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Industria y Comercio
SUPERINTENDENCIA

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
No. 06-019254- -00008-0000
Fecha: 2007-11-28 16:19:52 Dep: 2020 NUEVA CIUDAD
Tra: 2 PATENTES Eve: 1 RECDEPOSITO
Act: 400 OPOSICIONOBSERVLR Folios: 83

FORMULARIO ÚNICO DE OPOSICION

AS: 12.793

1. OPOSICION A:

- ☒ Patente de Invención.
- ☐ Patente de Modelo de Utilidad.
- ☐ Diseño Industrial.
- ☐ Esquema de Trazado para Circuitos Integrados.
- ☐ Marcas de Productos o Servicios.
- ☐ Marca Colectiva.
- ☐ Marca de Certificación.
- ☐ Lema Comercial.

2. SOLICITUD PUBLICADA

Número del Expediente: 06 019.254

Solicitante: CENTRAL CASTILLA S.A.
INTERCONTINENTAL SUGAR USA, LLC

Apoderado: DARIO CARDENAS NAVAS
Título de la nueva creación o denominación del signo: AZÚCAR ENTERA

Gaceta: 579

3. OPOSICION PRESENTADA POR:

SOLICITANTE	Nombre: MANUELITA S.A. Dirección: Km. 7 Via Palmira, Cerrito, Buga, Valle, Colombia Nacionalidad o Domicilio: Colombia Lugar de Constitución: Colombia Teléfono: Fax: E-mail:	**IDENTIFICACION** C.C. ☐ NIT ☐ C.E. ☐ Otro ☐ Cuál _____ Número: _____
REPRESENTANTE O APODERADO	Nombre: **JUAN PABLO CADENA SARMIENTO** Dirección: Carrera 7 No. 71-21, Torre B, Piso 4, Bogotá, Colombia Teléfono: 7442200 Fax: 7422200 E-mail: bricas@brigardycastro.com.co	**IDENTIFICACION** C.C. ☒ NIT ☐ C.E. ☐ Otro ☐ Cuál_____ No.: 80410337 Usaquén TP 57492

4. FUNDAMENTOS DE LA OPOSICION:

Causal: Incumplimiento de los requisitos de patentabilidad (Título II, Capítulo I, de la Decisión 486 de 2000). Se violan los Artículos 14 (las patentes de invención debe ser nuevas, tener nivel inventivo y ser susceptibles de aplicación industrial), 16 (una invención se considera nueva cuando no esta comprendida en el estado de la técnica), y 18 (una invención tiene nivel inventivo si no es obvia para una persona versada en la materia).

Justificación: Ver Anexo

5. Comprobante de pago No. 07- 83,066 **Fecha** 28-11-2007

6. ANEXOS

- ☒ Comprobante de pago de la tasa.
- ☒ Poderes, si fuere el caso.
- ☒ Pruebas de los fundamentos de la oposición.
- ☐ Documentos que acrediten el legítimo interés, si fuere el caso.
- ☒ Documento que acredita la existencia y representación legal de las partes, cuando sean personas jurídicas.

23 IV 86

7.

NOMBRE: JUAN PABLO CADENA SARMIENTO

FIRMA: _____

C.C. 80.410.337 de Usaquén TP 57492 del C.S.J.

Instrucciones para el diligenciamiento del presente formulario, ver reverso de esta página.

AS. 12.973

Señores
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISION DE SIGNOS DISTINTIVOS
E. S. D.

REF.:	EXPEDIENTE No. 06 019.254
TÍTULO SOLICITADO:	"AZÚCAR ENTERA"
PUBLICACIÓN:	Gaceta 579 de 31 de agosto de 2007, número de publicación 177.
SOLICITANTE:	CENTRAL CASTILLA S.A. y INTERCONTINENTAL SUGAR USA, LLC
PRIORIDAD:	US 60/741.148 del 01/12/2005
APODERADO:	DARIO CARDENAS NAVAS
TRÁMITE:	PRESENTACIÓN DE OPOSICIÓN

JUAN PABLO CADENA SARMIENTO, mayor de edad y vecino de esta ciudad, identificado como aparece al pie de mi firma, obrando como apoderado de MANUELITA S.A., con domicilio en Buga, Valle, Colombia, con legítimo interés solicito dentro de la oportunidad legal para el efecto concedida, y de acuerdo con dispuesto por las Articulos 42 y siguientes de la Decision 486, dar curso a la siguiente:

1. PETICION

Negar el beneficio de patente a la solicitud que reposa en el expediente No. 06 019.254, por no cumplir con los requisitos de patentabilidad a que se refieren los artículos 14, 16 y 18 de la Decisión 486, con base a los fundamentos y las consideraciones de hecho que exponen a continuación.

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2. LEGITIMO INTERES

Asistente a MANUELITA S.A., en su condición de industria de producción de azúcar, legítimo interés para presentar esta oposición, pues la eventual concesión de la solicitud restringiría injustificadamente el empleo de las técnicas comúnmente utilizadas para la obtención de cristales de sacarosa en detrimento de sus legítimos intereses comerciales y de la libre competencia.

3. FUNDAMENTOS DE LA OPOSICIÓN

3.1 La solicitud en referencia divulga un método para la obtención de azúcar entera que contiene vitaminas y minerales. Dicho método consiste en calentar un jarabe que contiene sacarosa para producir una masacocida, agregar a dicho jarabe al menos otro producto del paso de purga, evaporación o molienda y cristalizar el azúcar entera a partir de la masacocida. Dicho proceso carece tanto de novedad como de altura inventiva, como se verá a continuación de acuerdo con los siguientes argumentos.

- La anterioridad US 2 217 604 (en adelante D1) del 8 de octubre de 1940 enseña un proceso de refinación de azúcar que se lleva a cabo a bajas temperaturas y presión atmosférica. Dicha anterioridad enseña la recirculación de las masas que salen de las etapas posteriores del proceso así como también el uso de semillas para promover la formación de cristales, ver página 3, columna 5, línea 56 a columna 6, línea 32.

- La anterioridad CA 258 715 (en adelante D2) del 7 de agosto de 1956 divulga que el procedimiento convencional para la obtención de azúcar a partir de la caña o la remolacha involucra etapas repetidas de concentración y cristalización. Es claro, entonces, que el proceso de cristalización sugerido por la presente solicitud ya era conocido en el estado del arte.

- La anterioridad CA 724 614 (en adelante D3) del 28 de diciembre de 1965 enseña un método de formación de cristales de azúcar que

comprende la formación de las masas o massecuite, la incorporación de semillas y la subsiguiente supersaturación por cocción. La masa cocida es luego enfriada para promover la cristalización y llevada a centrífugas para separar los cristales de los materiales de desecho.

- La anterioridad US 4 164 429 (en adelante D4) del 13 de diciembre de 1977 describe, en la columna 1, líneas 15 a 48, un proceso de cristalización similar al divulgado por la presente solicitud, que comprende las etapas de formación de las masas cocidas, incorporación de un material semilla para la cristalización, supersaturación de las masas cocidas por calentamiento, y separación de los cristales por centrifugación.

- La anterioridad GB 2 206 293 (en adelante D5) del 22 de junio de 1987 enseña un método para la fabricación de azúcar por cristalización por concentración (supersaturación) al vacío de la solución de azúcar y posterior cristalización por enfriamiento.

- Por último, la solicitud misma, en las páginas 19 y 20 divulga que los métodos de cristalización empleados son convencionales.

De acuerdo con lo anterior, es claro que la solicitud carece tanto de novedad como de altura inventiva puesto que las etapas del proceso de formación de cristales de sacarosa ya eran ampliamente conocidos por el técnico con habilidad ordinaria en la materia desde los años 40's.

3.2 Aun si a modo de argumento se aceptara que ninguna de las anterioridades enseña o sugiere la incorporación de nutrientes como vitaminas y minerales, es claro para el técnico con habilidad ordinaria en la materia que la solicitud en cuestión no esta adicionando ningún reactivo externo al proceso. Como se puede apreciar en la página 1 de la solicitud el inventor dice que una fuente apropiada de vitaminas y minerales es un producto derivado del ciclo de purga, evaporación o molienda.

Para el técnico con habilidad ordinaria en la materia es común la recirculación de corrientes o el uso de corrientes provenientes de otras etapas del proceso para reducir los costos y mejorar la eficiencia. Por

ejemplo, el técnico con habilidad en la materia sabe del uso de los jugos provenientes de la clarificación para diluir las melazas resultantes del proceso de centrifugación con el fin de recircularlas a la etapa de evaporación, esto para maximizar la cantidad de azúcar que se extrae del proceso.

3.3 Por último, el solicitante sugiere la adición de vitaminas al azúcar entera, pero dicha característica no se menciona en el capítulo reivindicatorio, las tablas o el método tal como se describe. Cabe anotar que aunque se incorporara tal característica a las reivindicaciones ésta no restauraría la novedad o la altura inventiva de un proceso que, como lo demuestran las anterioridades D1 a D5, es ampliamente conocido en el estado de la técnica.

Por otro lado la adición de minerales y vitaminas al azúcar cristalizado es una imposibilidad técnica. En primer lugar el proceso de supersaturación se lleva a cabo a temperaturas que destruirían las vitaminas, y el proceso de cristalización separaría los minerales de los cristales de azúcar y los mismos serían arrastrados con las melazas en el proceso de centrifugación. Es obvio, entonces, el porque el solicitante no incluye esta caracteristica en las reivindicaciones.

Adicionalmente, la solicitud no representa más que un intento por parte del solicitante de patentar un método de cristalización conocido argumentando que se le han agregado nutrientes al azúcar cuando, en realidad, todo lo que el solicitante hizo fue caer en cuenta de la presencia de vitaminas y minerales en otras corrientes del proceso de fabricación de azúcar.

Según el solicitante las vitaminas y minerales provienen de otras corrientes como los jugos clarificados, las melazas clarificadas o las masacocidas provenientes de otras etapas del proceso de fabricación de azúcar. Es claro, entonces, que dicha "adicion de vitaminas y minerales" no es tal, y el método de la solicitud no es diferente de cualquiera de los métodos de las anterioridades, o de los conocimientos del técnico con habilidad ordinaria en la materia.

Por lo tanto ni la dicha adición de minerales y vitaminas, ni el proceso de cristalización son novedosos o inventivos, y el que el arte previo no enseñe o

sugiera la presencia de dichas vitaminas y minerales no es un argumento que automáticamente le restituya la novedad a un proceso ampliamente conocido y utilizado en el estado del arte.

4. FUNDAMENTOS DE DERECHO

Fundamento el presente escrito en el Capítulo I, los Artículos 14, 16, 18, 20, y el Artículo 42 (Oposiciones) de la Decisión 486 de 2000 de la Comisión de la Comunidad Andina de Naciones.

4. PRUEBAS

Ruego que se tengan como tales, sin perjuicio de que el Despacho considere oficiosamente, en particular de las allegadas por el suscrito a los expedientes mencionados en este escrito, las siguientes:

- Patente de Estados Unidos No. 2 217 604 titulada "MANUFACTURE OF CANE SUGAR", página 3, columna 5, línea 56 a columna 6, línea 32.

- Patente del Canadá No. 528 715 titulada "SUGAR CRYSTALLIZATION", página 2, líneas 13 a 23.

- Patente del Canadá No. 724 614 titulada "CRYSTALLIZATION OF SUGAR", página 1, líneas 8 a 32.

- Patente de Estados Unidos No. 4 164 429 titulada "PROCESS AND INSTALLATION FOR THE PRODUCTION OF SELLECTED CRYSTALLIZATION SEEDS FOR USE IN A SUGAR REFINERY", columna 1, líneas 28 a 58.

