2010

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IERDE Informal
Research Publications

1971

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PROCUREMENT EXECUTIVE

MINISTRY OF DEFENCE

EXPLOSIVES RESEARCH AND
DEVELOPMENT ESTABLISHMENT

INFORMAL PUBLICATIONS 1971

INFORMAL

# ERDE ~~Open~~ Publications 1971

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| N T No 32 | Testing materials by exposure - 'down under' and at home |
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| N T No 49 | A new one-stage pseudo-Grignard reaction |

# ERDE

## AUTOXIDATION RESEARCH

The oxidative degradation of organic materials and its inhibition covers a wide area of defence and general industrial applications, in particular food, textiles, plastics and rubber. The annual damage to the national economy which is due to oxidative degradation of polymers alone runs into millions of pounds and in defence applications the safeguards against oxidative degradation are expected to be outstandingly effective. This branch has, therefore, concentrated its efforts on the elucidation of the mechanism of autoxidation, including metal catalysis and synergism in antioxidant action, as well as the development of new antioxidants for commercial uses and defence applications.

## A NEW NICKEL CHELATE ANTIOXIDANT AND ULTRAVIOLET STABILIZER (1), (2), (3)

Most conventional antioxidants act as free radical acceptors. It has been known for some time that a combination of antioxidants can exhibit synergism, i.e. an inhibitory effect which is greater than that expected on the basis of additivity alone. Synergism is particularly significant when one antioxidant is mainly a free radical acceptor whilst the other is mainly a peroxide decomposer. The mechanism of synergism is not yet fully understood and the scope for further advances is significant. We found auto-synergism with a new nickel chelate antioxidant, i.e. bis(stilbenedithiolate)nickel, abbreviated to NiSDT. This nickel chelate combines uniquely in an outstanding manner the properties of a free radical acceptor, peroxide decomposer and UV stabilizer. An illustration of the outstanding efficiency of our antioxidant is given in the table overleaf.

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References


(1) C Copping and N Uri, British Patent Application.

(2) C Copping and N Uri, Discuss. Faraday Soc, 46, 202 (1968).

(3) N Uri, Israel J Chem, 8, 125 (1970). (Lecture delivered at L Farkas Memorial Symposium).



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INDUCTION PERIODS FOR THE AUTOXIDATION OF POLYPROPYLENE (HF-20) FILMS (0.2 mm THICK). (TEMPERATURE = 155°C)

| Antioxidant | Induction Period (hours) |
|---|---|
| None | <0.5 |
| 0.5% Ionox 330 | 625 |
| 0.5% A 2246 | 325 |
| 0.5% BHA | 26 |
| 0.5% propyl gallate | 18 |
| 0.5% Topanol CA | 205 |
| 0.5% PBNA | 145 |
| 0.5% Nonox ZA | 147 |
| 0.5% Flexone 5L | 176 |
| 0.5% DPPD | 428 |
| 0.5% NiSDT (the antioxidant of our invention) | 2012 |
| 0.25% Nickel dicyclohexyl dithiophosphate | 95 |
| 0.25% Ferro AM 101 | 29 |

Defence applications and commercial uses are being actively explored. Two British companies are at present carrying out tropical trials and other tests with polyolefins containing our NiSDT.

Enquiries should be addressed to:

Autoxidation Research

DD/1

# ERDE

AUTOXIDATION RESEARCH

## CHROMIUM DIPS* AS A NEW CATALYST FOR THE CURE OF LIQUID RUBBERS, ADHESIVES, ETC. (1), (2)

As a fall-out of CTPB propellant binder chemistry work by the Autoxidation Research Branch a new catalyst was found for the reaction between carboxylic acids and epoxides which could find widespread use in defence as well as commercial applications. A review of this work was presented by Dr N Uri at a joint IRI, PI and SCI Conference on Advances in Polymer Science and Technology III, held at the University of London 29 September-1 October 1970. This review paper has now been published (2) and a summary is available for distribution.

A licensing agreement has been concluded between the Ministry of Defence and Graesser Salicylates Ltd for the commercial exploitation of the catalyst which is expected to reach a production of at least five tons per annum in the near future. The extent of its ultimate use cannot as yet be foreseen but it could be very large if liquid rubber technology makes an impact. Defence applications in the field of adhesives are being actively explored.

Enquiries should be addressed to:

Autoxidation Research

DD/2

References

(1) A T Betts and N Uri, British Patent Application 38998/68.

(2) N Uri, Br Polym J, 3, 138 (1971).

*DIPS = 3,5-diisopropylsalicylato

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# ERDE

RESEARCH AND DEVELOPMENT OF INITIATING EXPLOSIVES

Initiators or primary explosives are so called because they are in the first stage of an explosive train. They must respond unfailingly to the stimulus of a very small amount of energy and yet must be safe to manufacture and load and be sufficiently stable and compatible to meet service and storage conditions.

WHY RESEARCH?

These requirements severely limit the choice in a class which can be described as chemical freaks. The few well-known initiators have disadvantages; from time to time they are reported either to fail or to explode inadvertently. Thus the traditional primary explosive, mercury fulminate, has good initiating properties but poor thermal stability so that it becomes unreliable, and eventually inert, after storage in hot climates; it is also chemically incompatible with aluminium. Its replacement is now almost complete but only by a number of newer explosives.

Much of the research is aimed at finding initiators with longer service life and better compatibility to eliminate troublesome and expensive replacement programmes. Modern requirements include stores to withstand both very low and very high temperatures and severe mechanical stress and the need to meet the sensitivity and other characteristics of more sophisticated designs and devices.

There is also the ever-present need to maintain and if possible increase the margin of safety at all stages of manufacture and in use; for example, by substituting an explosive less sensitive mechanically in an electrical device. Accidents are costly, yet the failure of an initiator even on one single occasion could waste much money and effort expended upon a weapon and the means of getting it to its target.

Research on sensitive explosives has the wider objective of relating chemical composition and physical properties, in which crystallization plays an important part. Some of these compounds, such as lead azide, are chemically simple but their crystalline properties are complex and the reason for their exceptional explosive properties is still not understood.

STANDARDIZATION OF EQUIPMENT AND PROCESSES

The ERDE Initiator Section has facilities, which are believed to be unique, for a 'pipeline' scheme of development from the first preparation of a new initiator in minute quantities up to the full manufacturing scale. An essential feature is the standardization of versatile equipment on both semi-technical and production scales. Thus production-type samples can be provided for assessment trials which are much more costly than the explosive. The new process and material can be demonstrated at ERDE and reproduced quickly in any one of the standardized units installed in many research establishments and factories in this country and overseas.

Another advantage is that a potential customer for a licence to manufacture can confirm that the process is in operation on a production scale and that any problems which might arise (such as the supply and specification of an ingredient) could be investigated at ERDE under the right conditions.

The success of this scheme has been dependent on three main provisions:

1 The availability of the specially designed buildings and equipment

2 Staff with experience of both laboratory research and factory development

3 Active collaboration with other sections in ERDE concerned with ingredients, compatibility, crystallography, sensitiveness and safety.

## THE RD1300 SERIES

Standardization is identified with a number in the RD1300 series. This became necessary when it was appreciated that the conventional chemical name was inadequate to describe the special characteristics of the composition even as a single substance. Thus under 'lead styphnate' there are several RD1300 compositions which are different either chemically or physically from the commercial explosive as described in the literature. These various modifications have been developed for such diverse applications as fuseheads, bridgewire electric igniters, conducting composition devices, primers and percussion caps and fuze delays.

Another example of a modification is RD1357 which is B type tetrazene in free-flowing granular form (UK Patent No 985,293); it is used as a sensitizing explosive and is in production in Pakistan with licence agreements in Belgium and Sweden.

The RD 1300 series includes new initiators which have been put into production for the first time with a wide range of applications from explosively-operated switches through detonators and fuzes to cable-cutters in release mechanisms, in fact wherever the convenient potted power of a small quantity of explosive can be put to work.

## CRYSTALS

A common problem with primary explosives is that when prepared by normal chemical methods the particle size and shape makes them unsuitable for technical use with difficult and possibly dangerous processing and handling. For example silver azide could not be utilized until such difficulties were overcome.

Special crystallization techniques have been developed with novel nucleation methods and the use of additives to modify either without diluting the explosive, or to phlegmatize it to make it safe to handle. Mechanical mixtures have been replaced by incorporating an insoluble component within the explosive crystals, new polymorphic modifications have been discovered and the phenomenon of spontaneous explosion during crystal growth has been studied.

Finally, the possible application of novel crystallization techniques to substances other than explosives (e. g. urea) may be noted.

Enquiries should be addressed to :

Explosives Branch

E/2

## USE OF PRESSURE/TIME STUDIES IN INITIATOR DEVELOPMENT

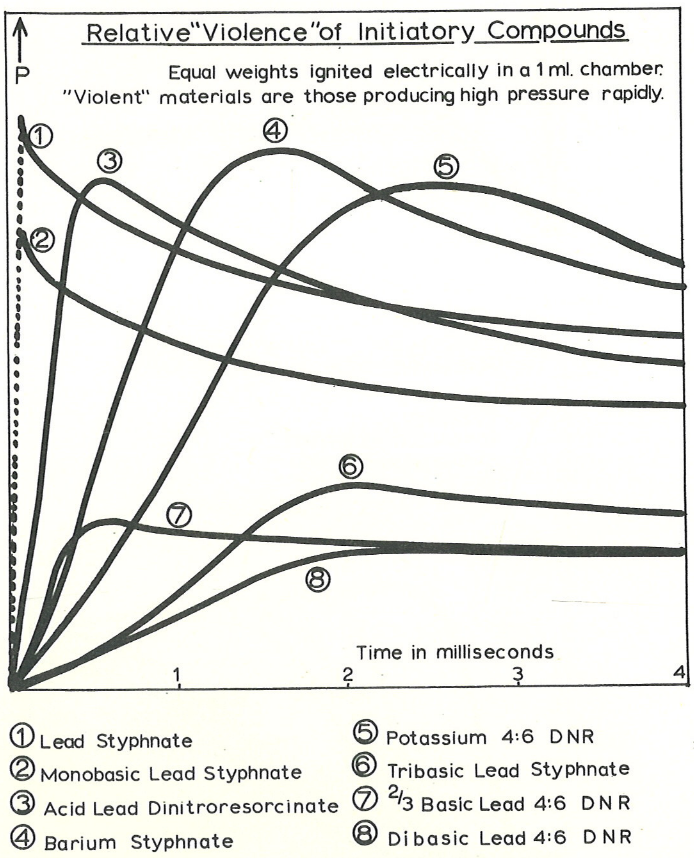
Igniting a small quantity of an initiating explosive or a device based on it in a small volume and observing the variation of pressure has been used for the following purposes:

1 assessment of the 'violence' or disruptive effect of the material;

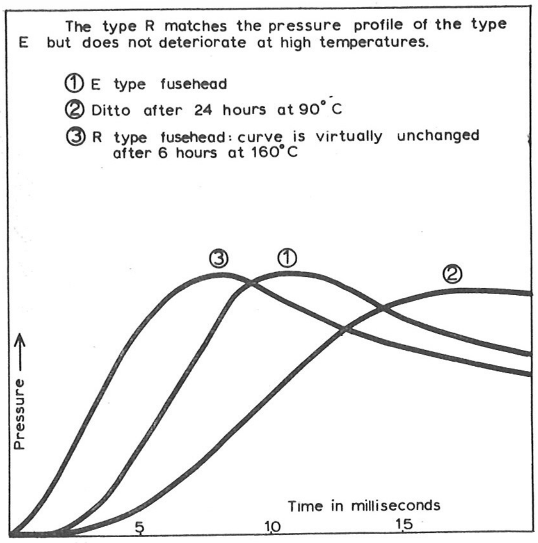
2 matching a pressure/time profile of desired shape;

3 determining the effect of exposure to an adverse environment.

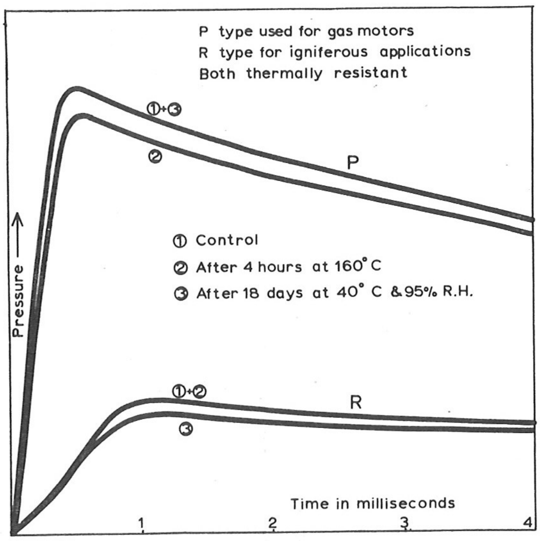
The equipment consists of a firing chamber into which are fitted a quartz crystal transducer and the device (a capsule or a fusehead) under test. Arrangements can be made to heat or cool the chamber or to fire into a large chamber at low pressure. The pressure change is displayed on an oscilloscope and the trace photographed.



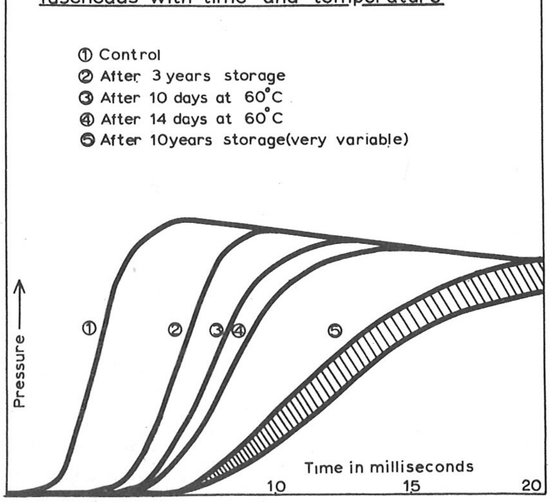
## High Temperature Fuseheads I




## High Temperature Fuseheads II




## Change in output of standard igniferous fuseheads with time and temperature




**Enquiries should be addressed to:**

**Explosives Branch**

# ERDE

EXPLOSIVES HAZARD ASSESSMENT

This exhibit illustrates some of the aspects of the work of the Sensitiveness Section, which deals with a wide range of problems connected with the preparation, storage and handling of explosive and other hazardous materials.

One of the principal responsibilities of the Section is the preparation of official Safety Certificates, this being a mandatory requirement before the manufacture of a new explosive composition in Royal Ordnance Factories may proceed. Conditions for handling and storage are laid down on the basis of the data contained in the Certificate. Methods for the tests stipulated for the Certificates are rigorously specified and members of the Section have taken an active part in the drafting of procedures. All of the following tests are carried out at ERDE

1 ROTTER IMPACT TEST

A small specified quantity of the explosive is struck by a falling weight. The criterion of a fire is the production of a specified volume of gas as measured in a gas burette. Sensitiveness is essentially a statistical phenomenon, and the result of a series of impact experiments with the material under test and with a standard explosive at differing heights of fall are analysed to give a Relative Median Height or 'Figure of Insensitiveness'.

2 MALLET FRICTION TEST

A small quantity of the explosive is placed upon an anvil, and struck a glancing blow by a mallet held in the operator's hand. A boxwood mallet is used with anvils of softwood, hardwood or York stone, and a mild steel mallet is used with anvils of mild steel, aluminium bronze and naval brass. Although reasonably consistent results are obtained by different operators, it is hoped in due course to replace this test by means of a friction machine currently under development.

3 TEMPERATURE OF IGNITION

A small specified quantity is heated at 5$^{o}$C per minute and the temperature of ignition is noted.

4 EASE OF IGNITION

A specified quantity is placed in a tube and subjected to the burst of flame and sparks emitted from a piece of Bickford fuse. The nature of the response (non-ignition, burning or explosion) is recorded.

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## 5 TRAIN TEST

An unconfined train of material is ignited at one end by a naked flame and observations are made of the liability of the sample to continue to burn and how vigorously it does so.

## 6 ELECTRIC SPARK TEST

Specified quantities of the explosive are subjected to sparks of energy 4.5 joules, 0.45 joules and 0.045 joules successively until a run of 50 trials at the same energy gives no ignition. These materials ignited with the lowest energy i.e. 0.045 joules are likely to need stringent safety precautions and so receive further testing (see leaflet on 'Electrostatic Hazards').

Extra tests are available for assessing the impact and friction sensitiveness of especially sensitive materials.

All of the statutory tests are carried out on relatively small quantities of material, usually in powdered form, and it is realised that not all the risks liable to be encountered in practice with explosives in the mass are covered. This has led to the development of a wide range of further tests, most of which involve larger quantities of explosive. These include:

(a) The various versions of the Gap Test, in which the material under test is initiated by a standard donor charge, the output of which is attenuated by a gap of brass shim or cardboard of variable thickness. The nature of the response (non-initiation, combustion or detonation) is determined by the use of either a witness plate behind the charge under test, or flag gauges. The results are analysed statistically as in the Rotter impact test.

(b) Sealed vessel tests in which the material is heated or ignited under heavy confinement. The nature of the response, combustion or detonation, is assessed from the state of the remains of the vessel, and is used for determining the safety distance category.

(c) A drop test in which a spigot is forced through the thin metal lid of a container filled with the explosive.

(d) An oblique impact test in which a charge is dropped at an angle on to a rough surface.

(e) A 'snatch friction' test in which a frictional force is rapidly applied to a small charge. This test differs from the mallet friction test in that friction occurs only between one rough surface and one surface of the explosive charge. (Tests (d) and (e) were originally developed at AWRE).

(f) A bonfire test in which the explosive contained in a wooden box is enveloped in flames from a 'standard bonfire'.

(g) The handling of liquid explosives introduces an additional hazard i.e. the adiabatic compression by pressure impulse of bubbles present in the liquid, leading to ignition of the vapour contained therein. Two tests are available which aim to reproduce this situation.

Enquiries should be addressed to:

Explosives Branch

E/4

# ERDE

## ELECTROSTATIC HAZARDS

Electrostatic charges are produced whenever separation of surfaces occurs, and the higher the electrical resistivity of the materials involved the larger are the charges observed. A discharge to a conductor at a lower potential, or to earth, may take place directly from one of the surfaces, or more usually via an induced charge on an adjacent insulated conductor. This discharge may ignite an explosive material or solvent vapour/air mixture in its path, with disastrous results.

In estimating the electrostatic risk in a process, information is needed on the magnitude of charge likely to be generated, and on the level of energy required for ignition of the explosive or flammable mixture. Two sources of energy are personnel who become charged as a result of their movements, and containers which become charged as a result of material being poured into them, or as a result of their contents being withdrawn. In this country the maximum energy that a person can acquire is reckoned to be 0.02 joules, i.e. the energy as a result of charging the capacitance of a human being, taken as 400 $\mu\mu$F, to a potential of 10 kilovolts. This figure of 0.02 joules provides a convenient level for subdivision of precaution, and the chief concern is with the handling of primary explosives, solvent/air mixtures and electrically initiated stores, since these can be ignited with energies less than this value.

In the case of primary explosives the energies for ignition by electrostatic spark are determined using an apparatus with either two steel electrodes, or one steel and one conducting rubber electrode. The first arrangement covers the occasions when sparks discharge from one metal body to another, and the second arrangement simulates the type of discharge from the finger of a charged person. This alternative method of test was developed at ERDE, following studies of the discharges from charged persons. With the two steel electrodes, tests with the two capacitances are sufficient to determine the threshold ignition energy. However with the conducting base electrode, it is necessary to cover a wider range of capacitance, leading to the determination of a minimum ignition energy and a minimum capacitance for ignition. The energy values obtained for most primary explosives are 1-100 microjoules. Those for solvent vapour/air mixtures range from 10 microjoules upwards.

Potentials attained by personnel as a result of walking and other movements are proportional to the leakage resistance to earth, and to avoid high values it is essential to reduce this resistance to less than 1 megohm for handling primary explosives. This is achieved by the use of footwear and flooring made sufficiently conducting by the incorporation of carbon black. However ⇨

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even if the operative's body is adequately earthed it is possible for significant charging to occur on outer clothing if this should become insulated from the body.

Powders and liquids of high electrical resistivity are readily charged on pouring from one container to another. This charge can be reduced to a safer level by increasing the conductivity of the powder or liquid, or by raising the relative humidity of the surrounding atmosphere to 65 per cent. Relative humidity and temperature have been found to be interdependent, enabling the hazard under conditions in less temperate climates to be assessed.

The electrostatic charging of containers and packaging for detonators and other electrically initiated stores is dependent on factors similar to those considered above. The replacement of natural products by synthetic materials has increased the magnitude of the charge attained on handling the container, and on withdrawal of its contents, since a much higher leakage resistance is presented - generally greater than $10^{10}$ ohms. Larger areas of contact and subsequent separation give rise to larger charges and hence potentially greater hazards. The charging of fabrics, and porous material in particular, is affected by their surface condition. The surface leakage resistance of most of these materials is reduced by raising the relative humidity.

These factors should be taken into account in designing the electrically initiated store and its container. If it is not possible to prevent electrostatic charges reaching the igniter, or the latter cannot be made sufficiently insensitive, the best solution is to reduce the electrical resistance of the container and packaging. This is generally achieved by incorporation of carbon black.

ERDE is the main adviser on electrostatic hazards in connection with flooring materials, footwear, clothing, container and packaging materials, and textiles to the Ministry of Defence and the Ordnance Board (Electrical/Explosives Hazards Committee) and advice is given at enquiries into accidents where it is thought that electrostatic factors are involved.

Enquiries should be addressed to:

Explosives Branch

# ERDE

## APPLICATIONS OF LINEAR CHARGES

Linearly distributed explosive charges are ideally suited for the generation of long duration pressure waveforms. This is because, when observed along the charge axis, the pressure wave duration from a charge of length L must be of order $L/_c$ where c is the sound speed; it is thus only necessary to choose L long enough in order to obtain any required duration. The more difficult problem is the determination of the required form of explosive distribution to produce the specified shape of waveform.

A second potentially useful property of the air blast from the linear charge is its spatial asymmetry. Observed along the perpendicular bisector to the charge, the blast waveform is very similar to that from a point source explosion, and is therefore of comparatively short duration. However, a larger proportion of the radiated energy is propagated in this direction as compared to the axial direction, so that the amplitude is usually much greater than that observed on-axis. Thus the possibility exists of propagating blast into preferred directions.

Since we cannot obtain both the extended duration property and the maximized energy property in the same direction the linear charge may seem at first sight an inefficient means of generating long duration waveforms. Compared, however, to the energy release that would be required from a conventional 'lumped' explosive charge, the linear charge is very efficient indeed, since in the former case wave duration is roughly proportional to $E^{\frac{1}{3}}$ whereas in the latter it is proportional to L (and therefore to E).

