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RECRYSTALLISATION OF RDX AT W.R.E.

I.R. JOHNSTON, R.H. WELDON and G.M. HENSEL

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SUMMARY

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Recrystallisation techniques for producing small scale batches of the explosive RDX in various particle sizes have been investigated. A plant has been set up and procedures have been established for the recrystallisation of RDX Class A, C, D or E from Australian manufactured RDX using either cyclohexanone or acetone. The potential capability for producing RDX in other particle sizes is also demonstrated.

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1. INTRODUCTION

RDX (cyclotrimethylene trinitramine) is an important explosive ingredient of many warheads due to its high performance and good stability at high temperatures. It is often desirable to have a high concentration of RDX in the fill and this high solids loading is achieved by blending RDX of various particle sizes. Those sizes of popular interest and given in the DEF(AUST) 382A Specification are:

RDX	nominal average particle size (μm)
Class A	220
Class C	330
Class D	600 - 700
Class E	13

It is possible to produce RDX to a specified class by treating RDX with solvents under different conditions, e.g., Class A by recrystallising from a rapid azeotropic distillation of a cyclohexanone/water system and Class E by dropping a warm solution of RDX in acetone into an excess of cold water.

Work on the cyclohexanone system is reported by Mills(ref.1) and Walsh(ref.2). A summary of overseas work involving various operating conditions is given by Tozer(ref.3).

This paper describes facilities set up at W.R.E. and sets down the operating procedures required for producing RDX Classes A, C and D using cyclohexanone and RDX Class E using acetone.

2. MATERIALS

2.1 Crude RDX

The crude RDX was manufactured by the Woolwich process at Albion Explosives Factory (AEF) and supplied in plastic drums containing 20 kg RDX as a water slurry. Prior to a cyclohexanone recrystallisation it was necessary to treat this material to remove insoluble impurities. The procedure was similar to the normal acetone recrystallisation process except that the solvent recovery and stripping stages were combined (see Appendix III).

2.2 Cyclohexanone (CX)

Cyclohexanone, commercial grade, was obtained from Explosives Factory, Maribyrnong (EFM) ex Swift & Co., South Melbourne. Before use, the cyclohexanone was purified by distillation as a low temperature azeotrope with water.

Under certain conditions, e.g., high nitric acid content of crude RDX or during the higher temperature stripping stage of the recrystallisation process, a cyclohexanone condensation dimer can be produced. The dimer discolours and contaminates the recrystallised RDX such that it will not pass the standard vacuum stability tests. Contamination was found to be avoided by carrying out the following steps:

- (a) decanting as much water as possible from the crude RDX,
- (b) discarding the first distillate portion which had flushed the condensers from the previous run,
- (c) discarding all stripping distillate, and
- (d) alcohol washing of recrystallised RDX during filtration.

2.3 Acetone

Acetone, commercial grade, was obtained from Monsanto, Melbourne. The acetone was purified before use by distillation, then subsequently recovered by azeotropic distillation with water.

2.4 Water

Distilled water was used in all recrystallisation processes.

3. PLANT

3.1 Recrystalliser plant

The plant was modelled on the one used by Mills(ref.1) and is depicted schematically in figure 1 and photographically in figure 2. The plant was assembled using QVF (Jobling) industrial glassware supported within a steel piping framework. The recrystalliser was a 100 l graduated cylindrical glass vessel fitted with a drain valve and a stainless steel cover plate which supported a stainless steel coiled heat exchanger and a stainless steel steam sparger pipe. The cover plate also incorporated a vapour outlet fitted with a glass reflux divider head, a water seal through which passed the shaft of an air powered stirrer and a feed opening with a cover plate through which passed the shaft of a mercury-in-steel dial thermometer (20° to 110°C). Above the reflux head was a large capacity glass condenser above which was a smaller secondary glass condenser.

The outlet side of the reflux head was fitted with a valve followed by a polytetrafluoroethylene (PTFE) flexible connector and a short section of pipe incorporating a "U" shaped sampling tube and valve. The reflux head also incorporated the stainless steel sensor bulb of a mercury-in-steel capillary dial thermometer (20° to 110°C) to monitor vapour temperatures during distillation.

The sampling section was connected, via a small condenser and a 2-way valve, to a 100 l graduated spherical glass receiver fitted with a drain valve. This valve was connected to a hand pump so that the contents recovered in the receiver could be transferred to the recrystalliser for a subsequent run. A hand pump was also provided to transfer the commercial grade solvents from drums to the recrystalliser for purification by distillation.

3.2 Dissolver

The preparation of RDX Class E and purified RDX (paragraph 2.1) required a separate dissolver vessel which consisted of an 80 l stainless steel vessel fitted with a stainless steel cover plate supporting a stainless steel coiled heat exchanger. The cover plate also incorporated a vapour outlet fitted with a medium capacity glass condenser, a PTFE seal through which passed the shaft of an air powered stirrer and a feed opening with a cover plate through which passed the shaft of a dial thermometer. The bottom outlet was fitted with a stainless steel drain valve and a length of nylon tubing so that the contents of the vessel could be "dropped", at a controlled rate, into the recrystalliser.

3.3 Control panel

Steam, water and air were piped to a control panel mounted adjacent to the plant. Regulation of steam and water pressures to the heat exchangers, sparger and condensers and also air to the stirrer motors was accomplished from the control panel. The vapour temperature at the reflux head was also monitored at the panel.

3.4 Plant operation

Calculation procedures and operating instructions for the recrystallisation of RDX from either cyclohexanone or acetone are set out in Appendices II and III respectively. The control panel gauges were graduated in Imperial units and, to avoid confusion, these units are quoted when referring to gauge readings.

3.5 Filtration equipment

All recrystallised RDX was filtered and washed several times. Two large (240 mm) Buchner funnels were used with No. 540 Whatman filter papers in conjunction with two laboratory water pumps.