- Patente del Reino Unido No. 2 206 293 titulada "MANUFACTURE OF SUGAR BY CRYSTALLIZATION BY COOLING", páginas 5 a 16, reivindicaciones, resumen y figuras.

5. ANEXOS

Para efectos del trámite correspondiente, me permito anexar al presente escrito los siguientes anexos:

5.1. Certificado de existencia y representación legal de MANUELITA, S.A.
5.2. Poder para actuar.
5.3. Recibo de pago No.07 – 83.066
5.4. Copia del presente escrito para el traslado al solicitante de la patente de la referencia.
5.5. Las mencionadas en el acápite de pruebas.

6. NOTIFICACIONES

El suscrito recibirá notificaciones en la secretaría de su despacho o en mi oficina localizada en Carrera 7 No. 71-21, Torre B – Piso 4, de esta ciudad.

Señor Superintendente,

JUAN PABLO CADENA SARMIENTO
C.C. No. 80.410.337 de Usaquén
Cra 7 No. 71-21 TORRE B – Piso 4

Bogotá, 30 de Noviembre de 2007
MEV/CH/ame

As.12973

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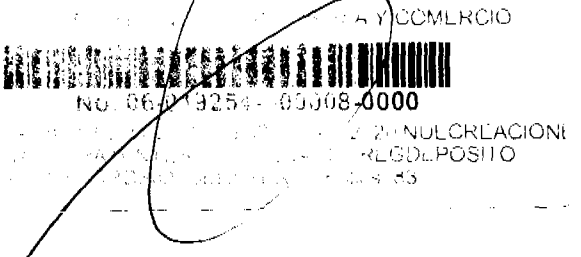
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 07 - 83,066
FECHA : OCTUBRE 9 DE 2007



***** C O N S I G N A C I O N *****

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PGO	Vr. PAGO
BRIGARD & CASTRO LTDA	CONSIGNACION	BANCO POPULAR	050-00110-6	49377419	09/10/2007	20,470,000.00

***** C O N C E P T O *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005-01-04 PRESENTACION DE OBSERVACIONES	12 MARCAS Y LEMAS COMERCIALES	256,000.00
		TOTAL : $	256,000.00

SON: DOSCIENTOS CINCUENTA Y SEIS MIL PESOS

RESPONSABLE : _____

RECIBO DE CAJA APLICADO AL EXPEDIENTE No. _____

Manuelita S.A.

Nit.: 891.300.241-9

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SEÑORA JEFE
DIVISION DE NUEVAS CREACIONES
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
E. S. D.

El abajo firmante, mayor de edad, vecino de esta ciudad, identificado como aparece al pie de mi firma, en mi condición de Representante Legal de la sociedad **MANUELITA S.A.**, con domicilio en Palmira, Valle, Colombia, por el presente confiero poder especial, amplio y suficiente, a los abogados **JUAN PABLO CADENA SARMIENTO** y/o **CARLOS HUMBERTO PALACIO E.** y/o **MARTIN EDUARDO TORRES C.**, para que en nombre de la sociedad que represento, adelanten todas las gestiones administrativas y judiciales de oposición al registro de la patente titulada **AZUCAR ENTERA**, solicitada por **CENTRAL CASTILLA S.A., INTERCONTINENTAL SUGAR USA, LLC.**, cuyo trámite se adelanta en el Expediente No. 06 019.254.

Los apoderados quedan ampliamente facultados para solicitar, desistir, transigir, pagar impuestos, presentar documentos, notificarse, interponer recursos, sustituir, reasumir y en general hacer todo cuanto fuere necesario para el fiel cumplimiento de este mandato.

Atentamente,

ALVARO F. GALEANO M.
C.C. No. 14.957.534 de Cali
MANUELITA S.A.

ACEPTO:

JUAN PABLO CADENA SARMIENTO
C.C. No. 80.410.337 de Usaquén
T.P. No. 57.492 del C.S.J.

En Palmira a 27 NOV. 2007 de 200 Ante el Despacho de la Notaría Tercera de este Círculo, se presentó,

Alvaro Francisco Gaitano

Mayor (s) de edad, con cédula (s) 14957134

y dijo (eron) que el contenido del documento es cierto y que la (s) firma (s) puesta (s) al pie del mismo (s) es (son) de su puño y letra y la (s) misma (s) usa (n) y acostumbra (n) en todos sus actos.

NOTARIO TERCERO

JORGE ENRIQUE NURI MOTTA
NOTARIO TERCERO
PALMIRA VALLE

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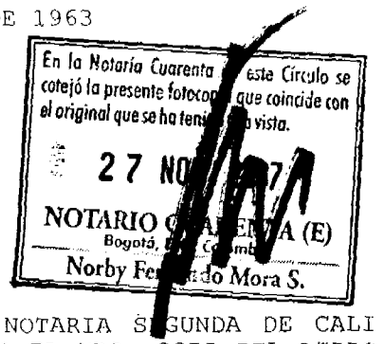
REPUBLICA DE COLOMBIA

CERTIFICADO DE EXISTENCIA Y REPRESENTACION

EL SUSCRITO SECRETARIO DE LA CAMARA DE COMERCIO DE PALMIRA

CERTIFICA

NOMBRE:MANUELITA S.A.
DOMICILIO: PALMIRA VALLE
DIRECCION COMERCIAL :-KM 7 VIA PALMIRA - EL CERRITO
DIRECCION NOTIFICACION JUDICIAL:-KM 7 VIA PALMIRA - EL CERRITO
CIUDAD:PALMIRA
MATRICULA MERCANTIL NRO. 606-4 FECHA MATRICULA : 23 DE JUNIO DE 1963
DIRECCION ELECTRONICA : j.murillo@manuelita.com
DIRECCION WEB : www.manuelita.com
AFILIADO



CERTIFICA

NIT : 891300241-9

CERTIFICA

QUE POR ESCRITURA NRO. 2247 DEL 25 DE NOVIEMBRE DE 1947 NOTARIA SEGUNDA DE CALI INSCRITA EN LA CAMARA DE COMERCIO EL 24 DE JUNIO DE 1963 BAJO EL NRO. 3275 DEL LIBRO IX ,SE CONSTITUYO LA SOCIEDAD DENOMINADA MANUELITA S.A.

CERTIFICA

QUE POR ESCRITURA NRO. 2819 DEL 30 DE ABRIL DE 1963 NOTARIA PRIMERA DE CALI ,INSCRITA EN LA CAMARA DE COMERCIO EL 24 DE JUNIO DE 1963 BAJO EL NRO. 3281 DEL LIBRO IX ,LA SOCIEDAD CAMBIO SU DOMICILIO DE CALI A PALMIRA .

CERTIFICA

QUE POR ESCRITURA NRO. 0841 DEL 17 DE MARZO DE 2005 NOTARIA TERCERA DE PALMIRA ,INSCRITA EN LA CAMARA DE COMERCIO EL 30 DE MARZO DE 2005 BAJO EL NRO. 3675 DEL LIBRO IX ,SE APROBO LA ESCISION PARCIAL DEL PATRIMONIO SOCIAL ENTRE (ESCINDENTE) MANUELITA S.A. Y (BENEFICIARIA(S)) ACEITES MANUELITA S.A., ACUACULTURA MANUELITA S.A., INVERSIONES LA RITA S.A. .

CERTIFICA

QUE POR ESCRITURA NRO. 4146 DEL 24 DE NOVIEMBRE DE 2006 NOTARIA TERCERA DE PALMIRA ,INSCRITA EN LA CAMARA DE COMERCIO EL 29 DE NOVIEMBRE DE 2006 BAJO EL NRO. 987 DEL LIBRO IX ,SE APROBO LA ESCISION PARCIAL DEL PATRIMONIO SOCIAL ENTRE (ESCINDENTE) MANUELITA S.A. Y (BENEFICIARIA(S)) MANUELITA INTERNACIONAL S.A. .

CERTIFICA

REFORMAS

DOCUMENTO	FECHA.DOC	ORIGEN	FECHA.INS	NRO.INS	LIBRO
E.P. 362	06/02/1953	NOTARIA SEGUNDA DE CALI	24/06/1963	3276	
E.P. 592	23/02/1953	NOTARIA SEGUNDA DE CALI	24/06/1963	3277	
E.P. 2264	18/06/1956	NOTARIA PRIMERA DE CALI	24/06/1963	3278	
E.P. 1702	14/05/1957	NOTARIA PRIMERA DE CALI	24/06/1963	3279	
E.P. 2044	03/05/1960	NOTARIA PRIMERA DE CALI	24/06/1963	3280	
E.P. 2280	18/10/1965	NOTARIA PRIMERA DE PALMIRA	20/10/1965	3941	
E.P. 852	11/05/1970	NOTARIA PRIMERA DE PALMIRA	16/05/1970	5257	
E.P. 587	30/03/1974	NOTARIA PRIMERA DE PALMIRA	02/04/1974	992	IX
E.P. 1029	30/07/1980	NOTARIA PRIMERA DE PALMIRA	01/08/1980	4476	IX
E.P. 695	29/05/1981	NOTARIA PRIMERA DE PALMIRA	08/06/1981	4986	IX

E.P. 1160	19/05/1988	NOTARIA PRIMERA DE PALMIRA	01/06/1988	8735	IX
E.P. 2810	08/11/1989	NOTARIA PRIMERA DE PALMIRA	09/11/1989	9556	IX
E.P. 3453	01/11/1991	NOTARIA TERCERA DE PALMIRA	13/11/1991	11548	IX
E.P. 1075	31/03/1993	NOTARIA TERCERA DE PALMIRA	07/04/1993	12841	IX
E.P. 1693	19/05/1993	NOTARIA TERCERA DE PALMIRA	02/07/1993	13033	IX
E.P. 4182	26/11/1993	NOTARIA TERCERA DE PALMIRA	26/11/1993	13306	IX
E.P. 2392	15/08/1996	NOTARIA TERCERA DE PALMIRA	22/08/1996	560	IX
E.P. 3191	30/09/1997	NOTARIA TERCERA DE PALMIRA	03/10/1997	1500	IX
E.P. 1317	07/05/2003	NOTARIA TERCERA DE PALMIRA	16/05/2003	1884	IX
E.P. 948	31/03/2005	NOTARIA TERCERA DE PALMIRA	04/04/2005	3709	IX
E.P. 3349	12/10/2005	NOTARIA TERCERA DE PALMIRA	28/10/2005	4421	IX
E.P. 3092	17/08/2007	NOTARIA TERCERA DE PALMIRA	22/08/2007	810	IX

