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## Peracetic Acid Pulping of Aspen Chips

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PERACETIC ACID PULPING OF ASPEN CHIPS

A THESIS SUBMITTED TO THE DEPARTMENT OF  
PAPER TECHNOLOGY AS A PARTIAL FULFILMENT  
OF THE REQUIREMENTS FOR A B.S. DEGREE

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AUGUST 8, 1966

## ABSTRACT

Ten cooks were carried out after an acceptable experimental procedure was found.

The brightness obtained was extremely high when compared to conventional cooks. All cooks gave a very low permanganate number. The yield ranged from 60 percent to 54 percent with 58 to 96 percent peracetic acid on the oven dry weight of the chips.

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## PERACETIC ACID PULPING OF ASPEN CHIPS

### Pulping and Bleaching with Peracetic Acid: Literature Survey

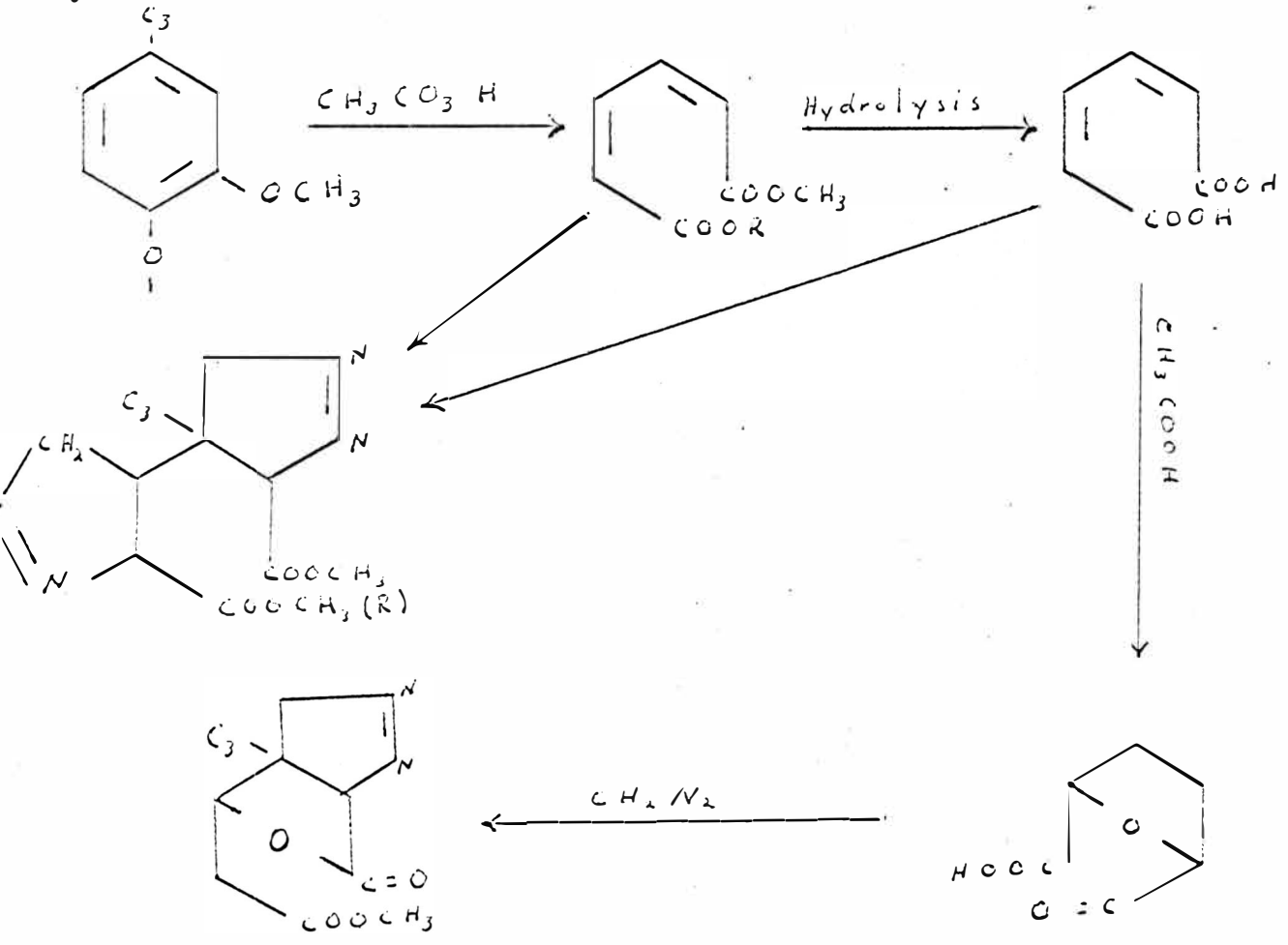
Very little work has been done with peracetic acid as a pulping or bleaching agent. Only recently have the potentials of peracetic acid been seriously investigated. The major drawback to using this acid has been the high cost of the acid. Also, peracetic acid, in concentrated form, can become quite unstable and decompose violently.

