

Oct. 12, 1954

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2,691,192

MANUFACTURE OF SMOKELESS POWDER

Filed Dec. 27, 1945

2 Sheets-Sheet 1

FIG. 4.

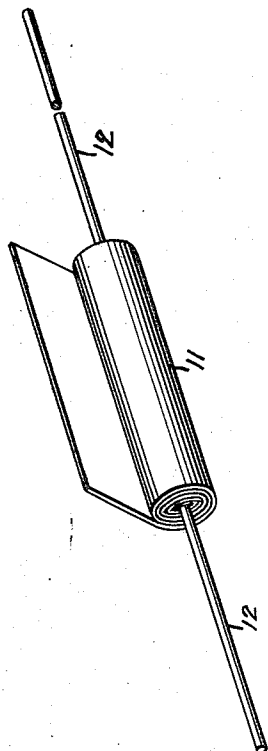


FIG 5

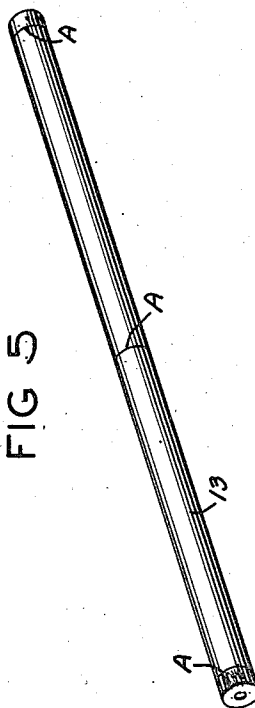
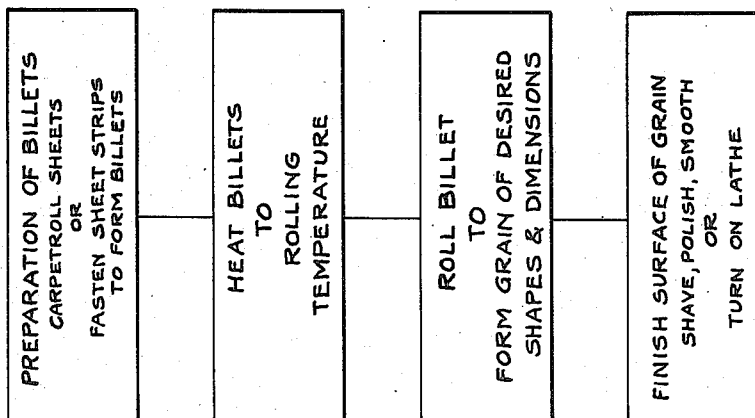


FIG. 1.



