

### Implementation of Data Banks

During the year progress has been made both in the consolidation of existing collections and in the application of computer retrieval to reference data collections. Microfilms of infra-red and ultra-violet spectra already circulated to regional laboratories have been updated by means of microfiche. These consist of 2876 and 1020 spectra respectively, the former has also now been stored on computer by means of the six largest peaks. A program is available for searching this file for unknowns; thus

once again by means of a telephone call to the Information Room, a regional scientist can reduce an unknown to a few possibilities. The final identification by means of the microfilm and/or his own spectra is left to the scientist himself.

A file of glass refractive index data, provided by all the regional laboratories from case-work, is being maintained. A program (CRE Report 87) has been written which determines the frequency of occurrence of a particular glass at a given RI or range of RI's.

The vehicle headlamp and auxiliary lamp collection originally containing 178 lamps is at present being updated. The range of manufacturers has been expanded to include the Japanese and East European vehicles, and the collection is being made more comprehensive by including all headlamp lenses available in the UK since January 1962. There is likely to be an additional 100 lamps added to the collection and photographs of these will be circulated together with the information as to vehicle or range of vehicles on which the lamps are used.

The investigation into a system for the comparison of colours of materials such as paint, fibres and hair has continued, the last two presenting great difficulties because of the size of sample normally encountered in case work. For paint, however, a system has been proposed, which can also be used, to a more limited extent, for fibres. In this system the samples are described by reference to a three dimensional colour block, the co-ordinates being hue, intensity and tone. These are represented by 1,266 coloured chips in the Methuen Book of Colour which was chosen from the other systems considered because of its low cost, ease of use and understanding. It also provided sufficient colours to produce a workable system.

In a series of trials in which over 800 colour comparisons were made, and in which the regional laboratories played an active part, it was found that for control samples of at least 1 $cm^2$ area an error of 1.1.1 (ie, one colour chip in every direction) included 90% of the comparisons.

Having thus established the reproducibility of the system for control samples a bank of information of paint colours for both cars and houses was established. This has been circulated to the regional laboratories with a request that they add their own control samples to the collection, which after a period of six months can then be collected for updating purposes.

The information on the frequency of occurrence of house and car paint colours (CRE Reports 85, 86 and 96) has been made available for computer retrieval. Three files of data have been created:

1. A simulated Methuen book for the occurrence of house paint colours (1,453 items).
2. A simulated Methuen book for the occurrence of car paint colours (514 items).
3. A file of data relating car manufacturers and ICI Reference number to Methuen notation (2,141 records).

Programs are available to search, update and modify all these files.

A collection of over 200 agricultural chemicals has been made, and with the assistance of regional laboratories, ultra-violet and infra-red spectra together with extraction data are now available for these samples.

A collection of over 2,000 pharmaceuticals has been made and it is hoped that liaison with manufacturers can be established so that samples of new drugs together with their analytical data can be made available to the regional scientists as soon as such pharmaceuticals are marketed.

Collections and indexing of boot, shoe and tyre patterns are being undertaken by external contracts and it is hoped that these will be available for computer accession and distribution in the near future.



### Communication Links

A detailed appraisal of the use of computers in and by the regional forensic science laboratories is being carried out. Obviously it would be preferable for such laboratories to have direct access to all the data banks established by CRE, however, in the short term it is not possible for nine laboratories to use the HP2100 on a time-sharing basis. It is felt that greater use would be made of the computer facilities if the human link via a telephone was removed. In an attempt to gauge the truth of this feeling and to establish the preferable form of inter-laboratory communications the following systems are under active consideration. It is hoped that trials will be organised to test them in the near future:

1. Telex links between two regional laboratories and CRE.
2. Telecopier machines in two regional laboratories and CRE.
3. A direct link between a regional laboratory and a central computer on a limited time-sharing basis.

In addition to the projects already outlined above, the Information Division was also responsible for the organisation of the six colloquia held last Winter, the details of which are shown in Appendix D and the attachment of twenty-four regional forensic scientists who each spent one week at CRE. Also in co-operation with the regional laboratories and six police forces a series of 20- minute video tapes dealing with various aspects of searching scenes of crime have been made since 1971. A list of these is shown in Appendix F.

### Quality Control

The quality control programme has been maintained during the year and a number of different materials have been circulated to the regional laboratories for examination. The trials included quantitative alcohol determinations on blood samples, examination of deflated car tyres and measurement of absorption wavelengths of drug samples both in the infra-red and the ultra-violet. In the trials the blood alcohol samples were submitted to the laboratories by the local police force as "routine' cases.

During the year a progress report on all the quality control trials conducted prior to July 1972 was prepared and circulated to the regional Directors. This included the determination of density and refractive index of glass samples, the grouping of blood and saliva, the qualitative and quantitative determination of barbiturates, the estimation of carboxyhaemoglobin in blood and the identification of drugs of misuse both in powdered form and in urine.

A complete summary and statistical analysis was prepared at the end of the tenth blood alcohol trial and this was also circulated to the regional Directors.

In most of these trials, the various quality control committees have played an active part, not only in the design of the trials, but also in suggestions for improvement of analytical techniques arising out of the results. We would like to express out thanks to the members of those committees, the composition of which is shown in Appendix E.

### Systems Analysis

Currently more than 100,000 cases are completed annually by the nine forensic science laboratories in England and Wales about half of which are blood alcohol analyses under the Road Traffic Act 1972. However, the case files created in each laboratory and the wealth of information they contain cannot readily be extracted at the present time.

The information they contain would be valuable for the following purposes:

1. The planning of research policy at CRE.
2. The feedback of valuable technical information to the laboratories themselves.
3. General management planning within the forensic science service.
4. The production of Home Office statistical returns.

A reporting officer in these laboratories is fully conversant with all the details of the case immediately prior to writing his report and it is proposed that at this stage he should complete a questionnaire attached to the case file. The design of this questionnaire and the subsequent collation of the information provided by it are under active study.

The following constraints are being applied to this design:

1. The information must be relevant to the points previously mentioned.
2. The amount of time needed to complete the questionnaire must be kept to a minimum.
3. The necessity for such a scheme must be apparent to the reporting officer.

At the present time, half-yearly statistical returns are made to the Home Office by each laboratory; such returns are processed manually. In the fields of glass and toxicology, technical information is already being collected routinely and with these as a starting point, several forms for information collection have been proposed. We are grateful to the staff of CRE and the various quality control committees listed in Appendix E for their help and active co-operation in the design of these forms which in some cases are already being used in limited pilot trials. It is expected that such trials will assist in deciding if the information requirements can be met under operational conditions. The regional scientists responsible for their completion will be able to make constructive criticisms of their design at an early stage. In this initial stage the data handling will be mainly manual.

Stage two will be the investigation into whether the handling should be manual or computer based, the design of optimum data collection cards and an appraisal as to whether all parts should be operated continuously or at sampling intervals.

Stage three will be the implementation of such a scheme.

By the time the system is operational, the results of the communication links' survey will be complete and all laboratories should have efficient access to the information within the data banks.

### Analysis and Searching Techniques

In forensic science where the amount of sample available for analysis may be limited, and where there is an ever increasing number of analytical techniques available for the analysis, it is important that maximum discrimination be achieved with minimum effort. Therefore, it is desirable that a measure be put on the amount of discrimination which can be achieved for a particular attribute or series of attributes.

This introduces the concept of discriminating power (DP) which is defined as the probability with which two samples selected at random can be discriminated. During the year a mathematical model has been developed which enables the discriminating power for any series of correlated attributes to be determined using the HP2100.

This concept of DP has been applied, with the help of the Toxicology Division, to the selection of TLC and GLC systems for the identification of basic drugs. Systems with high DP's have been selected and the marginal increase in DP's obtained by using additional correlated systems has been clearly



demonstrated. In the case of glass the two main attributes being refractive index and density, it has been demonstrated that the latter has the higher DP, and little increase is obtained by measuring both.

An appraisal of blood group systems using the same concept has begun in order to determine the cost effectiveness of those systems in current use. Although population statistics are accurately known for most blood group systems, no statistics appear to be available for the incidence of blood and other body fluids on clothing not related to crime. As a result of this, a survey has been carried out in an attempt to determine the incidence of blood and semen on male clothing, by using simple tests for peroxidase and acid phosphatase activity (CRE Report No. 104).

One hundred jackets and one hundred pairs of trousers which had been submitted for dry cleaning were examined for bloodstains, of these 58 were matching pairs, ie, were suits. A total of 13 blood stains were found on five of the one-hundred jackets, twelve of these stains were in the jacket linings. The remaining stain on a lapel, approximately 9 $mm^2$ in area represented the only blood stain found on the outside of the jackets examined.

A total of sixty-two blood stains were found on sixteen of the one-hundred pairs of trousers examined. The position, maximum dimension and area of each stain was recorded. Of the sixty-two blood stains found on the trousers thirty were found on the outside. The incidence of blood stains on trousers is therefore much higher than that on jackets. Similar tests were performed on fifty pairs of both men's and women's shoes arriving at a shoe repairers. Although no stains were visible, one pair gave a positive presumptive test for blood.

The inside and outside fronts of one hundred pairs of men's trousers were also examined above the knee. Acid phosphatase was detected on forty-four pairs and thirty-two of these showed at least one area of intense activity some of which were quite large. Although it is realised that this may be a biased sample because stained trousers need cleaning, it does represent an upper limit for the incidence of stains on men's trousers in this survey.

Forensic scientists have been searching clothing for contact traces for many years and by various methods they have been able to remove such traces to their own satisfaction. However, little or no work has been published on this topic and the following questions need to be answered.

1. What number and size distribution of samples can be expected to be transferred to various areas of the offender?
2. How do these decline with time after the initial contact?
3. How can such traces be preserved between the collection of items and searching?
4. What is the best method for collecting such traces?

Initially, a survey of packaging and searching procedures was made in four forensic science laboratories. As a result of this it was decided to concentrate on the two evidence types, glass and fibres.