CERTIFICA

VIGENCIA: 30 DE MARZO DEL AÑO 2073

CERTIFICA

LA SOCIEDAD TENDRA COMO OBJETO LAS SIGUIENTES ACTIVIDADES PRINCIPALES: 1) LA EXPLOTACION DE LA INDUSTRIAL DEL DULCE Y DE LOS NEGOCIOS RELACIONADOS CON LOS SECTORES DE LA AGROINDUSTRIA, GANADERO, PISCICOLA, ALIMENTICIO, METALMECANICO, QUIMICO, FARMACEUTICO, VETERINARIO, FINANCIERO, BANCARIO, CREDITICIO, TURISTICO, MINERO, SIDERURGICO, DE LAS OLEAGINOSAS, LA FLORICULTURA, LA FRUTICULTURA, LA HORTICULTURA, LOS PLASTICOS, LOS COMBUSTIBLES Y EL GAS. 2) LA EXPLOTACION DEL NEGOCIO DE LA CONSTRUCCION EN TODOS SUS ASPECTOS, LO MISMO QUE EL NEGOCIO DE LA ADMINISTRACION DE PUERTOS MARITIMOS O TERRESTRES DE SERVICIO PUBLICO, EL ARRENDAMIENTO DE MAQUINARIA AGRICOLA Y EL NEGOCIO DEL MONTAJE, PRODUCCION, DISTRIBUCION Y PRESTACION DE LOS SERVICIOS DE ENERGIA, AGUA, ALCANTARILLADO, TELECOMUNICACIONES, RADIO, TELEVISION, EDUCACION, SALUD, INFORMATICA, PUBLICIDAD, TRANSPORTE DE CARGA Y DE PASAJEROS Y RECOLECCION Y RECICLAJE DE BASURAS. 3) LA COMERCIALIZACION, IMPORTACION, EXPORTACION, DISTRIBUCION Y VENTA AL POR MAYOR Y AL DETAL DE PRODUCTOS PROPIOS O DE TERCEROS, ENTRE LOS CUALES SE ENCUENTRAN PERO SIN LIMITARSE A ESTOS. ALIMIENTOS, DETERGENTES, BEBIDAS ALCOHOLICAS, PASTAS, ACEITES. 4) LA PRODUCCION, TRANSFORMACION, IMPORTACION, EXPORTACION, COMERCIALIZACION Y DISTRIBUCION DE ALCOHOL CARBURANTE. 5) LA INVERSION O APLICACION DE RECURSOS O DISPONIBILIDADES A EMPRESAS EXISTENTES O A NUEVAS EMPRESA QUE SE CONSTITUYAN BAJO CUALQUIERA DE LAS FORMAS AUTORIZADAS POR LA LEY, SEAN NACIONALES O EXTRANJERAS Y QUE TENGAN POR OBJETO LA EXPLOTACION DE CUALQUIER ACTIVIDAD ECONOMICA LICITA. 6) ASESORAR A OTRAS EMPRESAS MEDIANTE LA PRESTACION DE SERVICIOS EN ASPECTOS TALES COMO: DESARROLLO DE CULTIVOS, DIRECCION, PLANIFICACION, DESARROLLO, FINANZAS, ADMINISTRACION, INVERSIONES, TESORERIA, CREDITO, SISTEMAS, CARTERA, MERCADEO Y EXPORTACIONES, VENTAS, SERVICIOS DE REPRESENTACION, AGENCIA, INTERMEDIACION COMERCIAL Y OTROS SIMILARES A LOS ANTERIORES, ADQUIRIR, DESARROLLAR Y GESTIONAR ESTABLECIMIENTOS COMERCIALES. 7) LA ADQUISICION, POSESION DE: PATENTES, NOMBRES COMERCIALES, MARCAS, SECRETOS INDUSTRIALES, REGISTROS SANITARIOS, LICENCIAS U OTROS DERECHOS CONSTITUTIVOS DE PROPIEDAD INDUSTRIAL, LA CONCESION DE SU EXPLOTACION A TERCEROS, ASI COMO LA ADQUISICION DE CONCESION PARA SU EXPLOTACION. 8) LA PROMOCION DE INVESTIGACIONES CIENTIFICAS O TECNOLOGICAS TENDIENTES A BUSCAR NUEVAS Y MEJORES APLICACIONES DENTRO DE SU OBJETO SOCIAL YA SEA DIRECTAMENTE O A TRAVES DE ENTIDADES ESPECIALIZADAS O DE DONACIONES A ENTIDADES CIENTIFICAS O CULTURALES O DE DESARROLLO AGRICOLA O DESARROLLO SOCIAL DEL PAIS. 9) OTORGAR GARANTIAS REALES O PERSONALES EN RESPALDO DE OBLIGACIONES PROPIAS Y/O DE COMPANIAS SUBORDINADAS O DE TERCEROS, PREVIA AUTORIZACION DE LA JUNTA DIRECTIVA.

PARAGRAFO: EN DESARROLLO DE ESTAS ACTIVIDADES PODRAN LOS REPRESENTANTES LEGALES DE LA SOCIEDAD DENTRO DE LOS PARAMETROS ESTABLECIDOS EN LOS ESTATUTOS EFECTUAR TODOS LOS ACTOS CONTRATOS, GESTIONES Y DILIGENCIAS QUE CONSIDEREN NECESARIOS O CONVENIENTES PARA EL EJERCICIO O CUMPLIMIENTO DE TODAS LAS MENCIONADAS ACTIVIDADES Y/O NEGOCIOS QUE COMPRENDEN EL OBJETO SOCIAL COMO POR EJEMPLO CULTIVAR CANA, PRODUCIR AZUCAR, MIEL, ALCOHOL Y DEMAS SUBPRODUCTOS, CELEBRAR CONTRATOS DE MUTUO, DONACIONES, HIPOTECAS, PRENDA, DE ASOCIACION PARA LA EXPLOTACION DE TIERRAS PROPIAS Y AJENAS, ESTABLECER AGROINDUSTRIAS PARA PROCESAR PRODUCTOS PROPIOS Y/O DE TERCEROS, CONSTRUIR OBRAS, PARCELACIONES, URBANIZACIONES, VIAS PUBLICAS QUE PRODUZCAN PEAJES, ETC, ADQUIRIR, EXPLOTRAR, ARRENDAR, GRAVAR Y ENAJENAR BIENES DE CUALQUIER CLASE, COMERCIALIZAR Y/O

MERCADEAR DIRECTAMENTE O MEDIANTE REPRESENTACION, AGENCIAS O SUCURSALES, OBTENER CONCESIONES Y PRIVILEGIOS DE CUALQUIER NATURALEZA, PATENTES DE INVENCION Y MARCAS DE PROPIEDAD INDUSTRIAL, CREAR O ESTABLECVER NEGOCIOS CON SUS RESPECTIVAS INSTALACIONES Y/O ADQUIRIR ACCIONES O DERECHOS SOCIALES Y/O PARTICIPAR EN LA CONSTITUCION DE OTRAS SOCIEDADES CUYO OBJETIVO COMPRENDA EN CADA CASO ALGUNA DE LAS ACTIVIDADES O NEGOCIOS RELACIONADOS EN ESTE ARTICULO ETC.

CERTIFICA

PRESIDENCIA: LA ASAMBLEA GENERAL DE ACCIONISTAS SERA PRESIDIDA POR EL PRESIDENTE DE LA JUNTA DIRECTIVA, A FALTA DE ESTE POR UNO DE LOS MIEMBROS PRINCIPALES O SUPLENTES DE LA JUNTA DIRECTIVA EN SU ORDEN, Y EN ULTIMO CASO, POR EL ACCIONISTA QUE DESIGNE LA ASAMBLEA.

LA JUNTA DIRECTIVA SE COMPONE DE CINCO (5) MIEMBROS PRINCIPALES QUIENES TENDRAN SUPLENTES NUMERICOS.

CORRESPONDE A LA JUNTA DIRECTIVA AUTORIZAR AL GERENTE PARA COMPRAR, VENDER O GRAVAR BIENES INMUEBLES INDEPENDIENTEMENTE DE SU CUANTIA Y PARA CELEBRAR LOS CONTRATOS RELACIONADOS CON EL OBJETO SOCIAL CUYOS VALORES EXCEDAN DE MIL DOSCIENTOS (1.200) SALARIOS MINIMOS MENSUALES LEGALES VIGENTES.

ATRIBUCIONES. LA JUNTA DIRECTIVA TENDRA ATRIBUCIONES SUFICIENTES PARA ORDENAR QUE SE EJECUTE O CELEBRE CUALQUIER ACTO O CONTRATO COMPRENDIDO DENTRO DEL OBJETO SOCIAL Y PARA TOMAR LAS DETERMINACIONES NECESARIAS EN ORDEN A QUE LA SOCIEDAD CUMPLA SUS FINES.

LA SOCIEDAD TENDRA UN GERENTE, QUE PODRA SER O NO MIEMBRO DE LA JUNTA DIRECTIVA Y HASTA TRES (3) SUPLENTES QUE LO REEMPLAZARAN EN EL ORDEN EN QUE SEAN DESIGNADOS, EN SUS FALTAS ACCIDENTALES, TEMPORALES O ABSOLUTAS.

PARAGRAFO: LA SOCIEDAD TENDRA TAMBIEN UN EMPLEADO DENOMINADO REPRESENTANTE LEGAL ESPECIAL, CUYA FUNCION EXCLUSIVA SERA LA REPRESENTACION LEGAL DE LA SOCIEDAD EN LOS PROCESOS LABORALES, CIVILES, COMERCIALES, ADMINISTRATIVOS, PENALES, CONTENCIOSO ADMINISTRATIVOS, DE JURISDICCION COACTIVA, ADUANEROS, FISCALES, DE POLICIA. REPRESENTARA IGUALMENTE A LA SOCIEDAD EN LAS AUDIENCIAS DESTINADAS A ABSOLVER INTERROGATORIOS DE PARTE. EL REPRESENTANTE LEGAL ESPECIAL TENDRA FACULTADES PARA CONCILIAR, DESISTIR Y HACER TODO CUANTO DERECHO SEA NECESARIO PARA LA DEFENSA DE LOS INTERESES DE LA SOCIEDAD. ESTE REPRESENTANTE LEGAL ESPECIAL TENDRA UN SUPLENTE ELEGIDO IGUALMENTE POR LA JUNTA DIRECTIVA PARA REEMPLAZARLO EN SUS FALTAS ABSOLUTAS, TEMPORALES, OCASIONALES O ACCIDENTALES. EL GERENTE Y SUS OTROS TRES (3) SUPLENTES PODRAN EJERCER EN CUALQUIER MOMENTO LAS FUNCIONES ATRIBUIDAS AL REPRESENTANTE LEGAL ESPECIAL Y A SU SUPLENTE.

REPRESENTACION LEGAL: EL GERENTE, O QUIEN HAGA SUS VECES, ES EL REPRESENTANTE LEGAL DE LA SOCIEDAD PARA TODOS LOS EFECTOS.

FUNCIONES: EL GERENTE EJERCERA TODAS LAS FUNCIONES PROPIAS DE LA NATURALEZA DE SU CARGO, Y EN ESPECIAL, LAS SIGUIENTES: 1. REPRESENTAR A LA SOCIEDAD ANTE LOS ACCIONISTAS, ANTE TERCEROS Y ANTE TODA CLASE DE AUTORIDADES, DE ORDEN ADMINISTRATIVO Y JURISDICCIONAL. 2. EJECUTAR TODOS LOS ACTOS U OPERACIONES CORRESPONDIENTES AL OBJETO SOCIAL, DE CONFORMIDAD CON LO PREVISTO EN LAS LEYES Y EN LOS ESTATUTOS Y QUE NO EXCEDAN EL VALOR DE MIL DOSCIENTOS (1.200) SALARIOS MINIMOS MENSUALES LEGALES VIGENTES A LA FECHA DE LA CELEBRACION DEL RESPECTIVO ACTO O CONTRATO. 3. PREVIA AUTORIZACION DE LA JUNTA DIRECTIVA, COMPRAR, VENDER O GRAVAR BIENES INMUEBLES INDEPENDIENTEMENTE DE SU CUANTIA Y CELEBRAR LOS CONTRATOS RELACIONADOS CON EL OBJETO SOCIAL CUYOS VALORES EXCEDAN DE MIL DOSCIENTOS (1.200) SALARIOS MINIMOS MENSUALES LEGALES VIGENTES. 4. AUTORIZAR CON SU FIRMA TODOS LOS DOCUMENTOS PUBLICOS O PRIVADOS QUE DEBAN OTORGARSE EN DESARROLLO DE LAS ACTIVIDADES SOCIALES O EN INTERES DE LA SOCIEDAD. 5. PRESENTAR A LA ASAMBLEA GENERAL EN SUS REUNIONES ORDINARIAS, UN INVENTARIO Y UN BALANCE DE FIN DE EJERCICIO, JUNTO CON UN INFORME ESCRITO SOBRE LA SITUACION DE LA SOCIEDAD, UN DETALLE COMPLETO DE LA CUENTA DE PERDIDAS Y GANANCIAS Y UN PROYECTO DE DISTRIBUCION DE UTILIDADES OBTENIDAS. ADICIONALMENTE, EL INFORME DEL GERENTE DEBERA CONTENER LOS NIVELES DE EXPOSICION DE LA SOCIEDAD A LOS DIFERENTES RIESGOS, TALES COMO, SIN LIMITARSE A ELLOS, LIQUIDEZ, ENDEUDAMIENTO, TASA DE CAMBIO. 6. NOMBRAR Y REMOVER LOS EMPLEADOS DE LA SOCIEDAD CUYO NOMBRAMIENTO Y REMOCION LE DELEGUE LA JUNTA DIRECTIVA. 7.