In 1960 a patent was granted to Wayman and Harris (7) for the pulping of wood with peracetic acid. In this process, a concentration of 10-40 percent by weight of peracetic acid was used. The pulping temperature was from 60-100°C and times of 6-360 minutes were used. In all cases, the reaction was carried out at atmospheric pressure. According to this patent, a white pulp was obtained with a low permanganate number, as compared to conventional pulps. The cupriethylenediamine viscosities (T230) of the pulps reported was from 39-5 cps. in a yield range of 56-44 percent on the original O.D. weight of the wood. It was also reported that wood wafers or defibered wood gave better results than conventional sized chips.

Leopold (2) prepared some softwood pulps using peracetic acid and obtained the following results: yield 75.3 percent, lignin 2.6 percent, acetyl 1.2 percent, carboxyl .26 meq./g, carbon .2 meq./g, glucan 45 percent, galactan 1.3 percent, mannan 9.5 percent, araban 1.0 percent, xylan 6.8 percent, pentosans 7.8 percent, and a D.P. of 2420.

Tompson and Kaustinen (3) prepared holocellulose pulps under very mild conditions with peracetic acid using white spruce. They were able to obtain a pulp of 66 percent yield with a Klason lignin content of .1 percent. A strength evaluation study was also undertaken with the following results: Tensile breaking length 13500m, zero span breaking length 19800m, burst factor 101, and a tear factor of 77.2.

Sarkanen and Suzuki (4) studied the oxidative delignification mechanisms of peracetic acid on Douglas fir. They found that practically all aromatic nuclei in the lignin were converted to muconic acid structures, containing some of the original methoxyl as methyl ester groups. They proposed the following oxidation mechanism for a peracetic acid lignin system.



There are three patents covering peracetic acid bleaching which were issued before 1950. The first is a Canadian patent issued to Dreyfus (5). This patent is for bleaching of wood pulp by treating the pulp with a combination of lower aliphatic peracid and a relatively concentrated lower aliphatic acid, as with peracetic and acetic acid, in the presence of a lead catalyst, 2-10 percent on the O.D. weight of the pulp. The second is a U.S. patent issued to the Celanese Corporation of America (6). In this patent, there is a concentration of 10-20 percent peracetic acid at room temperature for 5-24 hours, with a bleaching consumption of 10-30 percent. However, Rapson reported a much higher bleach consumption for similar pulps (7). The last patent is a Canadian patent issued to the Buffalo Electro-Chemical Company (8). This patent involves spraying a web of felted wood pulp with peracetic acid at a pH of 6-8 and then evaporating the water from the sheet. The patent states that the dry pulp should have of pH of less than 7.

Rapson (9) carried out quite an extensive investigation of ground-wood bleaching with varying amounts of peracetic acid. He reported that he was able to obtain, with cottonwood pulp, an 88 percent brightness with 30 percent peracetic acid on the O.D. weight of pulp and yield of 98 percent.

Most of the work done by Rapson was with western hemlock groundwood. He was able to obtain quite an increase in brightness with relatively small amounts of peracetic acid. Washing with SO<sub>2</sub> before and after bleaching, was reported to have resulted in substantially reducing brightness reversion and also giving a slightly higher initial brightness.

The effect of pH on bleaching showed that maximum brightness was obtained with a initial pH of 7-9.5. With a buffered bleach solution, the maximum brightness was obtained when the final pH was 7-8. The buffered bleach solution gave a larger brightness gain, but about half of this gain was lost upon aging.

