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(54) Title: SWEETNESS ENHANCED SUGARS AND SUGAR LIKE PRODUCTS

(57) Abstract: This invention comprises a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion, with optional ingredients for organoleptic or health benefits, and a method of making the same comprising steps of slow stirring, with or without seeding, accompanied with heating of a concentrated solution having dissolved solids concentration suitable for co-crystallization under above conditions comprising sucrose, a high intensity sweetener and optionally one or more of other ingredients. The sweetener of this invention has sucrose equivalent sweetness that is an integer or any other pre-defined sucrose equivalent sweetness, including SES precisely of 2 or 4. The high intensity sweetener ingredient in the sweetener may be sucralose or any other high intensity sweetener.

TITLE

SWEETNESS ENHANCED SUGARS AND SUGAR LIKE PRODUCTS.

FIELD OF INVENTION

The invention relates in general to sweetener and method of its preparation. It pertains particularly to preparation of a sweetener by co-crystallization of sucrose with one or more of a high intensity sweetener. Still more particularly the invention includes co-crystallization of sucrose and Sucralose.

BACKGROUND OF THE INVENTION

Sweetness enhanced sugar or sugar like products have been made by various ways.

Riegal's handbook of Industrial Chemistry (10th edition), 2003, Kluver Academic Plenum Publishers, New York discloses on pages 345 and 346 that compound products are made by manipulating crystal size and shape and incorporating another ingredient in a process called co-crystallization. A very hot (120° C) supersaturated sucrose solution is allowed to cool with agitation in the presence of a second ingredient, resulting in a dry, free flowing, agglomerated porous granule shown in figure in that book. Typical products include granulated brown sugar, powdered brown sugar, molasses granules, honey granules and some fondants. Flavors such as apple or peanut butter, may be co-crystallized with sugar. The process is said to protect flavors from volatiles and from oxidation.

Imperial Sugar (http://www.iscnewsroom.com/2010/03/15/co-crystallization-seeks-the-perfect-match/) disclosed that co-crystallization process was originally discovered in the early 1960's, when food researchers found that by heating a sucrose syrup to a certain temperature under controlled conditions, the sucrose

crystals develop a sponge like appearance, capable of bonding with other ingredients, such as molasses. This process was considered useful because it opens up a wide scope to mix ingredients that customers would prefer to give a free-flowing form of sugar with any grain size desired. Accordingly, sugars co-crystallized with honey, maple, molasses, cinnamon have been made. Brownies, cookies and pancakes with the stevia-sugar blend and with sugar have been made which tasted like the straight sugar and the stevia-sugar blend.

US 6,214,402 discloses a process for preparing a sugar co-crystallized sweetener composition comprising the steps heating sugar at approximately 120 degrees with water with agitation to form a mixture; seeding the said mixture with a premix comprised of N-[N-(3,3-dimethylbutyl)-L-.alpha.-aspartyl]-L-phenylalanine 1-methyl ester or salt thereof and sugar; removing said mixture from the heat; and allowing the mixture to cool with vigorous agitation.

US 6,875,460 discloses a co-crystallized polyol and hydrogenated maltodextrin sweetener composition comprising at least one polyol and a hydrogenated maltodextrin, wherein the composition is formed by co-crystallization of the at least one polyol and the hydrogenated maltodextrin. The co-crystallization is achieved by a process comprising: (a) co-melting the at least one polyol and the hydrogenated maltodextrin to form a melt; and (b) cooling the melt; wherein shear is applied to the melt during cooling.

EP0528604 discloses melt co-crystallized sobitol and xylitol.

US 20080292775 discloses a sweetener delivery system for sweetener compositions and methods of making them, the ingredients comprising at least one natural high-potency sweetener, wherein the delivery system is selected from the group consisting of a sugar or a polyol co-crystallized sweetener composition, an

agglomerated sweetener composition, a co-dried sweetener composition, and a cyclodextrin complex of a sweetener composition and methods comprising co-crystallizing the sweetener composition with a sugar or a polyol, agglomerating the sweetener composition, co-drying the sweetener composition, or preparing a cyclodextrin complex with the sweetener composition by (a) mixing a sugar or a polyol with water to form a mixture; heating the mixture to a temperature of at least about 120°C; seeding the mixture with a premix comprising the sweetener composition and the sugar or the polyol; and cooling the mixture, or (b) comprising the steps of providing a premix solution comprising the sweetener composition and a binding agent; heating the premix solution to a temperature effective to mix the premix solution; fluidizing a carrier; and applying the premix solution onto the fluidized carrier to form an agglomerate comprising the high-potency sweetener composition and the carrier, or (c) comprising the steps of compacting the sweetener composition to form compacts; and breaking up the compacts to form granules, or (d) comprising the steps of combining the sweetener composition, a plasticizer, and optionally a binder to form a wet mass; extruding the wet mass to form extrudates; and drying the extrudates to obtain extrudates of the sweetener composition, or (e) comprises associating the sweetener composition with alpha-, beta-, or gamma-cyclodextrin.

WO/1998/053706 disclosed a method of preparing a food additive, said method comprising (i) mixing crystalline carrier particles with an additive compound; (ii) applying to the thus produced mixture a solution of a crystalline solid under conditions such that the said carrier particles are partially dissolved; and (iii) allowing the mixture to co-crystallize and agglomerate so as to form particles of increased average size as compared to the said carrier particles.

US 20100034945 disclosed a process for co-crystallizing sugar with a natural sweetener product and eventually, an aggregate to give it special characteristics, characterized in that it comprises the steps of: Adding an antifoaming to the liquor or syrup (batch foot) in order to avoid the foam provoked by the natural sweetener in a percentage of about 0.006% to about 10%; mixing with mechanical stirring the sucrose solution (light liquor or concentrated solution) with 0.1% to 50% w/w of natural sweetener with respect to the amount of produced mass and, eventually, adding an aggregate in order to reach any special characteristic; concentrating the above mixture up to saturation point of sucrose, in a vacuum pan at a working pressure of about 60 KPa to about 75 KPa and a temperature between about 60°C. to about 75°C; adding a powdered sugar with isopropyl alcohol suspension, which is then used as seed for crystallizing the previous solution in the saturation zone; the amount is determined by different variables such as the weight of the crystal in the mass, the initial size of the seed and the desired final size; which relate in the following manner: suspension weight=crystal weight in mass*((initial size/final size)^3); feeding the light liquor or syrup in a constant and slow manner, maintaining the crystallized solution in the "meta-stable zone", in order to avoid the spontaneous generation of new crystals or dilution of those previously formed; this step is carried out until the equipment reaches 100% of its level, wherein the co-crystallized sucrose and natural sweetener crystal has reached a size of about 0.5 to about 0.7 mm average aperture; evaporating the remaining water until reaching about 88° and about 91° Bx; unloading the mass in order to carry out the separation of the crystals from the sugar juice or syrup through centrifuging at a velocity between about 1100 and about 1300 rpm; re-circulating the obtained sugar juice or syrup at the end of the co-crystallization process (centrifuging) as necessary while it does not lose the color established by the product specifications which correspond to a value between

about 1300 and about 1600 UI; recovering between about 1 and about 90% of the natural sweetener with a solvent in which said natural sweetener is soluble; bleaching the remaining syrup and natural sweetener mixture, with a bleaching product for obtaining a certain amount of a low calorie liquid product.

US 20100227034 disclosed a low-calorie sweetener composition, comprising a low intensity sweetener; and a high intensity sweetener which is applied onto the low intensity sweetener so as to form the low-calorie sweetener composition by (I) a process comprising: a) adjusting a moisture content of sugar granules to a level of about 0.5% to about 1.5%;b) distributing the sugar granules on a vibrating surface to form a layer with thickness of about 10 to about 100 mm on the vibrating surface; c) dissolving the high intensity sweetener in an aqueous alcohol mixture with an alcohol content of about 0.1% to about 99.9% (v/v) to obtain a solution with about 10% to about 70% (w/w) solids to make a high intensity sweetener solution; d) dispersing the high intensity sweetener solution onto the sugar granules while maintaining an intensity of vibration at about 1 to about 2000 vpm; e) drying the granules to form the low-calorie sweetener composition; Or (II) by a process comprising the steps of: a) making a sweetener solution by combining the low intensity sweetener, the high intensity sweetener and water; b) passing the sweetener solution through multiple-effect evaporators to make an evaporated sweetener solution; c) applying a vacuum to the evaporated sweetener solution to make a super-saturated solution; d) seeding the super-saturated solution with an additional amount of the low intensity sweetener to initiate crystal formation; e) allowing crystal formation to continue to complete crystallization in the solution; and f) separating the crystals from the solution to result in the low-calorie sweetener.

US20080014331 disclosed a method for increasing the sweetness of regular sugar made during the refining process of sugar beets and sugar cane, the method

comprising: a) a first step of forming a mixture of raw juice extracted from sugar cane or sugar beets with a milk of lime and a carbon dioxide gas in carbonation tanks so that the carbon dioxide bubbles through the mixture forming calcium carbonate and non-sugar particles in the mixture attach themselves to the calcium carbonate and settle to the bottom of the tanks; b) a second step of filtering the mixture, leaving a golden light brown clarified thin juice, c) a third step of boiling the juice under vacuum until a sufficient quantity of water is evaporated from the juice to form a concentrated solution having a consistency of pancake concentrated solution; d) a fourth step of filtering the concentrated solution in an after filter to remove all non-sugar materials to form a saturated sugar liquor; e) a fifth step of mixing the saturated sugar liquor with at least one high intensity sweetener in a large mixer; f) a sixth step of transferring the mixture the saturated sugar juice and the at least one high intensity sweetener to boiling pans and boiling the mixture of saturated sugar juice and at least one high intensity sweetener under vacuum until crystals begin to form a super sweet massecuite comprising a mixture of super sweet sugar crystals and super sweet concentrated solution; g) a seventh step of transferring the super sweet massecuite to centrifuges for separation by spinning rapidly in a perforated cylindrical basket to form a super sweet molasses concentrated solution thrown off through perforations in the perforated cylindrical basket with the super sweet sugar crystals remaining in the perforated cylindrical basket; h) an eighth step of processing further the super sweet molasses concentrated solution (if it is not used further commercially) that remains after the sugar crystals are initially separated by removing more super sweet sugar crystals from the remaining molasses to produce a super sweet sugar concentrated solution.

US20070026121 disclosed a solid sweetening crystal comprising a nutritive sweetener in intimate contact with a high intensity sweetener, wherein the crystal

comprises a matrix and a surface and contains an SSD of from about 0.01 g SES per g to about 300 g SES/g and an energy content of less than about 1 kcal/gram of SES. Illustrated method to prepare such crystal includes dissolving erythritol in water, maintaining the same at a slow boil and stirring it well until the materials dissolved. The solution was then removed from the heat and allowed to cool. When the beaker was cooled to the point at which it could be touched, a few additional erythritol crystals are added and the solution was allowed to sit quiescently. In about 1 hour, a significant crop of crystals emerged. The solution was then allowed to sit for another 3 hours at which time the crystals were separated from the remaining mother liquor, placed on a plate, broken by hand into smaller units to remove some of the agglomeration, and then allowed to air dry at room temperature overnight. Another example illustrated was for trehalose as the nutritive sweetener. It was seen in the laboratory of inventions of this patent specification that the method of this invention US20070026121 did not give co-crystallization when sucrose was used as nutritive sweetener. In large scale production adopting the patent process, even with erythritol is difficult like segregating by hand and breaking big crystals by hand etc.

In view of the prior art processes that required very carefully controlled complicated crystallization steps and several unit processes and yet significant quantity of costly high intensity sweetener was lost in the process; and with the fact that the processes were not able to yield low calorie sweeteners of exact sucrose sweetness equivalence, it was a great disadvantage and an improved process was required wherein the process would be simpler, more efficient and capable of yielding sweetener of precisely desired, precisely defined sweetness; such as exactly two times, four times etc.

SUMMARY OF THE INVENTION

This invention comprises a method of making a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion, comprising steps of: (a) obtaining a concentrated solution of sucrose and a high intensity sweetener with or without a stabilizer and with or without other optional ingredients accompanied by heating to a temperature, the resulting concentrated solution containing mixture of sucrose and the high intensity sweetener not containing impurities in a quantity that would result into formation of molasses or molasses like by-products after the step of co-crystallization during further steps of the process, the final concentration of the solution of sucrose, the high intensity sweetener and other optional ingredients being at least enough for getting co-crystallization of the dissolved sucrose with the high intensity sweetener by stirring, of the mixture of sucrose, the high intensity sweetener, with or without the other additional ingredients, for a period of time accompanied with heating to a temperature where caramelization will not occur, until co-crystallization is complete, the stirring being done, with or without seeding, (b) drying the moist co-crystallized mass with or without the separation of mother liquor at a temperature that promotes as much less residual moisture content as possible in the dried sweetener composition without caramelization to get a solid mass comprising free flowing crystals or comprising lumps of dried crystals, optionally, breaking lumps if required, and sizing the dry crystalline solids to get a sweetener composition that passes through desired mesh size.

In one embodiment of this invention, the said concentrated solution of sucrose and a high intensity sweetener, with or without other optional ingredients is obtained by: (i) adding one or more of a high intensity sweetener, with or without other optional ingredients, to a concentrated solution of sucrose prior to co-crystallization, or (ii)

adding sucrose with or without other optional ingredients to concentrated solution of a high intensity sweetener prior to co-crystallization, or (iii) adding one or more of a high intensity sweetener, with or without additional optional ingredients, to a concentrated solution made from process stream of regular sugar manufacturing process after removal of molasses from the crystallized sucrose, or (iv) adding sucrose to a concentrated solution of process stream, containing a high intensity sweetener in purified state produced in a process of manufacture of the high intensity sweetener, the said concentrated solution being free from organic solvent/s to such an extent that residual solvent will not interfere in the co-crystallization process.

In a further embodiment of this invention, the said obtained concentrated solution of sucrose, high intensity sweetener with or without other ingredients is obtained at a temperature and heated, avoiding caramelization, to a higher temperature, more particularly at a temperature of 80°C to 45°C, or at 50 to 70°C, or at 60°C,

The final concentration of the solution of sucrose and the high intensity sweetener acchieved before co-crystallization step could be around 250% dissolved solids or more,

To induce co-crystallization, the stirring of mixture of sucrose and the high intensity sweetener with or without additional optional ingredients may be at any rate, but preferred rate is 20-40 revolutions per minute, most preferably at 30 revolutions per minute; at a temperature of 80-40°C, most preferably at 60°C and for 3 to 32 hours, preferably to 16-32 hours.

The temperature of drying of co-crystallized moist mass may be between 40-45°C and the said residual moisture of the dried mass of co-crystal mass may be 0.1 – 0.4 %.

High intentisty sweetener may be selected for this invention from the group sucralose, Glycyrrihizin, Thaumatin, Monellin, Stevioside extract, Rebaudiside-A extract, Lo Han Guo Morgrosides extract, Brazzein, Curculin, pentadin, Mabinlin, Acesulfame K, Neotame, Talin, Citrose, Alitame, Cyclamate, Saccharin and Aspartame. For illustration in this invention, sucralose has been used, although examples from other high intensity sweeteners are also provided to show that sucralose may be replaced by any of other high intensity sweetener unless the high intensity sweetener is unstable at the process conditions of this invention.

In this invention, the said additional ingredients comprise one or more selected from a group consisting of an amino acid or derivative of an amino acid, a vitamin, a mineral, a flavor, an enhancer, or a prebiotic, or a probiotic, a Pharmaceutically active ingredient, an anti-oxidant, an energy booster, a derivative of a fat or an oil, a color and other natural products.

In one embodiment, a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion having pre-determined target of "Sucrose Equivalent Sweetness" abbreviated as SES, may be made with precise predetermined sucrose equivalent sweetness wherein mother liquor, that contains un co-crystallized components, is separated from the co-crystallized mass before drying of the moist co-crystallized mass by (a) performing a certain number of pilot experiments required statistically to find out what quantity of the high intensity sweetener is lost in mother liquor wherein a quantity of high intensity sweetener expected to give targeted sweetness is added in step a. of claim 1 to determine overages that need to be added to compensate for this loss, (b) dissolving the high intensity sweetener in step a. in a quantity with proper overages decided above that

shall give the desired sweetness in the co-crystallized dried mass, (c) preparing more than two batches as required within the statistical requirement for statistical uniformity, evaluating for actual sweetness or the content of the high intensity sweetener in the co-crystallized product, (d) combining batches with lower and higher than targeted sweetness in such a way that desired sweetness is achieved in the uniformly mixed product. The target of desired SES may be 1.15 times to 100 times, more particularly 2 times to ten times, still more particularly 2 times to six times, still more particularly 2 times to six times, still more particularly 2 times to four times, still more particularly 2 times or 4 times.

In another embodiment, a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion having pre-determined target of "Sucrose Equivalent Sweetness" abbreviated as SES, is be made when mother liquor is not separated from the drying of the co-crystallized mass in step c. comprising steps of (a) dissolving the high intensity sweetener in step a. of claim 1 in a quantity that is calculated to exactly give the desired sweetness in the co-crystallized mass, and (b) drying the co-crystallized mass without separating the mother liquor.

In one embodiment, this invention comprises a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, containing at least sucrose and at least one a high intensity sweetener, free flowing, non-dusty and with uniform sweetness in each 6 gram portion having defined "Sucrose Equivalent Sweetness", abbreviated as SES, that is precisely an integer or a whole number without a fraction or any other pre-defined SES; comprising SES of 2 times to 100 times, more particularly 2 times to 10 times, still more particularly 2 times to 6 times, still more particularly 2 times to 4 times, still more particularly 2 times or 4 times.

In a particular embodiment of this invention, the high intensity sweetener in the sweetness enhanced sweetener and used in the method of making the same is sucralose.