TOMAR TODAS LAS MEDIDAS QUE RECLAME LA CONSERVACION DE LOS BIENES SOCIALES, VIGILAR LA ACTIVIDAD DE LOS EMPLEADOS DE LA ADMINISTRACION DE LA SOCIEDAD E IMPARTIRLES LAS ORDENES E INSTRUCCIONES QUE EXIJA LA BUENA MARCHA DE LA COMPANIA. 8. CONVOCAR A LA ASAMBLEA GENERAL DE ACCIONISTAS A REUNIONES EXTRAORDINARIAS CUANDO LO JUZGUE CONVENIENTE O NECESARIO Y HACER LAS CONVOCATORIAS DEL CASO CUANDO LO ORDENEN LOS ESTATUTOS, LA JUNTA DIRECTIVA O EL REVISOR FISCAL DE LA SOCIEDAD. 9. CONVOCAR A LA JUNTA DIRECTIVA CUANDO LO CONSIDERE NECESARIO O CONVENIENTE Y MANTENERLA INFORMADA DEL CURSO DE LOS NEGOCIOS SOCIALES. 10. CUMPLIR LAS ORDENES E INSTRUCCIONES QUE LE IMPARTAN LA ASAMBLEA GENERAL DE ACCIONISTAS O LA JUNTA DIRECTIVA, Y, EN PARTICULAR, SOLICITAR AUTORIZACIONES PARA LOS NEGOCIOS QUE DEBEN APROBAR PREVIAMENTE LA ASAMBLEA GENERAL DE ACCIONISTAS O LA JUNTA DIRECTIVA, SEGÚN LO DISPONGAN LAS NORMAS CORRESPONDIENTES O LOS ESTATUTOS. 11. CUMPLIR O HACER QUE SE CUMPLA OPORTUNAMENTE TODOS LOS REQUISITOS O EXIGENCIAS LEGALES QUE SE RELACIONEN CON EL FUNCIONAMIENTO Y ACTIVIDADES DE LA SOCIEDAD. 12. SUMINISTRAR AL MERCADO PUBLICO DE VALORES Y A LOS INVERSIONISTAS EN GENERAL, EN LOS EVENTOS EN QUE ASI LO EXIJAN LAS NORMAS VIGENTES EN SU MOMENTO, TODA LA INFORMACION VERAZ, COMPLETA Y OPORTUNA ACERCA DE LA SOCIEDAD, SIEMPRE Y CUANDO DICHA INFORMACION NO SEA INFORMACION PRIVILEGIADA Y/O SE TRATE DE DOCUMENTOS QUE VERSEN SOBRE SECRETOS INDUSTRIALES Y/O DATOS QUE DE SER DIVULGADOS PODRIAN SER UTILIZADOS EN DETRIMENTO DE LA SOCIEDAD. 13. VELAR POR EL FIEL CUMPLIMIENTO DE LAS DISPOSICIONES CONTENIDAS EN LOS ESTATUTOS Y EN LOS ACUERDOS DE ACCIONISTAS QUE HAYAN SIDO DEBIDAMENTE REGISTRADAS ANTE EL SECRETARIO DE LA SOCIEDAD. 14. DAR A CONOCER A LOS INVERSIONISTAS Y AL PUBLICO EN GENERAL, MEDIANTE UN MEDIO DE COMUNICACIÓN QUE CONSIDERE IDONEO, EL CODIGO DEL BUEN GOBIERNO DE LA SOCIEDAD APROBADO POR LA JUNTA DIRECTIVA, ASI COMO LAS MODIFICACIONES Y/O ADICIONES QUE SE LE HAGAN AL MISMO. 15 NOMBRAR MANDATARIOS PARA ASUNTOS JUDICIALES O EXTRAJUDICIALES DIFERENTES A LOS QUE CORRESPONDAN A LOS REPRESENTANTES LEGALES ESPECIALES.

CERTIFICA

DOCUMENTO: ACTA No. 959 DEL 26 DE MARZO DE 2007
ORIGEN: JUNTA DIRECTIVA
INSCRIPCION: 29 DE MARZO DE 2007 No. 299 DEL LIBRO IX

FUE(RON) NOMBRADO(S):

GERENTE
ADOLFO LEON VELEZ VELEZ
C.C.8280548

PRIMER SUPLENTE
CARLOS ARTURO GUEVARA
C.C.16450888

SEGUNDO SUPLENTE
ALVARO FRANCISCO GALEANO MONTOYA
C.C.14957534

REPRESENTANTE LEGAL ESPECIAL SUPLENTE
ALVARO JAIR FERNANDEZ NOGUERA
C.C.94416781

REPRESENTANTE LEGAL ESPECIAL
NORMA CONSTANZA ARBELAEZ HERRERA
C.C.31991544

En la Notaría Cuarenta de este Círculo se cotejó la presente fotocopia que coincide con el original que se ha tenido a la vista.
27 NOV. 2007
NOTARIO CUARENTA (E)
Bogotá, D.C. Colombia
Norby Fernando Mo...

CERTIFICA

DOCUMENTO: ACTA No. 115 DEL 15 DE MARZO DE 2007
ORIGEN: ASAMBLEA GENERAL
INSCRIPCION: 20 DE ABRIL DE 2007 No. 401 DEL LIBRO IX

FUE(RON) NOMBRADO(S)

JUNTA DIRECTIVA

PRINCIPALES

PRIMER RENGLON
CESAR ZAMORANO ESTRADA.
C.C.6087401

SEGUNDO RENGLON
ARMANDO GARCES EDER.
C.C.6089759

TERCER RENGLON
SANDRA GIOVANELLI EDER.
C.C.31284750

CUARTO RENGLON
JUAN ANTONIO ZAMBRANO EDER.
C.C.16624526

QUINTO RENGLON
ALVARO GONZALEZ PRIETO.
C.C.14934154

SUPLENTES

PRIMER RENGLON
HAROLD ENRIQUE EDER GARCES.
C.C.79376787

SEGUNDO RENGLON
ALBERTO JOSE OTOYA D.
C.C.16258277

TERCER RENGLON
CARLOS ARTURO GUEVARA.
C.C.16450888

CUARTO RENGLON
RAMIRO MARINO.
C.C.19081313

QUINTO RENGLON
NORMA CONSTANZA ARBELAEZ HERRERA.
C.C.31991544

En la Notaría Cuarenta de este Círculo se cotejó la presente fotocopia que coincide con el original que se ha tenido a la vista.
27 AGO 2007
NOTARIA CUARENTA (E)
Bogotá, D.C., Colombia
Norby Fernando Mora S.

CERTIFICA

DOCUMENTO: ACTA No. 878 DEL 28 DE MARZO DE 2000
ORIGEN: JUNTA DIRECTIVA
INSCRIPCION: 03 DE MAYO DE 2000 No. 293 DEL LIBRO IX

FUE(RON) NOMBRADO(S):

CONTRALOR
ARTURO SUDUPE JIMENEZ
C.C.14434820

CERTIFICA

DOCUMENTO: ACTA No. 112 DEL 07 DE MARZO DE 2006
ORIGEN: ASAMBLEA GENERAL DE ACCIONISTAS
INSCRIPCION: 10 DE MARZO DE 2006 No. 179 DEL LIBRO IX

FUE(RON) NOMBRADO(S):

REVISOR FISCAL PRINCIPAL
LEONOR PATRICIA MARTINEZ
C.C.31290904

REVISOR FISCAL SUPLENTE
PATRICIA ORTIZ RENGIFO
C.C.31158295

En la Notaría Cuarenta del Círculo se cotejó la presente fotocopia con el original que se ha tenido a la vista.
27 NOV. 2007
NOTARIO CUARENTA (E)
Bogotá, D.C., Colombia
Norby Fernando Mo... S.

CERTIFICA

QUE POR ESCRITURA NRO. 1630 DEL 14 DE JUNIO DE 2001 NOTARIA TERCERA DE PALMIRA ,INSCRITA EN LA CAMARA DE COMERCIO EL 20 DE JUNIO DE 2001 BAJO EL NRO. 23 DEL LIBRO V EN LA CUAL EL DOCTOR CESAR HERNAN ZAMORANO ESTRADA CON C.C. NRO. 6.087.401 OBRANDO EN SU CALIDAD DE PRESIDENTE DE MANUELITA S.A., PROCEDE A ENCARGAR O CONFIAR INDEFINIDAMENTE AL DOCTOR ARTURO SUDUPE JIMENEZ CON C.C. NRO. 14.434.820, EXPEDIDA EN CALI LA GESTION DE FIRMAR EN NOMBRE DE MANUELITA S.A. LOS SIGUIENTES DOCUMENTOS QUE DEBAN PRESENTARSE DENTRO DEL TERRITORIO COLOMBIANO: A) LAS DECLARACIONES DE RENTA, DE VENTAS, DE TIMBRE; DE RETENCIONES EN LA FUENTE A TITULO DE IMPUESTO DE RENTA, DE IVA Y DE TIMBRE, LA RESPUESTA A LOS REQUERIMIENTOS TRIBUTARIOS; ASI COMO TAMBIEN LAS SOLICITUDES DE DEVOLUCION DE IMPUESTOS DE RENTA, DE VENTAS Y DE IMPUESTOS ADUANEROS, QUE DEBAN PRESENTARSE ANTE LAS RESPECTIVAS OFICINAS DE LA DIRECCION DE IMPUESTOS Y ADUANAS NACIONALES DIAN. B) LAS DECLARACIONES MUNICIPALES DE IMPUESTOS DE INDUSTRIA Y COMERCIO Y LAS DECLARACIONES MUNICIPALES DE RETENCION EN LA FUENTE A TITULO DE INDUSTRIA Y COMERCIO. C) LAS DECLARACIONES PRIVADAS DE RENTAS PARA DESTILACION DE ALCOHOL. D) LAS DECLARACIONES DE IMPUESTO PREDIAL. E) LAS DECLARACIONES DE IMPUESTO DE RODAJE. F) LAS DECLARACIONES DE IMPUESTO AL CONSUMO. G) LAS DECLARACIONES Y EL PAGO DE LAS CESIONES DE ESTABILIZACION QUE DEBAN PRESENTARSE ANTE EL FONDO DE ESTABILIZACION DE PRECIOS PARA EL PALMISTE, EL ACEITE DE PALMA Y SUS FRACCIONES Y TAMBIEN, LA DECLARACION DEL PAGO DE LA CUOTA PARA EL FOMENTO DE LA AGROINDUSTRIA DE LA PALMA DE ACEITE. PARAGRAFO: ASI MISMO QUEDA FACULTADO EL MENCIONADO MANDATARIO PARA FIRMAR CUALQUIER OTRA DECLARACION QUE REEMPLACE Y/O ADICIONE LAS INDICADAS EN EL CITADO DOCUMENTO. Y TAMBIEN LA CORRESPONDENCIA DIRIGIDA A CUALQUIER ENTE GUBERNAMENTAL SEA ESTE DEL ORDEN MUNICIPAL, DEPARTAMENTAL O NACIONAL RELACIONADA CON ASPECTOS TRIBUTARIOS Y/O CONTABLES DE MANUELITA S.A. SEGUNDO. LA VIGENCIA DE ESTE CONTRATO ES INDEFINIDA Y SOLO TERMINA CUANDO EXPRESAMENTE SE CANCELE EL CITADO PODER O MANDATO EN EL REGISTRO MERCANTIL DE LA CAMARA DE COMERCIO EN LAS CUALES SE HAGA INSCRIBIR LA CITADO ESCRITURA. TERCERO. QUEDA ENTEDIDO QUE EN CUALQUIER MOMENTO EL EXPONENTE, REPRESENTANTE LEGAL DE MANUELITA S.A. Y LOS DEMAS VICEPRESIDENTES EN SU ORDEN PODRAN EJERCER LAS ATRIBUCIONES OBJETO DE LA PRESENTE DELEGACION, EN ESPECIAL CUANDO EL MENCIONADO MANDATARIO SE AUSENTE TEMPORALMNTE O SE ENCUENTRE EN USO DE SUS VACACIONES.