F.M.C. recommends the following procedure for the analysis of peracetic acid: 10 milliliters of the cooled solution are pipetted into an Erlenmeyer flask containing 50-100 milliliters of cold water. Approximately 10 milliliters of 25 percent  $H_2SO_4$  are added, followed by 1-3 drops of a saturated solution of manganous sulfate. The hydrogen peroxide present is destroyed by adding 0.1N  $KMnO_4$  drop by drop, until a faint pink color lasting 5-10 seconds is obtained. A few crystals of potassium iodide are added and the liberated iodine immediately titrated to a colorless solution with 0.1N  $Na_2S_2O_3$ .



## Procedures

Aspen chips were obtained from Menasha Corporation, Otsego, Michigan. The chips were washed and sorted by hand, then subjected to two passes through a Bauer refiner, using the breaker plates. Only those chips which would pass through a  $\frac{1}{4}$  inch screen and be retained on a 0.0939 inch screen were employed. The acceptable chips were air dried at 75° F and 50 percent R.H. The dried chips were then mixed and sampled for moisture.

### A. Pulping

All cooks consisted of 50 grams of oven dry chips and 600 ml. of liquor, to give a liquor to wood ratio of 12 to 1. The chips were impregnated with cooking liquor, by cycling between 50 p.s.i. air pressure and the vacuum produced on a water aspirator for thirty minutes. The chips were cooked for four hours in a temperature bath which was held at  $100^{\circ} \pm 0.03^{\circ}$  C with refluxing.

The chips were defibered hot, in a Warring blender, and then washed on a Buchner funnel. Brightness and permanganate samples were removed at this time, and the pulp was air dried. After the pulp was dry, moisture samples were taken and the yield calculated.

### B. Beating and Strength Evaluations

Samples of each type of pulp were ball milled for one hour in a one pint ball mill. Canadian standard freeness were determined for both beaten and unbeaten samples. Sheets were made according to T 205m. The sheets were tested for tear, tensile, burst, and zero-span tensile, according to Tappi Standards.

### C. Brightness Measurements

All brightness measurements were made according to T 217m. Reported values represent an average of eight readings.

### D. Bleachability

Bleachability was determined by performing a 25 ml.  $\text{KMnO}_4$  test (Tappi Standard T 241m-50).

## Presentation of Data

Ten cooks were carried out using peracetic acid as the cooking liquor. In all ten cooks the amount of chemical was varied while the temperature and cooking time was held constant.

Table I shows the results obtained from these cooks. Included in this table are yield, amount of peracetic acid applied, permanganate number, brightness, freeness before and after ball milling, and results from strength tests before and after ball milling. Some of the more significant data is graphically represented in figures 1-5.

## Discussion of Results

All cooks were carried out in duplicate, so as to evaluate the accuracy of the yield values. In most cases, the maximum variation was 1.5 yield points. The yield levels of the pulps produced only varied from 59.7 percent to 53.4 percent when the amount of chemical, expressed as percent peracetic acid on the O.D. weight of the wood, was varied from 57.63 to 96.17 percent.

Brightness increased considerably over the same range of chemical application, ranging from 54.2 to 80.7 percent.

When the chemical applied is compared to the amount of chemical needed for the same yield with the bisulfite process, it is observed that four to five times as much peracetic acid is needed to obtain the same yield.

It is assumed that under the conditions employed this process is a holocellulose isolation. This assumption is based on the fact that in cooks 3-10 the permanganate numbers were very low, and the range of yields obtained was very small, when 67 to 92 percent peracetic acid was applied to the O.D. weight of the wood.