3.6 Sieving of RDX

Sieve analysis of recrystallised RDX was carried out as required by Annex L of DEF(AUST) 382A. A standard jet of distilled water was used to wash the RDX successively through a nest of five 100 mm diameter stainless steel sieves, made up with the following mesh sizes; BSS 18 (840 μm), BSS 30 (500 μm), BSS 52 (300 μm), BSS 100 (150 μm) and BSS 200 (75 μm). The Specification lists time limits for washing on each sieve, but more consistent results were obtained when washing was continued until no more appreciable quantities of RDX passed through each sieve. After drying, the amount of RDX retained on each sieve was weighed and expressed as a cumulative percentage.

4. RECRYSTALLISATION USING CYCLOHEXANONE

4.1 General

There are two distinct techniques for the recrystallisation of RDX from cyclohexanone. A partial solution method is preferred in the U.S.A., mainly to reduce batch times and increase overall production(ref.3). In the U.K. a complete solution method is adopted to reduce the total acidity and provide greater control over the final particle size(ref.2). Walsh(ref.2) preferred the complete solution technique and recommended this be adopted at Albion Explosives Factory. This method was later used at Explosives Factory, Maribyrnong (EFM) as reported by Mills(ref.1). In his work Mills studied four main variables, viz., RDX to cyclohexanone ratio, RDX to water ratio, distillation rate and stripping time and concluded that only the variation of distillation rate had any significant effect on the particle size and distribution of the recrystallised RDX.

Solubility tests confirmed the findings of references 1 and 2 in that the limit of solubility of RDX in cyclohexanone (CX) was in the order of 1:2.6. The initial run at W.R.E., batch CX1, used an overall mass ratio RDX/CX/water of 1:2.9:7.1, identical to that used by Mills(ref.1). As it seemed that the RDX did not completely dissolve, due to the presence of the water, in the allotted time of 45 min, an overall mass ratio of 1:3.2:7.1 was used in later batches. The use of excess cyclohexanone also ensured that the distillation rate was stabilized before recrystallisation began.

A total of 21 batches was recrystallised from cyclohexanone. The first 5 were investigational runs to determine the parameters involved in operating the plant. The RDX used for these batches was not purified (see paragraph 2.1) and the results, reported for reference only, were not considered. Of the remaining batches, 11 were of either Class A, C or D and 5 were of various particle sizes and distributions. The operating conditions and results for all individual batches are given in Appendix I. The specific requirements to produce RDX to either the Class A, C or D Specifications of DEF(AUST) 382A are given in the following paragraphs.

4.2 RDX Class A

After the experimental runs, a set of operating conditions required to produce RDX Class A, average particle size 220 μm , was determined. Appendix I lists the individual batches of recrystallised RDX, CX6, CX11, CX12, CX13 and CX19, which were produced by similar operating conditions and had similar properties. These batches were blended to produce a 40 kg lot of RDX Class A. The main operating conditions were:

Distillation Rate : 710 to 810 ml/min
Stirrer Speed : 150 - 170 rev/min
Sparger : operated continuously during distillation
at 35 kPa (5 lb/in²)

While the average particle size of the blended RDX was within the Class A Specification of DEF(AUST)382A, the amounts retained on the BSS100 and BSS200 sieves were slightly higher than the Specification values (see Appendix I). However, for W.R.E. purposes, this particular RDX was termed Class A. (See also figure 3).

4.3 RDX Class C

Batches of RDX Class C (CX8, CX16, CX17 and CX18) were produced by another set of operating conditions and had similar properties as detailed in Appendix I. These batches were blended to produce a 32 kg lot of RDX Class C with an average particle size of 330 μm . The main operating conditions were:

Distillation rate : 280 to 340 ml/min
Stirrer Speed : 130 rev/min
Sparger : not used during distillation

The overall particle size and distribution of the blended RDX was within the Class C Specification of DEF(AUST)382A. (See Appendix I and figure 4).

4.4 RDX Class D

Two batches which met the RDX Class D Specification of DEF(AUST)382A were produced. The first batch, CX20, was slightly more coarse with an average particle size of 766 μm compared to the second batch, CX21, value of 667 μm (see Appendix I and figure 5). The less coarse material is preferred at W.R.E. The main operating conditions for RDX Class D were:

Distillation rate : 150 to 160 ml/min
Stirrer Speed : 100 rev/min
Sparger : not used during distillation

4.5 RDX of other particle sizes

Further variations of distillation rate and/or stirrer speed yielded recrystallised RDX of different particle sizes which did not come within any class Specification of DEF(AUST)382A. The details of the batches concerned (CX7, CX9, CX10, CX14 and CX15) can be found in Appendix I.

Batches CX7, CX9 and CX10 used distillation rates or stirrer speeds between those used to produce Class A and C and it was found that the particle sizes of the recrystallised RDX were also between those two Classes. Batches CX14 and CX15 (and also CX19 mentioned in para 4.3) used a partial solution technique in an endeavour to reduce overall particle size and, while this was successful with batch CX14, two attempts to reproduce it with batches CX15 and CX19 failed for reasons unknown.

4.6 Discussion

Figures 3, 4 and 5 indicate the difference in particle sizes and distributions between the three classes of recrystallised RDX, viz., Class A (batch CX6), Class C (batch CX8) and Class D (batch CX21). These, taken with the sieve analysis results, demonstrate the narrow particle size distribution achieved in all batches, particularly those of Class A and C. The Class D material appears to contain some coarser particles where two or three individual crystals have agglomerated. The close control exercised over the distillation rate of each batch had allowed the recrystallisation process to proceed at a uniform rate. This also perhaps explains the uniformity of crystal shape noted in all batches.

RDX recrystallised from cyclohexanone, as covered in this report, varied in average particle size from 188 μm (batch CX14) to 766 μm (batch CX20). As batch CX14 could not be reproduced, the finest material that could be reliably produced was in the order of 220 μm . The other various sizes obtained demonstrated the potential capability for producing RDX of virtually any particle size between the above limits. It is also feasible that with more efficient distillation and/or partial solution techniques, finer RDX may be produced. The production of coarser RDX could be carried out, within practical limits, with lower distillation rates and stirrer speeds. However this material would have very limited use in present polymer bonded explosive (PBX) manufacture.