CERTIFICA

CAPITAL AUTORIZADO: $7,500,000,000
NUMERO DE ACCIONES: 3,000,000,000
VALOR NOMINAL: $2.5
CAPITAL SUSCRITO: $2,399,693,957.5
NUMERO DE ACCIONES: 959,877,583
VALOR NOMINAL: $2.5
CAPITAL PAGADO: $2,399,693,957.5

249

NUMERO DE ACCIONES: 959,877,583
VALOR NOMINAL: $2.5

CERTIFICA

QUE EL 04 DE MAYO DE 2005, BAJO EL NRO. 3800 DEL LIBRO IX, SE INSCRIBIO EN ESTA CAMARA DE COMERCIO UN DOCUMENTO PRIVADO DE FECHA 12 DE ABRIL DE 2005, RELATIVO A MODIFICACION DE GRUPO EMPRESARIAL, EN EL CUAL CONSTA:

MATRIZ: INVERSIONES MANUELITA S.A.

SUBORDINADA
MANUELITA S.A..
DOMICILIO : PALMIRA
NACIONALIDAD: COLOMBIANA.

OBJETO: LAS PRINCIPALES ACTIVIDADES DE ESTA SOCIEDAD SON: LA EXPLOTACION DE LA INDUSTRIA DEL DULCE Y DE LOS NEGOCIOS RELACIONADOS CON LOS SECTORES AGRICOLA, GANADERO, PISCICOLA, ALIMENTICIO, METALMECÀNICO, QUIMICO, FARMACEUTICO, VETERINARIO, FINANCIERO, BANCARIO, CREDITICIO, TURISTICO, MINERO, SIDERURGICO, DE LAS OLEAGINOSAS, LA FLORICULTURA, LA FRUTICULTURA, LA HORTICULTURA, LOS PLASTICOS, LOS COMBUSTIBLES, Y EL GAS, EN LOS CUALES PODRA INVERTIR, IMPORTAR O EXPORTAR, COMERCIALIZAR, TRANSFORMAR, DISTRIBUIR Y VENDER AL POR MAYOR Y AL DETAL YA SEA LOS PRODUCTOS PROPIOS O AJENOS O QUE LAS INVERSIONES SE REALICEN DENTRO O FUERA DEL PAIS. TAMBIEN HARA PARTE DEL OBJETO SOCIAL EL NEGOCIO DE LA CONSTRUCCION EN TODOS SUS ASPECTOS INCLUYENDO LA PARTICIPACION EN LICITACIONES PUBLICAS O PRIVADAS, LO MISMO QUE EL NEGOCIO DE LA ADMINISTRACION DE PUERTOS MARITIMOS O TERRESTRES DE SERVICIO PUBLICO; EL ARRENDAMIENTO DE MAQUINARIA AGRICOLA Y EL NEGOCIO DEL MONTAJE, PRODUCCION, DISTRIBUCION Y PRESTACION DE LOS SERVICIOS DE ENERGIA, AGUA, ALCANTARILLADO, TELECOMUNICACIONES, RADIO, TELEVISION, EDUCACION, SALUD, INFORMATICA, PUBLICIDAD, TRANSPORTE DE CARGA Y DE PASAJEROS Y RECOLECCION Y RECICLAJE DE BASURAS. IGUALMENTE PODRA LA COMPANIA RESPALDAR OBLIGACIONES DE SUS FILIALES O DE TERCEROS CON GARANTIA REAL O PERSONAL PREVIA AUTORIZACION DE LA JUNTA DIRECTIVA.

PARAGRAFO: EN DESARROLLO DE ESTAS ACTIVIDADES PODRAN LOS REPRESENTANTES LEGALES DE LA SOCIEDAD DENTRO DE LOS PARAMETROS ESTABLECIDOS EN LOS ESTATUTOS EFECTUAR TODOS LOS ACTOS, CONTRATOS GESTIONES Y DILIGENCIAS QUE CONSIDEREN NECESARIOS O CONVENIENTES PARA EL EJERCICIO O CUMPLIMIENTO DE TODAS LAS MENCIONADAS ACTIVIDADES Y/O NEGOCIOS QUE COMPRENDEN EL OBJETO SOCIAL COMO POR EJEMPLO CULTIVAR CANA, PRODUCIR AZUCAR, MIEL, ALCOHOL Y DEMAS SUBPRODUCTOS; CELEBRAR CONTRATOS DE MUTUO, HIPOTECA, PRENDA, DE ASOCIACION PARA LA EXPLOTACION DE TIERRAS AJENAS; ESTABLECER AGROINDUSTRIAS PARA PROCESAR PRODUCTOS PROPIOS Y/O DE TERCEROS; CONSTRUIR OBRAS, PARCELACIONES, URBANIZACIONES, VIAS PUBLICAS QUE PRODUZCAN PEAJES, ETC; ADQUIRIR, EXPLOTAR, ARRENDAR, GRAVAR, Y ENAJENAR BIENES DE CUALQUIER CLASE; COMERCIALIZAR Y/O MERCADEAR DIRECTAMENTE O MEDIANTE REPRESENTACION, AGENCIAS O SUCURSALES; OBTENER CONCESIONES Y PRIVILEGIOS DE CUALQUIER NATURALEZA, PATENTES DE INVERSION Y MARCAS DE PROPIEDAD INDUSTRIAL, CREAR O ESTABLECER NEGOCIOS CON SUS RESPECTIVAS INSTALACIONES Y/O ADQUIRIR ACCIONES O DERECHOS SOCIALES Y/O PARTICIPAR EN LA CONSTITUCIÓN DE OTRAS SOCIEDADES CUYO OBJETIVO COMPRENDA EN CADA CASO ALGUNA DE LAS ACTIVIDADES O NEGOCIOS RELACIONADOS EN ESTE ARTICULO, ETC.

PRESUPUESTO: INVERSIONES MANUELITA S.A. POSEE MAS DEL 50% DEL CAPITAL DE LA SOCIEDAD MANUELITA S.A. EJERCE LA DIRECCION SOBRE ELLA Y POR ENDE FORMA PARTE DEL GRUPO EMPRESARIAL.

CERTIFICA

QUE A NOMBRE DE LA SOCIEDAD FIGURA MATRICULADO EN ESTA CAMARA DE COMERCIO BAJO EL NRO.607-2 ESTABLECIMIENTO DE COMERCIO: MANUELITA S.A.
UBICADO EN: -KM 7 VIA PALMIRA - EL CERRITO DE PALMIRA
FECHA MATRICULA : 23 DE JUNIO DE 1963
RENOVO : POR EL AÑO 2007

POSTAL
197970-PRI

LUNES 27 AGOSTO 2007 04:52:30 PM

CERTIFICA

QUE LA SOCIEDAD EFECTUO LA RENOVACION DE SU MATRICULA MERCANTIL EL 27 DE MARZO DE 2007

CERTIFICA

QUE NO FIGURAN OTRAS INSCRIPCIONES QUE MODIFIQUEN TOTAL O PARCIALMENTE EL PRESENTE CERTIFICADO.
LOS ACTOS Y DOCUMENTOS REGISTRADOS QUEDAN EN FIRME CINCO (5) DIAS HABILES DESPUES DE LA FECHA DE SU INSCRIPCION, SIEMPRE Y CUANDO DENTRO DE DICHO TERMINO NO SEAN OBJETO DE RECURSOS EN LA VIA GUBERNATIVA.

DE CONFORMIDAD CON EL DECRETO 2150 DE 1.995 Y LA AUTORIZACION IMPARTIDA POR LA SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO LA FIRMA MECANICA QUE APARECE A CONTINUACION TIENE PLENA VALIDEZ PARA TODOS LOS EFECTOS LEGALES.
DADO EN PALMIRA A LOS 27 DIAS DEL MES DE AGOSTO DEL AÑO 2007 HORA: 04:51:59

EL SECRETARIO Aida Elena Vasso P.

En la Notaría Cuarenta de este Círculo se cotejó la presente fotocopia que coincide con el original que se ha tenido a la vista.
27 NOV 2007
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Oct. 8, 1940. G. E. STEVENS 2,217,604

MANUFACTURE OF CANE SUGAR

Filed Sept. 13, 1938 3 Sheets-Sheet 1

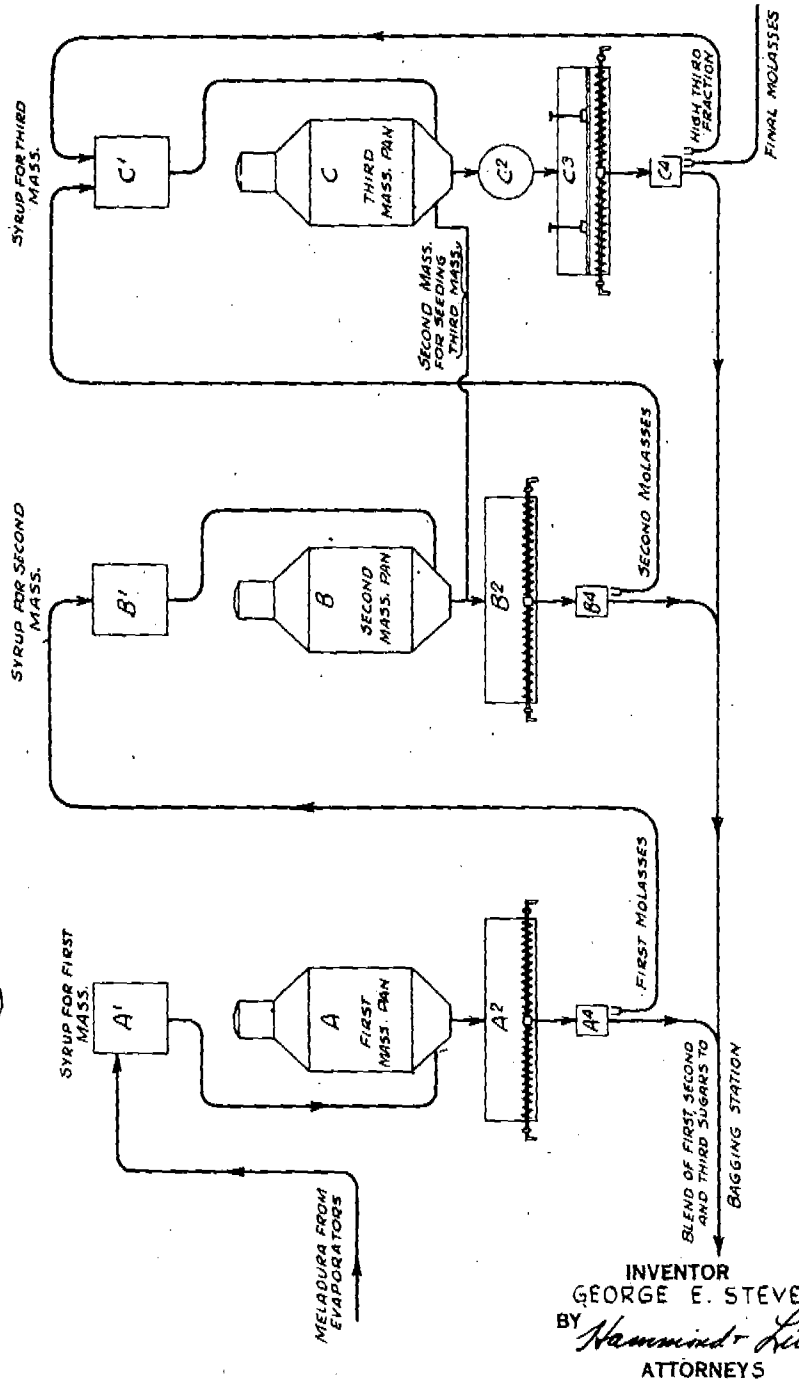

Fig. 1.