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2 Sheets-Sheet 2

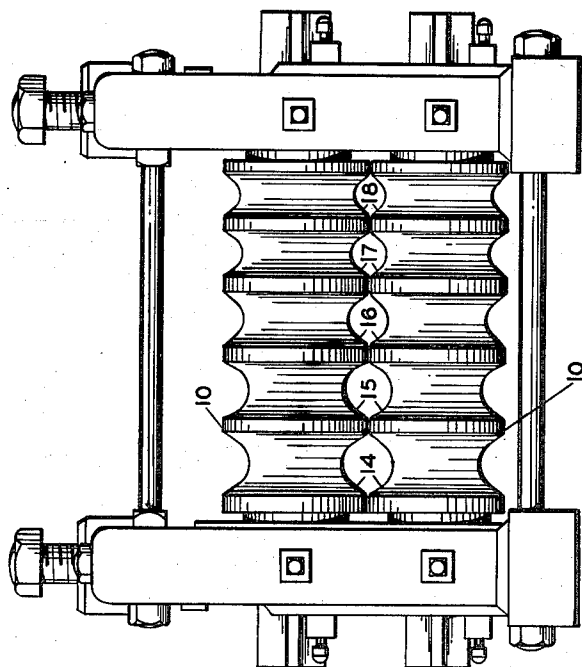


FIG. 3

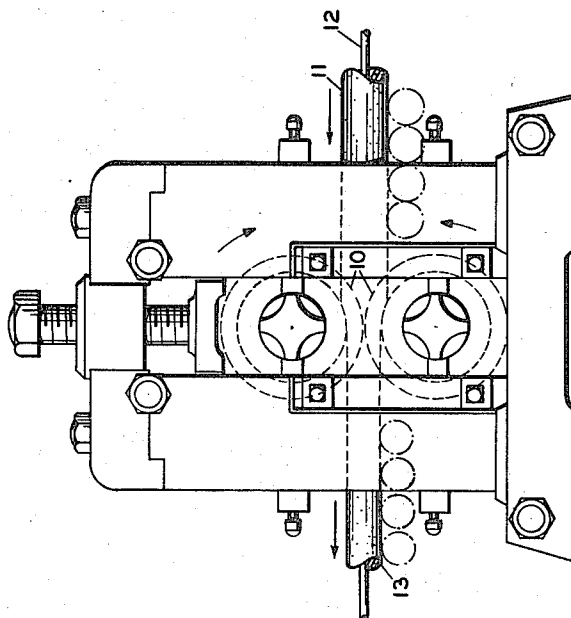


FIG. 2

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MANUFACTURE OF SMOKELESS POWDER

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America as represented by the Secretary of War

Application December 27, 1945, Serial No. 637,380

3 Claims. (Cl. 18—47.5)

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This invention relates to the preparation of smokeless powder. More particularly, it relates to the preparation of stick-type smokeless powder.

In the customary methods of manufacture of smokeless powder, the powder ingredients are incorporated in the presence of volatile solvents. The solvent serves to reduce the sensitivity of the powder ingredients during handling. It also assists in colloidizing the powder mass, and increases its softness and plasticity so that the product may be extruded readily at room temperature. After the extruded strand has been cut into grains of various lengths, the solvent remaining in the grains is removed by extracting with water or by drying in air. However, the rate of solvent removal decreases as the thickness of the grain increases. Where relatively large grains of relatively thick web are desired, the removal of solvent is so slow as to be impractical.

In the preparation of smokeless powder grains of large cross-sectional areas and lengths, commonly known as stick-type powder, it has therefore been necessary to employ powder which has been colloidized without the use of volatile solvents, but present methods of processing this type of smokeless powder have not been wholly satisfactory. The extrusion method commonly followed is a hazardous operation because relatively large amounts of dry powder are confined under extreme conditions of heat and pressure. Furthermore, this operation requires elaborate and costly equipment.

Now, in accordance with this invention, sticks of smokeless powder of both large and small cross-sectional areas are prepared by a simple and relatively safe method which is particularly adapted for use with solventless powders. This is accomplished by forming billets from smokeless powder in various forms and rolling the billets at elevated temperatures to produce shaped sticks or grains of the desired dimensions. A product is obtained in this manner which compares favorably with that prepared by extrusion techniques. If required, the surfaces of these sticks may be smoothed and polished.

The billets employed in this invention may be prepared from sheets, rods or other forms of smokeless powder. For example, smokeless powder sheets may be tightly rolled in the form of carpet rolls, or the sheets may be cut in strips and the strips fastened together to form various shaped billets. These sheets or strips are then consolidated in the course of the rolling process, producing a homogeneous stick. Solid or mono-

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perforated sticks, as desired, may be prepared by this process.

Having now indicated in a general way the nature of this invention, there follows a more detailed description of preferred embodiments thereof with reference to the accompanying drawings.

Fig. 1 is a flow chart showing a preferred method for preparing the sticks according to this invention;

Fig. 2 is a side elevational view of a rolling apparatus which may be employed in the practice of this invention;

Fig. 3 is a front elevational view of the apparatus illustrated in Fig. 2, showing a series of grooves for rolling sticks of circular cross-section;

Figure 4 is a perspective view of sheet powder being wrapped around a mandrel to form a carpetroll or billet thereon; and

Figure 5 is a perspective view of the carpetroll or billet after it has been rolled in the rolling apparatus illustrated in Figures 2 and 3, the mandrel having been removed, and the lines A indicating places where the rolled billet may be cut to produce shorter powder sticks therefrom.