### 1 SONIC BANG SIMULATION

Two types of linear explosive simulant have been designed, known as Mark 1 and Mark 2. Mark 1 matches the shape and spectrum of the N-wave (idealized sonic bang) up to frequencies of about 100Hz. It is thus ideal for use in studies of the effects of sonic bangs on buildings and other structures which are sensitive to these lower frequencies. To date, versions have been made for 100 ms and 200 ms N-waves.

Mark 2 simulant reproduces the bow and stern shocks of the sonic bang only, i. e. it simulates the N-wave in the audio-frequency part of the spectrum. It is thus suitable for physiological and psycho-social studies of the effects of sonic bangs. Versions can be produced corresponding to any duration of N-wave and the loudness is much more controllable than that from 'point-source' explosions.* ⇨

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In this country, these simulants have been used in studies of the response of window structures (RAE, Exercise BABEL), and of temporary shift of hearing threshold (APRE and ISVR joint exercise). Use of these simulants has been made in France; also in America by the US Army and US Forest Service.

## 2 SIMULATION OF ROCKET TAKE-OFF BLAST

The blast waveform at a point on the ground near a rocket launching pad has been simulated, for the British Aircraft Corporation, Bristol. This simulant enabled the adverse effects of take-off blast on launch control equipment to be assessed without actually launching rockets, thereby avoiding a very considerable expenditure of money.

* REFERENCE: Hawkins, S.J. and Hicks, J.A., Nature, 211, 2055, 1244 (1966).

Enquiries should be addressed to:

Explosives Branch

E/6

# ERDE

TIME-RESOLVED SPECTROSCOPY OF TRANSIENT EVENTS

The combination of spectrograph, photomultipliers, oscilloscopes and camera permits time- and spectrally-resolved photometry of transient events. In such an apparatus in use at ERDE, several exit slits in the focal curve of a spectrograph transmit narrow wavebands of visible and near infra-red radiation on to end window photomultipliers types EMI 9558 and Mullard 150 CVP. The electrical outputs from the photomultipliers are fed via coaxial cables into Tektronix oscilloscopes types 551 and 555 fitted with type C19 cameras. Care is taken that the photomultipliers are operated under conditions which preclude inter-dynode space-charge effects, so that deflection of the CRO trace is a linear function of radiation intensity. The spectral locations of the exit slits are determined by calibrations with an iron arc. Photometry is made quantitative in terms of spectral radiation intensity by calibrating the apparatus with a tungsten ribbon lamp which has itself been calibrated at the National Physical Laboratory. The time bases of the oscillopscopes yield the time history of the phenomena studied. The synchronization of the camera shutters, the CRO time bases and the transient event to be studied is achieved by the use of suitable delay circuits.

We have used this technique to measure temperatures both in condensed phase detonations and in gaseous shock waves. Anticipating the results, there are some detonation and strong shock waves where the temperature and pressure are high enough for the front to radiate as a grey or even a black body. Radiation intensity measurements made at two or more wavelengths may thus be used to solve Planck's equation to give temperature and emissivity.

$$I_\lambda = C_1 \epsilon \lambda^{-5} \left[ \left(\exp \frac{C_2}{\lambda T}\right) - 1 \right]^{-1}$$

DETONATIONS

As an example, our experiments on the liquid explosive nitromethane showed that its detonation front radiates as a black body at a temperature of about 3460°K. The high emissivity may be due in part to the presence of free carbon in the detonation products and it is intended to elucidate this point by experiments with carbon-free explosives.

A knowledge of detonation temperature is important in testing equations of state which are valid under the detonation conditions of 3000-4000°K and 200-400 kilobars. It enables an assessment to be made of the relative contributions of thermal and repulsion energies to the internal energy of the system. Under detonation conditions the repulsion energy may represent as much as 50 per cent of the total energy.

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INTENSE SHOCKS IN RARE GASES

We have used the same apparatus to investigate the radiation emitted by argon and other rare gases when strongly shocked by explosives. The high luminosity (millions of candlepower) of such shock waves is frequently utilized in high speed photography. Our experiments have shown that strongly shocked rare gases emit black body radiation; in the case of argon, (initially at 1 atm $20^{o}C$) the temperature was about $21000^{o}K$ when the shock was driven by the explosive RDX/TNT 60/40. Under these conditions the gas was about 75% ionized. The calculated shock pressure was 1000 atm and the calculated temperature based on hydrodynamic and thermodynamic considerations was $19880^{o}K$.

Work is at present in progress on the determination of detonation temperature of solid explosives. As most solid explosives are opaque the detonation wave may be observed only at the instant it emerges from the surface of the explosive. It is hoped that some insight will be gained into the kinetics and physics of the detonation process in new high-energy composite explosives.

Enquiries should be addressed to

Explosives Branch

E/7

# ERDE

## ELASTIC PYROTECHNICS - A NEW APPROACH TO AN OLD ART

Fireworks or pyrotechnics have been in use for centuries for purposes such as terrifying the enemy in war and celebrating the resultant victory. In modern warfare, pyrotechnics provide means of signalling with light or smoke, illuminating targets and guiding missiles, igniting rockets, setting time intervals (delays) and so on. The conditions under which they do so become more varied and exacting as shells fly faster, aircraft and rockets higher.

Pyrotechnic compositions consist of something to burn - fuel - and something to burn it - oxidant - chosen to suit the application. For example, for bright light, magnesium powder is included; for coloured light, a metallic salt oxidant, giving the flame this colour; slow-burning compositions are produced by using additives, a mild oxidant or an organic fuel.

Conventional pyrotechnic compositions are usually mixtures of powders which are pressed into containers often under high loads in heavy presses. Even so, many pressed charges crack fairly easily under shock or vibration giving rise to inconveniently rapid burning; they are also susceptible to damage by moisture and hazardous to mix and process.

Elastic pyrotechnics developed at ERDE can overcome these drawbacks in many cases. The constituents are bound together and protected by a rubbery matrix to form a robust, resilient charge; consolidation is carried out by hand-pressure in simple moulds, or extrusions can be made; 'loose' granular mixtures are non-segregating, in contrast to uncompressed powder mixtures. Charges are so little affected by moisture that they will function after a period of immersion in water.

Compositions are made by adding the oxidants in some instances, the fuels in others, to a liquid polymer, together with curing agents; mixing until the powders are wetted completely; then adding the other constituents in portions, mixing until a homogeneous mass is produced. In this way, fuel and oxidant particles are always separated by a film of liquid during mixing and the risk of an accidental ignition is small. The uncured composition, which may range in consistency from a coherent to a crumbly paste, can be kept in a refrigerator for several months and cured in about 16 hours at 70$^{\circ}$C.

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Compositions have been developed for the following applications:

- priming in rolled sheet form to replace gunpowder primed cambric and extruded cords, bare or encased in lead tubing;
- coloured signal, rocket tracking and illuminating flares;
- screening smoke;
- rocket igniters.

Enquiries should be addressed to:

Explosives Branch

E/9

# ERDE

HIGH SPEED PHOTOGRAPHY

Modern high speed cameras are essential tools in present day explosives research.

In ERDE wide use is made of both framing and streak cameras to study explosive behaviour both qualitatively and quantitatively.

Optical framing cameras may be divided into two distinct classes - those using 16 mm movie film giving picture rates up to about 18,000 per second, and those using larger format (35 mm) movie film and giving framing rates from about 100,000 to over 4 million per second. Examples of the first type used in ERDE are the Fastax and the Hycam, of which the Hycam Model K2001 is presently used in the Explosives Branch. This is a camera of the optical compensation type, and will give picture rates of 100-9000 per second in the full-frame mode and 200-18000 per second in the half-frame mode.

Such framing rates are appropriate in the study of flames and low order deflagrations as well as in the analysis of mechanical failure of apparatus under development. Present work includes an investigation into fragment attack using the Hycam in its 18,000 pps mode, in conjunction with suitable electronic flask, to photograph high velocity fragments.

For the study of extreme speed explosive phenomena, such as shock and detonation waves, ultra high speed cameras using the larger 35 mm format are necessary. The framing camera presently used is the Beckman and Whitley Model 189. This is of the Miller type in which the film is stationary and the light image is swept over it by a turbine-driven steel alloy mirror. Optical shuttering provides a series of 25 frames at rates of between $10^5$ and $4 \times 10^6$ per second depending on the type of turbine in use plus the fact that the camera is a non-continuous access model.

These extreme speeds give rise to certain synchronization problems. Commercial hot wire type detonators were found to have too variable a time delay and a programme of work was initiated to design a detonator that would have a maximum variability of plus or minus one frame at the maximum framing rate of 5000 rps turbine. The result was a high voltage spark initiated detonator containing a mixture of 98/2 PETN/aluminium powder with an operating time of 13.9 $\mu$sec and a standard deviation of 1.1 $\mu$sec. This has proved to be extremely reliable in many hundreds of firings.

To photograph non-luminous events such as non-reactive shock waves very intense front lighting must be employed. Standard electronic flash techniques cannot be used because of the limited intensity and normally destructive nature of the event. Recourse has to be made to the now standard technique of argon flash bombs. These are made within the Section and can be so constructed as to give the intensity and duration required to illuminate the event.

The main field of work in which the framing camera has been used in recent years is the study of shock and detonation waves in explosives. It is currently being used in an investigation into the initiation of explosives and propellants by shocks of known amplitude.

For quantitative measurements of detonation and shock-wave velocities the high-speed streak camera is indispensable. This, unlike the framing cameras, does not produce a series of discrete pictures but rather a space/time curve of the event photographed. It is basically similar to the framing camera already described except that there is no optical shuttering action, the light image, of slit form, being swept by a rotating steel mirror directly over a length of 35 or 120 mm film. Thus, knowing the writing speed and demagnification of the optics, velocity measurements can be readily calculated. The cameras in use are the Beckman and Whitley Model 339B with a maximum writing rate of 8.5 mm per micro-second and the Type RM120 with a maximum rate of 2 mm per microsecond.

References:

Special Detonators for Photographic Use: E L Kendrew and R M H Wyatt. Seventh International Congress on High Speed Photography, Zurich, September 1965.

# ERDE

## DETECTION OF EXPLOSIVES

Explosives have many legitimate uses in both military and civil life. However, illegitimate use of explosives, whether for personal or political ends, obviously introduces unexpected hazards into normal life. Lately some attention has been focussed on the problem of detecting the presence of explosives intended for illegitimate usage.

Explosives now commonly available may be classified into three families: (1) nitro bodies, (2) nitrate esters, and (3) fuel/oxidant mixtures. The first class is mainly the military preserve, and may be exemplified by such explosives as TNT, RDX, and plastic explosive. The second class, of which nitroglycerine and diethyleneglycol dinitrate (DEGN) are examples, find their main use in mining, quarrying, and civil engineering, in such forms as dynamite and gelignite. The third class are also blasting explosives: eg ammonium nitrate/fuel oil mixtures, or gelled aqueous slurries of nitrate salt mixtures. Gunpowder and other pyrotechnics would fall within this class, as do such hazardous, amateur mixtures as sugar/sodium chlorate.

None of these types of compound or mixtures possess any unique physical property (such as magnetism or radio-opacity) which would serve as the basis of a remote means of detection. The best hope in these lines would seem to be in the higher-than-average nitrogen content, which might be susceptible to neutron activation analysis.

It might be expected that the first two classes, comprising as they do organic molecules, would have appreciable vapour pressures, and that the low concentrations of vapours emitted might be susceptible to analysis. The range of vapour pressures is quite wide, running from the relatively volatile TNT and DEGN to the involatile RDX. There are severe problems connected with the sampling and transmission of traces of such vapours, but some success has been achieved with gas chromatographic analysis in this field.

Another method of trace gas analysis that is partially successful is based on permeation of a molecule such as nitroglycerine through a suitable membrane, followed by detection by electron capture. Unfortunately this method suffers from lack of specificity.

ERDE has developed a specific method of analysis for nitrate esters. This is based on pyrolysis of the ester by passage over a hot wire, followed by detection of the nitrogen dioxide thus produced. The nitrogen dioxide is detected by a sensitive dye-coupling reaction (modified Saltzmann reagent), such that the appearance of a pink colour indicates the presence of nitrogen dioxide, and thus by inference of a nitrate ester.

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$$\begin{array}{l} CH_2ONO_2 \\ CHONO_2 \\ CH_2ONO_2 \end{array} \xrightarrow{\text{heat}} 3\ NO_2 + \text{degradation fragments}$$

Nitric oxide and nitrogen dioxide naturally present in the air (as by-products of the internal combustion engine, cigarette smoking, etc) cause interference, but can be easily removed by passage of the air through a scrubbing train before passage through the pyrolysis furnace.

We have developed a small portable instrument using this method of analysis, and have successfully demonstrated that it will detect the presence of dynamite in brief-cases, parcels, drawers, etc. While the level of sensitivity of this instrument (ca. $10^{-9}$) is not as high as that of gas chromatography ($10^{-12}$) its low cost and easy portability render it suitable for wide-spread use by non-technical personnel. Its specificity makes it a useful adjunct to the more sensitive ($10^{-12}$) electron capture instrument.

Enquiries should be addressed to:

GC/1

General Chemistry Branch

# ERDE

## NON-METALLIC MATERIALS BRANCH

## HEAD OF BRANCH - DR. B. L. HOLLINGSWORTH

Non-Metallic Materials Branch undertakes basic and applied research on the chemistry and physics of polymers with particular regard to synthesis, characterization, degradation, and mechanical behaviour under a range of test conditions. The Branch offers an advisory service on the applications of polymers, and supervises long-term environmental testing at the Tropical Research Unit, which is a joint project with Australia.

## ACTIVITIES AND FACILITIES

### POLYMER PHYSICS AND ENGINEERING

Study of the physical properties of non-metallic materials and reinforced plastics: stress-strain curve, strength, fracture energy, damping capacity, creep, fatigue, stress relaxation, and dimensional stability. Effects of rate of deformation, temperature and moisture. Stress relaxation tests on rings and beams; dynamic mechanical properties of discs. Compression cell to study effects of dynamic, static, and creep loading.

### EQUIPMENT

Two Hounsfield E-type tensometers (extension rate, 0.0013-50.8 cm/min; temperature, $-80^{o}$ to $80^{o}$C); Baldwin machine; Goodbrand machine (fibre test). Avery Izod impact machines (capacity 17 kg.m; maximum loading rate, $18 \times 10^2$ kN/sec; photographic recording with 35 mm streak- or 12000 frames/sec Fastex-cameras). Flywheel machine (fracture energies at impact velocities of 130-1300 cm/sec). Ultrasonic equipment (150 kc/sec-2 Mc/sec; up to $80^{o}$C). Rotating beam machine (fatigue properties). Hounsfield Charpy Impact machine; falling weight and falling dart impact machines.

### POLYMER CHEMISTRY - SYNTHESIS, CHARACTERIZATION, AND STABILITY

Investigation of novel and potentially useful polymer systems; monomer and polymer synthesis, polymer fractionation, mechanism and kinetics of polymerization, reactions of polymers.

Characterization of polymers by molecular weight (viscometry, osmometry, light scattering, and end-group analysis), spectroscopy (IR, UV and NMR), dilatometry, bulk viscometry, compressibility and thermal expansion measurements, and by optical examination of polymer morphology.

Stability of addition and condensation polymers to heat, ultra-violet and high energy radiation; effects of chemical structure, impurities and environment. Mechanism and kinetics of degradation and its effect on molecular weight and physical properties.

EQUIPMENT

Molecular weight Hewlett-Packard 502 high-speed membrane osmometer (maximum operating temperature 130°C); Brice-Phoenix Series 2000 Universal light scattering photometer with MSE high-speed centrifuge.

Bulk properties Epprecht Viscometer ($0.1$-$10^6$ poise, $0^\circ$-$180^\circ C$); linear expansion apparatus for glass transition temperatures; pressure balance (temperature controlled to $\pm$ 0.001°C).

Spectroscopy PE R10 60 M/c NMR spectrometer (variable temperature probe, spin decoupling unit, probes for $^1H$, $^{11}B$, $^{19}F$, and $^{31}P$; PE 337 grating spectrometer (4000-400 $cm^{-1}$, ATR attachment); Unicam SP 500 spectrometer.

Microscopy Gilett and Sibert time-lapse microscope (35 mm and 16 mm (cine) photographic recording).

Degradation Mercury lamp, xenon arc, molten salt- and fluidized sand baths. Mectron "Climatest" Weathering Chamber.

Gas chromatography PE Model 801 (dual column analytical instrument, linear programming); Pye Model 105/15 (automatic preparative gas chromatograph, linear programming).

POLYMER DEVELOPMENT AND APPLICATIONS

Processing and curing of new rubbers and thermoplastics by injection moulding, extrusion, milling, and compression moulding. Measurement of physical properties, and the effect of contaminants (e. g. explosives, propellants, petrol, etc. ). Accelerated and tropical ageing trials to assess useful life. Investigation of failures. Manufacture of special components; antistatic and conducting rubbers.

## EQUIPMENT

Processing  Wide range of mills, mixers, hydraulic presses and injection moulding machines. Facilities for the synthesis of polyester-polyurethane rubbers.

Testing  Physical properties; permeability of materials to organic fluids and water; ageing ovens to simulate hot/dry and hot/wet conditions; continuous and intermittent stress relaxometers.

## JOINT TROPICAL RESEARCH UNIT, QUEENSLAND

Four sites are available for the exposure of small specimens to atmospheric weathering.

a  Innisfail  Latitude 17° 32'S. Average annual rainfall 140 inches. Average daily mean temperature 74°F: relative humidity 83 to 87. Sites are:

i  Hot/wet, jungle, i.e. sunlight screened off by foliation.

ii  Hot/wet, clearing.

b  Clump Point  Similar latitude and conditions to Innisfail but the site is

iii  Marine.

c  Cloncurry  Latitude 20° 43'S. Average annual rainfall 17 inches. Average daily mean temperature 78°F: relative humidity 39. The site is designated

iv  Hot/dry.

Commercial trials on plastics, rubbers, metals, adhesives, etc., can be undertaken.

## SOME RECENT PUBLICATIONS

Fibre filled thermoplastics, Part I, Asbestos, Composites, September 1969, p. 28.

"  "  "  " II, Reinforcement by high modulus fibres, Composites, December 1969, p. 80.

Conductive rubbers, Rubb. J., December 1969, p. 30.

Comparison of thermal and thermo-oxidative stability of polycarbonate, polyphenylene oxide, polysulphone and two polyarylates, Macromol. Chem., 132, 23 (1970).

Factors affecting strength of polyhydroxyether, J.A.P.S., 12, 1385 (1968).

Synthesis and properties of regular copolymers, Part I, Polymer, 10, 603 1969).

Ageing of polyurethane rubbers, Rubb. J. June/July/August 1971

NM/1

# ERDE

## SYNTHESIS OF NEW POLYMERS

One of the major interests of the Polymer Chemistry section at ERDE is in developing methods of synthesizing new polymers which might be commercially important or structurally interesting.

Recently, a method was developed here which appears to have very wide synthetic possibilities as it enables regular co-polymers to be prepared easily and in quantity. Generally, co-polymers have chains consisting of two different monomer units linked together in a random order. The relative proportions of the two components in the chains are, however, fixed by the properties of the species involved and cannot be systematically varied over a wide range. Regular co-polymers have the monomers joined in a specified and regular order and the method, by judicious choice of ingredients, enables varying proportions of the co-monomers to be incorporated.

As co-polymers have properties of both the corresponding homo-polymers but to a lesser degree, the technique opens up the possibility of tailor-making polymers to specific customer requirements by suitable selection of components.

The method relies on the reaction of a vinyl monomer with an alkali metal in the presence of a calculated amount of linking agent such as a dihalide, or diepoxide. A strong cation solvating solvent such as tetrahydrofuran is necessary, as is the absence of interfering species such as oxygen, water etc.

For example, co-polymers of styrene and methylene in various predetermined ratios have been prepared by reacting styrene with lithium in the presence of the $\alpha$, $\omega$- dihalide Br $(CH_2)_n$ Br where n can be systematically varied.

The following reaction sequences occur:

$$Li + CH_2 = \underset{Ph}{\underset{|}{CH}} \rightleftharpoons Li^+ \, {}^-\underset{Ph}{\underset{|}{CH}} \cdots CH_2 \qquad (1)$$

$$2\,Li^+ \, \underset{Ph}{\underset{|}{CH}} \cdots CH_2 \rightarrow Li^+ \, {}^-\underset{Ph}{\underset{|}{CH}} - CH_2 - CH_2 - \underset{Ph}{\underset{|}{CH}}{}^- \, Li^+ \qquad (2)$$

$$Li^+ \, {}^-\underset{Ph}{\underset{|}{CH}} - CH_2 - CH_2 - {}^-\underset{Ph}{\underset{|}{CH}} \, Li^+ + Br\,(CH_2)_nBr \rightarrow Li^+ \, {}^-\underset{Ph}{\underset{|}{CH}} - CH_2 - CH_2 - \underset{Ph}{\underset{|}{CH}} - (CH_2)_nBr + Li\,Br \qquad (3)$$

$$\sim\!\sim \underset{Ph}{\underset{|}{CH}} - CH_2 - CH_2 - \underset{Ph}{\underset{|}{CH}} - (CH_2)_n\,Br + Li^+ \, {}^-\underset{Ph}{\underset{|}{CH}} - CH_2 \sim\!\sim - \qquad (4)$$

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$$\rightarrow \sim\sim \underset{\mid \atop Ph}{CH} - CH_2 - CH_2 - \underset{\mid \atop Ph}{CH} - (CH_2)_n - \underset{\mid \atop Ph}{CH} - CH_2 \sim\sim + Li\ Br$$

The dimerization reaction shown in equation 2 appears to be followed almost invariably by the linking reaction (equation 3) rather than a polymerization step involving addition of more vinyl monomer. This is supported by nuclear magnetic resonance evidence which gives aliphatic to aromatic hydrogen ratios in close accord with those predicted by the reaction sequences above.

A large number of regular co-polymers have been prepared using monomers such as styrene, butadiene and isoprene etc. with many and varied linking agents, and the physical properties of these materials are presently being evaluated. The fact that diene monomers may be used with this technique enables rubbers also to be prepared with closely defined structures.

Enquiries should be addressed to:

NM/2

Non-metallic Materials Branch

# ERDE

## ERDE TENSILE CREEP FACILITY

SUMMARY

The ERDE Tensile Creep Facility minimises the need for skilled laboratory technicians while increasing the reliability of the data obtained and facilitating the flow of final information.