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MANUFACTURE OF CANE SUGAR

Filed Sept. 13, 1938 3 Sheets-Sheet 2

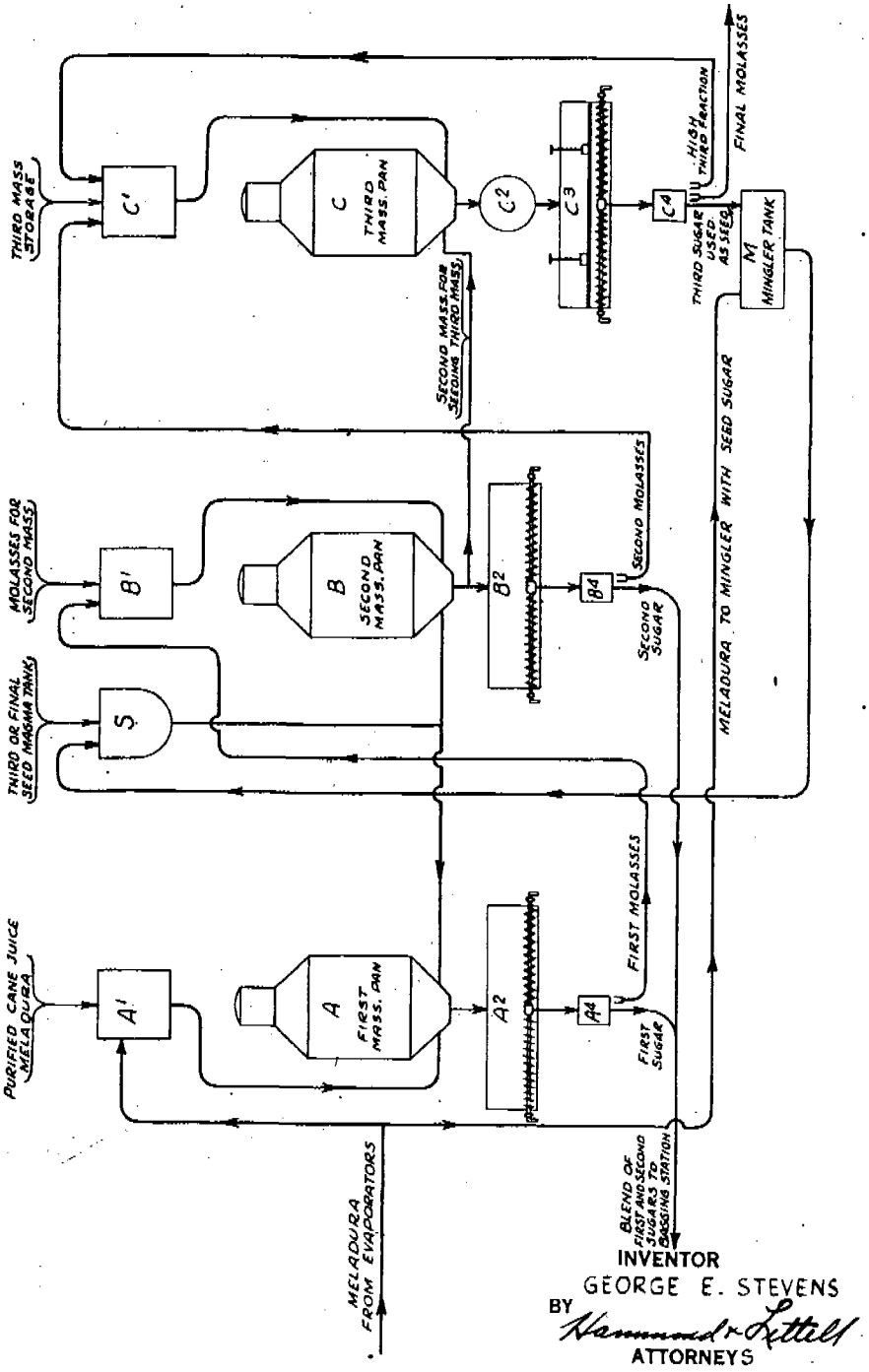

Fig. 2.

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MANUFACTURE OF CANE SUGAR

Filed Sept. 13, 1938 3 Sheets-Sheet 3

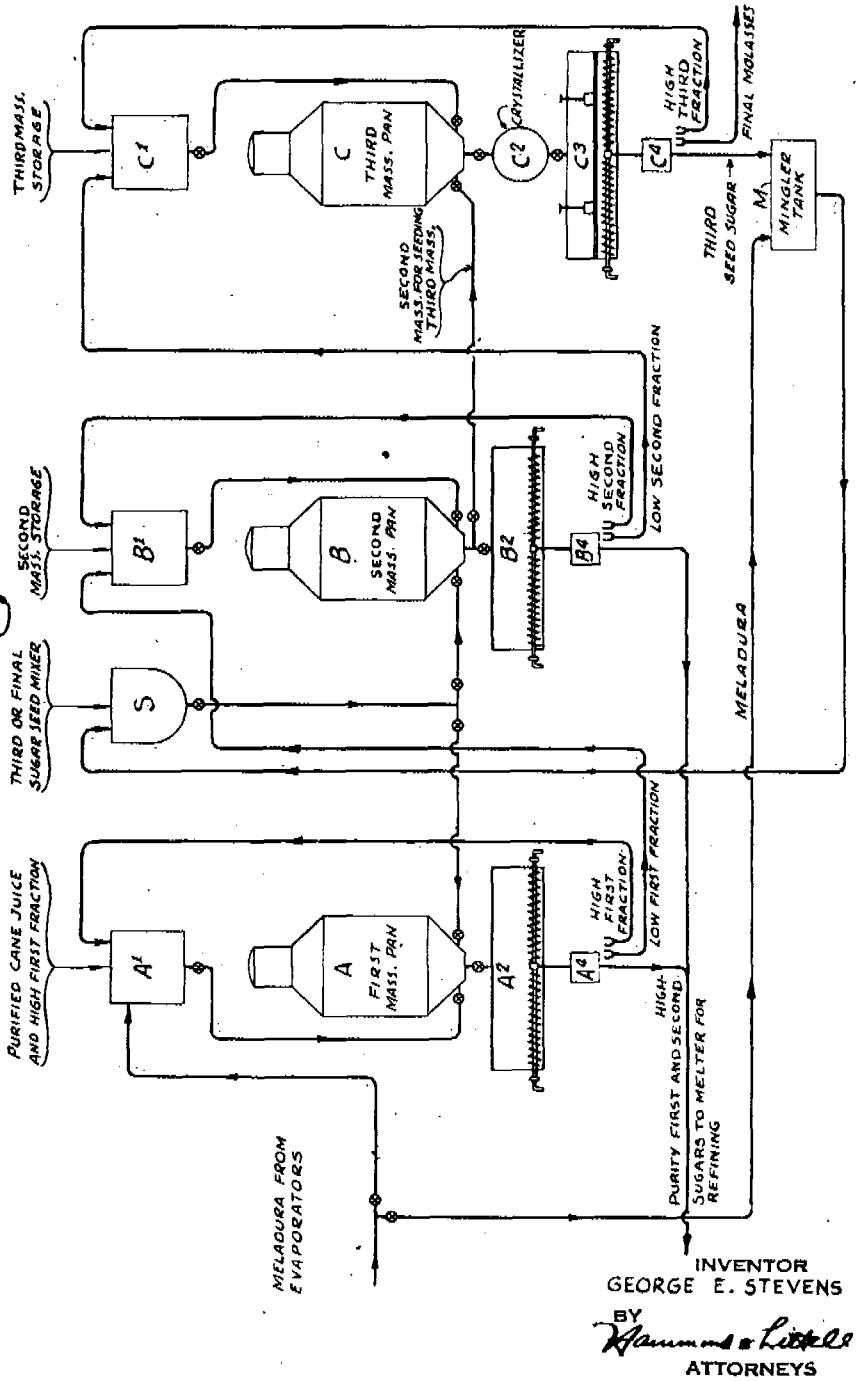

Fig. 3.

INVENTOR
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Patented Oct. 8, 1940

2,217,604

UNITED STATES PATENT OFFICE

2,217,604

MANUFACTURE OF CANE SUGAR

George E. Stevens, Denver, Colo., assignor to The Western States Machine Company, New York, N. Y., a corporation of Utah

Application September 13, 1938, Serial No. 229,647

11 Claims. (Cl. 127—62)

This relates to new and useful improvements in the manufacture of sugar and, more particularly, to improved processes for producing raw cane sugar, plantation white sugar, or sugar of high purity for direct refining from the concentrated and partially purified syrup, or meladura, obtained from evaporators in conventional plantation raw sugar manufacture.

It is general practice in the cane sugar industry to make raw sugar of about 96° average polarization from meladura by a cyclical series of crystallization and centrifuging treatments and to convert this raw sugar into refined sugar by separate treatments in refineries where the raw sugar is first subjected to an affination, or washing, treatment to remove most of its impurities and then melted and passed into a cyclical refining process. In some places, meladura is processed into plantation white sugar, sometimes known as "turbanado," by double-purging and washing the higher purity sugars to obtain slightly colored sugar of about 98 to 99 purity.

The sugar making process in all forms is cyclical in nature and involves a sequence of crystallization and separating treatments on materials of successively decreasing purities, in the course of which crystalline products are obtained from incoming raw material and non-sugars (non-sucrose materials) are progressively concentrated and segregated from the crystallized sugar in molasses or syrups. All process materials except the desired crystalline products and what is known as the "final molasses" are recirculated in the process to recover more of their sugar content, or, from another viewpoint, to effect further elimination of nonsugars from recoverable sugar values. The final molasses is the end material of the process, of low purity, from which additional sugar cannot be obtained economically by further crystallization. It is usually sold or otherwise disposed of at little return to the sugar producer, and in general its content of sucrose governs the extraction of sugar, or yield, from sugar end operations.

The desirability and economy of any particular sugar making process depend upon several interrelated factors, among which are the qualities of the desired product, the purity, or sucrose content, of the final molasses, and the effort and expense involved in recovering the product and segregating non-sugars into the final molasses. In general, the final molasses purity is kept below a more or less fixed point in all processes because of its direct effect on yields and returns, and in all processes there are minimum standards of product quality which must be met. Great variation occurs, however, with respect to effort and expense of manufacture, and this factor becomes troublesome in known processes whenever it is sought to improve the product without raising the final molasses purity. Similarly, attempts to simplify manufacture and to lower costs have often resulted in adverse effects with respect to other important factors, so that the usual process represents a compromise between conflicting tendencies.