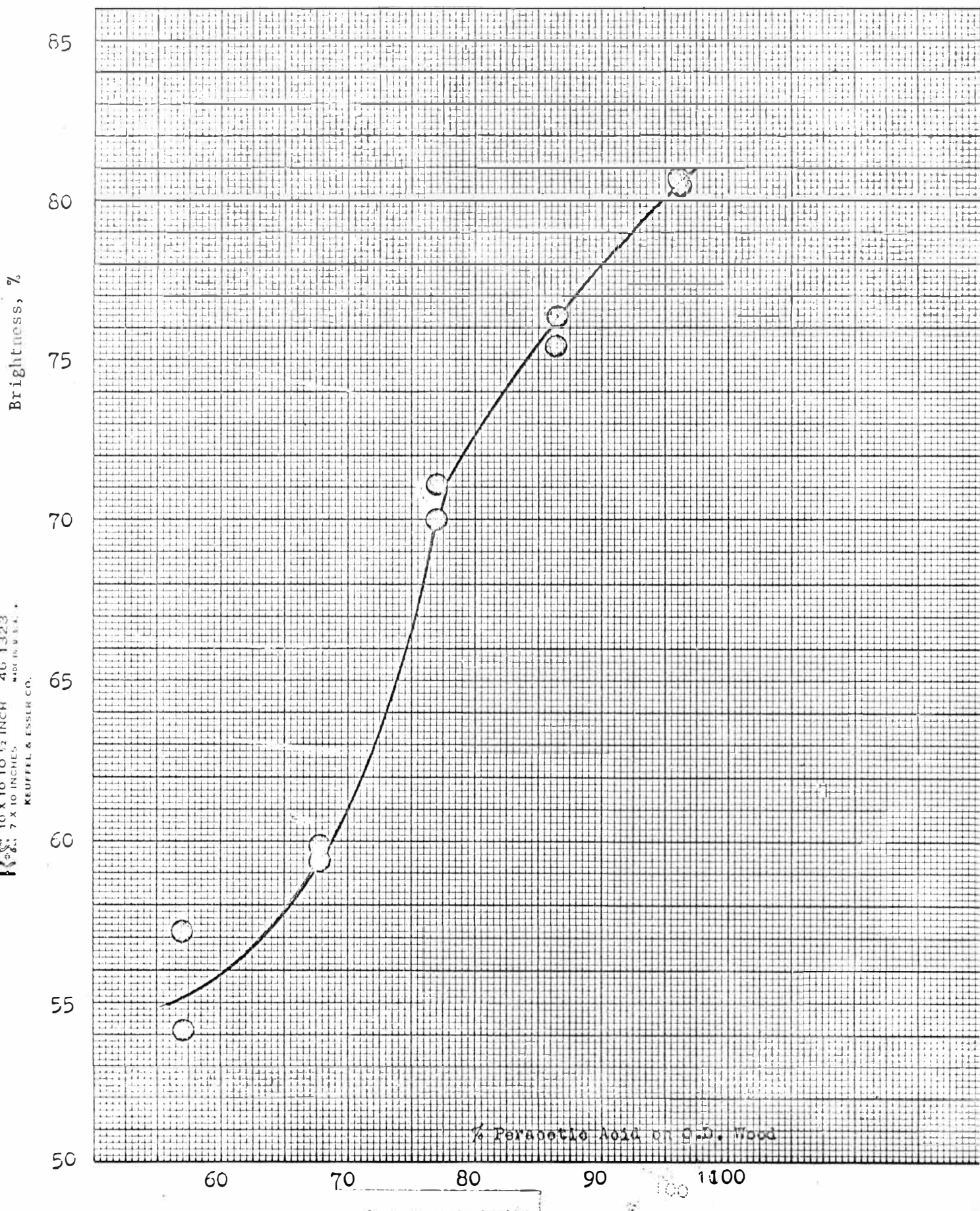
TABLE I

DATA OBTAINED FROM PERACETIC ACID COOLS

Cool Number	1	2	3	4	5	6	7	8	9	10
Total Peracetic acid, Basic C.D. Wood, %	57.7	57.7	67.3	67.3	76.9	76.9	86.5	86.5	96.2	96.2
Yield, %	59.7	57.4	56.0	56.3	55.3	54.0	53.7	54.4	55.5	55.8
Brightness, %	57.2	54.2	59.4	59.9	70.0	71.1	76.4	75.4	80.7	80.5
H <sub>2</sub> O <sub>2</sub> (25 ml) No.	5.3	5.4	3.1	3.4	2.0	1.3	1.5	1.9	1.4	1.7
Freeness										
No Beating		439		292		469		512		513
1 Hr. Ballmill		354		209		416		421		433
Breaking length, M										
No Beating		5426*		6772*		5874		6199		4072
1 Hr. Ballmill		5674		6917		7398*		7213*		7469*
Burst Factor										
No Beating		17.4*		27.1*		24.3		22.1		13.2
1 Hr. Ballmill		27.3		37.9		32.1*		31.2*		31.3*
Tear Factor										
No Beating		2.94		3.36		3.44		3.18		2.60
1 Hr. Ballmill		3.24		3.24		3.93		3.94		3.25
Zero Span Tensile lb/15mm		34.9		33.4		36.6		34.3		34.2

\* Values used in graphs IV and V.

