

SOME COMPARATIVE EXPERIMENTS ON THE FIREPROOFING OF
WOOD PULP

PURPOSE

The purpose of these experiments was not to produce
a pulp that was absolutely unaffected by fire. Nor was it the
aim of the writer to develop any one pulp to its maximum
efficiency.

SOME COMPARATIVE EXPERIMENTS ON

THE FIREPROOFING OF WOOD PULP

by

William McMurray Dix

A THESIS

Respectfully submitted to the
Department of Chemistry of Washington
and Lee University in partial fulfillment
of the degree of Bachelor of Science in Chemistry
June, 1931

Fireproofing. This term is applied to various methods of
making normally combustible materials fireproof as far as may
be, but in practice, the most that can be done with this is
to retard or delay inflammability. The subject is one of
great importance, especially in connection with the construc-
tion of theatres and other buildings where large numbers of
people are collected together and where special illumination
is required. The nature of or disposal of materials used in
theatres has become the subject of public regulation in many
countries, and it is usual to provide that no combustible

SOME COMPARATIVE EXPERIMENTS ON THE FIREPROOFING OF
WOOD PULP

PURPOSE

The purpose of these experiments was not to produce a pulp that was absolutely unaffected by fire. Nor was it the aim of the writer to develop any one pulp to its maximum efficiency, but solely to place upon a comparative basis the fire-retardent action of different concentrations of several substances with which the pulp may be treated to reduce its flammability, and upon this basis to state the observed differences and similarities of the pulp impregnated with these various chemicals.

INTRODUCTION

(From Encyclopaedia Britannica, 14th Edition 1929, V. 9, p. 281)

"Fireproofing. This term is applied to methods of making normally combustible materials fireproof as far as may be, but in practice, the most that can be done with them is to subdue or retard inflammability. The subject is one of great importance, especially in connection with the construction of theatres and other buildings where large numbers of people are collected together and where special illumination is required. The nature of or disposal of materials used in theatres has become the subject of public regulation in many countries, and it is usual to provide that no combustible

scenery shall be employed unless it has been treated with fire-retarding solutions. The London County Council regulations, for instance, provide that in theatres materials such as wood, canvas, textiles, etc., must be rendered non-inflammable by impregnating them with solutions, and names the following as having been found suitable:- (1) for scenery and coarse fabrics: a solution of 1 lb. of phosphate of ammonia and two lb. chloride of ammonia (sal ammoniac) in 1.5 gal. water; (2) for delicate fabrics and cotton wool: 10 oz. borax and 8 oz. boracic acid in 1 gal. of water. Stage wood-work must be thoroughly impregnated with phosphate of ammonia. It is, however, quite impossible to make wood-work really fireproof and the council therefore insist, as far as possible, upon the use of incombustible materials such as asbestos."

"United States.- The building code recommended by the National Board of Fire Underwriters is commonly used as a basis in the preparation of building ordinances of many cities. A solution which has been widely recognized for the fireproofing of fabrics is one made of three parts of ammonium phosphate, two parts of ammonium chlorate, and one part of ammonium sulphate, and about 40 parts of water. Lumber impregnated with fire-retardant chemicals is termed 'fireproof wood'. The industry had its beginning in the United States in 1895. The principle market for fireproof wood has been in New York city. Its building code prohibits the use of wood for floors and interior trim for buildings in excess of 150 ft. in height unless fireproofed in accordance with

rules prescribed by the superintendent. The treatment of the wood is commonly done by impregnating it with fire-retardant chemicals in closed cylinders under artificial pressure."

A great number of solutions of fire-retardant chemicals have been patented for use in and on wood, paper, cloth, and synthetic wallboard. Among them are solutions of ammonium phosphate, ammonium sulphate, ammonium bromide, zinc ammonium chloride, arsenious oxide, sodium tungstate, sodium silicate, chlorinated diphenyl, chlorinated naphthalene, very many others, and various mixtures of these and others. For instance, United States patent number 1,793,357 claims: "1. A composition for use in connection with fibrous material to bind and impart a fire resisting characteristic thereto and for preserving such characteristic consisting solely of ammonium phosphate, boracic acid, dextrine and water, the proportion of the ammonium phosphate being greater than that of the boracic acid, and the proportion of the dextrine being greater than the ammonium phosphate....."

The need for more work and better cooperation along this line is well brought out by the fact that the United States is less in control of her fire losses than any other world power today. The annual fire losses in this country per capita for the past five years shows neither a steady increase nor decrease, while that of Canada is steadily growing smaller each year. The total fire losses in the United States per year is steadily increasing, altho that of Canada is always decreasing at an appreciable rate.

The total fire losses in the United States for 1930 were about ten per cent higher than those for 1929, tho there were fewer big fires in 1930 than in 1929. Fire losses in 1930 amounted to approximately five hundred million dollars in this country.

METHOD

My method of testing the flammability of wood pulp was based upon the use of a standard flame and timing the period between the instant of placing the burner flame directly beneath the ball of pulp and the instant in which the pulp burst into flame. I always used the same burner, an ordinary Tyrrel with a hexagonal base, and had two blocks fixed in the shape of a wide V to fit the shape of the burner base. These blocks were glued and bound in permanent position upon the upper surface of the flat base of an iron stand, and acted as stops, enabling me to quickly place the burner in standard position by thrusting the base of the burner into this V. Above the burner, supported by an iron ring, was the wire gauze upon which I had placed the ball of pulp prior to placing the burner in position. This plain wire gauze was coarse and of open weave, permitting the free passage of air currents and heat waves, and was 7 cm. above the top of the burner when in position. The balls of pulp which I used for testing were made by taking approximately 0.1 gram of pulp and rolling it into a ball with my thumbs, index and middle

fingers of each hand, compressing it slightly to about 1.0 cm. in diameter. The standard flame that I used was at first defined as that flame coming from burner "A" (the Tyrrel burner described above) when the air and gas were so regulated as to give a column of unburned gas 1.5 cm. high when just enough air was admitted to dispel all traces of luminous carbon flame. However, in preliminary practice I found that this was too broad a definition, and on different days gave widely different times of ignition for samples of pulp taken from the same batch. I attributed this to fluctuations in the gas supply, carburetted gasoline being the source of fuel. For this reason, I redefined my standard flame to be that flame coming from burner "A", which, when placed with the burner-top 7 cm. below the wire gauze, ignited a standard ball of raw pulp as received from the Masonite plant in an average of between 11.0 and 12.0 seconds, five readings being taken. I found it quite difficult to produce this standard flame, but once so regulated I found it quite satisfactory.

At first, my procedure was to place the burner in position, then to place the wire gauze containing the pulp-ball in position above it, but the results varied widely. The method which I finally adopted was as follows: I regulated my flame to standard by adjusting air and gas and testing the ignition time of standard untreated pulp by the procedure which I am now describing: I made the pulp up into the usual ball, placed it in the center of the wire gauze, slid the burner into position below it as rapidly as possible without causing the flame to waver, pressing the starter of a tenth-

second stop-watch the instant that the burner reached position, and stopping the watch the instant that the pulp burst into a free flame, sparks being disregarded. The time recorded by the stop-watch I called the ignition time of the pulp. I found that to obtain the correct ignition time of any pulp it was essential that the pulp-ball be placed in exact position on the wire gauze so that the hot-air currents should be the same on all sides of the ball. Furthermore, I found that, altho I carried out all experiments in the hood, closed on three sides, it was necessary to have all nearby windows and doors closed and to stand back away from the burner while timing, so that my breath did not cause the flame to waver.

