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MINISTRY OF AVIATION SUPPLY

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

Technical Report for the Period 1 7 1970 to 31 12 1970

INVENTORY 1972

Explosives [S]

DDC
JUN 6 1972

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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

TECHNICAL REPORT

FOR THE PERIOD

1 7 1970 to 31 12 70

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EXPLOSIVES

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FIELD 3 ROCKET MOTORS AND EXPLOSIVES

3.1 SOLID ROCKET PROPELLANTS

(a) COLLOIDAL PROPELLANTS

(i) Smoke and Flash

Basic Mechanism of Platonisation RESTRICTED

To date it has not been possible to obtain platonised ballistics with double-base compositions containing ingredients added to increase energy level, and to minimise flash or resonance. To provide a background for the development of improved compositions the past work on platonisation has been reviewed.

The current experimental approach involves photographic investigation of propellant burning at pressures up to 10 MN/m^2 in high-pressure equipment. Unplatonised HSC propellant shows a smooth featureless surface, but with an experimental composition (F488/2236), platonised from 4 to 9 MN/m^2, hot spots which are clearly visible at 2 MN/m^2 become smaller and disappear at about 6 MN/m^2. The hot spots are relatively immobile and fibrous in appearance. Consequently, the possibility of inducing the required catalysis by the inclusion of treated fibrous materials is now being examined.

Reduction of Smoke from Rocket Exhausts CONFIDENTIAL

Effort has been directed largely to processing and firing of simulated Pointer charges, inhibited with polyacetal and modified cellulose acetate formations, for comparative smoke assessment. Emphasis was placed on polyacetal because of the outstanding smoke reduction reported previously with sustainer charges, and on modified cellulose acetate of improved flame resistance because it should be readily bondable to CDB or cordite.

Development of Improved Cellulose Acetate Formulations CONFIDENTIAL

Earlier work showed that cellulose acetate/triacetin (CA/TA) inhibition gave less smoke than the original standard CA but it was known that this type of inhibition would have a very limited storage life owing to the high nitroglycerine absorption which destroys resistance to hot gases. A programme of laboratory investigations to improve storage life has been conducted on a series of modified compositions including "smokeless" organic fillers and ingredients selected to improve char characteristics. To provide standard values various inhibitors, including the original standard Hypalon CL 2759, have been tested. Times for burn-through and weight losses were determined in a standard laboratory flame test. Tests were also conducted with added nitroglycerine to simulate the effect of prolonged storage. Laboratory burn-through times (seconds) under standard conditions are summarised in Table 1.

TABLE 1

	Composition alone	With added Nitroglycerine
Hypalon CL 2759	about 70	-
Cellulose acetate/triacetin (CA/TA)	26	18
CA/TA Asbestos	-	21
Lead citrate	-	28
Cotton Linters	-	28
CA/TA/Lead citrate/15 UF*	37	-
/25 UF	64	42
/40 UF	97	67
/65 polyacetal	67	30
*Finely powdered urea formaldehyde resin		

These results indicate that the storage life of modified CA/TA inhibition should be greatly increased by the use of fillers and additives which have given results comparable with those given by some Hypalons. It was therefore decided to mould two modified cellulose acetate formulation, (CA/TA/40 UF, CA/TA/65 polyacetal) into Pointer beakers for full assessment. ERDE prepared the special moulding powders and the beakers were injection moulded by RARDE, who also prepared further polyacetal beakers from three commercial moulding powder variants.

Bonding to Propellant of Modified CA and Polyacetal CONFIDENTIAL

In view of the promising results obtained with UF- and polyacetal-filled CA/TA, the possibility of obtaining good bonding in CDB charges has been assessed. No special treatment was necessary as adequate bonding for preliminary firings could be obtained by direct casting into modified CA/TA beakers. The laboratory bond-test figures were below normal with polyacetal-filled CA, due possibly to its reduced cohesive strength.

Satisfactory bonding of polyacetal to propellant was achieved by a "satinising" pre-treatment (soaking in perchlorethylene, dioxane and paratoluene sulphuric acid) of the internal surface of the beaker followed by coatings of the standard Redux-Formvar adhesive system.

The initial studies on the bonding of polyacetal to cast double-base propellant has been completed, and an ERDE report is in preparation. An apparatus has been devised for "satinising" polyacetal beakers without producing etch streaks down the outside of the beaker due to spillage of satinising solution. A roll coat method of applying Redux/Formvar lacquers was used in the preliminary trials but more recently it has been found that a spray-on technique produces a

better finish. As the polyacetal beakers are slightly under-sized, CDB processing problems have arisen and for conveneince we have standardised on "Modified Pointer" charges with a flat-ended boost.

Full Scale Smoke Evaluation Trials CONFIDENTIAL

Static firings have been conducted in heavy-weight 150 mm diameter motors, simulating the full scale Pointer charge, with burning times of five seconds for the boost phase and 25 seconds for the sustainer phase. Molybdenum probes were used in some firings to ensure fully developed secondary combustion, as the normal motors gave little secondary combustion and were virtually flameless.

Visual estimates of smoke throughout the burning times of the motors were made by a number of observers. All the systems of current interest are closely competitive except for polyacetal which shows a distinct superiority with respect to smokelessness; this is particularly marked when there is no secondary combustion. The static firings imply that polyacetal inhibition is superior to the new CL 8980 system being developed for Pointer and that it might give acceptable smoke even if a flashless charge were used. The next stage is to develop satisfactory inhibition systems using polyacetal and modified CA and to establish the degree of confidence required for trials at the firing ranges.

Qualification of New Inhibitors CONFIDENTIAL

As noted above, the bonding of polyacetal to CDB presents problems; there is a slight mismatch of physical properties and no information on storage capability.

Modified Pointer charges with flat-ended boosts have been cast in polyacetal Hostaform C 9010 beakers ("satinised" and Redux/Formvar coated). Twenty-seven charges have been produced; 25 were free of flaws and two showed signs of bonding failure at the inhibitor/boost propellant interface.

All the work to date has been conducted with the partially standardised roll-coating technique. Five Pointer charges inhibited with polyacetal ("satinised" and Redux/Fromvar coated) have fired satisfactorily at ambient temperature and one at +60°C without failures. The main emphasis has been on temperature cycling capability; charges have been cycled in aluminium cans, lined with 2 mm Hypalon, broadly representative of a Pointer motor. A severe thermal cycle was selected (16 hours -40°C, 8 hours ambient, 16 hours +60°C, 8 hours ambient) and as the tests are being conducted with the equivalent of a bare motor there is a degree of over-testing compared with even Land Service usage. Some charges have been subjected to repeated cycling to onset of failure whilst others have been fired after a limited number of cycles. Two charges have survived 38 and 61 cycles with no sign of failure whilst two charges failed after 17 cycles. One early charge (B19) failed on its first cycle when cooled to -40°C. The failure was unusual since the beaker cracked as well as giving separation of polyacetal from the propellant. Only one charge (B24) has been fired at -40°C after four cycles and gave increased boost

pressure (12 MN/m^2) with irregular burning and a short burning time for the sustainer. Further tests are planned, particularly to characterise the low temperature capability, which seems a problem area, and four charges which have withstood three cycles successfully are ready for test.

The nitroglycerine take-up of polyacetal is low, but to obtain confidence in the storage capability of polyacetal-inhibited charges, storage for many months at 50° or 60°C would be required. To obtain a quick assessment, Pointer charges were cast into "satinised" and Redux/Formvar roll-coated beakers which had been immersed in casting liquid for three weeks at 60°C (the increase in weight was 9 per cent). After ambient storage single charges fired satisfactorily at ambient and +60°C. The other two charges were subjected to $2\frac{1}{2}$ standard temperature cycles; subsequently one was fired satisfactorily at -40°C, the other is awaiting firing. This quick assessment suggests that storage at 60°C for some months should have little effect on performance but further trials are required.

A few charges inhibited with polyacetal filled CA/TA have been fired satisfactorily in the modified Pointer configuration at 60°C and ambient temperature.

Swingfire Power Cartridges CONFIDENTIAL

The Mechanite 14 gas generator propellant is very sooty even without the charge inhibition. Tubular K rounds with the minimum of inhibition for neutral-burning (ends only inhibited with ethyl cellulose) were fired at 4.7, 7.9 and 12.4 MN/m^2 (using venturis giving approximately correct expansion to atmospheric pressure to avoid secondary combustion) and copious soot was observed (mark 10/10) at all pressures.

New compositions are being developed to give reduced smoke by eliminating the phthalate ester coolant. A considerably reduced nitroglycerine content should permit stress-relieved modified CA/TA inhibition to give long Service life. Compositions containing 21 to 25 per cent nitroglycerine have been assessed and F547/471, containing 23 per cent NG and with a calculated energy level of 2900 kJ/kg proved to be the most suitable ballistically. Several formulations, based largely upon F547/471, have been tried with the express purpose of reducing the NG content even further.

A preliminary mock-up charge using F547/471 with CA/TA inhibition indicated that very marked smoke reduction could be obtained over the present charge design. It is proposed to devise a model smokeless cartridge on these lines within the Swingfire charge dimensions. Arrangements have been made with BAC (Stevenage) to supply an Ordnance Board test rig and the consumable parts required for this type of development.

(ii) Extruded Propellants

Quality Control Support to ROF's RESTRICTED

Quantities of tested ballistic additives have been supplied in the past six months as follows: 244 kg lead stannate and Additive C, 30 kg lead phthalate, 11 kg lead beta-resorcylate, and 3 kg Dixigloss carbon black. In addition, 23 kg of tested basic copper salicylate has been supplied to ICI (Nobel Division) for use in CDB.

A 150 kg batch of Analar cupric oxide, received from BDH, milled and assessed by ERDE, has been forwarded to ROF Bishopton. In view of the recent high rejection rate the manufacturer was visited and it was found that the firm had modified its manufacturing method, so that the correlation between the ballistic activity that had been established, and specific surface, no longer held. A new body of correlative data is being built up relating to the modified manufacture, in collaboration with QAD(Mats) Burghfield and the firm.

A high proportion of AU charges for a rocket ejection seat being manufactured at ROF Bishopton are being rejected at inspection. Comparative radiography and rolling experiments are being carried out to elucidate the causes of the apparently poor homogeneity.

Possible Export Order CONFIDENTIAL

An enquiry has been received from the Societe d'Etude de Realisations et d'Applications Techniques (SERAT) for propellant having a burning rate of at least 32 mm/s at 30 MN/m^2 and à good temperature coefficient. Compositions made during the development of propellant LU approach the required rate and energy level and no great difficulty is expected in manufacturing a suitable propellant. No information has so far been received on the type of weapon involved.

Improved Platonised Rocket Propellants

Cool Gas Generator Propellants CONFIDENTIAL

Work has continued on the development of a suitable propellant for the RPE packaged liquid expulsion charge based upon F3/61 composition. This is being supplied for current work but is not ideal. The original requirement was for a burning rate of not more than 4.8 mm/s at 8.3 MN/m^2, but the operating pressure has since been amended to 6.2 MN/m^2. Experimental compositions involving changes in nitrocellulose level, white lead content, energy level and use of SOA have been assessed. A cooler version of F3/61 containing 57.5% NC has given a burning rate of 4.8 mm/s at 6.2 MN/m^2 and is platonised over the range 5.8 to 9.5 MN/m^2. It was difficult to reduce the burning rate, though a reduction in NC content resulted in a slight decrease. Increase in white lead content resulted in a decrease in plateau pressures whilst a decrease in white lead content substantially increased both burning rate and plateau pressures.

New Ballistic Modifiers RESTRICTED

It has already been reported that the use of copper phthalocyanine with lead beta resorcylate in an LU matrix was unsuccessful in producing platonisation. Its replacement by pyrolysed material (prepared by heating copper phthalocyanine to one-third its original weight, thus increasing its copper content) in F 488/2288 has given a region of low slope from between 30 and 40 MN/m^2; further work is proceeding. Oxides of cobalt, nickel, antimony and tin have been tested as possible ballistic modifiers. Commercial samples of cobalt and nickel oxides were used in place of cupric oxide in F488/1480, a hotter version of KU, without success. Three formulations based upon an AU matrix and using either cobaltous or cobaltic oxides have also been prepared. Only the formulations using white lead together with either of the oxides showed some signs of platonisation, so that the observed effect is presumably due to the lead present. There was no difference in the ballistic activity of either cobaltous or cobaltic oxides.

Antimony trioxide was tried at two levels in EU in place of white lead without success (F547/481 and F547/473). The addition of litharge to F547/481 resulted in an incipient plateau (F547/482).

None of these metal oxides was sufficiently promising to justify further work.

Toughening of Sheet Propellant CONFIDENTIAL

RPE consider that the high temperature performance of the Crake motor could be improved if the mechanical properties of the 1.5 mm thick sheet LU could be improved. Further work has been conducted with the acetone/alcohol vapour treatment. A solvent uptake of 5 to 30 per cent was obtained with 1.2 to 1.5 mm sheet. The high solvent levels gave distortion after drying but in the range 5 to 25 per cent of solvent, the final sheet thickness, tensile strength, and elongation were increased very approximately by 1, 4 and 9 per cent respectively for each one per cent of solvent absorbed initially. Strand burning tests showed that the treatment had little or no effect on burning rate. Test motor assemblies treated similarly have shown cockling of the propellant, and propellant sheets treated to about 16 per cent solvent level have been supplied for motor assembly at RPE. In a further attempt to increase tensile strength without interfering with ballistic properties, one per cent of carbon fibres, 12 mm long, was incorporated into LU paste (without dixigloss) which was subsequently given the normal solventless hot rolling. Tensile test pieces, stamped from the sheet, showed no change in tensile strength but elongations were slightly reduced; Young's modulus was increased by about 10 per cent. Microscopic examination revealed that the fibres had been broken down to a mean size of 100 μm and they were aligned in the direction of rolling. Strand burning rates are awaited, but this approach shows little promise for platonised solventless cordite.

(iii) Cast Double-Base Propellant

Support of CDB Projects

The available staff have been concentrated on this item in view of the difficulties being experienced.

The Retriever Crisis RESTRICTED

ERDE senior staff have been involved in a large number of meetings to assist in explaining the causes of the abnormal performance from some stored "super-dry" Retriever boosts. Suitable action to establish the Retriever motor on a satisfactory basis has been recommended on the lines of the earlier ERDE work (TR 1/70/1) which linked ballistic drift of ATN(D28)/47 with both initial water content and water absorption. The special work conducted to provide basic information on problems likely to arise from the use of very dry casting powder and to allay MOD(N) suspicions on the basic reliability of correctly manufactured CDB has been reported (ERDE Memorandum 31).

Modified Plant and Procedures for Retriever Manufacture RESTRICTED

The thorough revision of Specifications for Retriever and liaison in connection with the "Get Well" programme has called for a major ERDE effort. Maximum water contents of 0.25 per cent for casting powder and 0.20 per cent for casting liquid have been accepted largely because IMI consider they cannot achieve lower levels consistently. (It is ERDE experience that their proposed water levels can be achieved when there is pre-evacuation before casting.) The reported ERDE work suggests that on prolonged storage at ambient conditions, ballistic drift of 5 to 10 per cent could be obtained using the maximum permitted VM levels.

To provide background information on the air conditioning levels required for processing at IMI, the equilibrium water contents (%) at various relative humidities have been determined on carefully pre-dried materials:

RH at 18°C	22%	32%	45%	55%	80%
Water in Powder	0.25	0.35	0.47	0.59	0.95
Water in Propellant	0.15	0.25	0.38	0.43	0.78

Some days are required for equilibrium to be attained, even with thin propellant samples, and years would be required in actual Retriever motors. It is quite clear that ambient humidities during storage and processing of casting powder should be as low as possible and the aim should be under 30% RH (in the absence of pre-evacuation before casting at IMI).

It is now generally agreed that the ballistic drift found with motor No 1862 was due to water being left in the motor after removal from the water tank used for ultrasonic testing. IMI has introduced a draining and air drying procedure. Observations there showed that the draining and swabbing out of

Retrievers leaves the surface covered with drops of water. Simulation tests with K-rounds indicated that no serious effects are to be expected with Retriever if the hot air drying procedure is strictly controlled.

