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The Continuous Disintegration and Nitration of
Cellulose Pulp Board: A Laboratory Study

L. H. Gerty

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TECHNICAL MEMORANDUM NO. 3/M/58

The Continuous Disintegration and Nitration of
Cellulose Pulp Board: A Laboratory Study

by

L.H. Gerty

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CONTENTS

	<u>Page</u>
1. Summary	1
2. Object	1
3. Introduction	1
4. Apparatus and Experimental Method	2
4.1 The Disintegrator	2
4.2 Cellulose Feed	2
4.3 Mixed Acid Feed	2
4.4 Hold-up Vessel Design and Construction	2
4.5 Spent Acid Retentions	3
4.6 Graded Washing in the Centrifuge	3
4.7 Stabilisation of Nitrocellulose	3
5. Results (Tables 1 - 3)	3
6. Discussion	5
7. Hold-up Vessel Design	6
8. Bulk Density and Acid Retentions (Table 4)	6
9. Post-Nitration Treatment	7
10. Conclusions	8
11. Acknowledgements	8

Figure 1.

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1. SUMMARY

A method has been demonstrated on a laboratory scale for the production of nitrocellulose by disintegrating and nitrating cellulose pulp board in a continuous manner. The method consists in the disintegration combined with partial nitration of cellulose in a swing hammer mill in the presence of mixed acid, followed by the completion of the nitration to equilibrium while the suspension passes through a specially-designed hold-up vessel. Hold-up times of 5 and 2½ minutes are required for nitrogen contents of 13.1 and 12.2 per cent respectively, using either a high alpha cellulose or the normal paper grade of sulphite pulp.

However, the acid usage and losses in the process were found to be higher than for present industrial practice such as the mechanical nitration of pulp-board shreds by the Hercules method. These disadvantages are considered to render the process uneconomic, and the work has been terminated.

2. OBJECT

2.1 To investigate a method of nitration of cellulose based on the combined disintegration and nitration of cellulose pulp board in a hammer mill.

2.2 To assess the merits of this method of nitration relative to the established processes for the production of nitrocellulose.

3. INTRODUCTION

The two main disadvantages of the displacement process for the nitration of wood cellulose are:

- (i) the high process labour costs involved,
- (ii) the high cost of converting the wood cellulose into a form suitable for nitration.

The production of wet-disintegrated wood-pulp, as used in Canada, requires about half the fuel needed to convert cellulose pulp board into the water-leaf paper used in Britain, but, in the former case, it has so far been necessary to use a soft high alpha cellulose board which reduces the cost advantage of this method of preparation and places a restriction on the supply of suitable pulp board.

A substantial proportion of the fuel required to produce wet-disintegrated pulp is consumed in re-drying the pulp after disintegrating in a hammer mill at a moisture content of about 50 per cent. The development work on which the Canadian process was based had shown that the product of a dry disintegration was unsuitable for displacement nitration on account of its low bulk density, high dust and 'fly' contents and uneven disintegration. These troubles could be avoided with wet-disintegrated pulp, but in this case the physical properties were fairly sensitive to changes in the moisture content of the board when disintegrated.

It occurred to the author that it might be preferable to disintegrate the board in a stream of liquid which would act as a cushion against which the cellulose could be disintegrated. The preliminary experiments, using water, showed that the disintegrated pulp consisted of a uniformly teased-out mass of

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fibres, and that the product was insensitive to changes in the ratio of water to cellulose at the high dilution levels used. The next stage was the substitution of mixed acid for water, resulting in a combination of the disintegration and nitration stages and the elimination of the need to re-dry the pulp after disintegration and before nitration.

This Memorandum describes the investigation of this method of combined disintegration and nitration.