### INTRODUCTION

An important aspect of engineering design data for polymers is the measurement of tensile creep under precise and controlled conditions. Creep itself and the many derivatives which may be obtained from the data, provide essential basic knowledge in respect of long term behaviour. Although in principle the method is similar to that of other workers [1, 2, 3] the guiding principle in the ERDE facility is to minimise the extent to which skilled laboratory technicians are required to measure accurately and calculate the numerous values obtained. Automation has been achieved and the major disadvantages normally encountered, those of drift and noise in electronic ancillaries, have been minimised to an extent where accuracy of measurement is unimpaired. The highly stable units are therefore ideally suited to the production of information for data processing.

### GENERAL DESCRIPTION

The basic creep machines are those designed by ICI and RAPRA, the former having been redesigned with respect to slide and alignment mechanisms.

A schematic diagram of the apparatus and data flow paths is shown in Figure 1.

The high sensitivity, low noise linear variable differential transformers (LVDT's) are engineered into directly mounted extensometers of low mass. The LVDT's are energised in an a. c bridge configuration which eliminates errors due to carrier fluctuations.

The output from the demodulator is logged electronically and recorded on punched tape which in turn is processed by an Elliott 903 digital computer. Programs for computer processing have been developed to provide tabulated data and permanent graphical displays suitable for direct use for design purposes. The basic curves produced are creep strain vs log time, log strain vs log time and creep compliance vs log time, which will be extended to include isochronous stress-strain curves, isometric curves and derivatives of the basic curves. Figure 2 shows an example of the computer graphic output.

REFERENCES


| | | |
|---|---|---|
| 1 | Darlington, M | Cranfield Institute of Technology - Private Communication |
| 2 | Turner, S | ICI, - Private Communication |
| 3 | Wright, D | Rubber and Plastics Research Association - Private Communication |


Technical Enquiries should be addressed to:

Non-metallic Materials Branch

NM/3

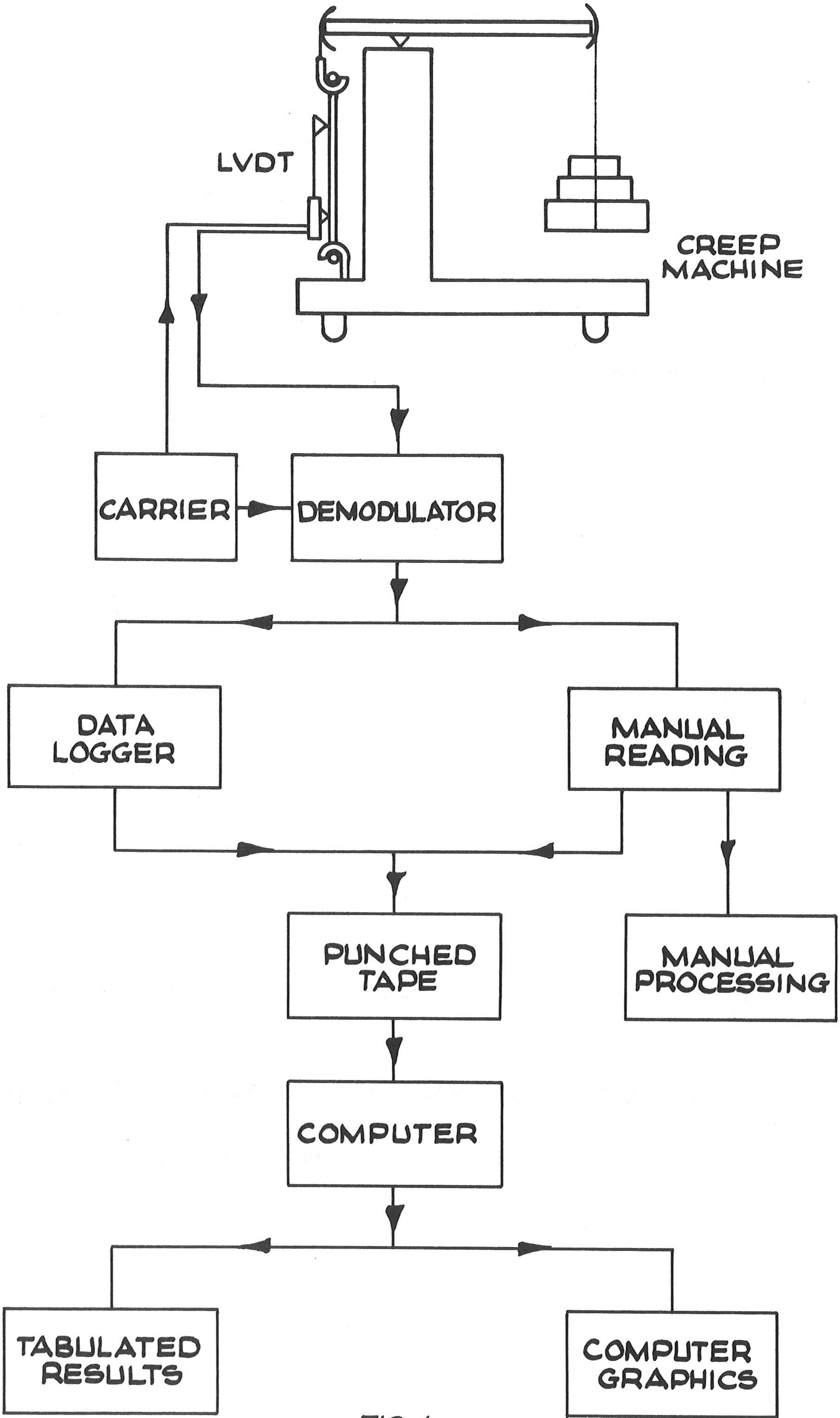
LVDT
CREEP MACHINE
CARRIER
DEMODULATOR
DATA LOGGER
MANUAL READING
PUNCHED TAPE
MANUAL PROCESSING
COMPUTER
TABULATED RESULTS
COMPUTER GRAPHICS

FIG. 1

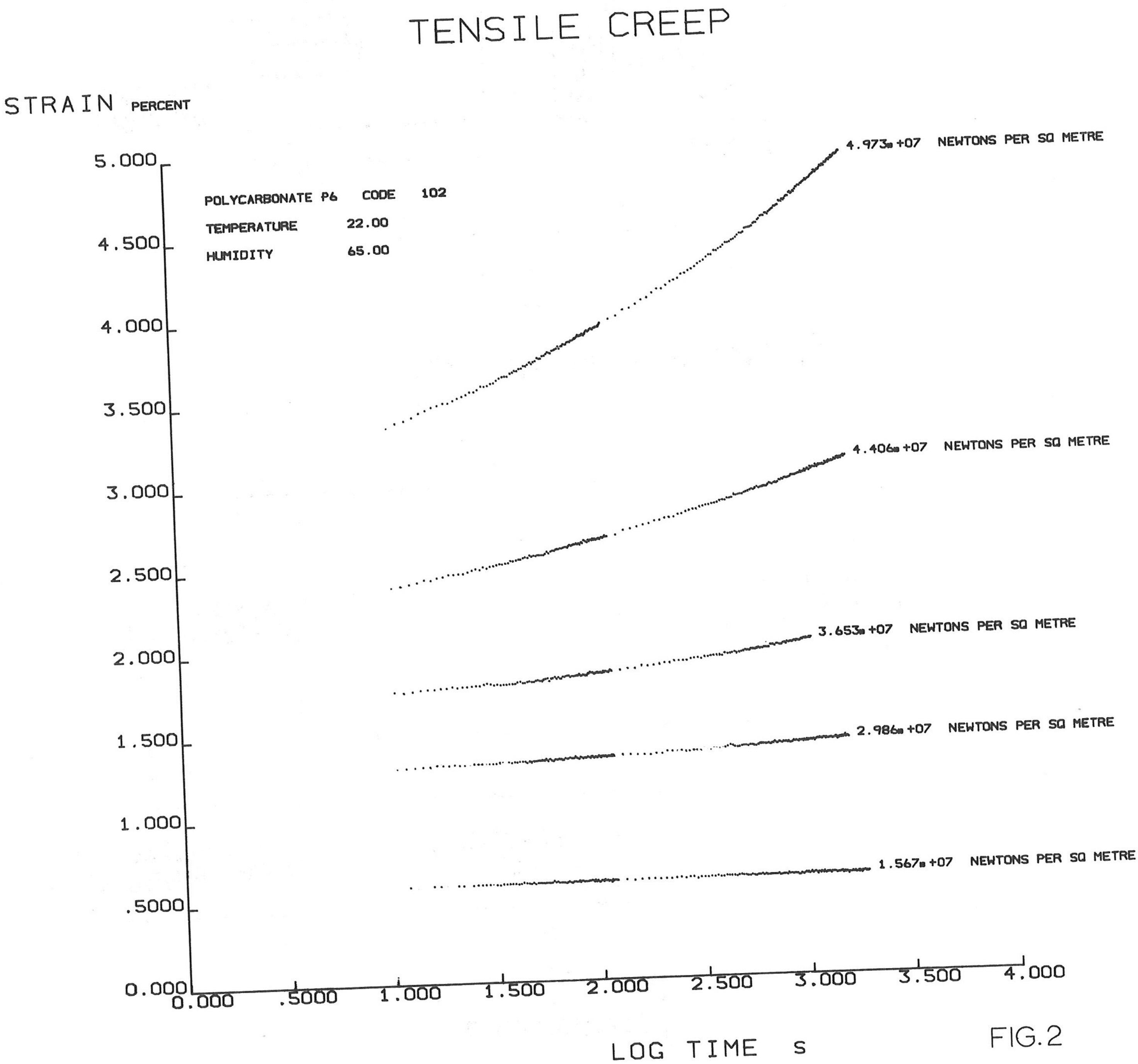
TENSILE CREEP
STRAIN PERCENT
POLYCARBONATE P6 CODE 102
TEMPERATURE 22.00
HUMIDITY 65.00
4.973 +07 NEWTONS PER SQ METRE
4.406 +07 NEWTONS PER SQ METRE
3.653 +07 NEWTONS PER SQ METRE
2.986 +07 NEWTONS PER SQ METRE
1.567 +07 NEWTONS PER SQ METRE
5.000
4.500
4.000
3.500
3.000
2.500
2.000
1.500
1.000
.5000
0.000
0.000
.5000
1.000
1.500
2.000
2.500
3.000
3.500
4.000
LOG TIME s

FIG.2

# ERDE

## APPLICATION OF DATA-PROCESSING TO MATERIALS TESTING


### SUMMARY

Data processing reduces the time required to obtain results from mechanical tests by several hundred per cent, and allows a more accurate and comprehensive assessment of material properties.


### INTRODUCTION

A large proportion of work on polymeric materials consists of routine mechanical testing, to measure tensile and flexural properties. Results from these tests form the basis for the characterisation of new materials, provide an assessment of the effect on properties of various parameters, e. g. temperature, environment, processing conditions, and assist in the investigation of in-service failures. On average, the time spent in calculating property values from experimental records can be between five to ten times that spent in conducting the test. The use of digital recording and subsequent computer processing provides the results much more quickly, and in addition has the following advantages:

1. Calculations are completed more rapidly

2. It is possible to obtain the maximum information from any one test, with a negligible increase in the calculation time

3. The results obtained are more accurate, and are not affected by the subjectivity of the observer

4. Staff are freed from this tedious task to do other work

5. Due to the rapid response of the recording system, the capabilities of the testing machine may be extended, without loss of accuracy.

### DESCRIPTION OF THE DATA COLLECTION SYSTEM

Mechanical tests are conducted on a Monsanto-Tensometer Type E tensile testing machine. This has been modified by the manufacturer so that electrical signals, derived from the load and extension measuring systems, are provided for data-logging purposes. These signals are in parallel to those that drive the chart


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recorder, and so are unaffected by any limitations of the recorder. An Electronic Associates Ltd 'Data Acquisition and Recording Terminal' (DART) measures the value of each signal in turn, and this value is then recorded on punched tape. Thus the test record consists of a series of alternate, discrete load and extension values, separated by a constant time interval. For normal purposes it has been found that a recording rate of five values a second is adequate but this can be extended to twenty a second with the existing punch if better resolution is required.

A manual entry unit developed by Electronic Associates Ltd is used to provide heading data for each test. Thumb wheel switches can be set to the values of specimen number, specimen dimensions, load range, testing rate, and this information can then be recorded on the data tape.

At the completion of a batch of tests, the logged data is processed on a computer (an Elliot 903), under the control of an appropriate program developed for this purpose.

Typical properties which are calculated are as follows:

Modulus

Stress )
)
Strain ) At yield and/or break
)
Energy )

In addition, the computer calculates various statistical properties for each batch of tests, e.g. mean, variance, coefficient of variation. An example of a set of calculated properties is given in Fig. 1. These results are from a tensile test on a thermoplastic, polycarbonate; programs have also been written to calculate properties from flexural test data, and for tensile tests on rubbers. All programs calculate properties similar to those shown in Fig. 1, and a simplified flow diagram is shown in Fig. 2. Modulus is determined by the numerical differentiation of the initial load/extension data, up to a pre-determined value of strain. Failure is detected as the region where the load values decrease, and the point of failure is determined by numerical interpolation.

A statistical comparison of results obtained by computer processing with those calculated by hand from chart records showed no significant difference. However, the time required to calculate the results on a computer, including the final typing of the output was about a tenth of that for manual calculation.

The data-logger and computer processing of data is also being used to obtain results from creep tests, and it is envisaged that the system will be applied to other test methods in the future.

A paper describing the system and its application to mechanical testing in more detail is awaiting publication.

Technical enquiries should be addressed to:

Non-metallic Materials Branch

NM/4

PDP101 N.M.SUPERINTENDENCY

THERMOPLASTIC MATERIALS.

TENSILE PROGRAM MK. 1.

VW = ENERGY ABSORBED/UNIT VOLUME IN GAUGE LENGTH.
TW = TOTAL ENERGY ABSORBED BY SPECIMEN.
IF NO VALUE IS GIVEN FOR VW,THEN THE STRAIN QUOTED UNDER THAT HEADING IS THE TOTAL SPECIMEN EXTENSION,DERIVED FROM CROSS-HEAD DISPLACEMENT.
* DENOTES SUSPECT SPECIMEN.

| | | | | | YIELD OR BREAK | | BREAK | | YIELD OR BREAK | | BREAK | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NO. | RATE | WIDTH | DEPTH | E | STRS | STRN | STRS | STRN | VW | TW | VW | TW |
| | MM/MIN | MM | MM | N/M↑2 X10-9 | N/M↑2 X10-7 | % CM | N/M↑2 X10-7 | % CM | J/CC | J | J/CC | J |
| 1 | 25.00 | 4.500 | 3.170 | 2.973 | 6.217 | 6.17 | 6.760 | 3.88 | 2.82 | 1.71 | ---- | 29.8 |
| 2 | 25.00 | 4.500 | 3.170 | .2055 | 2.613 | .477 | NO BREAK | | ---- | .622 | ---- | ----* |
| 3 | 25.00 | 4.510 | 3.180 | 3.020 | 6.149 | 6.15 | 5.479 | 2.44 | 2.78 | 1.67 | ---- | 16.6 |
| 4 | 25.00 | 4.520 | 3.180 | 2.988 | 6.158 | 6.09 | 6.238 | 3.24 | 2.75 | 1.70 | ---- | 23.5 |
| 5 | 25.00 | 4.520 | 3.180 | 3.110 | 6.150 | 5.97 | 5.515 | 2.52 | 2.77 | 1.75 | ---- | 17.1 |
| 6 | 25.00 | 4.510 | 3.160 | 3.044 | 6.189 | 6.09 | 5.830 | 2.73 | 2.78 | 1.72 | ---- | 19.1 |

STATISTICAL ANALYSIS.

| | | | | YIELD OR BREAK | | BREAK | | YIELD OR BREAK | | BREAK | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | WIDTH | DEPTH | E | STRS | STRN | STRS | STRN | VW | TW | VW | TW |
| | MM | MM | N/M↑2 X10-9 | N/M↑2 X10-7 | % CM | N/M↑2 X10-7 | % CM | J/CC | J | J/CC | J |
| MEAN | 4.512 | 3.174 | 3.027 | 6.173 | 6.09 | 5.964 | 2.96 | 2.78 | 1.71 | 0.00 | 21.2 |
| VARIANCE | .0001 | .0001 | .0029 | .0009 | .006 | .2910 | .359 | .001 | .001 | 0.00 | 30.6 |
| ST.DEV. | .0084 | .0089 | .0539 | .0297 | .076 | .5394 | .599 | .026 | .029 | 0.00 | 5.53 |
| C. OF V. | .1856 | .2816 | 1.781 | .4815 | 1.25 | 9.044 | 20.2 | .943 | 1.73 | 0.00 | 26.1 |
| N | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 0 | 5 |

FIG.1
-----

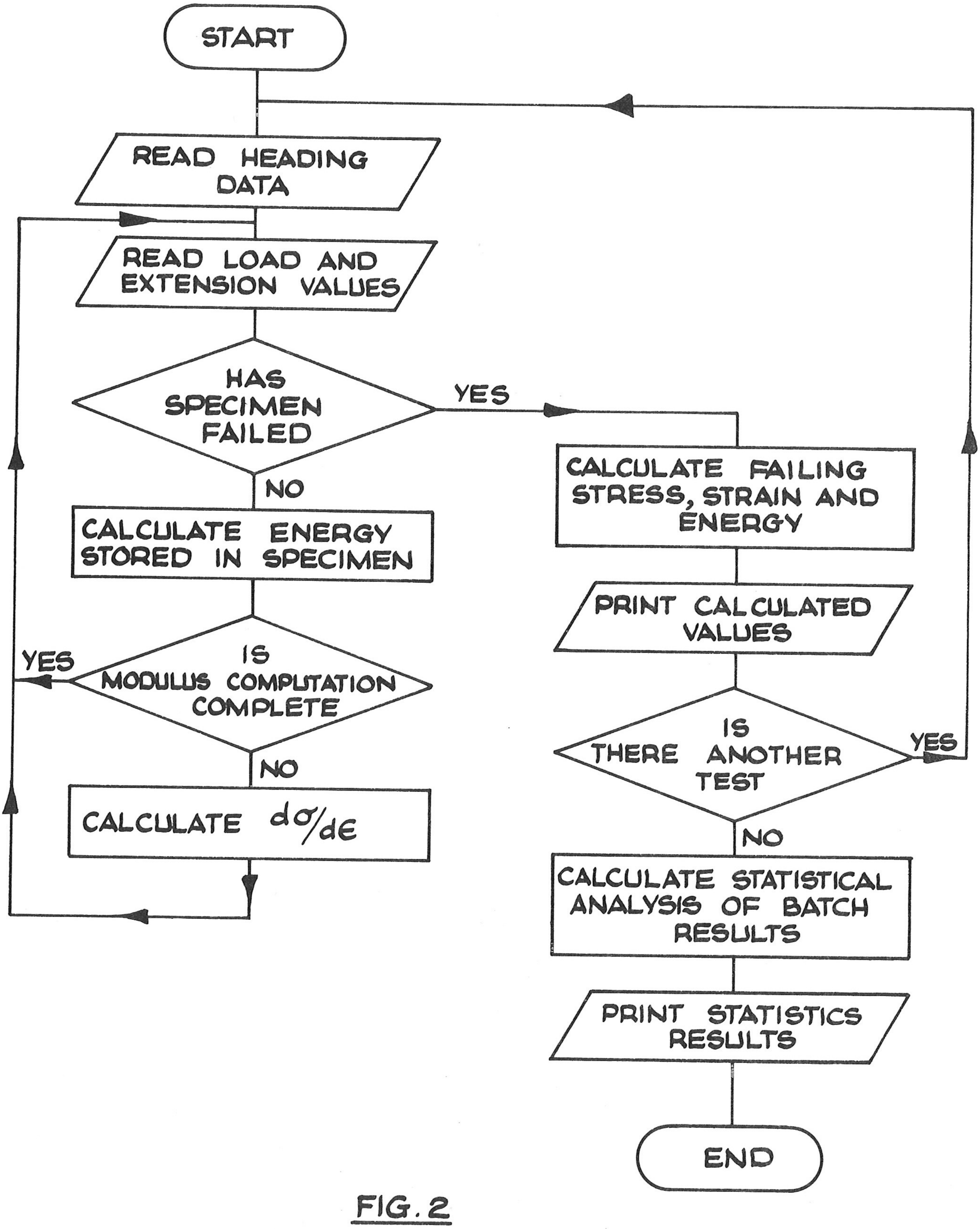
START
READ HEADING DATA
READ LOAD AND EXTENSION VALUES
HAS SPECIMEN FAILED
YES
NO
CALCULATE ENERGY STORED IN SPECIMEN
IS MODULUS COMPUTATION COMPLETE
YES
NO
CALCULATE $d\sigma/d\epsilon$
CALCULATE FAILING STRESS, STRAIN AND ENERGY
PRINT CALCULATED VALUES
IS THERE ANOTHER TEST
YES
NO
CALCULATE STATISTICAL ANALYSIS OF BATCH RESULTS
PRINT STATISTICS RESULTS
END

FIG. 2

# ERDE

JOINT TROPICAL RESEARCH UNIT, INNISFAIL, QUEENSLAND, AUSTRALIA

This Unit, whose offices and hot-wet exposure sites are situated near the small town of Innisfail, in longitude 146°E and latitude 17°S (between the tropic of Capricorn and the Equator) is operated by the Australian Department of Supply and the UK Procurement Executive, Ministry of Defence. Technical oversight of the work of the unit is in the hands of the Defence Standards Laboratories, Melbourne, Victoria and ERDE: the scientific and technical staff are drawn from these establishments. The primary purpose of the unit is to study the effects of the tropical environment on materials which are, or are likely to be used, in Service equipment in the 2 countries. Additionally, work is undertaken on a limited scale on agreed terms for other Government departments and for non-government organisations including commercial concerns. The exposure sites in use are:

At Innisfail (with resident staff): secondary tropical rainforest (jungle) and cleared tropical rainforest.

At Cloncurry (unattended but visited every 3 months) in longitude 140°E and latitude 21°S, 960 km west of Innisfail in an arid zone.

At Clump Point, 80 km south of the Unit (unattended) in 146°E and 18°S where specimens can be immersed from rafts moored in water of world average salinity with mean temperature of 23°C.

The climatic conditions for the 3 landward sites are given for the last 8 years in the attached figures. Conditions at Innisfail 'cleared' site are characterised by sustained high mean temperatures and relative humidities, with strong solar radiation which is, however, subject to periodic obscuration by heavy cloud. Conditions in the 'jungle' site are similar in respect of temperature but relative humidity is consistently higher and solar radiation much reduced. While the 'cleared' site is subject to very heavy rainfall, this is manifest in the 'jungle' as rainsplash. At both sites there is intense microbiological growth on organic materials (including paint on metals) where no precautions have been taken to prevent it. Conditions at the desert site include intense solar radiation, low relative humidity, a wider range of temperature both in means and extremes and very low rainfall. Microbiological activity is insignificant but windblown abrasive sand represents a hazard absent from the other sites.

