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SOLID CARBON DIOXIDE PRODUCT AND METHOD OF AND APPARATUS FOR MAKING IT

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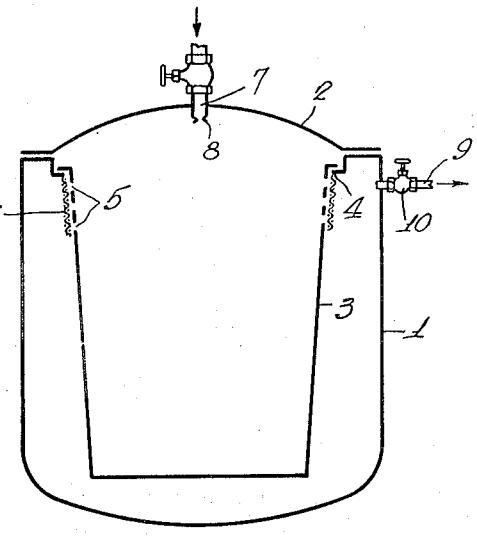


Fig. 1.

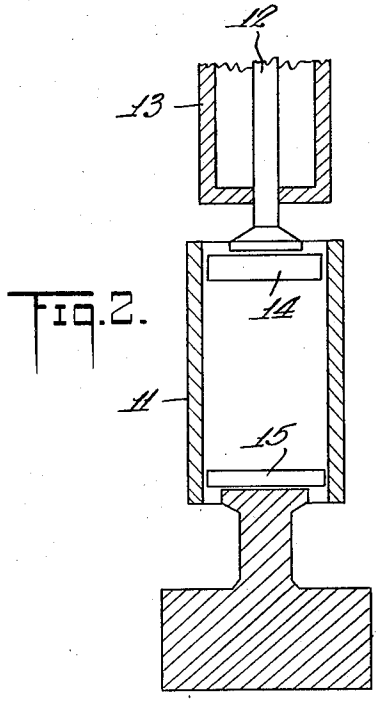


Fig. 2.

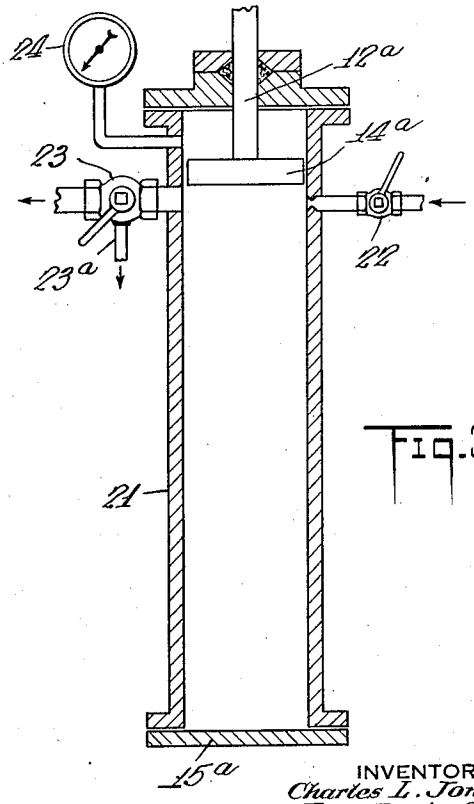


Fig. 3.

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SOLID CARBON DIOXIDE PRODUCT AND METHOD OF AND APPARATUS FOR MAKING IT

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11 Claims. (Cl. 62—121)

This invention relates to blocks of solid carbon dioxide having novel characteristics and qualities and to novel methods of producing such blocks. These novel features are the results of certain discoveries which in turn have resulted from making blocks in accordance with present-day commercial practice, supplemented by exhaustive experimental investigation, as will be evident from the following:

Solid carbon dioxide now in commercial use is made from very fine gritty crystals, produced by jet discharge and the rapid expansion of liquid carbon dioxide to approximately atmospheric pressure, as, for instance, in apparatus like that set forth in Martin Patent No. 1,659,434. These fine crystals are ordinarily compressed into sandy snow-white blocks, usually cubes of relatively large size, say 10 × 10 × 10, the size and the shape being a compromise with a view to getting large volume per unit of heat absorbing area, thereby to avoid excessive evaporation wastage, while also complying with the user's requirement of shape and size convenient for sawing into smaller sizes and shapes for special purposes. The present standard blocks weigh about 70 pounds per cubic foot or, say, 40 pounds for the 10 inch cube. These or similar standards of size, weight and shape are commercially approximated by having the fine grain crystals properly distributed in the mold and the gas expelled therefrom by tamping before pressing, as set forth in Martin Patent No. 1,659,435, but such blocks fail to meet the user's requirements, in certain particulars.