4. APPARATUS AND EXPERIMENTAL METHOD

4.1 The Disintegrator

The disintegrator was a swing hammer mill based on the design of the Raymond Laboratory mill manufactured by the International Combustion Company, but modified to suit the special duties for which it was required. The stainless steel rotor and hardened steel hammers were supplied by the I.C. Co. A mild steel casing was fabricated in the E.R.D.E. Workshops. The screw feed inlet was replaced by a narrow-bore stainless steel pipe with a funnelled end. The standard screen with $\frac{1}{8}$ -inch circular perforations was found to be the most satisfactory and was used throughout. A U-bend was fitted to the mill outlet to give a liquid seal of 9 inches. This was the equivalent of the 16-inch water gauge pressure head between the inlet and outlet sides of the mill when running against a sealed outlet.

The unit was powered by a $\frac{1}{2}$ -h.p. a.c. motor rotating at 10,000 rev/min. coupled direct to the rotor shaft. This shaft passed through a recessed collar in the back plate of the mill with a $\frac{1}{16}$ -inch clearance. No packing was needed for this gland.

4.2 Cellulose Feed

Pellets of Borregaard Super 1 rayon pulp, $\frac{1}{8} \times \frac{1}{8} \times \frac{1}{30}$ inch thick, or of Essvik Three Star sulphite paper grade of pulp, $\frac{1}{8} \times \frac{1}{8} \times \frac{1}{25}$ inch thick, were delivered to the funnel inlet to the disintegrator in one of two ways - either semi-continuously with a spoon at regular intervals, or continuously using a vibrating band conveyor.

4.3 Mixed Acid Feed

Mixed acid was fed from storage tanks via a constant-head feed tank and rotameter to the funnel inlet to the disintegrator. An electric heater was incorporated in the pipe line to bring the mixed acid to the required temperature before delivering it to the mill.

4.4 Hold-up Vessel Design and Construction

The hold-up vessel was a stainless steel cylinder 1 foot i.d. and 3 feet in length divided internally by a thin partition in the form of a single helical 'flight' of 3-inch pitch, welded on its inner edge to a $1\frac{1}{4}$ inch stainless steel shaft and to the casing along its outer circumference (Fig.1). At the inlet end of the vessel a ring 3 inches wide (i.e., a disc one foot in diameter with a six-inch concentric aperture) was welded on to the casing to form a chamber into which the nitration mixture from the disintegrator was fed. At the outlet end a helical-shaped baffle of $2\frac{1}{2}$ inches tapering to nil in one revolution was so positioned that it caused a continuous discharge of mixture from the hold-up vessel.

The vessel was mounted horizontally on bearings and was rotated at the

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appropriate speed by means of a V-rope and pulley drive from a geared down motor.

The fabrication of this vessel presented some difficulties. It was achieved in the E.R.D.E. Workshops by cutting out perforated stainless steel discs of the correct size and bending them to shape on a former. Each shaped disc was welded to the shaft. The casing was built up by welding shaped segments to the circumference of the screw. The tapering helical baffle was constructed by building on to the vessel an additional revolution of the helical screw from which the surplus metal was removed.

This vessel provided a working volume of $\frac{1}{2}$ ft³, and a capacity of 12 lb. of nitrocellulose per hour, when operated at an acid:cellulose ratio of 80:1 and a hold-up time of five minutes. Longer or shorter times of hold-up could be obtained by adjusting the speed of rotation of the vessel with a corresponding decrease or increase in throughput.

4.5 Spent Acid Retentions

Spent acid retentions were determined using a batch-operated stainless steel basket centrifuge of 7 inches i.d. rotating at a constant speed in the range 2000 to 3000 rev/min. to give a gravity factor in the range 400 to 900. The acid retentions of nitrated Hercules shreds were determined with the same centrifuge to obtain a fair comparison. For this purpose the mechanical nitrations of Hercules shreds were carried out in the laboratory.

4.6 Graded Washing in the Centrifuge

The acid-wet nitrocellulose remaining in the centrifuge after removal of the bulk of the spent acid was sprayed with wash liquors of varying compositions. These liquors were recovered little changed, having mainly channelled their way through the nitrocellulose.

4.7 Stabilisation of Nitrocellulose

Samples of nitrocellulose were stabilised in four water boils of 12, 12, 4 and 4 hours duration. The product was pulped to normal fineness and its alkalinity adjusted to 0.20 to 0.40% as CaCO₃ before analysis.