DETAILED DESCRIPTION OF THE INVENTON

For the purpose of this specification, "Co-crystal" is defined as a crystal that incorporates at least two chemical entities forming structural part of one and the same individual crystal, and not as agglomerates of two or more dfferent crystal species having a separate crystalline identity from each other and wherein one crystalline ingredient is dispersed over surface of aggregates of another crystalline aggregate. Thus, Co-crystal of this invention shall have XRD and DSC that would indicate that the co-crystals comprise at least a single crystalline entity/species comprising of at least two chemical entities and is a not a simple mixture of two separate crystal species of two chemical entities. It is, however, possible that the single co-crystal entity may entrap one or more other ingredients within the co-crystal or in intimate association with the co-crystalline mass. A process that would lead to formation of these co-crystals is termed as co-crystallization.

For purpose of this specification, "High Intensity Sweetener" is defined as the one that has a sucrose equivalent sweetness of at least 10 or more, Thus, for the sake of explanation, fructose and polyols are not considered as high intensity sweeteners.

One embodiment of this invention comprises a sweetness enhanced sugar or sugar like products as a co-crystallized product and a process of making thereof comprising making a highly concentrated solution made from crystalline sugar prepared in water at moderate temperature of about 40-65° C, adding to it and dissolving a high intensity sweetener such that the result is a supersaturated

concentrated solution, and allowing the same to co-crystallize., optionally with other ingredients too that may have some benefit to the consumers. An alternative embodiment of this invention comprises wherein the sweetness enhanced sugar or sugar like products as a co-crystallized product is made by a process comprising making a highly concentrated solution made from purified sucralose or a high intensity sweetener prepared in water at moderate temperature of about 40-65° C, adding to it and dissolving sucrose such that the result is a supersaturated concentrated solution and allowing the same to co-crystallize., optionally with other ingredients too that may have some benefit to the consumers

In one embodiment of this invention the supersaturated solution of sucrose is obtained by making a highly concentrated solution of sugar prepared in polar alcohol containing some quantity of water at elevated temperature, adding to it and dissolving a high intensity sweetener, the quantity of water being such that a highly concentrated solution of the sugar in water will be obtained when the added polar alcohol is distilled off.

In view of the prior art, whether the process starts from dilute solution of sucrose or starts with dissolving crystalline sucrose, a step of concentrating it under reduced pressure was an integral feature of the prior art processes, it was surprising that co-crystallization could be achieved without resorting to concentration under reduced pressure and without seeding. It was still further surprising that it was not necessary that the solution should reach saturation to achieve co-crystallizaiton by stirring at moderately high temperature; it resulted into co-crystallization, for example in Example 51, even if the if the concentration of dissolved solids was about 25 to142 % and the concentrated solution was kept continuously stirring; the time required for co-crystallization depended on temperature at which stirring was done and the concentration of dissolved solutes, higher the temperature or higher the

concentration of solutes, lesser was the time required. Thus, when the concentrated solution was stirred at about 60°C and concentration of the solutes was 25%, the co-crystallization occurred in 13 hour, whereas when concentration of solute was raised to 142%, the co-crystallization occurred in 3 hours. At the same time it was seen that there was no co-crystallization when (a) stirring was done at room temperature of 29°C, (b) when no stirring was done at 60°C, or (c) when the solution was kept standing at 60°C and seeding was done. Thus, stirring at temperature elevated relative to room temperature of 29°C was an essential condition for co-crystallization to occur within a reasonable time. The concentration of dissolved solids at which the co-crystallization will be achieved in this way will depend upon the temperature of the crystallizing mass to which it is heated and the concentration of the dissolved solids. In Example no. 51, the co-crystallizing mass was heated to maintain a temperature of 60°C, speed of stirring was 50 to 60 revolutions per minute and the co-crystallizaiton occurred in about 3 hours. A temperature of 60°C and dissolved solids content of 142% or more was selected as convenient for further experiments. By the method of the invention, the mother liquor that remains without crystallization is so small in quantity relative to the total mass of the co-crystallized out composition, that it needs to be removed only if shiny crystals are required, otherwise the whole moist mass could be dried with mother liquor to get a sweetener product that has sweetness enhanced than cane sugar, is non-dusty, having uniform sweetness in a 6 gram portion, is crystalline and has appearance of cane sugar. The instant invention is one of the simplest processes of making co-crystals that look like cane sugar, have enhanced sweetness as compared to cane sugar, is non-dusty, having uniform sweetness in a 6 gram portion and where no high-intensity sweetener is wasted and process steps are less.and the product is not amorphous nor is it a simple mixture of sucrose and high

intensity sweetener, but comprises co-crystals of sucrose and high intensity sweetener, as evident by XRD data. With less process steps, minimal equipment, low energy input, the overall process is more convenient and techno-economically more efficient. In one illustrative embodiment, about 333 % of sucrose solution was made in water at a moderate temperature. In illustrative experiment, temperature of dissolution was 30 - 55°C and Sucralose added was about 78% or less of the sucrose dissolved or 92% of Sucralose content in the total solid input. Optionally the solution is filtered. The filtrate was stirred slowly and continuously at temperature 45-70°C for 3-32 hrs until complete crystallization. The product was centrifuged, sediment collected and crystals were dried – with or without vacuum at 40- 45°C until moisture content reached to less than 0.2%. Lumps of dried crystals in the product were broken and the product sieved. Particle size achieved was such that about 54.1 % was retained on 50 mesh. The crystals passing through different mesh sizes are also prepared.

Throughout the specification, "sugar" and "sucrose" are the words used interchangeably to designate cane sugar / sucrose.

This process is illustrated in most examples by use of sucralose as high intensity sweetener, However, Sucralose may be replaced by many other high intensity sweeteners, natural as well as artificial / chemical derivatives, that includes, without limitation, Glycyrrihizin, Thaumatin, Monellin, Stevioside extract, Rebaudiside-A extract, Lo Han Guo Morgrosides extract, Brazzein, Curculin, pentadin, Mabinlin, Acesulfame K, Sucralose, Neotame, Talin, Citrose, Alitame, Cyclamate, Saccharin and Aspartame.

It is an embodiment of this invention that the process of the invention gives a very good control on quantity of sucralose that gets integrated uniformly throughout the

mass of the product so that the process makes it possible to make co-crystallized sweeteners over entire range of table top sweeteners and sweetened food product with entire range of permitted "label claims" and also sucrose equivalent sweetness i.e. specific defined or precise pre-defined multiples of sweetness of sucrose of a defined bulk density, such as one spoonful (6 g) of sucrose. Thus, as an illustration, with reference to Regulation (EC) No. 1924/2006 of the European Parliament and of the Council of 20th December 2006 on nutrition and health claims (label claims) made on foods, following co-crystal compositions are possible with sucrose and Sucralose as two ingredients: (a) "Energy free", containing 0.4 kcal or less per portion equivalent to 6 g sucrose equivalent sweetness (b) "Low Energy", containing the sweetener containing 4 kcal or less per portion equivalent to 6 g sucrose equivalent sweetness (c) "Energy Reduced" containing at least 30% reduction in energy on a portion that gives sweetness equivalent to 6 g of sucrose. In addition, it can give sweeteners with a defined portion of it with exact multiple of sweetness delivered by 6 g of sucrose such as a double sweetness will give about 50% reduction in calorie intake for same sweetness, a four times sweetness will give about 75% reduction in calorie intake for same sweetness, and so on.

In an aspect of this invention, co-crystals are produced in batches that are produced with targeted multiple of sweetness i.e. targeted sucrose equivalent sweetness, and the product has definitive and uniform sweetness in each 6 gram portion. The HPLC analysis for the content of high intensity sweetener i.e. sucralose in ordinary mixtures of sucrose and sucralose, and in sucrose co-crystallized with sucralose is illustrated by the data shown in Table 1 in Example 1(b). The sweetness taste profile was tested by the panel body formed in-house by the method described in Example 1(b).

In one embodiment, actual sweetness achieved for several batches is determined and two or more batches are mixed in such a proportion that practically achievable exact multiple of sweetness is achieved. Thus, in a further aspect, the invention comprises a process of preparation of co-crystals of sugar or sugar like product with at least one high intensity sweetener wherein several batches are targeted to produce desired defined multiple of sweetness in the defined portion of the co-crystalline sweetener, determining sweetness of each batch and mixing the batches such that resulting composition shall have statistically the desired sucrose equivalent sweetness. Thus, if a co-crystallized sugar like product is targeted that shall have sweetness of two spoonful of sugar for one spoonful of the enhanced sweetness sweetener product of this invention, targeted concentration of sucralose in the composition is 0.18%. This is achieved by making several batches of sucrose co-crystallized with sucralose by adding a calculated excess of sucralose that may be lost in mother liquor and carrying out co-crystallization and getting several batches in which the sucralose content is around 0.18%, some batches having little excess, others little deficient than 0.18%. From these batches, selection can be made of batches in such quantities that their mix will give concentration of sucralose very close to 0.18% upon uniform mixing and one spoonful of the composite composition gives sweetness of two spoonful of sucrose.

It is a further surprising finding and that also is a distinguishing feature of this invention over all prior art methods and an embodiment of this invention that the entire moist co-crystallized mass could be subjected to drying without removal of the mother liquor to get a low calorie sweetener composition that is dry, free flowing, non-dusty, did not segregate in handling, had uniform sweetness throughout the mass and had sucrose like crystalline cane sugar like in appearance, thereby resulting into 100% conversion of sucrose, the high intensity sweetener and, if

added, the other ingredients too into the low calorie sweetener. Thus the method of this invention is very simple, involving less number of unit processes and is highly efficient over prior art processes. This method has, also for the first time given a method to get means to produce sweetness enhanced low calorie sweeteners of precise per-defined sucrose equivalent sweetness. Thus, if a crystalline sweetener cane sugar like in appearance is desired to be produced that has exactly two time the sweetness of same bulk density sucrose, the method of this invention of direct drying without separation of mother liquor makes it possible to get a co-crystallized product of sucrose and sucralose that shall have exactly 0.18% sucralose incorporated in the composition, most of which is integrated into co-crystals and a minor proportion of which is unincorporated in co-crystals but are uniformly distributed so as to give uniform sweetness in any spoonful portion (6 gram) taken out from the same.

Co-crystallization of sugars or sugar like products and high intensity sweetener may also be made in presence of additional ingredients, which provide additional health benefits along with the sweetness property, as illustrated by examples; the said additional ingredients may individually or in combination be one or more of an amino acid or derivative of an amino acid, a vitamin, a mineral, a flavor or a flavor enhancer, or a prebiotic, or a probiotic, a Pharmaceutically active ingredient, an anti-oxidant, an energy booster, a fat or an oil, a color and any other natural product that would bring a benefit to consumer. The benefit may be a health benefit or an organoleptic benefit. The additional ingredients listed above may get uniformly distributed in the product.

An amino acids may include, without limitation, one or more of a, without limitation, Taurine, L-Arginine, L-Ornithine, L-Lysine, L-Carnitine, L-Methionine, L-Phenylalanine, L-Tyrosine, L-Cysteine, L-Glycine and S-Adenosyl methionine.

A Vitamin may include, without limitation, one or more of a Vitamin-A, Vitamin-B1, Vitamin-B2, Vitamin-B3, Vitamin-B5, Vitamin-B6, Vitamin-B12, Vitamin-B complex, Beta-carotine, Vitamin-C, Vitamin-D, Vitamiin-D1, Vitamiin-D2, Vitamin-E, Biotine, Choline, Folic acid,.

A mineral may include, without limitation, one or more of Selenium, Zinc, Boron, Calcium, Chromium, Iron, Magnesium, Potassium or Chromium picolinate.

A flavor or flavor enhancer may include, without limitation, one or more of a Coca powder, Coffee, Vanillin, Pista, Strawbeery, Mango, Orange, Chocolate, Dry Friut, Dry vegetables, Ice-cream, Yogurt, Wheet flour, Multigrain flour, Pepermint, Ginger, Apple, Citrius, Grape, Cherry, Ginseng, Peach, Wild berry, Tropical, -pome granate and Blue berry.

A prebiotic includes, without limitation, one or more of an Inulin and other fructooligosachhrides, Xylo-oligosaccharides, Gentio-oligoscaccharides, Galacto-oligosaccharides, Nigero-oligosaccharides, Malto-oligosaccharides, Soybean oligosaccharides.

A probiotic includes, without limitation, one or more of a Yeast, *Bacillus*, *Lactobacillus*, *Bifidobacterium bifidum*, *Bifidobacterium infantis*, *Bifidobacterium longum, Enterococcus faecium* and *Streptococcus thermophilus*.

A pharmaceutically effective ingredient includes, without limitation, one or more of a Bromelain, Chitosan, Salicin, Inosin, Myoglobin, Glucomannan, Guaranine, Yerbamate, Pectin, Pancreatin, Pantothenic, Spirulina, ATP, Conjugated linoleic acid, Hydroxy citric acid, Phaseolamin, 5-HTP, Adenosine receptor, caffeine, Theobromine, Theophylline, SCH58261, KW6002, ZM241385, GABA and Dehydro epiandrosteron.

An antioxidant includes, without limitation, one or more of a Alpha lipoic acid, Bilberry, CoQ10, Ginkgobioba, Glutathione, Grape seed extract, Green tea extracts, malatonine, Oligomeric proanthoscyanidins, Pycnogenol, Reseveritrol, Astaxanthin and Ergothineine.

An energy booster includes, without limitation, one or more of a Gotukola, Saint John's Wort, Wheet grass, Fennel, Kelp, Alfalfa, Red clover, Adenosine A (2A) and Common oats.

A fat or oil or their derivatives includes, without limitation, one or more of a Butter, Olive oil, Canola oil, Vegetable oil, Flax seed oil, Black currant seed oil, Primrose oil, Fish oil, Omega 3,6,9 poly unsaturated fatty acids, Milk, Condensed milk, Cheese, Nuts and DHA (Docosahexaenoic acid).

A color includes, without limitation, one or more of a Curcumin, Lycopene, Beta-carotene, Apocarotenal and Canthaxanthin.

Other natural productsthat may be considered as additional ingredients includes, without limitation, one or more of a Licorice root, Catnip, Passion flower, Lobelia, Hops, Skullcap, Gentian, Myrrh, Safflower, Bayberry root bark, Eucalyptus, Sarsaparilla, Slippery Elm, Valerian root, Ephedra, Guarana and kola nut.

The co-crystals of sweetener of this invention may be used as a sweetening ingredient of oral compositions, ingestible, partly non-ingestible as well as non-ingestible. Ingestible compositions may include, without limitation, one or more of a pharmaceutical composition, a nutraceutical composition, as an ingredient for a table top sweetener, as an ingredient of a sweet food product, as a cooking and baking ingredient for recipes and baked and cooked food products when all constituents of the sweetener of this invention are heat stable, as an ingredient of a beverage and as an ingredient of a sweet composition meant for reduced or low

calorie or weight control composition. Partly non-ingestible compositions include, without limitation, chewing gum. Non ingestible compositions include, without limitation, tooth paste and gargles. Co-crystals may also be used to retain flavor, aroma or volatile ingredients of pharmaceutical, nutraceutical or cosmetic use.

In a further embodiment of this invention, the sweetness enhanced compositions having sucrose as an ingredient shall possess better cane sugar like sweetness than sweetener compositions having Sucralose and no sucrose.

In a further embodiment, this invention comprises a sweetener that has appearance resembling cane sugar, and has controlled and definitive sweetness that is different than same weight quantity or same bulk density of sugar.

The ratio of sugar to water in this invention may range from - 0.3 to 3 times

The temperature of sweeteners dissolution may range from 30–95°C. Sugar to Sucralose ratio can range from 1: 11.5 to 1: 0.00025. The addition of the other sweeteners can be in any ratio to improve characteristics of the end product.

The dissolution of the said composition of sweeteners can be carried out in non-aqueous media including polar solvents, etc in which the sweeteners are able to dissolve or in mixture of solvents and in mixture of aqueous and non-aqueous solvents. However, at the time of inducing co-crystallization, organic solvents need to be removed.

Color of the solution can be improved by charcoal treatment or chemical oxidation including ozonization.

In large scale production process, the co-crystallization process steps of this invention are carried out in equipment like mass mixer, RCVD (Rotocon Vacuum drier) and similar kind of equipment capable of handling thick slurry formed towards end of crystallization. The process can be done in batches and also continuously.

Large scale manufacturing can be done under vacuum or without vacuum also. The process can be scaled up to several tons per day. Energy requirement for this process is minimal. The high intensity sweetener is retained over 99% in the product, if the drying of the product is done without isolating mother liquor i.e., the loss is negligible & hence this process is economically viable.

The crystallization is carried out by slow cooling and agitation with or without seeding of the components present in the solution. Seeding is alternatively repeated many times to accomplish complete crystallization. The temperature during crystallization may be varied from 65°C to 0°C in case of aqueous medium. In case of non-aqueous medium, temperatures can be reduced up to – 10°C.

In view of the prior art, at least one of the following steps are practiced as essential steps to get a co-crystallized product from sucrose: (a) heating the sugar syrup to a temperature higher than 100°C, (b) concentration under reduced pressure, (c) seeding concentrated syrup of sucrose and other ingredient by some quantity of sugar and (d) separating the mother liquor from the co-crystallized mass of the sweetener composition. It was surprising to find that in the method of this invention that none of the steps practiced as essential ones were essential to get final dried product that was low calorie, non-dusty, uniform in sweetness in each 6 gram portion and crystalline cane sugar like in appearance. Sucrose like appearance of the crystals of the co-crystalline product of this invention is an advantage from the point of view of aesthetic acceptability of this new sweetness enhanced sweetener by the consumers since they would prefer a sweetener which looks like sucrose and can be handled like sucrose, the only difference being use of less spoons of the sweetness enhanced sweetener than sucrose. Sucrose base of such sweeteners would make it sure that all organoleptic properties that are typical and exclusive to sucrose such as mouth feel, maillard reaction that produce agreeable flavors duing

cooking and baking would be available to the consumer and disagreeable after-tastes of high intensity sweeteners are also masked in the presence of sucrose. In the instant invention, co-crystallization was achieved even when the concentrated solution of sugar was not heated above 70°C, co-crystallizaiton was achieved at a temperature of about 55-60°C without seeding with sucrose and the final product was dried directly without separation of mother liquor. Since there was no loss of any sweetener ingredient in the process, this invention discloses a method wherein sweetener of any desired enhancement of sweetness with concurrent decline in calories for sucrose equivalent sweetness can be achieved accurately. For the purpose of this specification the term "Sucrose Equivalent Sweetness" is understood to mean sweetness of a sweetener in terms of sweetness of sucrose having same bulk, such as one teaspoon full, one spoon being defined as a spoon that becomes full with 6 gram of sucrose.