Humidity Indicators and Desiccant Devices for Retriever RESTRICTED

Consideration has been given at many meetings to the problem of using desiccants, and to sealing. ERDE has examined effects of location of desiccant and the possibility of finding indicating papers which might record the effective exhaustion of desiccant or the equilibrium RH present in the conduit. Three commercial cobalt salt papers have been assessed. They do not show high precision and it is dubious whether the claimed $\pm$ 5% is achieved, particularly in the RH region (20 - 40%) required, where colours tend to be a rather nondescript combination of pale blue and pale lilac. A special curling type test paper is promising, but has not yet been fully checked for reproducibility. It might be suitable for individual boxes of casting powder; after six weeks ambient storage with powder which gave slight yellowing, discs of curling paper continue to discriminate between powder at 22, 32 and 45% RH.

All these indicators, and also coloured silica gel, became discoloured on prolonged storage over ATN(D28)/47 propellant, depending on the degree of proximity. The original colours became overlaid with a yellowish-brown stain, possibly of sublimed stabiliser giving an overall greenish-brown appearance not clearly related to relative humidity. However silica gel protects the papers from this discoloration even after several weeks at 60°C, sufficient to simulate at least five years at 20°C. Nitroglycerine pick-up by silica gel and molecular sieve NC 4A are about 2 and 1% respectively after storage for 2 months at 60°C close to ATN(D28)/47.

A comprehensive note has been circulated to DGW(N), DGW(X) and others concerned, covering this work, as requested by the Retriever "Get Well Committee".

Re-selection of Dubious Retriever Motors for Range Trials CONFIDENTIAL

An urgent request to ERDE to advise on this subject has led to considerable correspondence. A small active group representing ERDE/IMI/QAD(N) is undertaking the selection and sampling of motors to enable the likely change in ballistics to be judged. Microradiography is to be carried out on samples, and attempts are being made to measure the relative humidities within the motors.

Improved Boost Propallent CONFIDENTIAL

In view of the difficulties being experienced by IMI with ATN(D28)/47 increased priority has been given to devising an improved composition. Ballistically this is a difficult problem as ATN represents many years development and also the structure and nature of CDB tends to lead to lower rates and poorer platonisation than equivalent extruded formulations. Attempts have been made to apply some of the advances in the letter to CDB and the results were made available to ICI/IMI in September. The systems which have been investigated include basic cupric salicylate/lead beta resorcylate, lead stannate/Ukarb 340 and lead phthalate/Dixigloss.

When processed normally as casting powder, only basic cupric salicylate/lead beta resorcylate has given well platonised CDB. K-motor firings have given plateaux or mesa burning rates ranging from 26.5 mm/s (between 15 and 20 MN/m^2) to about 32 mm/s (between 20 and 30 MN/m^2) depending on the amount of Dixigloss carbon black added. It is clear from the burning rate curves that significantly faster platonised rates will be obtained from similar formulations now being made containing still larger quantities of carbon black. The lower rate quoted above (26.5 mm/s) is obtained when no carbon black is present and the possibility of lowering the rate to the ATN(D28)/47 level (23 mm/s) by changes in the ratio of the ballistic modifiers and by omitting resorcinol is being explored.

Neither lead phthalate/Dixigloss nor lead stannate gave useful platonisation when normally processed casting powder was used. However, when casting powder prepared by solvent reworking of solventless rolled sheet was used the former gave plateau ballistics at around 20 mm/s between 6 and 10 MN/m^2 and this work is being extended. Similarly when this rather more complex processing was used for lead stannate casting powders, platonisation was achieved at around 17 mm/s at pressures between 9 and about 12 MN/m^2.

The more promising compositions are being subjected to testing for ballistic drift.

Composite Modified CDB

Use of CHOW CONFIDENTIAL

In connection with the CHOW development ERDE has reviewed SRS report 70/3 on CMCDB, in which variable storage results are reported for gassing/separation at the case bonded surfaces under accelerated test conditions. Test charges have also shown variable properties and a number of charges have been manufactured in which porosity (voids) is present initially or develops in a few weeks at 30°C. ERDE consider that obvious porosity is not acceptable in Service motors although IMI state it is tolerable.

CHOW "Keep Fit" Programme CONFIDENTIAL

DGW(X) requested ERDE to advise on the work required on CMCDB for CHOW. The technical aspects are to be agreed at the CDB Working Party and a preliminary ERDE note outlining the work required, mainly at ARDEER and IMI, has been discussed. Owing to staff limitations the ERDE contribution must be small.

ERDE considers that the low absolute density of production casting powder (95% of theory) is a major factor. A sample of a typical production lot of casting powder W86/89, when cast using ERDE procedures, gave soft castings owing to a low powder/liquid ratio of about 70/30. However the cracking test results on 50 mm cubes at 80°C were normal at nine to eleven days. No early porosity was observed and it appears that poor non-reproducible castings reported by IMI must be attributed to inadequate casting procedures for low density casting powders in large motor configuration.

Special Stabilisers for CMCDB CONFIDENTIAL

In normal CMCDB a special stabiliser greatly increases the resistance to high-temperature cracking. A complex replacement containing NMA, white lead and picrite has also been found to give similar results. Further work has been conducted with CMCDB compositions containing RDX, which received some development for the COMSAT application. The results show that even when the special stabiliser is present, cracking is much more rapid with this type of composition than with normal CMCDB.

No improvement in cracking life has been achieved by use of large amounts of conventional stabilisers such as NMA or NDPA nor by the use of a modification of the replacement stabiliser.

(iv) Gun Applications

Processing Techniques for Combustible Cartridge Cases RESTRICTED

The process modifications, involving higher temperature and longer time of pressing, devised to bring the ballistic properties of Bishopton manufactured cases more nearly into line with ERDE manufactured cases, have been introduced. The results of RARDE Trial B2(B) 450/70 showed that certain differences still persist. Slightly higher charge weights are required with Bishopton cases but the most significant difference was in firing intervals, where the ERDE cases showed a 14 millisecond advantage over the Bishopton cases, and 18 milliseconds over the bag charge. Analysis for solvent content and TVM indicate very slightly higher values for the Bishopton cases, which may account for some of the difference in firing interval. It is understood that the differences between the Bishopton and ERDE cases can be accommodated in Service, provided the ballistic level of the production cases remains constant.

Combustible Cartridge Cases: Research Aspects CONFIDENTIAL

The use of full length combustible cases in the 155 mm FH 70 gun presents considerable problems at the lowest charge weight, as operating pressures are of the order of 30 to 40 MN/m^2. To minimise these problems a small bottom charge container with screwed lid was devised for charges 1 and 2 which with an added extension piece would give the full length case. Sample cases were manufactured with an NC/Kraft ratio of 6/1 and 8 per cent resin, and supplied to RARDE for firing trials with a number of alternative ballistically small propellants. Although the firing results showed little evidence of debris, there was some black or white smoke, depending on the propellant used. Further modification of the container design, propellant or igniter may be necessary.

Experimental work has confirmed that replacement of the nitroglycerine in the "Service" water-proofing nitrocellulose lacquer, (Patent Application No 30953/69) by equal weight of either diethylene glycol dinitrate or triethylene glycol dinitrate does not change the water resistance of lacquered combustible cases. Samples of films of these alternative lacquers have been stored at 70°C for four weeks and retain their tensile strength similarly to the NG type.

Computerised Travelling Microscope for Granule Measurement RESTRICTED

The measurement of multi-perforated granules using a travelling microscope is somewhat tedious and time consuming in recording and making the required calculations. The Instruments section has devised a system involving a displacement transducer, electronic register and on-line Teletyper which produces data tapes which can be fed to a computer to give the required numerical records. First comparisons with purely manual assessment show marked savings in labour.

Low Temperature Correction of Low Erosion Gun Propellants

Assessment of F527/333 CONFIDENTIAL

Over the last two years, promising reductions in temperature corrections for pressure have been obtained in the higher temperature range in limited trials with F527/333 composition. This is based upon pyro-nitrocellulose (12.2% NC) with a similar force to NQ but an energy level about 250 kJ/kg higher and consequently containing 2 per cent titanium dioxide to minimise gun erosion. Large experimental batches have been processed in the 7-hole form for the 105 mm gun and as slotted tube for the 120 mm gun for firings at 21° and 52°C.

Also, to compare the relative merits of titanium dioxide and talc, in the 105 mm gun, large batches of experimental propellant F527/354 were made to the same chemical composition as F527/333 but with fine and coarse grades of talc replacing titanium dioxide.

The results obtained are summarised in Table 2.

TABLE 2

Equipment	Propellant	Variant	Temperature Correction for 10°C		Gun Erosion
			Pressure	Velocity	
105 mm	NQ (Service)		(27.8 MN/m)	(18.1 m/s)	-
	F527/333		37% Reduction	20% Reduction	55% of NQ
	F527/354	Fine Talc	35% Reduction	Similar to NQ	55% of NQ
	F527/354	Coarse Talc	27% Reduction	Similar to NQ	25% of NQ
120 mm	F527/333		37% Reduction over NQ	Similar to NQ	50% of NQ

The F527/333 composition has given reductions in pressure correction of about 35 per cent in both the 105 mm and 120 mm guns but the effect on the velocity correction is small. Replacement of fine titanium dioxide by fine talc gives similar results. The new composition with fine titanium dioxide or talc reduces gun wear to about half that of NQ. Coarse talc is less effective in reducing temperature corrections and gun erosion. In view of these results, effort has been devoted to optimising composition F527/333.

Optimisation of F527/333 Type Compositions and Granule Size — CONFIDENTIAL

The colloidal matrix of F527/333 is much hotter than NQ and if only one per cent of titanium dioxide were adequate, a significantly cooler propellant would result. Batches of two new compositions differing only in stabiliser have been made in seven-hole granular form, containing only one per cent of titanium dioxide, for firing in the 105 mm tank gun. The reduction in the proportions of the additive makes it possible to reduce the calorimetric value of the propellant by about 168 kJ/kg without any reduction in force relative to F527/333.

To enable a number of compositional effects to be assessed fairly rapidly, firings are being carried out by RARDE in the 30 mm RARDEN gun. The first compositions have been made in single-hole chopped tube, 2.5 and 5 mm long together with control NQ. The F527/333 composition in the 2.5 mm length gave a reduction of nearly a third and a half in the velocity and pressure corrections respectively compared with the NQ composition, so it appears that representative comparative results will be obtained. Replacement of carbamite by 2-NDPA as stabiliser gave poorer temperature corrections. The effect of cut length was considerable and confirmed earlier results on this factor. The results to date show that a considerable programme with various granule dimensions may be required to optimise temperature correction in the larger guns.

A batch of F527/333 with a novel cogged 7-hole granule, with different burning thicknesses and intended to function as a composite charge, proved to be slightly fast in the 105 mm gun, although the Closed Vessel had indicated that its vivacity was only 80 per cent relative to the usual 7-hole granule.

(v) Assessment

Ordnance Board Trials and FDS

68 mm SNEB Aircraft Rocket — RESTRICTED

Recently, three rounds which had failed due to electrical faults during practice firings from aircraft were subjected to static firing tests at 70°C. Although the rounds were from different lots and contained different batches of propellant, all burst immediately after ignition. Examination of a piece of propellant debris supplied to ERDE indicated an internal crack pattern characteristic of gas cracking.

Storage tests showed that the SNEB charge (TT 17 propellant) can be made to produce a very similar gas-cracking pattern if heated to the neighbourhood of 90°C for 24 hours.

Giant Viper (OB Procs 40628, 40947) RESTRICTED

This OB trial was for six months at 50°C; an additional charge has been stored at ERDE for up to 16½ months to give supplementary information. Some of the stored motors during the OB trial gave unacceptably large (± 8 per cent) variations in total impulse and the OB decided that a re-design was required. Our examination of the EU charge and inhibition failed to indicate changes of the required magnitude. Burning rate changes were small after even 16½ months at 50°C. Total energy of the propellant as determined by calorimetric values was 2863, 2863 and 2887 kJ/kg, after 0, 6 and 16½ months storage and clearly did not account for the changes in total impulse of the stored motors. No major chemical or physical changes occurred in the propellant after six months and the integrity of the charge and also the inhibition were regarded as satisfactory after 16½ months at 50°C.

ERDE considers that some variability in total impulse may be due to instrumentation or to factors such as weight or variable solvent retention during inhibition and is obtaining further information.

French Weapons SS11 and AS12 CONFIDENTIAL

Three separate limited storage trials have been proceeding to provide reliable information on the service life of the SS11. Failures appear to arise from inhibition burn-through rather than unbonding as originally expected and the temperature coefficient for NG migration (2.0 for 10°C) is regarded as relevant. The OB trial for 15 months at 50°C suggested a life of 6¾ years at 27°C.

Motors returned from Borneo were fired successfully after nine months at 40°C giving a life of over six years at 27°C but one of the two failed after 18 months at 40°C (estimated to be equivalent to 8/9 years at 27°C).

Motors stored for 7½ years in unconditioned storage at Boscombe Down are being stored at 40°C. Two were fired satisfactorily after 18 months at 40°C and correspond to total storage equivalent to 6½ years at 27°C.

Examination of the inhibitor residue after firing has been very helpful as it has been possible to make assessments on the inhibiting capacity remaining.

Representative AS12 charges have been examined after storage for up to nine months at 50°C as part of a 12 month trial.

Deerhound RESTRICTED

A motor failure after five years storage in the Natural life trial has lead to considerable activity and involved examining representative motors which had been stored under Service conditions and the devising of trials to determine what residual storage life there was still remaining.

Charge Inspection RESTRICTED

Radiographic examination has been carried out for IMI on 10 Chow rod charges which had been subjected to various storage treatments.

Ultrasonic testing has been conducted on ATN(D28)/47 cast propellant prepared from well dried ingredients as IMI had reported very high attenuation with Retriever motors manufactured under the new dry processing conditions so that they could not be inspected by ultrasonics. ERDE measurements on 12.5 mm thick samples of IMI propellants indicated that attenuation was actually lower with super-dry propellant whilst further tests through 150 mm lengths of ERDE processed propellants indicated little or no difference. It is now accepted that propellant attenuation was not the cause of the difficulty which has now been resolved.

To extend our capability for inspecting production motors, the ultrasonic tank has been increased in length to 5 m and new transducers prepared so that the scanning length is increased from 0.9 to 1.5 m. A new rotation frame assembly is to be installed to facilitate inspection.

Thermal Cycling Equipment UNCLASSIFIED

A fully automated cycling equipment has been installed to minimise labour usage in cycling trials. It can be programmed to operate automatically between two set temperatures within the limits -60°C to +60°C with or without an intermediate dwell at a set temperature representative, say, of ambient conditions. Dwell times at the set temperatures can be varied up to a limit of about four days. The rate of change of temperature between dwells is linear and can be varied, in 5 stages between 2°C/hour and 11°C/hour. The unit is capable of operating between the extreme limits with a load having a thermal capacity of 225 kJ per °C. The test cabinet, which has been designed to be suitable for storage of propellants, measures 2 ft by 2 ft by 6 ft long and each of its two shelves is capable of carrying a load of 250 lb. The "running-in" tests presented a number of problems but shortly it will be taken into use.

Quality Assurance Assistance UNCLASSIFIED

A new lot of SC cordite is being established as a calorimetric and strand burning standard.

The Smith bridge to be installed in a new calorimetric unit by DQA(Mats) is being tested and serviced.

Simplified Ballistic Testing of CDB RESTRICTED

Further modifications have been made to the high capacity strand burner now under development for testing CDB more economically. Owing to the temperature control provided by an externally mounted cooling coil proving inadequate, the apparatus has been re-designed not only to provide andimproved temperature control but also to give (i) simplified venting arrangements (ii) remote operation and (iii) access for the removal of excess condensation products within the system. More accurate assessment of the average pressure during firing will be achieved from a digital instrumentation channel. Comparative burning rate tests are

being carried out on a representative slow burning composition with a burning rate of about 6.2 mm/s at 7 MN/m^2 as square strands between 3 and 25 mm side and as 12.5 to 152 mm diameter end-burning charges. On the basis of successful firings with 51 mm diameter charges 2.5 mm silicone rubber inhibition has been standardised for all sizes above 25 mm; partial failures were obtained with 1.25 and 1.9 mm inhibition on 25 and 151 mm diameter charges.