Much of the work of the Unit consists of the routine exposure and withdrawal of materials and systems submitted by sponsoring organisations. Simple non-destructive tests such as visual assessment on defined scales, weighing and measurement are also a matter of routine. Good photographic facilities exist for illustrating routine reports. The photographs illustrating this note were taken by the Unit staff. Mechanical testing is undertaken on unexposed and withdrawn specimens, particularly on those which have been exposed under mechanical stress and which would, if returned to sponsoring organisations, be subject to indeterminate stress relaxations in ill-defined temperature conditions of variable duration. A wide range of materials and systems is examined. These include metals (the Unit is involved in BISRA's world-wide corrosion-mapping activity involving the use of standard panels of pure zinc and of copper-bearing steel) and paints as protection for metals. Light alloys feature strongly because of Service interest. A subject of particular interest is the effect of weathering on mechanically-stressed joints made with epoxy and Redux type adhesives (as widely used in current aircraft construction). These joints are stressed in chains on simple static stress rigs illustrated on the general view of the laboratories looking SW. Non-metallic materials such as thermoplastics[2], fibre-reinforced thermosetting resins, rubbers[3] and textiles are also exposed, some under static load. Materials used underwater in marine craft are tested by immersion. An example of the intense underwater biological activity studied appears in one of the photographs.

In addition to running the routine exposure trials, the staff of the Unit undertake studies of, inter alia, the temperatures attained in materials exposed to solar radiation (eg black PVC which can reach temperatures in excess of 70°C), conditions prevailing in storage shelters in the tropics and the spectral distribution of energy in solar radiation and its variation with season, time of day etc. An interest which is being developed jointly with ERDE is the use of thin films of rubbers and plastics as a means of rapidly evaluating the stability of these materials in natural environments. This approach has the attraction of eliminating the correlation step implicit in the use of environmental chambers to simulate outdoor weathering. Effects are accelerated because most of the irreversible changes induced by weathering operate through the surface. Thin films, with their high surface/volume ratio, show rapid changes on exposure out-of-doors. Materials subject to photochemical changes display characteristic changes in their UV, visible and IR absorption spectra. These can be repeatedly monitored on the same area of film and films can also be subjected to destructive testing. The rapidity with which spectroscopic changes occur in thin films of some materials make them useful as monitors of UV radiation and of other environmental effects. A review of thin film techniques in this area will be given to the Symposium on Climatic Testing to be held by the Society of Environmental Engineers on 27 October 1971 at Imperial College.[4] Results of long-term exposure trails on plastics will be published shortly by HM Stationery Office under the aegis of the Ministry of Defence Advisory Council on Materials. Polymers to be dealt with in early publications are polycarbonate, chlorinated polyether and polyacetal resins as well as glass-fibre reinforced polyester and epoxide resins. Other materials will be reported as results become available.

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1 B L Hollingsworth, New Technology No 32 September 1969 pp 3, 6

2 P Dunn, D Oldfield, R H Stacewicz, J Appl Polym Sci 14 No 8 2107-16 (August 1970)

3 P Dunn, S J Hart, J Inst Rubb Ind 3 No 2 1-5 (April 1969)

4 A Davis, D Gordon, Characterisation of Environments etc. Paper to be read at SEE Symposium on Climatic Testing, Imperial College, 27 October 1971

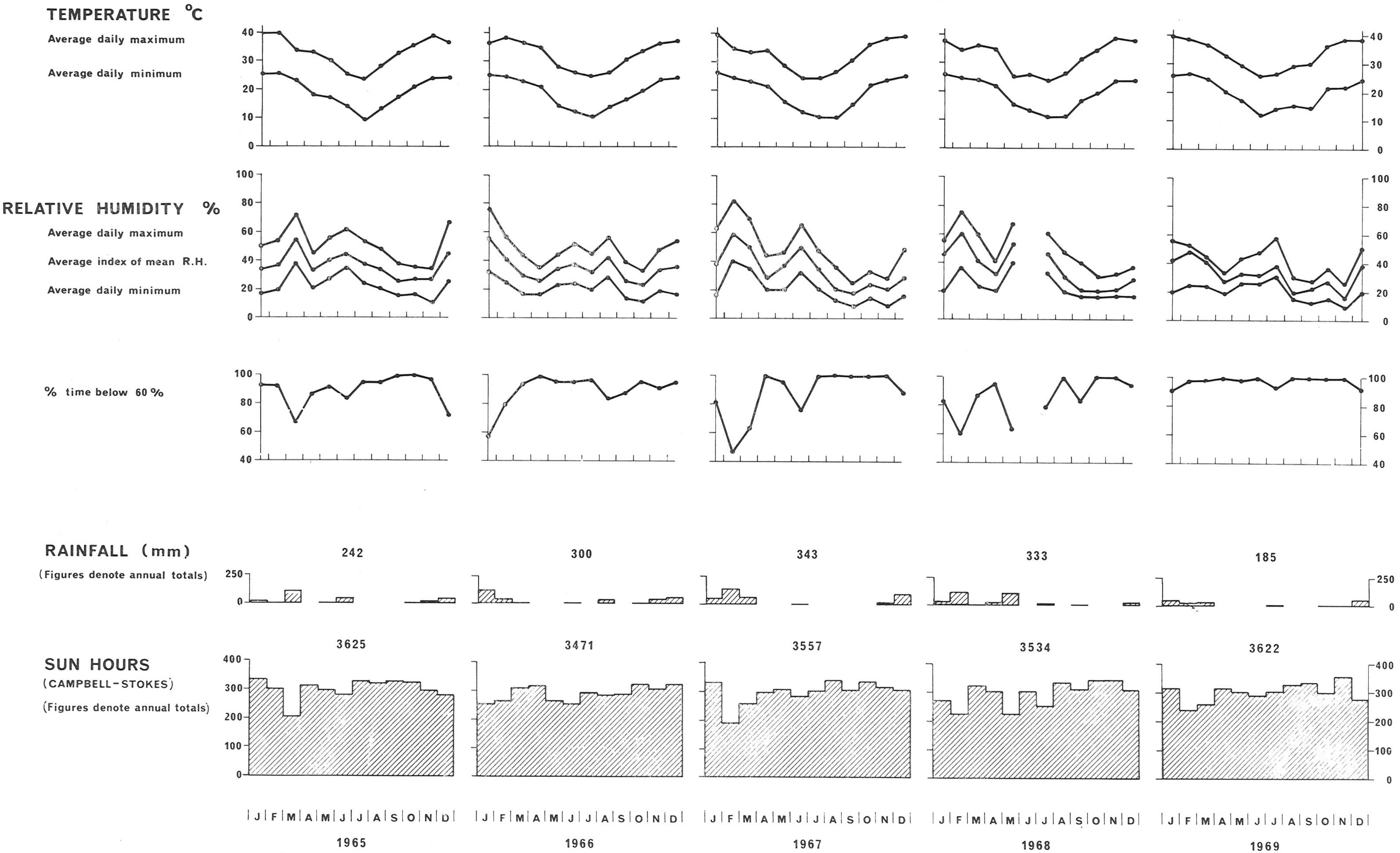


Enquiries should be addressed to:

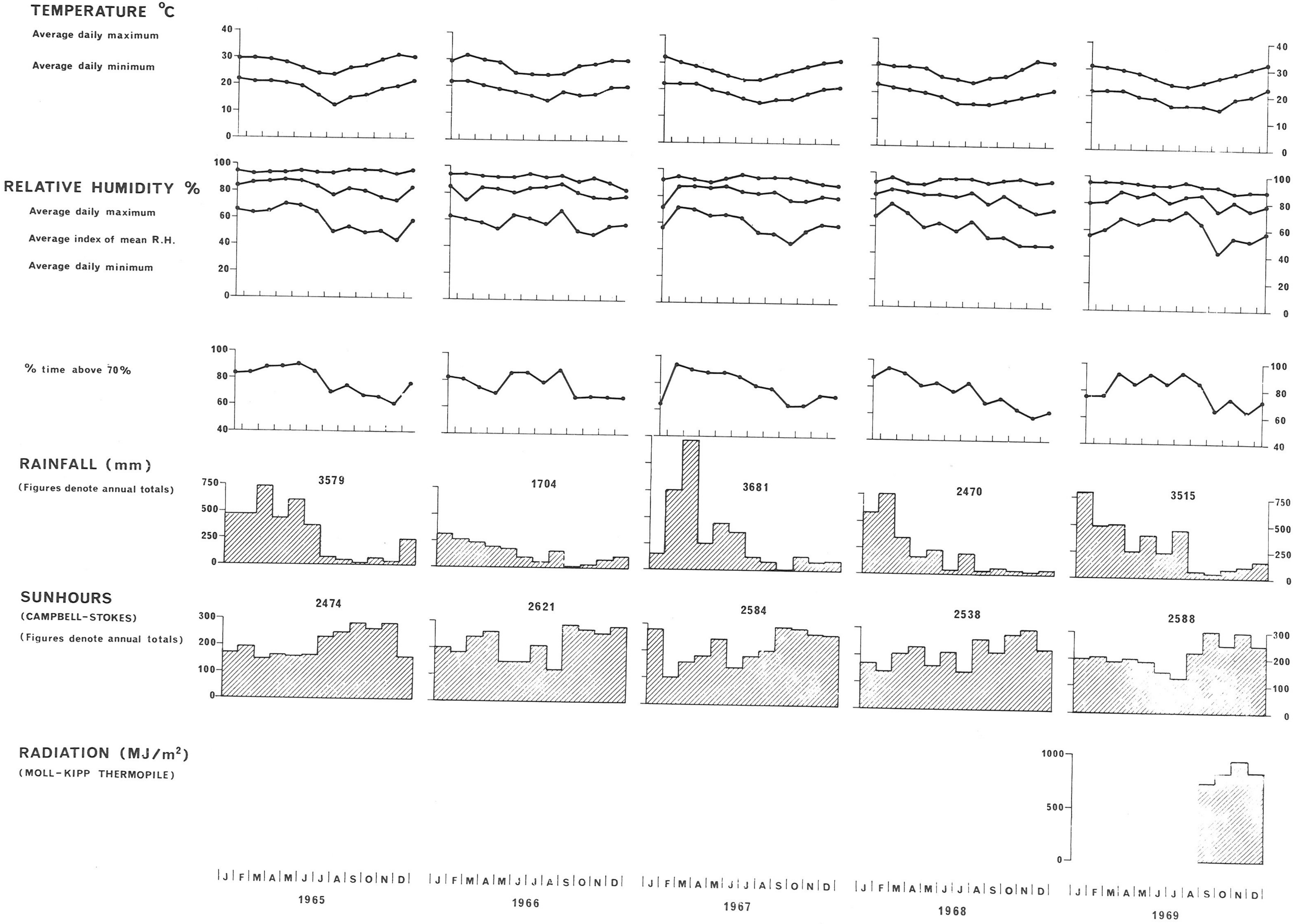
Non-metallic Materials Branch


NM/5

# CLONCURRY (HOT/DRY)



# ·NNISFAIL (HOT / WET CLEARED)



# NON-DESTRUCTIVE TESTING OF SOLID PROPELLANTS

## INTRODUCTION

Modern rockets are designed with lightweight cases for a high overall performance, and it is not feasible to allow factors of safety large enough to tolerate any badly flawed charges. Hence, it is essential to be able to detect such flaws [1]. Non-destructive testing is also required at ERDE to investigate the quality of charges made by experimental manufacturing techniques, and to study any defects that might develop in Service usage. The latter is generally simulated by standardized rough treatment, temperature cycling and hot storage.

Visual inspection is used wherever possible, but most modern propellants are opaque, and internal defects cannot be seen. The three methods mainly used at ERDE are radiography, charge deformation under vacuum (for air inclusions in plastic propellant), and ultrasonics. Application of the two latter techniques to propellants originated at ERDE.

The defects occurring in different solid propellants depend upon the various methods of manufacture and may include inhomogeneity, internal cavities, cracks, porosity, capillaries and lack of bonding. Acceptance standards are set up for each kind of flaw, depending on the nature of the rocket and its usage.

## RADIOGRAPHY

RARDE has used radiography (X-ray photography) for propellants since about 1930 and, from their technology, ERDE procedures have been developed since 1950. The ERDE equipment is limited to 400 kV, suitable for propellant charges up to about a foot in diameter.

In capable hands it is an excellent tool, especially useful when diagnosing the causes of charge failures. Its general limitations such as the need for protection from radiation hazards, cost, and delay time for processing - with hold-up in inspection runs, are well known. Propellant radiography requires high sensitivity techniques (e.g. small X-ray source, large film-focus distance, fine-grain film with thin lead screens), involving highly skilled viewers whose output is limited by fatigue.

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Assessments made at ERDE in recent years to improve or cheapen techniques include:

1 Use of cobalt 60 source - found too slow and/or to give poor resolution

2 Photographic paper in place of film - poor resolution

3 Electronic image intensifier - poor resolution and tube costly

4 Xero-radiography - good quality for smaller charges but no cheaper than conventional radiography. Special selenium-coated plates are costly, easily damaged and rather small.

The radiography section gives a comprehensive service to the whole Establishment, though most of the work is devoted to double-base propellants. An automatic Refrema film processing unit was introduced in 1966 and enables films to be viewed only ten or fifteen minutes after exposure, besides saving labour.

## ULTRASONIC INSPECTION

This method uses a beam of inaudible high-frequency sound waves which, owing to their short wave length, travel almost in straight lines, like light. They can be used to locate flaws, either by reflection of the radiation from the flaw, or by reduction of transmission through it. The transducers used for generating and detecting the radiation are discs composed of oriented piezo-electric crystals such as barium titanate or lead zirconate, with their flat faces plated with metal. An electric voltage between these faces is suddenly discharged, leading to a small change in dimensions of the disc, which vibrates at its natural frequency and emits a short damped train of ultrasonic waves. Pressure fluctuations generate a tiny oscillatory voltage between the metallized faces of the receiving transducers, which can be amplified and fed to a receiver. High frequencies ($2 - 5 \times 10^6$ cycles/second) give good resolution and are used in reflection sets to detect flaws in metal or absence of bonding, but they are rapidly attenuated in propellant and plastics. The ERDE-designed through-transmission set for plastics operates at a frequency of 370,000 cycles/second, with 60 pulses/second.

## ERDE ULTRASONIC FLAW DETECTOR

The first design was developed between 1952 and 1955(2), made commercially and adopted by the Inspectorates and Ordnance Factories for inspecting cordite rocket charges. It has also been exported to the Commonwealth and NATO countries.

The propellant charge is immersed on rollers in water, the acoustic coupling medium, and the transducers in watertight housings traversed on either side of it. The received signal viewed on an oscilloscope is distorted by reflections and 'hash' which, however, do not affect the first half-pulse travelling directly through the propellant. An electronic 'gate' is used to select this first signal, which is amplified and converted to a continuous DC current fed to a pen recorder. Any flaw deflects the pen, whose position on the chart can be correlated with the position in the charge that has caused it. The Mark III equipment is capable of inspecting propellant charges up to eighteen inches thick and is both rapid and cheap for production inspection. Inspection standards have been correlated with flaw sizes and the ultrasonic and ballistic effect of density variations of the order of one per cent have been investigated.

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1 Rocket Propulsion Technology, Vol 1, 269-279

2 British Plastics, July 1956, 262-264

ERDE

# THE COMBUSTION OF SOLID PROPELLANTS

The chemical energy stored in solid propellants is released by a process of combustion in which the initially solid reactants are converted, by a very complex sequence of chemical reactions proceeding at very high temperatures, to simple gaseous products. The development of sophisticated solid propulsion systems demands an understanding of the energy-releasing processes and research to this end is being actively pursued in all the leading countries.

Over the years ERDE has established recognized expertise in several areas: combustion and decomposition of systems containing nitrocellulose and liquid nitric esters; ballistic modification of these systems; the burning of ammonium perchlorate, a most important oxidiser in composite propellants; the combustion of aluminium in propellants and in the fundamental mechanisms involved in all these processes.

The rate of energy release depends on the surface area of propellant exposed and very high burning rates develop on ignition of dry guncotton or propellant 'crumble', compared with the relatively slow rates of properly-consolidated propellant. Very real hazards in the manufacture of solid propellants relate to the handling of finely divided energetic materials and propellant porosity or cracking presents dangers in usage. The phenomenon of the spread of flame over the propellant surface is not yet well understood.

Efficient usage of propellants requires that the energy-releasing reactions are complete before the products issue from the rocket nozzle. Two well-identified situations where this is not so are when compositions are used at low operating pressures and when aluminium powder is used as a high energy fuel without proper consideration of burning conditions. The inefficiency may be related to the fact that energy is released in several stages, and the later stages may not be complete inside the chamber. ERDE research into the control of ballistic behaviour of all types of solid propellant is internationally recognized.

The addition of aluminium powder and certain other light metals to propellants can theoretically give a very significant increase in specific impulse but many problems are associated with the efficient practical usage of such metals. Research at ERDE has shown that one of the most important phenomena in metal combustion is the natural growth of molten metal particles on the burning propellant surface. If conditions permit growth to large globules severe combustion inefficiency can arise.

In the combustion of real propellant systems all the individual features are related to each other and to their environment and continued research is necessary if a wider and more positive understanding and control of these complex systems is to be achieved.

P1/3

# ERDE

FACILITIES AVAILABLE AT BALLISTIC ASSESSMENT SECTION

## CALORIMETRY

Facilities are available for measuring heats of combustion of organic compounds containing any, or all of the following elements, carbon, hydrogen, nitrogen and oxygen, using an isothermal bomb calorimeter at 25°C. Temperature is measured by means of a platinum resistance thermometer in conjunction with a Smith's No. 3 Difference Bridge, to an ultimate accuracy of 0.0003°C in the working range of the apparatus. The precision apparatus, which is of a similar design to that used at the National Physical Laboratory, Teddington, is capable of giving results to an accuracy of the order of 0.01%.

Other equipment is available for the measurement of total heat evolution of pyrotechnic and propellant formulations.

## PRESSURE RECORDING AND MEASUREMENT

Two semi-portable, self-contained, analogue pressure recording systems are available for 'on-site' measurements.

### SYSTEM 1

| | |
|---|---|
| Maximum Range of Pressure: | 0-100 $MN/m^2$ (0-15,000 $lb/in^2$) |
| Transducer: | Quartz piezo gauge |
| Frequency Response: | Flat response within band 0-10 kHz |
| Recording Time Interval Range: | 0.005 s-1 s |
| Accuracy: | 2% |
| Record Dimensions: | 455 mm x 150 mm |

Facilities for synchronizing external events

### SYSTEM 2

| | |
|---|---|
| Maximum Range of Pressure: | 0-100 $MN/m^2$ (0-15,000 $lb/in^2$) |
| Transducer: | Strain gauged type |
| Frequency Response: | Flat response within band 0-100 Hz |
| Accuracy: | 2% |
| Record Dimensions: | Width 50 mm, length, as convenient |

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Permanent installations are also available for the recording of pressure and rate of change of pressure from explosions in closed systems at pressures up to 310 $MN/m^2$ ($4.5 \times 10^4$ $lb/in^2$) and rates of change of pressure up to $3.1 \times 10^5$ MN ($4.5 \times 10^7$ $lb/in^2$ s).

# ERDE

## COMPOSITE PROPELLANTS

A composite propellant is a mixture of chemicals containing both oxidizing and fuel components. Gunpowder is a well-known example although modern composite rocket propellants differ from gunpowder in that they are coherent rather than granular materials. These modern propellants contain an oxidizing agent such as ammonium perchlorate and a fuel which also acts as a binding agent for the solid ingredients. When the mixture is ignited in a rocket motor, high pressure gas is produced which escapes through a nozzle and thus impels the rocket forward by reaction.

Two principal types of composite rocket propellant are in use or under development in the UK. These are known as plastic propellant and rubbery propellant, respectively. Both are used in the form of a single block or charge which fits exactly the rocket motor case and is tightly bonded to it. Burning takes place either at the end or in a central hole. Both types of propellant have the merit that because of their plastic or rubbery nature they retain their bond to the casing in spite of differential thermal expansion. Bonding to the case is important because it protects the metal from the hot gases and enables a light-weight motor to be used thus increasing the overall performance.

## PLASTIC PROPELLANT

Plastic propellant is a British invention which has been developed at ERDE. It is a stiff putty-like material which uses polyisobutene as the binder and is easily made in a paste mixing machine. It is then deaerated and pressed into the rocket motor case by hydraulic pressure. It is cheap, safe to handle, chemically stable, and offers a wide range of burning rates.

A typical composition comprises ammonium perchlorate (77 wt per cent), aluminium powder (10 wt per cent), polyisobutene (11 wt per cent), wetting agents such as ethyl oleate, and a burning rate catalyst. A material of this composition has a specific impulse of about 245 seconds, a burning rate of 25. 0 mm per second, and is usable over a temperature range of about -20°C to + 50°C. By adjusting the components of the propellant, the burning rate may be varied within a range of about 2. 5 to 37. 5 mm per second.

Plastic propellant is not suitable for very large rocket motors exceeding about one metre in diameter or for applications where very low tempratures are encountered. Research is therefore being carried out also on the rubbery type of composite propellant.

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## RUBBERY PROPELLANTS

Rubbery propellants are formed by mixing a curable carboxyl-terminated polybutadiene (CTPB) binder with dried powdered oxidant at 60°C together with imine and epoxy curing agents. The slurry formed is filled into a motor casing and cured for several days at 60°C.

CTPB propellants originated some years ago in the United States. They have excellent rubbery properties which are retained at very low temperatures and since they have a high specific impulse they are suitable for high performance motors required to operate over a wide temperature range without a limitation on charge size.

A considerable variety of ballistic and physical properties can be obtained by changes to the composition of the propellant mixture. The range of burning rates so far obtained is less than with plastic propellant but future research is likely to produce considerable improvement.

## TESTING FACILITIES AT ERDE

Many requirements for the formulation and development of new composite rocket propellant compositions come to ERDE since this is the only United Kingdom Establishment where such work is carried out. Initial investigations are carried out on small scale equipment. Results are then applied to large-scale manufacture in order to establish reliable manufacture in order to establish reliable manufacturing procedures, and methods of quality control.