5. RESULTS

These are given in three tables.

/Table 1

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TABLE 1

The results obtained when Borregaard and Essvik cellulose pulp board pellets were disintegrated and nitrated in mixed acid at 30°C and at a ratio to cellulose of 80:1. Hold-up time 5 minutes. Nitrogen content 13.1% nominal.

	Borregaard Super 1 Rayon Pulp				Essvik Three Star Paper Grade Sulphite Pulp			
Nitrogen content, % not corrected. (Devarda)	12.98	13.05	13.04	13.08	12.90	12.98	12.86	12.92
Ether-alcohol solubility, %	23.4	24.1	24.0	22.8	26.5	25.0	27.1	26.8
Acetone solubility, %	99.6	99.8	99.7	99.8	99.7	99.9	99.5	99.6
Ash, %	0.46	0.34	0.32	0.28	0.43	0.53	0.39	0.45
Alkalinity, %	0.25	0.22	0.20	0.18	0.18	0.13	0.20	0.25
Heat Test, min.	18	10	10	15	23	28	18	20
Bergmann and Junk Test, mg. NO/g.	0.97	1.54	1.18	1.25	1.15	1.26	1.19	1.15

Mixed acid composition: 59.27% H_2SO_4 , 27.81% HNO_3 , 12.92% H_2O .

TABLE 2

The results obtained when Borregaard and Essvik cellulose pulp board pellets were disintegrated and nitrated in mixed acid at 30°C and at a ratio to cellulose of 80:1. Hold-up time 2½ minutes. Nitrogen content 12.2% nominal.

	Borregaard Super 1 Rayon Pulp				Essvik Three Star Paper Grade Sulphite Pulp			
Nitrogen content, % not corrected. (Devarda)	12.17	12.10	12.09	12.05	12.02	12.07	12.09	12.03
Ether-alcohol solubility, %	100	100	100	100	100	100	100	100
Acetone solubility, %	99.5	99.4	99.5	99.5	99.0	99.4	99.5	99.5
Ash, %	0.43	0.43	0.42	0.53	0.40	0.49	0.47	0.54
Alkalinity, %	0.24	0.25	0.22	0.25	0.32	0.29	0.31	0.35
Heat Test, min.	12	14	10	10	10	11	17	16
Bergmann and Junk Test, mg. NO/g.	-	-	-	-	-	-	-	-

Mixed acid composition: 58.63% H_2SO_4 , 26.43% HNO_3 , 14.94% H_2O

/Table 3 ...

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TABLE 3

Comparison of the spent acid retentions of mechanically nitrated Hercules shreds and disintegrated and nitrated Borregaard pulp board after centrifuging for two minutes at various basket speeds.

Type of Nitrocellulose	Centrifuge Basket Speed, rev/min.	Acid Retention of Nitrocellulose, Bulk as % on dry N.C.
Hercules shreds to 12.2% nitrogen	2050	147.5
	2550	128
	3000	119
Hercules shreds to 13.1% nitrogen	2100	129
	2440	128
	3000	123.5
Disintegrated and nitrated to 12.2% nitrogen	2050	176
	2550	153
	3000	133
Disintegrated and nitrated to 13.1% nitrogen	2000	159.5
	2500	154
	3000	132

6. DISCUSSION

In the preliminary experiments the cellulose pellets and mixed acid were fed to the hammer mill either separately or pre-mixed from a stirred vessel. The issuing slurry was sampled and stored for varying times before the spent acid was removed and the nitrocellulose drowned out in iced water. The following facts were established:

Nitrocellulose of 13.1 per cent nitrogen content could be obtained by the continuous disintegration and nitration of pellets, of high alpha or of a paper grade of board, using a mixed acid of the composition usual for the mechanical nitration process followed by a hold-up time of five minutes. Similarly, nitrocellulose of 12.2 per cent nitrogen content could be obtained with either grade of cellulose board and the appropriate mixed acid with a hold-up time of two and a half minutes.