It is an embodiment of this invention that the high intensity sweetener and the optional ingredients are added to a concentrated solution of sucrose or sucrose and the optional ingredients are added to a concentrated solution of sucralose, the respective concentrated solution being so much saturated that after the dissolution of the other sweetener component, which, as the case may be, would be sucralose or sucrose, accompanied with warming to 50-70°C, results into a supersaturated solution. This step, although very simple, was never contemplated by prior art workers despite its advantages if used, and resulted in this invention into simplification of the process, since no further concentration under reduced pressure is needed to get co-crystallization.

It is necessary, however, that the concentrated solution of the sucrose, the high intensity sweetener and, if added, additional ingredients do not contain any ingredients that are difficult to dry at 40 to 45°C with or without vacuum and would

lead to formation of significant quantity of molasses or molasses like mother liquor. Thus, for example, the concentrated solution of sugar should not be the one derived from sugarcane juice or similar sugar crop juice that contains hygroscopic impurities such as reducing sugars in an amount that would generate molasses. Thus, this element makes this process very much different than a prior art process of adding high intensity sweetener in a conventional large scale sucrose manufacturing process in supersaturated syrup of sucrose at the stage immediately before crystallization, since even at that stage, in conventional process, sucrose would be associated with un-separated molasses and the mass of sucrose plus high intensity sweetener plus molasses can not be dried after the step of co-crystallizaiton into a free flowing powder, removal of molasses becomes a must after co-crystallization step and appreciable quantity of the costly added high-intensity sweetener is lost in the molasses, needing an additional step of extraction of the high intensity sweetener from the molasses for recycle, adding to unit processes / steps of the process as well as adding to the cost, making the process inefficient.

Thus this invention also discloses a method of making a crystalline sweetener cane sugar like in appearance, free flowing, dust free low calorie sweetener of uniform sweetness in each 6 gram portion from a concentrated solution containing sucrose and a high intensity sweetener as at least two ingredients, comprising steps of: (a) dissolving a high intensity sweetener and optionally other ingredients in a concentrated solution of sugar at 50 - 70°C to get a supersaturated concentrated solution in an aqueous solvent, or dissolving sucrose, and optionally other ingredients to a concentrated solution of sucralose, the said concentrated solution not containing impurities in a quantity that would result into formation of molasses or molasses like by-products after the step of co-crystallization during further steps of the process free from molasses, (b) allowing the concentrated solution to co-

crystallize below 70°C to incorporate most of the high intensity sweetener in the co-crystals, with or without seeding, leaving behind none or negligible mother liquor containing non-co-crysrallized sucrose and high intensity sweetener, and (c) drying the crystallized wet mass to stabilize the same microbiologically, usually upto less than 0.2% moisture by drying at a temperature below 50°C. The drying may be done without the step of separation of mother liquor from the crystallized mass, wherein the advantage is that sweetness of the resulting composition can be managed mathematically and with precision. However, the appearance of the crystals is a little dull, which dullness is not seen if the mother liquor is removed. Hence, if desired, mother liquor may also be separated and then the crystalline mass dried, which gives crystals which are devoid of dullness shiny as compared to the crystal where mother liquor is not removed. Mother liquor can be recycled to next batch or several batches of mother liquor can be pooled and processed to get the product. However, when mother liquor is removed, there is loss of expensive sucralose in it and the precision of the control on sweetness of the resulting product is also compromised to some extent.

Above embodiment of direct drying provides yet another important difference between all the prior art processes that result into generation of mother liquor that needs to be separated from co-crystallized product leading to losses of the expensive high intensity sweetener and creating a problem of handling and disposal of the mother liquor and loss of the expensive high intensity sweetener in the mother liquor.

Above mentioned embodiment of concentrated solution, free from molasses and containing sucrose can be achieved very conveniently by dissolving crystalline sucrose as starting material for this process. In this way, the process of this invention is different from a process wherein concentrated syrup/concentrated

solution obtained from sugarcane juice in the process of sugar production obtained before start of the crystallization process is used for starting the phase of co-crystallization, to which is added high intensity sweetener and further process of co-crystallization follows. In this process, production of molasses and loss of appreciable quantity of the expensive high intensity sweetener in the molasses is inevitable, making the process inefficient.

The co-crystallized product that is optionally centrifuged / filtered, if sucrose like shine on the co-crystals is desired, and mother liquor is optionally recycled to the next batch or taken to further repeat crystallization process. The product obtained is then dried, lumps broken and sieved to get product of desired particle size. To get better shine on the crystals, one may resort to charcoalization or ozonization treatment any appropriate decolorization method to the concentrated solutions at appropriate time.

It is also an embodiment of this invention wherein the crystallized mixture is subjected to direct drying without subjecting to filtration or centrifugation or to any step of separation of mother liquor and the co-crystals. Such dried compositions of a mix of co-crystalline preparation and residual sucrose and sucralose that are not co-crystallized also have a visual appearance as cane sugar like crystalline preparation and have uniform distribution of sweetness. Hence, they can be used as low calorie sweeteners with efficiency as good as and with same advantages as the co-crystalline preparations from which mother liquor is separated before drying.

The product of co-crystallization of this invention may also comprise a product formed by:

1. adding one or more of a high intensity sweetener, with or without other optional ingredients, to a concentrated solution of sucrose prior to co-

crystallization, or adding a concentrated solution of sucralose and dissolving in it sucrose, or

2. adding sucrose with or without other optional ingredients to concentrat solution of a high intensity sweetener prior to co-crystallization, or

3. when one or more of a high intensity sweetener, with or without additional optional ingredients, is added to a concentrated solution made from process stream of regular sugar manufacturing process after removal of molasses from the crystallized sucrose, or

4. when sucrose is added to a concentrated solution of process stream, containing a high intensity sweetener in purified state, produced in a process of manufacture of the high intensity sweetener, the said concentrated solution being free from organic solvent/s to such an extent that residual solvent will not interfere in the co-crystallization process wherein,

5. in all above instances, the resulting concentrated solution containing mixture of sucrose and the high intensity sweetener did not contain impurities in a quantity that would result into formation of molasses or molasses like by-products after the step of co-crystallization during further steps of the process; the final concentration of the solution of sucrose, the high intensity sweetener and other optional ingredients being at least enough for getting co-crystallization of the dissolved sucrose with the high intensity sweetener by stirring of the mixture of sucrose, the high intensity sweetener, with or without the other additional ingredients, for a period of time accompanied with heating without caramelization, to a temperature until co-crystallization is complete. It was seen that this concentration of dissolved solutes for co-crystallizaiton to occur would depend on the concentration of dissolved

solids as well as temperature at which the solution is held. In an illustrative experiment, Example no. 51, it was observed that this concentration, when the solution was heated to 60°C and stirred for co-crystallization was around 25% for getting co-crystallization in 13 hours and 142% for getting co-crystallization in 3 hours. However, exact transition point between the concentration at which no co-crystallizaiton will take place when kept on stirring at elevated temperature for a long time and the concentration where it would start taking place would be lower than this.

The sweetness enhanced products of this invention may also be subjected to make conventional and unconventional derivatives including caramelization.

The product composition thus obtained will have reduced calories in comparison to sucrose composition of same sweetness. The nature of co-crystallized product shows desired cohesiveness and uniformity. The crystals are analysed by NMR, HPLC, DSC, XRD and organoleptically. Ref. fig 1 to 11.

BRIEF DESCRIPTION OF FIGURES

FIG. 1: NMR OF CO-CRYSTALIZED SAMPLE. Sucrose, 50 g, co-crystallized with 0.083 g Sucralose of SUCRALOSE Example No. 8.

FIG. 2: HPLC OF COCRYSTALLIZED PRODUCT. Sucrose, 50 g, co-crystallized with 0.083 g Sucralose of EXAMPLE No. 8.

FIG. 3: HPLC OF SUCRALOSE. Source of Sucralose – In-house Batch No. BL008.

FIG. 4: DSC OF PHYSICAL BLEND. Sucrose, 50 g, mixed with, 0.083 g of Sucralose and 0.5 mg of sodium acetate to get physical blended product (Example 12).

FIG. 5: DSC OF COCRYSTALLIZED SAMPLE 1. Sucrose, 50 g, co-crystallized with 0.083 g Sucralose of Example No. 8.

FIG. 6: DSC OF COCRYSTALLIZED SAMPLE 2. Sucrose, 50 g is co-crystallized with 0.083 g of Sucralose (EXAMPLE 9).

FIG. 7: DSC OF COCRYSTALLIZED SAMPLE 3. Sucrose, 50 g, co-crystallized with 0.083 g of sucralose (Example 10).

FIG. 8: DSC OF SUCRALOSE. Source of Sucralose – In house Batch N0. BL008.

FIG. 9: DSC OF SUGAR. Source of sucrose – cane sugar (sucrose) produced by Bannari Amman Sugars limited, Erode, Tamilnadu, pharmaceutical grade.

FIG. 10: XRD OF COCRYSTALLIZED PRODUCT. Sucrose, 50 g, co-crystallized with 0.083 g Sucralose of Example No. 8.

FIG. 11: XRD OF PHYSICAL BLEND PRODUCT. Sucrose, 50 g, mixed with, 0.083 g of Sucralose and 0.5 mg of sodium acetate and physical blended product.

The sweetness of the crystallized composition in comparison to sugar for same bulk density, i.e. sucrose equivalent sweetness, may range from 1.15 times to 550 times of sugar. The ratio of sugar to sucralose for achieving the sweetness is 1:0.00025 and 1:11.5 times quantity wise respectively.

This invention is illustrated by following non-limiting examples, and any obvious modifications and equivalents of the same are construed to be within the scope of the claims.

In following examples, "demineralized" is abbreviated as "dm"; "hours" is abbreviated as "hrs", "gram" is abbreviated as "g" and "milligram" is abbreviated as "mg".

Sucralose used in below described experiments is sourced from an in-house produced Batch No. BL008.

Sucrose used is cane sugar, pharmaceutical grade, produced by EID Parry, (Tamilnadu).

Example No. 1

(A) Testing sweetness by organoleptic evaluation by an in-house panel:

The tasting panel is formed in-house comprising of 9 members selected from the age group of 30-45 years age and from different departments.

At a time not more than 6 samples are tasted. The samples are coded.

An illustrative example is as follows

Sample 1: 6 g of sucrose was dissolved in drinking water and volume was adjusted to 50 ml and coded as say ' C'.

Sample 2: 12 g of sucrose dissolved in drinking water and volume adjusted to 50 ml coded as say ' E '.

Sample 3: 3 g product prepared having 2 times sweetness of sugar (HPLC sucralose content between 0.16%-0.18%) dissolved in drinking water and volume adjusted to 50 ml and coded as 'A'.

Sample 4: 6 g of product from the same batch as of sample 3 is dissolved in drinking water and volume adjusted to 50 ml and coded as 'D'.

Sample 5: 2 g product prepared having sweetness of 3 times of sugar (HPLC sucralose content of 0.32%-0.365) is dissolved in drinking water and adjusted to 50 ml and coded as 'B'.

Sample 6: 4 g product prepared having sweetness of 3 times of sugar (HPLC sucralose content of 0.32%-0.365) is dissolved in drinking water and adjusted to 50 ml and coded as 'F'.

The tasting of samples were done either by dropper or by spoon.

The observations and comments of panel members are tabulated as follows, and results are analysed.

Above method is adopted for samples having 4, 5, 6 and up to 550 times sweetness and concentration is adjusted according to number of times sweetness the sample as per sucralose content (HPLC).

OBSERVATIONS ARE RECORDED AS FOLLOWS

The evaluator was asked for following profile and recorded in the Form given below

1.SWEETNESS

2.BITTER AFTER TASTE

3.TEXTURE

4.SHARP TASTE

5. VISCOUS

6. FLAVOR

ORGANOLEPTIC EVALUATION FORM FOR SUPER SUGAR

DATE:

Name of Evaluator: _____

MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN A & B	MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN A & C	MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN B &C	COMMENTS
MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN D &E	MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN D & F	MORE / EQUAL SWEETER AND OTHER PROFILE BETWEEN E & F	COMMENTS

Sign of the Evaluator _____

Testing uniformity in sweetness in sweetener composition by HPLC analysis:

Seven samples were drawn from seven different points of the same batch from the container. Each sample was labeled and sent for the HPLC analysis, and analyzed as per the standard protocol for Sucralose content analysis. The results were tabulated as below -

TABLE 1

S.No	Sucralose content in 7 Samples						
Batch.no	Sample 1	Sample2	Sample3	Sample4	Sample5	Sample6	Sample7
T07	0.16	0.15	0.16	0.15	0.16	0.16	0.16
T11	0.2	0.19	0.2	0.2	0.18	0.18	0.19
T14	0.13	0.14	0.13	0.11	0.13	0.13	0.14

Example 2

Process of Co-crystallization of sugar and sucralose to get higher sweetness using water as solvent (Aqueous co-crystallization process)

100 g of sucrose was dissolved in 70 ml of demineralized (DM) water at 55°C. The sugar was dissolved completely. 25 g of Sucralose was dissolved in to this solution. 2 mg of sodium acetate was added and dissolved. The solution was filtered. The filtrate was concentrated at temperature 40-42°C and vacuum of 30 torr and 24 ml water was distilled off. The mass was kept for stirring and cooling. When temperature reached 20°C, 5 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs, the mass was further seeded with 3 g of powdered sugar. The mass was further kept at 5°C for 12 hrs and centrifuged. The mother liquor was separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached to 0.1%. The dried crystals weighed 120 g. The product was broken and sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with

appearance like commercial cane sugar. The Sucralose content in the sample was 19.5%. The particle analysis shows crystals have 51% retention on 50 mesh sieve and 49 % passing. The solids obtained from mother liquor weighed 4 g with Sucralose content of 28.9%.

Example No.3

Co-crystallization process with charcoalization

50 g of sucrose was dissolved in 70 ml of DM water at 55°C. Sugar was dissolved completely. 12.5 g of Sucralose was dissolved to this solution. 1 mg of sodium acetate was added and dissolved. 0.5 g of activated charcoal was added to the solution. The solution was filtered over hyflo. The filtrate was concentrated at temperature 40-43°C and at vacuum of 30 torr and 50 ml of water was distilled off. The mass was kept for stirring and cooling. When temperature reached 20°C, 2 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs., the mass was further seeded with 1.0 g of powdered sugar and further kept at 5°C for 12 hrs. The product was centrifuged. The mother liquor was separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 58 g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 18.9%. Recovered solids weighed 3.0 g with Sucralose content of 20.3%. Particle analysis showed 53.9% retaining on 50 mesh.

Example No.4

Aqueous-organic solvent Co-crystallization process using organic solvent along with water

50 g of sucrose was dissolved in 150 ml of methanol containing 35 ml of water at 60°C. The sugar was dissolved completely. 12.5 g of Sucralose was dissolved to this solution. The solution was filtered. 1 mg of sodium acetate was added. The filtrate was concentrated at temperature 40-42°C and vacuum of 30 torr. Methanol was distilled off completely. The mass was kept for stirring and cooling. When temperature reached 20°C 5 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs. The mass was further seeded with 3 g of powdered sugar and further kept at 5°C for 12 hrs and the product was centrifuged. The mother liquor was separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 62.5 g. And 2.1 g recovered from mother liquor. The product was broken and sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 19.6%. The particle analysis shows crystals have 56.5 % retention on 50 mesh.

Example No.5

Co-crystallization using aqueous organic solvent, water and charcoalization

50 g of sucrose was dissolved in 150 ml of methanol containing 35 ml of DM water at 60°C. The sugar was dissolved completely. 12.5 g of Sucralose was dissolved. To this solution 0.5 g of activated charcoal was added. The solution was filtered over hyflo. To the clear filtrate 1 mg of sodium acetate was added. The filtrate was concentrated at temperature 40-42°C and vacuum of 30 torr to distill off methanol completely .The mass was kept for stirring and cooling. When temperature reached 20°C, 5 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs. The mass was further seeded with 3 g of

powdered sugar and further kept at 5°C for 12 hrs. The product was centrifuged. The crystals were dried under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 62.0 g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 19.0%. Product from mother liquor recovered was 4.5g, the sucralose content is 19.6%. The particle analysis shows crystals have 56.5 % retention on 50 mesh sieve.

Example No.6

Co-crystallization of sugar and sucralose with other high intensity sweetener, aspartame using aqueous alcohol as solvent

50 g of sucrose was dissolved in 150 ml of methanol and 32 ml of DM water at 60°C. The sugar was dissolved completely. 12.5 g of Sucralose was dissolved to this solution. The solution was filtered. 0.5 g of aspartame was added to this solution and 1 mg of sodium acetate was added. The solution was concentrated at temperature 40-42°C and vacuum of 30 torr to distill off methanol completely. The mass was kept for stirring and cooling. When temperature reached 20°C, 5 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs. The mass was further seeded with 3 g of powdered sugar and further kept at 5°C for 12 hrs. The product was centrifuged. The mother liquor separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached to 0.1%. The dried crystals weighed 63.5 g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 17.5 %. The particle analysis shows crystals

have 50.2 % retention on 50 mesh sieve and the solids recovered from mother liquor weighed 5.6 g with Sucralose content of 19.2%.

Example No.7

Co-crystallization of sugar and sucralose with high intensity sweetener using water only as solvent

50 g of sucrose was dissolved in 48 ml of DM water at 55°C. The sugar was dissolved completely. 12.5 g of Sucralose was dissolved to this solution. 1 mg of sodium acetate was added and dissolved. Solution was filtered. 0.5 g of aspartame was added to the solution. The solution was concentrated at temperature 40-43°C and vacuum of 30 torr to distill off 30 ml of water. The mass was kept for stirring and cooling. When temperature reached 20°C, 2 g of powdered sugar in 1 ml of DM water was added as seed. The mass was cooled to 10°C and stirred for 6 hrs. The mass was further seeded with 1.0 g of powdered sugar, further kept at 5°C for 12 hrs and the product was centrifuged. The crystals were dried under vacuum at 45°C until moisture content reached to 0.1%. The dried crystals weighed 61.2 g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 18.9%. The particle analysis shows crystals have 45.7 % retention on 50 mesh sieve and recovered solids from mother liquor weighed 3.5 g with Sucralose content of 21.3%.