The user expects the block to be of standard density throughout so that when sawed into pieces of given size and shape, all pieces will afford the same amount of refrigerative work regardless of what part of the block it is sawed from. He also expects the block to be tough so that it will not break when being handled or shipped and will not crumble when being sawed into small pieces. The present standard blocks, however, are not of sufficiently uniform density, nor toughness and they rapidly deteriorate, becoming crumbly and sand like, sometimes within a day or two after making.

Our investigations seem to show that this is because the block is composed of hard, gritty, minute crystals that afford frictional resistance to transmission of pressure to the interior of the block; that have enormous surface area per unit volume and that have a large percentage of voids between crystals. When the pressure is applied, the gritty particles seem to weld at their points of contact sufficiently to afford structural resistance to further compression before pressure can be transmitted to the interior and more remote portions of the block. In any event, it has been found practically impossible to eliminate the voids between particles or to make the density uniform throughout the block by any of the methods heretofore known. The voids make the blocks bulky. Moreover, they connect one with another and form continuous passages or pores, affording opportunity for interior evaporation and flow of gas between different parts of the interior and the surface of the block, as is evident from the flocculent crater-like formation of frost on the surface of the block when it is exposed to the atmosphere. Without committing ourselves to details of theory, it seems to be a fact that in the present standard blocks, interior sublimation and outflow of gas facilitates rapid deterioration of the interior to a crumbly condition, while refreezing of the down flowing or outflowing gas increases the hardness and glassy brittle quality of other portions of the block, notably the exterior or other parts that are already densest.

Others have employed certain materials such as water ice and low freezing point liquids, with a view to lubricating the gritty particles and also to afford a cement or binder between them, but such adulterants are all objectionable, water because it is brittle when frozen and wet when melted, and the others for various reasons.

We have tried applying greater pressures, but the result is to increase non-uniformity as well as density, probably due to the sandy, gritty quality of the fine crystals, which prevents proper transmission of the increased pressure into the interior of the block. A thin layer of the block, at the plunger-contacting face, becomes glassy and hard, but it also becomes very brittle. Under very great pressures such as a thousand pounds or more per square inch, this brittle glassy formation may extend further inward, but it merges into a porous snow formation similar to that of the present standard commercial blocks, although the grain may be finer and the density greater. Using a relatively warm mold may result in similar vitrefaction of a thin skin on the other faces of the block. Such high pressure composite blocks are uncommercial for ordinary purposes, one objection being that they are of inferior toughness. Another is that when sawed in given sizes and shapes, the pieces are not standardized, those from the denser portions being too brittle and having far more refrigerant value than those from the snowy portion. Another quite unexpected objection is that ordinary saws which operate satisfactorily on products of relatively uniform density, either dense or porous, will break when used on the composite products. While we contemplate sawing off the dense layer and using it as a separate commercial product, it is evident that if these high pressure composite blocks have ever been produced by others, either accidentally or experimentally, they have not been used for

commercial purposes, because they do not answer present commercial requirements.

In this situation, the object of the present invention is to provide a block and method of making it economically and in large quantities, said block being superior as to internal stability, high uniform density throughout, and so much tougher than any solid carbon dioxide block ever before produced as to amount to a new order of toughness, said toughness resulting from a novel form of crystalline structure.

The block which we produce is difficult to describe because it is unlike anything of which we have knowledge. Its exterior, when free from frost of the atmosphere, appears somewhat like polished, more or less translucent, white, or cloudy agate or even more like a perfect, uncracked cake of camphor ice. Its interior or fracture surfaces appear somewhat like those of camphor ice and alum but differing from either as noticeably as they differ from each other.

