

Jan. 8, 1952

J. B. M. MICHON

2,582,054

PULPING PROCESS

Filed Sept. 12, 1946

2 SHEETS--SHEET 1

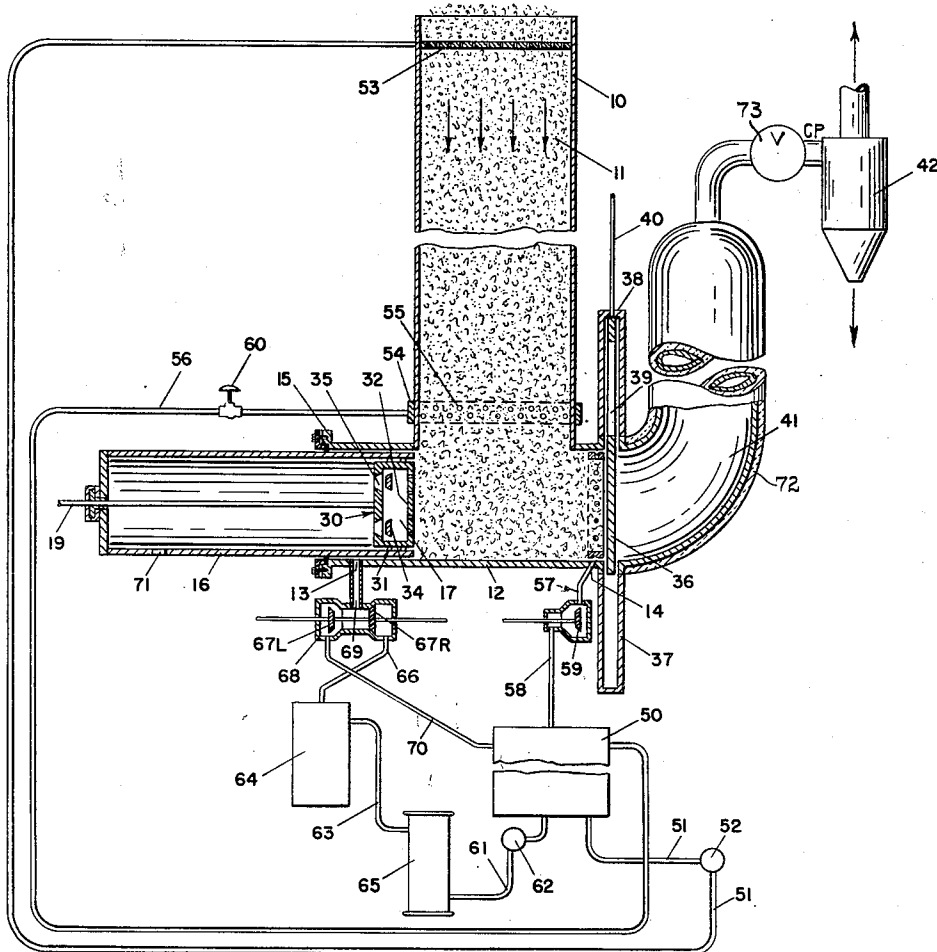


Fig. 1

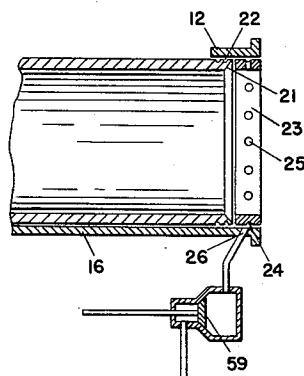


Fig. 2

INVENTOR.

JACQUES BERNARD MARIE MICHON

BY

Dale A. Bauer
ATTORNEY.