(b) COMPOSITE PROPELLANTS - PLASTIC PROPELLANT

Effect of Deaeration on the Burning Rate of Plastic Propellant RESTRICTED

The categorisation of plastic propellants for safety distance is largely decided on the basis of the behaviour of charges ignited under confinement in steel tubes using a test known as the large sealed vessel test or LSVT. In this test two types of ignition are used: the charge is either heated externally to ignition or it is ignited internally by a conventional SR 371C carton igniter. Afteriignition, the appearance of the tube, or tube fragments, is used to classify the result as a pressure burst, a low order explosion, an explosion, or a detonation. If both types of ignition give a pressure burst the propellant is classified as Category Y (no risk of mass explosion); if an explosion or detonation is obtained in both cases the propellant is Category ZZ (mass explosion risk with minor missile effect); if one type of ignition gives a pressure burst and the other an explosion, other tests must be used to decide the safety distance category.

With certain plastic propellant compositions difficulty has arisen with the LSVT because the results obtained depend to some extent on the technique used for filling the steel vessels and, to a greater extent, on whether or not the propellant is deaerated before it is tested (the undeaerated material always gives the more violent result). It has therefore been decided to carry out bonfire trials on various plastic compositions packed in boxes so as to obtain a more direct assessment of safety distance category. In the meantime, it seems that certain compositions must be treated as Category ZZ before deaeration and as Category Y after deaeration.

It is normal practice to deaerate plastic propellant immediately before it is filled into rocket motors because the deaeration process, which involves passing heated material through a vacuum pugmill, has a softening effect on the propellant which greatly facilitates charge pressing. Consequently, the propellant is normally stored in an undeaerated condition. However, if it is one of those compositions with a higher safety distance category before deaeration, it obviously would be advantageous to deaerate before storage to avoid the necessity for additional precautions. A difficulty with this procedure is that the material would have to be deaerated again before filling into motors, and it is possible that the double deaeration might affect the burning rate because of change of oxidant particle size.

To ascertain the effect of double deaeration on burning rate, 100 kg batches of three different propellant compositions were made and the burning rate of each composition was measured both by strand burner and K-round firings (a) before deaeration, (b) after deaeration by a single pass through the pugmill, and

(c) after double deaeration. With the undeaerated materials, great care was taken to obtain strands and motor fillings which were as free from voids as possible.

The propellants chosen were RD 2423, RD 2427 and RD 2428 which cover a wide range of ammonium perchlorate particle sizes and binder contents and also contain most of the other solid constituents used in plastic propellants. The compositions of these propellants are:

RD 2423 : 84.5% ammonium perchlorate of 30 μm average particle size, 2% copper chromate, 13.5% PSB1 binder.

RD 2427 : 42% ammonium perchlorate of 7 μm average particle size, 38% ammonium picrate, 5% aluminium powder, 2% oxamide, 13% PBS1.

RD 2428 : 74% ammonium perchlorate of 12.5 μm average particle size, 14% aluminium powder, 1% copper chromate, 11% PSB1.

With all three compositions, double deaeration had no measurable effect on burning rate over pressure range 4 to 10 MN/m^2 and it is concluded that deaeration prior to storage of propellant will not introduce additional problems of ballistic control.

Measurement of Particle Size of Fine Ammonium Perchlorate UNCLASSIFIED

In the last Technical Report, details were given of the use of ammonium perchlorate milled to an average size of 2 to 3 μm for obtaining compositions of high burning rate. Further work has been carried out with ammonium perchlorate milled to this size in a 100 mm diameter Micronizer and the installation of a larger mill for producing 25 kg/hr of very fine oxidant should be completed early in 1971.

A difficulty which has been encountered with the use of 2 μm material concerns the measurement of particle size. At ERDE, the normal method of determining the average particle size of ammonium perchlorate is by measuring the rate of flow of air under known pressure through a bed of powder compacted under standardised conditions. This method was adopted several years ago after investigating most of the other methods then available. It gives reliable and reproducible results for ammonium perchlorate in the size range of 5 to 30 μm, which covered the grades of milled oxidant in use at that time, but erratic results were obtained with both finer and coarser grades, possible due to non-uniformity in the compacted bed of material.

In the USA, where very fine ammonium perchlorate has been used by the propellants industry for several years, an intensive investigation of methods of measuring particle size resulted in the conclusion that for ammonium perchlorate finer than about 3 μm average size, the most reproducible results are given by centrifugal sedimentation using a Mines Safety Appliances (MSA) analyser. An MSA apparatus is being purchased by ERDE, but delivery is not expected until mid-1971 and it was therefore decided to investigate an alternative method based on gas adsorption.

Although gas adsorption methods have been used for measuring the surface areas (and hence the particle sizes) of very fine powders for many years, they are not normally suitable for routine control purposes because they are very time consuming and require highly skilled operatives. However, a method which avoids the use of high vacuum apparatus by measuring gas adsorption in a flow system was described by Nelson and Eggersen. This method is not sufficiently sensitive for the measurement of particle sizes coarser than about 3 μm, whereas it would be advantageous to have a single method suitable for all milled grades of ammonium perchlorate. Furthermore, the literature mentions several other disadvantages of the flow system method, connected with the accurate metering and flow control of the adsorbate and carrier gas. Nevertheless, it seemed there was scope for improving the method, particularly by using the precision mass flow controllers which have now been developed for temperature programmed gas chromatography.

The Nelson and Eggersen method, in brief, is to mix an adsorbate (for example, nitrogen) with a carrier gas (usually helium) and pass the mixture over the degassed powder sample. Adsorption takes place when the temperature of the sample is lowered to a temperature approaching the liquefaction point of the adsorpate, and desorption takes place when the temperature is subsequently raised. The level of nitrogen in the efflux gas in monitored continuously, for example with a thermal conductivity cell, and several desorption curves are obtained by repeating the experiment with different partial pressures of adsorbate. From the areas under the desorption curves, the surface area of the powder can be calculated using an equation due to Brunauer, Emmett and Teller.

Using precision mass flow controllers and monitoring the level of nitrogen in helium carrier gas with a gas chromatographic katharometer, it is possible to improve the sensitivity of the method so that the particle size of ammonium perchlorate as coarse as 30 μm can be measured reproducibly. Furthermore, the stability of the apparatus is such that the gas composition and flow rate remain constant over long periods.

The gas adsorption method always gave small particle size values by a factor of two to three, but it is by no means unusual for different methods of particle size measurement to differ to this extent and the gas adsorption method would be expected to give the lower values because presumably the entire surface, including that exposed by cracks and fissures, is measured. A comparison of the reproducibilities of the two methods applied to ammonium perchlorate and other powders finer than 2 μm average size is also being made, but results are not yet available.

Work in support of FOIL

Suitability of Plastic Propellant for FOIL CONFIDENTIAL

The parametric study carried out jointly by RARDE and HEL has indicated that the probable calibre of the motor required for FOIL will be 264 mm using a case-bonded star-centred charge. It was at first considered that the optimised solution would require a burning rate in the range 56 to 68 mm/s

at 28 MN/m^2 but although propellant studies were commenced with this range in view, a wider span of compositions was examined down to 40 mm/s at 28 MN/m^2. A recent reappraisal has indicated that the most likely range will be 50 to 56 mm/s.

In comparison with other propellants, plastic propellant offers many advantages for a motor of this type. It is inexpensive, has high specific impulse, can readily be obtained with the required burning rate, shows good ballistic reproducibility, storage life and vibration resistance, has a low temperature coefficient of burning rate, and, as has been demonstrated with Pipit motors, it will withstand acceleration environment. The main disadvantage of plastic propellant is concerned with its low-temperature behaviour, in particular its poor resistance at low temperatures to the very rapid straining imposed by ignition pressurisation of the rocket motor. During the past year, therefore, much of the effort on plastic propellant has been devoted to the characterisation and improvement of mechanical behaviour under rapid straining at low temperature.

Characterisation of Low-Temperature Behaviour CONFIDENTIAL

It has been previously reported that predicted failure temperatures (PFT's) of -32° to $-35^{\circ}C$ have been determined for RD 2428 in Pipit motors by evaluating the failure point of a characteristic extension of 5.3 per cent at a rate of strain of 160 min^{-1} using the extensibility master curve. These are the strain and strain rate expected for a Pipit motor on pressurisation. Four static firings of Pipit motors have taken place successfully at $-30^{\circ}C$. One motor ignited at $-40^{\circ}C$ burst violently indicating that the charge had cracked badly $5^{\circ}C$ below the lowest failure temperature predicted.

Comparison with Other Propellants CONFIDENTIAL

Master curves have been obtained for various types of propellant (plastic, CTPB, CMCDB and a modified plastic based on non-functional polybutadiene binder). From these, curves have been derived showing the relation between elongation-at-rupture and temperature at various strain rates. These depict quite clearly the superior low-temperature behaviour of CTPB in comparison with normal plastic propellant and CMCDB. For example, at a strain rate of 200 min^{-1}, the elongation at rupture of CTPB C37 at $-40^{\circ}C$ is 20 per cent, whereas for both RD 2428 and CMCDB F452/302/19 it is only 4 per cent. Furthermore, the curves for CTPB are very concave to the temperature axis at low temperatures so that appreciable extensibility is obtained only a few degrees above the brittle temperature (temperature at which extensibility is close to zero). The curves for CMCDB and plastic propellant are much flatter, or even convex to the temperature axis, the extensibility increasing only gradually with increasing temperature. The brittle temperatures for both CTPB and plastic are similar (about $-50^{\circ}C$ for a strain rate of 200 min^{-1} but very dependent on strain rate) whereas CMCDB has a higher brittle temperature (about $-42^{\circ}C$ for a strain rate of 200 min^{-1}, which is not so dependent on strain rate). The modified plastic propellant (which will be discussed later) has a very flat curve but at a high strain level so that the elongation-at-break at $-40^{\circ}C$ and a strain rate of 200 min^{-1} is about 10 per cent and the brittle temperature is below $-80^{\circ}C$.

Correlation of PFT with Motor Behaviour CONFIDENTIAL

Because of uncertainty regarding the relevance of PFT values to behaviour in a rocket motor, a trial is in progress designed to obtain a correlation between failure temperatures calculated from tensile testing and actual failure temperatures in a rocket motor. Two plastic propellant compositions (one aluminised and with 86 per cent solids, the other non-aluminised and with 85 per cent solids) have been filled into 2-inch XP motors which are being fired at progressively lower temperatures until two temperatures for each composition are found, these being the lowest temperature at which six out of six motors perform satisfactorily, and the highest temperature at which six out of six motors fail. Even with the same batch of propellant, a difference of perhaps 10°C between these two temperatures is to be expected due to variability in pressurisation strain and strain rate arising from variation in propellant surface finish and igniter characteristics. The firing records from successful firings are being used to determine, for each composition, the range of pressurisation strains and strain rates and from these, maximum and minimum values will be deduced by using $\pm$ 3 times the standard deviations. Thus, for each composition, it will be possible to compute maximum and minimum failure temperature values from tensile test data and these will be compared with the values obtained from the motor firings.

Although this trial is not yet complete, sufficient results (on 40 motor firings) have been obtained for a preliminary correlation between predicted and actual values; the agreement is very close. Thus, with the non-aluminised composition, six out of six motors functioned successfully at -42.5°C and two out of six burst at -55°C, (the temperature for complete failure has not yet been reached). The calculated failure temperatures are -41°C maximum and -59°C minimum. A similar correlation is being obtained with the aluminised composition and with a second batch of the non-aluminised composition. It can only be concluded from the results so far obtained that the effects of various factors neglected in calculating the failure temperatures largely cancel out.

Optimisation Studies CONFIDENTIAL

A programme of work is being carried out aimed at optimising a plastic propellant composition for FOIL. A range of compositions is being studied in two phases, initially to select appropriate burning rates and finally to consider physical properties, reproducibility, pressure exponent, temperature coefficient and specific impulse. The compositions cover the range of 11 to 14 per cent binder content, 7.5 to 30 μm initial average particle size of oxidant, and the use of copper chromate, silica, titanium dioxide and ferric oxide as burning rate catalysts. Each combination of binder content and catalyst is being used in conjunction with a range of oxidant particle sizes, and each of the resulting compositions is being subjected to ballistic assessment in the first phase of the programme. The compositions studied have produced results in the range of 40 mm/s to 68 mm/s and excellent correlation has been obtained for the variation of burning rate with average particle size, typical figures for the firing of E 4264 in K-rounds are shown in Table 3.

TABLE 3

Initial average particle size of oxidant (μm)	Burning rate at 28 MN/m^2 (mm/s)
30	49.1
15	57.1
10	65.6
7.5	59.8

With the most recent requirement forecast in the range of 50 to 56 mm/s this propellant is currently candidate for FOIL although its burning rate is high in the preferred range of ammonium perchlorate size.

A more detailed examination has commenced on another propellant, E 4265, which has a higher binder content (13 per cent plasticised polyisobutene). A batch prepared with ammonium perchlorate of 13.3 μm average particle size had a burning rate of 51 mm at 28 MN/m^2 and when the master curve for extensibility was constructed it showed a predicted failure temperature of -45°C giving a promising improvement compared with the -35°C predicted for RD 2428.

The systematic study of the candidate materials is continuing and it is expected that about eight compositions will be fully characterised by the end of the first phase of the programme.

Plastic Propellant Based on Non-functional Polybutadiene RESTRICTED

An attempt is being made to achieve a drastic improvement in the low temperature behaviour of plastic propellant by using binders other than polyisobutene.

As far as low temperature propellant properties are concerned, the two most important binder characteristics probably are the second order (glass) transition point and the temperature coefficient of viscosity. The former largely determines the brittle temperature of the propellant, at which extensibility becomes virtually zero, and has a controlling influence on properties well above the brittle temperature; the latter largely determines the plasto-viscosity and relaxation time (ie the flow and deformation characteristics) at low temperature. To obtain an improvement in low temperature propellant properties it is necessary to obtain liquid polymers with lower T_g's and temperature coefficients of viscosity than those of polyisobutene. The grade of polyisobutene used for plastic propellant has a T_g of -75° to -80°C and a temperature coefficient of viscosity, $\Delta \log \eta / \Delta (1/T)$, of about 3000.

A search of the literature showed there are very few commercially available polymers with lower T_g's than polyisobutene, except for polymers of butadiene which were quoted as having T_g's from -85°C to below -90°C. Four commercially available polybutadienes were obtained, Cis 4 grade 1203 (over 90 per cent cis-1,4 linkages) marketed by Phillips Petroleum Co, Intene 35S (36% cis-, 56% trans-, 8% vinyl-) marketed by International Synthetic Rubber Co, Taktene (cis-1,4) marketed by the Polymer Corporation and Cariflex BR 1202 grade marketed by Shell Chemicals Ltd.

The first two polymers are soft crepe rubbers which need considerable plasticization before they can be used as plastic propellant binders (a maximum viscosity of about 2×10^6 poise at 25°C is required). Sufficient plasticization could be obtained by mixing the polymer with, for example, isodecyl pelargonate (IDP), ethyl oleate or polybutene (probably low molecular weight polyisobutene). The polymers also had limited compatibility with CTPB (Thiokol HC 434). Both Taktene and Cariflex will compound to give suitable binders using standard plasticizers.

The second order transition temperatures of the base polymers and the plasticized materials were measured dilatometrically using ethyl alcohol as the confining liquid. The dilatometers were cooled to about -110°C in liquid nitrogen and allowed to warm at a rate of about 6°C per hour while measurements of the liquid level in the capillary were taken. With all experiments, a plot of capillary height against temperature showed a well-defined change in slope and the inflection point was taken to be the second order transition point. Some of the more interesting results are listed below:

	T_g =
PIB (propellant grade)	-75°C
PIB, 80: ethyl oleate, 20	-85°C
Cis 4 polybutadiene	-95°C
Cis 4, 50; ethyl oleate, 50	-97°C
Intene 35S	-98°C
Intene 35S, 60; ethyl oleate, 40	-105°C
Intene 35S, 50; polybutene, 50	-99°C
Intene 35S, 50; HC 434, 40; IDP, 10	-97°C
Cariflex BR 1202	-95°C

Viscosities over a range of shear rate and temperature were measured with a Rheomat 15 concentric cylinder viscometer. The viscosities of the polybutadiene materials were found to be much more dependent on shear rate than is the viscosity of PIB (for example, a 60-fold increase compared with a 20 per cent increase). At a given shear rate, however, the temperature coefficients of viscosity were much lower for the polybutadienes than for PIB (for example, 820 compared with 3000).