Enquiries should be addressed to:

Propellants 2 Branch

P2/2

# ERDE

## THE MANUFACTURE OF RUBBERY COMPOSITE PROPELLANTS

Rubbery propellants are made by mixing together various powdered solids and liquids. The result is a viscous slurry which can be readily cast into a rocket case, and then cured by heating for several days at about 60°C.

A typical composition contains ammonium perchlorate (oxidizer), aluminium (high energy fuel), a solid burning-rate modifier, a liquid polymer (e.g. carboxl-terminated polybutadiene, CTPB), a liquid curing agent, a cure catalyst and a plasticizer. The liquid polymer and the curing agent react to form the rubber matrix. The solids content is about 85 per cent by weight, of which the major portion is perchlorate.

The N550 mixing and filling plant, erected in 1962, was designed by ERDE. At present adapted for the filling of several small motors from a single mix, it could equally well be used for large motors requiring several mixes.

The central feature is a Baker Perkins stainless steel vertical mixer, capacity 300 kg, specially designed to handle viscous propellant slurries efficiently and safely. The two equal-speed, semi helical mixing blades rotate in fixed bearings. The water-jacketted vessel, which is of figure eight cross-section, is raised and lowered hydraulically. Blade-to-vessel clearance is 6 mm.

Mixing or discharging may be carried out in an inert atmosphere or under a vacuum of 5 mm of mercury. An interesting feature is the use of a labyrinth fluid seal to protect the blade shaft carbon seal from dust within the mixer, which could present a hazard. The fluid used is CTPB, and vacuum differential is catered for by suitable balance pipes.

Power drive is by a 25 hp electric motor through a Vulcan Sinclair fluid coupling and a Carter hydraulic infinitely variable speed gear, giving a blade rotation of 0 - 32 rev/minute. These are located in the adjacent bay.

All ingredients, except ammonium perchlorate, are normally added by hand loading, although facilities exist for remotely controlled additions of liquids and slurries. Ammonium perchlorate is added by remote control from a vibrated hopper, through a tubular vibro feeder and a vacuum-type butterfly valve in the lid of the mixer. The hopper is suspended from a tension load cell connected to an accurate weight indicator in the control room, which gives a check on rate and completeness of addition. ⇨

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The mix falls into an evacuated transfer vessel. The vessel is then lightly pressurized (up to 50 lb/sq in), causing the slurry to flow into the motor case which is fitted with a teflon-coated mandrel to give the required conduit shape. The motor assembly is then disconnected, inverted and some of the filling tools are removed. The propellant level is adjusted by hand, the motor is sealed and then transferred to the curing oven. Finally, the assembly is dismantled and the mandrel is removed.

All mixing and filling operations are remotely controlled from the reinforced concrete building to the south of the main entrance. Closed circuit television and an intercommunication system are installed. All electrical equipment within the processing bay is dust-tight, because of the use of fine ammonium perchlorate. Services are located outside the bay wherever possible.

The layout, all equipment and the methods of operation have been designed for maximum safety. Numerous interlocking devices and spring-loaded switches contribute to safety of operations. In the unlikely event of mechanical failure causing ignition at the mixing stage, an infra-red detector in the lid of the vessel operates a rocket-powered drencher containing 40 gallons of water. At the same time the mixer motor and vacuum circuits are interrupted. Should the system fail to operate automatically, it can be actuated manually at the control panel.

Enquiries should be addressed to:

Propellants 2 Branch

new technology

NUMBER 34 : NOVEMBER 1969

NEWS OF PRODUCTION, RESEARCH AND DEVELOPMENT FROM THE MINISTRY OF TECHNOLOGY



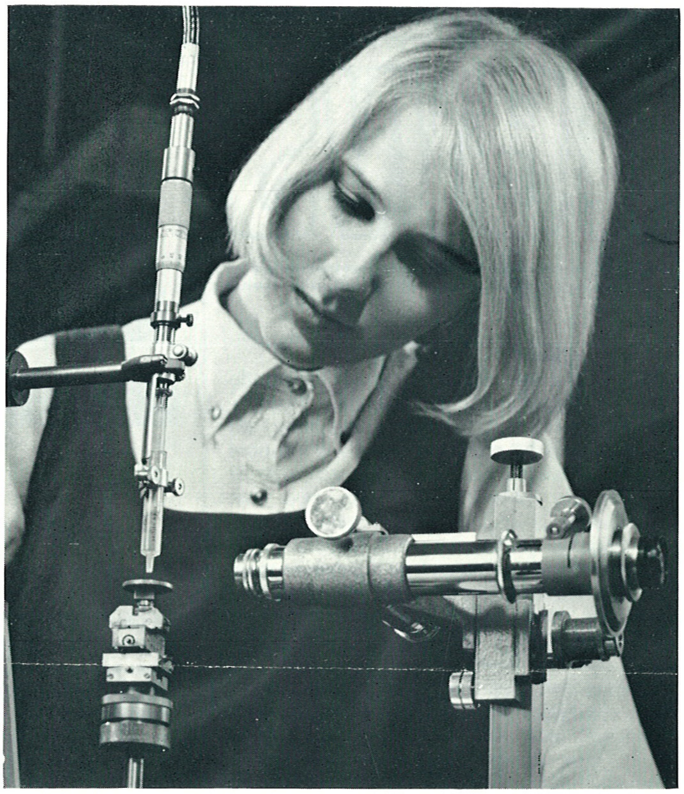
# ERDE advises on adhesives and sealants

In the Adhesion and Rheology Section of Mintech's Explosives Research and Development Establishment (ERDE), adhesives and sealants have been developed primarily for use during the assembly of equipment containing explosives or propellants, where it is vitally important to provide durable and reliable closures and joints between components fabricated in dissimilar materials. These adhesive and sealant compositions have received very wide use and for more than 30 years the Adhesion and Rheology Section has given advice on the choice and use of such materials to the designer of Service stores.

This service has now been extended to industry. Expert general advice can be given on the theory of bonding and sealing joints, and specialists are available to advise on such topics as the wetting of surfaces, rubber to fabric bonding, design of structural adhesive joints, methods of testing adhesives, the sealing of threaded joints and the rheology (deformation and flow behaviour) of adhesives and sealants.

Some problems demand experimental testing as well as advice, and this too can be provided to a limited extent. Equipment available includes an Instron universal testing machine with thermostatic control of the test specimen from minus 60 degrees C to plus 80 degrees C; an hydraulically operated high-speed test machine with high-speed Sanborn recorder and Tetronix oscilloscope; a variety of jigs for the preparation and testing of adhesive joints; apparatus for coating fabrics; and equipment for the measurement of surface tensions, contact angles, viscosities, plasticities, brittle points, glass transition temperatures, tensile and shear strengths, moduli and so forth. Facilities are available for the conditioning and storage of test specimens at controlled humidity and temperature.

Many enquiries concerning adhesives and sealants have already been received from industry, largely as the result of an effective demonstration of the work of the Adhesion and Rheology Section given during the ERDE Open Days in June 1968. Some of the problems dealt with include the use of adhesives in the production of electronic components and in clothing manufacture; replacement of brazing by adhesive bonding; preparation of laminates of paper, wood, plastics (including PVC) and metals; rubber to fabric bonding; high-speed adhesion in newsprint production; bonding of wax castings; development of permanently tacky labels easily removed by hot water; and sealing of joints in corrosive environments.



Measurement of contact angle at ERDE—the study of interfacial behaviour is an important aspect of adhesives development work.

A bibliography of published ERDE work on the bonding and sealing of joints is available.

**More information:** The Director, Explosives Research and Development Establishment, Waltham Abbey, Essex. Tel: Waltham Cross 23688.

information tool were completely traditional. No new questions were asked by management; no new answers were given.

This is not surprising. Few decision-makers know explicitly how they make decisions—the information they use, the manipulation they give it, the conceptual models they have of the decision-provoking situation, and the values they give to the variables affecting the decision. Having grown up with inadequate information sources and expensive, time-consuming information manipulative tools, many members of top and middle management in business firms don't believe in a world of adequate information quickly processed. 'Seat-of-the-pants' intuitive judgments are still frequent, and rare is the firm which spends enough effort to bring into its decision-making process all the pertinent outside information.

But increasingly the successful decision-makers in business believe in such a world. The hot item in business is 'management information systems', although there is no fixed definition of what a management information system is or does. Too often the management information system is restricted to computer manipulation and print-outs of financial, produc-

research, decision analysis, mathematical models of the corporate business, and risk and sensitivity analysis, are also helping decision-makers to make the best use of the vast amount of pertinent information.

The mere presence of sophisticated electronic and optical gear, and staffs capable of handling the new mathematical techniques, does not guarantee successful decision-making. Some managements have thought it would, only to find out after much time and cost that these new capabilities require a different style and method of management. The most successful exploiters of the new management capabilities have been those who match the flexibility of their management information system with an equally flexible organisational structure and set of activities. There have also been those who adapted their management practices to take full advantage of the potentialities of the system, rather than using the system as simply a new form of the traditional accounting system.

The cost of information and information systems to a firm is not insignificant. The information divisions mentioned earlier cost between two and ten per cent of total operating

# news

## 'The engineer is a woman'

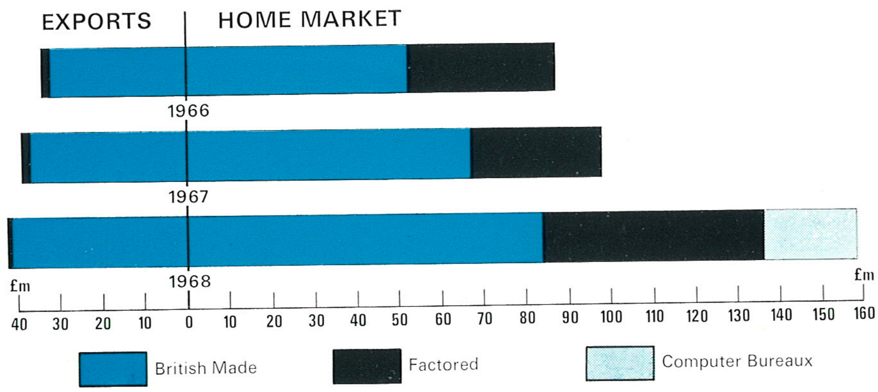
Five engineers 'star' in this 18-minute Mintech sponsored film. All are women—which means that in their fields they are outnumbered by men in the ratio of roughly 500:1. Nevertheless, the film sets out to explode the generally accepted view that engineering is an exclusively male domain. The country needs more professional engineers, and they can as well be women as men. To illustrate this, the five women engineers in the film, who range from a teen-age student apprentice to an R & D project manager and who work in mechanical, civil, electrical and electronic engineering, talk about education, training, working relationships, their individual jobs and their future prospects. In 16 mm, sound and colour, the film is available on free loan for non-theatrical use from the Central Film Library and its associated libraries in Scotland and Wales. Other enquiries to: J. Murray, Ministry of Technology, 42 Parliament Street, London, SW1. Tel: 01-930 4300, ext 567.

## Neutron radiography for NDT

A neutron radiography service is now being offered to industry by AERE's Harwell Nondestructive Testing Centre. The technique can be looked on as a complementary quality control technique to X-radiography and is similar in many ways. A beam of neutrons is used to produce an image of the internal details of an object and the final picture is obtained on photographic film. Hydrogen and hydrogenous materials can be readily radiographed in this way, where X-

## THE UK COMPUTER INDUSTRY

**Output of home industry, and factored hardware at current prices**




The total output of the computer industry of the United Kingdom is the sum of the value of hardware delivered together with the cost of installing and maintaining it, and any research and development on behalf of customers. In practice, the installation and maintenance separately identified by Mintech is only a fraction of the total effort, the remainder being merged in rental payments for the machines. Similarly, R & D performed on customer account is a very small proportion of the total 'in-house' R & D which the manufacturers must undertake all the time. To the output of the computer industry must be added the amount of hardware which is factored. This term is almost synonymous with 'imported'; the proportion of factored to British-made equipment is of considerable interest.

The UK market increased from £87 million in 1966 to £136 million in 1968, while exports increased over the same period from £34 million to £42 million. At the same time, the proportion of the home market met by factored equipment was 41 per cent in 1966, 31 per cent in 1967 and 38 per cent in 1968. Indications are that the 1969 figure will be higher.

An important feature of the computer market is the provision of software and other services. This covers a wide range of activities from writing programs to providing remote access to computers, and the service is provided by a variety of organisations. These include computer manufacturers themselves, specialist houses and service bureaux. It is a rapidly growing field, and Mintech figures per bureaux, compiled in 1968 for the first time, show that turnover in that year was over £22m.

Mechanical Engineering Group, National Research Development Corporation, PO Box 236, Kingsgate House, 66-74 Victoria Street, London SW1. Tel: 01-828 3400.

to much larger collections which use computers for storage, retrieval and updating. Several OSTI-supported projects in experimental handling of data are described. The publication is in effect the third edition of 'Critical Data in Britain', last issued May 1967.

# ERDE

ADHESION AND RHEOLOGY

ADVISORY CENTRE ON THE USE OF SEALANTS AND ADHESIVES

In this section, sealants and adhesives have been developed, primarily for use during the assembly of equipment containing explosives or propellants, where it is vitally important to provide durable and reliable closures and joints between components fabricated in many different materials. Our compositions are used very widely, and an advisory service is maintained to assist the designers for the three Services in their choice of suitable sealants and adhesives.

This service has been extended, and made available to industry through the Industrial Liaison Officers. Enquiries and requests for advice or assistance are welcome.

ADHESIVE JOINT STRENGTH

In recent years adhesives have been used increasingly as structural materials, and thus the strength of adhesive joints in structures is of growing engineering importance. It is usual to make joints in the Laboratory to simulate the structural joint, using adherends of the same materials with the same adhesive, cure cycle etc., and then to measure the strength and other mechanical properties of these joints.

In this Laboratory we are comparing various joint designs, with special emphasis on those which we have found relatively satisfactory or convenient; and we consider the difficulties encountered in relating the measurements made with them to both the strength of the structure, and to the fundamental adhesive strength, which should be independent of the method used in measuring it and a function only of the basic physical and chemical properties of the adherend surface and of the adhesive. With this knowledge, it should be possible to choose or design adhesives suitable for the stated end-use on any particular adherend under any particular stress conditions.

Tests developed here are preferable to the common overlap shear test, in that the adherend dimensions do not affect the measured strength. Thus the strength of a structure can be better assessed.

The reasons why thin joints are stronger than thick ones are also being examined. Reprints of relevant publications are available.

Enquiries should be addressed to:

Propellants 2 Branch

P2/4

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## ADHESION AND RHEOLOGY

### WETTING BY PROPELLANT INGREDIENTS

A prerequisite for good adhesion is that the adhesive must effectively wet the adherend. This is true not only in the case of structural adhesive joints, but also between the ingredients of the composite rocket propellants in which we are particularly interested. For example, plastic propellant is a dispersion of about 89% (by weight) of solids such as fine ammonium perchlorate crystals in a very viscous medium (about one hundred million times more viscous than water). The area of interface is about 600 sq ft per lb. So in the process of manufacture, $1\frac{1}{2}$ oz of viscous medium has to be spread over an area the size of this laboratory.

A knowledge of the interfacial properties is clearly essential to the understanding of the behaviour of these composites. To this end, we grow large crystals of ammonium perchlorate and measure the contact angles of the liquid ingredients on freshly cleaved surfaces, and we measure the surface tensions of the liquids. These measurements provide a fundamental thermodynamic basis for an understanding of the stability and rheological properties of the propellant.

### RHEOLOGY OF ROCKET PROPELLANTS

Rheology is the study of deformation and flow of materials, particularly non-ideal ones. A few examples, perhaps surprising, of rheological phenomena are on show in the Laboratory.

Rocket propellants must maintain adequate mechanical properties over a wide temperature range, both over long periods of storage and during the short times when they are actually functioning, under extreme stresses. For example, the plastic propellant described above must not slump nor change shape under its own weight during storage, and it must not be excessively deformed by the inertial forces acting on it during acceleration. Similarly, rubbery propellants must remain adequately extensible to resist without fracture the very rapid straining during ignition of the rocket, even at low temperatures.

In order to satisfy ourselves of the adequacy of rocket propellants, we measure such rheological properties as the yield stress, rigidity, extensibility, and uni- and multi-axial tensile properties as a function of temperature, using equipment such as the Instron testing machine. We also simulate inertial forces in the rocket motor by spinning model motors in a centrifuge.

Enquiries should be addressed to:

Propellants 2 Branch

P2/5

# ERDE

## REMOTE CONTROLLED PROCESSING OF HAZARDOUS CHEMICALS

Research and development work in ERDE frequently requires kilogramme quantities of chemicals which are not available from the trade and may never have been made in more than gramme quantities in the laboratory. Often the chemicals or intermediates in their preparation pose a formidable explosion, fire or toxicity hazard. In addition the reagents involved may be very powerful acids, alkalies, oxidizing or reducing agents of a highly corrosive nature and capable of producing violent reactions. Attempts to make kilogramme quantities of such materials by manhandling could obviously be very hazardous. Remote control is therefore advisable and enables these preparations to be carried out without any danger to the operating personnel.

The laboratory preparative method has to be critically examined and generally modified to make it suitable for remote controlled processing and/or remote control techniques developed to suit the process, keeping intrinsic safety in mind. Scale-up data has to be determined to enable heat and mass transfer, gas evolution and reaction control calculations to be made. Hazard appraisal corrosion, compatibility, desensitization, storage and waste disposal studies are also necessary. With this information the kilogramme scale plant is designed for remote control and operational procedures drawn up to ensure smooth and trouble free production.

There are two facilities in the Process Research Branch both being within mounds and with remote control operation. The essential difference between the two is that the building for this facility is designed to withstand an explosion equivalent to 2 kg of TNT without structural damage. The other facility has exceptionally good fume extraction and is now used principally where the major risk is toxic.

This facility, built of reinforced concrete, consists of two cells separated by a services room. The control room is outside the earth mound and vision of the plant is possible by binoculars through mirrors or by closed circuit television. A video-recorder is being developed by our Electronics Section working on a closed loop so that the last two minutes of operation is always on record. Thus it will be possible to analyse the sequence of events leading to an incident. When the facility is completed it will be possible to transfer material from the process cells to magazines or to the adjacent high energy explosive processing unit by small gauge railway, also remotely controlled. A transport system has been designed to convey samples from the cells to an explosion cupboard next to the control room. Analyses on these samples will be carried out from the control room by direct vision through a laminated safety glass window.

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT
WALTHAM ABBEY, ESSEX Waltham Cross 23688

Each process cell is fitted with an extraction fan and all electrical fittings are flameproof. Equipments for the remotely operated plants have been built almost entirely from glass, stainless steel, polythene and PTFE to overcome corrosion difficulties. Glass vessels also have the advantage that liquid levels, frothing, etc. can be easily observed thus reducing the instrumentation required. The general principle adopted is that the plant shall be static and the chemicals transferred from stage to stage. This avoids the need for bearings with the possibility of accidents due to friction. Reagents are kept below reactor level to avoid accidental spillage to the reactor and whenever possible chemicals are transferred by air blowing or suction. The use of valves in process streams is not desirable and is kept to a minimum. For safety reasons, all controls such as valves in service lines are operated by compressed air. The use of compressed air from the same main supply has the advantage that all systems can be arranged to give failure to safety. Operations are cut down to a minimum and the simplicity thus achieved is itself conducive to safety.

One of the difficulties for this scale of operation in that commercial equipment for remote control is generally too big and is not made to deal with the wide range of corrosive and explosive hazards that we encounter. It has been necessary to design small pneumatically controlled valves of glass and PTFE, special glass and polythene covered metal stirrers, glass air eductors, etc. Equipment is being built in units which can be easily joined together to enable chemicals using various unit processes to be produced with minimum delay. To date such operations as reactions with controlled feeds and temperatures, washing, filtration, liquid/liquid separation, distillation and drying have been carried out remotely. It is interesting to note that once the facility has been perfected it provides the easiest way of making chemicals quickly yet with maximum safety whether there is any obvious danger or not. This provides extra security against the unforeseen hazard.

Enquiries should be addressed to:

Process Research Branch

PR/1

# ERDE

## LIQUID EXPLOSIVES

Nitric esters are liquid explosives and as such have a range of uses in ERDE. The Process Research Branch has a facility for making batches of up to 60 kg of such nitric esters in a safe manner by remote control. The process consists of a number of stages.

### 1 PURIFICATION OF THE MATERIAL TO BE NITRATED

This is done in a high-vacuum, packed-column distillation plant in glass. There are two units with an output of about 15 kg/day each. They operate batch wise with automatic reflux division. Heat is supplied by Isopad jackets to a boiler. The column is 76 mm internal diameter packed with stainless steel Dixon gauze rings and is externally lagged and heated.

### 2 ACID FACILITY

This consists of an acid mixing plant capable of mixing oleum, sulphuric acid and nitric acid in any proportion. A typical analysis of a mixed acid for nitration is 55% $HNO_3$· 40% $H_2SO_4$, 5% $H_2O$ and less than 0.01% $HNO_2$. 700 kg are made in one batch. To meet the low nitrous acid content of the mixed acid, concentrated nitric acid from store has to be treated to remove nitrous acid. This is now done by countercurrent air-blowing in a packed column.

### 3 NITRATION

This is a batch plant with an output of about 60 kg of nitric ester per batch. For safety reasons the nitration building is mounded. The nitration is done by slowly adding the alcohol to the mixed acid with brine cooling to maintain about $10^{\circ}C$. The acid is separated off and denitrated in a denitration column. The whole operation is done by remote control, with closed circuit television, from a control room some 50 metres beyond the mound.

### 4 WASHING

The nitric ester after nitration is contaminated with acid and is unstable. Before use it is washed with water and sodium carbonate solution and possibly sodium sulphite solution till it passes the Abel heat test (10 min minimum at $180^{\circ}F$).

## 5 DESENSITIZING

Before use it is often necessary to desensitize the nitric ester (particularly nitroglycerine) by diluting it with a non-explosive constituent which is conveniently an ingredient of the final explosive or propellant. This is done in a mounded building in a separate small plant with accurate metering which is remotely controlled and uses fluid logic techniques.

## 6 DRYING

It is frequently necessary to dry the nitric esters intensively before use. A water content as low as 0.005% may be specified. This requires a vacuum of the order of 5 mmHg. In order to achieve the low moisture content in a short time a large surface area with low pressure drop is necessary. These conditions are met by running the nitric ester down 35 13 mm diameter wire spirals each two metres long. This system has the advantage of minimum hold-up and free drainage after use. The output is about 15 kg/hour of nitric ester.