All tests that can be applied externally or to the fracture surfaces show the internal structure to be substantially solid and free from through-extending voids or pores such as would permit internal evaporation and out-drainage of gas. One indication of this is that frost from the air forms on the surfaces at a much slower rate and is characteristically different in appearance and formation to that formed on the standard commercial blocks, the frost deposit being relatively smooth except for slight ridges along scattered lines where surface chilling of the block may have caused slight superficial check marks. These checks, when formed are characteristic, being entirely superficial and extremely minute, so that they do not materially increase the evaporation rate or the structural stability, strength or toughness of the block as a whole. Sometimes traces of impurities such as iron, being frozen out of the crystals, are visible as a slight veining effect.

We have discovered that these novel qualities, particularly the extraordinary toughness of our blocks, result not merely from decreasing the voids and eliminating the through extending pores, but also, and more important, from novel methods of recrystallization such that the block consists in large part, of relatively large fernlike crystals of substantial length, that are intermeshed, interlocked and solidly bonded as by plate welding, probably by amorphous solid carbon dioxide. We use long crystals for making our block and they appear laminated and quite flexible, so the tensile strength and flexibility of large individual crystals that appear in the block are likely to be important factors of its strength and toughness. Such a block is fundamentally different from the standard blocks wherein the voids are great and the structural strength depends on the point welding between minute crystals. The crystals as they appear on the fracture surfaces of our product are of relatively large size, say $\frac{1}{16}$ to $\frac{1}{4}$ inch in length and sometimes some of them are very much longer than this.

In the preferred embodiments of the invention, a 10-inch cube may weigh over 50 pounds as against the 40 pounds for the standard commercial block above described, and it can be subjected to shocks and mishandling without damage, that would completely shatter the ordinary commercial block. The less perfect embodiments of our invention may vary considerably in all of the above respects.

While some of the qualities of our product are distinguishable independently of the method of making, the best way and possibly the only way of making the preferred embodiments is by employment of the principles of our present method.

Whereas the prior art discloses no method of making crystals and necessarily no method of making dense blocks of solid carbon dioxide from crystals except the application of great pressure, we have discovered that in practice, time and heat may be made of equal or greater importance and that the size of the crystals to be compressed is so important as to constitute a distinct part or branch of our generic invention.

One point in the preferred method is to apply a pressure great enough for a time long enough or in a mold warm enough to cause adequate melting of the solid carbon dioxide and flow thereof to the interior of the mass of crystals being compressed, under such conditions that it will there recrystallize, mostly filling, but in any event completely sealing all voids; the recrystallizing being not in separate or minute crystals and not merely as welds but as a substantial growth of the original crystals or fragments thereof. That this actually takes place, under conditions which we establish, is evident from the fact that when minute crystals are mixed with the large ones, the minute crystals disappear and samples are frequently produced in which breakage of large crystals and recrystallizing of smaller ones, has resulted in a very noticeable tendency to uniform sizes, mostly about $\frac{1}{16}$ inch to $\frac{1}{4}$ inch in length.

One discovery as to this part of our invention is that where the crystals are made to exceed a certain size, and particularly where they include a substantial percentage of crystals of very large size, the resistance to compacting of the crystals under pressure in the mold, is decreased. It would seem that the large crystals afford far fewer and probably larger areas of contact, so that they slip, break and compact easily. Because of the enormously decreased number of contact surfaces, total pressure effective on each of the individual points or surfaces is greatly increased, so that it becomes possible with commercially attainable pressures applied for a reasonably short time, in a mold not too warm, to partially liquefy the solid, fill up and seal the voids, recrystallize the liquid, partly as growth of the larger crystal fragments and partly as amorphous bonds, so as to effectively unify a mass of the crystals sufficient to make a tough solid block of commercial thickness yet of substantially uniform great density from surface to center.

If the crystals employed average large enough to ensure relatively few contacts, breakage under pressure will not multiply the contacts enough to decrease individual contact pressures below the limit required for the results above described.