Apart from sieve analysis, the only other tests performed on the RDX at W.R.E. to date have been total and occluded acidity and vacuum stability at 100°C. All results have been within the respective specifications as quoted in DEF(AUST)382A. Future tests on vacuum stability will be carried out at the required 150°C when the equipment becomes available.

It is not intended to discuss the probabilities of how the shape of the recrystalliser vessel and the geometrical layout of the heating coils, stirrer and sparger contribute, one way or another, to the properties of the recrystallised RDX. However, large scale production vessels utilize stainless steel tanks fitted with panel heaters, thereby resulting in less turbulence(ref.3). A problem with the glass recrystalliser vessel used in this work was the length of connecting "spouts" between the bottom of the vessel and the drain valve. This held an amount of "stagnant" RDX which did not undergo normal stirring, heating or stripping. However, because this amount was only 6 to 8% of the total, no effort to overcome this problem was warranted.

5. RECRYSTALLISATION USING ACETONE

5.1 General

Recrystallisation from acetone produces either a fine RDX Class E or a slightly coarser Class F, depending on the mass ratio of RDX/acetone. Only the finer Class E required by present PBX work was produced during this current program. Compared to the recrystallisation using cyclohexanone, the process was straightforward with only one variable, the RDX/acetone ratio, controlling the final particle size. The limit of solubility of RDX in acetone was 1:5.6, which is the ratio reported to be used to produce Class F(ref.4). Class E, however, was produced with an excess of acetone, the ratio being 1:8.

The RDX Grade IA (4.5 kg dry weight) was dissolved in an acetone/water solvent by refluxing for 30 min in the dissolver vessel then dropped, with filtering, at a controlled rate into a specific quantity of water in the recrystalliser, during which time the temperature did not exceed 35°C. The

initial overall mass ratio was the same as that used by EFM(ref.4), i.e. RDX/acetone/water of 1:8.8. However due to a low recovery rate of 75% with the initial batch the ratio was changed to 1:8:12, the extra water assisting in two ways. Firstly, after dropping, the resultant liquor had a higher water content, the RDX was therefore less soluble and a higher yield of 85% of RDX was obtained. Secondly, the greater volume of liquid made it easier to control the temperature below 35°C during dropping.

As much as possible of the supernatant liquid was then siphoned off and the recrystallised RDX, with the remaining liquid, stripped for 30 min at 100°C to remove the last traces of acetone. The product was filtered by the Buchner process and washed with distilled water.

5.2 RDX Class E

A total of 9 batches was prepared to produce approximately 34 kg of RDX Class E. All batches were within the DEF(AUST)382A Class E Specification of 3% maximum retained on BSS 350 sieve, and all average particle sizes, as determined by Fisher method, were between 12.8 µm and 13.9 µm.

5.3 RDX Feedstock

22 more batches (95 kg) were prepared using the modified Class E procedure, as mentioned in paragraph 2.1, for use as feedstock for RDX recrystallised from cyclohexanone. Extra water was not necessary for this process and the mass ratio reverted to 1:8:8. The combination of the solvent recovery and stripping stages increased the RDX recovery rate to 96% but also increased the average particle size to 25 µm. While this was outside the Class E Specification, it could be useful if a slightly coarser grade of fine RDX were required.

5.4 Temperature control

The importance of maintaining the temperature below 35°C(ref.4) during the dropping stage was not readily confirmed except perhaps for safety reasons. During one particular batch, the temperature was allowed to rise to 40°C with no apparent affect on yield or particle size. However, to maintain consistency and as no real problems arose in keeping below 35°C, this condition was maintained.

6. CONCLUSIONS

The general findings of the work by Mills(ref.1) and Walsh(ref.2) and, to some extent, the overseas work reported by Tozer(ref.3) were confirmed, viz., that the distillation rate, with or without sparging, was one important variable in the cyclohexanone process. The work with the W.R.E. recrystallisation plant has shown that the stirrer speed was also important, especially when the finer classes of RDX were required. Mass ratios had very little effect and stripping time no effect, apart from the removal of the last traces of cyclohexanone. With set operating conditions, RDX was recrystallised reliably with reproducible particle sizes and distributions.

Prior to the cyclohexanone procedure, the RDX Grade IA should be purified by an acetone recrystallisation process.

Alcohol washing of products from the cyclohexanone recrystallisation process was found to assist in removing final traces of cyclohexanone.

RDX Class E was reliably produced from acetone with much less control required compared to the cyclohexanone process.

It is feasible that a whole range of alternative particle size RDX could be produced by changing operating conditions, thereby permitting better control of solids packing and mix viscosity in PBX production.

REFERENCES

No.	Author	Title
1	Mills, P.A.	"Investigation of the Recrystallisation of RDX from Cyclohexanone". Work Report No. EFM 1974/4
2	Walsh, J.	"The RDX Recrystallisation Process - Part of Engineering Project No. 628 - Additions to RDX Plant Albion".
3	Tozer, N.H.	"Report on Overseas Tour of Duty". Work Report No. AEF 1969/2, May 1969
4		Unpublished Laboratory Notes from Explosives Factory, Maribyrnong

APPENDIX I

RECRYSTALLISATION OF RDX FROM CYCLOHEXANONE -
OPERATING CONDITIONS AND RESULTS

I.1 Batch grouping

The grouping of batches, as followed in the main text and in this Appendix, is given below, with a summary of the main purpose for processing each batch. The details of each batch are given in the Table at the end of this Appendix.