### RELATION BETWEEN BRIGHTNESS AND CHEMICAL APPLICATION

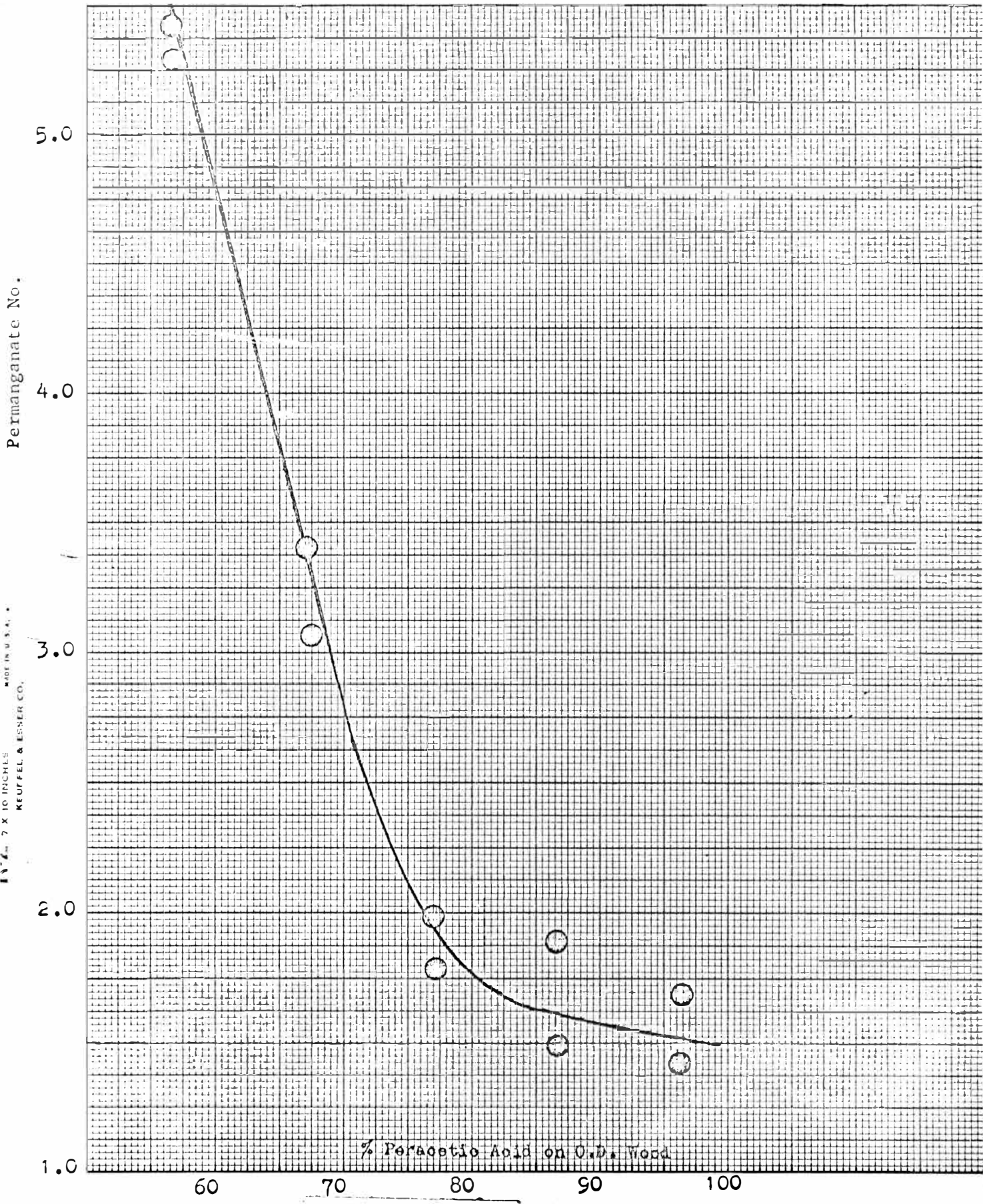


Brightness, %

% Peroxytic Acid on G.D. Wood

RS 10 X 10 TO 1/2 INCH 46 1323  
MADE IN U.S.A.  
KEUFFEL & ESSER CO.