Attempts to make plastic propellants with Intene 35S plasticized with polybutene were not very successful, the mix remaining as small non-coherent granules despite prolonged mixing. Better results were obtained using the mixture of Intene 35S, HC 434 and IDP (designated B615). Coherent compositions were made containing 12 to 15 per cent of B615 as binder with either lecithin or S101 as surface-active agent. These compositions had about half the extensibility

of normal plastic propellant at 25°C (for example, 44 per cent compared with 80 per cent) but extensibilities at low temperatures were very much higher than for normal plastic propellant. Even at -80°C, using the highest strain rate available with the Instron tester (28 cm min^{-1}), an extensibility of 8 per cent was obtained. Predicted failure temperatures under FFR2 conditions (5.3 per cent strain, 160 min^{-1} strain rate) ranged from -70° to -77°C, an improvement of about 30°C in comparison with normal plastic propellant.

A further series of compositions was prepared by blending together two propellants, one based on the binder designated B615 and the other based on plasticised polyisobutene. The predicted failure temperatures for compositions containing 100, 75, 60 and 0 per cent B615 were -76°C, -42°C, -34°C and -46°C respectively indicating that the increase observed in the room temperature extensibility on adding polyisobutene was accompanied by a deterioration in the low-temperature properties.

Initial results using Cariflex BR 1202 and Taktene have not shown any advantage in spite of the low glass transition temperature of the former. A formulation containing 14 per cent of Cariflex gave a predicted failure temperature of -13°C whilst with Taktene the value was about 0°C. It is possible that this poorer behaviour is associated with the fact that both these compounds are of the cis-1,4 type. The lower strain capabilities at high temperatures shown by the polybutadiene materials appear to be due to a reduced level of "cohesiveness" ("tack","spinbarkeit") in comparison with PIB.

Propellants for Raven RESTRICTED

The propellant used for the Raven motor is RD 2427 which contains unmilled ammonium perchlorate and 2 per cent of oxamide. This composition is platonised in the pressure range 7 to 10 MN/m^2 but current operating pressures are 3 to 4 MN/m^2 for Raven VIII and 6 to 7 MN/m^2 for Raven VI so that the platonisation is not utilised.

The ballistic control of RD 2427, based on strand burner and K-round measurements, has not been entirely satisfactory. Motors within a batch have shown variation in burning time and thrust-time characteristics, and nozzle sizes predicted from quality control results have not always given the expected burning time. The quality control results themselves have shown high variability and the propellant burning rate has been found sensitive to change in the source of oxidant and to any variation in manufacturing procedure.

These difficulties in ballistic control are not characteristic of faster burning compositions and seem to be associated with the use of oxamide and coarse ammonium perchlorate. In larger quantities oxamide is known to give extremely variable ballistics while coarse ammonium perchlorate is subject to considerable attrition during the mixing process so that variability in burning rate is to be expected. It would seem that the reproducibility of Raven propellant might be improved by the elimination of both coarse oxidant and oxamide. However, the elimination of oxamide would change the propellant density and specific impulse and hence the motor performance. It was therefore decided first to investigate the possibility of obtaining improved reproducibility by replacement of coarse oxidant only.

When the coarse oxidant in RD 2427 was replaced by oxidant milled to an average size of 30 μm, the platonisation characteristics disappeared but the burning rate at 7 MN/m^2 was unchanged at 4.0 mm/s. Trials carried out with this propellant (designated E 4040) in K-rounds, J-rounds and 5-inch diameter star-centred charge motors, demonstrated that satisfactory pressure-time records were obtained with no sign of combustion instability. Propellant manufacturing trials were then carried out to assess the effect of major changes in mixing conditions and oxidant source on burning rate. Four lots of propellant were manufactured using two oxidant sources (Sweden and ROF Bishopton) and two types of incorporator with very different mixing characteristics (Imperial and Heavy Duty). Each lot was blended by strand burner control to give burning rates as close as possible to 4.02 mm/s at 7 MN/m^2 (actual rates were 4.04, 3.97, 4.04 and 4.02). The coefficient of variance of strand burner results for each lot was found to be less than 1.5 per cent compared with a usual variance of 2.5 per cent for RD 2427.

Each lot of propellant was used for filling K-rounds, J-rounds and 5-inch star-centred motors at ERDE, and 5-inch and 10-inch motors at RPE. The strand burning, K-round and J-round results showed excellent correlation and all could be superimposed on a single plot of log pressure against log burning rate. The 5-inch motors gave results which deviated appreciably from the others and gave indications of erosive burning. However, RPE has found that if corrections are made for erosive burning, the 5-inch motor results are in good agreement with the others. Results for the 10-inch motors have not yet been reported.

Two further 1300 kg lots of E 4040 have now been made and have been delivered to RPE for assessment in full-size Raven motors.

CTPB Propellants

Ageing Behaviour of CTPB Propellants RESTRICTED

Much of the research effort on CTPB propellants is directed towards assessing and improving ageing behaviour. This work has been in progress for several years and data are now available indicating the effect of propellant composition and environmental conditions on the changes in propellant physical properties which occur during storage.

As far as propellant composition is concerned, two main types are being examined. Both use HC 434 as the binder prepolymer, but one type is cured with a mixture of methyl aziridinyl phosphine oxide (MAPO) and ERL 0510, a commercial trifunctional epoxide, whereas the other type has an all-epoxide cure obtained by using a mixture of ERL 0510 and difunctional DER 332. The mixed aziridine/epoxide cure can result in excellent propellant physical properties at low temperatures, but the aziridine cross-links are susceptible to hydrolytic and thermal degradation; the all-epoxide cure gives more stable cross-links but possibly at the expense of hardening on storage due to post-curing, and somewhat inferior low-temperature properties.

Several other compositional factors also influence physical properties and ageing behaviour; these include ratio of cross-linking agents, level of curative, presence or absence of cure catalyst and nature of solids, size and size distribution of solids. Early in this work, therefore, it was necessary to carry out formulation studies and small-scale manufacture to establish compositions with both types of curative which were optimised as closely as possible for mechanical properties and resistance to ageing. The resulting "optimised" compositions were then subjected to extensive storage trials under various environmental conditions.

With the mixed MAPO/ERL 0510 cure, the optimised composition consists of 79 per cent ammonium perchlorate (50 : 50 mixture of 300 and 30 μm average particle size), 5 per cent aluminium powder (15 μm average particle size), 2 per cent isodecyl pelargonate plasticizer, 14 per cent binder (HC 434, MAPO, ERL 0510 in 1 : 0.75 : 0.25 : equivalents ratio) and 0.1 per cent added iron linoleate curing catalyst. Samples of this propellant were cast into metal moulds which were sealed and stored at 60°C for one year. At intervals during storage, samples were withdrawn and uniaxial tensile properties measured at 60°C, 25°C, -40°C and -55°C. After storage for one year there was no significant change in physical properties apart from a slight increase in strength and modulus at 25°C.

A similar trial was carried out with the same propellant exposed to the atmosphere. Under these conditions, there was marked degradation after one year at 60°C; the strength and modulus measured at 60°C fell to one-fifth the original values and after extension the test pieces showed poor elastic recovery. This degradation, which also has been observed in rocket motor storage trials, is believed to be due to attack by atmospheric moisture. To check this point, further samples of the same composition were stored at 60°C in moderately sealed metal boxes containing molecular sieve desiccant so that the material was exposed to air under dry conditions. In this case, the strength and modulus increased during storage (for example, measurements at 25°C showed both maximum tensile stress and modulus to increase to twice the initial values after one year at 60°C), but the tensile extensibility was substantially unchanged. Trials are now in progress to determine the effect of the same storage conditions on constant strain capability which is believed to be more relevant to behaviour of a propellant charge on storage in a rocket motor. The observed hardening on exposure to dry air may be due to additional cross-linking resulting from the attack of atmospheric oxygen at the polymer double bonds.

The results so far obtained on the optimised MAPO/ERL 0510 composition confirm the conclusion already drawn from the results of rocket motor storage trials that it is necessary to seal rocket motors containing this type of propellant so as to prevent the ingress of atmospheric oxygen and water. With carefully sealed motors, the propellant physical properties will remain virtually unchanged after storage for at least one year at 60°C. If the efficacy of sealing is in doubt, then desiccant should be used to prevent softening of the propellant although under these conditions some surface hardening is to be expected at temperatures above 40°C. With the all-epoxy cure, preliminary storage trials in sealed moulds indicated the optimum binder composition

for resistance to ageing as a 1.0 : 0.4 : 0.6 ratio of HC 434 : ERL 0510 : DER 332 used in conjunction with 0.07 per cent chromium octanoate as catalyst. Propellant based on this binder has been made on the 50 kg batch scale and storage trials similar to those carried out on MAPO/ERL 0510 propellant are now in progress. So far, results for only 12 weeks of storage at 60°C have been obtained, but these show that the propellant hardens progressively in sealed moulds and the hardening effect is accelerated by exposure to air. This type of propellant always contains unreacted carboxyl, and attempts are being made to obtain a more stable system by using an excess of epoxide and by using other epoxides.

High Burning Rate CTPB Propellants RESTRICTED

Work has continued on increasing the burning rate of CTPB compositions. The first phase of the work is directed towards obtaining the fastest burning rates that can be achieved within the limits set by the present large-scale manufacturing facilities at ERDE. This means using perchlorate ground with a KEK mill, and hence no finer than 7 μm average size, and mix viscosities not higher than 6000 poise at 60°C one hour after completion of mixing.

Solid Catalysts RESTRICTED

The effect on burning rate of various solid catalysts at the one per cent level is being determined using a composition consisting of 80 per cent ammonium perchlorate, 5 per cent aluminium powder, 12 per cent HC 434 rubber, 2 per cent isodecyl pelargonate plasticizer, 1 per cent solid burning rate catalyst and 0.1 per cent (added) iron linoleate curing catalyst. The ammonium perchlorate has a trimodal size distribution of 35 per cent 7 μm, 55 per cent 60 μm and 10 per cent unmilled. With 1 per cent BDH ferric oxide, the burning rate catalyst usually used at ERDE, this particle size distribution gives a burning rate of 15 mm/s at 7 MN/m² and a mix viscosity of 5000 poise. Both faster and slower burning rates can be obtained with the same composition by changing the particle size distribution but this also changes the mix viscosity. Using a large number of different size distributions, it has been found that a plot of burning rate against mix viscosity gives a smooth curve. The fastest burning rate obtained by this means is about 19 mm/s with a mix viscosity of 12 000 poise, and the slowest rate is 12 mm/s with a mix viscosity of 3000 poise.

Substituting another solid catalyst for the BDH ferric oxide also changes both burning rate and mix viscosity. Hence, in order to compare the catalysts, the curve of burning rate against mix viscosity is being used to correct measured burning rates to a standard mix viscosity. In all cases, burning rates are being measured over a range of pressures (4 to 14 MN/m²) by static firings of 50 mm diameter test motors (K-rounds). From previous work (ERDE TR 1/70/3) it appeared that the largest effects on burning rate would be obtained with the copper chromates and iron oxides and these have now been studied in more detail.

Four different copper chromite catalysts have been examined. Three of these, designated Cu0202P, Cu1402P and Cu2000P, were supplied by the Harshaw Chemicals

Company and the fourth was obtained from Schuchardt (Germany). All four catalysts gave somewhat higher mix viscosities than is obtained with BDH ferric oxide. With Cu2000P, the mix viscosity was too high for the composition to be cast and the effect of this catalyst was assessed by using it in admixture with Cu1402P. Both Cu1402P and mixtures of Cu1402P and Cu2000P gave higher burning rates than BDH ferric oxide, but when the correction for mix viscosity was applied they showed no advantage over the ferric oxide. With Cu0202P and the Schuchardt catalyst, the viscosity corrected burning rates were higher than with BDH ferric oxide, the Cu0202P showing an increase of 2 mm/s and the Schuchardt catalyst an increase of 1.5 mm/s. It has since been learned that Harshaw Chemicals have stopped production of Cu0202P, the most effective of the four copper chromites tested.

Ten different types of iron oxide from various UK and USA sources are being examined. Several of these were obtained through BDRS Washington following a visit to the USA during which it was learned that certain ferric oxides had been found to give burning rate increases comparable to those obtained with the binder-soluble liquid ferrocenes. So far the most promising results have been obtained with Deanox iron oxides LY 25 and L 10 (UK). These materials gave the same mix viscosity as BDH ferric oxide but a burning rate of 19 mm/s at 7 MN/m^2, an increase of 4 mm/s. These catalysts have not yet been examined at higher levels, but with a US catalyst, Mapico Yellow 1000 (Columbian Carbon Company), which at 1 per cent concentration gave 18 mm/s at 7 MN/m^2, 2 and 3 per cent (the additional catalyst replacing ammonium perchlorate) gave 19 and 18.5 mm/s respectively without any alteration in mix viscosity. At a pressure of 28 MN/m^2 (of interest in connection with a possible motor project), the above compositions gave burning rates of 30 to 32 mm/s.

Liquid Ferrocene Catalysts RESTRICTED

In parallel with the work on solid catalysts, the effect of binder-soluble liquid catalysts on burning rate is being investigated. This work is at an early stage and, so far, only n-butyl ferrocene (NBF) in concentrations up to three per cent has been examined in a composition containing 71 per cent ammonium perchlorate (45 per cent 7 μm, 25 per cent 60 μm, 35 per cent unmilled), 15 per cent aluminium and 14 per cent plasticized HC 434 rubber (the plasticizer consisting of NBF and IDP). The strand burning rates obtained are shown in Table 4 in comparison with results for a similar composition containing no catalyst and one containing one per cent BDH ferric oxide.

TABLE 4

EFFECT OF N-BUTYL FERROCENE ON BURNING RATE AND VISCOSITY

AP	Al	Composition (wt %)				Strand Burning at 7 MN/m^2 (mm/s)	Casting Viscosity (Poise)
		Catalyst	IDP	Rubber	Iron linoleate (added)		
71	15	Nil	2	12	0.1	11	5000
70	15	1 Fe_2O_3	2	12	0.1	14	6000
71	15	1 NBF	1	12	0.1	18	7000
71	15	2 NBF	Nil	12	0.1	23	11000
71	15	3 NBF	Nil	11	0.1	26	8000

It can be seen that NBF is not as effective a plasticizer as IDP, the replacement of IDP by NBF resulting in an increased mix viscosity. However, the increase in burning rate with NBF is very marked even if a correction is made for the increase in mix viscosity. Thus, three per cent NBF gives a burning rate of 26 mm/s with a mix viscosity of 8000 poise compared with a rate of 16 mm/s which would be obtained with BDH ferric oxide at the same mix viscosity.

From experience with NBF in plastic propellant compositions and from American experience with NBF in CTPB compositions, it is known that high concentrations of this catalyst greatly increase sensitiveness to spark, friction and impact. Concurrently, therefore, sensitiveness testing is being carried out on both cured and uncured compositions containing NBF. Results on the compositions containing up to three per cent NBF are reassuring because the friction characteristics are very similar to those of the composition containing no catalyst, and there is only a slight increase in impact sensitiveness and a moderate reduction in temperature of ignition (in the worst case, from 252°C to 235°C).

The effect of NBF on physical properties and on ageing characteristics is also being assessed. The composition containing one per cent of NBF has been stored under sealed conditions for 26 weeks at 60°C without measurable change in strand burning rate or physical properties.

Rheology

Parallel-plate Plastometry UNCLASSIFIED

(i) Liquid Ingredients of Plastic Propellant

The main liquid ingredient of plastic propellant is polyisobutene (PIB) with a viscosity of about 100 kNs/m^2 (10^6 poise) at 25°C, when measured at a very low shear-rate by the falling-sphere (Stokes) method. This is within the

practicable range of the parallel-plate plastometer, the use of which has been extended to include this PIB and the mixtures of it with plasticizer and surfactants which are used as binders in propellant formulations. A cylindrical specimen is compressed between parallel plates and the plate separation recorded against time. The method allows a wide variation of strain-rate, from 10^{-3} to 10 s^{-1}, which is important when dealing with polymers, which are usually non-Newtonian. Preliminary measurements on an instrument not provided with temperature control were subject to a large experimental error, and will be repeated on a recently-completed plastometer of improved design in which thermostatted air is circulated. The measured viscosities at room temperature ranged from 100 to 1 kNs/m^2 (10^6 to 10^4 poises), and were found to be approximately inversely proportional to the square-root of the strain-rate.