The above is a description of the procedure which I employed in testing the flammability of all loose pulp. I was unable to devise an accurate method for timing the ignition of blocks of finished wallboard, etc.

SOME PRELIMINARY EXPERIMENTS

I made up a solution of 0.100 gram of $(\text{NH}_4)_2\text{PO}_4$ in 200 ml. of water and soaked 10.000 grams of dried pulp in it for five days. At the same time, I soaked 10.000 grams of dried pulp in 200 ml. of pure water for five days. At the end of this time, I filtered each by suction and dried each in the oven at 115°C for two hours. I collected 2.5 ml. of the filtrate from the treated pulp in a small test tube in another similar test tube, I placed 2.5 ml. of a solution of $(\text{NH}_4)_2\text{PO}_4$ identical with that into which I had placed the pulp. To each test tube I added the same amount of Magnesia mixture and

ammonium hydroxide and let the precipitate settle for 24 hours. At the end of this time, the volume of precipitate was greater in the test tube which had contained the filtrate from the wood pulp. Altho I am unable to account for the excess volume, it was evident that the amount of adsorption of the $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ by the wood pulp was negligible if at all.

I timed the ignition of 20 balls of pulp from the batch soaked in the $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ solution, 20 balls from the batch soaked in pure water, and 20 balls from the batch of plain pulp which had not been wet, but had been dried in the oven also at 115°C for two hours. All three batches had been cooled in dessicators. I used the same flame for each batch, tho it was not my conventional standard. The results are displayed in the following tables in seconds:

Ordinary pulp	Soaked pulp	Treated pulp
4.2	6.2	5.6
8.1	4.2	3.7
4.6	4.9	3.9
5.7	7.0	4.1
5.4	3.6	4.8
6.8	3.8	8.6
4.6	7.8	7.7
5.5	9.4	6.0
4.4	4.2	4.2
11.8	5.7	9.4
5.5	7.0	7.6
7.5	4.0	7.2
6.4	4.4	5.8
5.2	9.1	3.9
5.7	7.9	4.0
9.0	5.0	8.4
7.2	4.6	4.0
4.3	6.2	4.2
5.8	4.0	4.5
3.9	3.8	5.0
Means:	6.0	5.6

It is evident from these means that, if anything, soaking in such a dilute solution of $(\text{NH}_4)\text{H}_2\text{PO}_4$ makes the pulp still more flammable.

I cut some "Structural Insulation" into small blocks about three centimeters square, and treated them in the following ways:

- (1)- I left untreated.
- (2)- I smeared a 36% solution of sodium silicate over all six sides, wetting them thoroly.
- (3)- I wet with a 36% solution of potassium silicate.
- (4)- I wet with sodium silicate and sprayed with hydrochloric acid.
- (5)- I sprayed with sodium silicate solution and wet with hydrochloric acid.
- (6)- I sprayed with sodium silicate solution.
- (7)- I sprayed with sodium silicate solution and wet with alcohol.
- (8)- I wet with alcohol, then with hydrochloric acid, and sprayed with sodium silicate solution.
- (9)- I sprayed with "Araclor 1268" (a chlorinated diphenyl) solution in toluene, 200 grams of Araclor per liter of solution.
- (10)- I sprayed with a solution of $(\text{NH}_4)\text{H}_2\text{PO}_4$, 250 grams per liter of solution.

I dried each thoroly in the oven at 115 degrees centigrade.

- (1) burned readily with a free flame, but when this was blown out, the live sparks soon died out.
- (2) had a hard, transparent, glossy surface, and could be ignited neither to a flame nor to a free spark, tho the combustible gasses were distilled out and burned when a flame was held constantly below it for some time.
- (3) had a hard surface, darker than (1), but not glossy, and acted toward a flame as did (2).

- (4) was light colored, hard and stiff, but not glossy, having a uniform coating inside the surface. It burned readily with a free flame, and when this was blown out, burned easily with a free spark.
- (5) had a rough, scaly coating, easily rubbed off. It burned as did (4).
- (6) looked and acted toward a flame as did (2).
- (7) charred very easily and burned with a live spark, but would not give a free flame.
- (8) looked like (5) except that it was badly swelled. It burned as did (4).
- (9) burned even more readily than (1), and would not hold a live spark even momentarily.
- (10) would not give a free flame, but soon cracked open and and burned completely inside by spark leaving only the six black crusts that were the sides.

I found the following to be some of the physical properties of Araclor 1268:

Melting point: 130°C to 200°C

Boiling point: Above 360°C

Solubility in water: Practically insoluble

Solubility in alcohol: Practically insoluble

Solubility in acetone: Very slightly soluble

Solubility in ether: Slightly soluble

Solubility in Benzene: Soluble

Solubility in Toluene: Very soluble

Solubility in Paraffin oil: Soluble in all proportions in hot, but insoluble in cold.

The solubility in all the above Organic solvents is increased by heating, but the Araclor is reprecipitated on cooling.

Araclor 1268 is odorless and almost colorless, melting to an amber-colored liquid which darkens on further heating.

COMPARATIVE EXPERIMENTS

Using a 36% solution of sodium silicate (Sp. Gr. = 1.385), I made up the following solutions, placed 1.00 gram of dried pulp in each in a 50 ml. beaker bearing its number, and allowed to stand on my desk for several days until the volume of solution in each beaker had evaporated to about 15 ml.

No.	Ml. of 36% sol. added	Gm. of Na_2SiO_3 contained	Ml. of Water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
1.	0.02	0.01	25	25	1.00	15
2.	0.04	0.02	25	25	1.00	15
3.	0.07	0.03	25	25	1.00	15
4.	0.09	0.04	25	25	1.00	15
5.	0.11	0.05	25	25	1.00	15
6.	0.13	0.06	25	25	1.00	15
7.	0.16	0.07	25	25	1.00	15
8.	0.18	0.08	25	25	1.00	15
9.	0.20	0.09	25	25	1.00	15
10.	0.22	0.10	25	25	1.00	15
11.	0.44	0.20	25	25	1.00	15
12.	0.67	0.30	24	25	1.00	15
13.	0.89	0.40	24	25	1.00	15
14.	1.11	0.50	24	25	1.00	15
15.	2.22	1.00	23	25	1.00	15
16.	0.00	0.00	25	25	1.00	15

I filtered each by suction and dried each on its numbered watch-glass in the oven at 115°C for two hours. My first attempt at timing the ignition of these samples was

disregarded because the pulp balls were not placed upon the wire gauze with sufficient accuracy. However, I still had enough of each sample left to repeat the tests using a mean of three balls instead of five as I had intended. Below is given the time in seconds between the instant of placing the burner and the instant at which a free flame appeared on the pulp, i.e., standard conditions (pages 4 and 5 of this thesis):

No.	Gms. Na_2SiO_3 per gm. pulp	First ball	Second ball	Third ball	Mean time	Relative time
16.	0.00	8.2	11.8	14.2	11.4	0.0
1.	0.01	12.0	10.0	13.4	11.8	0.4
2.	0.02	12.6	11.0	12.8	12.1	0.7
3.	0.03	11.0	13.6	12.4	12.3	0.9
4.	0.04	12.2	12.0	15.4	13.2	1.9
5.	0.05	12.8	15.0	13.8	13.9	2.5
6.	0.06	11.0	18.2	14.4	14.5	3.1
7.	0.07	12.6	15.0	20.8	16.1	4.7
8.	0.08	18.4	(*)	15.6	17.0	5.6
9.	0.09	17.8	20.2	15.0	17.7	6.3
10.	0.10	18.8	20.4	16.2	18.5	7.1
11.	0.20	(*)	25.0	21.0	23.0	11.6
12.	0.30	32.8	(*)	(*)	(*)	(*)
13.	0.40	*	*	*	*	*
14.	0.50	*	*	*	*	*
15.	1.00	*	*	*	*	*

Symbols: * = Caught a spark and burned completely without a flame at all

() = Disregarded in the mean and relative time

IGNITION TESTS ON PULP TREATED WITH Na_2SiO_3 (Nos. 1-16)



I dissolved 20.000 grams of Araclor 1268 in toluene and made the solution up to 100.0 ml. As in the preceding experiment, I made up the following solutions, placed one gram of pulp in each, and let the solution evaporate over a period of several days.