### RELATIONSHIP BETWEEN PERMANGANATE NUMBER AND CHEMICAL APPLICATION

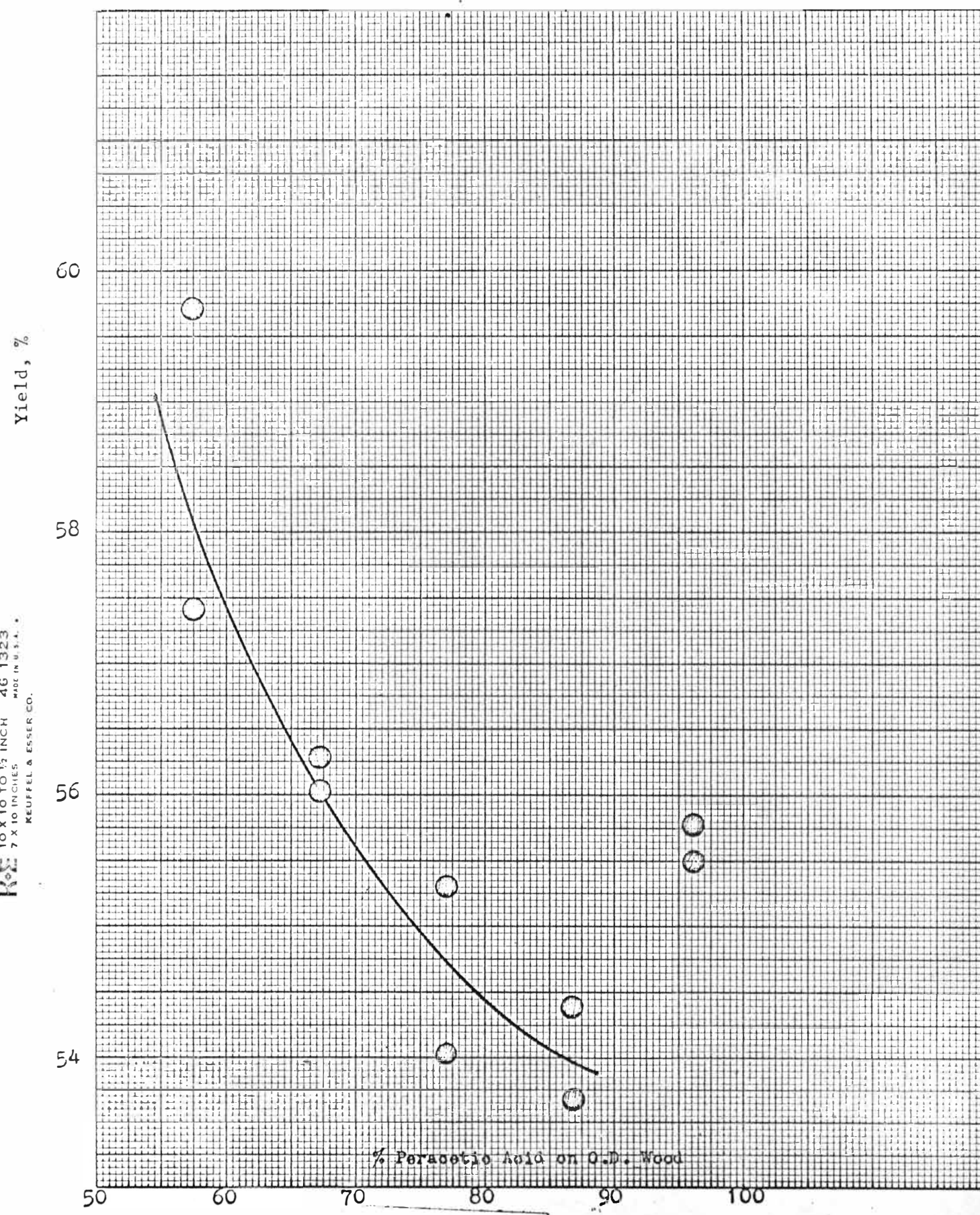


1 1/2" x 7" x 10" INCHES  
MADE IN U.S.A.  
NEUFFEL & ESNER CO.