(ii) Plastic Propellant (E3342/9A)

The parallel-plate plastometer is also suitable for examining plastic propellants, which are stiff heavily-loaded pastes. These materials exhibit a yield stress, below which there is negligible deformation; above the yield stress, the ratio of strain rate to stress in excess of effective yield stress is termed the plastoviscosity. The plastoviscosities of plastic propellant are about 10^2 times greater than the viscosity of the mixture of liquid ingredients (or binder) which they contain. Two separate approaches have been made, the first of which is an extension of a well-established routine for measuring the initial (ie, zero shear-strain) plastoviscosity; the second measures the plastoviscosity at various values of strain as well as strain-rate. The two methods are considered in turn below.

Initial (Zero-strain) Plastoviscosity

A compression experiment is normally performed with a standard load, and the decrease in height (h_o - h) is measured at short intervals of time. The strain, defined as $\ln(h_o/h)$, is plotted against time and extrapolated to zero time. The initial slope of the curve represents the initial rate of shear and is estimated. The initial plastoviscosity is taken as the shear stress (equal to half the compressive stress) divided by the initial shear-rate. In the present work the experiment has been performed at room temperature with various loads, over the range 0.4 to 75 kg. This resulted in initial strain-rates over the wide range from 10^{-3} to 10 s^{-1}, and measured plastoviscosities over the range 10 to 10^{-1} MNs/M^2 (10^8 to 10^6 poises), approximately inversely proportional to the square-root of the strain rate. Thus the heavily-loaded paste and its liquid ingredient alone seem to behave very similarly; their flow curves are of similar form.

Plastoviscosity at Finite Strains

The second approach to the measurement of the plastoviscosity of plastic propellant has been to use the same technique as was applied to the liquid ingredients. Results were complicated and apparently extremely erratic until strain itself as well as strain-rate was introduced as a parameter. At the same strain, the plastoviscosity was again approximately inversely proportional to the square-root of the strain-rate, and at the same strain-rate the logarithm of the viscosity was inversely and linearly related to the strain which the

material had undergone. That is, the plastoviscosity decreases exponentially and rapidly as the amount of deformation increases. It is postulated that this may be due to progressive alignment of the dispersed particles within the stream-lines, tending to reduce particle/particle interaction. It is important to distinguish this behaviour from the shear-hardening phenomenon, in which the yield stress increases with increasing strain. When a plastoviscosity measured at a finite strain is corrected for the strain-effect, ie when it is extrapolated to zero strain, then it is indistinguishable from the initial plastoviscosity measured at the same strain-rate by the method described above. This statement should be slightly qualified, since there exists a doubt as to the absolute value (within a factor of 2) of the strain in the finite-strain experiments and of the strain-rate in both the experiments. This and earlier work now make it possible to derive a rheological equation of state for plastic propellant. The flow equation for E3342/9A is of the form

$$\dot{\bar{\gamma}} = k[s-(f_o + a\gamma)]^2 e^{4.6b\gamma}$$

where $\dot{\bar{\gamma}}$ is the mean strain-rate, s^{-1}

k is a material constant (1×10^{-1} for E3342/9A)

s is the stress, kN/m^2

f_o is the yield stress at zero strain, kN/m^2 (3.7 for E3342/9A)

a is the shear-hardening coefficient of the yield stress, kN/m^2 (nearly zero for E 3342/9A)

γ is the strain

and b is the strain-thinning coefficient of the plastoviscosity (1.84 decades per unit strain for E3342/9A)

The purpose of deriving such an equation is to predict rheological behaviour in other geometries and conditions. An attempt is being made to predict the form of axial slumping which a cigarette-burning plastic propellant rocket charge might undergo on axial acceleration during launching.

Effect of Variations in Composition on Physical Properties of Polybutadiene Propellants at Low Temperatures UNCLASSIFIED

The WLF superposition technique has been applied satisfactorily to the uniaxial tensile properties of five polybutadiene propellant compositions of (a) various solids contents (84, 86 and 88 per cent by weight respectively in compositions C37/144, C79/5 and C78/3, but having a constant degree of crosslinking), and (b) various degrees of crosslinking (1.00, 1.06 and 1.12 equivalents of crosslinking agents per unit equivalent of polymer respectively in compositions C79/4, C79/5 and C79/6, but having a constant solids content of 86 per cent).

ERDE dumb-bells of each composition were tested at strain-rates ranging from 0.01 to 10 min^{-1} over a temperature range of -60° to $+20^{\circ}C$.

The WLF glass-transition temperatures derived are -80°, -80°, -72°, -79° and $-74^{\circ}C$ for C37/144, C79/4, C79/5, C79/6 and C78/3 respectively, which agree well with the corresponding measurements by differential scanning calorimetry, -80°, -79°, -74°, -75° and $-74^{\circ}C$. The properties of C37/144 agree closely with those reported in ERDE Tech Memo No 4/M/69 for the earlier batches C37/20, C37/68 and C37/72.

Over the temperature range -40° to $+20^{\circ}C$, with elongations greater than 10 per cent, the mean effective gauge length for these polybutadiene propellants in our equipment is 48 mm, but the rounded value of 50 mm (as used to date) is well within the experimental scatter and will continue to be used to estimate strains when they are not directly measured. When the strains fall below 10 per cent the effective gauge length increases. The maximum recorded in these tests was 140 mm with a photographically-measured strain at 1.1 per cent.

The effect of low temperatures on the properties of the propellant has been assessed at a constant true strain-rate of 100 min^{-1} (chosen to represent rates encountered in rocket motors on ignition). At temperatures above $-50^{\circ}C$, the solids content has a dominant effect upon the extensibility, the least solids giving the greatest extensibility. Below $-50^{\circ}C$ (or at strain-rates even higher than 100 min^{-1}) a reduction in crosslinking might be more effective in increasing extensibility. The least crosslinked propellant (C79/4) has a tensile modulus about three-quarters that of the lowest of the others over the range of temperature 0° to $-55^{\circ}C$.

Antioxidants and Cure Catalysts in CTPB UNCLASSIFIED

Experiments have been carried out on the method of preparation of CrDIPS catalyst. Attempts were made to simplify the method by use of double decomposition in aqueous solution, including the reaction of BaDIPS and chromic sulphate. None of these attempts led to a satisfactory improvement. Most DIPS chelates including CrDIPS, NiDIPS and CoDIPS had a tendency towards glass, rather than crystal, formation. In certain applications this could be important and attempts to prepare PbDIPS are in progress. FeDIPS spectra appeared to be different from those of other chelates and the assumption is made that iron forms only a very loosely bound complex.

The reaction between epoxides and carboxylic acids was studied in greater detail. Dibutyl ether was a suitable solvent for the study of various carboxylic acids which could be of commercial significance. Amongst those tested in Bu_2O, p-hydroxy benzoic acid and anthranilic acid were reactive in the system M/10 DER 332 + M/10 acid in the presence of 0.5 per cent CrDIPS. Comparable half-lives at $70^{\circ}C$ for hexoic acid, p-hydroxy benzoic acid and anthranilic acid were 90 minutes, 340 minutes and 210 minutes respectively. An interesting finding was the similarity in the activities between hexoic acid and CTPB in toluene solution although, with CTPB, particularly when the epoxide is used is ERL 0510, the reaction did not proceed to completion as was the case with hexoic acid.

CTPB/epoxide cures were studied with a view to comparing concurrent changes in the infrared spectrum, the mix viscosity and curometer amplitudes. One of the main findings was that the curometer amplitude was a function not only of viscosity but also of the type of epoxide used. Viscosities were measured by a Brookfield Viscometer or a Wallace viscometer/gelation timer which allowed measurements with small quantities of a material in a confined space. With different epoxides substantially different curometer amplitudes were obtained when certain viscosities of the curing CTPB had been reached. Invariably there was a greater response with ERL 0510 than with DER 332; at a viscosity of 4000 poises significant Weissenberg effects occurred with a cure involving ERL 0510. The well known effect in which the flow of the liquid polymer is cut by scissors and the upper half retreats against the force of gravity clearly indicated that, under those conditions, elastic liquids were obtained. The conclusion was therefore drawn that the Wallace-Shawbury Curometer, which is insensitive to viscosity changes as such, can be utilised for ascertaining the appearance of elastic liquids.

More experiments were carried out with epoxides and LAMA (the Diels-Alder adduct of conjugated linoleic acid and maleic anhydride) and epoxides in the presence of CrDIPS as a cure catalyst and NiSDT as a stabiliser. Both these were incorporated by means of methylene chloride into LAMA prior to use. NiSDT at a concentration of 0.5 per cent was an outstanding stabiliser for LAMA by comparison with phenolic antioxidants such as BHA, Antioxidant 2246, or even synergistic combinations with DLTDP. At 100°C Ni SDT-stabilised LAMA exhibited induction periods which were between one and two orders of magnitude greater than those obtained with equivalent concentrations of conventional antioxidants and/or synergistic combinations. NiSDT also stabilised the final epoxy adhesive. A standard Araldite adhesive (AV100 + HV100) which was used to seal silica discs to Pyrex flasks was very efficiently protected against intensive unfiltered UV irradiation whilst the joint was immersed in water at 50°C. Under the best experimental conditions the LAMA/ERL 0510/ DER 332/CrDIPS/NiSDT combination produced stronger metal-metal adhesion than the above-mentioned Araldite combination. It can be used at elevated temperatures as well as at room temperature. At room temperature at least 2-3 days have to be allowed for the cure; at 70°C a few hours were sufficient, whilst at 100°C and 120°C periods of 1-10 minutes (depending also on addition of catalyst concentration) were adequate. For use at higher temperatures the recommended stabilisation by the addition of NiSDT is particularly important.

Cure Chemistry of CTPB Propellants RESTRICTED

In the early stages of the manufacture of rubbery propellants the rate of cure is usually monitored by observing changes in mix viscosity. Laboratory studies have shown that the extent of reaction in relatively simple mixtures (for example, those containing low levels of ammonium perchlorate) can be followed by infrared spectophotometry, and it was of interest to correlate these measurements with corresponding viscosity changes.

Changes in the viscosity of CTPB gumstocks (containing CTPB and epoxide in a 1:1 ratio, isodecyl pelargonate, and chromium octanoate) have been studied in relation to extent of reaction. Viscosities do not increase rapidly until

the extent of reaction is 0.6, and even here no system showed a viscosity greater than 200 poise. Values obtained on a Brookfield Syncrolectric Viscometer are listed in Table 5.

TABLE 5

Epoxide	Temp	Extent of Reaction at 0.1	1.0	10.0 kilopoise
DER 736	80°C	.845	.905	.915
DER 332	80	.82		.89
(MAPO)	80	.75	.82	-
ERL 0510	80	.63	.73	.77
ERL 0510/DER 332	80	.67	.795	-
O-glycidyl derivatives: 1,3,5-trihydroxy benzene	80	.62	.735	.765
	60	.50	.715	.76
O-glycidyl derivatives: 1,3,5-trihydroxy-/1,3-dihydroxybenzene	60	.55	.77	.80
ERL 0510/DER 332	60	.55	.755	-

Chemical Analysis and Quality Control

(i) Detection of Nitroglycerine RESTRICTED

Improvements have been made in the design of the ERDE nitroglycerine (NG) vapour detector following field tests carried out in collaboration with ROF Bishopton. The detector has been re-calibrated using accurate aliquots of NG vapour, obtained by evaporating weighed solutions of NG in methylene chloride, and the design has been simplified to facilitate use by unskilled operators.

(ii) Bisdinitropropyl Acetal/Formal (BDNPA/F) Mixtures CONFIDENTIAL

The ERDE nitrite-value test, developed for the routine analysis of NG, has been applied to the mixed nitroplasticiser, BNDPA/F, with a view to monitoring changes in impurity levels effected by various washing and filtration procedures. This test, which may be applied to samples as received or after brief pre-heating, appears to respond to both organic and inorganic nitrites, nitroso compounds, and nitrogen dioxide.

Various methods for repurifying old stocks of nitroplasticiser mixture, known to contain N-phenyl β naphthylamine (PBNA) antioxidant and its degradation products (see below), were investigated, and the degree of purification was

assessed by improvement in colour, nitrite-value, and Abel heat test. A good method is to treat an ethereal solution of the red mixture (nitrite-value 57 mgm/g) with activated charcoal, by trituration or by passage through a bed of charcoal/alumina. This gives a yellow product with nitrite-value < 1 mgm/g; it is important, however, to ensure that ether solvent is completely removed - failure to do so results in very low Abel heat test figures: for example 2 min instead of about 60 min. Ether is a better solvent than ethylene- or methylene chloride, methanol or acetone.

Ineffective purification procedures include: treatment with alumina, molecular distillation, washing with water, dilute acid or dilute alkali, and low temperature crystallisation from ethanol.

Thin layer chromatographic analysis showed that commercial samples of BDNPA/F, known to contain PBNA, also contained N-nitroso PBNA. This degradation product appears to enhance gassing rates of the mixture, judging by the results of Will gas evolution tests: the presence of N-nitroso PBNA (0.1%) increases gassing by a factor of three.

A general conclusion from this study of purification and storage stability of BDNPA/F mixtures is that the compounds are best mixed when required for use, and that it is probably better to store the separate ingredients without addition of PBNA.

(iii) Spectroscopic Analysis of Colloidal Propellants CONFIDENTIAL

Nitrocellulose and HMX

Simultaneous measurement of the nitrocellulose (NC) and HMX contents of an advanced CMCDB propellant can be made accurately by infrared spectroscopy if acetonitrile-d_3 is added to the propellant extracts. The internal standard is necessary since dimethylsulphoxide (DMSO) solvent attacks the windows of the infrared cell, changing the path length during the determinations. A small correction is required when aluminium powder is present. Accuracy is $\pm\frac{1}{4}\%$ (absolute) for NC, and $\pm\frac{1}{2}\%$ for HMX.

Molybdenum Trioxide

A rapid method (< 1 hr) is being developed for determining molybdenum trioxide in colloidal propellant. A perchloric acid digest is treated with alkali and sulphuric acid to yield a solution which is suitable for analysis by atomic absorption spectrophotometry.

(iv) Igniter Powder UNCLASSIFIED

A small sample of an igniter powder, heavily contaminated with mercury from a density determination, was analysed. Free carbon was separated from the propellant by centrifuging an acetone solution, and from mercury by flotation in bromoform. Final analysis depended on combustion to carbon dioxide; the other components were determined by established methods.

(v) Combustible Cartridge Cases UNCLASSIFIED

Quantitative analysis still presents problems; it is hoped that the position will improve when a new batch of Schweitzer's reagent (for dissolving cellulose) is obtained.

Residues from thirteen cases fired by RARDE were analysed. Some contained NC, cellulose and styrene-butadiene rubber, but in others the only recognizable components were cellulose and/or styrene-butadiene rubber.

Compatibility, Stability and Climatic Testing

Foreign Propellants: Stability Assessment CONFIDENTIAL

(i) A specimen of multitubular, single-based propellant, from Russian 30 mm ammunition, has been subjected to the NATO stability test, STANAG 4117. The propellant, stabilised with 1.15 per cent diphenylamine, gave results which were very similar to those obtained from comparable propellants made in Canada, the US and in this country.

(ii) Two types of Carl Gustav 84 mm recoil-less ammunition of Swedish manufacture have been assessed in respect of the chemical stability of the explosive fillings and the compatibility of non-metallic components. No adverse features were found in either the HEAT Round FFV 551 or in the HE Round FFV 64C.

Stability of Multron R18-TDI Polyurethane Rubber CONFIDENTIAL

We have studied the resistance of this rubber to hydrolysis when cured with varying proportions of TDI. Although higher proportions of TDI increase the crosslink density and improve the quality of the rubber, the hydrolysis reaction chiefly affects the ester functions in the polymer and appears to proceed at a very similar rate, notwithstanding the degree of cure. In consequence, the time taken for the rubber to reach a seriously degraded condition in a moist environment is not greatly improved.