Thus there are several known processes for the manufacture of raw cane sugar, but the most common is a three-massecuite process which, in comparison with known two-, four- and five-massecuite processes, usually provides the most satisfactory combination of efficiency factors. The two-massecuite process is simpler, but the product must be of very poor quality in order to keep the final molasses at a suitable purity, and large volumes of massecuite must be boiled and treated for a given output of sugar. The four- and five-massecuite processes produce some sugar of better quality than known three-massecuite processes, but they introduce complications in manufacture. Moreover, all of these processes for producing raw sugar present need for improvement in several important respects. I have found that avoidable reintroduction of once-concentrated non-sugars into higher purity stages of treatment takes place, and that there is too much intermixture of relatively pure and relatively impure materials, so that the directness of the route from incoming raw material to a low purity end material and over-all process efficiency are materially impaired. The only practicable procedure now used for reducing the recirculation of non-sugars is to employ a "double-purging" process on sugar from crystallizer massecuite, but this requires additional work and equipment capacity, also introduces new syrups into the process for sugar recovery, and does not fully overcome the evil. Moreover the usual processes do not permit of continuous control over the quality of materials used in the several massecuite boilings, and they lack the degree of flexibility that would be desirable in a more satisfactory system.

When plantation raw sugar, or "turbanado," is made in a raw sugar factory using the usual three-massecuite process, it is common practice to subject first and second sugars to double-purging treatments in order to obtain a product of suitable quality, and attendant operations produce molasses or syrups which further affect

processing efficiency adversely. When refined white sugar is made from the raw sugar, whether near the raw sugar factory or after shipment of the sugar to foreign areas, the raw must be subjected to affination treatment in order to improve its quality and render it suitable for efficient refining.

In view of the above-mentioned and other characteristics which I have found existing in present-day plantation cane sugar manufacture, it is an object of this invention to provide new and improved processes for making raw cane sugar, plantation white sugar or sugar of high purity for direct refining by which the economy and efficiency of sugar manufacture may be greatly increased while producing products of better quality when desired and keeping the final molasses purity at a suitable low point.

Another object of this invention is to provide processes for the manufacture of sugar from meladura which involve the treatment of much less materials than known processes for a given output of sugar and still give a high extraction or yield of crystallized sugar having qualities which may be equal or considerably superior to present-day standards.

Another object is to provide improved processes avoiding to the maximum extent the intermixing of materials of relatively high purity with materials of relatively low purity to form massecuites, and avoiding the recirculation of materials in which impurities have once been concentrated into higher purity stages of treatment. Thus, the most direct route from starting material to a low purity end material is provided, and the functions of the processes are performed with minimum effort, minimum equipment and minimum costs of production.

Another important object of the invention is to provide processes involving novel treatment of crystallizer massecuites and to produce final molasses of usual low purity and a high yield of crystallizer sugar of much greater than usual purity that may be marketed as raw sugar or recirculated to advantage without further treatment, this being accomplished efficiently and economically and without requiring separate treatment of third sugar such as by the double-purging process required in present practice.

Other features of the invention lie in the provision of improved three-massecuite processes for producing cane sugar from syrups which give optimum results in respect of product quality, effort and expense of manufacture, and net yields or extraction of product; in keeping the first, or highest purity, massecuite at a higher purity than in known three-massecuite processes; when making high purity sugar, in regulating and controlling the purity of the second massecuite by fractionation and distribution of centrifugal run-offs to keep the same at a proper purity in relation to a third or crystallizer massecuite of fixed low purity; in eliminating double-purging treatments when making plantation white sugar; and in eliminating affination treatment when making refined sugar.

Still another object of the invention is to provide a three-massecuite process for the manufacture of high purity cane sugar direct from meladura in which each massecuite is boiled from a standard liquor of controlled purity, the liquor for first massecuite being continually produced as a composite of incoming meladura and another process material of controlled purity and the liquor for second and third massecuites being continually produced as composites of other process materials of controlled purities.

Among further features, my improved processes enable greater control over product quality and greater flexibility of operation, permitting ready adaptation for different conditions of operation or for the production of different types of products.

In one embodiment of the invention, raw cane sugar is manufactured from meladura by an optimum three-massecuite process involving the direct production of marketable raw sugar having good keeping and refining qualities from each of the three massecuites, the maintenance of third massecuite at a fixed low purity, and the maintenance of first massecuite at an abnormally high purity. The third, or crystallizer, sugar produced by this process may be blended with first and second sugars and shipped to market, or it may be mingled with meladura and used as seed for first or second massecuite boilings or for both.

In another embodiment of the invention, cane sugar of extra high quality having a purity of 99 to 99.5 is produced directly from the first and second massecuites, and this may either be marketed as plantation raw sugar of better than usual quality or melted and passed through a conventional refining process for the production of refined sugar, without intermediate affination treatment. In this embodiment also the first massecuite is kept at an unusually high purity. The second massecuite is maintained in proper relation to third massecuite purity by fractionation and distribution of centrifugal run-offs. It will be understood that the production, direct from meladura, of sugar that is suitable for refining without affination treatment offers important advantages and economies to the refined sugar producer.

The accompanying drawings present illustrative flow charts indicating the treatment and distribution of materials in the sugar end operations of a cane sugar factory according to several embodiments of my invention, as explained in greater detail hereinafter. The indications of units of equipment in this flow chart are merely diagrammatic. It will be understood that one or more units of each piece of equipment may be used, depending upon the nature and capacity of the equipment and the character and volume of sugar end operations, and also that other equipment and operations which have no material bearing on this invention may be used in any system for practicing the present process.

According to the illustrative embodiments, three-massecuite processes are provided including high-, intermediate- and low-purity massecuites which will be referred to, respectively, as first, second and third, or crystallizer, massecuites. The several massecuite boilings take place in vacuum pans designated on the flow chart by the characters A, B and C, respectively. The vacuum pans are supplied with materials from storage tanks A^1, B^1 and C^1, respectively. The A and B pans discharge their strikes into mixers A^2 and B^2. The C pan discharges into crystallizer apparatus C^2, in which the third, or crystallizer, massecuite is cooled to promote the growth of crystals and the exhaustion of sucrose from the mother liquor, and from this the crystallized third massecuite passes into a mixer C^3. Each mixer supplies charges of massecuite to centrifugals, designated A^4, B^4 and C^4 for the respective stages, in which take place the op-

erations of purging mother liquor from sugar crystals and, in some instances, of washing the crystals and fractionating centrifugal run-offs into higher-purity and lower-purity fractions, as described more particularly hereinafter. A tank M is provided for mingling third sugar and meladura when the former is used as seed for first and second massecuite boilings, and a storage tank or mixer S receives seed magma from M and holds it for delivery to the vaccum pan A or B in such cases.

All of the above-mentioned equipment may be constructed in a manner now well-known in the art. It is important, however, that the mixers A^2, B^2 and C^3 be equipped to permit accurate regulation and control over the consistency of the massecuites and to keep uniform the temperature and fluidity of successive charges of massecuite as withdrawn for treatment in the centrifugals. This may be accomplished, with maximum capacity and minimum dissolution of sugar grain, by use of the processes and apparatus disclosed in my United States patents, Reissue No. 20,556 and No. 2,086,951. It is also important that the centrifugals be capable of substantially uniform operation and of efficient elimination of molasses from the particular types of massecuite which they treat, that they be equipped with means for effecting sharp separation around their curb walls between syrups flowing from the walls at different time intervals (except as pointed out below) and that purging, washing and syrup-separating operations in each centrifuging cycle be kept under accurate and timed control and adapted for controlled variation to suit requirements when qualities of the massecuites undergoing treatment are subject to change. Each centrifuging cycle on a particular massecuite that is to be washed in the centrifugals should involve a substantially uniform period during which the sugar crystals are purged of substantially all of the mother liquor that can be eliminated efficiently by centrifugal force, a uniform time and period for applying controlled amounts of wash water after such purging, and a uniform time for actuating the syrup separating means. Centrifugal processes in which the charge being centrifuged is subjected to high centrifugal forces in excess of 800 times its weight may be employed to advantage, particularly at the C^4 station. Centrifugal apparatus possessing these operating capacities has been developed by Eugene Roberts and described in his issued patents and pending applications, and is now well-known in the American sugar industry.

The sucrose bearing starting or raw material for the processes herein disclosed consists of concentrated cane juice, or meladura, which enters the sugar end operations of the factory from the evaporators. In the manufacture of raw cane sugar according to an embodiment of this invention, as illustrated diagrammatically in Figure 1 of the drawings, the meladura at a normal purity between about 83 and 89 is passed to storage tank A^1, from which it is withdrawn from time to time and introduced into vacuum pan A for boiling into first massecuite. Each strike of first massecuite may be grained by boiling meladura, by leaving a portion of a previous strike in the vacuum pan, or, in another embodiment as described below, by the use of high-purity seed magma from the mixer tank S. In this embodiment the practice of leaving a portion of previous strike in the pan is preferred since it results in raw sugar of larger grain size and better refining qualities. In any event, the first massecuite purity is kept at least as high as the purity of incoming meladura, and somewhat higher in said other embodiment, so that it is possible to produce first sugar having better qualities than obtainable by prior practice. Further, this practice in forming the first massecuite avoids the addition of relatively impure process materials to meladura and thereby increases the extraction of sugar from first massecuite boilings per unit quantity of massecuite and reduces the total amount of massecuite necessary for a given output of sugar, with consequent savings in equipment requirements and in the effort and expense of manufacture.

The boiling operations at A may be carried out in known manner to produce first massecuite of relatively high net crystallization, so that the mother liquor in the massecuite attains a purity about 20 or more points below the massecuite purity. After the massecuite has been formed it is dropped from the vacuum pan at A into the mixer A^2, where the temperature and consistency of the material are kept substantially uniform and at a point well suited for efficient centrifuging by means of the improvement disclosed in my prior patents, above identified. Suitably conditioned charges of the massecuite are withdrawn successively from the mixer A^2 and treated in the centrifugals A^4, which preferably are capable of operation at high speeds and on controlled cycles so as to enable regulation of total molasses elimination.

In the manufacture of raw cane sugar for importation into domestic markets it is usually of little or no value to produce sugar having a polarization much in excess of 97°, since the usual market and duty structures are based upon raw sugar of 96° average polarization. Sugar polarizing 96° to 97° or higher, as desired, is easily obtained from my high purity first massecuite by simple purging without washing, so that no washing is employed at A^4 in this embodiment of the invention except under abnormal operating conditions, as when the meladura is much below standard purity or when the massecuite is "smeared" or otherwise boiled in a manner preventing effective centrifuging. Hence centrifugals equipped with washing and syrup separating means and time controls are not required at A^4 in this embodiment.

The raw sugar produced at A^4 is discharged from the centrifugals and passed onward for bagging, as indicated on the flow chart. The first molasses produced at A^4, at a purity between about 63 and 69, is passed directly to the B^1 storage tank for use as the principal material for second massecuite boilings.

In preparing the second massecuite, as in the case of the first massecuite, the vacuum pan may be grained by boiling first molasses, by leaving a portion of a previous strike in the pan, or, in another embodiment described below, by using high purity seed magma from S. Again, the massecuite at B is boiled to a relatively high net crystallization, so as preferably to obtain a drop of about 20 points or more between the purity of the second massecuite and the purity of the mother liquor. The dense crystallized massecuite is dropped into the mixer B^2, where its temperature and consistency are kept uniform and controlled by use of my patented improvements, and from B^2 charges of the massecuite are withdrawn successively and treated in centrifugals at B^4. In a small factory the same mixer and centrifugals may be used for both first and second massecuites.

The second massecuite is of such quality that marketable raw sugar of desired polarization and other qualities may normally be produced by simple purging without washing. In no event should any washing required at B^4 be sufficient to raise materially the purity of the centrifugal run-offs, so that all run-offs may be collected together as second molasses and passed to the C^1 storage tank for use in boiling third massecuites, as indicated on the flow chart.