The best results were obtained when the cellulose pellets and mixed acid were fed separately to the hammer mill inlet, the acid being at 30°C, and at a ratio of 80:1 of cellulose by weight.

Pre-mixing of mixed acid and cellulose for periods of up to fifteen minutes before feeding them to the disintegrator gave rise to inferior nitrogen contents and ether-alcohol solubilities. This was undoubtedly caused by the progressive dilution of the mixed acid as it penetrated into the cellulose pellets, with the

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result that, even after disintegration, the composition of the mixed acid in the cellulose fibres must be restored to that of its surroundings by the slow process of diffusion. Thus, if pre-mixing is to be of any benefit, really long times of contact before disintegration are necessary to allow equilibrium to be reached and to benefit from the lower shredding energies known to be required after prolonged contact with mixed acid. It is not considered, however, that disintegration of the pulp board after nitration merits serious consideration, for the complications added to the process would far outweigh any benefit to be obtained.

The acid to cellulose ratio of 80:1, though not necessary to obtain uniformity of nitrogen content, was required so that the slurry would be sufficiently fluid to be evenly discharged from the liquid seal on the mill outlet. This liquid seal was found to be necessary in order to prevent the passage of large volumes of air through the disintegrator, causing heavy loss of nitric acid as vapour and dilution of the mixed acid to a degree dependent upon the relative humidity of the atmosphere. Mixed acid temperatures above 40°C caused greater degradation of the nitrocellulose and gave rise to ether-alcohol solubilities of about 35 per cent at the 13.1 per cent nitrogen content level.

Use of the paper grade of pulp board instead of the high alpha cellulose board resulted in a slightly lower mean nitrogen content at the 13.1 per cent level. This difference could be overcome by making a suitable adjustment to the composition of the mixed acid.

7. HOLD-UP VESSEL DESIGN

Much thought was given to the design of a hold-up vessel capable of continuous operation in conjunction with the disintegrator/nitrator, and from which the acid nitrocellulose suspension could be discharged strictly on the basis of first-in first-out after the pre-determined time of hold-up. It was realised that variations in the time of hold-up about the mean had to be avoided, as otherwise the mean nitrogen content and ether-alcohol solubility of the nitrocellulose would be adversely affected. The thick porridge-like consistency of the suspension issuing from the hammer mill increased these design difficulties. However, the cylindrical vessel which could be rotated at the appropriate speed and which contained the fixed helical screw operated satisfactorily and discharged the nitration mixture on a semi-continuous basis. The design and fabrication of this vessel are detailed in Section 4.4.

8. BULK DENSITY AND ACID RETENTIONS

The degree of disintegration required in order to obtain rapid nitration appeared to correspond with the separation of the cellulose pulp board into fibres. In this form the bulk density is low, and a high ratio of mixed acid, over and above that required for nitration, was necessary in order to be able to transport the suspension. The amount of spent acid retained after centrifuging was found to be higher than for the denser product, such as mechanically nitrated Hercules shreds, as Table 4 shows. The wringing time was two minutes at about 2500 rev/min., for which the gravity factor is about 620.

/Table 4

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TABLE 4

Type of Nitrocellulose	Acid Retentions Expressed as % Weight of N.C.	
	12.2% Nitrogen	13.1% Nitrogen
Disintegrated and nitrated high alpha cellulose	153	154
Hercules shred mechanically nitrated	128	128

The disintegrated and nitrated paper grade of pulp required longer spinning times to attain the acid retentions of the high alpha cellulose board.

Attempts to reduce the degree of disintegration in the hammer mill in order to increase the bulk density of the product, and so reduce both the ratio of mixed acid to transport the pulp and the amount of spent acid retained after centrifuging off surplus acid were not successful. When a screen with larger perforations was used in the hammer mill irregular disintegration resulted. Some of the pulp was discharged as separated fibres and some as hard nodules.