Enquiries should be addressed to:

Process Research Branch

PR/2

# ERDE

PROCESS STUDIES

## CONSTANT RATE EVAPORATIVE FEEDER

This has been developed in ERDE to enable the decomposition of methyl nitrite (b.pt. -12°C) to be studied but it has general application as a constant rate feeder. A constant evaporation rate without a large volume of liquid requires a large heating surface area to volume ratio, good heat transfer, stable boiling conditions and a constant temperature heat source. These conditions are met by use of a narrow bore coil which is one leg of a circulatory system. By inducing boiling at the base of the coil the mean fluid density is reduced and the vapour-liquid mixture rises to be replaced by liquid from the reservoir. As the fluid rises up the coil the gas and liquid reach equilibrium and the pressure is virtually independent of the liquid level in the reservoir. A constant rate feed can therefore be taken off through a suitable orifice. To enable the system to function smoothly it is essential to initiate boiling at the right point. This is achieved by the use of a small reed vibrator in the liquid at the bottom of the coil.

## HARTRIDGE-ROUGHTON MIXER

Certain propellant ingredients are prepared by precipitation from solutions of electrolytes. Their ballistic properties are greatly influenced by the mixing conditions. The preparation of these materials can be studied in a rapid mixer of the Hartridge-Roughton design. The two streams to be mixed flow continuously and at high velocity through tangential jets into a tiny mixing chamber. The combined stream, partially mixed, leaves the mixing chamber through a tube in which mixing is quickly completed by the turbulent swirling motion. Nearly complete mixing can be achieved in one millisecond. This approaches the ideal precipitator in which the rate of mixing is rapid compared with the rate of nucleation. Thus the effect of reaction parameters such as concentration, pH and recycle can be studied without the complicating mixing factor. Also, the rate of precipitation can be measured by using light sensitive probes at intervals along the observation tube. This technique can be used to study other aspects of precipitate quality. Results can be applied to plant scale production.

## MOLECULAR STILL

In the preparation of chemicals for propellants and general research, high purity is often required. As the chemicals are frequently heat sensitive, low temperature distillation with low residence time is essential. The 'molecular' still is really a short path, high vacuum distillation unit with a wiped wall, ⇨

thin film heating section and is produced by Edwards High Vacuum Ltd. The material being heated at any one time is about 5 ml and the duration of heating about 20 seconds, the distillation rate varying from 200 to 800 ml/hr depending on the conditions. Operating pressures down to $10^{-3}$ mmHg can be achieved. We have modified the unit by making the heating section of electrical conducting glass which has the twin advantages of low heat capacity and full vision. The consequent rapid response facilitates control and makes it easy to avoid overheating. Anything abnormal such as gum or residue formation can be observed and given attention at an early stage.

Enquiries should be addressed to:

Process Research Branch

PR/3

# ERDE

CRYSTALLIZATION STUDIES

Crystal size and shape play an important part in the manufacture and behaviour of propellants and explosives. Materials such as ammonium perchlorate and RDX are required in sizes ranging from 5 to 500 micrometers, and in a variety of size distributions and shapes, according to the particular application.

Most of the materials used are crystallized from solution and it has been necessary to study the mechanisms involved in this process in order to establish a more precise control over the operation.

Much research on crystallization in the past, because of the complexity, has been carried out on the laboratory scale except in isolated circumstances. This forms an excellent background but does not contribute much knowledge on the variables encountered in larger crystallizing plant, and consequently the approach to crystallization in industry, by necessity, has been largely empirical.

Studies in the Process Research Branch have been directed towards bridging the gap between the academic and the practical approaches, by investigating the significance of the variables encountered in a variety of crystallizing equipment. The majority of the work has used ammonium perchlorate because this is readily available in a pure state and in large quantities, and forms no hydrated solid phases.

A study has been made of an Oslo type classifying crystallizer which has provided useful information. More recently a programme has been undertaken using a computer to solve a set of complex implicit equations which has been produced to predict the product size distribution from a Kestner recirculating evaporative crystallizer.

These showed that nucleation in solution is a difficult problem and a study of this process has been done in two crystallizing systems. In one, a cooled agitated batch vessel, the closest possible control over such variables as the rate of cooling has been achieved in order to obtain reproducible results. The equipment is able to give repeatable crystallizations and it is possible to follow supersaturation changes and crystal size variations at the same time, the latter by a photo-extinction method.

The second is a cooled agitated continuous feed vessel, in which the agitation conditions can be accurately measured, together with close temperature control. By combining the information from these experiments with other information on the rate of crystal growth obtained in the laboratory, it is possible to build up a clear picture of the mechanisms of crystallization in a large crystallizer. Similar considerations often apply wherever crystalline materials are produced, and the subject is of importance in industry generally.

Enquiries should be addressed to:

Process Research Branch

PR/4

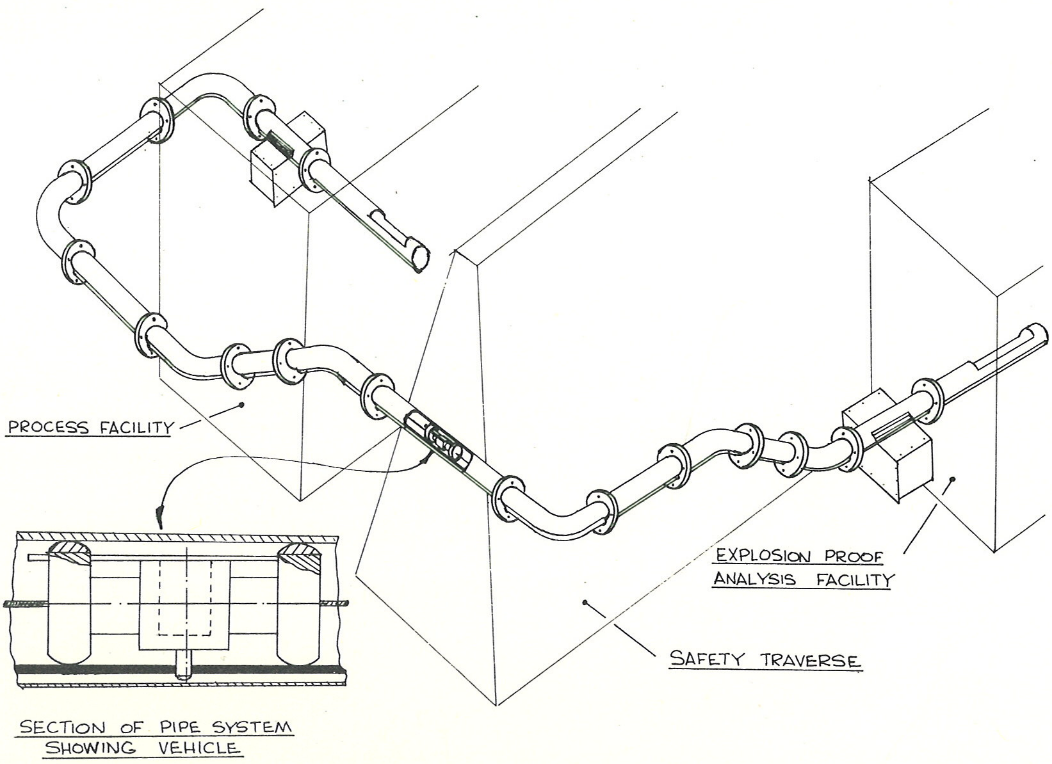
# ERDE

REMOTELY CONTROLLED SAMPLING TRANSPORT SYSTEM

A prototype transport system has been devised to carry liquid or solid explosive samples by remote control from the process building to an explosion proof analysis facility.

The design uses a modified 76 mm internal diameter UPVC pipe through which passes a transport vehicle (mole) with a volume capacity of 5 $cm^3$. The mole is an articulated design to enable it to negotiate bends and irregularities in the pipe. The sketch below shows the basic design features. The centre section of the wall can be fitted with a compatible plastic liner and the end sections are rimmed with PTFE to reduce friction. A special leaf-proof valve is fitted to the centre section which is alternately opened and closed at the limits of travel of the mole, at the process bay and analysis facility. The mole is pulled in either direction by wire winding on to drums and its position is controlled automatically from the control room.

The system has been designed with a number of standard, easily assembled units, so that the system can be dismantled and reassembled and/or modified with the minimum of effort.

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Enquiries should be addressed to:

Process Research Branch

PR/5

PROCESS RESEARCH SECTION

## Aligned Short Fibres in Reinforced Plastics

Continuous fibres have received much attention in the past as they lend themselves to manual processing techniques suitable for laboratory investigation. The standard metre length in which carbon fibre was first produced was ideal for this purpose. However, widespread engineering use calls for mechanised, fast and relatively inexpensive fabrication processes. This requires either continuous filament or short fibres. Work at ERDE has concentrated on the development of processes to handle short fibres.

### The Case for Short Fibres

a Short fibres are cheaper than continuous ones. Asbestos is cheaper than glass fibre. At present continuous carbon fibre is 2 to 3 times more expensive than chopped fibre and a difference in price is likely to continue as it is cheaper to make material for use as short fibre, e.g. made as heavier tows (1) or from staple precursors.

b Short fibre aligned prepreg possesses excellent handling properties (due to cross-plying by a few misaligned fibres). This, combined with slippage of the short fibres, enables the prepreg to be draped and moulded to complicated three dimensional shapes and double curvatures.

c Short fibre alignment offers much greater flexibility when complex orientation patterns are contemplated. Individual ply layers can be as thin as a fibre diameter so that pseudo-random or cross-plied prepreg can be built up in a 'balanced' condition. This greatly eases the subsequent processing to produce cross-plied composites of the required thickness with no tendency to warp on moulding. More complex alignment patterns, e.g. circumferential in a plane or conical pose difficulties with continuous fibres (2,3) but can readily be formed using short fibres. Programmed alignment to match predicted stress distributions such as those encountered in rotating discs opens a wide field of application. Techniques are being developed at ERDE to produce these types of prepreg and preform.

d Short fibres lend themselves to mechanical processing and composite felts of mixed fibres can be made easily. If required the mixture proportions can be varied over the preform or high performance expensive fibres can be used only in selected areas to give local reinforcement or as a skin for sandwich type construction.



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e The strongest fibres so far known, whiskers, are generally available only as short fibres. These can only be handled by short fibre processes.

f As expensive, high performance fibres come into use, it is logical to recover them from prepreg off-cuts and possibly even from disused hardware. Such recovered material, even if originally made from continuous fibre, can only be effectively re-used as short fibres.

g The preceding arguments are only persuasive if it can be shown that short fibres do reinforce effectively. Model studies (4) show that absolute length is unimportant and fibres need only have an adequate ratio of length to diameter in order to carry high stresses as a reinforcement. In practice, using ERDE processes, it has been possible to consistently utilise approximately 70 per cent of the fibre strength and 90 per cent of the fibre modulus in the alignment direction for most short fibre thermoset systems.

## ERDE Processes for Short Fibres

For optimum, reproducible properties in composites control of length and diameter of short fibres is necessary and fast, economic, wet processes have been developed for cleaning and grading short fibres and whiskers. Sorting for diameter is achieved by conventional hydrocyclone techniques and for length by a new centrifugal screening process (5, 7). To obtain optimum reinforcement it is necessary to pack the maximum amount of fibre or whisker into a composite without significantly damaging the fibres in doing so. This is best achieved by alignment and again novel wet processes have been developed for this purpose (6, 7).

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4 A Kelly "Strong Solids" Clarendon Press, Oxford, 1966, pp 143-147.

5 Composites, 1, 114-117 (1969)

6 H Ziebland et al. Seventh International Reinforced Plastics Conference. The British Plastics Federation, Brighton 1970, 6/1-6.

7 H Ziebland et al. New Technology, 1971, 46, 4-8.



Enquiries to:

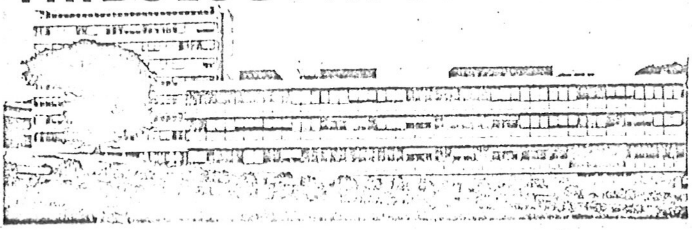
Mr H Ziebland
Process Research Branch

# new technology

NUMBER 32 : SEPTEMBER 1969

NEWS OF PRODUCTION, RESEARCH AND DEVELOPMENT FROM THE MINISTRY OF TECHNOLOGY


## TRIBOLOGY AT SWANSEA




**by A. R. Lansdown**

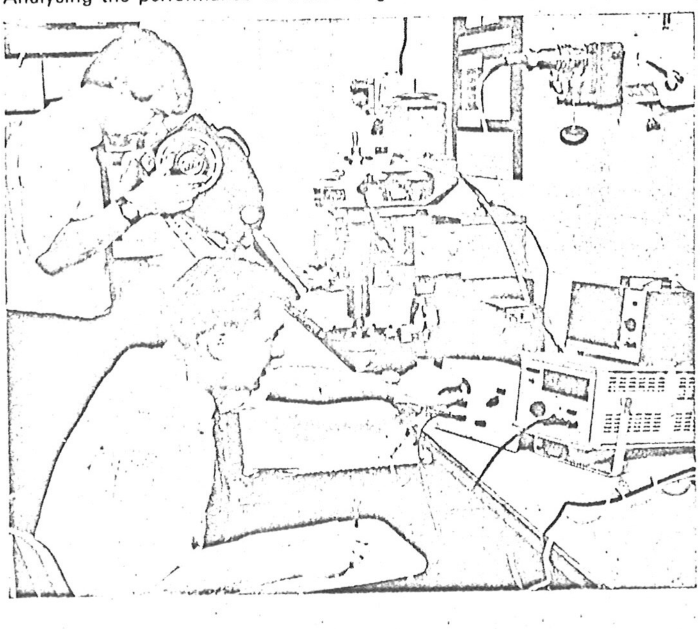
*Manager, Industrial Unit for Tribology, University College of Swansea*


The Industrial Unit for Tribology was established at the University College of Swansea late in 1968. Its purpose is to assist industry by investigations into all types of problem involving moving surfaces – wear, friction, bearing failures, lubrication, metalworking, and so on. Because of its geographical position it has a special interest in the problems of the process industries – metallurgical, chemical, mining, and refining – but it has also worked on vehicles, engines and machine tools.

Set up with the support of the Ministry of Technology, which advanced £40,000 to assist during its first two years, the Centre must make itself self-supporting within the first four years of its life.

The work of the Centre tends to fall into four main categories: plant design and maintenance, specific metalworking and processing problems, testing and evaluation of materials and lubricants, and the behaviour of rolling bearings and gears.

Analysing the performance of a new angular contact ball bearing design



A vital trend in modern manufacturing is to increased utilisation of plant. In the chemical industry, for example, plant is expected to operate continuously, with no standby components and only annual shutdowns for maintenance.

In these circumstances the cost of unscheduled maintenance and breakdowns becomes enormous. In the 1966 DES report '*Lubrication (Tribology)*' more than half of the potential annual savings were related to maintenance and breakdown. The figures were £230 million per annum for maintenance and replacement, and £115 million for consequential losses. Relate this £345 million per annum to the size of your company and the result may shock you.

> Industry is becoming increasingly aware of the savings which can be achieved through a combined approach to the problems of wear, friction and lubrication and their relations to design—savings which have been put as high as £500 million a year.
>
> The Ministry of Technology is producing an exhibition of tribological examples showing their practical advantages in commercial and engineering terms, together with a film dealing with tribology problems in an industrial setting.
>
> Both will receive their first showing at an industrial event to be held in the Angel Hotel, Cardiff, on October 28. This is being arranged by the Ministry in conjunction with the South Wales branch of the Institution of Mechanical Engineers, the CBI (Wales) and the Industrial Unit for Tribology at the University College of Swansea.
>
> To accompany the display and film, a one-day conference on various aspects of tribology in industry has been organised by the Institution of Mechanical Engineers.
>
> Admission to the display and conference is free. Further details and invitations are available from: Ministry of Technology, Office for Wales, 69 Park Place, Cardiff. Tel: Cardiff 37671.

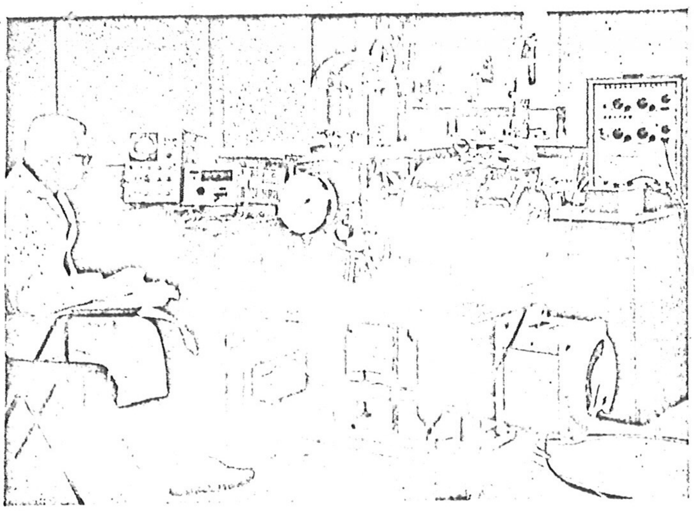
Of course it isn't that simple, but a representative of one large chemical company has stated that the cost of tribological problems to his company is enough to make the difference between net operating profit and operating loss.

The Centre offers a consultancy service on the lines of management consultancy, in which Centre staff will survey a plant and indicate to management the areas in which improvements can be made. Such surveys can be even more useful when carried out at the design stage.

Any sort of tribological problem can occur in the process industries, but some factors are particularly important. They include integrated plant and high throughput (the need for reliability), difficult environments (abrasion and corrosive wear), contamination (filters and seals) and on-line quality control (measurement techniques).

Rigs are available in the Centre for testing filters and seals. In quality control one achievement by the unit has been the invention of a device which measures directly the 'shape' of metal strip as it leaves a rolling mill.

The Centre has a number of test rigs for measuring the friction and wear properties of materials under different loads,



A test in progress on the Centre's controlled environment wear rig

speeds and environments. This work can be done for the manufacturer of materials, lubricants or additives. For example, one such manufacturer commissioned the Centre to study the wear behaviour of a new reinforced resin intended for high-temperature bearing use. But such tests can be of even more value to the user, and the Centre can take a material intended for use in a plant and test it under fully representative conditions for a fraction of the cost of a full-scale plant test and without any of the associated risks. There is also a well-equipped metrology laboratory; and one technique of special interest is computerised 'micro-cartography' of surfaces, soon to be supplemented by a scanning electron microscope.

The Centre is unusually well equipped to study the behaviour of rolling contacts. One major current contract is for a study of the performance of ball bearings used in jet engines, and this work has already led to improved understanding of factors causing premature failure of such bearings. Similar principles are applied in a study of the problems in metal rolling being made for the steel industry. Other techniques are used to measure the stresses in bearings, gears, slides or any other type of moving contact.

The above areas are those in which the Centre has special interest, expertise or facilities, but work is carried out on all sorts of other problems, such as theoretical study of a new seal design, a check on the performance of a new oil filter in service, an analysis of used lubricants, application of dry lubricants to machine tools, and reduction of contamination in lubricant systems. Advice can often be given on such problems immediately in answer to a telephone call, and in such cases the Centre makes no charge for its help.

The senior staff of the Centre consists of the manager and two senior engineers, who cover between them the fields of lubricants and materials, rolling contacts, and sliding contacts. They are supported by junior engineers, technicians and secretaries, and other facilities such as workshop, draughting, library and reproduction are provided by the College.

### Bridging operation

Apart from its own internal activities, the Centre can act as a bridge between industry and other College departments, or can obtain the help of these departments in attacking industrial problems. Of particular interest for the Centre's activities are the metallographic and fabrication facilities in metallurgy, control systems work in electrical engineering, heat transfer in mechanical engineering, finite element techniques in civil engineering, extrusion in chemical engineering, surface and discharge phenomena in physics, and spectroscopy in chemistry. Contact between the Centre and College departments is facilitated by the fact that Professor F. T. Barwell combines the appointments of Director of the Centre and Chairman of the School of Engineering.

The Centre also has informal agreements whereby the more specialised expertise of University College, Cardiff, on the microbiology of lubricants, and of Southampton University on gas bearings can be applied to problems where necessary.

Charges for the Centre's work are agreed with the client in advance, and no charges are made without prior agreement. If you have a problem with which the Centre may be able to help, or even if you don't think you have any problems but would like a second opinion, you are cordially invited to contact: The Manager, Industrial Unit for Tribology, University College of Swansea, Singleton Park, Swansea SA2 8PP. Tel: Swansea 24561 (day or night). □

### TRIBOLOGY DIARY

| | | |
|---|---|---|
| Sep. 22–26 | Tribology in Iron and Steelworks. | London |
| Oct. 23–24 | Effects of temperature on Lubrication systems. | Paisley College of Technology |
| Nov. 24–26 | Tribology in Powder Metallurgy. | Eastbourne |
| Nov. 26–28 | 3rd Industrial Lubrication and Tribology Exhibition. | London |

More information: Mr D. Brown, Institution of Mechanical Engineers, 1 Birdcage Walk, London SW1. Tel: 01-930 7476.

A two-day symposium and demonstration to explain the principles of tribology and to show how they can be applied in industry will be held at the National Centre of Tribology, Risley, Warrington, Lancs., on September 30 and October 1, 1969. Registration fee: £22 10s.

## ADVISORY COMMITTEE ON LEGAL UNITS OF MEASUREMENT

The Minister of Technology, Mr Anthony Wedgwood Benn, in a written Parliamentary Answer has announced the appointment of an Advisory Committee on Legal Units of Measurement, to advise him on that part of the metric legislation which will provide for the legal definition of units of measurement and related matters concerning physical standards.

An extract from the written reply follows:

'The legislation which the Government have undertaken to introduce to facilitate the adoption of the metric system in this country, will include provision for defining the metric units of measurement which are to be authorised for all future legal purposes and for relating them to all other units at present in use.

'In principle, the Government accept, as a basis for the changeover to the metric system, the International System of Units as recommended by the Conference Generale des Poids et Mesures, which was set up by international agreement in 1875 by the Convention of the Metre. The UK joined this Convention in 1885.

'While, however, there is international agreement on the units of measurement to be used for the great majority of purposes, there are nevertheless some areas of genuine difficulty, which will require expert consideration and the widest consultation with all the relevant interests, both within Government and throughout the economy, as well as internationally, before a complete system of measurement can be defined for purposes of the proposed legislation.