The number and size distribution of glass fragments found on the ground at various distances and angles from windows at which bricks were thrown have been studied. The results have shown that beyond 1.5 metres from the window the majority of glass fragments were in the size range 0.1 to 0.5 mm. The tests were repeated with individuals standing at different positions in front of the window. The position, number and size distribution of fragments found on the clothing were recorded and found to be similar to that on the ground at similar positions. This size distribution, is in fact similar to that found on clothing not related to crime (CRE Report No. 40). Most fragments were found on the surface of the garments rather than in pockets and turn-ups, however, in each case fragments were recovered from hair combings. Samples of glass from the backward fragmentation experiments were used to determine the persistence on clothing. The results showed that the larger the fragments the shorter their persistence, and that fragments persisted longer on sweaters than on jackets. Although neither of these results is unduly surprising, the fact that only about 10% of the small fragments and 2% of the larger fragments were still on a jacket after one hour is significant. This indicates that really valuable information may be provided by the finding of glass on the surface of a suspect's clothing.

Samples of glass from backward fragmentation are also being used to determine the efficiency of searching techniques, but this work is still in progress.

The study in relation to fibres is a very difficult one; very little is known concerning the factors which control the transfer and persistence of foreign fibres on clothing. In order to observe the transferred fibres, samples of wool and acrylic material have been tagged with a fluorescent dye, thus enabling individual fibres to be readily visible under ultra-violet light. These materials have been knitted into squares which have been pinned to polystyrene blocks and thus used as a standard method of transference. The number, size distribution and persistence has been recorded for transfer to wool and acrylic garments.

The results have been surprisingly reproducible and virtually independent of type of fibre, type and material of garment and surface characteristics. Approximately 80% of the foreign fibres were lost in the first two hours although a small percentage persisted for a lot longer. There is still a great deal of work to be carried out, but these intitial results suggest that the forces controlling the transfer and persistence of fibres may not be purely mechanical. Electrostatic attraction and charge decay are at present under investigation.



## 9. TOXICOLOGY DIVISION

The work of the past year has seen the conclusion of some short-term projects and an increasing commitment into the radioimmunoassay of drugs in blood samples. In addition, assessments have been made of certain current methods used in the regional laboratories such as those which are used to isolate drugs from viscera.

By employing resources in this manner it is hoped not only to provide toxicologists with new methods of detecting and assaying drugs, but to point the way to the most efficient and economical use of existing methods with regards to both time and manpower.

The progress which has been made during the year with the various topics of research is described in greater detail in the following pages.

### 'Difficult' Drugs

*Quaternary ammonium compounds.* The method of ion-pair-extraction of these compounds from body-fluids and tissues using bromothymol blue was outlined in the Annual Report for 1972.

The technique proved of use in examining some tissues received from a regional laboratory for the weedkilling compound Paraquat ('Weedol') in a case of fatal ingestion.

Paraquat could be obtained from the filtrate, obtained after deproteinizing a liver macerate with sodium tungstate and sodium bisulphate, by adjusting it to pH 7.5 and after addition of bromothymol blue solution, carrying out extraction of the ion-pair complex with dichloromethane. Paraquat could then be obtained from the concentrated dichloromethane extract either by running it on the TLC systems described in CRE Report No. 83, or by shaking it directly with a small amount of 0.5N sulphuric acid.

A case was received from a regional laboratory in which the highly potent muscle relaxant Pancuronium was possibly involved. From circumstantial evidence it was thought that not more than 8mg of the drug could have been injected into the arm.

It was found that Pancuronium could not be extracted by dichloromethane in the form of a bromothymol blue complex, but as with other large-molecule quaternary amines like Tubocurarine it could be extracted by the same solvent as the iodide from alkaline deproteinized filtrates.

Like so many compounds of its class, Pancuronium does not absorb UV light and so detection was made on a TLC plate using the colour reaction (purplish-brown) with potassium iodoplatinate.

From visual comparison with spots of standard solutions of Pancuronium run concurrently on the TLC plate, and from studies on the recovery of the drug after addition to visceral slurries it was calculated that the 200g liver sample contained about 50μg of Pancuronium. None could be detected in the urine (100ml) or blood (40ml) of the deceased person. Pancuronium, unlike many other compounds of its class is stable to alkali, but not to hot acid. The protein precipitation therefore had to be carried out at room temperature to avoid decomposition of the drug.

The work on quaternary ammonium compounds to date has opened up a new approach to the screening of tissues and body fluids for this class of drug. Many quaternary amines are highly toxic and the detection of the small amounts involved (micrograms) is often rendered more difficult by their chemical inertness typified by lack of UV absorbance, and poor response to colour reagents. All of them are very water soluble and cannot be extracted directly into organic solvents. The ion-pair extraction studies have indicated that large-molecule quaternaries are most effectively extracted as the iodide whereas for the small molecule ones (eg, suxamethonium, paraquat), extraction as their bromothymol blue complexes offers the best chance of isolating them.

*Fluoroacetamide.* This very poisonous compound is the active constituent of the rat-bait 'Fluorakil' and cases of fatal animal poisoning have been reported with it. Although it is very strictly controlled, it is used to kill vermin in sewers and ships and in our experience, rigid control of poisons does not completely rule out their use in criminal poisoning.

The extraction and detection of fluoroacetamide from tissues incurs inherent difficulties arising from the compound's water solubility, its complete inertness *per se* to colour reactions, its lack of absorbance in the ultra-violet and the very low tissue-levels expected on account of its high toxicity. Experiments designed to detect fluoroacetamide on a TLC plate by spraying it with a phenoxyethanol – silver nitrate solution followed by UV irradiation, were not successful.

A general colour-test for amides was modified for application to TLC plates, and this was found to detect spots of fluoroacetamide containing approximately 5$\mu$g of the compound. The plate was sprayed with an acid solution of hydroxylamine hydrochloride and heated. This treatment converted the amide spot to a hydroxamic acid. This was then over-sprayed with aqueous ferric chloride which produced a pink to magenta colour.

After addition of fluoroacetamide to water or urine, saturation with potassium carbonate and shaking with 2 equal volumes of dichloromethane extracted all the poison. With blood containing added fluoroacetamide, deproteinization with sodium tungstate/sodium bisulphate followed by the treatment of the aqueous filtrate as described above proved successful. Fluoroacetamide is a neutral compound and can be extracted from acid or alkaline solution, but the latter gave a clearer extract.

Future work will be directed at developing a clean-up procedure prior to extraction of blood containing low levels of fluoroacetamide, and also to invoking the use of organic mass-spectrometry to confirm that the coloured spot obtained on the TLC plate is from fluoroacetamide. It is also hoped that this work will be expanded in due course to include the mono-and trifluoroacetates.

*Assessment of protein-precipitation techniques.* (CRE Report No. 97). Six different protein-precipitating techniques were investigated to compare their efficiency at releasing thirty-six basic drugs which had been added in turn to blood samples. After protein-precipitation by each of the agents under study, the acid filtrate was given a clean up with ether, and then made alkaline with ammonia and extracted with the organic solvent in which the drug under test was most soluble. The solvent was then washed by shaking it with water and the drug extracted from it by shaking with 0.5N sulphuric acid. This acid extract was then examined in the UV spectrophotometer in the spectral range appropriate to the drug under study.

The protein-precipitation methods examined were (1) sodium tungstate/bisulphate at 90°C (Valov); (2) N hydrochloric acid saturated with ammonium sulphate at 60°C (Nickolls); (3) N hydrochloric acid containing aluminium chloride (5% w/v) and citric acid (5% w/v) at 60°C (Stevens); (4) 5-7N hydrochloric acid at 90°C (Dubost-Pascal); (5) Trichloroacetic acid solution (10% w/v); (6) Perchloric acid solution (10%) at 60°C (Neuberg-Strauss).

Results showed that protein-precipitants based on hydrochloric acid (3 and 4) produced the highest recoveries of the largest number of drugs added to the blood samples, while inferior recoveries generally resulted where an oxyacid or its salt (such as $WO_4^{=}$, $SO_4^{=}$. -COOH, or $ClO_4^-$ in 1, 2, 5 and 6 respectively) was used as precipitant.

A guide was also provided as to which drug(s) would not survive the conditions of any particular method. Phenothiazines were converted to sulphoxides by all methods where hot dilute acids were used (ie, all methods except 4), but in method 4 where more concentrated acid was used the sulphoxides reverted to the parent compound. Dextropropoxyphene was stable to hot dilute acids but was completely decomposed in method 4 yielding an unsaturated product possessing 20 times the UV-absorbance of the parent drug.



**Putrefaction**

Data on the basic compounds which result when liver samples were 'artificially' putrefied by leaving samples for long periods in a refrigerator at 4°C, in a container in the open air at 23°C for 2-3 weeks and buried in the ground for 2 weeks were obtained. In addition liver samples from eight decomposing corpses were also analysed.

All the samples from the corpses yielded varying amounts of one or more of the six main compounds which had been encountered already during the 'artificial' putrefaction namely nicotinamide, 1- and 2-phenylethylamine, thymine, tryptamine and tyramine.

Results of the putrefaction project have proved of use on several occasions in regional laboratories when endogenous substances have interfered with the identification of drugs in viscera, and indeed in determining whether a basic substance encountered is a drug or a product of putrefaction. This was particularly useful in the investigation of a fatal aircraft accident carried out in conjunction with the Royal Air Force.

**Radioimmunoassay**

The previously used radioimmunoassay for digoxin involving the use of $\beta$-emitting tritium labelled digoxin has been replaced by a method involving $\gamma$-emitting $^{125}I$ labelled digoxin. Only four hours are needed for the completion of an assay compared to the four days previously required with the $\beta$-emitter.