Batch	Text ref.	Appendix ref.	Remarks
CX1-CX5	4.1.3	I.2	Not considered in final analysis because of use of inferior RDX feedstock
CX6, CX7, CX8		I.3	Variation of distillation rate
CX9, CX10	4.5	I.4	Variation of stirrer speed
CX11, CX12 CX15	4.2	I.5	Production of RDX Class A
CX14, CX15 CX19	4.2 and 4.5	I.6	Partial solution technique
CX16, CX17 CX18	4.3	I.7	Production of RDX Class C
CX20, CX21	4.4	I.8	Production of RDX Class D

I.2 Batches CX1 to CX5

Because batches CX1 to CX5 used impure RDX Grade IA, the results obtained were not directly compared with later batches. The operating conditions of batch CX1 were similar to those used for a "standard run" at EFM(ref.1). The distillation rate was maintained at 325 ml/min with no sparging. The product had a slightly larger particle size than that obtained by EFM but was found to be within the limits of the coarsest recrystallised RDX (Class D) covered by Specification DEF(AUST)382A.

In an immediate attempt to produce finer crystals, live steam sparging was introduced in batch CX2 with an increased distillation rate of 713 ml/min. To ensure complete dissolution of the RDX, the RDX/cyclohexanone (CX) ratio was increased to 1:3.2. During filtration, the recrystallised RDX was washed with absolute ethanol in an attempt to remove oily droplets noted on the filtered material and identified as cyclohexanone. Tests showed this technique was successful and the procedure was adopted for all future runs. The particle size was found to be much less than that for batch CX1 with a distribution close to the DEF(AUST)382A Class A Specification.

Batch CX3 was an attempt to confirm that the distillation rate could be used to control particle size. A distillation rate (485 ml/min), between those used in the first two batches, was used with sparging. However, the particle size was not as large as expected when compared with either batch CX2 or its duplicate run, batch CX4. The reason for this was not established but it was observed that the RDX Grade IA material contained differing amounts of impurities, drum to drum, and only when these

impurities were eliminated as mentioned in paragraph 2.1, were consistent results obtained.

Batch CX4 was a repeat of batch CX2 and the particle sizes and distributions of the two batches were found to be similar.

A partial solution technique was used in batch CX5 to determine whether this had any significant affect on particle size or distribution. A mass ratio of RDX/CX, 1:2.3 was used, leaving 12% of the RDX undissolved. The distillation rate was 760 ml/min with sparging. Although the particle size was slightly larger than batches CX2 and CX4, no conclusions were drawn until later batches were prepared using the purified RDX as feedstock.

I.3 Batches CX6, CX7 and CX8

The introduction of larger batches (8 to 8.5 kg dry RDX) and the use of purified RDX as feedstock occurred with batch CX6. A higher stirrer speed of 150 rev/min was inadvertently used - previously 120 rev/min. This occurred as a result of an air motor overhaul and reintroduction without speed check. The three batches were run to specifically determine the affect of distillation rate on particle size. Common operating conditions were standard mass ratio RDX/CX/water of 1:3.2:7.1, and stirrer speed of 150 rev/min. The distillation rates were 710, 515 and 339 ml/min respectively, with the former two using live steam sparging. The table in this Appendix shows that the particle size increased as distillation rate decreased with batch CX6 almost to Class A Specification and batch CX8 almost to Class C Specification of DEF(AUST)382A. Batch CX6 which was run under similar conditions to batches CX2 and CX4, except that the impurities were not present and the stirrer speed was higher, yielded a finer product.

I.4 Batches CX9 and CX10

These two batches were run identical to batch CX6 except that the stirrer speed was restored to the original 120 rev/min. The particle sizes and distributions were similar to those of batches CX2 and CX4 which demonstrated that the finer product of batch CX6 was probably due to the increased stirrer speed. This led to the observation that batches of this size (8.5 kg dry RDX) required approximately 150 rev/min to "hold" all of the RDX in suspension in the liquid. It was probable that, when the RDX crystals were contained in a slower moving or stagnant mass, due to the stirrer speed being less than 150 rev/min, additional growth occurred on these crystals resulting in larger particle sizes. Conversely, when the stirrer speed was 150 rev/min, the majority of the crystals was kept moving with the greater probability of a larger number of smaller crystals being formed. At stirrer speeds in excess of 150 rev/min, no further decrease in particle size was noticed. (See batches CX12 and CX13).

I.5 Batches CX11, CX12 and CX13

It was desired to produce a large quantity of RDX, similar to Class A, as produced in batch CX6. Three more batches were processed with similar operating conditions except for the higher stirrer speed of 170 rev/min for batches CX12 and CX13. Sieve analysis results showed that all four batches (CX6, CX11, CX12 and CX13) were of very similar particle size and distribution.

I.6 Batches CX14, CX15 and CX19

Partial solution techniques were again investigated as in batch CX5. The mass ratio was the same but the stirrer was held at a higher speed of 160 rev/min. The results of batch CX14 were encouraging in that a finer product was obtained than for batch CX6, with the particle size and distribution well within the Class A Specification of DEF(AUST)382A.

However, two attempts to reproduce batch CX14 failed, when batches CX15 and CX19 produced a particle analysis very similar to batch CX6. The reasons for batch CX14 being finer and difficult to reproduce cannot be satisfactorily explained at this stage.

I.7 Batches CX16, CX17 and CX18

As mentioned in paragraph I.3, batch CX8 was almost to Class C Specification. It was also desired to have a large quantity of RDX Class C for PBX work and consequently batches CX16, CX17 and CX18 were produced. Slightly lower distillation rates and stirrer speeds than for batch CX8 were used to ensure the recrystallised RDX was within the Class C Specification of DEF(AUST)382A. In the event, RDX Class C was successfully produced.

I.8 Batches CX20 and CX21

These two batches were attempts to produce the very coarse RDX Class D. The operating conditions for batch CX20 were a distillation rate of 106 ml/min with no sparging and a stirrer speed of 80 rev/min. Because the distillation rate was slow, less RDX (6 kg) had to be used to keep within normal batch times. To maintain the liquid level above the heating coils, extra water was required and this led to a mass ratio RDX/CX/water of 1:3.2:9.0. It was considered that the extra water would not affect the recrystallised RDX as the cyclohexanone could only be distilled with water as the normal azeotrope. (See also Mills(ref.1)). In appearance the product was similar to large crystals of sugar; while it was within the Class D Specification, was too coarse for PBX work.