No.	Ml. of solution added	Gm. of Araclor contained	Ml. of Acetone added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
17.	0.00	0.00	25	25	1.00	0
18.	0.05	0.01	25	25	1.00	0
19.	0.10	0.02	25	25	1.00	0
20.	0.15	0.03	25	25	1.00	0
21.	0.20	0.04	25	25	1.00	0
22.	0.25	0.05	25	25	1.00	0
23.	0.30	0.06	25	25	1.00	0
24.	0.35	0.07	25	25	1.00	0
25.	0.40	0.08	25	25	1.00	0
26.	0.45	0.09	25	25	1.00	0
27.	0.50	0.10	24.5	25	1.00	0
28.	1.00	0.20	24	25	1.00	0
29.	1.50	0.30	23.5	25	1.00	0
30.	2.00	0.40	23	25	1.00	0
31.	2.50	0.50	22.5	25	1.00	0
32.	5.00	1.00	20	25	1.00	0

I dried each on its numbered watch-glass in the oven at 115°C for two hours, and made the following ignition tests under standard conditions. Time is in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
17.	15.4	13.0	11.4	10.0	8.4	11.6	00.0
18.	11.4	11.0	13.8	12.6	10.2	11.8	.2
19.	11.6	13.2	14.2	10.6	11.0	12.1	.5
20.	10.0	14.8	10.8	13.0	13.6	12.4	.8
21.	11.2	13.8	14.0	10.6	14.0	12.7	1.1
22.	14.8	13.8	14.0	12.8	12.8	13.4	1.8
23.	13.8	14.0	14.0	10.2	15.6	13.7	2.1
24.	14.0	14.0	(24.0)	12.0	14.2	13.5	1.9
25.	13.8	15.0	13.0	14.2	14.0	14.0	2.4
26.	14.4	14.0	13.0	14.0	16.2	14.3	2.7
27.	15.0	14.2	15.8	15.0	13.8	14.8	3.2
28.	15.0	16.0	20.0	18.6	16.0	17.1	5.5
29.	16.8	18.6	18.4	25.6	18.0	19.5	7.9
30.	16.2	20.8	22.0	24.6	19.4	22.6	11.0
31.	24.2	28.6	20.2	26.8	20.6	24.0	12.4
32.	45.6	59.2	37.2	45.8	44.2	46.5	34.9

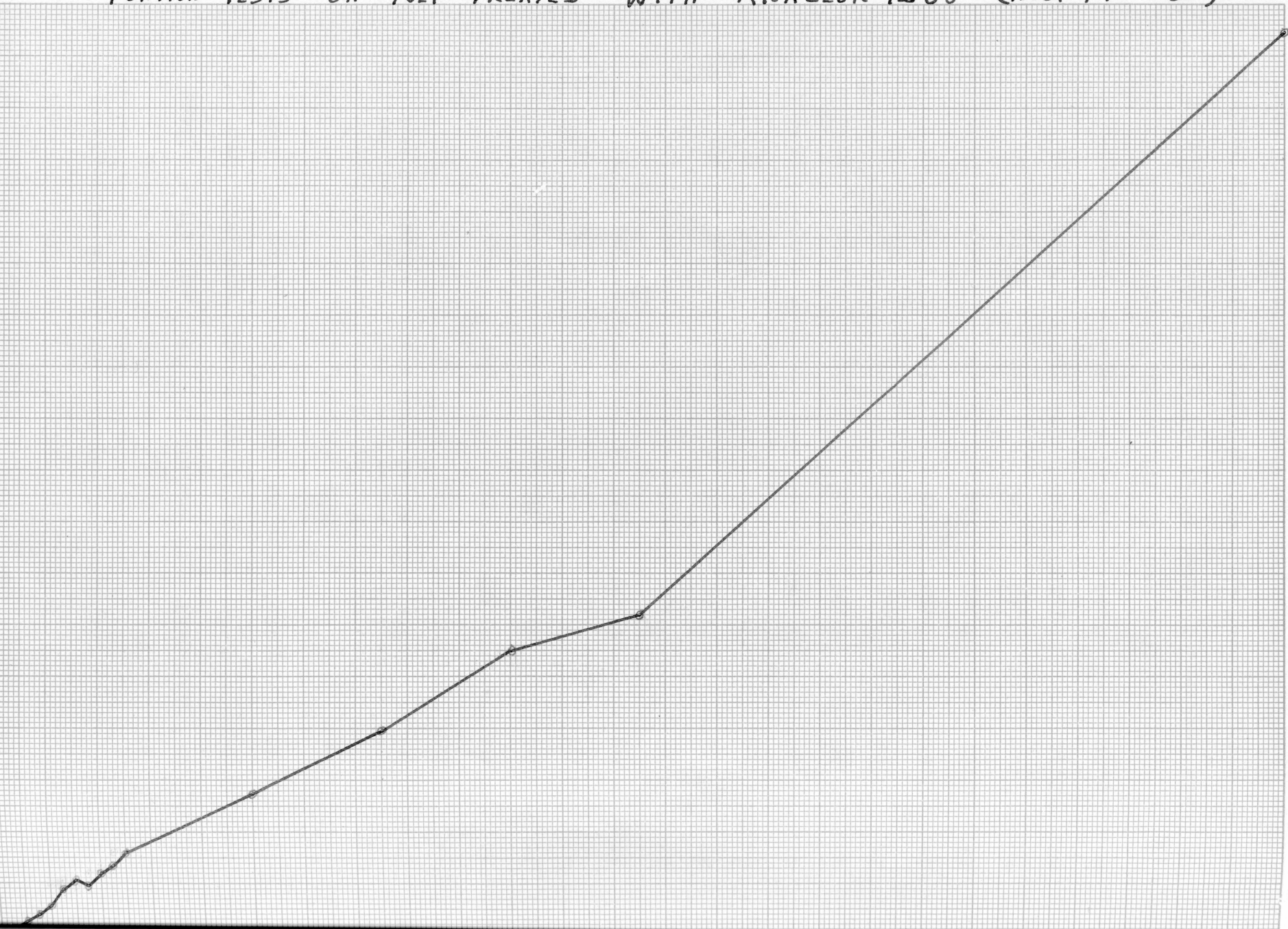
Symbol: () = Disregarded in mean and relative time.

It is a characteristic of this pulp that it would never catch a live spark nor even darken until apparently all of the Araclor distilled out leaving the untreated pulp, which then burned with a free flame. This is in strong contrast with the pulp treated with sodium silicate, which was easily charred and readily acquired a live spark. Another feature of pulp treated with Araclor is that in all the above samples except the last two, the pulp looks and feels like untreated pulp.