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2 SHEETS--SHEET 2

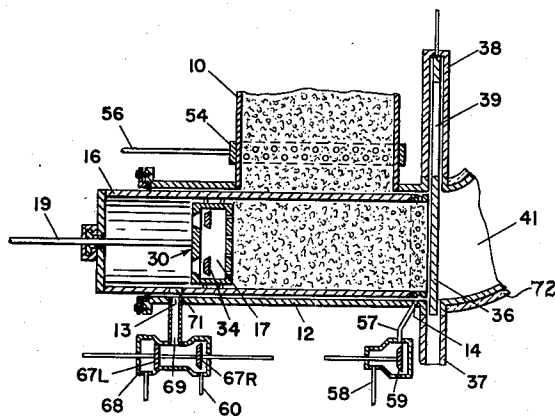


Fig. 3

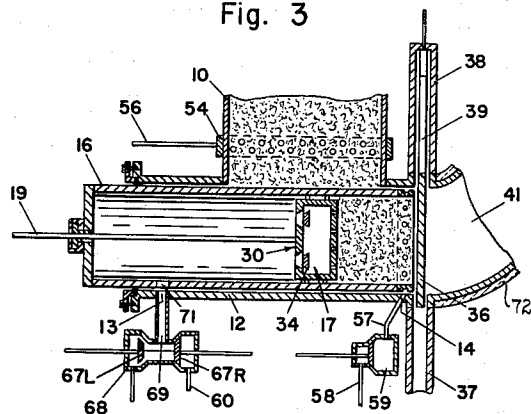


Fig. 4

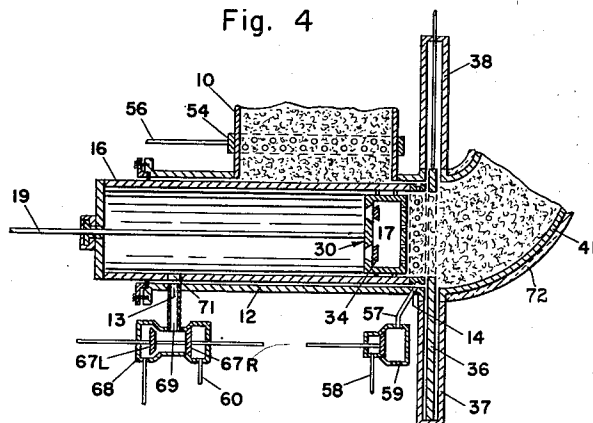


Fig. 5

INVENTOR.

JACQUES BERNARD MARIE NICHON

BY

Dale A. Bauer
ATTORNEY.

UNITED STATES PATENT OFFICE

2,582,054

PULPING PROCESS

Jacques Bernard Marie Michon, Paris, France,
 assignor to La Cellulose Du Pin, Paris, France

Application September 12, 1946, Serial No. 696,603
 In France September 15, 1945

4 Claims. (Cl. 92-13)

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This invention relates to the making of cellulosic pulp by alkaline processes. The raw materials that are useful in the making of such pulps are many, including straw, esparto, bamboo, rushes, bagasse and corn stalks, papyrus, asparagus, pea and bean vines, grasses, and particularly wood. Inasmuch as wood is the most important of pulp sources, the invention will be described in particular relation to the pulping of wood chips, but it is to be understood that this particular description is not a limitation, the process being applicable to the other raw materials with changes in conditions of treatment adapted to their particular state. The process will be described in particular relation to the soda and sulfate processes which are alkaline processes in commercial use in many parts of the world, but it is to be understood that the process is equally applicable with other alkaline cooking liquors.

It is an object of this invention to make pulp having a high percentage of hemi-celluloses, to control the degradation of the hemi-celluloses, and celluloses, while removing the liquor, to remove liquor from cellulosic raw material without dissolving the hemi-celluloses, and to reduce the quantity of cooking liquor to that which is actually needful.

Another object of the invention is to reduce the time required to pulp cellulosic material such as chips and, by producing a superior pulp, to reduce the time required for refining it.

Another object of the invention is to make pulp continuously and of high quality by moving a stream of chips along a path and operating upon them to perform the pulping during their progress.

Another object of the invention is to recover the liquor used for lignin removal and to employ it repeatedly.

Another object of the invention is to remove the lignin separately from the hemi-celluloses of woody material.

Yet another object of the invention is to construct novel apparatus adapted to handle cellulosic raw material in accordance with the process of the invention.