An increased distillation rate of 154 ml/min for batch CX21 enabled the normal mass ratio to be used. The stirrer speed was 100 rev/min with no sparging during distillation. The particle size and distribution of the recrystallised RDX were within the Class D Specification and compatible with the particular PBX manufacturing requirement.

RECRYSTALLISATION OF RDX FROM CYCLOHEXANONE - OPERATING CONDITIONS AND RESULTS

Batch No.	Mass ratio		Distillation rate (ml/min)	Stirrer speed (rev/min)	Sieve analysis % retained					Wt. Av. part. size (µm)	Remarks	
	RDX	CX			Water	18	30	52	100			200
CX1	1	2.9	7.1	325	120	3.6	67.2	89.7	98.0	99.0	571	
CX2	1	3.2	7.1	713	120	0.1	0.6	20.6	90.9	98.3	252	
CX3	1	3.2	7.1	485	120	0.0	0.3	26.0	95.5	98.4	266	
CX4	1	3.2	7.1	760	120	0.0	0.7	24.9	96.2	99.3	266	
CX5	1	2.3	7.1	760	120	0.1	1.4	35.6	93.3	98.7	283	
CX6	1	3.2	7.1	710	150	0.0	0.3	7.7	76.7	95.6	212	Class A
CX7	1	3.2	7.1	515	150	0.0	0.5	20.3	96.8	97.7	257	
CX8	1	3.2	7.1	339	150	0.1	2.8	48.6	93.1	98.1	309	Class C
CX9	1	3.2	7.1	780	120	0.0	0.6	28.0	91.0	98.4	265	
CX10	1	3.2	7.1	774	120	0.0	0.8	28.1	90.4	97.8	264	
CX11	1	3.2	7.1	760	150	0.0	0.2	8.3	81.0	96.3	216	Class A
CX12	1	3.2	7.1	760	170	0.0	0.6	9.5	81.1	97.9	221	Class A
CX13	1	3.2	7.1	810	170	0.0	0.2	8.3	79.9	97.8	217	Class A
CX14	1	2.3	7.1	808	160	0.3	0.8	3.0	60.6	96.6	188	
CX15	1	2.3	7.1	826	160	0.3	0.6	3.7	83.2	99.0	214	
CX16	1	3.2	7.1	312	130	0.2	7.2	53.8	90.5	98.2	329	Class C
CX17	1	3.2	7.1	279	130	0.8	14.3	63.8	94.0	99.1	371	Class C
CX18	1	3.2	7.1	296	130	0.5	8.8	50.8	89.5	98.3	328	Class C
CX19	1	2.3	7.1	787	170	0.1	0.9	9.6	82.2	96.8	225	Class A
CX20	1	3.2	9.0	106	80	71.0	90.4	96.4	99.2	99.8	766	Class D
CX21	1	3.2	7.1	154	100	46.0	72.8	88.3	99.0	100.0	667	Class D
DEF (AUST) 382A SPECIFICATION												
Class A												
Class C												
Class D												
EPM RDX CLASS C												
EPM RECRYSTALLISED RDX(ref.1)												
EPM RDX CLASS A (by sieve sizing)												
					0-4	0-20	10-70	55-95	110-270			
					max	50-70	70-90	min	310-460			
					1			80				
					0.1	74.0	98.0	99.0	414			
					0.0	81.0	94.0	95.0	500			
					0.0	17.0	36.0	90.0	210			

APPENDIX II

RECRYSTALLISATION OF RDX FROM CYCLOHEXANONE -
CALCULATION AND OPERATING PROCEDURES

II.1 Calculation procedures

Let mass ratio RDX/CX/water = 1:a:b

A = weight of dry crude RDX (kg)

B = Aa = weight of cyclohexanone (kg)

C = Ab = weight of distilled water (kg)

x = moisture content of RDX (%)

F = volume of aqueous phase available
in receiver (litres)

Calculation of charges

$$\text{Wet RDX} = \frac{100 A}{100-x} \text{ (kg)} \quad (X)$$

Aqueous phase required

$$= 1.11 \left(C - 0.064 B - \frac{Ax}{100-x} \right) \text{ litres} \quad (D)$$

Cyclohexanone phase required

$$\text{If } D \leq F \quad = 0.11 \left(10 B - C + \frac{Ax}{100-x} \right) \text{ litres} \quad (E)$$

$$\text{If } D > F \quad = 1.12 B - 0.10 F \text{ litres} \quad (G)$$

Make-up water required (only when $D > F$)

$$= C - 0.91 F - 0.06 G - (X-A) \text{ litres} \quad (H)$$

NOTE: The above calculations were based on sufficient cyclohexanone phase always being available. Commercial cyclohexanone required purification by low temperature azeotropic distillation with water, thereby producing the appropriate phase. The distillation also served to flush out the recrystalliser system.

II.2 Operating procedures

II.2.1 Purified RDX was supplied under water in plastic drums. During storage the RDX settled sufficiently so that when the surface water was carefully decanted, the remaining RDX/water had the desired paste-like consistency. Ideally 30-40% water was present as determined by weight difference after oven drying a 20 g sample at 105°C for 2 hours. From the moisture content, the amount of wet RDX containing the desired amount of dry RDX was determined.

II.2.2 The volumes of water and cyclohexanone required were determined as outlined in Part 1 of this Appendix. Approximately three quarters of the water and all of the cyclohexanone were pumped into the recrystalliser. The wet RDX was loaded into the recrystalliser using the remaining water and/or make-up water to rinse the container.