Gas Cracking in Solid Propellants

(i) Colloidal Propellant - Gas Evolution Studies CONFIDENTIAL

Further results obtained after vacuum storage of a colloidal propellant at temperatures between 32° and 50°C have confirmed that, for the particular propellant under investigation, the rates of decomposition as indicated by gas evolution in this temperature range are some five times greater than would be expected by extrapolation from higher temperature data. At 50°C the reaction rate slowed down to the lower predicted rate after 10 to 14 weeks, but at 32°C gas evolution proceeded at a constant rate for at least one year with no sign of decrease. The propellant studied was not new and the higher decomposition rates are considered to be more indicative of its actual behaviour in storage than an extrapolation from high temperature tests. The activation energies derived from Arrhenius plots of both the fast and slow reactions are virtually

the same (~ 36 kcal/mole). At higher temperatures the higher rate reaction is completed in a comparatively short time and hitherto it has tended to be dismissed as a transient phenomenon during the early stages of testing.

(ii) Gas Transport Properties in Solid Propellants RESTRICTED

In studies on the cracking of solid propellant motors due to build-up of internal pressure, the solubilities and diffusivities of gaseous decomposition products assume a major role. Internal pressure, P, is determined by Fick's law of diffusion, the Henry's law solubility constant, S, and the gas generation rate, Q, according to the equation

$$\frac{\partial P}{\partial t} = D\nabla^2 P + Q/S$$

where D is the diffusivity and t the time.

For propellants of low volumetric solids loading these transport parameters can readily be evaluated by studying the progress to equilibrium conditions when gas is allowed to diffuse from a high pressure reservoir through a thin membrane into a vacuum. After a time-lag, from which the diffusivity can be calculated, steady-state permeation is obtained which gives the permeability P; the relationship P = DS then gives the solubility constant. However, with propellants of high volumetric solids loading this method is liable to give misleading results because of the inordinate effect of gas transport through minute pinholes and vacuoles in the membrane and filler binder boundaries.

More reliable results can be obtained by studying sorption and desorption kinetics, the gas passing into and not through the propellant. The quantity, q, of gas adsorbed (or desorbed) at a time t, on suddenly changing the surrounding gas pressure from the original pressure, can be related mathematically to the diffusivity, time and sample geometry. For a square-sectioned rod of infinite length the relationship is

$$Q = \frac{q - q_o}{q_\infty - q_o} = 1 - \left[\frac{8}{\pi^2}\sum \frac{1}{n^2} e^{-n^2 Dt}\right]^2$$

where q_o is the gas concentration at the original pressure, q_∞ is the equilibrium concentration at the new pressure and n an odd positive integer, and dimensions are in π units.

Methods have been developed, based on gas-handling techniques and gas chromatography for the determination of q, q_o and q_∞ using 10 × 1 × 1 cm rods of propellant, end-diffusion being prevented by coating with aluminium foil. From the equilibrium values at known pressures, the solubility constant S is readily obtained.

These methods are applicable to all colloidal and composite propellants and to explosives; another more rapid method for determination of diffusivities, based on the kinetics of the early stages of sorption is under development.

Compatibility and Stability Testing: New Techniques

Heat Flow Calorimetry UNCLASSIFIED

Preliminary investigations are in progress into the applicability of this method as a means of detecting incompatibility between propellants and other substances. Four materials known to exhibit incompatibility with propellants in the 80°C Silvered Vessel Test have been tested. The heat output of the propellant in contact with them at 80°C was at higher levels than was given by the propellant alone, and there was a correlation between the indicated degree of incompatibility and the SV Test result. There are, however, unexpected features in the heat flow records, particularly some very high but transient heat outputs at the commencement of the tests. A further understanding of the processes which are responsible will be required before the method can be considered reliable. Its great potential advantages over existing methods are that results are obtained within a very few days, whereas most other tests of compatibility with propellants require two to four weeks. Also, eventually, it is hoped to reduce the temperature of operation and so obtain information more readily related to behaviour at ordinary temperatures.

3.2 CHEMICAL ENGINEERING RELATED TO PROPELLANTS AND EXPLOSIVES

(a) REMOTE PROCESSING

Hydrazine Diperchlorate UNCLASSIFIED

Modifications to the pound-scale plant have been completed; an automatic drowning device has been fitted, and the filtration stage changed to give better visibility during the sulphur dioxide work. The technical sulphur dioxide contained impurities and equipment was installed for purification by distillation. Alternative methods of manufacture and drying are under review, including drying by azeotropic distillation.

Hydroxylammonium Perchlorate UNCLASSIFIED

Two methods of manufacture have been tried in the laboratory:

1 Double decomposition of hydroxylammonium sulphate and barium perchlorate. This process involved precipitation and troublesome separation of barium sulphate. The concentration of the liquor and drying of the hydroxylammonium perchlorate were not successful.

2 Double decomposition of hydroxylammonium chloride and sodium perchlorate in dry methanol, followed by distillation with butanol. During this procedure sodium chloride is precipitated and removed and the water present is taken up in the butanol/water azeotrope. The anhydrous hydroxylammonium perchlorate is salted out by addition of methylene chloride and stored under carbon tetrachloride. This method seems quite workable and samples of the product have been sent to the Advanced Explosives Research Group for assessment.

(b) NON-REMOTE PROCESSING

XIII Isocyanate UNCLASSIFIED

A further six kilograms were made for SE Branch during the period.

Barium Nitrate UNCLASSIFIED

The production of barium nitrate, recrystallised to Spec 1A for supply to MOD and IMI at about 600 kg per month, was continued. It became apparent that material well within specification but which was not acceptable to MOD could be produced. MOD require material to a closer size range than is specified. Investigations showed that it was possible to produce near-to-size range on the plant directly and material to a closer specification was achieved by blending.

Investigations both in the laboratory and on the plant with the object of avoiding crystal aggregation showed that aggregates are formed in the crystalliser itself irrespective of the rate of agitation.

Ultra-Fine Perchlorate UNCLASSIFIED

Work on this project was restarted during the period, but the problem of particle size measurement of this material is still not solved. Specific surface measurement by air permeability is of limited value and in the absence of a better method characterisation is by microscopic examination.

A freeze-drying technique, which seems preferable to ball milling, is being reinvestigated, using Aerosol OT. Despite reported American claims for 0.4 μm material by this method, the product obtained was of the 2 - 5 μm particle size which is coarser than micronised material.

Hexanitrostilbene Manufacture UNCLASSIFIED

Due to the pressure of other work this was not pursued very actively during the period. A process has been developed for the recovery of solvents, with recycling, so that only three parts of tetrahydrofuran are lost for one part of HNS. After about five recycles of tetrahydrofuran there was a sudden and large drop in the yield of hexanitrostilbene. Attempts to determine whether this was due to build-up of impurities, or the presence of excess water, or the use of poor quality hypochlorite, have not yet been successful.

Recovery of Lead Stannate RESTRICTED

There are in ERDE large quantities of lead stannate which does not conform to the current specification. Direct use by blending with high burning-rate material is not considered worth while; the other alternatives are to convert it into commercially saleable lead and/or tin salts, or into acceptable lead stannate. Treatment of the lead stannate with nitric acid gives lead nitrate solution and stannic oxide but further conversion of the stannic oxide to sodium stannate is not practicable in our existing equipment. Reaction of the lead stannate with sodium hydroxide solution gives mainly litharge and sodium stannate solution with some sodium phenolate. It is possible to react these in the usual way to give lead stannate and indeed a quantity of some 25 kg was made by this technique which, however, was considerably discoloured.

It seems likely that a long and costly joint programme of testing would be necessary before a successful recovery process could be proved, and it is doubtful whether this can be justified.

Crystallisation Research UNCLASSIFIED

Experiments with the laboratory-scale continuous crystalliser were performed at various impeller speeds and production rates, which showed that provided all crystals were in suspension, the rate of growth was independent of size. The

rate of growth was linear with supersaturation within the range investigated and, per unit saturation, was proportional to the quarter root of the power input of the stirrer.

The rate of nucleation was proportional to the surface area of the crystals in suspension and the rate of nucleation per unit area of crystal surface was linear with supersaturation, above a value of about 0.4 per cent supersaturation, and increased with increasing stirrer speed.

This suggests that nucleation, within a continuous crystalliser of this type and under these operating conditions, is caused by attrition either through collision of crystals with themselves and/or the wall of the vessel. The product crystals were quite regular in shape and showed no sign of such damage which may, however, have been very small and repaired by rapid growth at broken surfaces.

3.3 HIGH EXPLOSIVES, INITIATORS AND THEIR INGREDIENTS

(a) DETONATION STUDIES

Underwater Explosives RESTRICTED

Experiments have been carried out to study the effect of the specific surface (S_o) of ammonium perchlorate on the detonation velocity of composite explosives in the Poly-X class.

Cylindrical charges ranging in diameter from 25 mm to 50 mm were prepared using ammonium perchlorate with S_o = 1.6×10^4 m^2/m^3, 5.0×10^5 m^2/m^3, 1.0×10^6 m^2/m^3, 1.5×10^6 m^2/m^3. The maximum concentration of ammonium perchlorate studied was 50 per cent by weight. The two most interesting compositions were those containing 50 per cent and 40 per cent ammonium perchlorate; the former having the higher calculated energy release.

Plots of detonation velocity against specific surface show similar trends for both compositions; but a comparison between the two plots reveals that with coarse ammonium perchlorate the 50 per cent composition shows a lower detonation velocity than the 40 per cent composition; whilst with fine ammonium perchlorate the position is reversed. Tentatively, the following observations and inferences may be drawn from the results obtained so far:

1. In all compositions containing ammonium perchlorate, the detonation velocity increases with specific surface.

2. When S_o is greater than 1.0×10^6 m^2/m^3, detonation velocity no longer appears to be a function of S_o suggesting that the major portion of the total chemical energy is released before the C-J plane.

3. Coarse ammonium perchlorate with S_o about 1.6×10^4 m^2/m^3 removes energy from the reaction zone by acting as a heat sink rather than contributing to the chemical reaction.

Underwater Shock Data for Poly X-75 and Torpex 2 CONFIDENTIAL

32 kg cylindrical charges of Poly X-75 are being tested at NCRE for their damage effectiveness against underwater targets. More tests could be carried out against each target if it were possible to use 0.5 kg charges instead. However, US experience with composite explosives of the Nitronol type has cast doubt on whether the underwater shock parameters of this type of explosive scale with charge weight in a simple manner. For this reason, a careful comparison of the parameters for 0.5 kg and 3.5 kg charges has been made. This revealed that the peak shock pressure, the impulse and the energy flux are considerably reduced at small stand-off distances for the smaller charges when compared with the 3.5 kg charges on a normal scaling basis. Only at larger stand-off distances is the scaling valid.

In response to a request from NOL, ERDE underwater shock data for Torpex 2 (3.5 kg cylindrical charges) were collated and the regression lines or curves based on 40 measurements quoted. Work continues on the present TNT charges (underwater standard) and on H4027-A2 (a Nitronol composition).

Rocket Motor Blast Simulation CONFIDENTIAL

Attempts to predict the pressure field from static firings of BLACK CAP have given widely different results; pressures obtained by the application of scaling laws to some predictions made by BAC for Gosling motors range from one-half to one-seventh of those predicted by RPE on the basis of ionization measurements and a mathematical model of jet structure.

Experiments at RPE are planned to determine the kinetic pressure field of BLACK CAP and results are expected shortly.

Instrumentation UNCLASSIFIED

A new technique for producing a spiral time-base oscilloscope using a conventional CRO as display unit has been devised. The technique offers considerable advantages over previous spiral CRO designs, notably that a radial signal deflexion is easily added to the display and that the circular frequency and collapse speed are readily adjustable. No L-C circuits are necessary in the design.

Tenders have been invited for design and construction of a prototype equipment using the new technique and a Technical Note is in preparation.

(b) COMPOSITE EXPLOSIVES AND INGREDIENTS CONFIDENTIAL

Poly-X Compositions

An NCRE survey of paddle wheel target damage, submarine model damage and Scorpion (destroyer) shock trial results has indicated that Poly X-75 seems to be equally effective against both weak and strong targets, and that on an equivalent weight basis it is 1.2 ± 0.05 times more effective than Torpex 2. Although this figure is less than was hoped for, at larger stand-offs, from the ERDE free field measurements, the increased close in effectiveness against strong targets has heightened interest in the use of this filling in the NAST 7511 torpedo warhead. Confirmatory firings are to be carried out at NCRE and three 70 lb charges and three 6 lb charges in each of Poly X-75 and Torpex 2 have been prepared for these trials.

The MK 44 torpedo warhead has been selected as a suitable vehicle for carrying out filling and hazard trials (in collaboration with DGW(N) and OB) with Poly X-75, and a supply of empty cases has been arranged through AUWE.

Further measurements at ERDE have confirmed that Poly X-97(33 per cent aluminium) shows significant increases in shock wave impulse and time constant compared with Poly X-75, and this may deserve following up in the light of AUWE and NCRE target response studies.

An attempt to influence the shock parameter by concentrating the aluminium into the central part of the charge gave disappointing results; peak pressure, time constants and impulse were all reduced in comparison with a control charge of uniform composition (Poly X-98).

Nitranol, PBXN 103 CONFIDENTIAL

Twenty 8 lb charges of this composition have recently been supplied to us by NOL, White Oak. An assessment programme is in hand and both the OB and DGW(N) are to be kept informed of results.

New Compositions CONFIDENTIAL

The lithium perchlorate composition reported in TR/70/1 gave underwater shock impulse below that of Torpex 2, but time constants 60 per cent greater. Further exploration of lithium perchlorate systems is needed, and work continues on processing problems, including vacuum coating of the oxidiser with aluminium to reduce oxidiser solubility. The viscous solutions formed by this oxidiser in a wide range of binder materials have been examined; glycerol solutions containing up to 66 per cent oxidiser may be fluid enough to be workable. However, the RMH (rotter impact test) falls from 142 cm at room temperature to 16 cm at about 80°C, and some caution is called for in hot processing.

Aluminium Hydride CONFIDENTIAL

The material supplied through AFRPL has been found insensitive by itself, but preliminary mixes with the Poly-X binder, both with and without ammonium perchlorate, have exhibited RMH values between 2 and 5 cm and have given ignitions at the lowest loading (0.5 kg) on the BAM friction test. (A mix with sand, of roughly similar hardness, replacing the hydride, gave a BAM result of 6 kg). Autoignition tests have shown no stability problems in the absence of ammonium perchlorate, but with the oxidiser present early exotherms occur; the effects of stabilisers have not yet been explored. Effort is being concentrated on using the remaining hydride to establish the performance obtainable from a simple aluminium hydride/Poly-X binder composition.

Oxidisers CONFIDENTIAL

Laboratory testing of stability, sensitiveness and purity of hydrazine diperchlorate and hydroxylamine perchlorate samples is continuing, but larger quantities of these oxidisers have not yet become available.

Slurry Explosives CONFIDENTIAL

A large amount of background information has now been gathered from the literature, particularly from patents, and discussions have been held with IRECO (Dr M A Cooke) Rio Tinto Zinc, ICI, and Explosive and Chemical Products Ltd, who are the only companies concerned with slurry explosive manufacture in the UK.

Nine commercial products have been procured, and a test programme has been commenced covering the following properties: gel stability over the range -26°C to +50°C, gas evolution at 50°C, resistance to salt and fresh water, pourability, sensitiveness, shock sensitivity, detonation properties and sensitivity to fragment attack. Information and samples of Guar gum and synthetic gelling agents have been acquired from several sources, and preliminary exploration of gelling agent/cross linker behaviour in saturated nitrate solutions has been carried out. Ranges of fuel and nitrate oxidiser grades have been selected for investigation, and some small batches (1 kg) of typical explosive compositions have been made using a small domestic mixer. It does not seem likely that sophisticated equipment will be required for larger batch production.

(c) SENSITIVENESS

Fragment Attack on Propellant and Rocket Motor Segments CONFIDENTIAL

Fragment attack of the fast burning plastic propellant RD2428 has been carried out. Key velocities for ignition have been established for various experimental arrangements as before (values in m/s; where two values are given, the first is the maximum impact velocity for non-ignition, and the second the minimum velocity for ignition).