In Fig. 2, a set of grooved rollers 10 is shown in the act of rolling a monoperoforated billet. The unrolled billet 11 has a mandrel 12 extending therethrough. The mandrel is of a size commensurate with the size of perforations desired and is centrally located within the billet. The rollers are operated in the direction shown by the arrows such that they act to draw the billet between them and exert a compressing, consolidating action upon the billet, while simultaneously exerting an elongating action upon the billet. In this manner, a rolled billet or semi-finished stick 13 is thrust forth which is smaller in cross-section but greater in length than the original unrolled billet 11.

In Fig. 3 is shown a front view of the grooved rollers of the same apparatus. The unrolled billet, depending upon its size, may be passed through any of the roller sets 14, 15, 16, 17, or 18, or any combination of these. Thus, for example, a billet may be passed one or more times through roller sets 15, 16, and 17, each pass, in general, corresponding to an operation of the type shown in Fig. 2.

For certain purposes, it may be more advantageous to arrange the groove series on a tandem-type mill in which the grooved rolls, for example, might be placed in line so that the billet would feed directly from one groove to the next.

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In the above illustrations, grooved rolls which rotate continuously in one direction are shown. Other rolling methods, however, may be employed. It would be possible to employ semi-circular grooved rolls or dies which are rocked back and forth over the billet, compressing the powder to the desired configuration or in the manufacture of a monoperforated stick compressing the powder against a mandrel which controls the diameter of the perforation.

The grooves of dies of this type are tapered, one end being slightly larger than the outside diameter of the unrolled billet. As the dies roll over the billet, the gradually diminishing cross-section of the grooves results in a compression of the billet to the size desired.

The following examples are given to illustrate the practical application of the invention:

Example 1

This example describes the manufacture of smokeless powder sticks 1.31 inches outside diameter x 0.31 inch inside diameter x 11.81 inches long. A solventless powder in the form of sheets 23 inches x 12 inches x 0.065 inch was used. The powder had the following formula:

Percent by weight	
Nitrocellulose containing 12.24% nitrogen	49.25
Nitroglycerin	41.50
Ethyl centralite	9.00
Methyl cellulose	0.16
Candelilla wax	0.09
Total	100.00

The ends of these sheets were planed to an angle of 4°. Two sheets with the beveled edges abutting were laid in overlapping relationship upon two similarly arranged sheets, and then wrapped about an 0.312-inch diameter steel mandrel. A billet three inches in diameter and twelve inches long was thereby formed. This billet was tightened in a carpet-rolling machine and the free edge of the outermost sheet was secured by tape. The billet was placed in an oven, heated to 70° C., and kept there for about 10.5 hours to heat the charge uniformly to a suitable temperature for final rolling. The billet was removed and rolled through a series of grooved rolls maintained at a temperature of 70° C., whose grooves had the general design shown in Fig. 3. The billet was passed through each groove twice and rotated 90° between each pass. Powder was forced out of the grooves into the clearance between the rolls in the form of "fins." These "fins" were cut off after each pass. The resulting rolled billet or stick was approximately 33 inches long and 1.5 inches in diameter. This represented a decrease in diameter of 50% and an increase in length of 1.75%.

Two pieces, each 13 inches long, were cut from the center of the stick after removal of the mandrel. These pieces were transferred to steel center-drilled mandrels 24 inches long and 0.312 inch in diameter and then cooled in water to 10° C. The sticks were then mounted on a lathe using the mandrel as a centering core and shaped to a diameter of 1.31 inches and a length of 11.81 inches. These operations were then repeated until 35 finished powder sticks were obtained.

The sticks were burned in a vented chamber having a two-inch inner diameter and a length of 18 inches. The tests indicated that the sticks were well-consolidated and that they burned very regularly under normal working pressure in static tests in a typical rocket motor.