% Paraacetic Acid on O.D. Wood



### RELATIONSHIP BETWEEN YIELD AND CHEMICAL APPLICATION

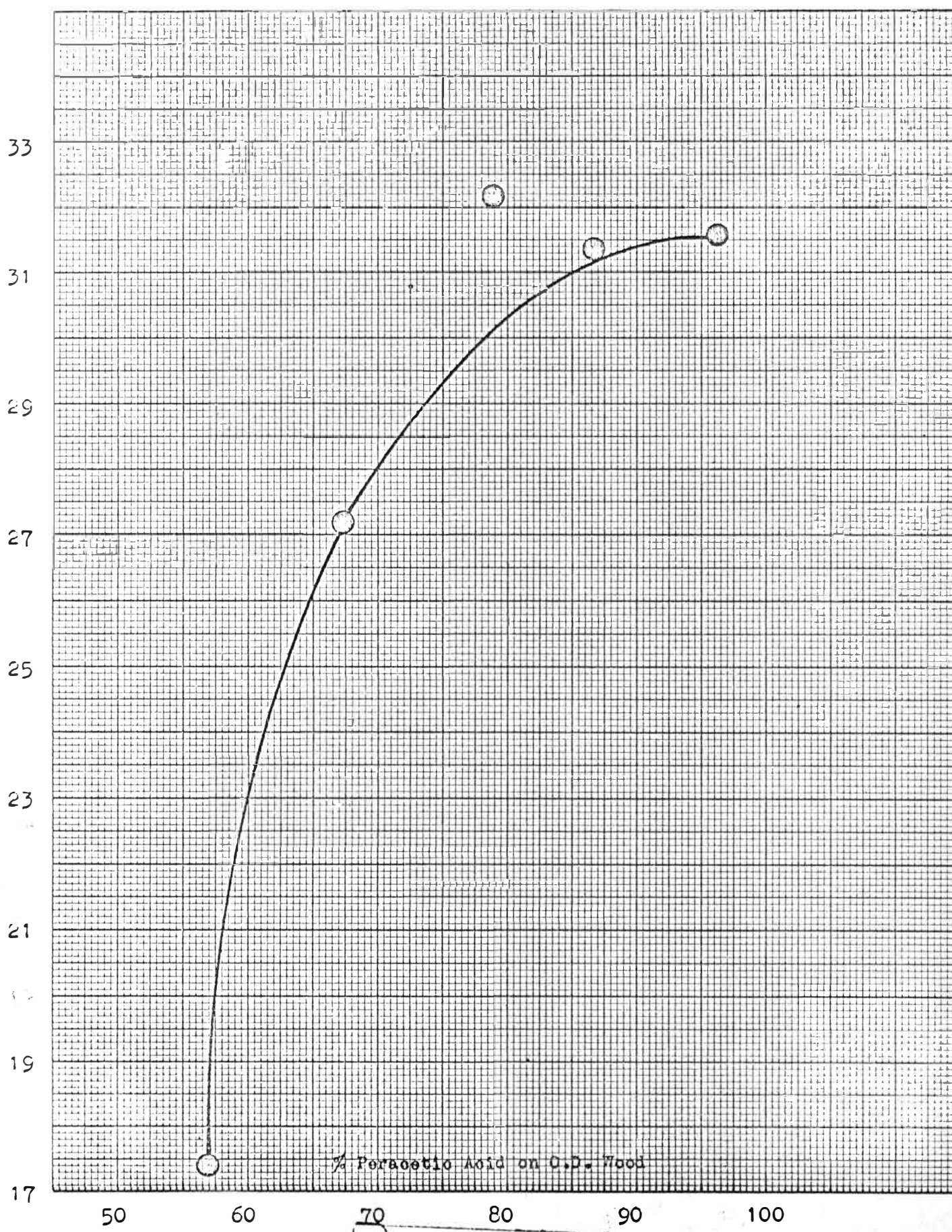


K&E 10 X 10 TO 1 1/2 INCH 4G 1323  
7 X 10 INCHES  
MADE IN U.S.A.  
KEUFEL & ESSER CO.