- II.2.3 The vessel was closed and the stirrer turned on and adjusted to the desired speed. The cooling water and heat exchanger steam valves were carefully turned on, in the former case to remove air from the condensers and in the latter to remove water from the steam lines. When the water and steam flows became smooth, the valves were fully opened.
- II.2.4 When the liquid began to boil, the first 500 ml of distillate was discarded via the sample valve. The system was then allowed to gently reflux to dissolve the RDX. After 45 min the reflux head valve was opened and the distillate collected in the receiver.
- II.2.5 If steam sparging was required, the sparger valve was opened, and together with the heat exchanger steam, adjusted so as to produce the desired distillation rate as determined by recording the time every 4 l. All steam pressures, temperatures, stirrer speeds, distillation rates and volumes were recorded on the appropriate log sheet.
- II.2.6 When the constant temperature of the cyclohexanone/water azeotrope had ceased and the vapour temperature just began to rise, all further distillate was discarded via the two-way valve, as it was likely to contain the contaminant cyclohexanone condensation dimer (CCD).
- II.2.7 This was also the commencement of the stripping stage. If the live steam sparger was not already on, the appropriate valve was opened and together with the heat exchanger steam, adjusted so that the original distillation rate was followed as closely as possible.
- II.2.8 After 55 min stripping the heat exchanger steam, sparger steam and stirrer were turned off and the sparger vent opened to prevent siphoning.
- II.2.9 When the system had cooled sufficiently, the condenser water was turned off and all volumes recorded. RDX was washed from the re-crystalliser walls with distilled water.
- II.2.10 As much water as possible was siphoned off before the RDX/water mixture was drained from the vessel.
- II.2.11 The remaining water was filtered off using Buchner funnels and the RDX washed with absolute ethyl alcohol (2 l/funnel), followed by distilled water. The RDX was stored under water in high density plastic drums in the appropriate magazine.

APPENDIX III

RECRYSTALLISATION OF RDX FROM ACETONE -
CALCULATION AND OPERATING PROCEDURES

III.1 Calculation procedures

Let mass ratio RDX/Acetone/water = 1:a:b

A = weight of dry crude RDX (kg)

B = Aa = weight of acetone (kg)

C = Ab = weight of distilled water (kg)

x = moisture content of RDX (%)

y = S.G. of acetone/water solvent

Calculation of charges

$$\text{Wet RDX} = \frac{100 A}{100 - x} \text{ (kg)}$$

Acetone/water solvent required

$$= \frac{0.26 B}{1 - y} \text{ litres}$$

Water required in recrystalliser

$$= C - B \left(\frac{0.26}{1 - y} - 1.26 \right) - \frac{Ax}{100} \text{ litres}$$

NOTE: The above calculations were based on sufficient acetone/water solvent always being available. The solution was recovered from previous batches by azeotropic distillation up to 75°C.

III.2 Operating procedures

III.2.1 Crude RDX Grade IA was supplied under water in plastic drums. During storage the RDX settled sufficiently so that when the surface water was carefully decanted, the remaining RDX/water had the desired paste-like consistency. Ideally 20 to 25% water was present as determined by weight difference after oven drying a 20 g sample at 105°C for 2 hours. From the moisture content, the amount of wet RDX containing the desired amount of dry RDX was determined.

III.2.2 Approximately 50 litres of acetone/water solvent was pumped from the receiver into the recrystalliser and stirred for 2 min at 80 rev/min. The S.G. of the solvent solution was determined using a hydrometer. (S.G. reading should not be greater than 0.830).

III.2.3 The volumes required of acetone/water solvent and of water were determined as outlined in Part 1 of this Appendix. Approximately three quarters of the acetone/water solvent was transferred to the dissolver, followed by the RDX, using the remaining solvent to rinse the RDX container. The vessel was then closed and the stirrer turned on.

III.2.4 Excess solvent was drained from the recrystalliser and the required volume of water added.

- III.2.5 The dissolver condenser cooling water and heat exchanger steam valves were carefully turned on, in the former case to remove air from the condenser and in the latter to remove water from the steam lines. When the water and steam flows became "smooth" the water valve was fully opened and the steam pressure adjusted to 10 lb/in².
- III.2.6 The heating process was carefully monitored by observing the temperature and at 55°C the steam valve was adjusted so that it was only just "cracked" open. Refluxing commenced at 57 to 58°C and was observed by the condensing vapours in the lower section of the condenser. At all times it was ensured that the condensing vapours did move more than half way up the condenser. Refluxing was continued for 30 min when the dissolver heat exchanger steam and stirrer valves were turned off.
- III.2.7 The contents of the dissolver were "dropped" into the water in the recrystalliser, with stirrer operating, via a nylon hose and filter system, the flow being regulated so that the recrystalliser temperature did not exceed 35°C.
- III.2.8 The stirrer was turned off and, when the precipitated RDX had settled, the supernatant liquid was siphoned off (for later recovery) and distilled water added to bring the liquid level up to 40 litres.
- III.2.9 The recrystalliser stirrer, heat exchanger steam and condenser cooling water valves were turned on and when the temperature reached 100°C, the sparger steam valve was opened and the stripping stage continued for 30 min. All distillate was discarded.
- III.2.10 All steam, stirrer and cooling water valves were closed and the system allowed to cool. The RDX was drained from the recrystalliser, filtered and washed using Buchner funnels and stored in high density plastic drums in the appropriate magazine.
- NOTE: For modified procedure for the preparation of purified RDX, delete steps III.2.8 and III.2.9 and replace with steps III.2.8A and III.2.9A below.
- III.2.8A The recrystalliser, heat exchanger steam and condenser cooling water valves were turned on and all distillate was collected in the receiver up to 75°C. After 75°C the distillate was discarded.
- III.2.9A When the temperature reached 100°C, the sparger steam valve was opened and the stripping stage continued for 30 min.