The sample of blood is centrifuged to obtain the serum and this is then serially diluted to provide dilutions from 1:2 to 1:16. The serum radioactive digoxin (containing a digoxin-tyrosine conjugate labelled with $^{125}I$) and digoxin antibody are incubated at room temperature for 1½ hours and then centrifuged to separate the antigen-antibody complex. A scintillation counter is used to count the radioactivity present in the precipitated complex. The amount of radioactivity finally bound to the antibody is inversely proportional to the amount of digoxin present in the sample. A calibration curve (curve A in Figure 1) is constructed using known concentrations of digoxin in serum and an unknown serum solution is assayed with reference to it. The results for dilutions agreed to within ±10% for the assay to be satisfactory. At the moment the inter-assay coefficient of variation is 8.4%, but it is hoped to reduce this if an automated solid scintillation counter can be purchased for $\gamma$-counting.

The specificity of the method has also been improved compared to the previously used method. Other cardiac glycosides such as digitoxin and ouabain which are chemically very similar to digoxin (Figure 2) do not interfere significantly in the assay for digoxin (Figure 1). However, both lanatoside C and deslanoside both bind to the digoxin antibody to the same extent as digoxin because all three molecules have the same genin, differing only in the sugar chain. This enables the assay to be used for all these three drugs although differentiation between them is impossible with the present antibody.

During the last year 27 samples have been analysed for cardiac glycosides (1 for lanatoside C and 26 for digoxin) for regional laboratories which is double the number for 1972.

Much higher values than the therapeutic levels (ca, 1ng/ml digoxin in serum) have been encountered, the highest being 71ng/ml. It is noted with regret that a number of cases of accidental overdosages involved young children.

A radioimmunoassay for LSD in blood and urine is in the process of evaluation. The method originally took 2 days to complete, but using a new commercially available antibody, it now takes only 4 hours. Much interference from naturally occurring compounds in urine has been encountered and work is continuing. Several methods of improving the specificity of the assay have been tried, but all were unsuccessful. In conjunction with the Poisons Reference Centre (New Cross Hospital) and the Microbiological Research Establishment (Porton Down) another antibody to LSD, which is raised to a

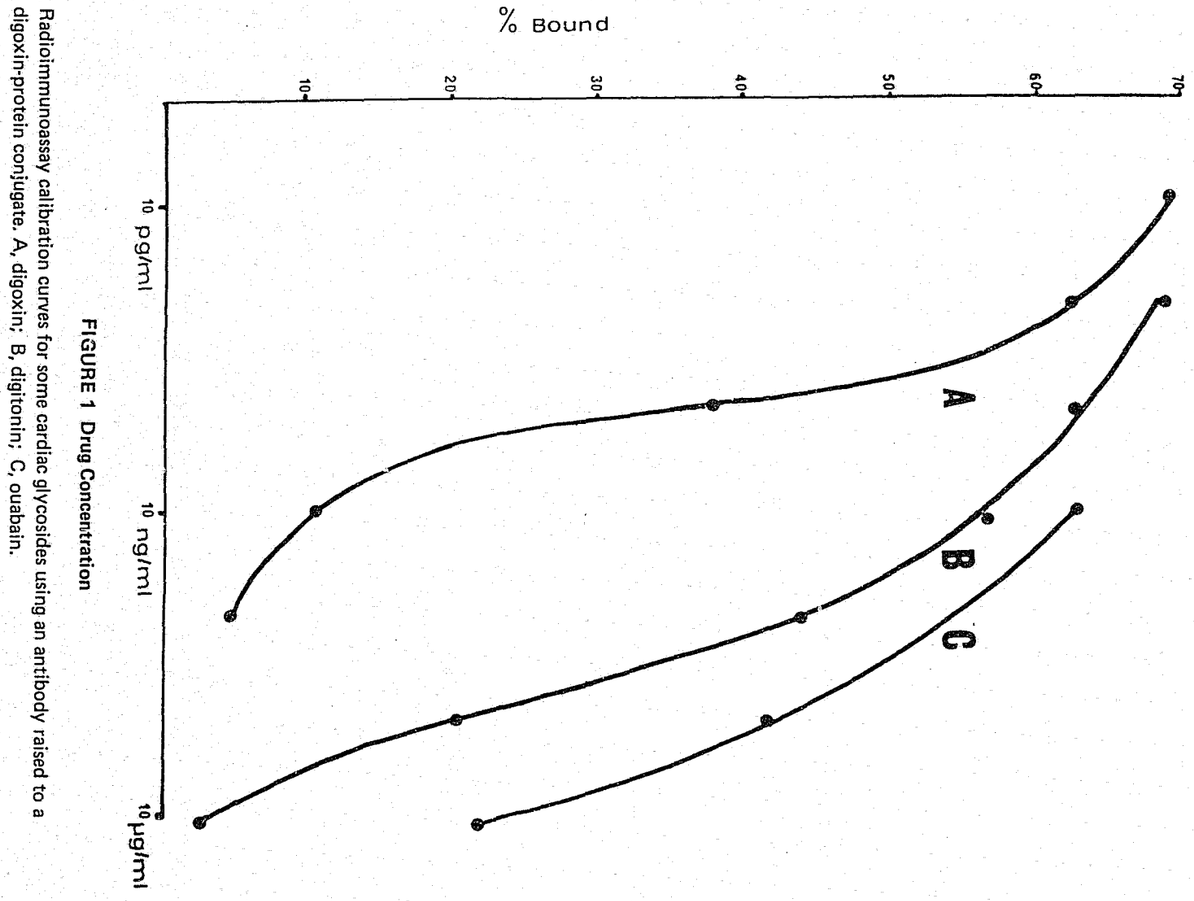



**FIGURE 1 Drug Concentration**

Radioimmunoassay calibration curves for some cardiac glycosides using an antibody raised to a digoxin-protein conjugate. A, digoxin; B, digitonin; C, ouabain.





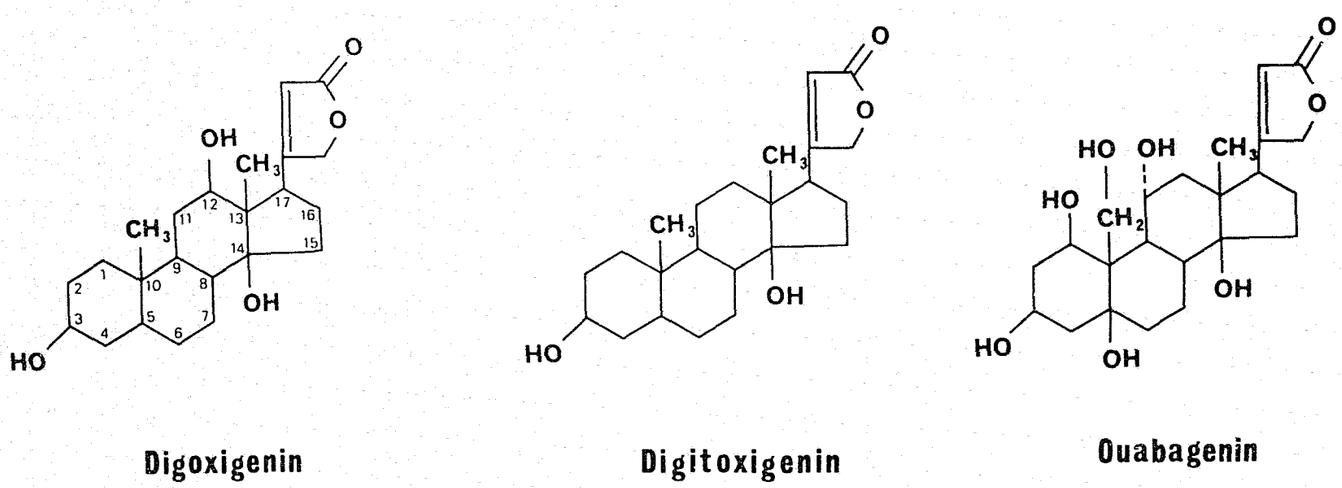



**FIGURE 2** The aglycones of some cardiac glycosides. Sugar chains are attached at the 3 position.

conjugate formed by joining a protein to the amide nitrogen atom of lysergic acid ethyl amide (Immunogen II, Figure 3) is being prepared. The antibody in current use has been raised to a conjugate of protein joined at the 1 position (Immunogen I, Figure 3). By the use of the two antibodies it may be possible to identify conclusively LSD in biological fluids.

It is hoped that a method for the radioimmunoassay of $\Delta^9$-THC will be available from a commercial organisation in the next year and this will be evaluated as soon as it is available.

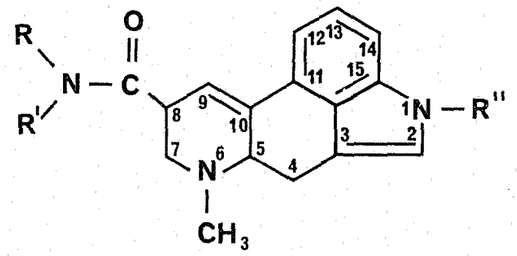


FIGURE 3

Lysergic acid amide: R = H, R′ = H, R″ = H

LSD: R = $C_2H_5$, R′ = $C_2H_5$, R″ = H

Immunogen I: R = $C_2H_5$, R′ = $C_2H_5$, R″ = $CH_2$ – NH – Protein

Immunogen II: R = $C_2H_5$, R′ = $(CH_2)_5$. COO. Protein, R″ = H

**Enzyme Inhibition and Drug Intoxication**

The investigation into possible relationships between drug intoxication and the levels of cyclic AMP in urine and catecholamines in brain are continuing.

**Cyclic AMP**

Urinary levels of cyclic AMP were not found to follow a circadian excretion pattern. The hourly variation in the cyclic AMP/creatinine ratio was determined and was found to be well within the 'normal' range. The cyclic AMP content of a urine sample stored in a bladder at 4°C was found to be stable for at least five days. Urine samples stored at the same temperature were found to have stable cyclic AMP levels for at least 30 days. Alcohol (15gm-90gm) tea, coffee, exercise and therapeutic doses of amphetamine sulphate and amylobarbitone did not significantly alter the cyclic AMP/creatinine ratio. The cyclic AMP/creatinine ratio is currently being determined for urine samples from people who have died of natural causes as well as patients treated with a variety of drugs. Urine samples from overdose cases will be analysed in the very near future.