In the normal processes of producing pulp from wood chips, the hemi-celluloses are dissolved in the same step that dissolves the lignin, and first, because the hemi-celluloses dissolve at a lower temperature in a cooking liquor of selected concentration. It is a part of this invention to retain hemi-celluloses in the pulp by raising the temperature of the cooking liquor and of the chips, to the temperature at which lignin dissolves, so

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quickly that a large proportion of the hemi-celluloses remain undissolved. This action depends upon the chemical fact that the hemi-celluloses, which dissolve at a lower temperature, do not dissolve readily in alkaline digesting liquor at the temperature at which lignin dissolves. Consequently, by raising the temperature of the digestion mass quickly to the temperature at which lignin dissolves, the hemi-celluloses are retained. In combination with this discovery are others which also contribute to the success of the process. Heretofore, it has been necessary to cook the chips in a quantity of liquor greater than is established by this invention to be necessary or desirable for the production of the best product. It is also a part of my discovery that improved results are secured if the digestion liquor containing the lignin is drained off the cellulosic mass before the final cooking step, the cooking taking place at a temperature above that at which the hemi-celluloses are dissolved in the presence of only that amount of cooking liquor that is absorbed in the cellulosic mass.

It is a still further discovery, forming a preferred but not necessarily an essential part of the invention, that a further improvement in the end product is secured if the chips are impregnated with hot digesting liquor not under pressure prior to the digesting step. Thus, in the preferred form of the invention, which is about to be described in detail, the chips are first impregnated with the cooking liquor, then drowned in a lake of the cooking liquor under pressure at a temperature above that at which hemi-celluloses dissolve, and then cooked in that quantity of liquor which is retained after the liquid is drained off.

In order to carry out the invention in a commercial manner, novel apparatus has been devised which is described herein and diagrammatically shown in the accompanying drawings.

The above and further objects and novel features of the invention will more fully appear from the accompanying drawings when the same are read in connection with the following detailed description. It is to be understood, however, that the drawings are for purposes of illustration only and are not intended as a definition of the limits of the invention, reference for that latter purpose being had primarily to the appended claims.

In the drawings, wherein like reference characters refer to like parts throughout the several views:

Fig. 1 is a vertical sectional view diagrammatically illustrating an apparatus embodying

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principles of the invention and adapted to carry out the novel process;

Fig. 2 is an enlarged detail of a portion of Fig. 1; and

Figs. 3, 4, and 5 are sectional details of the digester at succeeding stages of the cycle.

In the drawings, the numeral 10 indicates a stand pipe, which may be made of sheet metal riveted or welded together, which is open at the top to receive chips 11 which are added thereto by implements at a rate which prevents the stand pipe from being emptied by withdrawals from below. This stand pipe serves particularly for the impregnation of the chips with hot digesting liquor at normal pressure, the temperature of which approaches 100 degrees C. under favorable circumstances. The size of the stand pipe is so related to the rate of withdrawal from below that the chips are well impregnated with the liquor before they are sealed off in the digester. The liquid supply system is related to all the parts of the apparatus and will be described as a unit in connection therewith after the mechanical parts of the apparatus have been completely described.

At its base the stand pipe opens into a cylindrical or tubular digester 12 which is open at both ends, except for sealing means, and which is provided with apertures 13, 14 in the bottom of its wall. It has an external flange 15 at one end which serves to mount a packing gland that seals the joint between it and the inner tube 16. The leading edge 21 of the inner tube 16 is interiorly bevelled as shown in Fig. 2 and exteriorly provided with a sealing labyrinth 22. The inner tube or cylinder 16 fits the digester wall snugly and the grooves or indentations of the labyrinth constitute a joint which is effective for its purpose. The sharp edge of the inner tube abuts, in its sealing position, a perforated ring 23 having an exterior annular groove 24 which connects through the holes 25 with the interior of the digester. The groove is itself aligned at one portion of its circumference with an outlet orifice 26 in the wall of the digester. It would be possible for the liquid contents to escape therefrom even when the digester is sealed except for other sealing means which are provided and which will be described hereinafter in connection with the fluid system.

Within the inner tube is a hollow piston 30 attached to the piston rod 19. The hollow piston is sealed within the tube by piston rings 31 and has a foraminous pressure wall 32, and a valved wall 33. In the valved wall are a plurality of valves 34 adapted to seal the piston when seated in conical seats 35. The valves 34 are normally spring biased to closed position. However, the presence of liquid within the inner tube 16 under sufficient pressure will serve to open them.