TABLE 6

	Projectile A (cylindrical)	Projectile B (fronted with 30° half angle cone)
1) Bare propellant	~ 700	482 - 502
2) Propellant with metal plate stood off by 25 mm	511 - 550	553 - 578
3) Plate bonded to propellant as in rocket motor	509 - 531	490 - 472

In A(1), B(1), A(2) and B(2), although the face attached was bare, the curved face of the charge was bound by Lassotape, as otherwise bare charges merely broke up without ignition at impact velocities up to 1070 m/s; with the plate-bonded charges (3), binding with Lassotape made no difference to the results. With projectiles B it is clear that plate bonded to the propellant provides much less effective protection than when stood off; this effect is similar to that observed with ATN/D28/47 propellant but the general level of sensitiveness to such stimuli is less with RD2428.

Sensitiveness of Poly-X Explosives CONFIDENTIAL

The sensitiveness of Poly-X75 at low temperatures to fragment attack was studied under conditions similar to those of previous work (TR 2/69/1 and TR 1/70/1). At -25°C a cylindrical charge 127 mm (5 inches) diameter by 25 mm (1 inch) thick, attacked by a 14 g (0.5 oz) steel ball, 15.1 mm (19/32 inch) diameter at an impact velocity of 2050 m/s (6710 ft/s) in the centre of one plane face, was completely penetrated without ignition. At -30°C and 2090 m/s (6780 ft/s) the charge ignited and was completely consumed, and its supporting angle-iron framework was heavily distorted; at a similar temperature and 1750 m/s (5760 ft/s) however the charge merely broke up and the frame was not damaged. Uncased cylindrical charges 76 mm (3 inches) diameter by 229 mm (9 inches) length but with aluminium alloy plates at front and rear, attacked at about 900 m/s (2950 ft/s), produced increasingly violent responses (decreasing quantity of Poly-X remaining unconsumed, increasing damage to metal frame) as the temperature was reduced from -25°C to -45°C. With the material cast in steel tubes 89 mm (3.5 inches) od and 4.75 mm (3/16th inch) wall thickness, but without front plates a similar effect was observed. At -30°C and impact velocity of about 825 m/s (2700 ft/s) the tube split but remained in one piece; the angle iron frame, while heavily distorted, was not broken up, and a good deal of the Poly-X remained unburnt albeit in small pieces. At -48°C and about 2070 m/s (6800 ft/s) however, a violent explosion was achieved, the tube being split into small pieces and the angle iron frame shattered, and no unburnt Poly-X recovered. These results, in combination with those reported in TR 1/70/1, indicates little or no difference in fragment attack sensitiveness between Poly-X75 and Poly-X98 either in ambient or low temperatures.

In order to assess the possibility of greater explosive response from cracked charges, Poly-X75 cast in steel tubes as above were stored at 80°C for about two days to produce cracks (as shown by X-ray). At ambient temperature and impact velocity of 895 m/s (2940 ft/s) ignition occurred but much material remained unburnt; the steel tube was split but the frame was undamaged. At 2040 m/s (6690 ft/s) and 2140 m/s (7020 ft/s) the results were similar but rather more violent, the frames being distorted. At -30°C and about 2040 m/s (6700 ft/s) the tube was broken into a few pieces but some unburnt Poly-X was still recovered; at -50°C however, with similar impact velocity the tube was thoroughly fragmented, the state of some of the pieces indicating a partial detonation of the Poly-X. It is therefore evident, when comparison is made with the results reported in TR/1/70/1, that greater explosive output is obtained under similar conditions from a cracked charge.

Sensitiveness of PETN RESTRICTED

Because of variations in sensitiveness figures for PETN over the years, AWRE have supplied two types for impact friction and temperature of ignition measurement. They were (1) XN Type, Batch 442, and (2) Commercial grade, Batch RK316. The mean F of I results for six determinations were respectively 60 and 58 on the two ERDE Rotter machines for the XN type, and 51 and 49 for the commercial type. In the friction tests and ignition tests no significant differences were obtained between the two samples. The tests have been completed and will be reported through the Sensitiveness Collaboration Committee.

Large Sealed Vessel Tests on Gunpowder UNCLASSIFIED

Large sealed vessel tests have been carried out on gunpowder (a) to widen the type of compositions studied by this test and (b) to see whether the results agree with its known explosive risk. Two igniter tests and two heated tests were performed with G40; the former gave considerable fragmentation and both would be classed as borderline between a low order explosion and an explosion (LOE/E). The latter gave less violent results, one being classed as a pressure burst (PB) and the other as borderline between a pressure burst and a low order explosion PB/LOE. This diminished effect was thought to be the result of some slow and not very exothermic reaction taking place during the heating, which uses up some of the available energy so that the ultimate bursting reaction is less violent. The overall results however confirm the explosive risk category for gunpowder.

Pressure Vessel Testing for RRE UNCLASSIFIED

A "pressure puller" for the manufacture of crystals of semi-conducting materials has been developed at RRE. The apparatus is essentially a watercooled cylindrical stainless steel vessel, of internal volume 2.4 litres, with various attachments and parts. The design working pressure is 20 MN/m^2 (3000 psi) at approximately 500°C. A considerable safety factor was known to exist but interest was shown in assessing the mode and consequences of failure should this occur due to excessive pressure rise. Two preliminary experiments have been carried out using burning cordite to pressurise the vessel and recording by high speed photography and pressure gauges.

The results showed a blast overpressure of approximately 2 psi at about 2 m distance which is well in excess of the maximum tolerable in a laboratory; in consequence the design of the vessel will be re-examined.

Electrostatic Problems UNCLASSIFIED

1 Advice was sought by CILSA on the proposal to fit plastic type rollers for a conveyer gravity runway where steel rollers are not allowed. These rollers were found to have a resistance of 10^{13} ohms, and consequently high potentials were obtained when two of them were rolled over a metal plate to simulate the movement of a metal cased store. Solutions involving partial or complete change to anti-static materials were put forward.

2 CAD Bramley are working on an explosive dust extractor and a prototype has been tested. The hose and collecting vessel was made largely of plastic material which was found to have a high resistance. Tests showed that the inner metal cylinder housing the filter could be charged to 11 kV when 1 oz talc was sucked up. Substitution of anti-static hose for the flexible hose showed only a small improvement, thus it appears essential to construct the whole extractor of anti-static or conducting material.

3 Two samples of Mipolam anti-static flooring have been tested for surface and transverse resistance. One made to the MOD specification gave satisfactory results and the other, the standard material, obeyed the surface resistance requirements but had rather a low transverse resistance. A floor is being laid for the Initiator Section with the former material.

(d) INITIATORS AND IGNIFEROUS EXPLOSIVES

Lead Styphnate — UNCLASSIFIED

Stores have been filled with VH2 composition using RD1367 and are under trial. Manufacturing trials of basic lead styphnate compositions RD1346 and RD1349 have confirmed that styphnic acid, both special and recrystallised (CS1936) from ROF Bridgwater, can be employed.

Other Nitrobody Salts — RESTRICTED

Thallous styphnate has been prepared and characterised and its explosive properties investigated.

The cerous salts of picric acid, 2.4 dinitrophenol, 2.4 dinitroresorcinol 4.6 dinitroresorcinol, styphnic acid and trinitrophloroglucinole have been prepared and are being evaluated as prospective igniferous compositions.

The F type of lead trinitrophloroglucinate has shown promise as a priming composition in wirebridge caps and is being developed.

The investigation of salts of nitramines has been resumed with the preparation of the sodium and lead salts of ethylenedinitramine.

Lead Azide — UNCLASSIFIED

The possible causes of poor quality in a series of production batches of Service lead azide have been investigated and traced to high alkalinity of sodium azide solution; this has now been corrected and normal quality restored.

Tetrazole Compounds RESTRICTED

Attempts have been made to increase the stab sensitiveness of lead azotetrazole by crystallisation with silver and thallium azotetrazole. Preliminary work on preparation and assessment of thallous and cadmium azotetrazole has been carried out and a detailed study of the reactions of the azotetrazole anion with acids is in progress.

Potassium Picrate UNCLASSIFIED

In support of technical applications by AWRE, a laboratory scale method has been developed to prepare potassium picrate in a suitable physical form with the prospect of a feasible production-scale process.

Overseas Collaboration UNCLASSIFIED

Sweden: A visit has been paid to the Swedish National Defence Research Institute and Government filling factory for discussions on research and development of primary explosives. Representatives of the Industrial Laboratory (Swedish Match Corporation) have visited ERDE in connection with research projects.

Pakistan: In support of the licence agreement, demonstrations at ERDE and background technical information have been given on lead azide RD1343 production.

Israel: Demonstrations of the manufacturing processes for RD1343 and RD1357 have been given to the technical representative and a licence agreement has been signed.

Australia: A licence agreement has been completed for the production of tetrazene (RD1357) by Messrs ICI (ANZ).

RD1362 HD (lead azide/coprecipitated boron) RESTRICTED

RD1362 has been prepared successfully on the 5 lb scale using standard equipment. An investigation into the reaction of RD1362 with the atmosphere has been completed and recommendations made regarding appropriate storage and loading conditions.

Delay Detonator RESTRICTED

The prototype stage has been reached in the development of a 30 millisecond delay detonator for gassy conditions using RD1362, RD1343 and RD1347 compositions. For military use and for application to non-gassy conditions an extended range of delays from 10 to 300 milliseconds is available; uses for experimental purposes are planned.

Coprecipitation Delay Compositions RESTRICTED

RD1368 (barium chromate/boron) has been approved for use in the L25 fuse, following successful production and user trials.

Work has continued on compositions using fuels other than boron, with the main emphasis on tungsten, molybdenum and titanium, and a number of promising compositions have been made. One lead chromate/titanium composition has the very fast burning rate of 0.01 sec/in. Interesting compositions have been made using lead and barium tungstate, and molybdate and mixed lead/barium chromates, all of which can be prepared in granular form. Some of the compositions appear to have potential as the igniting layer on fuse-heads.

Boron UNCLASSIFIED

Further investigation of boron produced at Bridgwater has shown some variability in the products from nominally identical runs. Some of the material would probably be usable for RD1368. Elastic igniting compositions can be made from any of the boron produced at Bridgwater, slow curing being overcome by the addition of up to 3 per cent of magnesium oxide.

Elastic Pyrotechnics UNCLASSIFIED

Most of the recent work has been confined to elastic processing with ad hoc studies of matters of concern to RARDE and ROF Glascoed. Mixes of the SR(E) 794 type (B/KNO3/Thiokol) have been found to be slowed considerably by the inclusion of 5 per cent Eccospheres (hollow V/F spheres) and by the use of high purity boron, both results being contrary to expectations. The effect of added water has also been studied and it was shown that curing was progressively accelerated by increasing water additions; the products became increasingly "short" after curing and the rate of burning was somewhat decreased as initial water content increased.

The effect of the use of silicon and mixtures of silicon and boron in SR(E) 794 on sensitiveness has been studied preparatory to the development of such compositions at ROF Glascoed.

A few flame compositions containing titanium instead of magnesium have been made with CTPB binder; these overcame gassing but did not burn well.

(e) INGREDIENTS OF EXPLOSIVES

Nitroaromatics

i 4,6-Dinitroresorcinol RESTRICTED

Work on this initiator intermediate has now been completed, and its preparation has been demonstrated on a pilot-plant scale.

ii Chloronitrophenols RESTRICTED

Work on the catalyzed halogenation of nitrophenols has been completed. In general, where the hydroxyl group is activated by the presence of o- or p-nitro groups halogenation may be effected either by thionyl chloride/dimethylformamide or by an equimolar quantity of phosphorus oxychloride on the pyridinium salt prepared in situ in a suitable solvent. With m-nitro groups no substitution can be made. However, on occasion the activation effect can be too great; thus while trinitrophloroglucinol could be converted into chlorostyphnic acid by the use of one mole of thionyl chloride, attempts to introduce a second and third chlorine always resulted in complete decomposition.

It appears that either of these methods may offer considerable economic advantage over traditional mixed-acid nitration for the preparation of picryl chloride.

iii Trinitrotoluene - Analysis UNCLASSIFIED

A colorimetric method for small amounts of TNT has been examined to assess its suitability for determining the extent of contamination of rubbers exposed to TNT. The method depends on formation of a deeply coloured Meisenheimer complex in diaminoethane/dimethyl sulphoxide solution. Good results were obtained with known amounts of TNT. No colour formation was observed with extracts from untreated rubbers.

iv Temperature-resistant Explosives RESTRICTED

The Shipp synthesis of polynitroaromatics - involving generation of the trinitrobenzyl anion in tetrahydrofuran - has been extended to the preparation of dipicrylmethane, but the product is very dermatitic.

Examination of an authentic sample of nonanitroterphenyl (NONA) by DSC, showed that the material previously prepared in these laboratories could not have been NONA. A sample of 3-chlorohexanitrobiphenyl has also been examined.

v p-Nitromethylaniline RESTRICTED

Methylation of 4-nitroaniline by formaldehyde in sulphuric acid has been investigated. It is possible to prepare n-methyl-4-nitroaniline (PNMA) in good yield by this method, but a suitable means of purifying the crude product has still to be found.

Nitramines

i HMX/RDX - Manufacture RESTRICTED

An investigation has been started into the methods of manufacture of HMX. Work to date has served to emphasise the need for absolute reproducibility in the parameters governing the initial simultaneous, three-feed addition, and in the

subsequent two-feed addition. An experimental rig based on three microsyringe pumps has permitted the required degree of accuracy and reproducibility to be attained. A similar apparatus using DCL reciprocating pumps (with a feed accuracy of ± 2%) has given markedly different results. An analytical method based on infra-red absorption in DMSO solution has been developed to analyze the final HMX/RDX mixture; at the moment there is an unresolved discrepancy between the results of this method and those obtained by the conventional differential solubility method.

Some work has been done on NMR analysis of various compounds involved in the nitrolysis of hexamine. Initial results were promising in that it proved possible to differentiate RDX from HMX in nitric acid solution, but there are still experimental difficulties to be resolved before this technique can be applied to kinetic measurements.

ii RDX Compositions - Thermal Stability CONFIDENTIAL

Preliminary experiments at 140°C with the compositions UR/60 and UR/4 (both nominally 80% RDX) show that the initial stages of the decomposition follow first order kinetics. The rate of disappearance of RDX from UR/60 is twice that from UR/4, and some eight times faster than the decomposition of RDX itself.

Perchlorates

i Hydrazine Diperchlorate - Analysis RESTRICTED

It appears to be possible to differentiate the weakly bound and strongly bound perchloric acid in hydrazine diperchlorate by titrating with sodium acetate in acetic acid to the first end-point and then adding acetic anhydride before titrating to the second end-point. End-points were determined potentiometrically. So far it has not been possible to separate free perchloric acid from the two types of bound acid by this method.

ii Dimethylammonium Perchlorate - Polymorphism UNCLASSIFIED

Further observations on the polymorphism of this material show that it exists in a form stable below 300K in a birefringent (biaxial) modification; above this temperature in an isotropic (cubic) modification. The transition is easy to induce and is reversible. Material stored in a cool magazine is biaxial; on the warm stage of a microscope it becomes isotropic. Neither of these dimorphs correspond with Stammler's (1966 Advances in X-ray Analysis) tetragonal and therefore optically uniaxial form, nor does the transition point correspond with his figure of 311K; in addition EMR work at Royal Holloway College indicates two transitions at 311K and 313K. We find the material to be isotropic up to its melting point (453K).

Initiatory Explosives

i Compatibility of Silver Azide with Rubbers and Plastics RESTRICTED

No further instances of incompatibility with silver azide have been found. Specimens of silver azide degraded over various plastic materials have been supplied to RARDE for performance tests. The results suggest that when the product of partial decomposition is metallic silver (as in the majority of cases so far), the effects on performance are much less harmful than may be the case when other products are formed.

ii Compatibility of Basic Lead Azotetrazole (RD1355S) with Metals RESTRICTED

Tests of the effects of basic lead azotetrazole (RD1355S) on various metals under extreme conditions of humidity and temperature show that tin is the most resistant of those tested with no apparent effect after four weeks at 60°C, 100% RH and in a high concentration of carbon dioxide.

Aluminium and silver were slightly attacked but only when carbon dioxide was present, moisture alone having no effect. Copper and to a greater extent cadmium are more susceptible to corrosion, but no information regarding the potential hazards from the products is yet available.

iii Sodium Azotetrazole - Decomposition UNCLASSIFIED

Gaseous products formed by reaction of sodium azotetrazole with excess 5N hydrochloric acid and 5N nitric acid at room temperature have been studied by gas chromatography. With hydrochloric acid, nitrogen (2 moles) is produced and with nitric acid 4 moles of nitrogen and 0.5 moles CO_2 were recovered.