**Catecholamines**

The concentrations of the neurotransmitters 5-hydroxytryptamine (5HT) noradrenaline and dopamine have been reported to be significantly raised in certain areas of the brain after the administration of monoamine oxidase inhibitors. Monoamine oxidase inhibitors are very difficult to detect chemically because of their small therapeutic dosage and also because their effects occur for several weeks after the last dose.

For the investigation, hind brains, caudate nuclei and hypothalami were removed at the post mortems, and deep frozen. The three areas were then analysed for 5HT, dopamine and noradrenaline respectively.



The hind brain was homogenised in dilute hydrochloric acid, and 5HT extracted with butanol. The 5HT was returned to an aqueous acid phase by adding heptane to the butanol and shaking the mixture with dilute hydrochloric acid. Concentrated hydrochloric acid was used to activate it for fluorometric measurement.

Caudate nuclei and hypothalami were homogenised in dilute hydrochloric acid and after precipitation of proteins with perchloric acid, the catecholamines were adsorbed from the filtrate onto alumina at pH8. They were subsequently eluted from this adsorbent with dilute perchloric acid and the fluorescence of the catecholamines measured after the formation of their tri-hydroxyindole derivatives. At present 'normal' neurotransmitter levels in the specific areas of the brain mentioned are being determined, prior to the examination of cases where monoamine oxidase inhibitors have been taken.

We are most grateful to Dr H Johnson, St Thomas' Hospital, London for samples of brain tissue.

**Assessment of Analytical Techniques for the Identification of Drugs**

It is important that the forensic toxicologist should be able to choose the most effective techniques to identify an unknown compound in the shortest possible time. Factors such as the reproducibility of the system, the distribution of values measured using a particular technique over the drug population and inter-system correlation have all to be considered. Until now, there was no easy way of evaluating the effectiveness of the techniques available, so that a comparison could be made between them.

However, the concept known as the Discriminating Power of a system or technique has been developed to include all the above features. The effectiveness of a technique or a series of techniques used in combination, for the identification of an unknown drug can now be expressed as a single figure.

Discriminating power is defined as the probability that two drugs selected at random from a large population would be discriminated by the technique used. The discriminating power can therefore be used to compare the techniques in terms of their effectiveness.

Before the evaluation could start it was imperative that a representative sample of drugs should be chosen out of all the drugs used in the UK. Over 3,000 drugs are known and 100 basic drugs were selected from these after searching through the records of two regional laboratories for 1970-2 to find which of these were the most commonly encountered.

Paper and thin-layer chromatography were the first systems to be examined in this way and the eight systems studied are given in Table 2. It can be seen that system 6 which was suggested and investigated by CRE for the analysis of basic drugs (CRE Report No. 36) is third in power of discrimination and arrangements are now in hand to scan the scientific literature for all commonly-used paper and thin-layer systems in order to determine their discriminating powers. It was noted during these evaluations that the simultaneous chromatography of a standard mixture of drugs, was necessary for good reproducibility. This provided coefficients of variation of less than 10% in Rf values of the drugs under examination.

The research revealed high correlations between some chromatographic systems, eg, as shown in Figure 4. Figure 5 shows what can be achieved by a selective choice.

The largest discriminating power found was with system 7 ($DP_7 = 0.75$) and when used in combination with other systems much higher discriminating powers could be obtained. For example:

2 systems combined (7 + 3), $DP_{3,7} = 0.93$

3 systems combined (7 + 3 + 6), $DP_{3,6,7} = 0.98$

4 systems combined (7 + 3 + 6 + 1), $DP_{1,3,6,7} = 0.99$

TABLE 2

Paper and Thin-Layer Systems Studied

| Type | System No. | Plate or Paper | Solvent | Discriminating Power |
|---|---|---|---|---|
| TLC | 1 | Silica gel dipped or prepared with 0.1M KOH | Cyclohexane:benzene diethylamine (75:15:10) | 0.73 |
| | 2 | Silica gel dipped or prepared with 0.1M KOH | Methanol | 0.69 |
| | 3 | Silica gel dipped or prepared with 0.1M KOH | Acetone | 0.75 |
| | 4 | Silica gel dipped or prepared with 0.1M $KHSO_4$ | Methanol | 0.66 |
| | 5 | Silica gel dipped or prepared with 0.1M $KHSO_4$ | Ethanol (95%) | 0.67 |
| PC | 6 | Whatman No.1 paper dipped in 5% sodium dihydrogen citrate | Butanol:water:citric acid (870:130:4.8g) | 0.74 |
| | 7 | Whatman No.1 paper dipped in 10% tri-butyrin in acetone | Acetate buffer pH 4.58 run at 95°C | 0.75 |
| | 8 | Whatman No.1 paper dipped in 10% tri-butyrin in acetone | Phosphate buffer pH 7.4 run at 86°C | 0.55 |



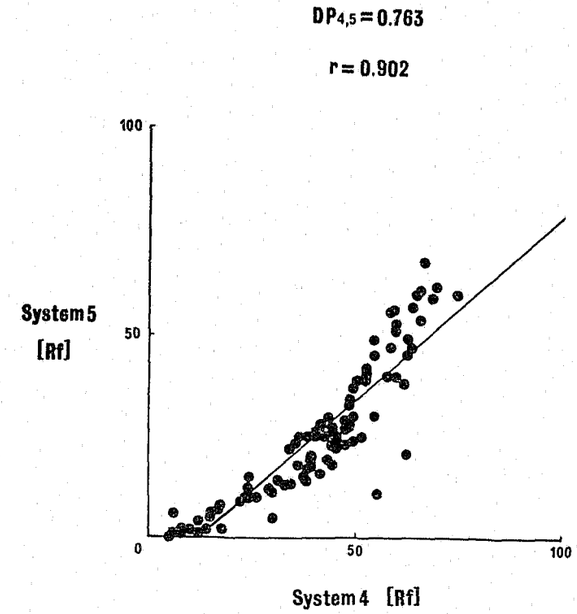



FIGURE 4

The correlation of Rf values of 100 basic drugs using system 4 (silica gel with 0.1N $KHSO_4$/methanol) and system 5 (silica gel with 0.1N $KHSO_4$/ethanol, 95%). Correlation coefficient = 0.902.

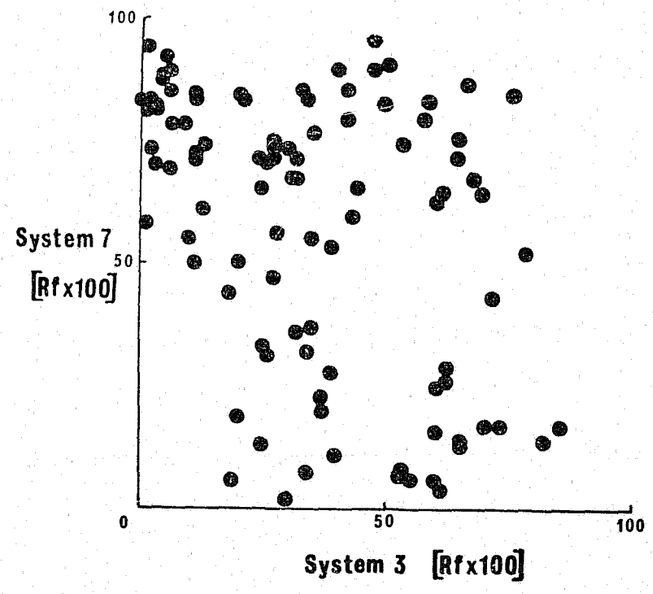



FIGURE 5

The correlation of Rf values of 100 basic drugs using system 3 (silica gel with 0.1N KOH/acetone) and system 7 (Whatman No.1 paper dipped in 10% tributyrin in acetone/acetate buffer pH4.58 run at 95°C). Correlation coefficient = -0.43.

Gas-liquid chromatographic systems have been examined in a similar way. Discriminating powers from 0.97 to 0.75 were obtained for the eight columns studied. These differences were not due essentially to the different spread of chromatographic values as with paper and thin-layer chromatography but were due to the failure of the columns to elute some of the drugs (see Figure 6). From the results obtained it has been decided that a collection of retention indices for basic drugs on an SE-30 column is desirable.

Ultra-violet and infra-red spectra have also been evaluated for drug identification in a similar manner. Of the 100 drugs studied in acidic solution by UV spectroscopy, valuable additional information was gained in 54 cases by re-running the compound in alkaline solution.

Work is being carried out, in conjunction with the Information Division, to place all the analytical data concerning drugs on the CRE computer, and to design a satisfactory searching system to enable toxicologists to obtain a read-out of all possible identities from the results of their analyses. They can then choose the best method for final identification of the drug in question.

Clearly the impact of mass spectrometry into toxicology has to be taken into account in planning future work and, as recorded elsewhere in this Report, an evaluation of mass spectrometry has already begun. GC-MS-computer links are already being installed in CRE.

Liaison with the Royal Air Force in the field of aviation toxicology has continued.