In Fig. 1 the apparatus is shown in the first position before digestion has begun and after the stand pipe has been filled with chips. The impregnating liquid has been admitted and has made it way down through the stand pipe so that the chips within the digester are submerged in the hot liquid. The valves 34 are closed so that the liquid cannot escape through the piston.

The end of the digester opposite the inner tube is closed by means of a valve 36 of sliding panel type which is shown in the illustration to be mounted for vertical sliding movement in a well 37, 38. The sliding panel is solid at its lower portion, but is provided with an opening 39 in its upper part. The solid portion of the panel is of

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sufficient extent to seal the end of the digester and the opening is sufficient to permit the entire contents of the digester to be ejected by the piston when the panel has been lowered into the section 37 of the well. A rod 40 is diagrammatically shown as a means for moving the sliding panel, the rod passing through the end of the well which is provided at that point with a stuffing box. The motion of the panel may be secured by hand or by machine. Also connected to the well, on the other side of the panel from the digester is a cooking tower 41 into which the contents of the digester are pushed from time to time, the previous digester charges being advanced upwardly through the tower by each subsequent ejection from the digester. Thus, the cooking material is advanced step-wise until at the upper end of the tower it is ejected through a steam separator 42, the steam escaping upward and the pulp being discharged downward to a beating engine or other selected parts of the refining system.

The raw cellulosic material is inserted in the stand pipe until it is filled, and thereafter as required to keep an adequate supply and to provide for satisfactory impregnation. The inner tube 16 is withdrawn to the position shown in Fig. 1, and the sliding panel 36 is elevated to the position shown in Fig. 1. The raw material therefore fills the stand pipe and the portion of the digester between the piston and the panel. Into this raw material the impregnating liquid is poured by the means shown in the drawing or by other means. The rate of progress of the raw material through the stand pipe is so related to the flow of impregnating liquid that satisfactory impregnation is secured before the chips are sealed in the digester.

The digester having been filled with impregnated raw material, the inner tube 16 is forced into engagement with the ring 23, sealing off the digester from the impregnator. The digester is now completely sealed and hotter liquid under pressure can be admitted through the valve 17 and retained in the digester for a period sufficient to complete that stage of the operation. Upon completion thereof, the liquid is drained off the raw material, the valve 36 is lowered into the well portion 37, and the piston forces the solid contents of the digester, together with the absorbed liquid, through the opening 39 into the tower 41, after which the panel is returned to its sealing position and the piston and inner tube are retracted to admit another charge of impregnated raw material. The cooking tower 41 is maintained at the most satisfactory temperature by whatever heating means are desired.

The apparatus for supplying liquid to the various stages of the process is an important part of the invention. It is to be understood, however, that other valve arrangements and other sources and means of supply can be devised within the principles herein set forth and claimed.

One digesting liquid is preferably employed in all the steps of the process although digesting liquor of different concentration or of different constitution could be employed if any advantage were obtained thereby. The digesting liquors employed are preferably of alkali type such as soda or sulfate digesting liquors. A satisfactory concentration is 70 grams of active alkali (NaOH or NaOH+Na₂S) per litre. This liquid is contained in a reservoir 50 which is diagrammatically indicated in Fig. 1. The reservoir is connected by a pipe line 51 in which is a pump

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52 with an annular spray 53 located at the upper part of the stand pipe and preferably snugly fitting the wall thereof so as not to form a barrier to the passage of raw materials. Wetting of the materials toward the center of the stand pipe is cared for by suitably directing some of the spray orifices. The liquor makes its way downward through the raw materials into the digester and the lower part of the stand pipe, in which is located a means for controlling the level of the liquid. As shown, this comprises a ring 54 sealed in liquid-tight fashion to the outside of the stand pipe and connected to its interior through holes 55 in the wall thereof. A pipe 56 connects the ring with the reservoir so that any excess of liquor beyond that required to fill the digester and the stand pipe to the level of the ring is drawn off and returned to the reservoir. The ring 23 is connected with the reservoir 50 by means of a pipe 57, 58 in which is located a valve 59. When the valve is opened it connects the interior of the digester with the reservoir so that the liquid therein is drained off. When the valve is closed, the digester and the stand pipe may be filled to the level of the ring 54. By means of a suitable valve 60 the level of the liquid in the stand pipe may be raised materially, and by reversing the flow of liquid through the stand pipe, it may be raised to a sufficient height to apply substantial pressures to the raw materials in the digester. In general, however, it is preferred to operate in the manner hereinabove suggested.