It was of course realised that the scope for reducing the degree of disintegration was strictly limited, since any move in this direction would result in increased times of nitration which would prove an embarrassment to a continuous process. Furthermore, as the degree of disintegration was reduced the nitration stage would become increasingly dependent upon the physical properties of the cellulose pulp board used.

It was therefore concluded that it was not possible to make a satisfactory compromise based on the continuous disintegration and nitration of pulp board involving disintegration to some stage intermediate between Hercules shreds and separation into fibres.

9. POST-NITRATION TREATMENT

Whereas the combined disintegration and nitration is operated continuously the post-nitration processing can be carried out batch-wise or continuously. The various possibilities are equally applicable to the product of the nitration of Hercules shreds and to that of the disintegration and nitration process. If, for example, some of the spent acid retained by the nitrocellulose after centrifuging can be removed by a series of graded washes this applies to the products of both nitrations.

The preliminary attempts to examine the possibilities of graded washing by the batchwise treatment of acid wet nitrocellulose with wash liquors of varying acidities in a centrifuge were not successful. The liquors did not displace much acid from the nitrocellulose, but largely channelled their way through the nitrocellulose to be recovered practically unchanged.

It is interesting to note that, in the few cases examined, the stability of the nitrocellulose was brought up to the required standard in the normal four-boil sequence prescribed for displacement-made nitrocellulose.

/10. CONCLUSIONS

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10. CONCLUSIONS

10.1 Nitrocellulose can be produced by the continuous disintegration and partial nitration of cellulose pulp board in a hammer mill in the presence of mixed acid, followed by the completion of the nitration to equilibrium while the suspension passes through a specially designed hold-up vessel. Hold-up times of 5 and $2\frac{1}{2}$ minutes are required for nitrogen contents of 13.1 and 12.2 per cent respectively.

10.2 The degree of disintegration achieved, viz., the separation of the pulp board into cellulose fibres, leads to low bulk density of the cellulose, causing high acid usage and losses through:

- (i) The high acid to cellulose ratio (80:1) required in order to obtain a free-running slurry from the mill.
- (ii) The high residual acid content of the nitrocellulose after the centrifugal separation of spent acid.

10.3 A compromise, in balancing the degree of disintegration and the rate of nitration, between the process as operated and that of the mechanical nitration of Hercules shreds is not possible. Comminution of the pulp board short of complete separation into fibres resulted in an uneven disintegration and nitration. The higher acid usage and losses are, therefore an integral part of the disintegration nitration process.

10.4 The disadvantages of the higher acid usage and losses are considered to render the process uneconomic as compared with the mechanical nitration of Hercules shreds.

10.5 It is considered that these drawbacks are not outweighed by the advantages of the disintegration nitration process, among which are the following:

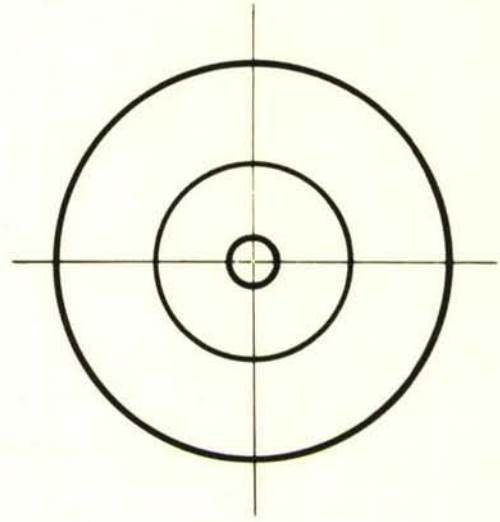
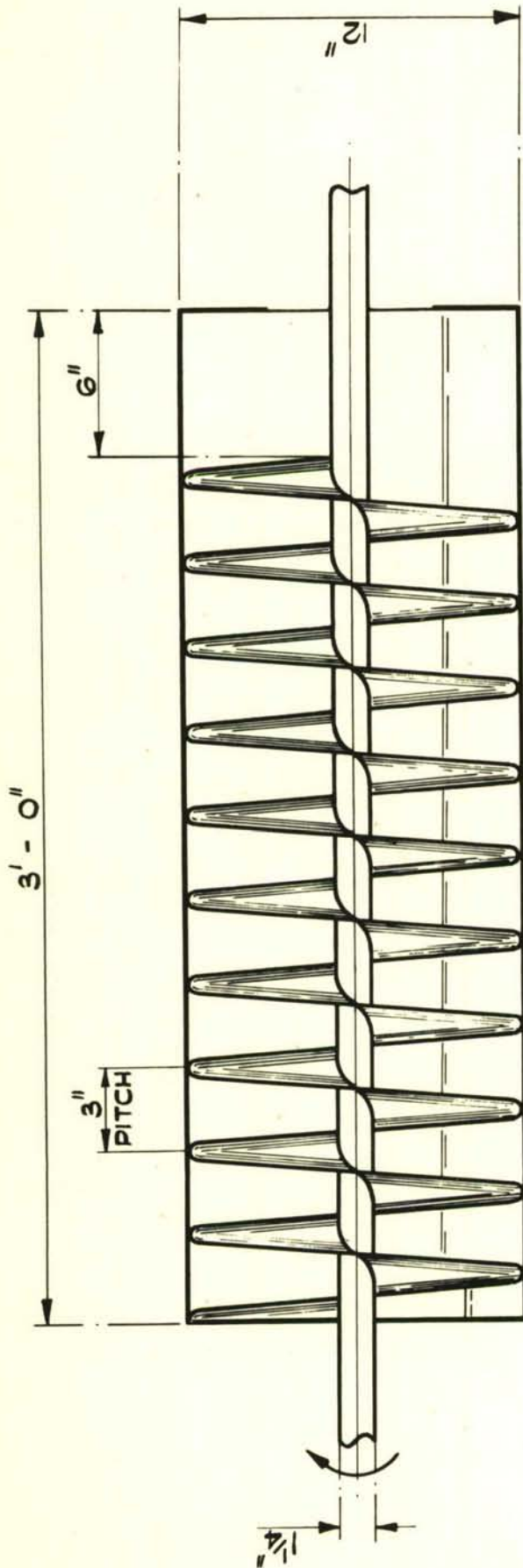
- (i) An ordinary paper grade of sulphite pulp board can be used. It is not necessary to use a soft, highly refined, pulp as is required for the Hercules shredding process.
- (ii) The disintegration nitration can be operated continuously. Spent acid removal could be operated batchwise or continuously; however, with stabilisation carried out batchwise, a change from continuous to batch operation at some stage is necessary.
- (iii) An overall saving in labour would be expected as a result of the continuous operation of the nitration process.
- (iv) Adequate stability appears to be possible with the standard four-boil treatment employed for displacement-made Service nitrocellulose.

11. ACKNOWLEDGEMENTS

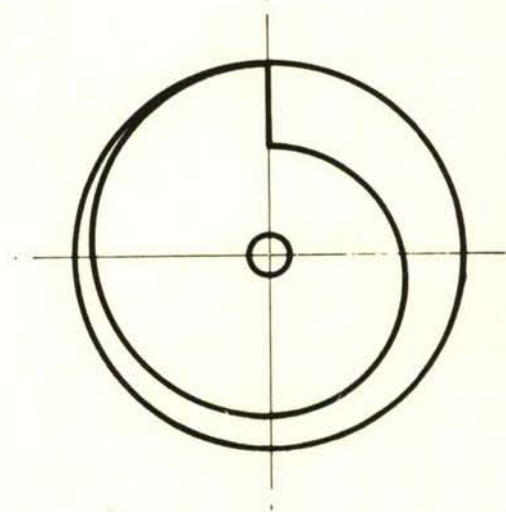
Thanks are due to Mr. C.H. Miller who carried out some of the experimental work covered by this report.

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HOLD-UP VESSEL. FIG. 1.

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However, the acid usage and losses in the process were found to be higher than for present industrial practice such as the mechanical nitration of pulp-board shreds by the Hercules method. These disadvantages are considered to render the process uneconomic, and the work has been terminated.

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