Crystals of proper size for commercial practice of our methods may be made by properly regulated, slow freezing of the liquid, as by applying an external refrigerant to the liquid as in various prior patents such as Josephson 1,659,431; or, preferably, by methods utilizing the principles of self intensive cooling by exaporation.

One method is to discharge high pressure liquid carbon dioxide into a chamber in which is maintained a back pressure corresponding to the triple point of carbon dioxide, approximately 75 pounds absolute per square inch. Expansion of a certain amount of the liquid will reduce the chamber to triple point temperature and thereafter part of

the liquid will gasify, part will remain a liquid and part will freeze. In this situation, the crystals of carbon dioxide will form and grow until finally all of the liquid is either crystallized or gasified. If the chamber is first charged with liquid at a pressure above the triple point, say 75 pounds absolute, the source then cut off and the back pressure in the chamber regulated down to the triple point, the liquid may be boiled off as slowly as desired, giving the crystals any desired amount of time for formation, the rate of gas released and time of crystallization determining how large the average size of the crystals will be. Any suitable apparatus may be used for this purpose, as for instance that disclosed in the patent to Jones, No. 1,877,180, to which reference is hereby made for the details. For present purposes, the main features of the apparatus may be sufficiently understood from the accompanying drawing, in which

Fig. 1 is a diagram showing the essentials and following some of the details of the evaporator of said application:

Fig. 2 is a diagrammatic view of a compressor that may be used to form the block in accordance with the present method; and

Fig. 3 is a diagram showing a single chamber for both the evaporator and compressor.

In Fig. 1 there is an exterior casing 1 of strength sufficient to sustain not only a triple point pressure of approximately 75 pounds, but a considerably higher pressure, which may be employed as a charging pressure, say 150 pounds, plus a safety factor, or say, 500 pounds per square inch. In the top of this casing is a large manhole normally securely closed by a cover 2, which may be removed to insert a can 3, preferably suspended by the rim on ledges 4. This can is preferably gas-tight up to the level of outlets 5 which are provided for escape of gas. These outlets may be guarded by a screen 6, which is preferably carried by the manhole frame so that the can 3 may be lifted out and replaced without disturbing it. The liquid carbon dioxide is supplied through a pipe 7, which extends through the cover to discharge the liquid within the can 3. In the present case, we have diagrammatically indicated the discharge inlet as being an expansion nozzle 8, which may be an ordinary snow making nozzle, although this is not essential. The casing is provided with an outlet 9, controlled by valve 10, whereby any desired back pressure may be maintained within the casing 1 and thereby upon the inlet nozzle 8 and the surface of any liquid within the can 3. By adjusting the valve 10, the rate of escape of gas may be governed so that liquid within the can 3 will be boiled off and crystallized as slowly as may be desired, thereby insuring a desired average size for the crystals, crystals of relatively enormous size being easily attainable by sufficiently slow evaporation.

Almost any expansion or evaporator chamber could be used in this way, if strong enough to stand the pressure, provided with the necessary valves and arranged for removal of the crystals.

In Fig. 2, 11 is a mold in which the crystals may be compressed to form a block by means of a plunger 12, preferably operated by hydraulic cylinder 13 of known or desired type. The plunger applies the pressure through a follower 14 which may fit the mold loosely enough to permit escape of gas and air, at least until such time as the pressure becomes sufficient to form a more impervious seal of solid carbon dioxide. The bottom of the mold may be integral, but in the present case we have shown it as consisting of a follower pressure plate 15, like 14. When this form is used it permits the block to be compressed within the mold by inward pressure at both ends so that for a given compression the central portion of the block does not have to slide on the walls, thereby reducing wall friction. Where large crystals are used, however, this friction is greatly reduced and a mold with an integral bottom may be employed.