Example No.8

Aqueous Co-crystallization process with direct crystallization without concentration and without cooling below 48°C to give cocrystallized product having 40 times sweetness of Sugar, with stabilizer.

79 g of sucrose was dissolved in 32 ml of DM water at 55°C the sugar was dissolved completely. 7.0 g of Sucralose was dissolved to this solution. 1 mg of sodium acetate was added and dissolved. The solution was filtered. The filtrate was stirred slowly at temperature 48 to 53°C for 28 hrs until complete crystallization. The product was centrifuged. The mother liquor was separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 81.0 g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 8.1 % by HPLC. The solids recovered from mother liquor weighed 3.0 g with Sucralose content of 8.6%, with particle size such that 54.1 % was retained on 50 mesh.

Example No.9

Co-crystallized product of Sugar, Sucralose with stabilizer having two times sweetness of Sugar

50 g of sucrose was dissolved in 20 ml of DM water at 50°C. The sucrose was dissolved completely. 0.083 g of Sucralose was dissolved to this solution. 0.5 mg of sodium acetate was added and dissolved. The solution was filtered. The filtrate was stirred slowly at temperature 48 - 54°C for 28 hrs. Observed for complete crystallization. The product was centrifuged. The mother liquor separated and analyzed for Sucralose content. The crystals were dried at under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 47.4 g. Further the product was sieved on 50 mesh sieve. The Sucralose content in the sample was 0.16 % by HPLC and recovered solids from mother liquor weighed 1.2 g with particle size such that 52.0 % was retained on 50 mesh. The product was crystalline

in nature, non-dusty, with appearance like commercial cane sugar. The NMR of the product has been recorded and was as shown in FIG 1. The HPLC of the product has been recorded and is as shown in FIG 2. The DSC of the product has been recorded and is as shown in FIG 5. The XRD of the product has been recorded and is as shown in FIG 10.

Example No.10

Co-crystallized product of Sugar, Sucralose with stabilizer having two times sweetness of Sugar

50 g of sucrose was dissolved in 20 ml of DM water at 54°C. The sucrose was dissolved completely. 0.091 g of Sucralose was dissolved to this solution. 0.5 mg of sodium acetate was added and dissolved. The solution was filtered the filtrate was stirred slowly at temperature 48-56°C which led to crystallization. Crystallization was allowed to proceed for 29 hrs for crystallization. The product was centrifuged. The mother liquor separated and analyzed for Sucralose content. The crystals were dried under vacuum at 45°C until moisture content reached 0.4%. The dried crystals weighed 47.3g. Further the product was sieved on 50 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The Sucralose content in the sample was 0.179 % by HPLC. Solids recovered from mother liquor weighed 1.5 g and particle size was such that 55.3 % was retained on 50 mesh.. The product has double the sweetness of sugar. The DSC of the product has been recorded and was as shown in FIG 6.

Example No.11

Co-crystallized product of Sugar, Sucralose with stabilizer

50 g of sucrose was dissolved in 20 ml of DM water at 56°C. The sucrose was dissolved completely. 0.074 g of Sucralose was dissolved to this solution. 0.5mg of

sodium acetate was added and dissolved. The solution was filtered the filtrate was stirred slowly at temperature of 48-54°C for 28 hrs. Crystallization occurred. The product was centrifuged. The mother liquor was separated and analyzed for Sucralose content. The crystals were dried at under vacuum at 45°C until moisture content reached to 0.2%. The dried crystals weighed 46.3 g. Further the product was sieved on 50 mesh sieve. The Sucralose content in the sample was 0.142 % by HPLC. Solids recovered from mother liquor weighed 1.4 g. With Sucralose content of 0.15% and particle size such that 53.1 % was retained on 50 mesh. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product has double the sweetness of sugar. The DSC of the product has been recorded and is as shown in FIG 7.

Example 12

Co-crystallized product having nearly 250 times sweetness of Sugar

28 g of sucrose was dissolved in 25 ml of DM water at 55°C. The sucrose was dissolved completely. 22g of Sucralose was dissolved to this solution 0.5mg of sodium acetate was added and dissolved. The solution becomes clear and was filtered. The filtrate was stirred continuously at 48-52°C for 31 hrs. Crystallization occurred. The product was centrifuged, mother liquor separated and processed separately. The crystalline solids dried at 45°C under vacuum, the moisture content reached 0.1 %. Lumps of dried crystals were broken and sieved. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. 52% of the product was retained on 50 mesh. Yield obtained was 46.7 g, and further 2.4 g solids recovered from mother liquor. The HPLC result shows Sucralose content of 43.8% and the moisture content was 0.2%.

Example 13

Product of Physical blend of Sugar and Sucralose (for comparison with co-crystallized product)

To 50 g of sucrose, 0.083 g of Sucralose and 0.5 mg of sodium acetate are added and physically blended. The moisture content will be less than 0.2%. The product weigh was 50.5 g. The mixture was dusty although predominantly it looked like cane sugar. Five different samples were picked from this same batch at random. Sucralose content of the five different samples, as measured by HPLC were 0.103, 0.107, 0.11, 0.105 and 0.1107%. The DSC of the physically blended product was recorded as shown in FIG 4. The XRD of this product is recorded as shown in FIG 11.

Example No. 14

Co-crystallized product having 11 times sweetness of Sugar

Co-crystallized product was prepared to get sweetness 11 times sweeter than sugar as depicted in this example. 9.8 kg of sugar was dissolved in 4 L of demineralized water at 55°C in 20 L stainless steel equipment having stirring arrangement and jacketed system. The sugar was dissolved completely. 200 g of sucralose was dissolved to this solution. 240 mg of sodium acetate was added and dissolved. The solution was filtered. The filtrate was kept for crystallization under stirring at temperature 60-65°C. The mass was kept for stirring for 8 hours till crystallization is complete. . The product was centrifuged. The mother liquor quantity of 200 ml was separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.1% and the weight of dried crystals was 9.8 kg. Further the product was sieved on 50 mesh (297 micron) sieve. The sucralose content in the sample was 1.98% and sugar content as expected. The particle analysis shows crystals have 51 % retention on 50 mesh (297 micron) sieve

and 49 % passing. The co-crystallized product was tested for sweetness by dissolving sample in water equivalent to 11 times of sugar. The organoleptic test was performed with panel selected in-house consisting of 9 members. The opinion of panel members reveals the product has 11 times sweetness of sugar of same bulk density, which was also confirmed by HPLC result of sucralose content.

Example No. 15

Co-crystallized product for studying the stability of product with and without stabilizer

The co-crystallized product was prepared with and without stabilizer as followed in this example. Parallel experiments were conducted with all the quantities of inputs and parameters remaining same. 100g of sugar was dissolved in 40 ml of DM water at 50-55°C. The sugar was dissolved completely. 0.2g of sucralose was dissolved to this solution. In one experiment 1.0 mg of sodium acetate was added as stabilizer, whereas in another experiment done in same way, no sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 58-59°C. After 3 hrs, 2 g of sugar was added as seed. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor was separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to around 0.1%. Further the product was sieved on 50 mesh (297 micron) sieve. The product obtained with stabilizer is 97g and product obtained without stabilizer is 96.8g.

Stability procedure : The samples were packed in sealed containers and stored in stability chamber which is conditioned for 75% humidity at temperature of 40°C for 1month. Then the samples were drawn and analyzed for sucralose content by

HPLC, and studied for the intactness of the sucralose. This is the accelerated stability study. However all the samples were studied as per the standard protocol.

The sucralose content in the samples are as follows –

	Product with Stabilizer Sample before Stability Study	Product with Stabilizer Sample after 1 month	Product without Stabilizer Sample before Stability Study	Product without Stabilizer Sample after 1 month
Stability conditions		Temp : 40°C RH : 75		Temp : 40°C RH : 75
Sucralose content (%)	0.17 %	0.17 %	0.169 %	0.169 %

Conclusion : The accelerated study shows no sucralose loss, whether the samples were prepared using stabilizer and without stabilizer.

Example No. 16

Co-crystallized Product having 3 times sweetness of Sugar

The co-crystallized product was also prepared without using any stabilizer and having 3 times sweetness of sugar as followed in this example. 100g of sugar was dissolved in 40 ml of Demineralized (DM) water at 50-55°C, the sugar was dissolved completely. 0.4g of Sucralose was dissolved to this solution. The solution was filtered. The filtrate was stirred at temperature 57-59°C. After 3 hrs, 2 g of sugar was added as seed. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.1% the dried crystals weighing 98g. Further the product was sieved on 50 mesh (297 micron) sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.38 % and sugar content as

expected. The product was tested for sweetness in-house panel. The sweetness profile shows product was 3 times sweeter than sugar. The taste is sharp, without any bitterness.

Example No. 17

Co-crystallized Product having 4 times sweetness of Sugar

The co-crystallized product was also prepared without stabilizer and having sweetness 4 times of sugar as followed in this example. 100g of sugar was dissolved in 40 ml of Demineralized (DM) water at 55-58°C. The sugar was dissolved completely. 0.6g of sucralose was dissolved to this solution. The solution was filtered. The filtrate was stirred at temperature 58-59 oC. After 2 hrs, 1 g of sugar was added as seed. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.12% the dried crystals weighing 98g. Further the product was sieved on 50 mesh (297 micron) sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.49% and sugar content as expected. The product has four times sweetness of sugar. Sweetness taste profile is sharp and no bitterness.

Example No. 18

Co-crystallized Product having 5 times sweetness of Sugar

The co-crystallized product was prepared with stabilizer and sweetness of 5 times of sugar as shown in this example. 100g of sugar was dissolved in 40 ml of Demineralized (DM) water at 56-59°C. The sugar was dissolved completely. 0.8g of sucralose was dissolved to this solution. 2.4 mg of sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 56-59°C. After 2 hrs, 2g

of sugar was added as seed. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor was separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.13% the dried crystals weighing 99g. Further the product was sieved on 50 mesh (297 micron) sieve. The sucralose content in the sample was 0.76% and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar.. The product has five times sweetness of sugar. . Sweetness taste profile is sharp and no bitterness.

Example No. 19

Co-crystallized Product having 6 times sweetness of Sugar

The co-crystallized product was also prepared with stabilizer and sweetness of 6 times of sugar and without seeding as shown in this example. 99.0g of sugar was dissolved in 40 ml of Demineralized (DM) water at 56-59°C. The sugar was dissolved completely. 1.0g of sucralose was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 56-59°C. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.1%, the dried crystals weighing 98.6g. Further the product was sieved on 50 mesh (297 micron) sieve. The sucralose content in the sample was 0.98% and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product has nearly six times sweetness of sugar. Sweetness taste profile is sharp and no bitterness.

Example No. 20

Co-crystallized Product having 7 times sweetness of Sugar

The co-crystallized product was also prepared with stabilizer and sweetness of 7 times of sugar and without seeding as shown in this example. 98.8g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60-62°C. The sugar was dissolved completely. 1.2g of sucralose was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 60-62°C. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 45°C until moisture content reached to 0.14%, the dried crystals weighing 99.6g. Further the product was sieved on 50 mesh (297 micron) sieve. The sucralose content in the sample was 1.1% and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product has seven times sweetness of sugar. Sweetness taste profile is sharp, without bitterness.

Example No. 21

Co-crystallized Product having 8 times sweetness of Sugar

The co-crystallized product was also prepared with stabilizer and sweetness of 8 times of sugar and with seeding as shown in this example. 98.6g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60-62°C, the sugar was dissolved completely. 1.4g of sucralose was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 62°C. The mass was stirred to complete crystallizationand product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 40-45°C until moisture content reached to 0.1%, the dried crystals weighing 97.0g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 1.38% and sugar content as expected. The

product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product has eight times sweetness of sugar of same bulk density as per taste and also by HPLC analysis of sucralose content.

Example No. 22

Co-crystallized Product having 9 times sweetness of Sugar

The co-crystallized product was also prepared with stabilizer and sweetness of 9 times of sugar and with seeding as shown in this example. 98.4g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60-62°C. The sugar was dissolved completely. 1.6g of sucralose was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was filtered. The filtrate was stirred at temperature 62°C. The mass was stirred to complete crystallization and product was centrifuged. The mother liquor separated and analyzed for sucralose content. The crystals were dried at 40-45°C until moisture content reached to 0.17%, the dried crystals weighing 98g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 1.51% and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product has nearly nine times sweetness of sugar. The sweetness profile is sharp, without any bitterness.

Example No. 23

Co-crystallized Product of Sugar and Sucralose along with other high intensity sweetener (Saccharin)

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose as shown in this example. 99.6g of sugar was dissolved in 40 ml of Demineralized (DM) water at 58°C. The sugar was dissolved completely. 0.2 g

of sucralose and 0.27 g of saccharin was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was stirred at temperature 62°C. The mass was seeded with 1 g of sugar. The mass was stirred to complete. The crystals were dried at 40-45°C until moisture content reached to 0.11%, the dried crystals weighing 99.5 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.19% and saccharin is estimated by HPLC, which is 0.25%. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the in-house panel. The sample had about 2.5 times sweetness with respect to sugar. The taste is sharp with faint bitter taste.

Example No. 24

Co-crystallized Product of Sugar and Sucralose along with other high intensity sweetener (Acesulfame-K)

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose as shown in this example. 99.6g of sugar was dissolved in 40 ml of Demineralized (DM) water at 58°C, the sugar was dissolved completely. 0.2 g of sucralose and 0.27 g of Acesulfame-k was dissolved to this solution. 2.4mg of sodium acetate was added. The solution was stirred at temperature 57°C. The mass was seeded with 1 g of sugar. The mass was stirred till complete crystallization of product. The crystals were dried at 40-45°C until moisture content reached to 0.14%, the dried crystals weighing 99.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.18% and sugar content as expected. The acesulfame-k content by HPLC is 0.23%. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar.. The product was tested for sweetness and taste profile by the panel, in-

house. The sample had about 2.5 times sweetness with respect to sugar. The product is sharp in taste.

Example No. 25

Co-crystallized Product of Sugar and Sucralose along with low intensity sweetener (Fructose)

The co-crystallized product was also prepared with another low intensity sweetener along with sucralose and sugar as shown in this example. 95.0g of sugar was dissolved in 40 ml of Demineralized (DM) water at 58°C, the sugar was dissolved completely. 0.2 g of sucralose and 4.8 g of fructose was dissolved to this solution. The solution was stirred at temperature 57-60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.16%, the dried crystals weighing 99.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.19% and sugar content as expected. The fructose content is specified by input certification. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness is nearly 2 times sweeter than sugar, with sharp and pleasant taste.

Example No. 26

Co-crystallized Product of Sugar and Sucralose along with Polyols (Sorbitol)

The co-crystallized product was also prepared with polyol along with sucralose and sugar as shown in this example. 95.0g of sugar was dissolved in 40 ml of Demineralized (DM) water at 56°C, the sugar was dissolved completely. 0.2 g of sucralose and 4.8g of sorbitol was dissolved to this solution. The solution was

stirred at temperature 57-60°C, the mass was seeded with 1 g sugar. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.13%, the dried crystals weighing 98.0g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.2% and sugar content as expected. The content of sorbitol is specified by input certification. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness is nearly twice of sugar. The taste is sharp.

Example No. 27

Co-crystallized Product of Sugar and Sucralose along Polyols (Xylitol)

The co-crystallized product was also prepared with polyol along with sucralose and sugar as shown in this example. 95.0 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 54°C, the sugar was dissolved completely. 0.2 g of sucralose and 4.8g of xylitol was dissolved to this solution. The solution was stirred at temperature 60°C., the mass was seeded with 1 g sugar. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.15%, the dried crystals weighing 98.0g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.16% and sugar content as expected. The xylitol content is specified by input certification. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The product is double the sweetness compared to sugar.

Example No. 28

Co-crystallized Product of Sugar and Sucralose along with Vitamins (therapeutically significant supplementation of Vitamin C, providing 20% supplementation of the RDA in three serves; About 0.18g Vitamin-C available per serving)

The co-crystallized product was also prepared with vitamin along with sucralose and sugar as shown in this example with vitamin-C. 93.8 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 50°C., the sugar was dissolved completely. 0.2 g of sucralose was dissolved to this solution. The solution was stirred at temperature 60°C., the mass was seeded with 1g sugar. 6g of Vitamin-C (Ascorbic acid) added and the mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.15%, the dried crystals weighing 98.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.18% and sugar content as expected. The Vitamin-C content is estimated by HPLC which is 5.8%. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. Product was double the sweetness of sugar. The taste is acceptable.

Example No. 29

Co-crystallized Product of Sugar and Sucralose along with natural colors (to provide 0.006g of Curcumin per serving)

The co-crystallized product was also prepared with natural color along with sucralose and sugar as shown in this example with Curcumin. 99.6 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 49°C, the sugar was dissolved

completely. 0.2 g of sucralose and 0.2g of curcumin was dissolved to this solution. The solution was stirred at temperature 60°C, the mass was seeded with 1 g sugar. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.13%, the dried crystals weighing 99.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.18% and sugar content as expected. Curcumin is quantified by input certification. The product was uniformly yellow in color. The product was tested for sweetness and taste profile by the panel, in-house. The sweetness was 2 times of sugar and taste profile is acceptable.

Example No. 30

Co-crystallized Product of Sugar and Sucralose along with energy booster (therapeutically significant supplementation of St. John Wort, providing 20% supplementation of the RDA in three serves; 0.018g of St. John Wort per serve)

The co-crystallized product was also prepared with energy booster along with sucralose and sugar as shown in this example with St. John Wort. 99.2 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 59°C, the sugar was dissolved completely. 0.2 g of sucralose and 0.6g of St. John Wort was dissolved to this solution. The solution was stirred at temperature 62°C. The mass was stirred to complete. The crystals were dried at 40-45 °C until moisture content reached to 0.12%, the dried crystals weighing 99.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.18% and sugar content as expected. The St. John wort is quantified by input certification. The nature of the product was off-white. The product was crystalline in nature, non-

dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness was 2 times of sugar and taste profile is acceptable.