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Example 2

This example describes the manufacture of sticks of smokeless powder 1.31 inches outside diameter x 0.30 inch inside diameter x 12 inches long. Sheets of solventless smokeless powder of the following formula were used:

Percent by weight	
Nitrocellulose containing 12.8% nitrogen	56.81
Nitroglycerin	28.00
Ethyl centralite	4.00
Dinitrotoluene	11.00
Candelilla wax	0.08
Methyl cellulose	0.11
Total	100.00

The dimensions of the sheets used were 24 inches x 11 inches x 0.065 inch. Five-degree bev-els were produced on the ends of the sheets by planing. The sheets were then arranged in overlapping fashion as in Example 1 and rolled about an 0.30-inch diameter mandrel bar to form a billet three inches in diameter and 11 inches long. The billet was heated at 75° C. in an oven for about seven hours. It was then rolled through a series of grooved rolls preheated to 70° C. similar to those illustrated in Fig. 3. The rolled stick was finished as in Example 1 into two sticks 1.31 inches outside diameter x 0.30 inch inside diameter x 12 inches long.

Burning tests conducted on this powder indicated that the powder was satisfactorily consolidated and had a uniform burning rate.

Example 3

Sheets of smokeless powder 110 inches x 13 inches x 0.08 inch were prepared employing the following ingredients:

Percent by weight	
Nitrocellulose containing 12.2% nitrogen	49.50
Nitroglycerin	41.50
Ethyl centralite	9.00
Total	100.00
Candelilla wax (added)	0.08
Methyl cellulose (added)	0.16

The sheets were planed on the ends to give a 3-4° beveled edge and were then heated at 70° C. for 30 minutes to soften them for the next operation. Four sheets were overlapped lengthwise as in Example 1 and wrapped tightly about an 0.75-inch diameter steel mandrel to form a billet 13 inches long and 6.25 inches in diameter. The free edge of the outermost sheet of the billet was taped to keep it from unrolling. The billet was heated in an oven at 70° C. for about 13 hours. It was then removed from the oven and passed, in order, twice through each groove of rolls similar to those shown in Fig. 3. The billet was rotated 90° between each pass. The "fins" formed were trimmed off after each pass. The stick finally produced was about 60 inches long and 2.75 inches in diameter. This represented about a 360% increase in length and a 56% decrease in diameter. The stick was cut to leave the best 44-inch length on the mandrel. This section was cooled for two hours in water at 5° C. and then mounted in a metal-turning lathe and finished to a stick about 2.65 inches outside diameter x 0.75 inch inside diameter x 43.6 inches long.

These sticks compared favorably in performance as a rocket propellant with sticks prepared by extrusion of solventless smokeless powder compositions through a die.

Example 4

Cordite S. U. solventless smokeless powder sheets 23 inches x 12 inches x 0.065 inch were prepared from the following composition:

	Percent by weight
Nitrocellulose containing 12.24% nitrogen	49.25
Nitroglycerin	41.50
Ethyl centralite	9.00
Candelilla wax	0.09
Methyl cellulose	0.16
Total	100.00

The sheets were cut into strips 10 inches x 1.74 inches for use in the top and bottom portions of the desired rectangular billet and 10 inches x 0.68 inch for use in the middle section of the billet in forming a square perforation. The strips were laid in an orderly pile to form a billet 10 inches x 3.5 inches x 1.74 inches having an 0.375-inch square perforation through the center. The strips were secured in place by tape. The billet was heated at 70° C. in an oven for 14 hours. When the billet was removed from the oven, an 0.375-inch diameter steel mandrel was inserted through the perforation and the billet rolled at 69° C. through rolls carrying a series of grooves of rectangular outline at first and a gradual transition to the desired typical outline shown in Fig. 3. The billet was passed through each groove once and the "fins" formed during rolling were trimmed off after each pass.

A well-consolidated stick 37 inches long and about 1.20 inches in diameter was obtained which contained no air streaks. The stick was surface-finished suitable for firing. Thus, billets of rectangular cross-section wherein the powder sheets are laid up in a desired orientation may be used in accordance with the present invention.

In each of the above examples, solventless smokeless powder was employed. The use of this type powder is preferred in the preparation of sticks of relatively large diameter because of the difficulty of solvent removal from thick finished sticks. However, the method according to this invention is well adapted for use with solvent types of smokeless powder.