In Fig. 3, 21 is a chamber long enough so that it may be used first as a crystallizing chamber and then as a block-compressing chamber. As in Fig. 1, it may be used for snow making and then for triple point boiling, a liquid inlet and valve 22 and a gas outlet and back pressure valve 23 being provided; preferably also a pressure gauge 24; also a plunger 12*a* of a hydraulic press operating a piston head 14*a* which is held in the retracted position during crystal making. It is here indicated as fitting closely but not gas-tight, although in the present case it may well be gas-tight because the crystals formed in the chamber 21 do not contain any undesirable air and the pure gas in the voids of the crystals may be compressed, liquefied and frozen in situ. This form of apparatus, however, makes possible another feature of our method, which consists of a final step after the crystal forming has been completed which consists in completely releasing the pressure and then turning the valve 23 to connect with a vacuumizing conduit 23*a*. In this way the sublimating point of the crystals may be lowered, thereby reducing the internal temperature from, say, —110° F. down to —140° F. or —150° F. As explained below, such sub-cooling of crystals by vacuum accomplishes in a better way the sub-cooling that is accomplished by air.

The chamber 21 is hermetically sealed at the bottom by a closure 15*a*, which may be a breech block of any known or desired type capable of withstanding the enormous pressures to be applied through the compressing piston 14*a* and of being conveniently open for ejecting the finished block.

This apparatus will be recognized as analogous to that shown in Slate Patent 1,643,590 and, as in said patent, the liquid may be charged in at the bottom of the chamber and caused to follow the retreat of the piston until it uncovers the vent outlet 23.

As to the factor of warmth of the mold, it is to be noted that although the mold gets very cold, and may be insulated by collected frost or otherwise, it continues to absorb some heat from the atmosphere. Consequently, the length of time the material is compressed in the mold will be a factor of the amount of heat that will be imparted to it by the mold. This factor may be controlled in various ways and, in special cases, special heating means or insulation, permanent or adjustable may be employed.

In practice of our method, either with apparatus shown in Figs. 1 and 2 or that shown in Fig. 3, the crystals are likely to vary greatly in size according to the time they remain beneath the surface of the liquid under crystallizing conditions of temperature and pressure. Some of them may be wide, thin leaves, 2 inches or more in length and quite flexible, while others may be of smaller size, grading down to a small amount of what we call "popcorn snow". Conditions can be controlled to make the sizes and the proportions of the smaller sizes relatively to the larger sizes, well within the limits required

for the purposes herein set forth. In general, the smaller sizes should not be less than 100 mesh, that is, .01 inch. It will be realized that crystals as small as this are really of very great size as compared with ordinary snow jet crystals now commonly employed. Even so, the percentage of smaller crystals must be limited in favor of larger ones if best results are to be secured.

We find that when we press such coarse crystals with sufficient pressure, the time need not be objectionably long nor the mold objectionably warm, in order to produce truly solid, uniform, tough, stable block of over 90 pounds per cubic foot density, regardless of the distribution of the charge in the mold prior to pressing. The coarse crystals behave as though they flow in the mold, but fine crystals do not.

An important factor seems to be low initial teperature of the crystals in the interior of the mass. If they are exposed to the air or are vacuumized, the crystals sublimate and chill down to —120° to —150° F., according to the percentage of effective air or of vacuum, as the case may be. This is far below the normal freezing point of —110° F. and probably still more below the freezing point under pressure in the mold. If the crystals are sufficiently subcooled, in this or any other way at the time the great pressure is applied in the mold, minute fragments can easily melt, but the large ones embody large refrigerant values, sufficient to condense gas and freeze liquid whether sucked in or driven in through the passages that initially exist between the fragments or that may be produced interiorly by the pressure.

Any well-known method of pressing may be employed that will apply sufficient pressure, the preferred pressure, even for coarse crystals being over 1,000 pounds per square inch for blocks 10 inches deep. It will be understood, however, that these pressures are given for illustration only as a smaller pressure may be caused to produce a similar result if the time, amount of heating, sizes of crystals or thickness of the block are properly regulated to produce the desired liquefaction and recrystallization. The structure of the product is the best criterion of whether these have been properly coordinated.

To illustrate the desirable properties attained, it may be stated that products made in identical apparatus and under the same identical conditions as our product, but from finer crystals, are less uniform and generally lower, in density and very much more fragile, as judged by the breakage in dropping the various products or tumbling them under identical conditions. When ordinary snow is used, even the densest products show fracture surfaces that are snow white, very smooth, and follow directions of pressure applied in the making. There is none of the jaggedness and crystalline cleavage displayed by our product.