Non-Explosive Ingredients

i Ferrocenes CONFIDENTIAL

A number of ferrocenes have been synthesized; considerable experience with separation and analytical techniques was required before reproducible results could be obtained. In general it was possible to reproduce published work, but the preparation and isolation of novel compounds met with only limited success.

Formylferrocene, FeCHO, mp 120°C was prepared easily (53% yield) by the action of n-methylformanilide and phosphorus oxychloride on ferrocene. The crude product was difficult to purify, but it was found that column chromatography was a better technique than differential extraction.

Hydroxymethylferrocene, $FeCH_2OH$, mp 76°C, was prepared easily (96% yield) by the reduction of formylferrocene with ethanolic sodium borohydride.

Bis(ferrocenylmethyl)acetal, $(FeCH_2O)_2CHMe$: condensation of hydroxymethylferrocene with acetaldehyde gave a material, possibly polymeric, which analyzed correctly for the expected structure. This material deflagrated on grinding.

Bis(ferrocenyl-1-butyl)acetal, $(FeCHPrO)_2CHMe$: condensation of 1-hydroxybutylferrocene with acetaldehyde gave a material in good yield (95% w/w); however, although the iron content is correct the C/H analytical data do not fit the expected structure, and this product has yet to be fully characterized.

1,1'-Diacetylferrocene, $FeAc_2$, mp 128°C, (84%) and 1,1'-bis(1-hydroxyethyl)ferrocene, $Fc(CHOHMe)_2$, mp 67°C, (96%) were produced easily, the latter by reducing diacetylferrocene with ethanolic sodium borohydride.

Acetylferrocene, FeAc, mp 77°C, (63%) was prepared easily then converted into 1-hydroxyethylferrocene, FcCHOHMe, mp 78°C, (98%) by reduction with ethanolic sodium borohydride.

Samples of properly-characterized ferrocenes are being evaluated as burning-rate catalysts.

ii Epoxy Curing Agents RESTRICTED

Cyanoethyl Derivatives

Epoxide derivatives of various cyanoethylamines have been prepared by treating the appropriate amine (itself synthesized by reaction of acrylonitrile with ammonia or an alkylenediamine) with epichlohydrin. Boron trifluoride appears to be a better catalyst than lithium hydroxide for the synthesis. The following compounds have been synthesized and obtained in a relatively pure state: N-glycidyl-N,N-bis-(2-cyanoethyl)amine; N,N'-diglycidyl-N,N'-bis(2-cyanoethyl)-ethylenediamine; and N,N'-diglycidyl-N,N'-bis(2-cyanoethyl)trimethylenediamine. These compounds are being evaluated as crosslinking reagents for CTPB, particular regard being paid to their effect on mechanical properties.

Ferrocene Derivatives

Attempts to prepare epoxy derivatives of ferrocene have met with partial success. It has been possible to effect reaction between either hydroxymethylferrocene or 1,1'-bis(1-hydroxyethyl)ferrocene and glycidol or epighlorhydrin to give products which contain both the ferrocene moity and an epoxide group. However, some difficulty has been encountered in determining epoxide content, and these products have not yet been characterized as individual pure compounds.

Nitramino Epoxides

A quantity of N,N-diglycidylmethylenedinitramine has been prepared for further examination, and two new epoxynitramines have been synthesized: N-glycidyl<u>iso</u>-propylnitramine and N-glycidyl<u>tert</u>butylnitramine.

Larger-scale Preparations

The following epoxides have been synthesized on a larger scale for evaluation as propellant ingredients: O-glycidyl derivatives of benzene; 1,2,3-trihydroxybenzene; and ethylene glycol.

iii Platonising Agents for Colloidal Propellants CONFIDENTIAL

Samples of basic copper β-resorcylate, basic copper terephthalate, lead citrate (phase A, phase B, and phase D), lead tartrate, lead malate, the lead salts of 4- and 5-amino- and acetamidosalicylic acids, and lead acetamidobenzoate have been submitted for ballistic assessment.

iv Dimethyl Phthalate - Calibration Standard UNCLASSIFIED

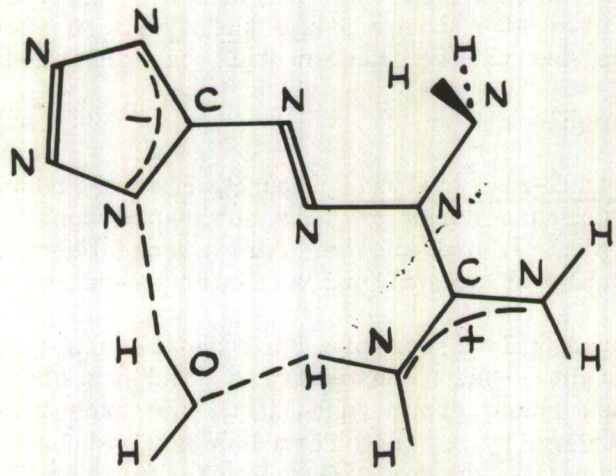
Preliminary investigations suggest that refractive index and melting point may be of value in specifying pure dimethyl phthalate.

The literature gives a melting point of 272K, but a sample of unpurified "Laboratory reagent" grade had already a setting point of 272.5K.

Crystallography

i Tetrazene UNCLASSIFIED

Refinements of the structure parameters of the A and B crystal forms of this substance have now been completed, final R-Values being 0.046 and 0.026 respectively. The preferred systematic name for the revised structure of tetrazene is the zwitter ion (guanidinium) form of 1-amino-1-[(1H-tetrazol-5-yl)azo] guanidine hydrate rather than that quoted in TR 1/70; the A and B forms are related as polymorphs, and have the same molecular conformation, the A-form being the thermodynamically stable one at ambient temperatures.



The mechanism suggested by Patinkin et al (JACS, 1954, 77, 562) for the formation of tetrazene by the reaction of sodium nitrite and an aminoguanidinium salt in neutral solution involves the reaction of tetrazole-5-diazonium ion, formed as an intermediate, with unchanged aminoguanidine. The revised structure now presented for tetrazene supports this view of the course of the reaction, but the position of coupling is now normal rather than abnormal as previously suggested, since it is observed in other cases that such coupling reactions involving substituted hydrazines usually occur at the substituted nitrogen atom.

A preliminary account of the results of this work has been published (Chem Commun, 1971, p 2).

ii RDX RESTRICTED

A report from Ohio State University Research Foundation in 1959 by P M Harris reported the results of an X-ray study of the structure of RDX (AFOSR-TR-59-165). While the overall picture of the structure may be correct, there are some features of the results which indicate that further refinement is necessary, eg the N-O bondlengths show an unacceptably large spread, and until such features are accounted for some reservations must be felt in accepting the results on the rest of the structure.

In an attempt to clarify this situation, a new set of X-ray data has been collected using the automatic diffractometer; a spherical crystal of purified RDX was employed, and the first set of data comprises approximately 2400 planes ($\sin\theta/\lambda_{max} = 0.704$) measured using molybdenum radiation; a further set has been collected out to $\sin\theta/\lambda_{max} = 0.766$, but a large proportion of the additional intensities are below the level of measurement. It is believed that these results will not be affected by radiation damage or absorption to a significant extent.

Least-squares refinement of the atomic positional and thermal parameters derived from the previous study has been started, using the new data, but only one cycle has so far been carried out. At a later stage the hydrogen atoms will be sought in difference-Fourier maps and the refinement will be continued.

iii Dimethylammonium Perchlorate UNCLASSIFIED

Stammler et al (Advances in X-ray Analysis, Vol 9, Plenum Press, 1966) reported the existence of two polymorphic forms of this substance, one stable above 38°C and having a tetragonal lattice, and another form stable below this temperature and having a lattice probably of monoclinic or lower symmetry.

This lower-temperature, optically isotropic form has been obtained in a well-crystallised form from slightly-warm benzonitrile, and has been shown to have a face-centred cubic lattice, space group Fd3m, with the exceptionally large lattice parameter of 30.6 Å; the density of this form has not yet been measured, but is less than 1.59, and this implies approx 190 molecules/unit cell, which compares with the maximum required by this space group, 192, for a molecule occupying a general position in the unit cell.

It has now been found that this cubic phase, on standing at 15°C, transforms to a different phase which is optically biaxial; X-ray powder patterns of this new phase have been obtained, and lath-shaped crystals of it, grown from benzonitrile at approximately 15°C, have been used to obtain single crystal data. This phase has an orthorhombic lattice, space group Pcn2 or Pcnm, with lattice parameters (Å) a = 9.2, b = 10.8, c = 6.3; gentle warmth causes it to transform to the cubic phase.

Analysis UNCLASSIFIED

Attempted Detection of 4-Aminodiphenylamine in 2-Nitrodiphenylamine by Chromatography

In studies of the purity of commercial 2-NDPA it was of interest to determine whether the material contained 4-aminodiphenylamine. Primary amines and other basic impurities were concentrated in the mother liquor by crystallisation of 2-NDPA from 90 per cent acetic acid. Neutralisation of the acid followed by ether extraction and evaporation further concentrated the impurities which were then dissolved in acetone. The solution was chromatographed on Kieselgel $HF_{254 + 366}$ using petroleum ether bp 40 - 60°C/ethyl acetate [4 : 1 (v/v)] developing solvent. An authentic sample of 4-aminodiphenylamine developed on the same plate was detected at R_f 0.1 as a blue spot on exposure of the chromatogram to hydrochloric acid vapour. Comparison of spot positions and sizes showed only a faint trace of material at a position corresponding to that of 4-aminodiphenylamine. Since the limit of detection was 4 μg it appears that this impurity, if it is present at all, accounts for less than 0.005 per cent of the 2-NDPA sample.

General Analysis UNCLASSIFIED

i Nitrogen Dioxide in Nitromethane

The current procedure has been improved by substituting Saltzman's reagent (sulphanilic acid/N-1-naphthylethylene diamine dihydrochloride) for sulphanilic acid and α-napthylamine as diazotisation and coupling agent. Less than 1 ppm nitrogen dioxide is easily detectable and the determination may be completed spectrophotometrically or by visual comparison with standards.

ii Rubbers

Several natural rubber samples were analysed to determine differences in type of rubber, fillers, accelerators, antioxidants and degree of cure. Pyrolysis/infrared spectroscopy was used to identify the rubber. This technique was also used to identify polyester and polyether polyurethanes, styrene-butadiene and nitrile-butadiene rubbers. The acrylonitrile to butadiene ratio in the last sample was estimated by comparison of spectra with standard samples.

Differential Scanning Calorimetry

Over 80 samples have been examined in studies on polymer glass transition temperatures, compatibility behaviour, decomposition temperatures, and impurity determination. The thermochemical behaviour of the initiatory explosives, tetrazene and cadmium azotetrazole, was observed under reduced pressure.

Boron UNCLASSIFIED

Total boron content can be determined either by fusion or by oxidation with a mixture of concentrated sulphuric and nitric acids ("nitrose method"), followed by titration of the boric acid formed. Amorphous boron can be determined from its reaction with silver perchlorate; it has been found that magnesium borate can interfere if amorphous boron is determined by reaction with dilute nitric acid. Combined amorphous and boron suboxide is best determined by oxidation with ceric sulphate. Crystalline boron is obtained by difference.

Delay Compositions UNCLASSIFIED

Schemes have been tested for the analysis of delay compositions containing lead- and/or barium chromates mixed with metallic powders.

Weight loss separation (treating the sample first with sodium hydroxide to dissolve the lead chromate, then with perchloric acid to dissolve barium chromate) is accurate to ± 1%. Greater accuracy (± 0.2%) is obtained by precipitating the lead or barium from solution, with concentrated sulphuric acid and determining as the sulphate. A scheme has been developed for identification and estimation of the metal; it involves fuming with acid followed by treatment with fusion mixture.

LIST OF TECHNICAL REPORTS AND NOTES
ISSUED BY ERDE IN THE PERIOD 1 7 70 to 31 12 70

Classification Key:
S = SECRET
C/D = CONFIDENTIAL/DISCREET
C = CONFIDENTIAL
R = RESTRICTED
U = UNCLASSIFIED
No classification = UNLIMITED

Classification of title shown in brackets

Number	Classi-fication	Author	Title
TR 4	C	E A Baker	Very Fast Burning Cordites (U)
TR 8	R	J W Grindlay	The Preparation and Properties of Polyepoxides (U)
TR 21	R		The Sensitiveness of Barium Styphnate - Summary of Tests at ERDE, RARDE and AWRE (U)
TR 24	U	A J Dedman & A L Lovecy	Electrothermal Ignition and Propagation in Solid Explosives: Part 9: Proving Performance by Working Metals Under Water
TR 28	U	M J Healey & W A W Smith	Smokeless Rocket Propellants: Pyrolysis of, and Smoke Production from, some Organic Components
TR 30	C/D	C G Lawson	Low Energy Solid Propellants: Problems of Steady Burning. Safety of Power Cartridges (U)
TR 34	U	J F Sumner	The Development of a Friction Sensitiveness Test for Explosives and Propellants Part 1: Investigation of Design Requirements
TR 35	U	P J Hart & J Scrivener	CTPB Propellants: Part 3: Adhesion to Various Rubbers
TR 38	C	E A Baker	Solventless Rocket Cordites. Effects of Wet Paste Storage on Ballistics (U)
TR 42	C	F H Doe	Castable Rubber Explosives: Polyurethane/RDX Compositions (U)

LIST OF TECHNICAL REPORTS AND NOTES
ISSUED BY ERDE IN THE PERIOD 1 7 70 to 31 12 70 (cont'd)

Number	Classi-fication	Author	Title
TR 43	C/D	R T M Fraser Diane Howse & N C Paul	The Preparation of Basic Copper Salicylate: Part 2: Minor Phases (U)
2/69/1	S/D		Technical Report for the Period 1 7 1969 to 31 12 1969 Explosives
1/70/1	S		Technical Report for the Period 1 1 1970 to 30 6 1970 Explosives
TN 12	R	C H Miller & A M Whittaker	Manufacture of Fuseheads Using ERDE Compositions: Part 3: Performance of Fuseheads and Equipment for Assessment (U)
TN 13		R W Bryant W A Dukes & R A Gledhill	Tensile Properties of Plastic Propellants RD2435, E3342 and E4058
TN 18		W E Batty J A Bell Patricia Clements D F Debenham J R C Duke & D C Mullenger	Some Solvates of BSX
TN 19		A K Clarke J M Jenkins J G Rankine	Precipitation Unit for Initiators (600 ml Gross Capacity Vessel)
TN 20		H C Turner	A Design for a Hydrostatic Pressure Chamber for Calibrating Piezoelectric Pressure Transducers
TN 24	U	R B Holt & C G Lawson	A Dual Thrust, Single CDB Propellant Rocket Motor: Preliminary Static Firings

PAPERS BY ERDE STAFF PUBLISHED IN THE OPEN LITERATURE
IN THE PERIOD 1 7 70 TO 31 12 70

Authors	Title	Journal Reference
R P Ayerst M I Phillips	Crystallization from Agitated Ammonium Perchlorate Solutions: Some Aspects of Nucleation and Growth Symposium on Industrial Crystallization organised by the Institute of Chemical Engineers, 17th April 1969, p 56	
R P Ayerst M I Phillips	A Study of the Operation of a Pilot-Scale Forced Circulation Evaporating Crystallizer for Ammonium Perchlorate Symposium on Industrial Crystallization organised by the Institute of Chemical Engineers, 17th April 1969, p 177	
A L Lovecy	The Role of Shock in the Initiation of Secondary Explosives	Combust Flame, 1970, 15, 299
F I H Tunstall	The Determination of Gases Evolved from Stored Propellant Compositions by Gas Chromatography	Chromatographia, 1970, 3, 411 (ERDE Offprint No 15)

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Date of Search: 18 November 2008

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Title: Explosives: technical report for period 1 July 1970 - 31 December 1970
Availability Open Document, Open Description, Normal Closure before FOI Act: 30 years
Former reference (Department) ERDE TR/2/70/1
Held by The National Archives, Kew

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