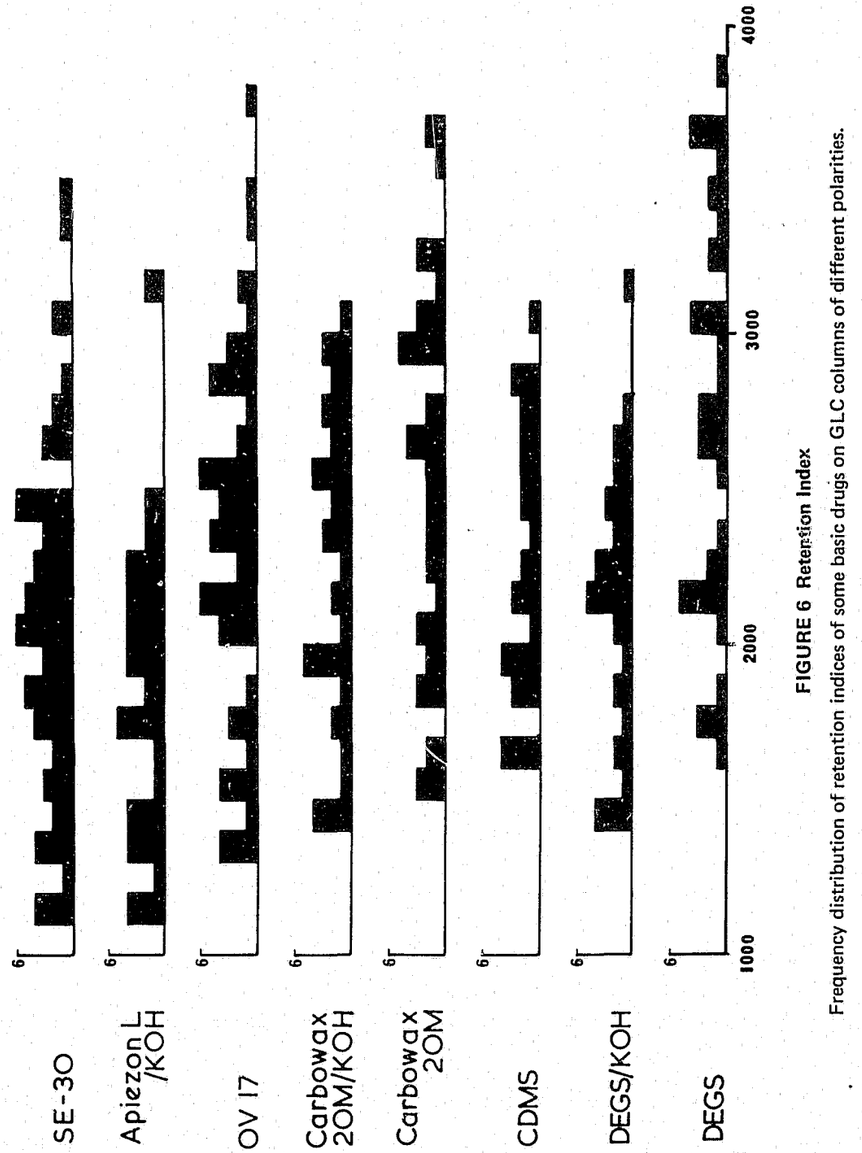



**FIGURE 6** Retention Index

Frequency distribution of retention indices of some basic drugs on GLC columns of different polarities.

## 10. LIAISON WITH OTHER GOVERNMENT DEPARTMENTS

In a field with such diverse interests as forensic science it is desirable that liaison be maintained with many people and organisations. It is a pleasure to accord our thanks to the following Government departments who have been particularly helpful to us during the year – the Police Scientific Development Branch, the Police Research Services Branch and the Drugs Branch of the Home Office, the Laboratory of the Government Chemist, the Chemical Defence and Microbiological Research Establishments, the Transport and Road Research Laboratory, the National Research Development Corporation and the National Institute for Research in Dairying.

Finally, it is always prudent for a tenant to take an opportunity to pay respect to his landlord and neighbours, but it is with no sense of obligation to propriety that we record our gratitude to the Atomic Weapons Research Establishment and the Home Counties Forensic Science Laboratory respectively for the splendid co-operation which they have extended to us.



# 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, 5502) | | |
| **Biology Division** | | |
| Dr P H Whitehead | PSO | 5947 |
| Mr E R Rutter | SSO | 5937 |
| Mr J G Sutton | SSO | 5937 |
| Mrs J A Brech | HSO | 5937 |
| Dr A E Kipps | HSO | 5937 |
| Mr P E Burdett | HSO | 5937 |
| Dr L A King | SRF | 4289 |
| **Chemistry Division** | | |
| Mr G W Walker | PSO | 5947 |
| Mr B German | SSO | 7574 |
| Dr J V Worthington | SSO | 5505 |
| Dr A Butterworth | HSO | 5952 |
| Dr R J Dudley | HSO | 5505 |
| Mrs D Morgans | SO | 7574 |
| Mrs I B Beattie | ASO | 5930 |
| **Drugs of Abuse Division** | | |
| Dr D A Patterson | PSO | 5947 |
| Mr P J Gomm | SSO | 5948 |
| Dr P J Twitchett | HSO | 5948 |
| **External Contracts Division** | | |
| Dr M D G Dabbs | PSO | 5930 |

| Name and Division | Rank | Telephone Extensions |
|---|---|---|
| **Information Division** | | |
| Mr V J Emerson | PSO | 6585 5947 |
| Mr K W Smalldon | SSO | 4289 |
| Mr M Swain | SSO | 6996 |
| Mr C Brown | SSO | 6996 |
| Mr C A Pounds | SSO | 4289 |
| Mr G W Owen | HSO | 5930 |
| Mr J Porter | HSO | 5505 |
| Mr M C D Harold | SO | 6996 |
| Mr S W Brandish | CA | 6996 |
| **Toxicology Division** | | |
| Dr H M Stevens | PSO | 5947 |
| Mr A C Moffat | SSO | 5938 |
| Mr P Owen | SO | 5938 |
| **Technical Staff** | | |
| Mr D J Nicholson | P and TO III | 5783 |
| **Clerical Staff** | | |
| Mr S Jones | CO | 5942 |
| Mrs S M Webb | Typist | 5783 |
| Mrs M A Golding | Typist | 5783 |
| Mrs M R E F Dry | CA | 5942 |
| **Non-Technical Staff** | | |
| Mrs S C Jones | Cleaner | |
| Mr C H Nicholson | Driver | |
| Mr L Rowbottom | 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 | L A King | Therapeutic salicylate levels in micro blood stains can be detected. Further studies on other drugs in progress. |
| (ii) Viral antibodies | L A King | Studies in progress. |
| **Discrimination Studies Using Genetic Parameters** | | |
| (a) Semen | | |
| (i) Acid phosphatase | J G Sutton | Work started. |
| (ii) Other semen enzymes | J G Sutton | To be started. |
| (b) Saliva | | |
| (i) Amylase genetic variants | A E Kipps | Work started. |
| (ii) Other salivary enzymes | A E Kipps | To be started. |
| (c) Blood | | |
| (i) Studies on established enzyme variants | P E Burdett | Work on quantitation and simplification started. |
| (ii) Laurell electrophoresis | P E Burdett<br>M D G Dabbs | Protein work by external contract. Work on enzymes to be started. |
| **Immunology Studies** | | |
| (a) Use of Latex | | |
| (i) Species identification of blood and tissue | A J Brech | Technique shown to be viable. Early trials planned. |
| (ii) Serology | A J Brech | To be started. |
| (iii) Other antigens | A J Brech | To be started. |
| (b) General Studies | A J Brech | Development studies and appraisal of new techniques continued. |

| *Subject* | *Staff* | *State of Progress* |
|---|---|---|
| **Correlation Studies** | | |
| | | Studies into possible correlations between genetic enzymes and serological groups to be started. |
| **Automation** | | |
| Blood | E R Rutter<br>M D G Dabbs | Continuous flow and batch processing machines being developed and by external contract. |
| **Botanical Studies** | | |
| Pollen | A E Kipps | Work in progress. |

## CHEMISTRY DIVISION

**Head of Division — Mr G W Walker**

| | | |
|---|---|---|
| **Organic Analysis** | | |
| (a) Pyrolysis-gas Chromatography | M D G Dabbs | Studies on rubbers and fibres being done by external contracts. |
| (b) Mass Spectrometry | J V Worthington | The Micromass 12 is to be interfaced to the HP2100 Computer. Cost benefit analysis studies of the mass spectrometer in progress. Data collections on computer to be started. |
| (c) Blood Alcohol | A Butterworth | Work in relation to the Road Traffic Act in progress. Pye liquid injector under test. External contracts operative. |
| (d) Explosive Residues on Hands | J V Worthington | To be started. |
| **Inorganic Analysis** | | |
| (a) Mass Spectrometry | | Absolute quantitation work being developed. Studies on reduction of sample size started. |
| (i) Glass | B German<br>D Morgan | Survey work continuing. |
| (ii) Trace elements in liver tissue | B German<br>D Morgan | To be started. |
| (b) Laser-Spark Emission | B German | Initial work completed. Quantitation and discrimination on glass and paint to be studied. |



| *Subject* | *Staff* | *State of Progress* |
|---|---|---|
| (c) Emission Spectroscopy | B German | Studies on quantitation of arc and plasma emission to be started. |
| (d) Flameless Atomic Absorption | B German<br>D Morgans<br>M D G Dabbs | Studies on firearms' residues completed. Work on glass to be started. External contracts operative. |
| **Soil Analysis** | R J Dudley | Work has started. |
| **Automation** | | |
| (i) Refractive Index of glass | M D G Dabbs<br>I B Beattie | Prototype machine almost completed. External contract for production prototype negotiated. |
| (ii) Density gradient | M D G Dabbs | External contracts to be placed. |
| **Computer HP 2100** | A Butterworth | Disc store installed. Interfaces to MM12 and MS702 to be fitted for data handling. Direct link to MS702 now operational for machine control. Links to other machines in development stage. |