In the preparation of smokeless powder compositions of this invention, nitrocellulose having a nitrogen content between about 12.0% and about 13.3% by weight has been found operable. Preferably, the nitrogen content should be between 12.0% and 12.7%. The composition may contain, in addition to nitrocellulose, explosive and nonexplosive plasticizers, stabilizers, and selected fillers. The ingredients may be included in any combination within the following ranges: Between about 20 and about 95%, preferably between 45 and 80% of nitrocellulose; between about 0 and about 60%, preferably between 20 and 50% of explosive plasticizer; between about 0 and about 30%, preferably between 0 and 15% of non-explosive plasticizer; between about 0 and about 20%, preferably between 0 and 5% of stabilizer; and, between about 0 and about 60%, preferably between 0 and 15% of fillers.

Among the explosive plasticizers which have been found satisfactory are trinitroglycerin, dinitroglycerin, nitroglycide, dinitrochlorohydrin, nitroglycol, tetranitrodiglycerin, dinitrotoluene, dinitrodiglycol, etc. Nonexplosive plasticizers which may be used include ethyl and other centralites, (diphenyl-ureas), ethyl sebacate, dimethylphenyl-o-tolylurea, ethyl succinate, ethyl

phthalate, triphenyl phosphate, benzyl benzoate, ethyl oxalate, sym-dialkyl ureas, DNX oil, etc. Stabilizers which may be used include diphenylamine, diphenylnitrosamine, carbazol, the centralites, etc. Fillers such as waxes, methyl cellulose, oxanilide, starch, hydrocellulose, etc., may be employed.

The billets in accordance with this invention may be prepared from various forms of smokeless powder. Generally, billets are most conveniently prepared from sheets of smokeless powder. However, billets may also be prepared from blocks or amorphous masses of smokeless powder, and where certain shaped sticks are desired, it is preferable to employ billets approaching these forms. Thus, where a square stick is to be prepared, square billets are preferably employed. By this technique, the rolling period is shortened and excessive waste of the material through "fin" formation is avoided.

For the production of cylindrical sticks, cylindrical-type billets are preferred. The process of preparing such a billet is illustrated in Examples 1, 2, and 3. Cylindrical billets are most readily prepared from smokeless powder sheets of the following dimensions: Between about 0.025 inch and 0.300 inch, preferably between 0.065 inch and 0.200 inch in thickness; between about 24 inches and about 400 inches, preferably between 100 inches and 300 inches in length; and, between about 10 inches and about 45 inches, preferably between 15 inches and 35 inches in width. In forming the billets, one or several sheets of smokeless powder, the exact number depending upon the thickness of the sheets and the size of billets required, are arranged in layers. Preferably, the layers are overlapped lengthwise. It is desirable to have a minimum number of sheet surfaces in the billet; thus, the thickest sheets possible which are still limber enough to be wound tightly should be used. Sheet ends in the billet increase the chance of including air in the strand during rolling. The sheets employed should therefore be as long as possible. It is preferable, for example, to roll two long sheets rather than to roll three short sheets in order to obtain a billet of a certain diameter. If the finished stick is required to be monoperforated, the sheets may be wrapped around a mandrel bar whose diameter is the size of the intended perforation. Beveling the ends of the sheets, preferably prior to rolling, aids in obtaining a stick with a smooth perforation and with a minimum number of streaklike cavities and pockets of entrapped air. The angle of the beveled ends should be between about 2 and about 6°. To avoid excessive inclusion of air in the strand during rolling, it is imperative that the billet be wound as tightly as possible. It should be well secured against loosening during handling prior to rolling. This may be accomplished, as shown in the examples, by taping the edges.

Another method of billet formation which may be employed as convenient for the preparation of rectangular and oddly shaped billets is illustrated in Example 4. The smokeless powder sheets are cut into strips of varying length and width depending upon the dimensions of the intended billet. The strips may then be coated with plasticizer, stacked, and cemented into a solid stick under pressure. In some cases where the presence of excessive plasticizer is undesirable, the strips may be pressed tightly together, thereby securing them in place and effecting consolidation of the strips during rolling.

Besides the methods indicated above, the formation of a perforation or perforations may be accomplished by piercing or drilling a solid block of powder.

The rolling process is expedited by heating of the billets to a suitably elevated temperature prior to rolling, although this is not essential. This may be accomplished by heating the billets in an oven at temperatures between 20 and 90° C., preferably between 50 and 80° C. The upper temperature of the limit is governed by safety considerations. With this exception, the exact temperature used will be dependent upon the thermo-plastic characteristics of the composition being worked. In rolling, the billet is passed through grooved rolls which are heated to a temperature at which the smokeless powder composition is workable.