IGNITION TESTS ON PULP TREATED WITH ARACLOR 1268 (Nos. 17-32)

RELATIVE
TIME
IN
SECONDS

36
32
28
24
20
16
12
8
4



KEUFFEL & ESSER CO., N. Y., NO. 368-14
Millimeters, 10th lines heavy

Using the same 36% solution of sodium silicate that I used in samples 1 - 16, I made up the following solutions (identical with numbers 1 - 16), and after adding 1.00 gram of pulp to each, I stirred each and left them for several days until the volume of solution in each had evaporated to about 15 ml. :

No.	Ml. of 36% sol. added	Gm. of Na_2SiO_3 contained	Ml. of water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
33.	0.00	0.00	25	25	1.00	15
34.	0.02	0.01	25	25	1.00	15
35.	0.04	0.02	25	25	1.00	15
36.	0.07	0.03	25	25	1.00	15
37.	0.09	0.04	25	25	1.00	15
38.	0.11	0.05	25	25	1.00	15
39.	0.13	0.06	25	25	1.00	15
40.	0.16	0.07	25	25	1.00	15
41.	0.18	0.08	25	25	1.00	15
42.	0.20	0.09	25	25	1.00	15
43.	0.22	0.10	25	25	1.00	15
44.	0.44	0.20	25	25	1.00	15
45.	0.67	0.30	24	25	1.00	15
46.	0.89	0.40	24	25	1.00	15
47.	1.11	0.50	24	25	1.00	15
48.	2.22	1.00	23	25	1.00	15

I filtered each by suction and replaced the samples in their beakers. I added 15 ml. of a very weak solution of HCl to each, stirred each well and let stand for four hours. I then filtered each again by suction and dried them in the

oven at 115°C for two hours. The samples were now so brittle that they fell to a powder when I tried to make balls out of them for the ignition tests. I wet some of the pulp and made a ball of it. but after drying, it could not be picked up without falling to a powder. Thus, ignition tests were impossible.

I repeated the above identically except that instead of filtering the pulp from the 15 ml. of Na_2SiO_3 solution and then adding HCl to the pulp, I added just enough HCl to the 15 ml. of solution containing the pulp to make it acid to litmus. I stirred each and left them standing for four hours. I then tested each again with litmus and found them still acid; so I filtered them by suction and dried them in the oven at 115°C for two hours on their watch-glasses numbered 33 - 48, inclusive. The pulp was now very brittle, but not too brittle for me to make balls of it. Using standard flame and method, I obtained the following ignition times in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
33.	12.5	12.0	11.0	11.3	12.2	11.8	00.0
34.	10.2	11.3	12.4	13.0	12.5	11.9	.1
35.	11.7	12.3	12.2	10.6	11.8	11.7	-.1
36.	10.2	11.4	13.0	11.4	12.0	11.6	-.2
37.	13.1	11.9	11.7	10.2	12.4	11.9	.1
38.	11.9	12.4	13.1	11.8	11.3	12.1	.3

(Continued on the next page)

(Continued from previous page)

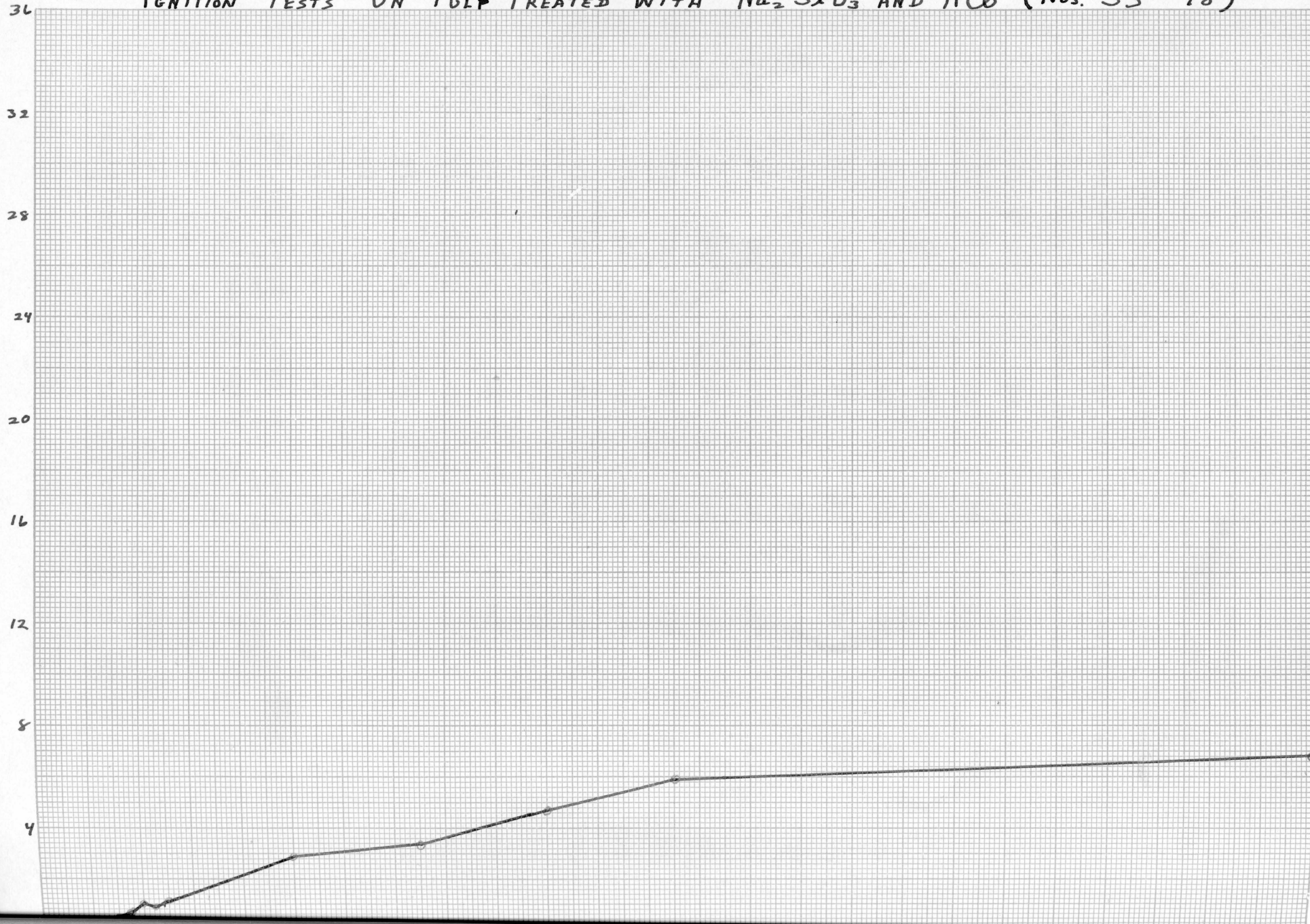
No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
39.	14.0	11.1	12.6	12.7	11.0	12.3	.5
40.	12.8	13.0	10.7	15.1	11.1	12.5	.7
41.	11.4	11.3	12.4	12.9	16.0	12.8	1.0
42.	11.7	12.0	15.2	14.3	10.4	12.7	.9
43.	12.3	10.0	13.3	12.6	16.3	12.9	1.1
44.	15.7	14.0	11.3	16.6	15.7	14.7	2.9
45.	16.3	15.9	16.2	13.6	17.2	15.2	3.4
46.	17.3	17.0	17.2	14.3	16.7	16.5	4.7
47.	13.5	19.3	18.7	17.0	14.0	17.7	5.9
48.	18.5	16.3	14.3	19.6	21.8	18.6	6.8

Only in sample 48 was the silicic acid visible, as a white incrustation. All samples charred easily, held a live spark easily, and as the above table shows, burned with a free flame easily, tho not as readily as the untreated pulp in most samples. The negative values in the "Relative time" column are probably due to experimental error. A graphic representation of the flammability of numbers 33 - 48 is shown on page 19 of this thesis.