## DRUGS OF ABUSE DIVISION

**Head of Division — Dr D A Patterson**

| | | |
|---|---|---|
| **Tablet, Powder, Capsule Identification** | P J Gomm | Prototype colour testing. Automatic analyser completed. Production prototypes under operational trial. |
| **Drug Trend Monitoring** | D A Patterson<br>P J Gomm<br>P J Twitchett | Liaison with Central Drugs Intelligence Unit established. Operational research in progress. |
| **High Pressure Liquid Chromatography** | P J Twitchett | To be evaluated. |
| **Identification of Stereoisomers** | P J Twitchett | Circular dichroism shown to be successful. Assessment of other techniques continuing. |
| **Cannabis** | R M Barrett (Research Fellow Australia) | Pyrolysis of cannabinols being studied. |
| **Collection of Reference Standards and Compilation of Analytical Data** | D A Patterson<br>M D G Dabbs | Continuing satisfactorily. External contract in progress. |

| *Subject* | *Staff* | *State of Progress* |
|---|---|---|
| **EXTERNAL CONTRACTS DIVISION** | | |
| Initiation and Supervision of all External Contracts | M D G Dabbs | |
| **INFORMATION DIVISION** | | |
| **Head of Division — Mr V J Emerson** | | |
| **Computerised Information Retrieval Service** | M Swain<br>C Brown<br>M C D Harold<br>S Brandish | Move from time-shared computer to CRE computer — September 1973; 12,000 reprints on file. |
| (a) 'In-House' Searching | | |
| (b) Use of UKCIS Computer Agency | | |
| **Implementation of Data Banks** | | |
| (a) Infra-red (IR) spectra | C Brown | 2,876 spectra on microfilm available; computer retrieval system operational. |
| (b) Ultra-violet (UV) spectra | C Brown | 1,020 spectra on microfilm. |
| (c) Collection of Pharmaceutical Drugs | C Brown | 2,000 samples now available. |
| (d) Collection of Agricultural chemicals | G W Owen | UV; IR and extraction data available for 200 compounds (UV and extraction data provided by regional laboratories). |
| (e) Register of Human Toxicology | M Swain<br>C Brown | Details of unusual poisonings analysed by regional laboratories published. |
| (f) Refractive Index and Density of Glass | C Brown | Glass population studies including all crime cases handled by regional laboratories. Statistical studies on discrimination going on. |
| (g) Collection of Headlamps and Spot Lamps | J Porter | 178 samples available. Updating in progress. |
| (h) Shoe and Boot Patterns | C Brown<br>M D G Dabbs | Subject of an external contract. |
| (i) Tyre Patterns | C Brown<br>M D G Dabbs | Subject of an external contract. |
| (j) Car Paints | J Porter<br>C Brown | Colour index for top coats and undercoates for cars completed. Computer retrieval to be started. |
| (k) House Paints | J Porter<br>C Brown | Statistical studies will be made on returns from regional laboratories. |



| *Subject* | *Staff* | *State of Progress* |
|---|---|---|
| **Literature Presentation** | | |
| (a) Literature and Data Presentation to Regional Laboratories | M Swain<br>M C D Harold<br>S Brandish | Selected papers and data supplied monthly. |
| (b) Microfilm and Microfiche to Regional Laboratories | M Swain<br>M C D Harold | Full collections have been supplied. |
| (c) Preparation of Computer/ Microfilm Retrieval System for Commercial Exploitation | C Brown | A start has been made. |
| **Communication Links** | | |
| (a) Video Tapes | G W Owen | In co-operation with regional laboratories and six police forces, a series of 20-minute video tapes dealing with searching scenes of crime, have been made. |
| (b) With Regional Laboratories | | A start has been made to study the best way to provide rapid comprehensive communication links. |
| (c) Computer Linkages | C Brown | Detailed appraisals of the use of computers by regional laboratories are being done. |
| (d) Quality Control | G W Owen<br>K W Smalldon | Analytical samples are circulated to regional laboratories. |
| **Systems Analysis** | K W Smalldon | A detailed study of work done in regional laboratories is being made for future planning of research. |
| **Analyses and Searching Techniques** | | |
| (a) Glass | C A Pounds<br>K W Smalldon | Methods are being studied of the transference of glass to clothing and on the efficiency of searching. |
| (b) Fibres | C A Pounds<br>K W Smalldon | As above |
| (c) Clothing | G W Owen<br>K W Smalldon | The incidence of blood and semen on shoes and clothing not related to crime is being studied. |
| (d) Blood Group Systems | G W Owen | A statistical appraisal of biological systems in an attempt to provide maximum information in return for minimum analytical effort. |

| | *Subject* | *Staff* | *State of Progress* |
|---|---|---|---|
| (e) | Toxicological Date | K W Smalldon | Correlation parameters for TLC and GLC systems with the concept of a 'discriminating power' has been introduced. |

**TOXICOLOGY DIVISION**

**Head of Division — Dr H M Stevens**

**Difficult Compounds**

| | | | |
|---|---|---|---|
| (a) | Quaternary Ammonium Compounds | H M Stevens | Work completed unless problems are encountered with new compounds in regional laboratories. |
| (b) | Protein Precipitation Techniques | H M Stevens | Six different protein precipitation methods have been evaluated. |
| (c) | Fluoroacetamide and Fluoroacetates | H M Stevens | Methods for extracting and detecting fluoroacetamide in water, urine and blood have been investigated. Work is continuing. |
| **Putrefaction** | | H M Stevens | Studies on basic compounds completed. Acid and neutral compounds yet to be started. |

**Radioimmunoassay**

| | | | |
|---|---|---|---|
| (a) | Digoxin | A C Moffat | Methodology work completed — now an operational service. |
| (b) | LSD | A C Moffat | Methodology being evaluated. |
| (c) | Other Drugs | A C Moffat | To be started. |
| **Assessment of Analytical Techniques** | | A C Moffat | Practical work on correlation and discriminating power in TLC and GC systems is proceeding. |

**Biochemical Toxicology**

| | | | |
|---|---|---|---|
| (a) | Cyclic AMP in Urine | P Owen | Methods of assay worked out — typical case material has yet to be investigated. |
| (b) | Catecholamines in Brain Tissue | P Owen | Methods of assay worked out — typical case material has yet to be investigated. |

**Automated Analysis**

| | | | |
|---|---|---|---|
| Drug Extraction from Urine | | E R Rutter | Four-channel machine for morphine, amphetamine, barbiturates and methaqualone completed. Method for routine assay of urine samples for creatinine completed. |



## APPENDIX C

### REPORTS PUBLISHED FROM JANUARY 1967 TO DATE

| *Report No.* | *Title* | *Date* |
|---|---|---|
| 1 | Detection of Firearm Residues on Hands and Clothing. | May 1967 |
| 2 | A Study on Suitable Commercially Available Stable Diazonium Compounds for use in the Acid Phosphatase Test. | July 1967 |
| 3 | The Use of Absorption Spectrophotometry in Assessing the Colour Changes in Ageing Bloodstains. | October 1967 |
| 4 | Dry Cleaning Processes (Detection of Residues). | October 1967 |
| 4(a) | The Effect of the Dry Cleaning Process on the Serological Examination of Bloodstains. | January 1969 |
| 5 | DELETED | |
| 6 | Atomic Absorption Spectrophotometry I — Introduction. | January 1968 |
| 6(a) | Analytical Notes on the Determination of Cobalt by Atomic Absorption Spectrophotometry. | March 1968 |
| 6(b) | Atomic Absorption Spectrophotometry — Analysis of Lead in Tissue, Blood and Urine. | July 1968 |
| 6(c) | Determination of Thallium in Biological Material by Flame Spectrophotometry Methods. | February 1969 |
| 7 | Exclusion of Urinary Barbiturates by Gas Chromatography. | January 1968 |
| 8 | Infra-Red Retrieval Systems. | |
| 9 | Percentage Incidence of Microscopic Hardwood Characteristics. | March 1968 |
| 10 | Quantitative Morphology of Human Head Hair Part I — Shaft Diameter. | April 1968 |
| 11 | Emulsion Breaking in Toxicological Analysis. | April 1968 |
| 12 | MS702 Inorganic Mass Spectrometer. | May 1968 |
| 13 | The Bacterial Production of Ethyl Alcohol. | May 1968 |
| 14 | The Work of the Central Research Establishment from its Inception to May 1968. | May 1968 |
| | Note on the Sadtler Collection of Standard Infra-Red and Ultra-Violet Spectra. | |

| Report No. | Title | Date |
|---|---|---|
| 15 | Micro Infra-Red Spectroscopy of Gas Chromatography Fractions. | September 1968 |
| 16 | A Simple Infra-Red Spectrum Retrieval System for use in Forensic Science Laboratories. | November 1968 |
| 17 | Application of the Fluorescent Antibody Technique to the Detection of Blood Group Antigens in Stains. | January 1969 |
| 18 | The Detection of Cannabis Constituents in the Mouth and on the Fingers of Smokers. | January 1969 |
| 19 | Haptoglobins, Part I. | February 1969 |
| 20 | Paint Strippers. | February 1969 |
| 21 | An Investigation into Forensic Problems Associated with Cannabis. | February 1969 |
| 22 | Heterogeneity in Glass. | March 1969 |
| 23 | A Comparison of Motor Manufacturers Production Colours. | March 1969 |
| 24 | The Extraction of Phenylbutazone from Blood and Plasma. | March 1969 |
| 25 | The Identification of Paint Resins and Other Polymeric Materials from the Infra-Red Spectra of their Pyrolysis Products. | May 1969 |
| 26 | The Hot Stage Microscope. | June 1969 |
| 27 | The Technique of Antigen/Antibody Crossed Electrophoresis. | July 1969 |
| 28 | Haptoglobins, Part II. The Typing of Haptoglobins by Agar-Gel Electrophoresis. | July 1969 |
| 29 | The Determination of Carbon Monoxide in Blood and Tissue. | August 1969 |
| 30 | Report on Colour Comparison of the Colour Cards of Some Manufacturers of Household Paints. | October 1969 |
| 31 | Evaluation of the Carl Zeiss (W Germany) MPM Microscope Photometer as a 'Colour' Measuring Instrument for use with Paints. | November 1969 |
| 32 | Research at the Central Research Establishment. | October 1969 |
| 33 | Comparative Trial UV Spectrophotometers. | November 1969 |
| 34 | 'Normal' Levels of Cadmium in Human Liver and Kidney in England. | January 1970 |
| 35 | Storage of Blood Samples. | January 1970 |