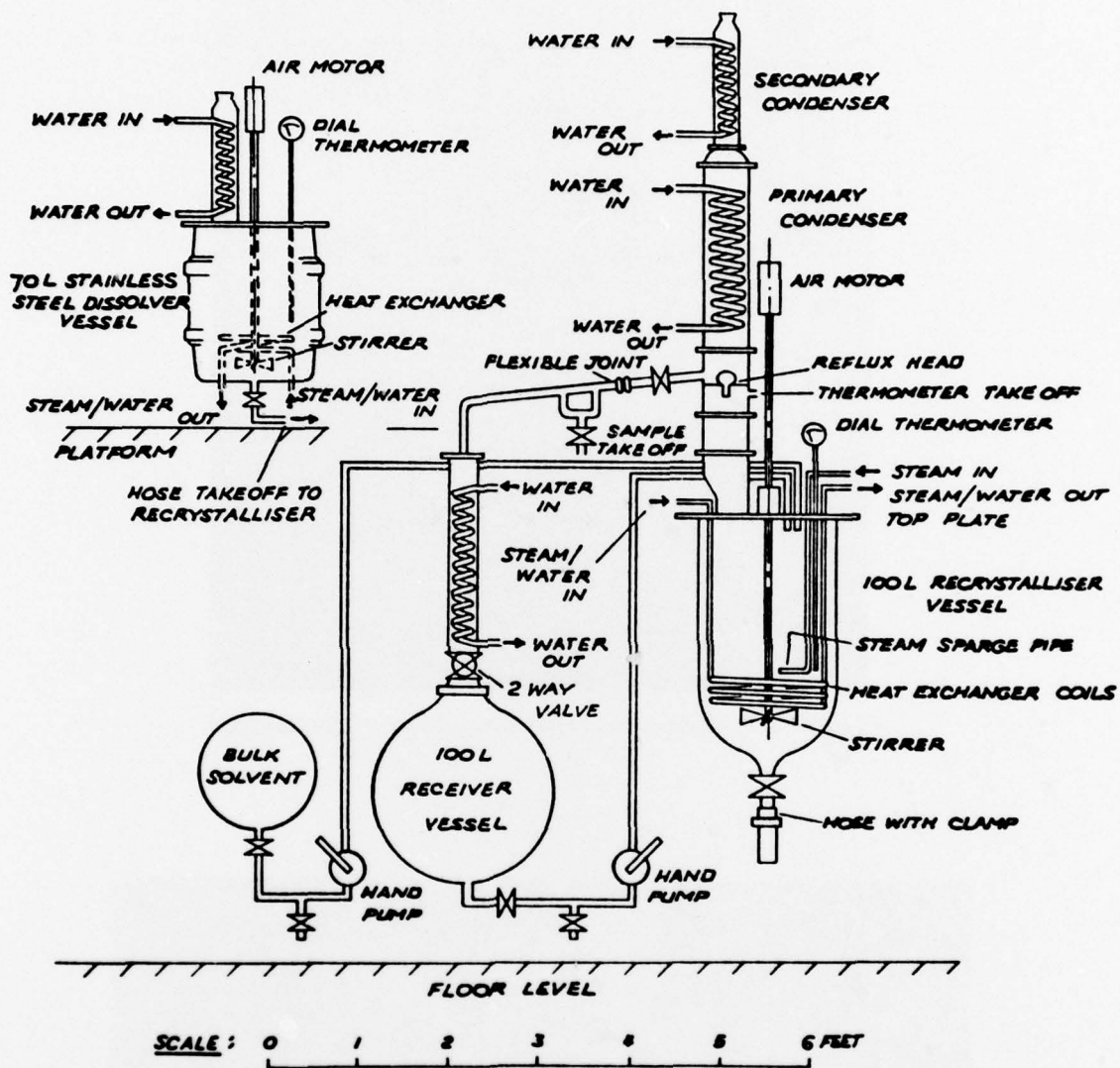


Figure 1. Recrystallisation plant - schematic diagram

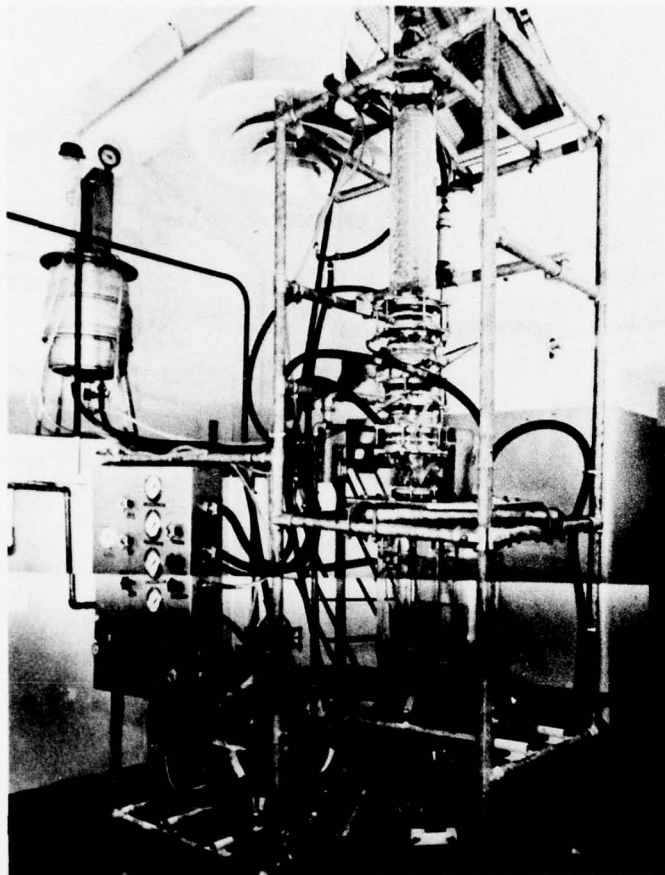


Figure 2. Recrystallisation plant - photograph

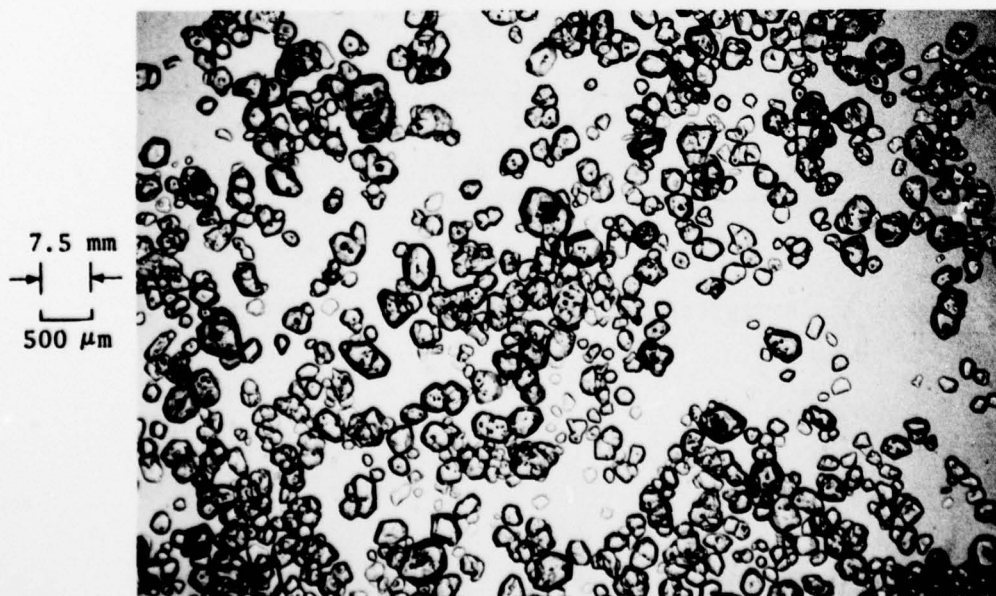