Example No. 31

Co-crystallized Product of Sugar and Sucralose along with Prebiotics (therapeutically significant supplementation of Inulin, providing 20% supplementation of the RDA in three serves)

The co-crystallized product was also prepared with prebiotics along with sucralose and sugar as shown in this example with Inulin. 99.6 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 62 °C, the sugar was dissolved completely. 0.2 g of sucralose and 0.2g of inulin was dissolved to this solution. The solution was stirred at temperature 63°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45 °C until moisture content reached to 0.11%, the dried crystals weighing 98.0 g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.18% and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness profile is like sugar and has double the sweetness of sugar.

Example No. 32

Co-crystallized Product of Sugar and Sucralose along with Anti-oxidant (therapeutically significant supplementation of Green Tea extract, providing 20% supplementation of the RDA in three serves; 0.018g of Green tea extract available per serve)

The co-crystallized product was also prepared with an antioxidant along with sucralose and sugar as shown in this example with green tea extract. 99.2 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 62°C, the sugar was dissolved completely. 0.2 g of sucralose and 0.6g of green tea extract was dissolved to this solution. The solution was stirred at temperature 63°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45 °C until moisture content reached to 0.14% the dried crystals weighing 99.0g. Further the product was sieved on 20 mesh (841 micron) sieve. The sucralose content in the sample was 0.17 % and sugar content as expected. The estimation of green tea extract was by input certification. The product was slightly pinkish and crystalline in nature. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel, in-house. The sweetness was 2 times sweetness of sugar and the taste profile is acceptable.

Example No. 33

Co-crystallized Product of Sugar and Sucralose along with another natural sweetener (Maltose)

The co-crystallized product was also prepared with other natural sweetener along with sucralose and sugar as shown in this example with maltose. 95.0 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 58°C The sugar was dissolved completely. 0.2 g of sucralose and 4.8g of maltose was dissolved to this solution. The solution was stirred at temperature 63 °C. The mass was stirred to complete crystallization. The crystals were dried at 40-45 °C until moisture content reached to 0.14% the dried crystals weighing 99.5 g. Further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.18 % and sugar

content as expected. The maltose content was by input certification. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness was sharp in taste and has sweetness 2 times of sugar.

Example No. 34

Co-crystallized Product of Sugar and Sucralose along with high intensity sweetener (Acesulfame-K, to get double sweetness)

The co-crystallized product was also prepared with other high intensity sweetener along with sucralose and sugar as shown in this example with Acesulfame-K. 99.83 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 59°C. The sugar was dissolved completely. 0.162 g of sucralose and 0.013 g of Acesulfame-K was dissolved to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.12% the dried crystals weighing 99.0 g. Further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.16 % and sugar content as expected. Acesulfame-K content was 0.012%, analyzed by HPLC. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The sweetness was 2 times of sugar. The taste profile is sharp, pleasant without any bitterness.

Example No. 35

Co-crystallized Product of Sugar and Sucralose along with amino acids : therapeutically significant supplementation of Methionine, providing 20% supplementation of the RDA in three serves; 0.06g of L-Methionine per serve:

The co-crystallized product was also prepared with proteins and amino acid along with sucralose and sugar as shown in this example with L-Methionine. 97.7 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60.0°C. The sugar was dissolved completely. 0.2 g of sucralose and 2.1g of L-Methionine was dissolved to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45 °C until moisture content reached to 0.14% the dried crystals weighing 97.8 g. Further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar.The sucralose content in the sample was 0.19 % and sugar content as expected. L-methionine content was 1.9%, as estimated by Kjeldhal method. The particles are crystalline. The sweetness is double of sugar. Taste profile is acceptable.

Example No. 36

Co-crystallization process by duplicating the Sucralose crystallization process (In the manufacture of Sucralose) to get the co-crystallized product

The process of co-crystallization was applied to sucralose crystallization in the plant. The same process was carried out in lab scale to get co-crystallized product of sugar and sucralose. The details of the process are as follows. 100.0 g of sucralose was added to 100 ml of Demineralized (DM) water at 55°C. After the solution was clear, 50 g of sugar was added and continued stirring for 90 minutes at 55°C. Mass was cooled to 20 °C and stirred at 20 °C for 1 hr. Product crystallized out. The mass was filtered. The wet cake of 109 g was obtained which was dried at 40-45 °C. The product obtained was 107.0 g. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The moisture content was 0.16% and

sucralose content was 46.2%. The sweetness profile is nearly matching to sugar and sweetness is nearly 270 times of sugar.

Example No. 37

Co-crystallized product of Sugar, Sucralose and other high intensity sweetener (Saccharin) to get the product with double the sweetness of Sugar

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose and sugar as shown in this example with saccharin. 99.62 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 57°C. The sugar was dissolved completely. 0.10 g of sucralose and 0.275 g saccharin was dissolved to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.14%. The dried crystals weighing 99 g. Further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.09% and sugar content as expected. The saccharin content was 0.25% by HPLC. The sweetness was 2 times of sugar and taste profile is sharp, with slight bitterness.

Example No. 38

Co-crystallized product of Sugar, Sucralose and other high intensity sweetener (Acesulfame-K) to get the product with double the sweetness of Sugar

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose and sugar as shown in this example with Acesulfame-K. 99.6 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 57.0°C. The sugar was dissolved completely. 0.10 g of sucralose and 0.3 g with Acesulfame-K was

dissolved to this solution. The solution was stirred at temperature 60.0°C. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.15 %. The dried crystals weighing 99.5 g further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.091 % and sugar content as expected. Acesulfame-K content was 0.26% by HPLC. The product was tested for sweetness and taste profile by the panel in-house. The sweetness was 2 times of sugar. The taste was sharp, almost matching that of sugar.

Example No. 39

Co-crystallized product of Sugar, Sucralose and other high intensity sweetener (Stevia) to get the product with double the sweetness of Sugar

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose and sugar as shown in this example with stevia. 99.62 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 59°C. The sugar was dissolved completely. 0.10 g of sucralose and 0.275 g with stevia were dissolved to this solution. The solution was stirred at temperature 60 °C. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.17% the dried crystals weighing 99.0g. Further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.09 % and sugar content as expected. The Stevia content was by input certification. The product was tested for sweetness and taste profile by the panel in-house. The product has nearly 2 times sweetness and good taste profile.

Example No. 40

Co-crystallized product of Sugar, Sucralose and other high intensity sweetener (Neotame) to get the product with double the sweetness of Sugar

The co-crystallized product was also prepared with another high intensity sweetener along with sucralose and sugar as shown in this example with Neotame. 99.13 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 59°C. the sugar was dissolved completely. 0.083 g of sucralose and 0.0038 g with Neotame was dissolved to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.16% the dried crystals weighing 99.0g, Further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.08 % and sugar content as expected. The Neotame content was by input certification. The sweetness of product is twice of sugar. The taste is sharp, without any bitterness.

Example No. 41

Co-crystallized product of Sugar, Sucralose along with flavor (Vanilla)

The co-crystallized product was also prepared with a flavor along with sucralose and sugar as shown in this example with Vanilla. 97.8 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 63°C, the sugar was dissolved completely. 0.2 g of sucralose and 2.0 ml of vanilla flavor was dissolved to this solution. The solution was stirred at temperature 60 °C. The mass was stirred to complete crystallization. The product was dried at 40-45 °C until moisture content reached to 0.11.% the dried crystals weighing 98.0g Further the product was sieved on 20 mesh sieve. The

product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.17 % and sugar content as expected. The product was twice the sweetness of sugar. The taste was sharp, pleasant vanilla flavor.

Example No. 42

Co-crystallized product of Sugar, Sucralose along with flavor (Orange)

The co-crystallized product was also prepared with a flavor along with sucralose and sugar as shown in this example with orange. 97.8g of sugar was dissolved in 40 ml of Demineralized (DM) water at 59°C, the sugar was dissolved completely. 0.2 g of sucralose 1.6 mg of sodium acetate and 2.0 ml of orange flavor was dissolved to this solution. The solution was stirred at temperature 62°C.The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.12% the dried crystals weighing 97g, further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.18 % and sugar content as expected. The particles are crystalline. The product was twice the sweetness of sugar. The taste was pleasant with orange flavor.

Example No. 43

Co-crystallized product of Sugar, Sucralose along with flavor (Strawberry)

The co-crystallized product was also prepared with a flavor along with sucralose and sugar as shown in this example with strawberry. 97.8g of sugar was dissolved in 40 ml of Demineralized (DM) water a 59°C, the sugar was dissolved completely. 0.2 g of sucralose, 2.0 g of strawberry flavor was dissolved to this solution. The solution was stirred at temperature 60 °C. The mass was stirred to complete crystallization.

The product was dried at 40-45 °C until moisture content reached to 0.12% the dried crystals weighing 97.2 g further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.18 % and sugar content as expected. The particles are crystalline. The product has 2 times sweetness and good strawberry flavor.

Example No. 44

Co-crystallized product of Sugar, Sucralose along with DHA (Docosahexaenoic acid):

The co-crystallized product was also prepared with DHA. 99.6 g of sugar was dissolved in 40 ml of Demineralized (DM) water a 60°C, the sugar was dissolved completely. 0.2 g of sucralose and 0.2 g of DHA was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45 °C until moisture content reached to 0.12%. The dried crystals weighing 98.0g further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.19 % and sugar content as expected. The product has 2 times sweetness and faint taste/flavor of DHA.

Example No. 45

Co-crystallized product of Sugar, Sucralose and minerals (Molybdenum chelate: therapeutically significant supplementation of Molybdenum providing 20% supplementation of the RDA in three serves;)

The co-crystallized product was also prepared with minerals along with sucralose and sugar as shown in this example with Molybdenum content. 99.6 g of sugar was dissolved in 40 ml of Demineralized (DM) water a 60°C. the sugar was dissolved completely. 0.2 g of sucralose and 0.0016 g of Molybdenum chelate (in-house product) was added to this solution. The solution was stirred at temperature 60°C.The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.12% the dried crystals weighing 99.0g, further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.17 % and sugar content as expected. The molybdenum content was by input certification. The product has double the sweetness of sugar with no appreciable metallic taste.

Example No. 46

Co-crystallized product of Sugar, Sucralose by complete crystallization without separating the mother liquor to get the product of double sweetness of Sugar

The co-crystallized product was prepared by complete crystallization without filtration and without separation of mother liquor .99.8 g of sugar was dissolved in 40 ml of RO water a 60°C. The sugar was dissolved completely. 0.18 g of sucralose was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.15% the dried crystals weighing 99.0g. Further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.18 % and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for

sweetness and taste profile by the panel in-house. The product has 2 times sweetness.

Example No. 47

Co-crystallized product of Sugar, Sucralose by complete crystallization without separating the mother liquor to get the product of four times sweetness of Sugar

The co-crystallized product having 4 times sweetness was prepared by complete crystallization without filtration and without separation of mother liquor . 99.5 g of sugar was dissolved in 40 ml of Demineralized (DM) water a 60°C. the sugar was dissolved completely. 0.54 g of sucralose was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.15% the dried crystals weighing 99.0g further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.53 % and sugar content as expected. The particles are crystalline. The product was tested for sweetness and taste profile by the panel in-house. The product has 4 times sweetness. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. Even in case of mother liquor separation the crystallization is complete and in case of without mother liquor separation, no striking difference is observed in the appearance of the crystals, It is only when observed very minutely, shining of crystals observed in case of mother liquor separation was just a little more bright.

Example No. 48

Co-crystallized product of Sugar, Sucralose along with therapeutically significant supplementation of Theobromine, providing 20% supplementation of the RDA (1.3mg of Theobromine per serve)

The co-crystallized product was also prepared with therapeutics along with sucralose and sugar as shown in this example with Theobromine. 99.8 g of sugar was dissolved in 40 ml of Demineralized (DM) water a 60°C, the sugar was dissolved completely. 0.2 g of sucralose and 0.043 g of Theobromine was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The product was dried at 40-45°C until moisture content reached to 0.14% the dried crystals weighing 98.5g, further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.17 % and sugar content as expected. The theobromine content was 0.04% by HPLC. The particles are crystalline. The product has 2 times sweetness and has good taste profile.

Example No. 49

Co-crystallized product of Sugar, Sucralose to get the product of 1.25 times sweetness of Sugar without separation of mother liquor

The co-crystallized product having 1.25 times sweetness was prepared by complete crystallization without filtration and without separation of mother liquor .100.0 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60°C. The sugar was dissolved completely. 0.01g of sucralose was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization.

Time taken for crystallization was about 4 hrs. The crystals were dried at 40-45°C until moisture content reached to 0.12.% the dried crystals weighing 99.0g further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.01 % and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The product has 1.25 times sweetness. The crystals are sugar like.

Example No. 50

Co-crystallized product of Sugar, Sucralose to get the product of 2 times sweetness of Sugar using less quantity of water and without separation of mother liquor

The co-crystallized product having 2 times sweetness was prepared by using low water ratio1:0.3 (sugar: water) complete crystallization without filtration and without separation of mother liquor. 99.8g of sugar was dissolved in 30 ml of Demineralized (DM) water a 60°C. the sugar was dissolved completely. 0.2 g of sucralose was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.13% the dried crystals weighing 98.0g further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.22 % and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The product has 2 times sweetness. Product has sugar like appearance has good taste profile.

Example No. 51

Co-crystallized product of Sugar, Sucralose to get the product of 2 times sweetness of Sugar using high quantity of water and without separation of mother liquor

The co-crystallized product having 2 times sweetness was prepared by using high water ratio 1:3.0 (sugar: water) complete crystallization without filtration and without separation of mother liquor. 99.8g of sugar was dissolved in 300 ml of Demineralized (DM) water a 60°C. The sugar was dissolved completely. 0.2 g of sucralose was added to this solution. The solution was concentrated to remove about 200 ml water, the concentrated solution was about 142% dissolved solids, which was further stirred at temperature 60°C for 3 hours. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.16% the dried crystals weighing 98.0g. Further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.20 % and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product was tested for sweetness and taste profile by the panel in-house. The product has 2 times sweetness and sweetness profile is like sugar.

When the same experiment was repeated without removal of water, i.e. when the concentrated solution was stirred at about 60°C and concentration of the solutes was 25%, the co-crystallization occurred even in that case, although it took a longer period of stirring for 13 hours.

When the same experiment was repeated wherein stirring was done at room temperature of 29°C, there was no co-crystallization. Also no co-crystallization occurred in repeats of above experiment when (a) no stirring was done for co-

crystallization step at 60°C, or (b) seeding was done to induce co-crystallization but the solution was kept standing without stirring at 60°C, Thus, stirring at an elevated temperature relative to room temperature of 29°C was an essential condition for co-crystallization to occur within a reasonable time. In this experiment, temperature of 60°C was used as an elevated temperature. However, any other temperature could have been used unless it led to caramelization; higher the temperature, lesser is the time required for co-crystallization.

Example No. 52

Co-crystallized product of Sugar, Sucralose to get the product of 1.15 times sweetness of Sugar without separation of mother liquor

The co-crystallized product having 1.15 times sweetness, low ratio of sugar:sucralose(1:0.00025) was prepared by complete crystallization without filtration and without separation of mother liquor. 100.0 g of sugar was dissolved in 40 ml of Demineralized (DM) water at 60°C. The sugar was dissolved completely. 0.025 g of sucralose was added to this solution. The solution was stirred at temperature 60°C. The mass was stirred to complete crystallization. The crystals were dried at 40-45°C until moisture content reached to 0.15% the dried crystals weighing 99.0g further the product was sieved on 20 mesh sieve. The sucralose content in the sample was 0.02 % and sugar content as expected. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar.. The product was tested for sweetness and taste profile by the panel in-house. The product has 1.15 times sweetness. Product has sugar like appearance.

Example No. 53

Co-crystallized product of Sugar, Sucralose to get the product of 550 times sweetness of Sugar without separation of mother liquor

The co-crystallized product having very high sweetness about 550 times sweetness ,high ratio of sugar:sucralose(1:11.5) was prepared by complete crystallization without filtration and without separation of mother liquor. 8.0 g of sugar was dissolved in 140 ml of Demineralized (DM) water , the sugar was dissolved completely 92.0 g of sucralose was added to this solution and dissolved completely. The mass was stirred to complete crystallization at 60-65°C. The crystals were dried at 40-45°C until moisture content reached to0.14.% the dried crystals weighing 97.2 g ,further the product was sieved on 20 mesh sieve. The product was crystalline but the prduct did not have crystalline appearence. The sucralose content in the sample was 91.7 % and sugar content as expected. The particles are crystalline. The product was tested for sweetness and taste profile by the panel in-house. The product has nearly 550 times sweetness.

Example No. 54

Co-crystallized product of Sugar, Sucralose along with Probiotics (therapeutically significant supplementation of *Bacillus coagulans*, providing 20% supplementation of the RDA in three serves; 200 million spores of *Bacillus coagulans* per serve)

The co-crystallized product was also produced with probiotics, as shown in this example with *Bacillus coagulans* having strength of 6 billion cfu/gm. prepared along with sugar and sucralose by complete crystallization without filtration and without separation of mother liquor. 98.8 g of sugar was dissolved in 40 ml of Demineralized

(DM) water at 55-60°C, the sugar was dissolved completely 0.2g of sucralose and 1.0 g of *Bacillus coagulans* powder containing its spores was added to this solution. The mass was stirred to complete crystallization at 50-55°C. The product was dried at 40-45°C until moisture content reached to 0.14% the dried crystals weighing 97.2 g, further the product was sieved on 20 mesh sieve. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The sucralose content in the sample was 0.17 % and sugar content as expected. The spore count was 70million spores/g estimated by microbiological plate-count method. The total spores were retained. The product has 2 times sweetness, and had sharp taste.