I made up an aqueous solution of $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ containing 25.000 grams of $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ in 100.0 ml. of solution. From this solution, I measured out the following amounts of $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ in a series of beakers numbered 49 - 65, number 64 being of a concentration hitherto omitted from the series. I added 1.00 gram of dried pulp to each

IGNITION TESTS ON PULP TREATED WITH Na_2SiO_3 AND HCl (Nos. 33-48)

RELATIVE
TIME
IN
SECONDS



beaker, stirred each thoroly, and left standing for several days until the volume of solution in each beaker had evaporated to about 15 ml.:

No.	Ml. of sol. added	Gm. of $(\text{NH}_4)\text{H}_2\text{PO}_4$ contained	Ml. of water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
49.	0.00	0.00	25	25	1.00	15
50.	0.04	0.01	25	25	1.00	15
51.	0.08	0.02	25	25	1.00	15
52.	0.12	0.03	25	25	1.00	15
53.	0.16	0.04	25	25	1.00	15
54.	0.20	0.05	25	25	1.00	15
55.	0.24	0.06	25	25	1.00	15
56.	0.28	0.07	25	25	1.00	15
57.	0.32	0.08	25	25	1.00	15
58.	0.36	0.09	25	25	1.00	15
59.	0.40	0.10	25	25	1.00	15
60.	0.80	0.20	24	25	1.00	15
61.	1.20	0.30	24	25	1.00	15
62.	1.60	0.40	23	25	1.00	15
63.	2.00	0.50	23	25	1.00	15
64.	3.00	0.75	22	25	1.00	15
65.	4.00	1.00	21	25	1.00	15

I filtered each by suction and dried them on their numbered watch-glasses in the oven at 115°C for two hours. After allowing them to cool, I obtained the following ignition times for the samples. I used the standard flame and method, and recorded the time in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
49.	12.4	12.4	10.2	11.0	11.6	11.5	00.0
50.	9.8	15.6	9.0	11.4	11.6	11.7	.2
51.	11.0	12.5	11.2	10.8	12.0	11.5	.0
52.	11.9	9.6	14.8	14.2	10.0	12.1	.6
53.	20.3	16.4	11.0	15.1	9.5	14.5	3.0
54.	15.9	11.4	14.8	16.7	15.6	14.9	3.4
55.	10.0	16.4	11.8	18.8	18.0	14.8	3.3
56.	15.4	19.3	17.7	11.5	14.0	15.6	4.1
57.	12.5	15.0	17.3	16.2	17.0	15.6	4.1
58.	14.3	17.3	15.4	14.8	15.5	15.5	4.0
59.	18.3	19.0	10.2	16.5	17.0	16.2	4.7
60.	17.5	15.5	16.4	16.6	16.5	16.5	5.0
61.	19.4	22.7	30.2	25.0	22.4	23.9	12.4
62.	22.0	25.2	25.0	24.0	24.3	24.0	12.5
63.	23.0	26.2	(*)	25.0	25.2	24.8	13.3
64.	29.6	(*)	(*)	30.2	(*)	29.9	18.4
65.	*	*	*	*	*	*	*

Symbols: * = Burned completely by spark without any flame.

() = Disregarded in mean and relative time.

As indicated above, these samples showed a tendency to catch a live spark when the amount of Ammonium phosphate was too much to permit a free flame. This was not as marked as with the samples treated with sodium silicate, however.

IGNITION TESTS ON PULP TREATED WITH $(NH_4)H_2PO_4$ (Nos. 49-65)

RELATIVE
TIME
IN
SECONDS

36

32

28

24

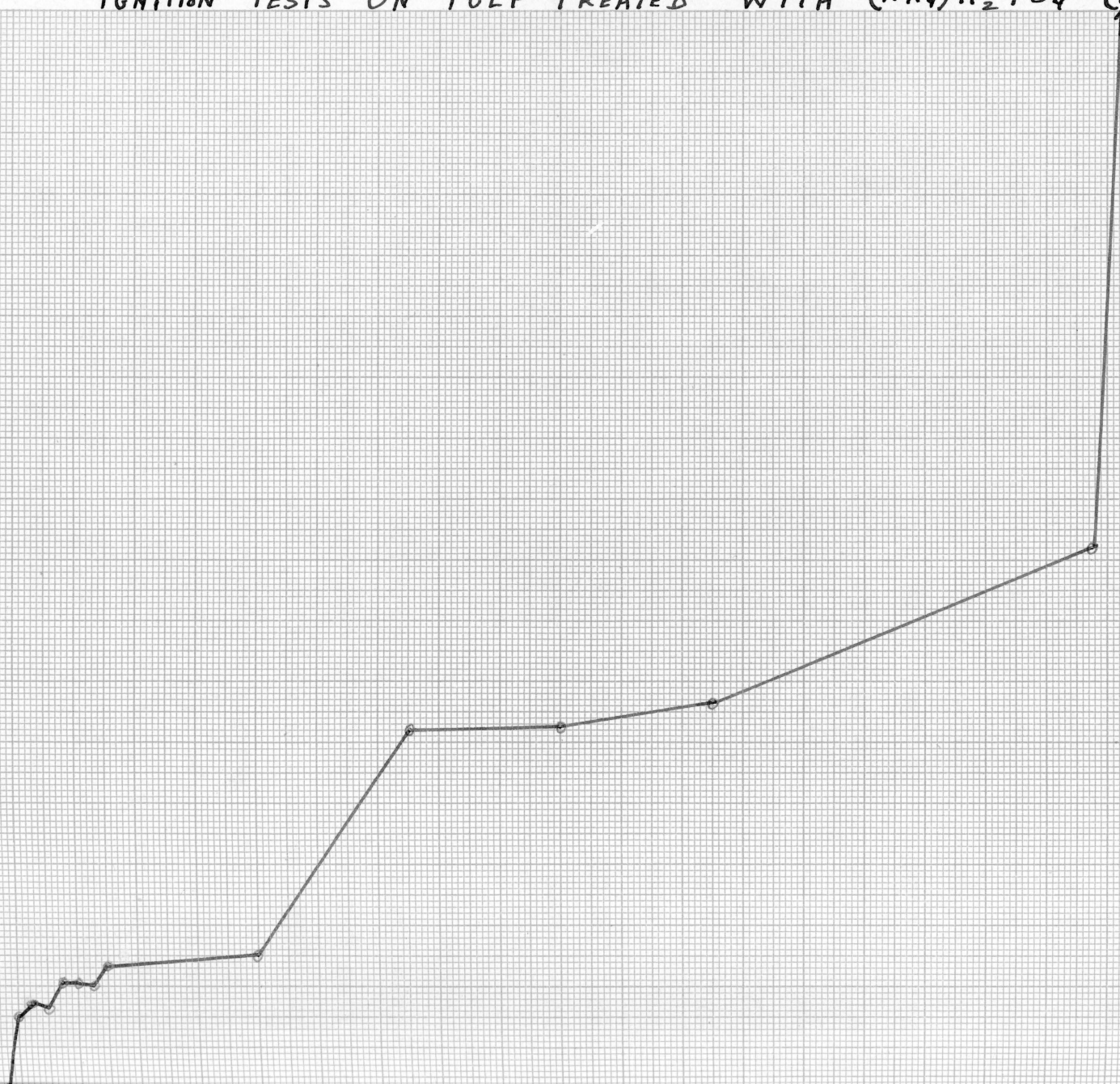
20

16

12

8

4



I measured out accurately 17.20 ml. of an 85% solution of H_3PO_4 (Sp. Gr. = 1.710) and diluted it with water to exactly 100.0 ml. of solution. This gives a solution of 25.0 grams of H_3PO_4 in 100.0 ml. of solution.