| Report No. | Title | Date |
|---|---|---|
| 36 | The Identification of Alkaloids and Synthetic Basic Drugs. | January 1970 |
| 37 | The Determination of Carbon Monoxide in Blood and Tissue, Part II. | February 1970 |
| 38 | Second Interpol Forensic Science Colloquium, Paris, 25 28 November 1968. | February 1970 |
| 39 | The Relationship Between Chemical Composition and Geographical Origin of Cannabis. | May 1970 |
| 40 | Glass and Paint on Clothing — Report of a Survey. | July 1970 |
| 41 | Saliva Grouping. An Analysis of the Inter-Laboratory Trials and Experiments Performed at the Central Research Establishment. | July 1970 |
| 42 | Composition of Illicit Diamorphine Samples. | August 1970 |
| 43 | Identification Tests for Bases Formed During Putrefaction of Visceral Material. | September 1970 (Amended Jan 71) |
| 44 | Some Physical Properties of a Large Number of Window Glass Samples: Report of a Survey. | October 1970 |
| 45 | Trial of a Commercial Anti-Human Haemoglobin Preparation. | October 1970 |
| 46 | Reference Analytical Data for some Hallucinogens. | November 1970 |
| 47 | Some Physical Properties of Window Glass Broken in the Course of Criminal Activity in the City of Liverpool. | |
| 48 | Reference Analytical Data for Some Ergoline Alkaloids. | March 1971 |
| 49 | Report on the LKB Density Gradient Former. | April 1971 |
| 50 | A Method for Detecting Tubocurarine in Tissue. | April 1971 |
| 51 | The Chromatographic Separation of Mixtures of Benzodiazepine Drugs. | April 1971 |
| 52 | Infra-Red Spectrophotometry of Some Benzilates and Related Compounds. | April 1971 |
| 53 | Identification of Benzoctamine — A New Anti-depressant Drug. | May 1971. |
| 54 | A Reference Collection of Chromatograms of the Pyrolysis Products of Some Paints and Plastics. | June 1971 |
| 55 | Spectrophotometry of Some Newer Benzodiazepines. | July 1971 |
| 56 | The Detection of Sex Chromatin in Bloodstains on Solid Substrata. | August 1971 |

| Report No. | Title | Date |
|---|---|---|
| 57 | A Case Involving the Administration of Known Amounts of Arsenic and its Analysis in Hair. | September 1971 |
| 58 | Thin-Layer Chromatography of Lysergide and Other Ergot Alkaloids. | September 1971 |
| 59 | Sample Preparation for Tritium Counting in the Application of the Digoxin Radioimmunoassay Technique to Lysed Blood. | September 1971 |
| 60 | Determination of Lead and Copper in Hair by Non-Flame Atomic Absorption Spectrophotometry. | January 1972 |
| 61 | A Survey of Headlamps and Auxiliary Lamps used on Cars in the United Kingdom. | January 1972 |
| 62 | A Spectrophotometric Method for Screening Urine Samples for Amines, Including Amphetamine. | January 1972 |
| 63 | The Refractive Index of Glass Encountered in Casework — Report of Results Returned 1971 (Glass Committee). | February 1972 |
| 64 | Improved Y Chromosome Fluorescence in the Presence of Magnesium Ions. | February 1972 |
| 65 | Reference Infra-Red and Ultra-Violet Spectra for some Intermediates in the Synthesis of Drugs of Abuse. | February 1972 |
| 66 | The Probability of Occurrence of Window Glass Having Given Values of Refractive Index. | March 1972 |
| 67 | The Use of Spark Source Mass Spectrometry for the Analysis of Glass. | March 1972 |
| 68 | The Use of Spark Source Mass Spectrometry for the Analysis of Glass Fragments Encountered in Forensic Applications — Part II. | March 1972 |
| 69 | Reference Analytical Data for Some Narcotics. | April 1972 |
| 70 | The Variation in Refractive Index Across Specific Windscreens. | April 1972 |
| 71 | Report on the Use of Spark Source Mass Spectrometry in a Number of Recent Forensic Science Cases. | May 1972 |
| 72 | Effect of Sample Size on Colour Discrimination. | May 1972 |
| 73 | The Identification of Certain Interfering Substances in the Determination of Barbiturates in Human Liver. | June 1972 |
| 74 | The Radioimmunoassay of Digoxin in Post Mortem Blood. | August 1972 |
| 75 | An Evaluation of Trace Element Analysis in the Forensic Investigation of Fibre Samples. | October 1972 |



| Report No. | Title | Date |
|---|---|---|
| 76 | Infra-Red Spectrophotometric Data and Retrieval System for Drugs of Abuse. | October 1972 |
| 77 | Immunological Identification of Human Semen. | November 1972 |
| 78 | Preliminary Report on the Differentiation of Blood of Avian Species. | December 1972 |
| 79 | The Identification of Male Bloodstains by Y Chromosome Fluorescence. | January 1973 |
| 80 | A Review of the Technique of Organic Mass Spectrometry in the Context of Toxicology and Drug Analysis. | January 1973 |
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| 82 | An Evaluation of Common Methods of Paint Analysis. | January 1973 |
| 83 | The Extraction of Quaternary Ammonium Compounds from Body Fluids and Tissues. | April 1973 |
| 84 | A Comparison of Some Presumptive Tests for Blood. A Non-Carcinogenic Substitute for Benzidine in the Detection of Blood. | April 1973 |
| 85 | Identification of Vehicles from Paint Flakes using the Methuen Colour System and Manufacturers' Colour Cards. | April 1973 |
| 86 | The Application of a Standard Colour System to Forensic Science. | April 1973 |
| 87 | The Retrieval of Data from the Glass Survey by Means of the CRE Computer. | April 1973 |
| 88 | A Report on an Investigation into the Trace Elements Present in Vehicle Headlamp and Auxiliary Lamp Glasses. | April 1973 |
| 89 | The Calculation of Discriminating Power for a Series of Correlated Attributes. | May 1973 |
| 90 | The Discriminating Power of Density and Refractive Index for Window Glass. | May 1973 |
| 91 | Ethanol Oxidation by Human Erythrocytes. | May 1973 |
| 92 | Report on the Refractive Index of Glass Encountered in Casework During 1972. | May 1973 |
| 93 | Small Survey of British Container Glass Using Spark Source Mass Spectrometry with Electrical Detection. | May 1973 |
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| *Report No.* | *Title* | *Date* |
|---|---|---|
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| 96 | Identification of Vehicles from Paint Flakes Using Methuen Colour System and Pyrolysis Gas Chromatography. | June 1973 |
| 97 | The Assessment of Various Protein Precipitation Methods in the Extraction of Basic Drugs from Tissues. | July 1973 |
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| 100 | Optimum Use of Chromatography for Basic Drugs: (2) Paper and Thin-Layer Chromatography. | July 1973 |
| 101 | Optimum Use of Chromatography for Basic Drugs: (3) Gas-Liquid Chromatography. | July 1973 |
| 102 | Hair Sexing in Forensic Science. | August 1973 |
| 103 | RESTRICTED | August 1973 |
| 104 | Blood and Semen Stains on Outer Clothing and Shoes not Related to Crime — Report of a Survey. | August 1973 |
| 105 | Agricultural Chemicals — Analytical Data. | September 1973 |
| 106 | A Small-Scale Study of Acrylic Fibres by Spark Source Mass Spectrometry. | October 1973 |
| 107 | The Detection of Salicylate in Bloodstains. | October 1973 |
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## APPENDIX D

### COLLOQUIA HELD AT CRE SINCE OCTOBER 1972

**'Colloquium on Alcohol'**

Friday, 17 November, 1972

The Stability of Ethanol in Stored Blood

Part I — A Factorial Investigation by D Neylan, Metropolitan Police Laboratory.

Part II — Mechanisms of Alcohol Loss by K Smalldon, Central Research Establishment.

Discussion

Automated Analysis of Ethanol Using the Perkin Elmer F40

Part I — Basic Operation of the F40 by M Wright, North Western Laboratory, Chorley.

Part II — Investigations Using the F40 by D Rudram, Metropolitan Police Laboratory.

Discussion

Alcohol Dehydrogenase

Part I — Introduction by A Scaplehorn, Central Research Establishment.

Part II — Practical Demonstration by P Gomm, Central Research Establishment.

Alcohol — The Future by A Scaplehorn, Central Research Establishment.

Discussion



**'Colloquium on Motor Vehicle Accidents'**

Friday, 8 December, 1972

Some Examples of Brake Pipe Failure by P B Wilson, North Eastern Laboratory, Harrogate.

Discussion

Some Problems Encountered in Lamp Examinations by D Dolan, Home Counties Laboratory, Aldermaston.

Discussion

On the Spot Accident Investigation by C Blamey, Road Research Laboratory, Crowthorne.

Discussion

Tyre Examinations — Where do we go from Here? by R Grogan, Dunlop Tyre Company.

Discussion

Component Failures Associated with the Use of Wide Wheels and Wheel Spacers by R W Wootton, South Western Laboratory, Bristol.

Discussion

(i) Liquid Chromatographic Characterisation of Used Engine Oils.

(ii) Pyrolysis Gas Chromatographic Characterisation of Automotive Paints by B B Wheals, Metropolitan Police Laboratory.

Discussion

'Colloquium on External Contracts'

Friday, 16 February 1973

Multi-element Atomic Absorption Spectrophotometry by Professor T S West, Imperial College.

Automatic Extraction of Viscera.

Part I — Dr H M Stevens, Central Research Establishment.

Part II — Dr R D Cowling, International Research and Development Co Ltd.

Recent Research in Microspectrofluorimetry by B Towell, Royal Military College of Science.

Spectroscopic Applications of Photon Counting Techniques by A Bowd, Royal Military College of Science.

Pyrolysis Gas Chromatography of Textile Fibres.