'The Government have therefore decided that I should appoint an Advisory Committee to advise me on that part of the metric legislation which will provide for the legal definition of units of measurement and related matters concerning physical standards. My intention is that this Committee should be composed of experts from the principal Departments and Research Councils concerned, the Metrication Board, the British Standards Institution and other widely representative bodies whom I propose to invite to participate in the Committee's work. I have appointed Mr A. H. A. Wynn, Head of my Standards Division, to be Chairman of the Committee.' □

# newtechnology

Number 49 August 1971 A supplement to *Trade and Industry*


# Optical Processing for Better Picture Intelligibility

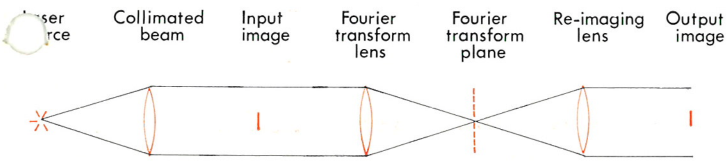

by J. D. Redman

**Atomic Weapons Research Establishment, Aldermaston**


Just as an electronic signal can be analysed into its constituent temporal frequencies, so a pictorial scene can be analysed into constituent spatial frequencies, and it follows therefore that a picture can be processed to improve its intelligibility by optical methods. This article reviews some of the techniques employed for the purpose by the Optics Branch of AWRE's Applied Physics Division.

In the AWRE optical-processing system the picture—usually in the form of a transparency—is illuminated by a collimated laser beam, and the light it diffracts is collected by a converging lens. This very simple optical system has the remarkable property of being able to perform the operation of Fourier transformation: if the transparency is placed at the front focus of the lens, its Fourier transform is produced at the back focus. The distribution of light in the back focal plane of the lens can be regarded as a map of the spatial frequency content of the original picture, and predetermined frequencies can hence be selectively removed by placing obstacles in this plane. For example, low frequencies can be removed by putting an opaque spot in the centre of the Fourier transform plane,

*Figure 1. Basic optical system used in processing.*




and high frequencies can be eliminated by inserting a plate containing a hole with a radius corresponding to the highest spatial frequency to be left in the picture. After filtering, the picture is viewed by means of a re-imaging lens, as shown in Figure 1. The photographs in Figure 2 exemplify the results obtained: low-pass filtering (the filtering operations are termed low-pass and high-pass by analogy with electronics) has removed the dots from the halftone, and in the other photograph high-pass filtering has taken out the low frequencies and emphasised the highlights.

Inherently, an optical filter has a much sharper cut-off than its electronic counterpart. Moreover, whereas, in the case of the electronic filter, the input is a function of one variable (time), in optics the input may be a function of two variables—the spatial co-ordinates. Consequently optics can be used to perform filtering operations for which there are no electronic analogues. One example—directional filtering to remove lines oriented in a particular direction—is illustrated in Figure 3.

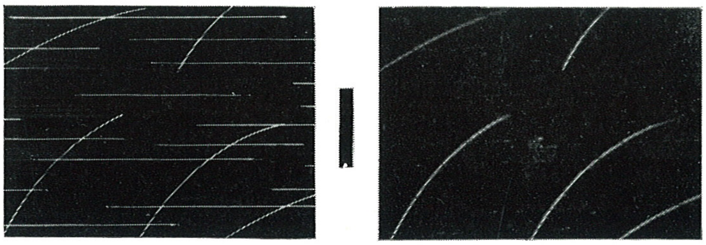
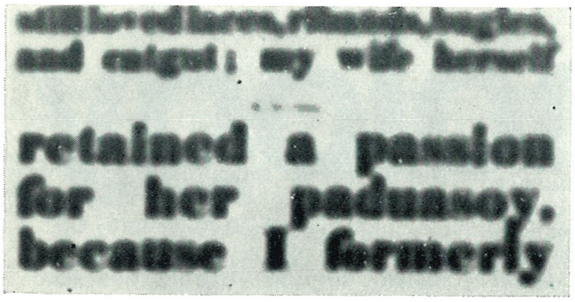


*Figure 3. Directional filtering. Left: input. Centre: filter. Right: output.*

This result was obtained by inserting into the transform plane an opaque mask (its shape is shown in the figure) arranged to point in a direction at right angles to the lines required to be removed. Optical processing can therefore be used to clean up a picture by eliminating unwanted features. Optics can, however, be employed also for much more sophisticated applications: for example, to deblur pictures—provided that the nature of the blur is known or can be estimated.

The blurring process can be looked at as the convolution of an ideal picture with a blurring function: it is as though the

*Figure 2. Amplitude filtering. Left: low-pass. Right: high-pass.*



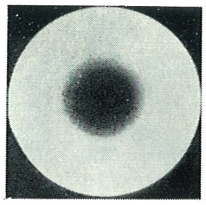
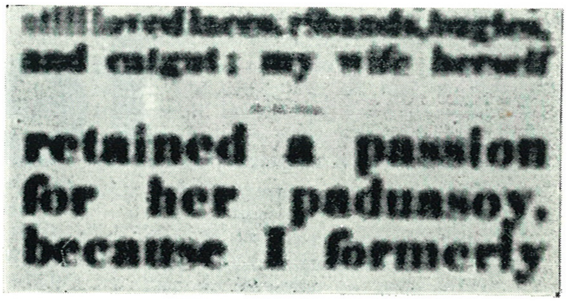



Figure 4. Use of least-squares filter. Left: input. Centre: filter. Right: output.




ideal picture had been scanned by a scanner of poor impulse response. Happily for our purpose, convolution in the space domain is equivalent to multiplication in the Fourier domain. In other words, the blurring could be considered as having been achieved by transforming the ideal picture into the Fourier domain, multiplying it by the transform of the blurring function, and then retransforming the final result. Clearly, therefore, if we know the transform of the blurring function, and can multiply by its inverse, the picture can, in principle, be deblurred. As always, however, things are never quite so simple in practice. There may, for example, be zeros in the transform of the blurred picture (ie, certain frequencies may be irretrievably lost), and care must be taken not to enhance noise. Consequently the basic inverse filter normally has to be extensively modified to achieve any significant improvement in the usefulness of a picture. Figure 4 illustrates the effect of using a filter which takes noise into account.

In this example only amplitude filtering was required (see Figure 5). Often, however, it is necessary—for instance, in the

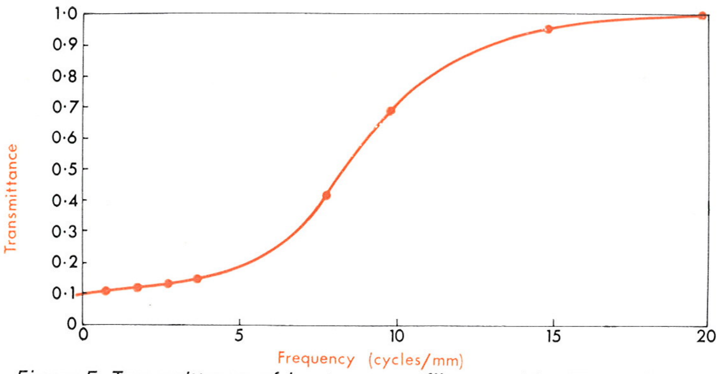
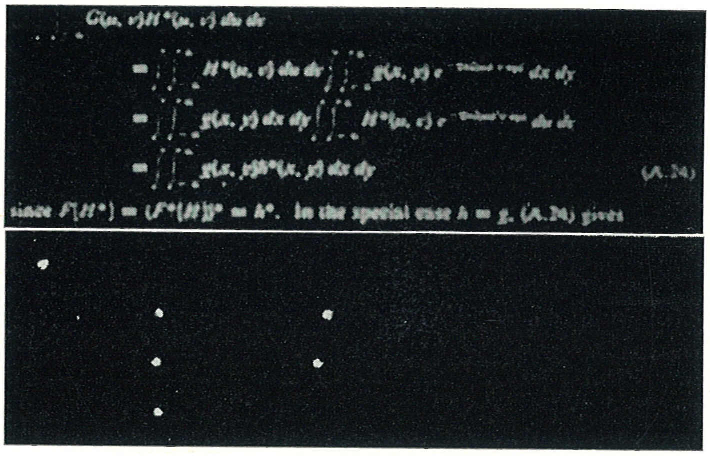



*Figure 5. Transmittance of least-squares filter used for Figure 4.*

correction of linear smear—to use a filter which also modifies the phase of the light passing through it. Phase filters may be produced directly by depositing thin films on to optically flat substrates, but this technique is practicable only in very simple cases. Where more complicated filters are required, a holographic technique may be employed. The simplest holographic filter is used to perform the cross-correlation operation. The AWRE procedure is as follows. It we want to cross-correlate two functions, we make a hologram of the Fourier transform of the first and illuminate it with the Fourier transform of the second (applying the familiar optical system shown in Figure 1), the cross-correlation function being then displayed in the final image plane. In effect the hologram serves as a filter which multiplies the Fourier transform of one function by the complex conjugate of the Fourier transform of the other. The final imaging lens retransforms the result, giving an output such as that illustrated in Figure 6.

*Figure 6. Cross-correlation. The dots in the lower part of the field correspond to the positions of the double integral signs in the input (top).*



In the absence of noise, the general deblurring problem can be solved by means of an inverse filter—one whose amplitude transmittance is inversely proportional to the Fourier transform of the blurring function: in other words, if the Fourier transform of the blurring function is H, where H may be complex, a filter with an amplitude transmittance of 1/H

*Figure 7. Holographic deblurring.*

(or, in other terms, $H^*/(HH^*)$ or $H^*/|H|^2$ where $H^*$ is the complex conjugate of H) is required. The problem may, therefore, be reduced to that of producing two filters to be used in series. One has to have transmittance $H^*$, and the other transmittance $1/|H|^2$. The second filter, which is entirely real, may be achieved merely by exposing a photographic emulsion to the Fourier transform of the blurring function, and developing in such a way that the amplitude transmittance is inversely proportional to intensity. The $H^*$ component is produced holographically, as described earlier. An example of holographic deblurring is shown in Figure 7.

Optical-processing methods are obviously less versatile than digital ones, but, where they *are* applicable, they enable large volumes of data to be handled quickly and economically. Many of the practical difficulties which in the past have prevented them from being widely employed have now been overcome, and interest in optics is reviving.

**More information:** Mr. J. D. Redman, Applied Physics Division, Atomic Weapons Research Establishment, Aldermaston, Reading RG7 4PR. Tel: Tadley 4111.

# A New One-Stage Pseudo-Grignard Reaction

by D. H. Richards

**Explosives Research and Development Establishment, Waltham Abbey**

The Grignard reaction—Victor Grignard was awarded the Nobel Prize for its discovery in 1912—is a two-step process, involving reacting an alkyl or aryl halide (RX) with magnesium in ether to form the Grignard reagent RMgX, and then reacting the reagent with aldehydes, ketones or esters to produce a wide variety of alcohols. The process is one of the most important in synthetic organic chemistry, and has found wide application in the pharmaceutical and fine-chemical fields, and the Explosives Research and Development Establishment's discovery (during an investigation exploring organometallic routes to new copolymers and small molecules) of a new technique for carrying out Grignard-type reactions in a simple one-stage operation, and with very high product yields, is hence a significant development. Patents covering the process have been filed, and information on this aspect can be obtained from the National Research Development Corporation.

In a typical reaction, a solution of the carbonyl compound and the organohalide is prepared in tetrahydrofuran, with the halide in slight excess (~10 per cent) of that required by the stoichiometry of the reaction. This solution is then slowly added to a stirred suspension of lithium pieces in tetrahydrofuran, with the reaction vessel blanketed by an inert gas, and a highly exothermic reaction ensues. The rate of the reaction is controlled by the rate at which the reagents are added (the addition takes place over 1-2 hours, by which time the reaction is essentially complete), and the temperature is maintained at ambient or below. On completion of the reaction, the excess lithium pellets are filtered off, and the solvent is removed for re-use by vacuum distillation. Hydrolysis of the residue generally results in a phase separation, and the alcohol—comprising the upper layer—can then be washed and dried; if there is no phase separation, the alcohol is extracted by ether.

As the table shows, the method is of wide scope. Alkyl and aryl bromides have been used the most extensively, but chlorides and iodides also undergo the reaction. The process is applicable equally to aldeyhydes, ketones and esters, gives substantially higher yields than those achieved by the conventional Grignard reaction, and, in addition to offering the advantage of being a one-stage process, has an easier work-up procedure. The temperature of reaction does not have a great effect on the yield of product (and does not therefore require careful control during synthesis), and, although for optimum yield a moderate excess of alkyl halide has to be present, the concentration of the reactants is not a significant factor in determining the conversion. Nor does the size of the lithium pellets greatly influence the efficiency of the process—in fact, the yields listed in the table were obtained with systems where the lithium pellets were cut from conventional lithium rod by means of scissors.

The reaction appears to progress via the *in situ* conversion of the alkyl halide into the alkyl lithium and the rapid attack of this reagent on the carbonyl compound: eg for an aldehyde,

$$RX + 2\,Li \rightarrow R^-Li^+ + LiX$$
$$R^-Li^+ + R^1CHO \rightarrow RR^1\,CHO^-Li^+$$

It was hoped that it would be possible to substitute sodium for the more expensive lithium, but no reaction took place when the attempt was made—due apparently to the fact that the sodium halide generated during the initial reaction is very insoluble in the reaction medium and is deposited on the surface of the sodium metal, thus inhibiting further reaction. Lithium halide, being more covalent, is much more soluble in tetrahydrofuran, and hence does not inhibit in this way. Although lithium is therefore necessary for the reaction, it does not follow that the cost of the operation is high. Lithium halide is the only inorganic material present, and can easily be recovered by distillation of the aqueous layer and either electrolysing on site or returning to the supplier for processing. Similarly the solvent can be recovered for further use by distillation at the prehydrolysis stage, and can be used directly without further purification. Since yields are high and the reaction time is short, the new process therefore offers a cheap way of manufacturing a variety of organic alcohols.

**More information:** Dr. D. H. Richards, Non-Metallic Materials Branch, Explosives Research and Development Establishment, Waltham Abbey, Essex. Tel: Waltham Cross 23688.

Dr. R. S. R. Parker, National Research Development Corporation, Kingsgate House, 66 Victoria Street, London, S.W.1. Tel: 01-828 3400, ext 379.

**Yields from Lithium and Organic Halides**

| Carbonyl compound | Halide | Product | % Yield obtained | % Yield from Grignard method |
|---|---|---|---|---|
| Paraformaldehyde | n-hexyl bromide | 1-heptanol | 72 | — |
| Paraformaldehyde | n-dodecyl bromide | 1-tridecanol | 80 | — |
| Propionaldehyde | ethyl bromide | 3-pentanol | 90 | — |
| Propionaldehyde | methyl iodide | 2-butanol | 51 | 47 |
| n-Hexaldehyde | methyl bromide | 2-heptanol | 70 | — |
| Acetaldehyde | n-pentyl bromide | 2-heptanol | 39 | — |
| n-Butyraldehyde | n-pentyl bromide | 4-nonanol | 73 | 73 |
| n-Butyraldehyde | n-butyl bromide | 4-octanol | 73 | — |
| n-Butyraldehyde | n-propyl bromide | 4-heptanol | 89 | — |
| Acrolein | ethyl bromide | hept-1-en-3-ol | 90 | — |
| Methacrolein | n-butyl bromide | 2-methyl-hept-1-3n-3-ol | 93 | — |
| Benzaldehyde | bromobenzene | benzhydrol | 95 | 55 |
| Benzaldehyde | chlorobenzene | benzhydrol | 100 | — |
| Benzaldehyde | iodobenzene | benzhydrol | 64 | — |
| Acetone | bromobenzene | dimethylphenyl carbinol | 34 | — |
| Mesityl oxide | bromobenzene | 1, 3-dimethyl-1-phenylbutadiene | 32 | 40 total yield |
| | | 1, 1-dimethyl-3-phenylbutadiene | 36 | |
| Benzaldehyde | allyl bromide | allyl phenyl carbinol | 71 | Poor |
| Methyl benzoate | methyl iodide | dimethylphenyl carbinol | 74 | 78 |
| Methyl methacrylate | n-butyl bromide | 2-methyl-3-butyl-hept-1-3n-3-ol | 89 | 15–20 |
| Benzaldehyde | cyclohexyl chloride | cyclohexylphenyl carbinol | 60 | — |
| Benzaldehyde | cyclohexyl bromide | cyclohexylphenyl carbinol | 72 | — |
| di-n-butyl ketone | n-butyl bromide | tri-n-butyl carbinol | 91 | — |
| n-Decylaldehyde | n-decyl bromide | eicosane-10-ol | 49–50 | — |
| Ethyl formate | n-decyl bromide | heneicosane-11-ol | 60+ | — |
| Ethyl formate | n-butyl bromide | nonanol-5 | 91 | — |

# Applied Research in the Rheology of Non-Newtonian Fluids at Warren Spring Laboratory

This article gives a general outline of the work on the rheology of non-Newtonian fluids being conducted at Warren Spring Laboratory. Further articles, describing specific aspects of this work, will appear in the next issue of *New Technology*. More information on WSL rheological know-how and services can be obtained from: Dr. D. C.-H. Cheng, Materials Handling Division, Warren Spring Laboratory, Gunnels Wood Road, Stevenage (Tel: Stevenage 3388).

Rheology—'the science of flow of matter; the critical study of elasticity, viscosity and plasticity'—is a discipline of obvious importance to the chemical and process industries, particularly where the fluids being handled are non-Newtonian.

Non-Newtonian? . . . Newton defined viscosity as the ratio of shear stress (force per unit area) to shear rate (velocity gradient) in the flow of a fluid, and in the case of gases and liquids—Newtonian fluids—viscosity is constant with respect to shear rate. There exists, however, a wide range of fluid-like materials—the so-called non-Newtonian fluids—which do not behave in this way: their viscosity is not constant with respect to shear rate and/or their flow behaviour cannot be fully described in terms of viscosity alone. Non-Newtonian fluids—polymer melts and solutions, biological fluids, paints, printing inks, greases and clay suspensions, and certain foodstuffs are examples—can be classified (1) as pseudo-plastic or as dilatant (depending on whether viscosity decreases or increases with shear rate), (2) as thixotropic (viscosity varies with time, although the shear rate remains constant), or (3) as visco-elastic (the fluid exhibits elastic behaviour). Briefly, if, as in the case of a high-polymer melt or solution, the fluid consists of or contains high-molecular-weight molecules, it will exhibit elastic as well as viscous behaviour: it will be visco-elastic. The pseudo-plastic, dilatant and thixotropic types of non-Newtonian fluid comprise dispersions or mixtures of solid particles (or of droplets of liquid) in a suspending liquid medium. If the particles or droplets are small enough for the electrochemical interaction between them to dominate over gravitational forces, they will remain in suspension, and, even if the fluid is thixotropic, the recovery times may be such as to enable it to be treated as purely viscous under continuous-flow conditions. If the particles are sieve-size or larger, the gravitational forces will be dominant and the particles will tend to sediment; flow behaviour is then determined by the interplay between the gravitational pull and the drag and lift resulting from the interaction between the particle motion and the flow of the suspending liquid.

The three types of non-Newtonian fluid differ profoundly in their flow behaviour therefore, and consequently they pose problems to the chemical and processing industries—problems which loom ever larger as more and more products are developed and the need to modernise and automate manufacturing systems in such traditional industries as food and ceramics becomes more compelling. In this context, data on rheological properties can be used not only to characterise the fluid from the processing point of view, but also as a tool in quality control and in optimising the design and operation of the processing plant. Rheological data can shed light on such 'practical' properties as the texture of foodstuffs and the spreading behaviour of paints and inks; in some cases, they can relate the properties of an intermediate product directly to the quality of the final product; and, not least, they can provide a correlation with plant performance which can be used as a means of process control and of designing new 'flow' equipment (eg, pipelines, pumps, agitators, extruders, heat exchangers) on the basis of laboratory tests. They have hence become virtually indispensable to the chemist, process engineer and plant designer. Determination of this information is, however, a specialist task, requiring experienced staff and considerable facilities . . . And it is here that the Materials Handling Division of Warren Spring Laboratory comes into the picture. Set up in 1962 to deal with the problems of storing, transporting, feeding and mixing slurries, pastes, powders and granules in a factory, the Division has two sections, one concerned with the mechanics of bulk solids, the other with non-Newtonian materials. Discussion of the bulk solids section will be postponed to a future issue of *New Technology*: in this article we will restrict ourselves to reviewing the services, facilities and technical and economic advantages which the rheological section offers industry.

WSL's basic research on applied rheology has two main aims: to establish fundamental data on the properties of non-Newtonian materials and on the performance of handling equipment and plant; and to apply this knowledge in the evaluation of techniques and design procedures, and in the development of equipment and plant, relating directly to specific industrial needs. The three constituents of the research programme—the rheological properties of non-Newtonian materials; research on unit operations and the performance of process equipment; and the development of instrumentation for process plant—are described below.

## 1 Rheological properties of non-Newtonian materials

Research on determination of **viscosity** has centred on purely viscous fluids; ie, fluids with negligible elastic and time-dependent properties, whose viscosity can be determined by the shear-stress/shear-rate relationship alone. Mathematical formulae permitting the derivation of various parameters have been established for all viscometric flows (including, in some cases, those involving non-isothermal conditions), and, since in practice viscometric flows are limited by end effects, wall slippage or the onset of secondary or turbulent flow conditions, these aspects have also been studied. The wide range of materials covered by this work necessitates the use of a variety of viscometers, including a specially developed low-viscosity tube viscometer, the Davenport Extrusion Rheometer, the Ferranti Portable Viscometer, the Contraves Rheomat-15 Viscometer, the Ferranti-Shirley Cone-Plate Viscometer, and the Sangamo Weissenberg Rheogoniometer.

WSL's work on **thixotropy** was prompted by the property's importance in the production of paints, printing inks, greases, clay slurries, pigment pastes, and other industrial materials. The Laboratory's research in this field is continuing on the basis of a concept explaining thixotropic behaviour in terms of the structure's breaking down on shearing and building up on rest.

The properties of **pastes** are also being investigated—initially to provide a reference point for subsequent work on unit operations involving such fluids. The first aim is development of a theoretical framework for experimental measurement.

## 2 Research on unit operations

**Pipeline transport** is the simplest type of unit operation. The principles of design of pipelines for Newtonian fluids are well established, but not until WSL's work was a *comprehensive* design procedure evolved for non-Newtonian materials. The WSL procedures (which will form the subject of an article in next month's *New Technology* were developed after a combination of fundamental studies, generalisation from published information, and experimental work on a pipeline rig.

In another aspect of operations research, WSL—with the approval of potential users, the active co-operation of pump manufacturers and advice from the trade and research associations—has embarked upon a programme of work aimed at providing information enabling the most suitable type and size of **metering pump** to be selected for use in feeding a particular non-Newtonian fluid. The intention is to establish performance data for a range of typical pumps (eg, peristaltic, plunger, diaphragm and screw), and so correlate pump characteristics with the rheological parameters of the pumpable fluids. A similar exercise is under way (1) to shed light on the performance of the **industrial mixers** now being used for mixing non-Newtonian fluids and pastes, and (2) to develop new forms of mixer.