The reservoir 50 is also connected to the digester by means of a pipe line 61, in which is a pump 62, leading to a reheater 65 and through a pipe line 63 to a high pressure storage reservoir 64 in which the liquor can be stored until needed. The pressure in the system at this point will be equal to that which is generated by the pump 62 at the temperature furnished by the reheater 65. Under the conditions of pressure therein pertaining, the digesting liquor remains liquid at temperatures above its normal boiling point. The temperature and pressures under which this liquid is maintained are materially higher than those at which the hemi-celluloses dissolve. The capacity of the system is such that materially large quantities of liquid may be mixed with the cellulosic raw material suddenly. Thus, the cellulosic raw material may be, and preferably is, drowned in about four times its weight of the super-heated liquor. The drowning is accomplished by means of a pipe line 66 which connects with a valve 67R which operates on a single stem with valve 67L in the valve chamber 68. As shown in Fig. 1, the valve 67R is closed and the valve 67L is open. Consequently, any liquor in the pipe 69 will drain from the valve chamber 68 through the valve 67L to the reservoir 50 through the pipe line 70.

During the impregnating stage of the process, before the high pressure and temperature liquor is admitted to the cellulosic materials in the digester, the valves 34 are closed. When the inner tube 16 is sealed within the digester an opening 71 is aligned with the opening 13 in the digester wall and is in position to receive a discharge from the pipe 69. The valve 59 is at that time open in order to drain off the low temperature liquid from within the digester. After the draining the valve 59 is closed and the valve 67R is opened while the valve 67L is simultaneously closed. The hot liquid from the high pressure system then passes through pipe line 66, valve 67R, pipe 69, and openings 13, 71 into the inner tube, forcing open

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the valves 34 and filling the digester with the super-heated liquid. The operation of the valves 59 and 67R can be automatically regulated to avoid the formation of any air pockets within the digester.

The digestion is carried on until the solution of lignin is substantially completed and is terminated before the hemi-celluloses are dissolved or too far degraded. When this point has been reached the valve 59 is opened and the liquid is drained from within the chamber, leaving the digested raw material and some residual liquid. This stage is illustrated in Fig. 3.

If desired, means may be provided, operable through the center of the piston rod to unseat the valves 34 and permit the escape of liquids through pipe 69 and valve 67L, valve 67R being simultaneously unclosed.

The piston 30 is now thrust against the digested cellulosic material and compresses it in a compact mass as shown in Fig. 4, simultaneously squeezing out of it substantially all superfluous liquor. Under normal conditions, this amount will roughly equal the weight of the cellulosic material. It is my discovery that this amount of liquor is sufficient to produce an excellent pulp and that material advantages of efficiency and economy are attained by the removal of excess liquors as accomplished by this process and apparatus.

The gate 36 is now opened and the charge in the digester is expelled by the piston into the cooking tower 41, as shown in Fig. 5. After the parts of the digester have been returned to their initial position, the digesting cycle may be started again.

The first impregnation, in tower 10, may be at such concentration, temperature, and time as will produce a dissolution of any fraction of hemi-celluloses desired, or to avoid dissolving any hemi-celluloses. The temperature of the alkali liquor during impregnation in tower 10 may approach 100° C. without dissolving the hemi-celluloses, the concentration of alkali may be about 70 grams per liter, and impregnation may continue for about a half hour. After this impregnation the impregnating liquor is drained off, a charge is sealed off from the mass undergoing impregnation, and about four times the weight of the chips of hot alkali liquor at a temperature at which lignin dissolves and above that at which hemicellulose dissolves namely circa 170-180° C., is suddenly made to drown the drained chips. This raises the temperature of the wood so suddenly to the high temperature at which lignin dissolves that the hemicelluloses are affected little or none. The total temperature rise occurs in about five minutes or less, which does not give the liquor time to dissolve the hemicelluloses. The chip mass is in a sealed chamber or digester during the drowning with the 180° C. liquor and the pressure in the chamber corresponds to the temperature. This is the digesting period and it lasts until the lignin has been dissolved. The liquor is then drained off again and the chips are thrust into the cooking tower where they cook in their absorbed liquor at normal pressure and without the addition of heat or more liquor. The tower should be insulated to retain the heat in the digested mass. The digested mass is moved through the cooking tower by injections of later batches from the digester.