Into beakers numbered 66 - 82, I measured out the following amounts of H_3PO_4 , diluted to 25 ml. each, added 1.00 gram of dried pulp, and left standing for several days until the volume of solution in each beaker had evaporated to about 15 ml.:

No.	Ml. of 85% sol. added	Gm. of H_3PO_4 contained	Ml. of water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
66.	00.00	00.00	25	25	1.00	15
67.	0.04	0.01	25	25	1.00	15
68.	0.08	0.02	25	25	1.00	15
69.	0.12	0.03	25	25	1.00	15
70.	0.16	0.04	25	25	1.00	15
71.	0.20	0.05	25	25	1.00	15
72.	0.24	0.06	25	25	1.00	15
73.	0.28	0.07	25	25	1.00	15
74.	0.32	0.08	25	25	1.00	15
75.	0.36	0.09	25	25	1.00	15
76.	0.40	0.10	25	25	1.00	15
77.	0.80	0.20	24	25	1.00	15
78.	1.20	0.30	24	25	1.00	15
79.	1.60	0.40	23	25	1.00	15
80.	2.00	0.50	23	25	1.00	15
81.	3.00	0.75	22	25	1.00	15
82.	4.00	1.00	21	25	1.00	15

I filtered each by suction, dried them in the oven at 115°C for two hours, and under standard conditions obtained the following ignition times in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
66.	11.0	10.6	12.0	12.2	12.8	11.7	00.0
67.	12.8	10.5	12.2	12.6	11.4	11.9	.2
68.	10.0	13.4	13.0	12.0	11.5	12.0	.3
69.	13.0	12.4	12.0	11.6	11.6	12.1	.4
70.	11.2	14.0	13.3	12.0	11.6	12.4	.7
71.	14.3	11.0	12.4	13.3	13.2	12.8	1.1
72.	14.1	14.9	11.2	12.2	14.7	13.3	1.6
73.	12.0	16.1	15.0	13.7	13.2	14.0	2.3
74.	11.2	19.0	16.2	14.6	12.5	14.7	3.0
75.	15.3	16.8	16.2	12.2	17.3	15.5	3.8
76.	16.0	22.2	13.0	14.2	16.5	16.4	4.7
77.	13.2	29.3	18.4	22.0	17.3	20.0	8.3
78.	29.5	28.2	(*)	(*)	29.0	28.9	17.2
79.	(*)	(*)	39.3	(*)	(*)	39.3	27.6
80.	*	*	*	*	*	*	*
81.	*	*	*	*	*	*	*
82.	*	*	*	*	*	*	*

Symbols: * = charred completely without flame

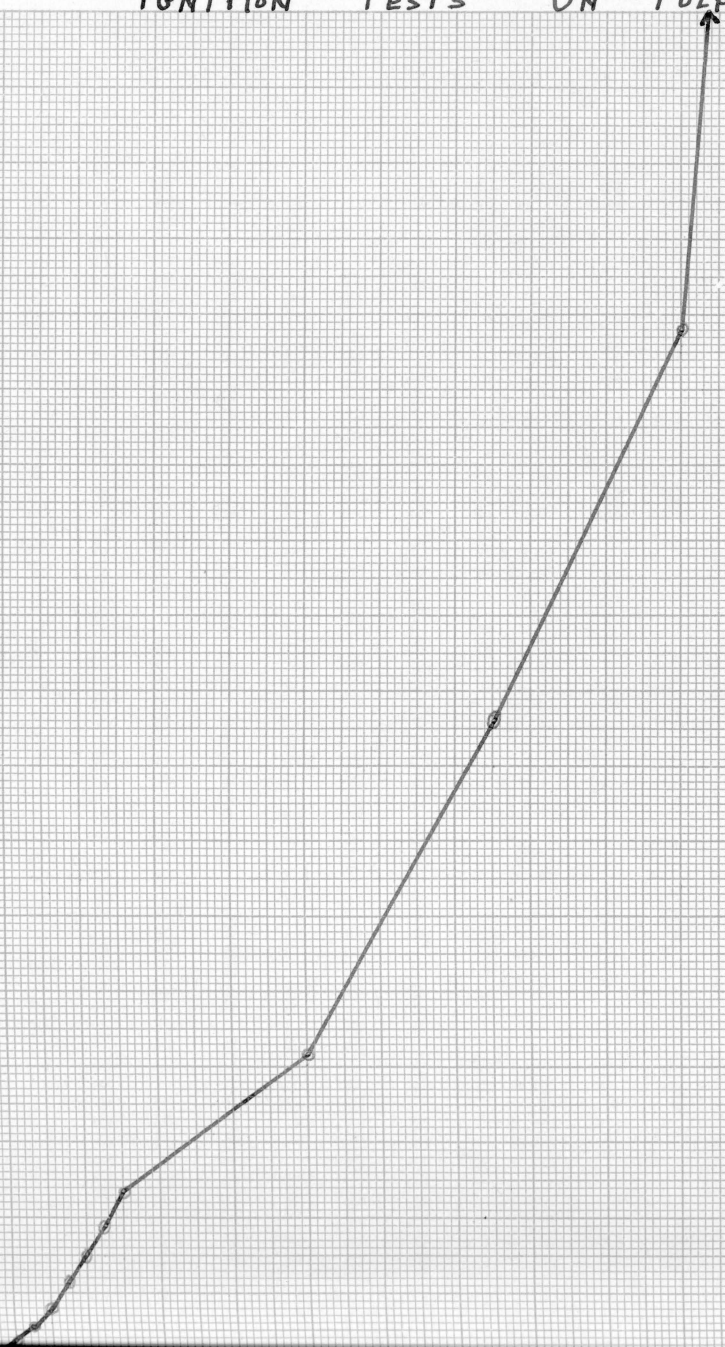
() = Disregarded in mean and relative time

Because of the dehydrating action of concentrated H_3PO_4 , the last five samples were slightly charred while drying, and all of them more quickly than normal while being tested.