Example No.55

Co-crystallized product of Sugar, Sucralose in large scale production using Mass-mixer as crystallizer

The co-crystallization process was carried out on large scale, the equipment used for crystallization was Mass Mixer having jacket and ribbon type blade. 150 kg of sugar dissolved in 60L of Demineralized (DM) water followed by 0.3 kg of sucralose and 3.6g of sodium acetate. The temperature kept at 65-70°C. In 1 hr the dissolution completed. The mass was filtered over nutsche filter. The clear solution was charged to mass mixer. The mass was kept under agitation at 60-65°C. After 3 hrs the mass was seeded with 150g of sugar. The seeding was done two more times after interval of 3hrs. The crystallization was completed as indicated by moisture content. The mass was centrifuged with RPM of 900. The mother liquor of 17.2 kg collected and analyzed for sucralose content and recycled in next batch. The crystallized product dried at 40-45°C till moisture content drops below 0.15 %. The product was sieved on 20, 40, 60, mesh sieves. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product

weighing 134.0 kg was sampled. The analysis shows sucralose content 0.19% by HPLC. Also product was subjected to taste and sweetness test by the panel. The product has double the sweetness of sugar and is sharp without any bitterness or metallic. Also the batch has uniformity in sweetness as well as Sucralose content as analysed by HPLC.

Example No.56

Co-crystallized product of Sugar, Sucralose in large scale production using Mass-mixer as crystallizer by recycling the mother liquor

The co-crystallization process was carried out on large scale, by recycling mother liquor from previous batch. The equipment used for crystallization was Mass Mixer having jacket and ribbon type blade. 150 kg of sugar dissolved in 60.0 L of Demineralized (DM) water followed by 0.3 kg of sucralose and 3.6 g of sodium acetate. The mother liquor of 17.2 kg from previous batch was also charged, the temperature kept at 65-70°C. The dissolution completed. The mass was filtered over nutsche filter. The clear solution was charged to mass mixer. The mass was kept under agitation at 60-65°C. After 3 hrs the mass was seeded with 150 g of sugar. The seeding was done two more times after interval of 3 hrs. The crystallization was completed as indicated by moisture content being around 5%.the moisture content reveals the water quantity present in the mass and completion of crystallization is determined. The mass was centrifuged with RPM of 900. The mother liquor of 16.5 kg collected and analyzed for sucralose content and recycled in next batch. The crystallized product dried at 40-45°C till moisture content drops below 0.15 %. The product was sieved on 20, 40, 60, mesh sieves. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The product weighing 147.9 kg showing yield of 98.6%. The product was sampled. The analysis

shows sucralose content 0.17% by HPLC. Also product was subjected to taste and sweetness test by the panel. The product has double the sweetness of sugar and is sharp without any bitterness or metallic. The batch has uniformity in sweetness as well as Sucralose content as analysed by HPLC.

Example No.57

Co-crystallized product of Sugar, Sucralose in large scale production using Vacuum drier as crystallizer

The co-crystallization process was carried out on large scale by using different equipment and without separation of mother liquor and without using stabilizer . The equipment used for crystallization was a vacuum drier having jacket. In this experiment, Rotocon Vacuum drier was used. 50 kg of sugar dissolved in 20.0 L of Demineralized (DM) water followed by 0.085 kg of sucralose The temperature kept at RT (room temperature). At the time of the experiment, room temperature was 26 °C. The dissolution completed. The clear solution was charged to ROTOCON VACUUM DRIER. The mass was concentrated under vacuum at 40-45°C. The seeding was not done. The crystallization was completed as indicated by moisture content. The mass was unloaded and dried at 40-45°C to get product of 49.5 kg having crystalline nature. The product was sieved on 20, 40, 60, mesh sieves. The product was crystalline in nature, non-dusty, with appearance like commercial cane sugar. The yield was 99%. The product was sampled. The analysis shows sucralose content 0.18% by HPLC. Also product was subjected to taste and sweetness test by the panel. The product has double the sweetness of sugar and is sharp without any bitterness or metallic. The batch has uniformity in sweetness as well as Sucralose content as analysed by HPLC.

Example 58

Experiments were conducted as per the procedure described in patent application publication no. US20070026121 for achieving co-crystallization.

In one experiment Erythritol is replaced with sugar and remaining other conditions are maintained as provided in one of the example given in the patent. The crystals are not formed at all in spite of keeping for several days. In another experiment erythritol is used as per patent example followed as per the example. It is observed that crystals are formed as mentioned. It is evident that example works for Erythritol and not for sugar.

Experiment details

A. WITH ERYTHRITOL

109 G of erythritol is dissolved in 105 ml of Demineralized (DM) water and 5.3 g of sucralose is dissolved. Temperature kept at 60°C dissolution is completed. The mass is allowed to attain 30°C

Observation; crystals developed after 12-14 hrs.

B. WITH SUGAR

109 g of sugar is dissolved in105 ml of water and 5.3 g of sucralose is dissolved. The temperature kept at 60° C till dissolution completed. The mass is allowed to attain 30° C.

Observation: No crystals developed even after several days

Conclusion: The method described in US20070026121 does not work for getting co-crystals of sugar with sucralose as it works for getting co-crystals of erythritol with sucralose.

Example 59

Co-crystallized product of other Sugars : Dextrose and Sucralose without separation of mother liquor

The co-crystallized product was prepared using dextrose and Sucralose. 99.7g of dextrose was dissolved in 40ml of RO water at 55-60°C. After complete dissolution 0.25g of Sucralose was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.11%, the dried product weighed 98g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The sucralose content was 0.23% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was two times than the Sugar. The taste profile was sharp and acceptable.

Example 60

Co-crystallized product of other Sugars : Lactose and Sucralose without separation of mother liquor

The co-crystallized product was prepared using lactose and Sucralose. 99.6g of lactose was dissolved in 200ml of RO water at 55-60°C. After complete dissolution 0.34g of Sucralose was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.20%, the dried product weighed 98.5g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The sucralose content was

0.33% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was almost two times than the Sugar. The taste was not sharp and no bitter taste observed.

Example 61

Co-crystallized product of other Sugars : Maltose and Sucralose without separation of mother liquor

The co-crystallized product was prepared using Maltose and Sucralose. 99.6g of Maltose was dissolved in 70ml of RO water at 55-60°C. After complete dissolution 0.33g of Sucralose was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.10%, the dried product weighed 99g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The sucralose content was 0.32%by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was almost two times than the Sugar. The taste was not sharp and no bitter taste observed.

Example 62

Co-crystallized product of other Sugars : Fructose and Sucralose without separation of mother liquor

The co-crystallized product was prepared using Fructose and Sucralose. 99.6g of Fructose was dissolved in 30ml of RO water at 55-60°C. After complete dissolution 0.4g of Sucralose was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.15%, the dried product weighed 98.5g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The sucralose content was

0.39% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was four times than the Sugar. The taste profile was acceptable.

Example 63

Co-crystallized product of other Sugars and other high intensity sweeteners : Maltose and Acesulfame-K without separation of mother liquor

The co-crystallized product was prepared using Maltose and Acesulfame-K. 99g of maltose was dissolved in 55ml of RO water at 55-60°C. After complete dissolution 1g of Acesulfame-K was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.20%, the dried product weighed 98.5g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The Acesulfame-K content was 0.92% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was two times than the Sugar. The taste profile was different from the product made from Sugar and Sucralose.

Example 64

Co-crystallized product of other Sugars and other high intensity sweeteners : Dextrose and Acesulfame-K without separation of mother liquor

The co-crystallized product was prepared using Dextose and Acesulfame-K. 99g of dextrose was dissolved in 40ml of RO water at 55-60°C. After complete dissolution 0.75g of Acesulfame-K was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.15%, the dried product weighed 99g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The Acesulfame-K

content was 0.74% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was nearly two times than the Sugar. The taste was not sharp.

Example 65

Co-crystallized product of other Sugars and other high intensity sweeteners : Lactose and Acesulfame-K without separation of mother liquor

The co-crystallized product was prepared using Lactose and Acesulfame-K. 99g of lactose was dissolved in 270ml of RO water at 55-60°C. After complete dissolution 1.02g of Acesulfame-K was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.20%, the dried product weighed 99.5g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The Acesulfame-K content was 0.98% by HPLC. The product was crystalline, however, appearence of the product was not like cane sugar. The sweetness was two times than the Sugar. The taste was not sharp.

Example 66

Co-crystallized product of Sugar and other high intensity sweeteners : Sucrose and Acesulfame-K without separation of mother liquor

The co-crystallized product was prepared using Sucrose and Acesulfame-K. 99g of Sucrose was dissolved in 40ml of RO water at 55-60°C. After complete dissolution 0.6g of Acesulfame-K was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.20%, the dried product weighed 99g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The Acesulfame-K

content was 0.59% by HPLC. The nature of the product was crystalline and appearance was like cane sugar. The sweetness was nearly two times than the Sugar. No bitter taste and is sharp in taste.

Example 67

Co-crystallized product of Sugar and other high intensity sweeteners : Sucrose and Aspartame without separation of mother liquor

The co-crystallized product was prepared using Sucrose and Aspartame. 99g of Sucrose was dissolved in 40ml of RO water at 55-60°C. After complete dissolution 0.8g of Aspartame was added and dissolved. The solution was filtered and allowed for crystallization at 55-60°C. The crystallized product was dried at 45-50°C until moisture content reached 0.18%, the dried product weighed 99.5g. The small lumps were broken and sieved 20 mesh (841 micron) sieve. The Aspartame content was 0.79% by HPLC. The nature of the product was crystalline and cane sugar like in appearance. The sweetness was almost two times than the Sugar. The taste is acceptable, sharp and no bitter taste observed.

CLAIMS

1. A method of making a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion, comprising steps of:

   a. obtaining a concentrated solution of sucrose and a high intensity sweetener with or without a stabilizer and with or without other optional ingredients accompanied by heating to a temperature, the resulting concentrated solution containing mixture of sucrose and the high intensity sweetener not containing impurities in a quantity that would result into formation of molasses or molasses like by-products after the step of co-crystallization during further steps of the process, the final concentration of the solution of sucrose, the high intensity sweetener and other optional ingredients being at least enough for getting co-crystallization of the dissolved sucrose with the high intensity sweetener by stirring of the mixture of sucrose, the high intensity sweetener, with or without the other additional ingredients, for a period of time accompanied with heating to a temperature where caramelization will not occur, until co-crystallization is complete, the stirring being done, with or without seeding,

   b. drying the moist co-crystallized mass with or without the separation of mother liquor at a temperature that promotes as much less residual moisture content as possible in the dried sweetener composition without caramelization to get a solid mass comprising free flowing crystals or comprising lumps of dried crystals, ,optionally, breaking

lumps if required, anc ıg the dry crystalline solids to get a sweetener composition ıasses through desired mesh size.

2. The method of claim 1 wherein

a. The said concentrated solution of sucrose and a high intensity sweetener, with or without other optional ingredients is obtained by:

i. adding one or more of a high intensity sweetener, with or without other optional ingredients, to a concentrated solution of sucrose prior to co-crystallization, or

ii. adding sucrose with or without other optional ingredients to concentrated solution of a high intensity sweetener prior to co-crystallization, or

iii. adding one or more of a high intensity sweetener, with or without additional optional ingredients, to a concentrated solution made from process stream of regular sugar manufacturing process after removal of molasses from the crystallized sucrose, or

iv. adding sucrose to a concentrated solution of process stream, containing a high intensity sweetener in purified state produced in a process of manufacture of the high intensity sweetener, the said concentrated solution being free from organic solvent/s to such an extent that residual solvent will not interfere in the co-crystallization process.

b. the said obtained concentrated solution of sucrose, high intensity sweetener with or without other ingredients is obtained at a temperature and heated, avoiding caramelizaiton, to a highe temperature, more particularly at a temperature of 80°C to 45°C, or at 50 to 70°C, or at 60°C,

c. the said final concentration of the solution of sucrose and the high intensity sweetener being 250% dissolved solids or more,

d. the said stirring of mixture of sucrose and the high intensity sweetener with or without additional optional ingredients is done at 20-40 revolutions per minute,

e. the said stirring of mixture of sucrose and the high intensity sweetener with or without additional optional ingredients is stirred at a temperature of 80-40°C,

f. the said stirring of the mixture of sucrose and the high intensity sweetener is done at 30 revolutions per minute,

g. the said period of time of stirring of the mixture of sucrose and the high intensity sweetener, with or without the other additional ingredients, accompanied with heating is 3 to 32 hours, more particularly 16-32 hours.

h. the temperature of drying of co-crystallized moist mass being between 40-45°C,

i. the said residual moisture of the dried mass of co-crystal mass being 0.1 – 0.4 %.

3. A method of claim 1 wherein:

   a. the high intensity sweetener comprises at least one selected from the group sucralose, Glycyrrihizin, Thaumatin, Monellin, Stevioside extract, Rebaudiside-A extract, Lo Han Guo Morgrosides extract, Brazzein, Curculin, pentadin, Mabinlin, Acesulfame K, Neotame, Talin, Citrose, Alitame, Cyclamate, Saccharin and Aspartame.

   b. the said aqueous solvent being water,

   c. wherein the said additional ingredients comprise one or more selected from a group consisting of an amino acid or derivative of an amino acid, a vitamin, a mineral, a flavor, an enhancer, or a prebiotic, or a probiotic, a Pharmaceutically active ingredient, an anti-oxidant, an energy booster, a derivative of a fat or an oil, a color and other natural products.

4. A method of claim 3 wherein:

   a. the said amino acid comprise one or more selected from the group consisting of Taurine, L-Arginine, L-Ornithine, L-Lysine, L-Carnitine, L-Methionine, L-Phenylalanine, L-Tyrosine, L-Cysteine, L-Glycine and S-Adenosyl methionine,

   b. the said vitamin comprises one or more selected from the group consisting of Vitamin-A, Vitamin-B1, Vitamin-B2, Vitamin-B3, Vitamin-B5, Vitamin-B6, Vitamin-B12, Vitamin-B complex, Beta-carotine, Vitamin-C, Vitamin-D, Vitamiin-D1, Vitamiin-D2, Vitamin-E, Biotine, Choline, Folic acid,

c. the said mineral comprises one or more selected from the group consisting of Selenium, Zinc, Boron, Calcium, Chromium, Iron, Magnesium and Potassium,

d. the said flavor comprises one or more selected from the group consisting of a Coca powder, Coffee, Vanillin, Pista, Strawberry, Mango, Orange, Chocolate, Dry Fruit, Dry vegetables, Ice-cream, Yogurt, Wheat flour, Multigrain flour, Peppermint, Ginger, Apple, Citrus, Grape, Cherry, Ginseng, Peach, Wild berry, Tropical, pomegranate and Blue berry,

e. the said pre-biotic comprises one or more selected from the group consisting Inulin and other fructooligosaccharides, Xylo-oligosaccharides, Gentio-oligoscaccharides, Galacto-oligosaccharides, Nigero-oligosaccharides, Malto-oligosaccharides, Soybean oligosaccharides,

f. the said probiotic comprises one or more selected from the group consisting a Yeast, Bacillus, Lactobacillus, Bifidobacterium bifidum, Bifidobacterium infantis, Bifidobacterium longum, Enterococcus faecium and Streptococcus thermophilus,

g. the said pharmaceutically effective ingredient comprises one or more selected from the group consisting of a Bromelain, Chitosan, Salicin, Inosin, Myoglobin, Glucomannan, Guaranine, Yerbamate, Pectin, Pancreatin, Pantothenic, Spirulina, ATP, Conjugated linoleic acid, Hydroxy citric acid, Phaseolamin, 5-HTP, Adenosine receptor, caffeine, Theobromine, Theophylline, SCH58261, KW6002, ZM241385, GABA and Dehydro epiandrosteron,

h. the said antioxidant comprises one or more selected from the group consisting of Alpha lipoic acid, Bilberry, CoQ10, Ginkgobioba, Glutathione, Grape seed extract, Green tea extracts, malatonine, Oligomeric proanthoscyanidins, Pycnogenol, Reseveritrol, Astaxanthin and Ergothineine,

i. the said energy booster comprises one or more selected from the group consisting of a Gotukola, Saint John's Wort, Wheet grass, Fennel, Kelp, Alfalfa, Red clover, Adenosine A (2A) and Common oats,

j. the said fat or oil or their derivatives includes, without limitation, one or more of a Butter, Olive oil, Canola oil, Vegetable oil, Flax seed oil, Black currant seed oil, Primrose oil, Fish oil, Omega 3,6,9 poly unsaturated fatty acids, Milk, Condensed milk, Cheese, Nuts and Docosahexaenoic acid,

k. the said color comprises one or more selected from a group consisting of a Curcumin, Lycopene, Beta-carotene, Apocarotenal and Canthaxanthin.

l. the said other natural products comprises one or more selected from a group consisting of a Licorice root, Catnip, Passion flower, Lobelia, Hops, Skullcap, Gentian, Myrrh, Safflower, Bayberry root bark, Eucalyptus, Sarsaparilla, Slippery Elm, Valerian root, Ephedra, Guarana and kola nut.

5. A method of claim 1 of making a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-

dusty and with uniform sweetness in each 6 gram portion having pre-determined precise pre-defined target of "Sucrose Equivalent Sweetness", abbreviated as SES, wherein mother liquor is separated from the co-crystallized mass before drying in step c. claim 1, the said method comprising following steps:

a. performing a certain number of pilot experiments required statistically to find out what quantity of the high intensity sweetener is lost in mother liquor wherein a quantity of high intensity sweetener expected to give targeted sweetness is added in step a. of claim 1 to determine overages that need to be added to compensate for this loss,

b. dissolving the high intensity sweetener in step a. in a quantity with proper overages decided above that shall give the desired sweetness in the co-crystallized dried mass,

c. preparing more than two batches as required within the statistical requirement for statistical uniformity, evaluating for actual sweetness or the content of the high intensity sweetener in the co-crystallized product,

d. combining batches with lower and higher than targeted sweetness in such a way that desired sweetness is achieved in the uniformly mixed product.