The pulp which is produced by this invention is novel and of superior quality. It is a slow

pulp, it contains a high proportion of hemi-celluloses and it is refined by the usual refining steps of beating, etc., in about half to one-quarter the time required with pulps made by the alkaline processes of the prior art.

The cooking tower 41 is thermally insulated by a suitable insulating layer 72. A valve 73 is mounted on the top of the cooking tower. The shutting and opening positions of the valve 73 correspond to the shutting and opening positions of the valve 36. The shutting position of these valves and the thermal insulation 72 maintain the pressure and the temperature of the pulp in the cooking tower.

The process is economical of reagents and enables the liquor from digestion to be used repeatedly.

The process is economical in fuel consumption because it requires far less fuel to accomplish a result equal to or superior to that attainable in the prior art for the equivalent quantity of pulp.

The total time required for the pulping of standard materials is less than that required by the processes of the prior art. It requires less than five minutes to reach the temperature at which lignin dissolves, and only about half an hour is required for the cooking. This advantage is added to that which is obtained in the quicker beating of the pulp.

Many and obvious are the changes which may be made in the means of delivering the superheated liquor to the digester; that which has been particularly described is deemed to be merely illustrative. Furthermore, the means of sealing off the digester during the lignin removal can be substituted by equivalent means if there is an advantage in doing so. The means particularly described is of simple construction and satisfactory operation.

As many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

What is claimed is:

1. The method of making cellulosic pulp that comprises impregnating natural ligno-cellulosic material with hot alkaline digesting liquor, immersing the material quickly in a large quantity of preheated alkaline digesting liquor under pressure at a temperature circa 170-180° C., digesting the material in said liquor, draining off the liquid from the digested mass, and cooking the mass by its absorbed liquor.

2. The method of making cellulosic pulp that comprises impregnating natural cellulosic-ligneous material in an excess of alkaline digesting liquor having a concentration requisite to the dissolving of lignin and a temperature approaching 100° C., draining off the liquor, immersing the material suddenly in several times its weight of preheated alkaline digesting liquor which is at a temperature above that at which hemicellulose dissolves and circa 170-180° C. at a pressure corresponding to that temperature, digesting the material in the same liquor until the lignin dis-

solves, removing the liquor from the material, removing the mass, and cooking it under thermal insulation in its absorbed liquor in substantial confinement by its retained heat until the pulping is complete.

3. The method of making cellulosic pulp that comprises impregnating natural cellulosic material with alkaline digesting liquor containing about 70 grams of active alkali per litre at a temperature approaching 100 degrees C. for about a half an hour, immersing the material quickly in about four times its weight of alkaline digesting liquor preheated under pressure to a temperature of about 180 degrees C., digesting it in said liquor to remove lignin, draining the liquor from the digested mass, and cooking the mass in confinement, by its retained liquor, at about 170 to 180 degrees C. for about a half an hour.

4. The method of making cellulosic pulp that comprises impregnating natural cellulosic-ligneous material in an excess of alkaline digesting liquor having a concentration requisite to the dissolving of lignin and a temperature approaching 100° C., draining off the liquor, immersing the material suddenly in several times its weight of preheated alkaline digesting liquor which is at a temperature above that at which hemicellulose dissolves and circa 170-180° C. at a pressure corresponding to that temperature, digesting the material in the same liquor until the lignin dissolves, removing the liquor from the material, and cooking it under thermal insulation in its absorbed liquor in substantial confinement by its retained heat until the pulping is complete.

JACQUES BERNARD MARIE MICHON.

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