The importance of uniform density, toughness, and stable structure in storage cannot be overemphasized since these factors have limited the commercial practicability of the products of the prior art severely, and have made it imperative that solid carbon dioxide be used within a day or so after it has been produced to avoid structural changes in storage.

Owing to its non-porous and dense nature, our product is relatively stable and less subject to these changes.

It is important to note that blocks pressed from crystals will not have commercial structural strength and toughness unless the crystals are of sufficient length to enmesh and hold the mass together, and, in general, the crystals visible to the naked eye in the finished product should be over $\frac{1}{16}$ inch and mostly over $\frac{1}{4}$ inch in length, although with very large crystals in the product it is fair to suppose that others of them may be less than $\frac{1}{16}$ inch long without sacrificing all of the novel properties of our product, because they are scattered among the large crystals, embedded in and bonded by the amorphous solid. While the densities of these component elements of the block may be slightly different, it is evident that the distribution throughout the block is so uniform that for all practical purposes the densities and refrigerant values are substantially the same for usable pieces taken from any part of the block.

While our product as ordinarily made is translucent to a marked degree, it is possible to increase the translucency and under special conditions to get substantially transparent blocks. For instance, if the crystals are either exposed to air sufficiently or are evacuated in a closed machine, they will be cooled by evaporation to a temperature considerably below the normal freezing point of —110° F. and possibly as low as —140° F. If these supercooled crystals are brought in contact with pure carbon dioxide gas, they are capable of condensing some of it to liquid by absorbing sensible heat. Similarly they will freeze liquid formed by great pressure and by warmth of the walls of the mold. This condition exists when the block is pressed. If the initial temperature within the block is low enough, it will be sufficient to solidify all the residual liquid or gaseous carbon dioxide within the mass and there will be no gas bubbles; if, as in Fig. 3, air is excluded and a high enough vacuum applied, the result will be an absolutely clear block without any air bubbles.

Referring again to Figs. 1 and 3, there are several composite methods of producing and growing crystals. For instance, the liquid may be discharged in the chamber through the ordinary snow jet, the gas outlet being free so as to maintain a pressure near atmospheric, say 10 pounds above atmosphere. After a desired amount of ordinary carbon dioxide snow has thus been made, the pressure may be raised to the triple point of 75 pounds, or preferably, above it, say 85 pounds or 100 pounds, so that the carbon dioxide will be discharged into the chamber as a liquid to wet down and preferably submerge the snow. Thereupon the supply may be cut off and the liquid boiled off as slowly as may be desired to permit any desired amount of recrystallizing or growing of the snow crystals during evaporation of the liquid.

While theories as to the details of obscure internal phenomena of recrystallization are not essential to practice of our invention, such of them as have been offered will be found helpful working hypotheses, regardless of their accuracy, and in this connection it seems desirable to note a theory as to sizes of crystals. Where ordinary snow crystals are used, they are all so minute that no matter how much they may be sub-cooled, their individual heat absorbing capacities remain minute and their individual heat absorbing areas per unit volume are corelatively great. Moreover the pores or passages between them become exceedingly minute under pressure. Consequently, when gas and liquid formed by the heat of the mold is forced or sucked into these capillary passages all the crystals are available to absorb all of

the surplus heat. The result is that all of the heat of the penetrating gas or liquid is quickly used up in the outermost layers of the block, and the entrances of the inward passages are quickly choked by refreezing of the inflowing gas and liquid, consequently the bonding of the crystals in the interior of the block is almost entirely by such minutely localized heating and point welding as can be effected by plunger pressure. On the other hand, the larger the sizes of the crystals at the time they are compacting by slipping and breaking under pressure, the greater the heat absorbing capacity and the less the heat absorbing area of the individual crystals and, although the number of inwardly extending passages is much fewer, their average cross-section for easy flow of gas and liquid from the mold walls to the interior is correspondingly multiplied. Consequently, if the crystals are of large average size, the smaller ones can be completely remelted and the voids filled without exhausting the heat absorbing capacity (refreezing ability) of the larger crystals and fragments. Such a theory would tend to explain why lagre sizes, and also why mixed sizes, are desirable and will assist the maker in selecting average sizes suitable for the thickness of the desired block and regulating all of the other factors above described as contributing to best practice of our invention.