6. The method of claim 5 wherein the pre-determined precise pre-defined target of desired SES is 1.15 times to 100 times, more particularly 2 times to ten times, still more particularly 2 times to six times, still more particularly 2 times to six times, still more particularly 2 times to four times.

7. The method of claim 6 wherein the target of desired SES is 2 times or four times.

8. The method of claim 1 of making a sucrose equivalent sweetness enhanced sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion having pre-determined target of "Sucrose Equivalent Sweetness" abbreviated as SES, when mother liquor is not separated from the drying of the co-crystallized mass in step c. of claim 1, comprising steps of

    a. dissolving the high intensity sweetener in step a. of claim 1 in a quantity that is calculated to exactly give the desired sweetness in the co-crystallized mass, and

    b. drying the co-crystallized mass without separating the mother liquor.

9. A sucrose equivalent sweetness enhanced sweetener containing at least sucrose and at least one a high intensity sweetener with cane sugar like crystalline appearance, free flowing, non-dusty and with uniform sweetness in each 6 gram portion having defined "Sucrose Equivalent Sweetness", abbreviated as SES, that is precisely an integer or a whole number without a fraction or any other pre-defined SES.

10. The sweetener composition of claim 10 wherein the SES comprises 2 times to 100 times, more particularly 2 times to 10 times, still more particularly 2 times to 6 times, still more particularly 2 times to 6 times, still more particularly 2 times to 4 times.

11. The sweetener composition of claim 11 having SES of 2 times or 4 times.

12. A method of claim 1 of making a sucrose equivalent sweetness enhanced sweetener wherein the high intensity sweetener is sucralose.

13. A sucrose equivalent sweetness enhanced sweetenerof claim 9 wherein the high intensity sweetener is sucralose.

FIG. 1

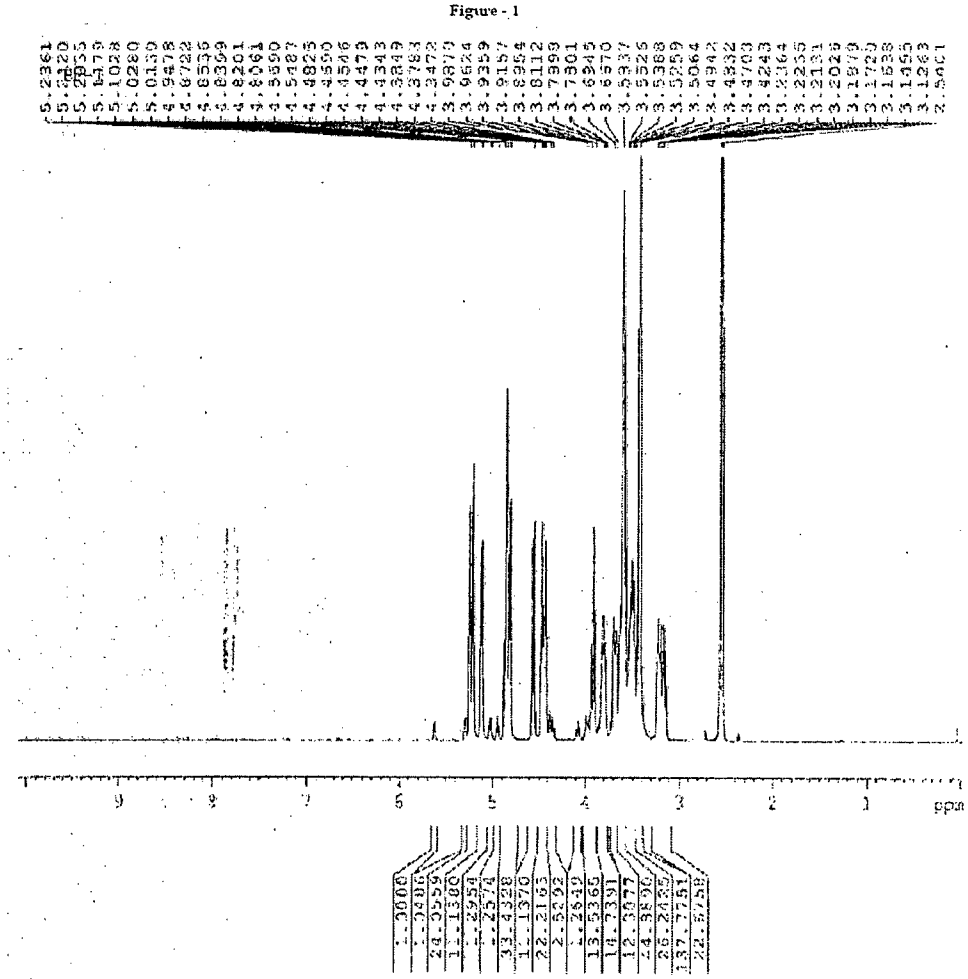
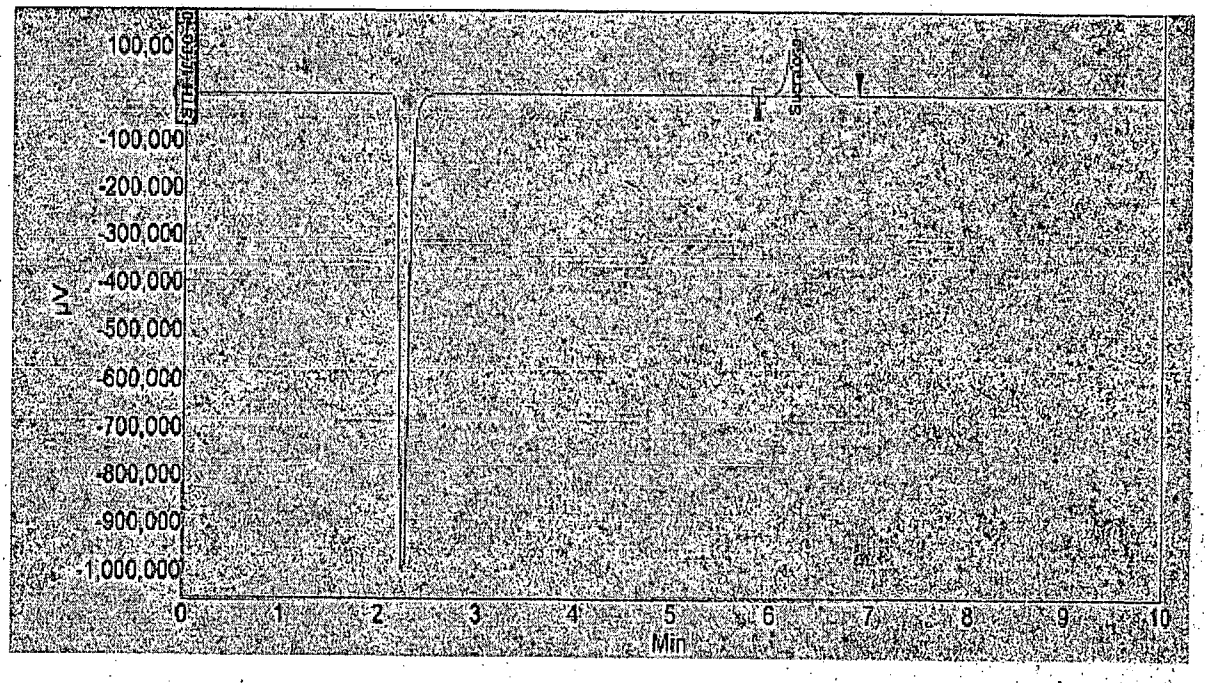


FIG 2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2
3
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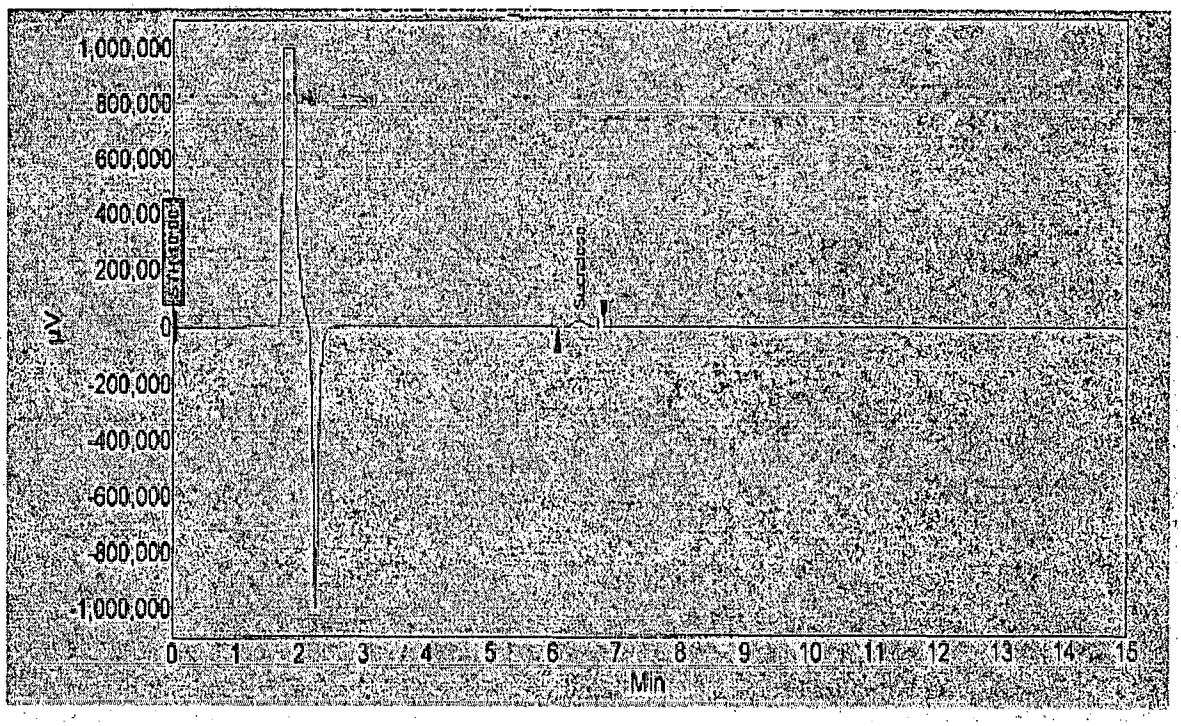
5
6
7
8
9
10
Min

FIG 3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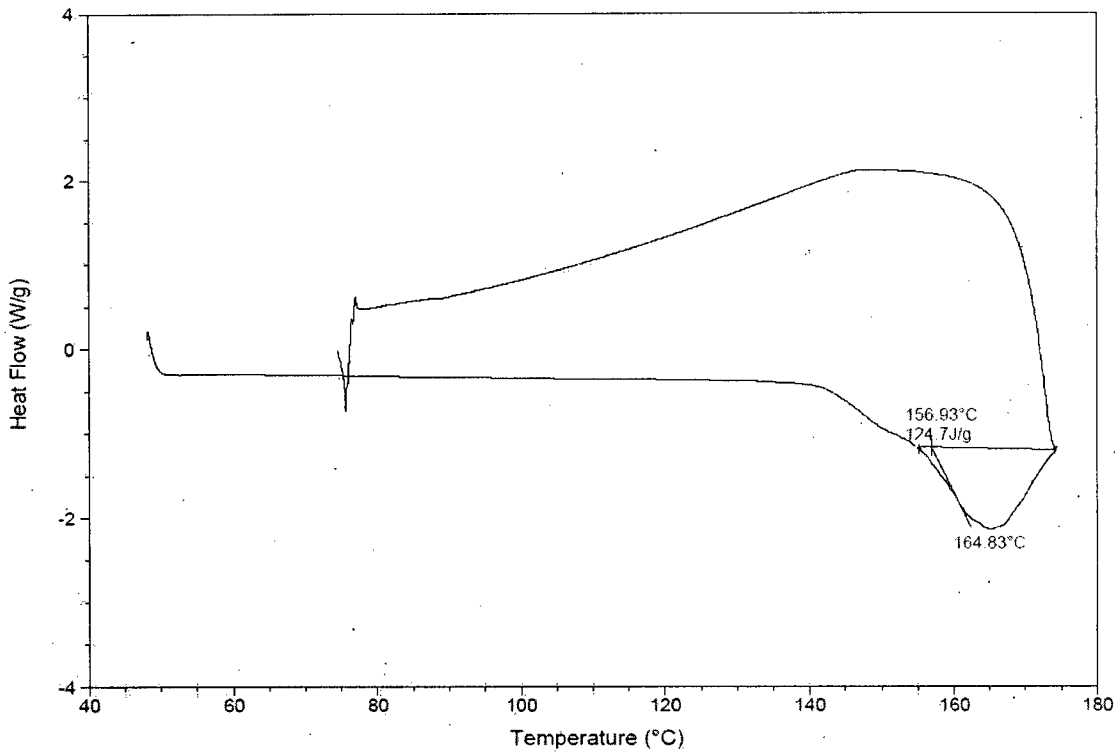
µV
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
Min

FIG 4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Temperature (°C)
156.93°C
124.7J/g
164.83°C
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-2
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60
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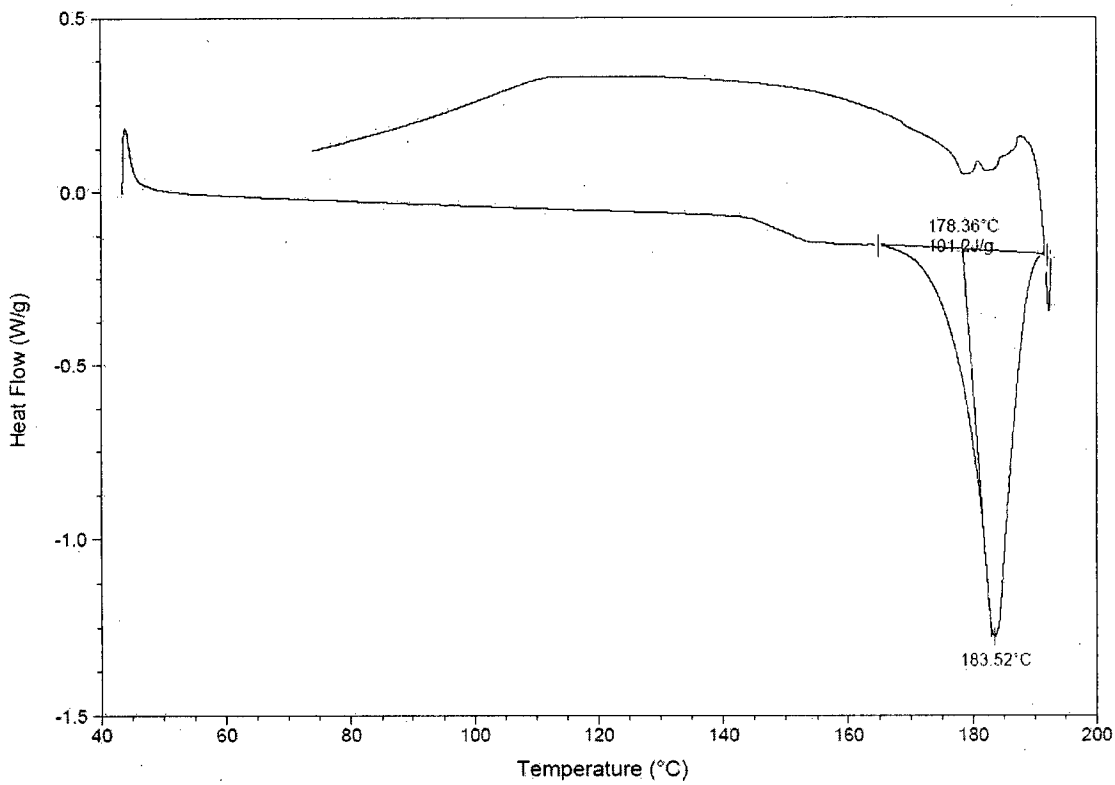
100
120
140
160
180

FIG 5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Heat Flow (W/g)
178.36°C
101.2 J/g
183.52°C
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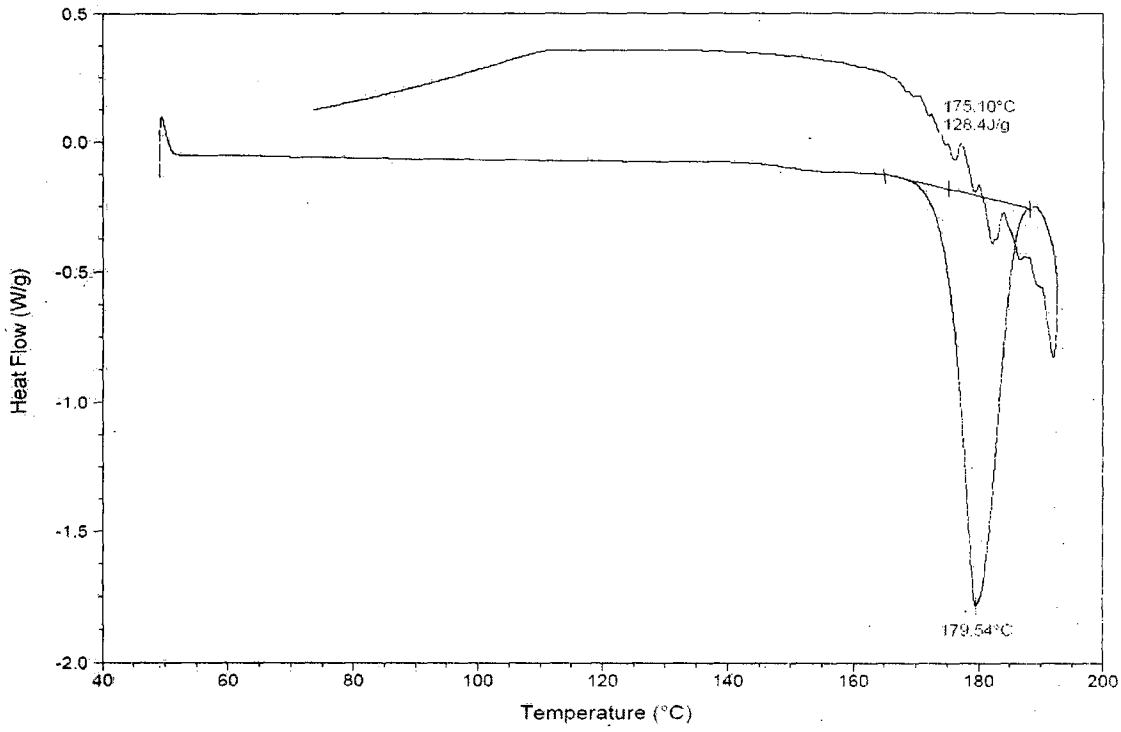
60
80
100
120
140
160
180
200
Temperature (°C)