### 3 Research on process-plant instrumentation

The trend towards continuous operation and automation has focused attention on the lack of suitable process-plant instruments. At WSL two types of instrumentation are at present under investigation: flow meters and process viscometers.

The work on **flow meters**—the tests were conducted on meters of orifice (up to $1\frac{1}{2}$ in. in size), turbine and variable-area type, handling a range of bentonite and lapronite slurries and a suspension of starch particles—has shown that, hydrodynamically, they behave the same with non-Newtonian as with Newtonian fluids, though mechanical effects, such as jamming of the turbines or retention of the float, limit some of the meter's usefulness for non-Newtonian applications. Since development of a satisfactory procedure for continuous paste mixing is dependent on the development of a suitable method of flow measurement, work is also in progress to produce a meter suitable for installation in a 4-in. pipe forming part of a continuous mixing rig.

The **process viscometers** at present commercially available were designed to handle Newtonian fluids, which can be specified by one parameter—viscosity; and they therefore operate at a single flow rate or rotational speed. In general, however, multi-point measurements are necessary where the fluid is non-Newtonian: hence WSL's development work on process viscometers. Preliminary research to gather rheological and other relevant data on the fluids and the pertinent processes revealed the need for an automatic viscometer capable of continuous measurement on a process stream. Two were subsequently developed: one, a modified Rheomat-15 viscometer, will be described in next month's issue of *New Technology*; the other—an elastico-viscometer—operates on the oscillatory mode, and enables the elastic property of a fluid to be determined in addition to its viscosity.

# Sponsored Research

**In the rheological field, as in all the others in which it is involved, Warren Spring Laboratory's aim is to apply the results of its research directly to industrial practice, and, with this aim in mind, it undertakes contract work on behalf of firms encountering difficulties with non-Newtonian fluids. Companies wishing to avail themselves of this service should contact the Laboratory (either directly or via the nearest Department of Trade and Industry regional office or the local Industrial Liaison Officer), so that an agreed programme of work can be drawn up. The whole research can be conducted by the Laboratory, or, alternatively, arrangements can be made for the company's staff to work alongside the Laboratory's, or even to work alone using the Laboratory's facilities, but, whatever the arrangement, the closest liaison is maintained with the sponsor, enabling progress to be reviewed and, if necessary, aims redefined and methods adjusted. To date, such sponsored projects have been concerned solely with specific unit operations, but the increasing range of its basic research programme is rapidly placing WSL in a position also to tackle assignments entailing renovation or redesign of an entire process.**

**Another of Warren Spring's rheological services—the organisation of training courses for companies requiring them—will be described in the next issue of *New Technology*.**

### Some case histories

WSL has conducted sponsored projects on behalf of firms in such widely different fields as the confectionery, pharmaceutical, engineering, oil, industrial-waste, paint, photographic and electrical industries.

Some examples:

★ To overcome difficulties due to sagging of a polysulphide sealant used in the civil-engineering and building industry, WSL employed the Weissenberg rheogoniometer to obtain the basic rheological data needed to correlate the sagging behaviour of alternative materials, and then compared these data with those established using the Brookfield Synchrolectric viscometer as a factory control instrument.

★ Using a Brabender Plastograph, WSL determined the rheological properties of a mixture of coke particles, pitch and other minor additives at elevated temperatures, and showed that a critical composition existed at which the amount of pitch present was just sufficient to coat all the particles, and which would hence ensure optimum quality in the final product.

★ Using a Ferranti-Shirley cone-and-plate viscometer, WSL established rheological data for a rubber-latex/solid-fillers/thickener/protective-additives dispersion, which, taken in conjunction with other relevant physical properties, enabled the characteristics of the dispersion to be correlated with its performance. The exercise revealed a relationship between optimum performance and high viscosity, and enabled the company to maintain product quality by adjusting the thickener concentration.

★ The rheological behaviour of various blends of butter under steady-state shearing in the Ferranti-Shirley cone-and-plate viscometer was studied as a function of the formation of 'gritty blends'. The work showed that grits occurred as a result of differences in rheological properties, and, it not only provided a solution to the problem, but also led to the development of a new type of mixer applicable to any paste-like material.

★ The dynamic shear property of 'bouncing putty' (a high-molecular-weight silicon polymer) has been determined over the range 0.5 to 10 Hz and −20 to +55°C to supply data for the possible design of anti-vibration devices for high-speed rolling stock.

★ Using the Davenport extrusion rheometer, a range of solid/liquid dispersions employed as additives in the cable-making industry were subjected to viscometric measurements to determine the relative performance of the various dispensing systems proposed for use in pre-packing the materials to facilitate loading of the mixing machines.

★ The design of a mixing unit for production of an air-free metal-powder paste demanded that the shearing action of the pump recirculating the mixture be lower than the critical value preventing degradation of the solid particles present in the paste. WSL established this critical value, so enabling determination of the size of pump required.

(*continued on next page*)

## RHEOLOGY RESEARCH
*continued*

★ An acid/flavour/colouring additive dispersed in a syrup/pectin solution employed in continuous manufacture of pectin jellies was maintained at the required distribution by using a pipe section filled with spheres as an in-line mixer.

★ Various studies of agitation in chemical reactors have been conducted to obtain scale-up data enabling the power requirement of full-scale plants to be established from pilot-plant results.

Two other examples of agitation and mixing investigations:

The yield from a process involving precipitation of an organic chemical by the reaction of liquid and powdered materials was increased greatly by optimising the speed of agitation and by appropriately modifying the geometry of the agitator.

After various trial-and-error attempts to increase the uniformity of continuous mixing achieved by high-speed extrusion screw pumps, a company turned to WSL. The Laboratory conducted a mathematical analysis of the mixer, which showed that the performance of the production screw could be improved by using shallow screw depths, but that even better results would be achieved by using an annular mixer.

## B.C.S./I.E.E. Course in Electrical Measurements

The British Calibration Service is co-sponsoring a Vacation School in Electrical Measurement Practice to be held at York University from September 19 to October 1, 1971. Organised by the Institution of Electrical Engineers, and the third in a series which started in 1968, the School will cover the best modern practices in making accurate electrical measurements at dc and low frequencies (up to 100 kHz). The course should prove of particular interest to those engaged in the operation of measurement laboratories and test and calibration facilities, and to those involved in teaching electrical measurement, and is designed to suit both professional engineers and those interested in the field who are without formal qualifications beyond, say, ONC.
**More information:** Miss J. M. Edwards, Divisional Secretary, Science Education and Management Division, I.E.E., Savoy Place, London, WC2R 0BL.

# British Calibration Service: Approved Laboratories

A ready-reference list of BCS-approved laboratories was published in *New Technology* Number 45 (April 1971). Since then, the following additional laboratories have received BCS approval:

Electrical (dc and lf)
**Calibration Laboratory,**
**Bennett Brown (Technivision) Ltd.,**
No. 9 The Trading Estate,
Station Approach, Fleet, Aldershot, Hants.
Mr. J. G. Baggs,
Fleet 21666.

Electrical (dc and lf)
**Standards Laboratory,**
**Calibration Systems Ltd.,**
Blackwater Station Estate, Camberley, Surrey.
Mr. J. D. Cook,
Camberley 28121.

Electrical (dc and lf)
**Electrical Measurements Laboratory,**
**Naval Ordnance Quality Assurance Establishment,**
Janson Street, Sheffield, S9 2LJ.
Mr. R. R. Butters,
Sheffield 49261, ext 7 or 10.

In addition, BCS approval of the **Standards Laboratory, Wayne Kerr Co. Ltd.,** has been extended to cover hf measurements.

The following changes in telephone numbers have been notified:

Winfrith Mechanical Standards Laboratory,
Dorchester 3111.

Cranfield Metrology Laboratory,
Bedford 51551.

# news

## Computer evaluation of a range of alternatives in a design situation . . .

Facilities at DTI's Computer-Aided Design Centre, Cambridge, were used recently to perform an exercise in which a sophisticated computer model was used by an architect for evaluating a range of alternatives in a design situation. The model used was the LUBFS Environmental Model, developed at the Centre for Land Use and Built Form Studies by Dean Hawkes and Richard Stibbs, and the architect user was Georges Soukiassan, Assistant Chief of Building Management at the World Health Organisation in Geneva. Computer file storage facilities were donated by Applied Research of Cambridge.

The LUBFS Environmental Model—a computer system constructed for use as a research tool—collates a series of established environmental design processes by relating them to a single data structure capable of accepting data describing virtually any building. The 'environment', as simulated by the model, is the visual, thermal and acoustical description of conditions within a building.

The range of analysis in the exercise was:

1. Daylight-factor calculation, allowing evaluation of any side-lit room with windows in any number of walls.
2. Artificial-lighting installation design, enabling the number of fittings required and the total wattage to be calculated from a basic fitting specification and the required level of illumination.
3. Thermal-performance evaluation, which calculates heat flows through the outside surfaces of each room for clear and cloudy, winter and summer days.
4. Acoustic evaluation, calculating the noise levels in the room resulting from outside sources, and permitting comparison with a specified maximum acceptable noise level.

The model is available at the CAD Centre for general use by architects and planners.

## . . . And computer-aided design of continuous prestressed concrete beams

Developed for the design of continuous prestressed concrete beams by the CAD Centre, Cambridge, and Stirling, Maynard and Partners, consulting engineers, the 'PRESBEAM' program assumes that the shape of the beam has been determined prior to the start of the program and that the applied bending moments have been generated by other means. This situation coincides exactly with bridge problems, where the shape is often strongly influenced by aesthetic considerations and where the applied moments are an envelope resulting from multiple moving-load cases and an elaborate three-dimensional analysis. Using the program, the designer types appropriate commands on a storage-tube display console: he can thus first display an elevation of the beam and any optional preliminary guess at a prestressing system, then analyse the loaded beam and display the resulting stresses, and finally, if the results are unsatisfactory, he can manipulate the prestressing system and display the stresses until a satisfactory design has been obtained. A permanent record of a design may be requested at any stage. Savings of £200-£300 in design costs can be achieved during a single session using 'PRESBEAM'.
**More information:** Chief Consultant, The Computer Aided Design Centre, Madingley Road, Cambridge, CB3 0HB.

## Government Chemist on show

The environmental aspects of its services to other Government departments will be stressed when the Laboratory of the Government Chemist puts its work on show at open days on October 20 and 21. The work of the Laboratory's divisions concerned with food and nutrition, agricultural materials, radio-chemistry and water and health services will be prominent, and there will be displays involving food additives and contaminants, vitamins, pesticides, effluents, drugs and medicines, dental materials and bacteriology. Other exhibits will include analytical examination of beer, tobacco, hydrocarbon oils, minerals and industrial atmospheres, plus recent developments in the automation of analytical methods. Tickets, free, from: Dr. D. C. Abbott, Laboratory of the Government Chemist, Cornwall House, Stamford Street, London, S.E.1.

## Degradation of grinding belts

The Fulmer Research Institute has been awarded a two-year contract (funded jointly

by English Abrasives Ltd. and the Department of Trade and Industry) to study the mechanism of degradation of grinding belts and the effect of the grinding operation on the mechanical properties of the component. Extensive use will be made of the Institute's combined facility for electron microscopy/microprobe analysis.
**More information:** Dr. J. A. Coiley, Fulmer Research Institute, Stoke Poges, Slough, Bucks SL2 4QD. Tel: Fulmer 2181.

## The real cost of computing

How much does computing really cost, how can this cost be determined, what overheads should be included and what indirect costs are involved? These are some of the questions to be discussed at a National Computing Centre conference to be held in London's National Film Theatre on September 7. The conference aims to help both users and potential users make the right computer decisions from a true identification of all the relevant costs.
**More information:** Peter Wheeler, Public Relations Officer, National Computing Centre, Quay House, Quay Street, Manchester M3 3HU. Tel: 061-832 9731.

## All aspects of joinery

*Joinery*, by C. H. Tack of the Forest Products Research Laboratory, covers all aspects of the subject and draws together information otherwise obtained only by studying many different publications. Emphasis is laid on the importance of using wood at the appropriate moisture content. £1 from HMSO and booksellers.

## Mines research contributes to other industries

Examples of the way in which research carried out by DTI'S Safety in Mines Research Establishment can be applied to non-mining industries are to be found in SMRE's annual report now published. They include work on anti-slip studs for boots; research into the safety problems that could be created by a breach in the cooling duct of a nuclear reactor; and development of a device for detecting explosive fuel mists of the type that might be produced in ducts in the Concorde's fatigue-testing rig. Notable among mine safety research described in the report is work on instruments for detecting and measuring gases that pollute the mining environment. *Safety in Mines Research* 1970 is 60p from HMSO.

## Engineering in medicine—and textiles

Two new films have joined the *Engineers for Tomorrow* series, which aims to encourage able young people to consider engineering or technology as a profession. *Engineering in Medicine* looks at some of the branches of medicine in which the doctor and the engineer work together to raise the quality of medical treatment and rehabilitation. *The Engineer in Textiles* shows something of the enormous scope for the young engineer in today's highly automated textile industry. Both films are sponsored by DTI and produced through the Central Office of Information, and are available on free loan for non-theatrical use from the Central Film Library and its associated libraries in Scotland and Wales.

## Forty years' experience of temperature measurement

*Calibration of Thermometers*, by the late C. R. Barber of the National Physical Laboratory, reflects over 40 years' experience by the author in the field of temperature measurement. The term 'thermometer' covers a wide range of measuring instruments—including platinum resistance, gas and mercury-in-glass thermometers, thermocouples, and radiation pyrometers. 35p from HMSO.

## Computer Composing

Now available from HMSO (price 80p). *Computer Composing*—a report by C. J. Duncan, Director of Photography at Newcastle University—covers a five-year research programme conducted, on behalf of the Department of Trade and Industry, to provide the printing industry both with a series of computer programmes enabling all the operations of copy preparation, editing and photo composition to be performed, and with a wholly automatic system of preparing material from computers for publication in an improved, readable form.

## Group Technology appreciation

The DTI-sponsored and UKAEA-managed Group Technology Centre at Blacknest in Berkshire is offering a series of one-day Group Technology appreciation seminars during the coming months. Group Technology is a technique for producing small-batch component runs with the same economies as in continuous flow-line production. Seminars will be held this year on October 5, November 9 and December 7; and in 1972 on January 18, February 15, March 14, April 18, May 16, June 13 and July 11. Course fee is £15.
**More information:** The Secretary, Group Technology Centre, Blacknest, Brimpton, near Reading, Berks RG7 4RS. Tel: Tadley 4111 exts 5951/5864.

## 'Pressure-Probe Methods for Determining Wind Speed and Flow Direction'

A 125-page monograph of the above title, written by D. W. Bryer and R. C. Pankhurst of the National Physical Laboratory (and available—price £3.50—from HMSO), reviews the present state of the art, and should prove a valuable source of reference both to the specialist and the non-specialist. The monograph covers measurement of total pressure, static pressure, stream velocity and flow direction, construction of pressure probes, and the role of such associated apparatus as orientation and traversing devices and manometers.

## Computer abstracts

*International Computer Bibliography*, Volume 2, 1968/70, is the second part of a comprehensive compilation of abstracts relating to computer books in several languages. Volume 1 contained 6,000 entries and Volume 2 has a further 1,200. All aspects of computer technology and applications are covered, with 39 countries contributing. Volume 1 is £20, and Volume 2 £10, from NCC Publications, The National Computing Centre, Quay House, Quay Street, Manchester M3 3HU.

## Survey of Information Projects—3rd edition

Because the number of information projects undertaken has expanded so rapidly, a third edition of the booklet *A Survey of Information*

*Through a licensing arrangement with DTI's Warren Spring Laboratory, Biggs Wall and Co., Hitchin Road, Arlesey, Beds., can now offer, as a complete unit, all the components and fabricated sections required for treatment of oil-polluted beaches and inshore waters. For sea operation, the equipment is mounted on a small- or medium-size boat, and the dispersant is sprayed on to the oil slick from booms projecting over both sides at the stern, and then agitated by trailing surface breakers to produce an emulsion suitably stabilised for dispersal. To clear beaches, oil is sprayed by hand from two lances connected to the same basic equipment.*

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*Projects* is to be published by the Department of Trade and Industry. A large number of questionnaires sent out by the Department have been completed and returned but DTI is keen to make the survey as comprehensive as possible and would like to hear of any projects that may not have been so far covered. Companies, associations, university departments, libraries and other organisations with an active interest in information or document retrieval who have not so far received a copy of the questionnaire are invited to request one from: Department of Trade and Industry, Systems and Automation Division, Room 206, Dean Bradley House, 52 Horseferry Road, London, S.W.1. Tel: 01-799 5688 ext 172.

## A dynamic performance test for lathes

The Machine Tool Industry Research Association has developed a practicable cutting-test procedure for comparing the metal-removing capabilities of lathes. The test, which was developed under a contract from the Department of Trade and Industry, is described in a report, just published by MTIRA, which also describes the work done in developing the test procedure and gives instructions for carrying out the test and for interpreting the results. Despite the very large number of factors which can affect the cutting performance of a lathe, the relatively simple test procedure will give results which will usually be accurate to plus or minus 10 per cent, provided that the conditions of test are controlled in the manner described. Although the test procedure described relates only to lathes—the most common type of machine tool and the one using the simplest cutting methods—similar tests could now be formulated for other types of machine tool. The project involved the formulation of a cutting test procedure, identification of the factors having most effect on the liability of a machine tool to chatter, and the specification of acceptable limits on the values of the quantities involved, the specification of test conditions, and the determination of the repeatability of the results obtained. In addition, because of the need to be able to predict, from tests on one machine of a given type, the likely behaviour of other, nominally-identical, machines, consideration was also given to the way in which the test results are to be interpreted for this purpose. The report is £12 from MTIRA, Hulley Road, Macclesfield, Cheshire.

## Continued Government support for CoID

The Government is to continue support of the Council of Industrial Design to enable it to carry out its essential educational and promotional role, the Secretary of State for Trade and Industry, Mr. John Davies, has informed the House of Commons in a written answer. Announcing the result of his review of the Council, the Secretary of State said: 'I am satisfied that the Council has had a significant and beneficial influence on the standards of industrial design in this country and that there is a continuing need for Government support to enable it to carry out its essential educational and promotional role. Earlier I asked the Council, in collaboration with the Council of Engineering Institutions, to consider how progress might be made toward improving the standards of engineering design, and to this end an Engineering Design Advisory Committee has been established. I am convinced that the Council can give a valuable lead in this field. Accordingly I have now asked that it should adjust its priorities and, over time, give greater emphasis to this work.'

## Guarding machinery in foundries

Methods of guarding certain machines used in foundries are described in a report published by the Department of Employment. *Guarding of Foundry Machinery* (HMSO, 40p) is the first report of a sub-committee of the Joint Standing Committee on Health, Safety and Welfare in Foundries, and is directed not only to users, but also to machine makers, on whose efforts progress in the development of guards will largely depend. The report says: 'We should like to see all makers of machinery consciously designing it for intrinsic safety and selling machines with guards already fitted.' It deals in particular with moulding machines, core-making machines, and sand mixers and mills, which experience has shown to be particularly dangerous and for which there are no easily available commercial guards. Examples are given in each case of guards which have been used for several months in the day-to-day conditions of normal production.

## Improving welded-product design

A Welding Institute Conference at the Bloomsbury Centre Hotel, London, from November 16-19, will put into perspective some of the experience and research of the past years which could be used as design parameters, and will also examine recent research which has led to new engineering philosophies. Six technical sessions will cover: structural and stress analysis of welded fabrications; determination of strength criteria and design considerations related to welded joint properties; the design, inspection and quality control relationship; design organisation; and education and training trends. Many overseas delegates are expected.
**More information:** The Welding Institute, Abington Hall, Abington, Cambridge, CB1 6AL. Tel: Linton 162.

## A 'clinic' for materials-handling problems

A new approach to materials-handling problem solving which will enable industrialists to talk directly to experts who may have the solutions is being initiated by the National Materials Handling Centre. Each month the Centre is to hold a one-day 'problem clinic' dealing with a specific aspect of materials handling, at which experts will describe particular problems they have solved. John Williams, NMHC director, describes the clinics as 'development accelerators'. He says: 'New materials-handling techniques are constantly being introduced and it is important that potential users should have the opportunity to learn how they are being used and to assess their value. It is equally of value for the researcher, developer, engineer or manufacturer to gain an appreciation of the problems his equipment may be required to solve and that he should do this at an early stage in its development'. The clinics, to be held at Cranfield Institute of Technology, are open to members and non-members of the NMHC and are free of charge. The programme is as follows:

| | | |
|---|---|---|
| 1971 | Sept. 23 | Minicomputers in materials handling |
| | Oct. 21 | The air-cushion principle |
| | Nov. 18 | Expendable pallets |
| | Dec. 16 | Handling on building and construction sites |
| 1972 | Jan. 20 | Fire risks in warehouses |
| | Feb. 17 | Modular dimensions |
| | Mar. 16 | Selling in the Common Market |
| | April 13 | Order selection techniques |
| | June 1 | Sorting systems |
| | June 29 | Robots and their uses |

Reservations in writing to: Mrs. Enid Wilson, National Materials Handling Centre, Cranfield Institute of Technology, Cranfield, Beds.

## Patents Office information retrieval services

A new booklet, *About Patents*, describes in detail the information retrieval services provided by the Patent Office—the largest technical publisher in the country—and how to make use of them. The use of inexpensive Patent Office services for finding prior patent specifications dealing with any aspect of manufacture can prevent waste of time on the development of ideas which are not, in fact, novel. Manufacturers can also be kept aware of what is being patented in their own particular fields. The booklet also deals briefly with applying for a patent, what happens to the application, the exploitation of a patent including licensing, and how to obtain details of accepted patents, and contains a list of publications available from the Office and from other bodies concerned with invention and patent literature. The booklet is available free from Sale Branch, The Patent Office, Orpington, Kent, BR5 3RD.

**More information:** Where possible, items in *New Technology* indicate the best sources of further information. Readers are advised also to seek information on matters relating to *New Technology* from the following DTI regional offices: Edinburgh, 031-225 1822; Cardiff 62131; Newcastle upon Tyne 27575; Manchester, 061-832 9111; Leeds 38232; Nottingham 46121; Birmingham, 021-643 8221; Bristol 21071; South East and East Anglia, 01-799 5688. In Northern Ireland, information is given by the Dept. of Industrial and Forensic Science, Belfast 21722.

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