Figure 3. RDX Class A - batch CX6

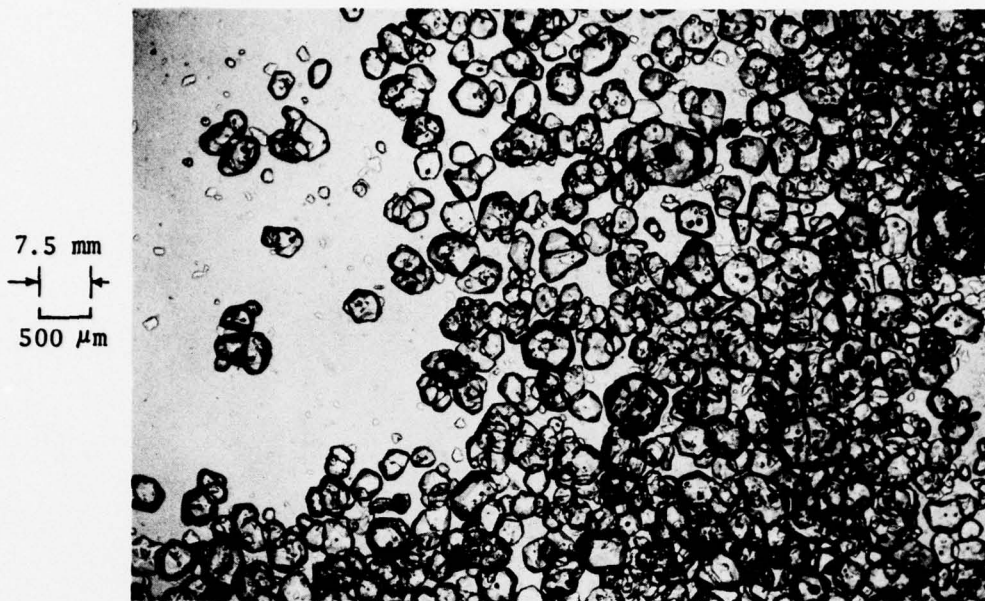


Figure 4. RDX Class C - batch CX8

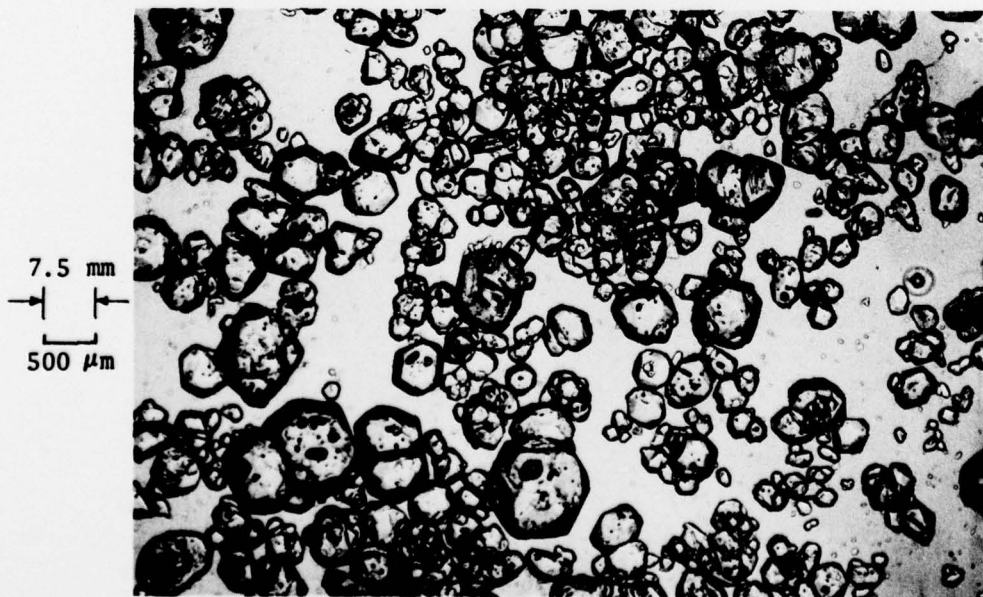


Figure 5. RDX Class D - batch CX21

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