IGNITION TESTS ON PULP TREATED WITH H_3PO_3 (Nos. 66-82)

RELATIVE
TIME
IN
SECONDS

36
32
28
24
20
16
12
8
4



I weighed out 25.00 grams of $(\text{NH}_4)_2\text{CO}_3 \cdot \text{H}_2\text{O}$ and immediately dissolved it in cold water, making the solution up to 250 ml. Using this solution, I made up the following solutions in beakers numbered 83 - 99. To each solution, I added 1.00 gram of dried wood pulp, stirred thoroly, and left on my desk for several days until the volume of solution in each beaker had evaporated to about 15 ml. :

No.	Ml. of sol. added	Gm. of $(\text{NH}_4)_2\text{CO}_3 \cdot \text{H}_2\text{O}$ contained	Ml. of water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
83.	00.00	00.00	25	25	1.00	15
84.	0.10	0.01	25	25	1.00	15
85.	0.20	0.02	25	25	1.00	15
86.	0.30	0.03	25	25	1.00	15
87.	0.40	0.04	25	25	1.00	15
88.	0.50	0.05	24.5	25	1.00	15
89.	0.60	0.06	24	25	1.00	15
90.	0.70	0.07	24	25	1.00	15
91.	0.80	0.08	24	25	1.00	15
92.	0.90	0.09	24	25	1.00	15
93.	1.00	0.10	24	25	1.00	15
94.	2.00	0.20	23	25	1.00	15
95.	3.00	0.30	22	25	1.00	15
96.	4.00	0.40	21	25	1.00	15
97.	5.00	0.50	20	25	1.00	15
98.	7.50	0.75	17.5	25	1.00	15
99.	10.00	1.00	15	25	1.00	15

I filtered each by suction, dried them in the

oven at 115°C for two hours, and under standard conditions, obtained the following ignition times in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
83.	11.5	12.0	11.6	11.2	11.3	11.5	00.0
84.	11.0	10.8	11.8	10.9	11.6	11.2	-.3
85.	11.3	11.7	11.6	11.8	10.8	11.4	-.1
86.	11.2	10.9	12.0	11.5	11.8	11.5	0.0
87.	12.5	12.0	11.3	11.1	10.0	11.4	-.1
88.	11.1	11.2	10.9	12.1	11.1	11.3	-.2
89.	11.8	11.5	11.6	10.8	12.3	11.6	.1
90.	12.2	11.6	10.1	11.3	11.5	11.5	0.0
91.	11.0	11.4	11.6	10.6	10.7	11.3	-.2
92.	12.1	11.9	11.2	12.0	10.7	11.6	.1
93.	11.5	11.7	11.1	12.0	12.4	11.7	.2
94.	11.4	11.8	11.6	12.3	12.4	11.9	.4
95.	13.0	12.8	12.9	12.1	11.6	12.5	1.0
96.	13.2	12.5	12.9	11.8	13.2	12.7	1.2
97.	12.3	14.0	11.9	12.8	13.5	12.9	1.4
98.	13.3	13.5	13.3	13.4	13.8	13.5	2.0
99.	16.0	14.8	13.2	14.8	15.9	14.9	3.4

The negative figures were probably due to experimental error and to the fact that a large amount of the Ammonium carbonate had already decomposed in the oven while being dried. The pulp was odorless, and looked like untreated pulp. Except for the slight difference in time, it burned like untreated pulp.

IGNITION TESTS ON PULP TREATED WITH $(NH_4)_2CO_3 \cdot H_2O$ (Nos. 83-99)

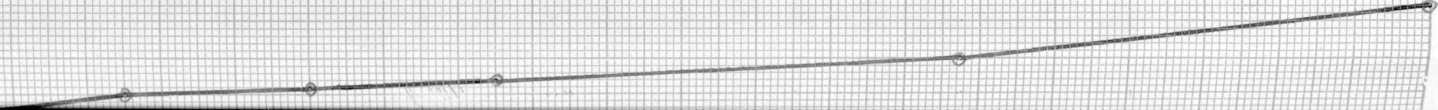
R
E
L
A
T
I
V
E

T
I
M
E

I
N

S
E
C
O
N
D
S

36
32
28
24
20
16
12
8
4



I weighed out 5.000 grams of $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$, dissolved it in water, and made up to exactly 50.0 ml. of solution. Using this solution, I made up the following in beakers numbered 100 - 116. To each solution, I added 1.00 gram of dried pulp, stirred thoroly, and left on my desk for several days until the volume of solution in each beaker had evaporated to about 15 ml.:

No.	Ml. of sol. added	Gm. of $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ contained	Ml. of water added	Approx. ml. sol. at start	Gms. pulp added	Approx. ml. sol. at end
100.	0.00	0.00	25	25	1.00	15
101.	0.10	0.01	25	25	1.00	15
102.	0.20	0.02	25	25	1.00	15
103.	0.30	0.03	25	25	1.00	15
104.	0.40	0.04	25	25	1.00	15
105.	0.50	0.05	24.5	25	1.00	15
106.	0.60	0.06	24	25	1.00	15
107.	0.70	0.07	24	25	1.00	15
108.	0.80	0.08	24	25	1.00	15
109.	0.90	0.09	24	25	1.00	15
110.	1.00	0.10	24	25	1.00	15
111.	2.00	0.20	23	25	1.00	15
112.	3.00	0.30	22	25	1.00	15
113.	4.00	0.40	21	25	1.00	15
114.	5.00	0.50	20	25	1.00	15
115.	7.50	0.75	17.5	25	1.00	15
116.	10.00	1.00	15	25	1.00	15

I filtered each by suction, dried them in the

oven at 115°C for two hours, and under standard conditions obtained the following ignition times in seconds:

No.	First ball	Second ball	Third ball	Fourth ball	Fifth ball	Mean time	Relative time
100.	10.2	13.3	10.3	11.6	12.2	11.5	0.00
101.	10.8	11.0	13.2	12.0	11.1	11.6	.1
102.	11.6	11.9	12.3	11.2	11.9	11.8	.3
103.	12.0	12.1	11.6	11.5	11.3	11.7	.2
104.	11.6	12.7	11.8	12.1	11.1	11.9	.4
105.	11.6	12.1	12.7	11.9	12.8	12.2	.7
106.	12.0	11.8	11.9	12.9	14.6	12.6	1.1
107.	14.1	13.4	12.8	13.2	11.5	13.0	1.5
108.	13.1	13.8	13.2	14.6	12.6	13.5	2.0
109.	13.9	13.8	13.2	13.6	13.9	13.7	2.2
110.	13.7	13.6	13.7	14.2	14.3	13.9	2.4
111.	15.2	15.0	15.2	16.1	16.2	15.7	4.2
112.	18.0	18.3	17.0	17.5	17.8	17.7	6.2
113.	20.0	19.2	17.4	20.6	20.1	19.5	8.0
114.	22.0	24.5	19.0	19.4	(*)	21.2	9.7
115.	23.5	22.9	(*)	23.0	24.1	23.3	11.8
116.	(*)	28.0	30.7	28.3	(*)	29.0	17.5

Symbols: * = Burned entirely by spark without flame.

() = Disregarded in mean and relative time.

This pulp was odorless, and felt like untreated pulp, but shaded off from the normal tan color to an even olive green in number 116. Except for the difference in time, it burnes as does untreated pulp.