Part I — Dr R W May, Home Counties Laboratory, Aldermaston.

Part II — Dr B Sagar, Shirley Institute.

Enzyme Inhibition in Drug Detection Systems by Dr Townshend, Birmingham University.

Any Suggestions for Future Contract Work.

Discussion



'Colloquium on Drugs and Poisons Analysis'

Friday, 16 March, 1973

The Measurement of Effectiveness for Correlated Chromatographic Systems.

Part I — Basic Concepts by K W Smalldon, Central Research Establishment.

Part II — Application to PC, TLC and GLC by Dr A C Moffat, Central Research Establishment.

Resolution of Optical Isomers by Dr P J Twitchett, Central Research Establishment.

Murder by Poisoning by P Rees, East Midlands Laboratory, Nottingham.

The Potential of High Pressure Liquid Chromatography in Drug Analysis by B B Wheals, Metropolitan Police Laboratory.

The Radioimmunoassay of Drugs by Dr A C Moffat, Central Research Establishment.

Quaternary Ammonium Compounds – A Gleam of Hope by Dr H M Stevens, Central Research Establishment.

Drug Abuse Trends by Dr D A Patterson, Central Research Establishment.

A Case of Murder Involving Organophosphorus Poisoning by Dr Wilson, North Eastern Laboratory, Harrogate.

Discussion

## 'Colloquium on Information'

Friday, 13 April, 1973

Information Requirements in Forensic Science and Possible Sources by Dr E F Pearson, Central Research Establishment.

Incidence of Blood and Seminal Stains on Male Outer Garments by G W Owen, Central Research Establishment.

Review of Progress in Information Retrieval by M Swain, Central Research Establishment.

Information and Forensic Science by A Fawcett, East Midlands Laboratory, Nottingham.

Analysis of the Information from Ten Blood Alcohol Trials by K W Smalldon, Central Research Establishment.

Information Retrieval from Collections by C Brown, Central Research Establishment.

Discussion



## 'Colloquium on Inks'

Friday, 18 May, 1973

Surveys of Handwriting Media

Surveys for 1971 and 1972 – Introduction by R A Page, South Wales and Monmouthshire Laboratory, Cardiff.

Part I — Ball Point Pens by Dr D Baxendale, South Wales and Monmouthshire Laboratory, Cardiff.

Part II — Fibre Tipped Pens by Dr R N Totty, South Wales and Monmouthshire Laboratory, Cardiff.

Part III — Fountain Pen Inks by Dr D Baxendale, South Wales and Monmouthshire Laboratory, Cardiff.

Part IV — Carbon Papers by Dr R N Totty, South Wales and Monmouthshire Laboratory, Cardiff.

The Evidential Value of Ink Analysis by R A Page, South Wales and Monmouthshire Laboratory, Cardiff.

Analytical Methods

Spectra of Ink-Lines by R M Kevern, Leicester.

Electrophoresis by Dr R N Totty, South Wales and Monmouthshire Laboratory, Cardiff.

The Examination of Inks by Electron Probe Microanalysis by Mrs R C Robeson, R H Keely and C Van Essen, Metropolitan Police Laboratory.

Spectrophotometric Analysis by M G Hall, South Wales and Monmouthshire Laboratory, Cardiff.

Discussion

## APPENDIX E

### COMPOSITION OF QUALITY CONTROL COMMITTEES

Close co-operation with the Committees in each subject area has been maintained during the year. The current composition of each Committee is as listed below:

### BODY FLUIDS COMMITTEE

| | |
|---|---|
| J L Fish (Chairman) | Cardiff Laboratory |
| G W Owen (Secretary) | Central Research Establishment |
| J G Craddock | Nottingham Laboratory |
| M Pereira | Metropolitan Police Laboratory |
| P H Whitehead | Central Research Establishment |

### FIBRE COMMITTEE

| | |
|---|---|
| P G W Cobb (Chairman) | Home Counties Laboratory |
| B Rees (Secretary) | Home Counties Laboratory |
| R Cook | Metropolitan Police Laboratory |
| A S Fawcett | Nottingham Laboratory |
| K W Smalldon | Central Research Establishment |
| N T Weston | Birmingham Laboratory |
| C Wood | Chorley Laboratory |

### GLASS COMMITTEE

| | |
|---|---|
| F L Cann (Chairman) | Chorley Laboratory |
| P D B Clarke | Home Counties Laboratory |
| K Feenan | Chorley Laboratory |
| D Goodwin | Metropolitan Police Laboratory |
| G W Walker | Central Research Establishment |

### PAINT COMMITTEE

| | |
|---|---|
| I G Holden (Chairman) | Nottingham Laboratory |
| H Bamford | Chorley Laboratory |
| R W May | Home Counties Laboratory |
| K W Smalldon | Central Research Establishment |
| C F Tippett | Cardiff Laboratory |



### TOXICOLOGY AND DRUGS COMMITTEE

| | |
|---|---|
| A S Curry (Chairman) | Central Research Establishment |
| H M Stevens (Secretary) | Central Research Establishment |
| M J Bailey | Chorley Laboratory |
| N Dunnett | Home Counties Laboratory |
| J S Fox | Bristol Laboratory |
| J V Jackson | Metropolitan Police Laboratory |
| K J Kimber | Newcastle Laboratory |
| M Loveland | Birmingham Laboratory |
| G Osborne | Harrogate Laboratory |
| G W Owen | Central Research Establishment |
| P O Rees | Nottingham Laboratory |
| A Richardson | Cardiff Laboratory |

### TYRE COMMITTEE

| | |
|---|---|
| C F Tippett (Chairman) | Cardiff Laboratory |
| K W Smalldon (Secretary) | Central Research Establishment |
| P D B Clarke | Home Counties Laboratory |
| T R Watson | Birmingham Laboratory |
| B Wright | Metropolitan Police Laboratory |

## APPENDIX F

### LIST OF VIDEO TAPES AVAILABLE*

| TITLE | FORCE | LABORATORY |
|---|---|---|
| Introduction to Scenes of Crime | Sussex | Home Counties Laboratory |
| Hit and Run | Durham | Newcastle Laboratory |
| Casts and Impressions | Durham | Newcastle Laboratory |
| Sexual Offences | W Yorkshire | Harrogate Laboratory |
| Drugs, the Police and the Scientist | W Yorkshire | Harrogate Laboratory |
| Searching the Crime Scene — A Case of Murder | Lancashire | Chorley Laboratory |
| Materials for the Expert (Packaging) | Lancashire | Chorley Laboratory |
| Breaks | W Mercia | Birmingham Laboratory |
| Road Safety Act 1967 | W Mercia | Birmingham Laboratory |
| Firearms | | Nottingham Laboratory |
| Documents Examination | | Birmingham Laboratory |

*Copies are not held at CRE. Enquiries should be made to the relevant Regional Laboratory.



## APPENDIX G

### 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 to first open the window and use the handle on the outside of the carriage!

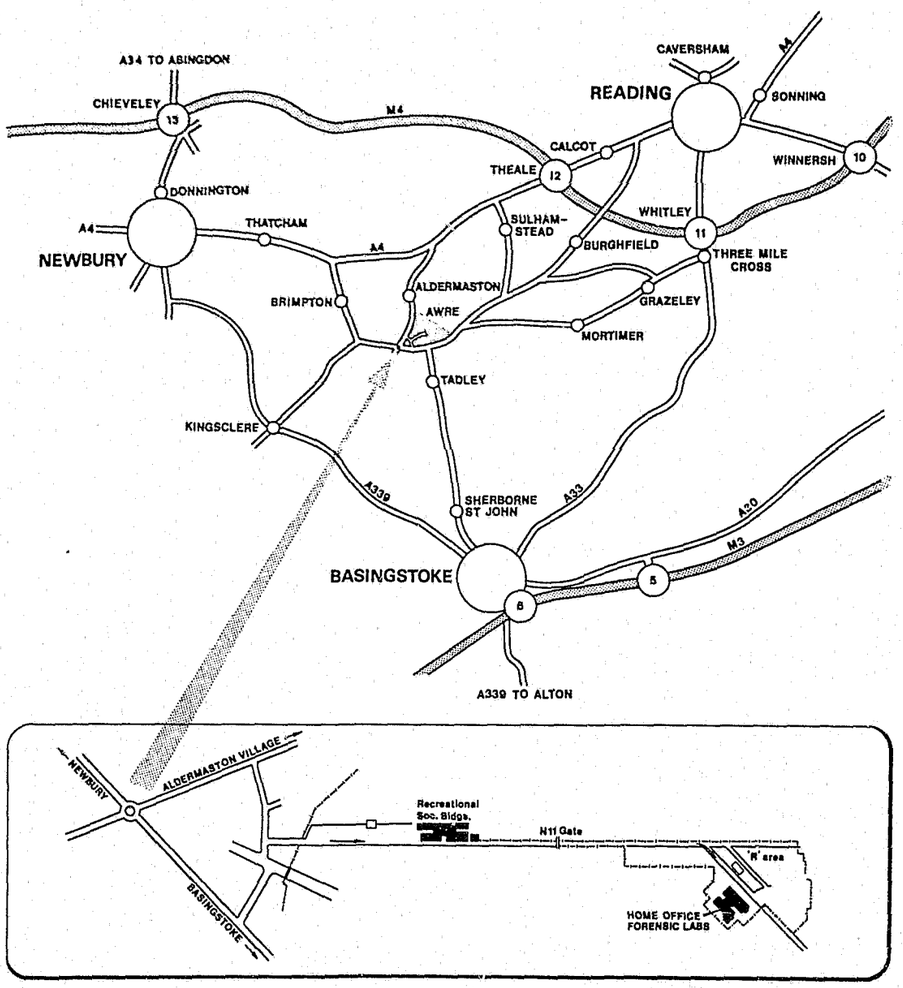


FIGURE 7. APPROACH ROADS TO AWRE AND ACCESS MAP TO HOME OFFICE LABORATORIES

END