As an illustration, a quantity of crystals of mixed sizes, say, .01'' to 2.'', sufficient for a 10'' cube, subjected to pressure of 1800 pounds per square inch, more or less, in a fairly warm mold, produced a tough block having practically uniform density of over 96 pounds per cubic foot, together with other above described novel details of appearance, fracture surfaces, frosting, low evaporation rate, etc.

We claim:

1. In a method of making blocks by compressing a mass of solid carbon dioxide crystals, the step which includes freezing liquid carbon dioxide to produce crystals, the freeezing being at a regulated rate to effect the production of a product characterized by the major portion thereof consisting of crystals over $\frac{1}{8}$ inch in length; then breaking the crystals and compressing them to form the block.

2. The method of making blocks by compressing carbon dioxide crystals, which includes subcooling a quantity of pre-formed crystals many of which are of relatively great size and applying pressure sufficient to produce substantial recrystallization and solidification in regions distributed throughout the block.

3. The method of making blocks by compressing carbon dioxide crystals, which includes sub-cooling a quantity of the crystals to a temperature substantially below their normal freezing point and applying pressure sufficient to produce substantial recrystallization and solidification in regions distributed through the block.

4. The method of making blocks by compressing carbon dioxide crystals, which includes subcooling a quantity of the crystals to a temperature substantially below their normal freezing point and applying great pressure to said mass in a mold substantially above said temperature for a time sufficient to produce substantial recrystallization and solidification in regions distributed throughout the block.

5. The method of making blocks by compressing pre-formed carbon dioxide crystals, which includes vacuumizing a mixture of crystals of greatly different sizes to cause accelerated evaporation sufficient to sub-cool them to a desired extent and applying heat and pressure in amounts and for times corelated to the average size of the sub-cooled crystals so as to produce substantial recrystallization and solidification in regions distributed throughout the block.

6. The method of making blocks by compressing pre-formed carbon dioxide crystals, which includes assembling a mixture of crystals of greatly different sizes sub-cooled to a desired extent and applying heat and pressure in amounts and for times corelated to the average size of the crystals so as to melt the smaller ones and to produce substantial recrystallization and solidification in regions dnstributed throughout the block.

7. The method of making solid carbon dioxide, which includes slowly evaporating liquid from and at the triple point, breaking the mass thus formed, and pressing it in a mold to a density of 95 pounds or more per cubic foot.

8. In combination, a source of liquid carbon dioxide under high pressure and means for controlling discharge thereof, a crystallizing chamber therefor capable of safely withstanding pressures substantially above the triple point of the liquid, a gas outlet and valve means for adjusting the same to impose desired back pressures near the triple point in said crystallizing chamber, a removable closure through which the crystallized products may be withdrawn, means for vacuumizing said chamber and a piston for subjecting the crystals to high pressure in said chamber.

9. In combination, a source of liquid carbon dioxide under high pressure and means for controlling discharge thereof, a crystallizing chamber therefor capable of safely withstanding pressures substantially above the triple point of the liquid, a gas outlet and valve means for adjusting the same to impose desired back pressures near the triple point in said crystallizing chamber, a removable closure through which the crystallized products may be withdrawn and a piston for ejecting the product when the closure is removed.

10. In combination, a source of liquid carbon dioxide under high pressure and means for controlling discharge thereof, a crystallizing chamber therefor capable of safely withstanding pressures substantially above the triple point of the liquid, a gas outlet and valve means for adjusting the same to impose desired back pressures near the triple point in said crystallizing chamber, a removable closure through which the crystallized products may be withdrawn and means for vacuumizing said chamber.

11. In combination, a source of liquid carbon dioxide under high pressure and means for controlling discharge thereof, a crystallizing chamber therefor capable of safely withstanding pressures substantially above the triple point of the liquid, a gas outlet and valve means for adjusting the same to impose desired back pressures near the triple point in said crystallizing chamber and a removable closure through which the crystallized products may be withdrawn and a piston for subjecting the crystals to high pressure in said chamber.

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