IGNITION TESTS ON PULP TREATED WITH $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ (Nos. 100-116)

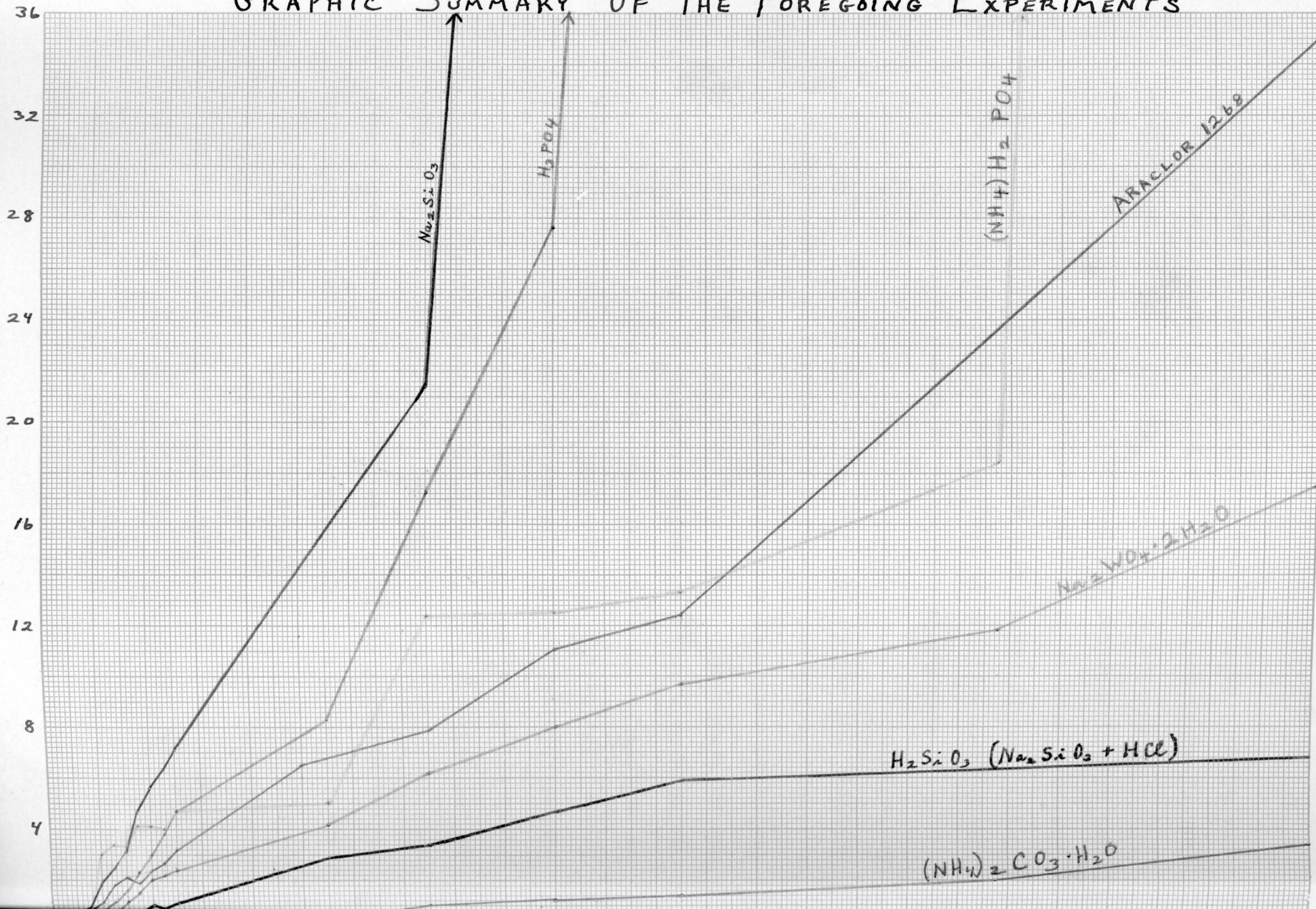
RELATIVE
TIME
IN
SECONDS

36
32
28
24
20
16
12
8
4



GRAPHIC SUMMARY OF THE FOREGOING EXPERIMENTS

RELATIVE TIME IN SECONDS



CONCLUSIONS

An examination of the graphic summary shows that Sodium silicate is the most effective fireproofing agent encountered during these experiments. Such a conclusion as that, however, is recognized as unsound and false when we recall that, of all the samples tested, the pulp treated with Sodium silicate caught a live spark most readily and burned most easily without a free flame. As a flameproofing agent, Sodium silicate is without doubt the most effective of all those tried, but for the purpose of making the pulp less combustible, it is far from the best when added to the loose pulp. Sodium silicate must not be condemned, however, as useless for fireproofing, for "Structural Insulation", coated with a layer of Sodium silicate or potassium silicate, was as incombustible as brick, except for the gasses which distilled out of it when heated for a long time. As a coating, Sodium and Potassium silicates gave almost perfect results, but showed up poorly as a fireproofing agent in loose pulp.

Phosphoric acid shows up second best in the graphic summary, but is probably more useless than nothing at all, for it reacts with the pulp, makes it brittle, and chars it even at 115°C . Its action upon the pulp resembles that of concentrated sulfuric acid.

Leaving the next three in the order of the graphic summary until last, let us look at the last two for a moment: Silicic acid ($\text{Na}_2\text{SiO}_3 + \text{HCl}$), and Ammonium carbonate.

It will be recalled that I had considerable difficulty getting a pulp treated with silicic acid which could be made into balls for testing. It was the most brittle of all the samples, being reduced to a powder by the fingers. As the graph shows, the flame-retarding action of the silicic acid is not very great, and it shares with sodium silicate the property of holding a live spark in the fibers of the wood. The silicic acid is probably dehydrated to silica at the temperature necessary for combustion.

Ammonium carbonate is out of the question as a permanent fireproofing agent because it is not stable even at room temperature. It is probably all driven out as NH_3 , CO_2 , and water before the temperature has reached the ignition temperature of untreated pulp except when there is a large amount of the Ammonium carbonate present.

Mono-ammonium phosphate, as well as I can interpret its graph, shows up very favorably in comparison with all the others mentioned so far. It will be remembered that Ammonium phosphate is mentioned and recommended by both the London County Council and by the National Board of Fire Underwriters of this country. My tests show that pulp treated with Mono-Ammonium phosphate has a slight tendency to acquire a live spark, tho this may not be any more marked than in untreated pulp. The appearance and physical condition of the treated pulp is normal, and as the graph shows, the flame-retardant action of the phosphate is

very noticeable. It is recommended by the writer as a very good, tho not the best, fireproofing agent for loose pulp.

The appearance and physical condition of pulp treated with Sodium tungstate is normal except that the color tends toward a beautiful olive green instead of the normal tan-buff. Altho its flame-retardant action is not as strong as that of Ammonium phosphate, it is less prone to catch a live spark on being heated, tho it sometime does. Its type of action as a fireproofing agent is very probably different from that of Ammonium phosphate. Ammonium phosphate seems to fireproof by giving off NH_3 gas. Sodium tungstate, too, gives off a gas, H_2O , at 100°C ., but this probably helps very little. However, at 698°C Sodium tungstate melts, giving a film of non-reacting salt about the fibers. The residue from Ammonium phosphate is Phosphoric acid, whose action upon pulp has already been described. Sodium tungstate is recommended by the writer as equal in value as a fireproofing agent to Ammonium phosphate.

Araclor 1268 stands out from the foregoing tests as the best all-round fireproofing agent used in the tests. As a flameproofing agent, it ties with Ammonium phosphate, and has the advantage over Ammonium phosphate in that it neither chars nor is in any other way affected by fire until all of the Araclor has distilled out. It even retains its normal color for a time noticeably longer than pulp

treated with any other chemical used in these experiments. It will not catch a live spark even for an appreciable instant, if at all. After the pulp was treated with the Araclor, in those samples containing as much as 0.5 gram of Araclor per gram of pulp the Araclor was visible as a white wax on the pulp. However, when this pulp was heated, the pulp absorbed the excess Araclor, which did not reappear after cooling. Pulp treated with Araclor 1268, heated and cooled, has the normal color and appearance of untreated pulp. Contrary to being made more brittle, it is softened slightly.

In view of the above facts, the writer recommends Araclor 1268 as the best all-round fireproofing agent of all the substances employed in these experiments.