RELATIONSHIP BETWEEN BURST  
AND CHEMICAL APPLICATION

7 X 10 INCHES  
40 1323  
MADE IN U. S. A.  
KRUFFEL & ESSER CO.



% Peroxyacetic Acid on O.D. Wood

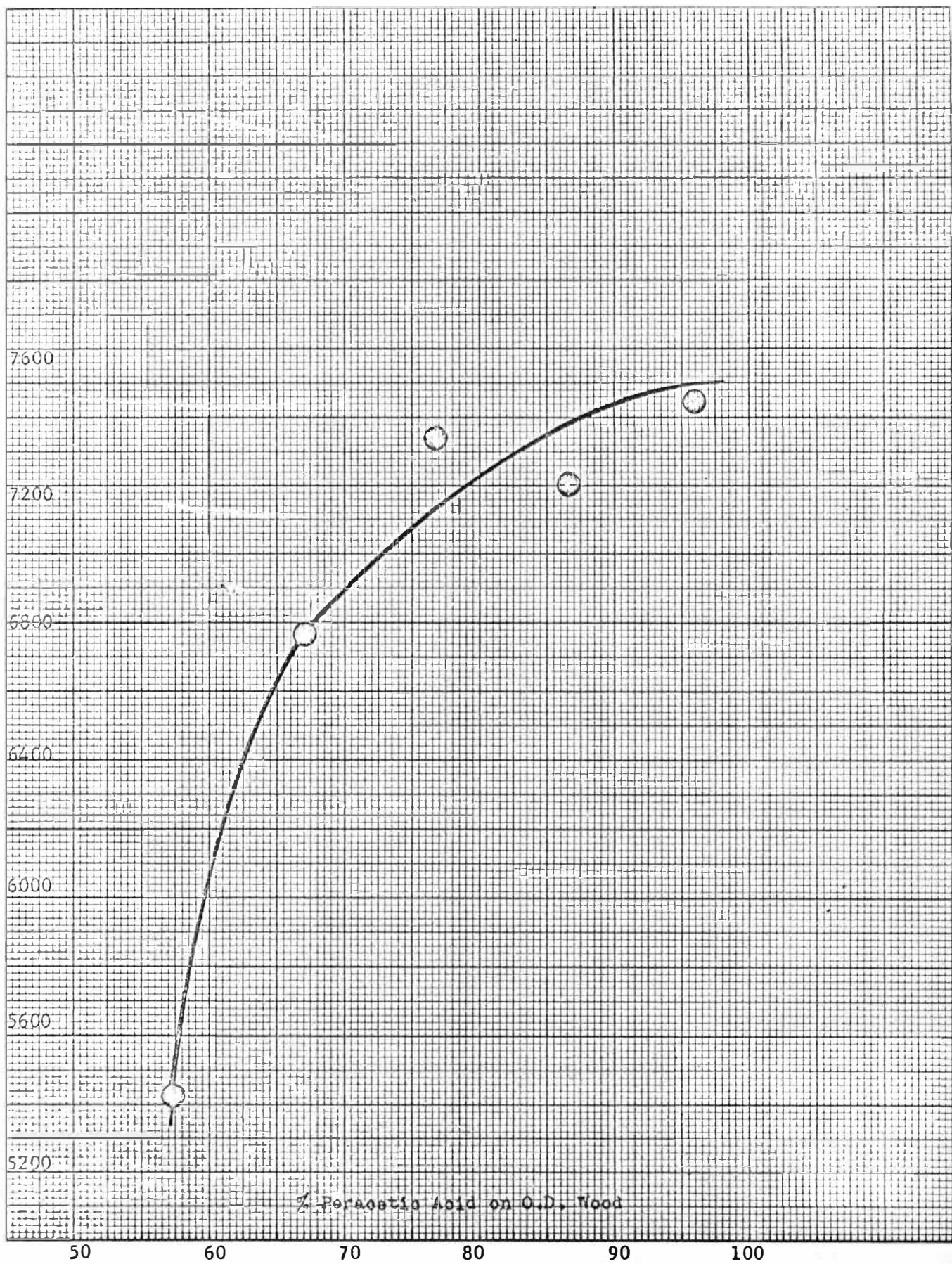
17 19 21 23 25 27 29 31 33  
50 60 70 80 90 100

RELATIONSHIP BETWEEN BREAKING LENGTH  
AND CHEMICAL APPLICATION

Breaking Length, M

MADE IN U.S.A.  
KEUFFEL & ESSER CO.

1 1/2" x 7" x 10" INCHES



% Peroxy Acid on O.D. Wood

Observations and Conclusions

1. Peracetic acid pulping of aspen chips under the conditions employed is economically unfeasible.
2. The brightness of the pulp produced was very high in comparison with sodium or magnesium bisulfite pulps. This difference being at least thirty points at all yield levels.
3. The permanganate number of the peracetic acid pulp was much lower than that obtained with sodium or magnesium bisulfite pulps at comparable yield levels.
4. The physical strength of the peracetic acid pulp was about the same as would be expected with sodium or magnesium bisulfite pulps.
5. Penetration of the wood chips with the cooking liquor was extremely difficult, under the conditions employed.
6. Tear and zero-span tensile tests showed no definite relationships when compared with the amount of chemical applied.

### ACKNOWLEDGMENT

Sincere appreciation is extended to Truman Pascoe whose guidance was invaluable.

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