FIG 6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Heat Flow (W/g)
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60
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140
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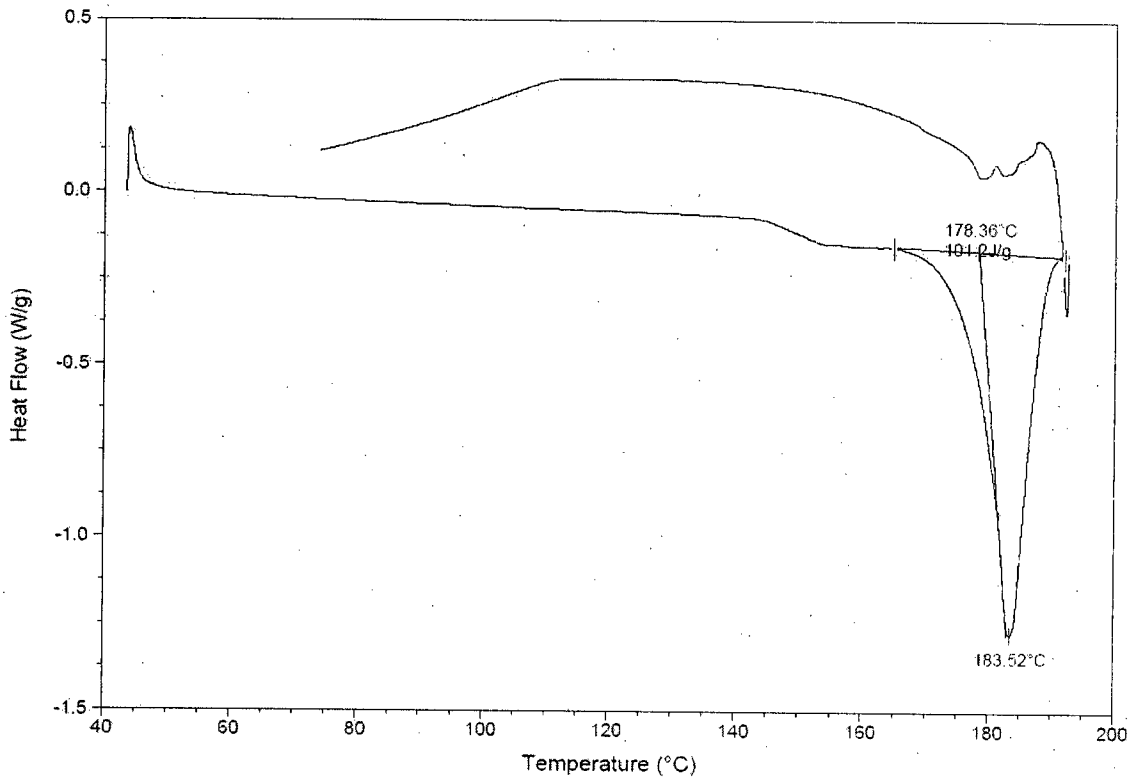
180
200
Temperature (°C)
175.10°C
128.4J/g
179.54°C

FIG 7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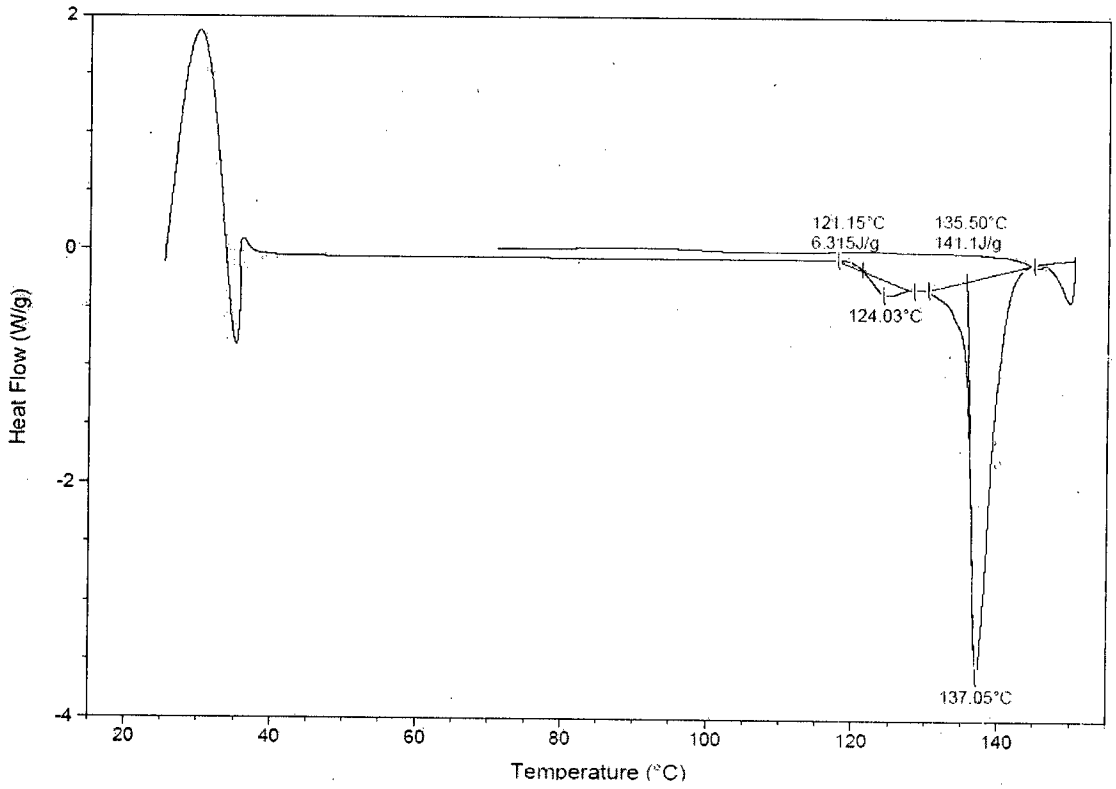
180
200
Temperature (°C)

FIG 8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Heat Flow (W/g)
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40
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100
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Temperature (°C)
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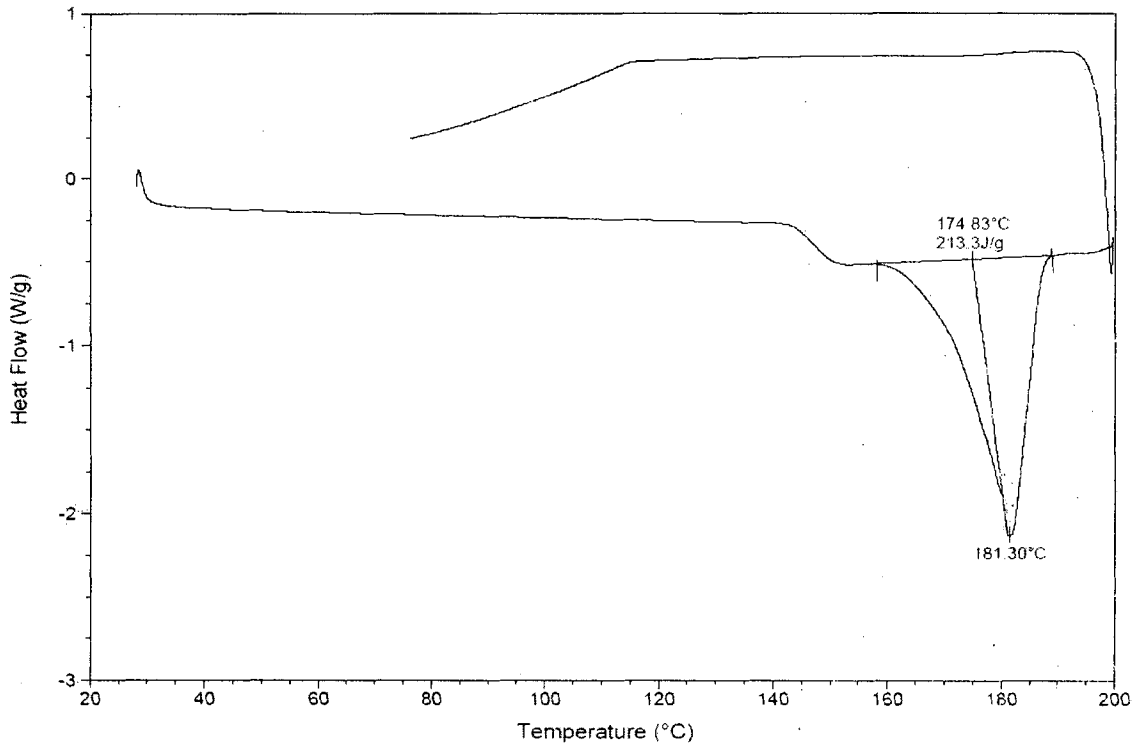
6.315J/g
135.50°C
141.1J/g
124.03°C
137.05°C

FIG 9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Heat Flow (W/g)
20
40
60
80
100
120
140
160
180
200
Temperature (°C)
174.83°C
213.3J/g
181.30°C

FIG 10

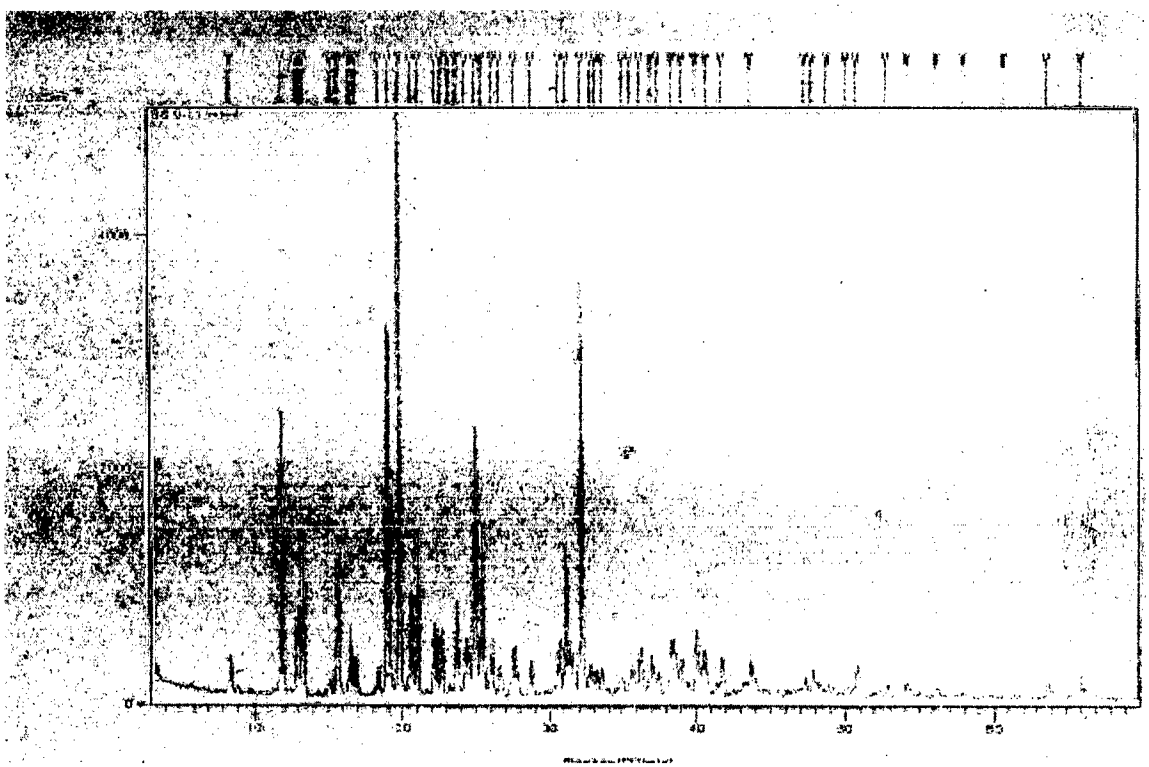
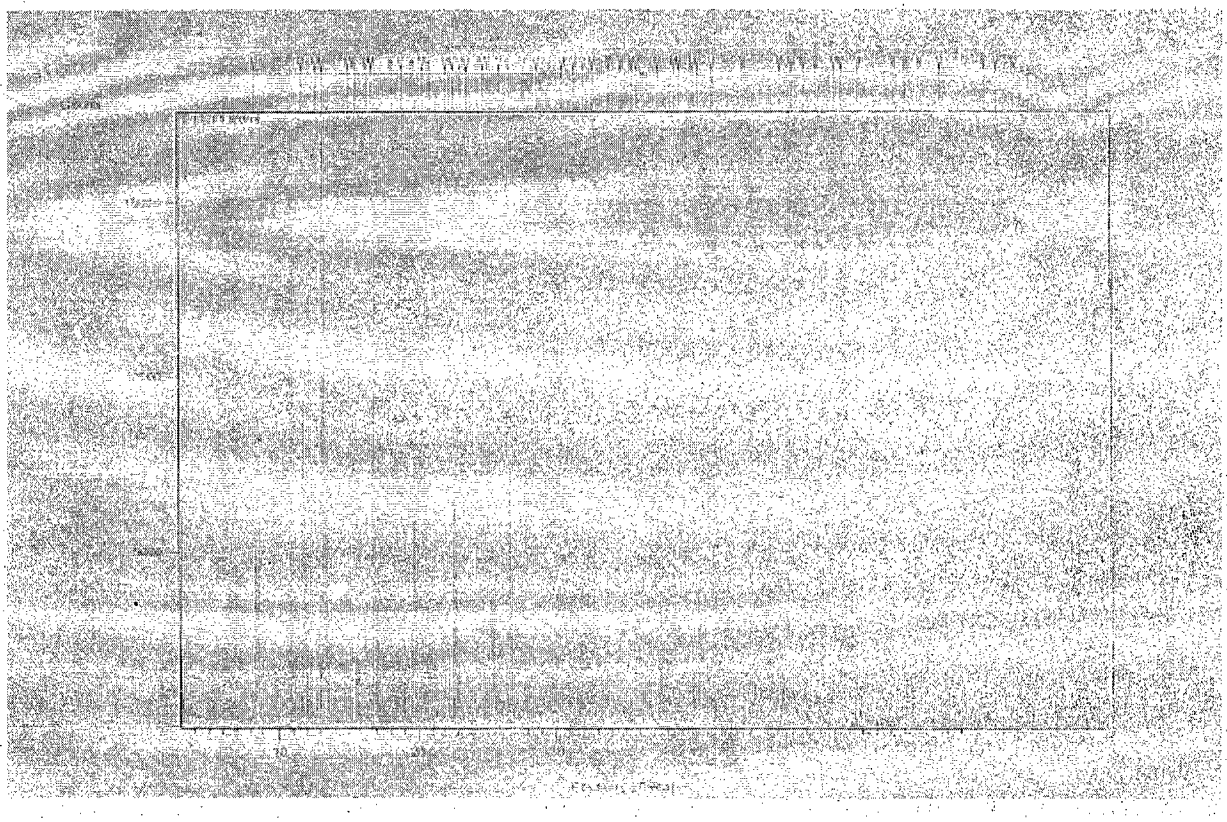


FIG 11



INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2012/001064

A. CLASSIFICATION OF SUBJECT MATTER

A23L 1/236 (2006.01) A23L 1/09 (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

STN CAS Online: CAPLUS, FSTA - Keywords (sucrose, sucralose, high intensity sweetener, crystal) and like terms
EPOQUENET: EPODOC, WPI - Keywords (sucrose, sucralose, high intensity sweetener, crystal) and like terms, IPC/ECLA: A23L/-, A23G/-

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Documents are listed in the continuation of Box C	

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
12 October 2012	12 October 2012
Name and mailing address of the ISA/AU	**Authorised officer**
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Email address: pct@ipaustralia.gov.au Facsimile No.: +61 2 6283 7999	Jocelyn Chan AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No. 0262832914

INTERNATIONAL SEARCH REPORT		International application No.
C (Continuation).	DOCUMENTS CONSIDERED TO BE RELEVANT	**PCT/IB2012/001064**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2008/0014331 A1 (BADALOV) 17 January 2008 abstract; paragraphs [0020], [0027], [0029], [0030], [0034], [0043], [0061]-[0068], tables 1-3	1-13
X	US 6214402 B1 (FOTOS et al.) 10 April 2001 abstract; column 3, lines 11-63; examples; column 5, lines 44-53	9-11, 13
X	CN 101933568 A (HEBEI TBL SCIENCE AND TECHNOLOGY CO LTD) 05 January 2011 abstract; page 2; paragraphs [0037]-[0040], [0090]; examples at pages 5-7	1-4, 9-13
X	US 2010/0034945 A1 (ARANGO MORENO) 11 February 2010 abstract; paragraphs [0045], [0051]; claim 1; fig. 2	1-4, 9-13
A	WO 2006/115680 A1 (MCNEIL NUTRITIONALS, LLC) 02 November 2006 abstract; page 7, paragraph [0029]; examples	1-13
P,X	US 2011/0195169 A1 (ALKEM LABORATORIES LTD.) 11 August 2011 examples 16-25; paragraph [0069]	1-4, 9-11

INTERNATIONAL SEARCH REPORT Information on patent family members	International application No. PCT/IB2012/001064

This Annex lists known patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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		US 2010112154 A1	06 May 2010

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

INTERNATIONAL SEARCH REPORT Information on patent family members	International application No. **PCT/IB2012/001064**

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Patent Document/s Cited in Search Report		**Patent Family Member/s**	
Publication Number	**Publication Date**	**Publication Number**	**Publication Date**
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		US 2010112156 A1	06 May 2010
		US 2010112158 A1	06 May 2010
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		WO 2011059954 A1	19 May 2011

End of Annex

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.