The rate of reduction of the billet which may be employed is determined by the flow and cohesive characteristics of the powder composition being worked. A slow rate of reduction favors complete welding of the sheets in the billet and keeps at a minimum the chance of including air in the grain. A high rate of reduction gives a stronger consolidating effect but it also increases "fin" formation and the tendency of the billet to break or crack during rolling.

In order to effect total consolidation of the laminate of the billets in the case of the more common types of solventless smokeless powder, a total reduction in cross-section of at least 50% in the course of the groove series is required. Reductions in cross-section as high as 200% may readily be achieved. Usually, however, a reduction between 60 and 90% is sufficient, and is preferred. Peripheral speed of the rolls is between 5 and 100 feet per minute and preferably between 5 and 50 feet per minute.

The design of the grooves used may vary considerably. Grooves of square, rectangular, elliptical and circular cross-sections have been found satisfactory. Flared grooves are preferred since flares reduce the amount of "fin" formation produced during rolling. The choice of groove design should be such as to alter successively the shape of the billet to the shape of the final product and to reduce the cross-sectional area of the billet sufficiently to obtain complete consolidation. The billets may be rotated between passes through the same or succeeding groove so as to further enhance the consolidation process and reduce "fin" formation.

The surfaces of the rolled sticks may in some cases be rough. In this event, the surfaces may be finished by use of cutting and polishing devices, such as shaving dies, lathes or polishing buffers. For example, the stick may be passed between two or more cylindrical revolving rolls with knife edges cut in a helical pattern on each roll. The axes of the rolls are set at an angle with one another so that the stick passed therethrough is reduced in diameter and smoothly finished. A shaving die may be placed at the break of the final grooves in the rolls so that the rolls, in addition to their function in elongating and consolidating the powder grain, would force the stick through the shaving die. The stick would be centered in the die during the operation by the use of sliding guides or rolls. The shaved stick may then be polished by the action

of additional grooved rollers. Dependent upon the physical characteristics of the stick, however, a cooling stage may be found necessary between the rolling and shaving operations to prevent mushrooming over the cutting edge of the die.

The sticks of smokeless powder produced in accordance with this invention are characterized by uniform burning properties and where plasticizer is not employed in the preparation of the billets, by substantially complete homogeneity. Compared with powder sticks prepared by extrusion methods, these sticks have comparable uniform burning rates under normal working pressures.

This process is less hazardous than extrusion methods in the production of solventless powder grains because the application of heat and pressure to large quantities of dry powder in a confined space is avoided. Construction of heavily barricaded buildings is not necessary because of the reduction of this hazard. Less complicated equipment is required.

What I claim and desire to protect by Letters Patent is:

1. The process of preparing perforated, stick-type smokeless powder which comprises forming a laminated billet from sheets of smokeless powder by wrapping said sheets about a mandrel, heating said billet to a temperature between about 20 and about 90° C., passing said billet between grooved rolls at a temperature between about 20 and about 90° C. which reduces the cross-section of the billet by compression while simultaneously elongating the billet whereby a uniformly dense, homogeneous stick is produced, removing the mandrel, and cutting the stick into predetermined lengths.

2. The process of preparing perforated, stick-type smokeless powder which comprises forming a laminated billet from sheets of smokeless powder by wrapping said sheets about a mandrel, heating the billet, passing said billet between grooved rolls which reduces the cross-section of the billet by compression while simultaneously elongating the billet whereby a uniformly dense, homogeneous stick is produced, surface-finishing the stick, removing the mandrel, and cutting the stick into predetermined lengths.

3. The process of preparing monoperforated, stick-type smokeless powder which comprises forming a billet of smokeless powder about a mandrel, heating said billet, passing said billet between grooved rolls which reduces the cross-section of the billet by compression while simultaneously elongating the billet whereby a uniformly dense, homogeneous stick is produced, removing the "fins" produced during said rolling operation, cooling the stick, finishing to size, removing the mandrel, and cutting the stick into predetermined lengths.

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