It will be noted that the massecuite in this embodiment is made entirely from first molasses without combining purer syrups therewith Thus the most direct concentration of impurities and production of sugar are effected, resulting in maximum extraction with minimum massecuite boiling and centrifuging operations and reducing losses of recoverable sugar, due to inversion and other chemical changes, to a minimum. The use of a single standard syrup for each of the first and second massecuites makes it unnecessary to compound different materials for the boilings and lends extraordinary simplicity to operation.

The second sugar produced at B^4 is passed onward for bagging with first sugar. Both first and second sugars, in addition to their desirable polarization, have very good refining qualities due to the high purity and uniform qualities of the crystals themselves, and their keeping qualities and factor of safety

$$\left(\frac{\text{moisture}}{\text{100-polarization}}\right)$$

are excellent.

Starting with second massecuite at a purity between 65 and 75 and obtaining a drop of about 20 points or more in the purity of the mother liquor, the second molasses passed to C^1 for third massecuite boiling will have a purity between about 45 and 55. This may be somewhat low for use alone in the production of a third sugar of high purity. My preferred practice in preparing the third massecuite, therefore, is to grain the C vacuum pan with a portion of second massecuite drawn from a B strike, this giving high purity seed crystals upon which to build sucrose extracted from syrups from C^1 without sacrificing crystallization values obtained by operations at B.

The massecuite purity at C is thus kept at a workable point between about 50 and 60. At this purity, crystallization of sugar cannot be carried out to the optimum point by boiling at C, due to the large concentration of non-sugars and the viscosity of the massecuite. According to usual practice, therefore, the massecuite is boiled at C to develop the grain as far as practicable and then dropped into a crystallizer C^2 where it is cooled, with or without stirring, to increase its supersaturation and deposit more sugar on the grain. This, of course, is carried out as far as practicable, and when using my patented improvements for conditioning the massecuite at C^3 a state of high density and high net crystallization may be reached without preventing efficient treatment of the massecuite in the centrifugals at C^4.

In the practice of my processes it is important to conduct the boiling of the third or crystallizer massecuite so that the massecuite is free from objectionable fine grain or "smear" (false grain). It is also important to avoid "smearing" a properly boiled third massecuite through improper operation of the crystallizers, for reasons which will appear below.

After crystallization at C^2 the material passes to mixer C^3 where it is reheated immediately before purging to a controlled temperature giving it a good consistency for purging, but without substantial dissolution of sugar grain. Reheated charges from C^3 are introduced into the centrifugals at C^4 and are centrifuged at high centrifugal forces in a manner ensuring elimination of more than 90% of the mother liquor within a definite time interval. The previous treatment of the massecuite is important to this phase of operations. A smeared massecuite, or one with grain of varying size including very fine grain, is difficult to purge and forms a troublesome skin on the inside of the centrifugal basket due to the resistance of the sugar wall to molasses filtration, and due to the separating effect of high centrifugal forces. A massecuite that has not been reheated to a relatively high temperature before purging does not enable the requisite molasses elimination under efficient operating conditions. By keeping the massecuite of fairly uniform and of medium or large grain size and substantially free of smear, reheating it uniformly under controlled conditions before purging and applying high centrifugal forces in purging the regular elimination of more than 90% of the mother liquor within a definite and comparatively short time interval may be realized. Thereafter, using time-controlled centrifugal operations, a fixed and small amount of wash water is applied to the sugar remaining in the revolving centrifugal. This water should be at a temperature at least as high as, and preferably considerably higher than, the temperature of the reheated massecuite in order to prevent the remaining molasses from cooling and adhering more strongly to the crystals. Within several seconds after the wash water is first applied (e. g., 1 to 3 second) the syrup-separating mechanism on the centrifugal is actuated to divert all syrups subsequently flowing from the curb wall into a separate collecting trough. After completion of washing, the centrifugal may be brought to low speed, and its contents of sugar may be discharged and passed onward for blending with first and second sugars, bagging, and shipment to raw sugar markets.

The third sugar produced by this process regularly attains a polarization of 96° to 97° or higher and a purity of 98 to 99. It has very good refining and keeping qualities, and its factor of safety lies below .33 and usually below .25. The molasses flowing from the curb wall before actuation of the syrup-separating mechanism is collected and disposed of as final molasses. Its purity is at least as low as, and usually somewhat lower, than obtained by present processes, which may be explained by the facts that the mother liquor close to the sugar crystals is more exhausted of sucrose than the remainder of the mother liquor and that the treatment described above, eliminating practically all of the mother liquor before washing, results in a final molasses of somewhat lower purity than otherwise obtained.

The syrup flowing from the curb wall of the centrifugal after actuation of the syrup-separating means attains a purity close to the third massecuite purity. It is collected as a "high third" run-off fraction and returned to the storage tank C^1 for admixture with incoming material for third massecuite boilings.

Thus, by the treatment above described, the maximum amount of concentrated impurities is diverted from the process in the final molasses, the maximum proportion of remaining concentrated impurities is segregated from the third

sugar in a run-off fraction which may be treated efficiently in the lowest-purity stage of the process, all without increase in final molasses purity or appreciable loss of crystallized sugar values, and a crystallized product of high purity is obtained which may be sent direct to market for refining, or recirculated as described below, without further treatment.

In another embodiment of this invention, as illustrated diagrammatically in Figure 2, the first, and second raw sugars may be bagged and shipped to market, and the high purity third sugar (97 purity or higher) may be passed to mingler tank M for mingling with meladura. The resulting magma of high purity is then passed to storage tank or mixer S, from which portions are withdrawn from time to time and introduced into the A and B vacuum pans for use in the seeding of strikes of first and second massecuite. This practice raises the purity of the first massecuite above the meladura purity and permits further increase in the quality and direct yield of first and second sugars, all without loss of crystallized sugar values inasmuch as most of the third sugar becomes the seed of first and second sugar crystals. Again, the recirculation of once-concentrated impurities is avoided to the optimum extent, and the product of raw sugar of good quality may be carried out with a minimum of massecuite boiling, with maximum yield, and at a minimum of effort and expense.

In still another embodiment of this invention, as illustrated diagrammatically in Figure 3 of the drawings, a three-massecuite process for the production of high purity cane sugar direct from meladura is provided in which the treatment and distribution of process materials take place in a manner ensuring maximum product qualities, maximum yields, and minimum requirements of work and equipment in obtaining such qualities and yields. The crystalline products obtained according to this embodiment may be marketed as plantation white sugar of extraordinarily high quality, or they may be melted and passed directly into a refining process for the production of refined cane sugar without intermediate affination treatment.

The equipment used according to this embodiment may be the same as described hereinabove, except that the centrifugals at A^4 and B^4 should be equipped with sprayers for applying washing fluid to the sugar, with means for sharply separating syrups flowing from the curb walls, and with adjustable time-controlled means for governing the time for applying a washing fluid and actuating the syrup-separating means in each centrifuging cycle; for example, the same as used at C^4 in all embodiments of the invention. Suitable equipment of this type is now well known and is disclosed in the pending application of Eugene Roberts, Serial No. 732,114, filed June 23, 1934, now U. S. Patent No. 2,145,633.

In the embodiment now described, boilings of first, second and third massecuites at A, B and C, respectively, are made from standard liquors or syrups, one for each stage, preferably after graining the A and B pans with seed magma from S and the C pan with a portion of massecuite from B.

The liquor for first massecuite is continually formed in the storage tank A^1 as a composite of incoming meladura and a "high first" run-off fraction from the first massecuite purgings. The liquor for second massecuite is continually formed in tank B^1 as a composite of a "low first" run-off fraction from the first massecuite purgings and a "high second" run-off fraction from the second massecuite purgings. The liquor for third massecuite is continually formed at C^1 by combining a "low second" run-off from the second massecuite purgings with the "high third" run-off fraction described hereinabove. High purity seed magma for seeding A and B pans is obtained by mingling high purity third sugar with meladura at M and passing the resulting magma to storage at S, and seed massecuite for use in the C pan may be obtained by cutting over a portion of a strike of second massecuite, as described hereinabove.

In this embodiment of the invention, the purity of the first massecuite is kept considerably higher than the meladura purity, and the purity of the second massecuite is maintained in normal relation to a third massecuite of fixed low purity by fractionation and distribution of centrifugal run-offs. Materials of controlled purities are treated throughout the process, and the process is adapted to produce the optimum results at each stage and to provide a novel correlation of stages resulting in optimum efficiency for the whole.

Boilings at A, B and C and the subsequent treatment of resulting massecuites at A^2 and B^2 and C^2 and C^3, respectively, may take place substantially as described in connection with embodiments for the production of raw sugar. At all of the centrifugals, A^4, B^4 and C^4, however, centrifuging operations are carried out so that most of the mother liquor, preferably more than 90%, is regularly eliminated from the respective sugars within a definite time interval for each stage, and thereafter a fixed amount of wash water is applied to the sugar remaining in the centrifugals. The wash water is preferably applied in the form of a fine spray and at a temperature at least as high as the temperature of the massecuite when ready for purging, which increases its effectiveness in removing adhering impurities from the sugar, makes its effect substantially uniform throughout the sugar wall, and minimizes the amount of wash water needed for proper results.

The separation of syrups flowing from the curb walls of the C^4 centrifugals is performed in the same manner as in the production of raw sugar, thus producing a final molasses of usual low purity, a "high third" run-off fraction of approximately third massecuite purity, and a third sugar of high purity (at least 97, and generally 98 to 99) by means of a small quantity of wash water and without further treatment of the sugar.

In the centrifuging of the first massecuite the separation of syrups flowing down the curb wall is effected at a predetermined interval after applying the wash water, which is regulated so that a portion of the wash syrup flows into one trough with the bulk of the mother liquor to form the "low first" run-off fraction mentioned above, and so that the remainder of the wash syrup is collected separately, at a controlled and relatively high purity (substantially as high or higher than the first massecuite), as the "high first" run-off fraction. In this manner three materials of distinct and controlled purities are produced from the first massecuite-first sugar of unusually high quality (purity usually 99 to 99.5) and two separate run-off fractions, the high first fraction, consisting essentially of wash syrup, and the low first fraction, consisting essentially of green syrup with a small predetermined amount of wash syrup.

Using high purity seed magma for graining the first massecuite and using a liquor or syrup consisting of meladura and the high first fraction for first massecuite boilings, the purity of the first massecuite will range between 85 and 90, which is considerably higher than available with present practices. The drop in purity from first massecuite to low first fraction is about 20 to not more than 25 points, so that the material leaving the high purity stage for treatment as the principal constituent of second massecuite has a purity range usually between 65 and 70 and not more than 75.

In order to produce a low second run-off fraction of suitable purity (e. g., 45 to 55) for use as the principal material in the boiling of third massecuite of fixed low purity, particularly when the high first fraction is of high purity, the second massecuite is kept at a predetermined purity (from 65 to not more than 75) by control over the separation of syrups flowing from the curb walls of the B^4 centrifugals. In this instance the syrup separation is effected at a predetermined time before wash syrup resulting from the washing of second sugar is able to reach the trough in which mother liquor, or green syrup, is collected as the low second fraction. Thus, with uniform purging operations, a substantially definite and regulated amount of mother liquor is collected with the wash syrup from the second sugar in each centrifuging cycle to form a high second run-off fraction of substantially predetermined purity and volume. The proportion of mother liquor to be collected with the wash syrup in each cycle depends upon the purity fixed for the third massecuite (and consequently for the second massecuite) and the purity and volume of the low first fraction.

The high second fraction, consisting essentially of wash syrup from second massecuite purgings, is passed directly to storage at B^1 where it is combined with the low first fraction to form the standard liquor for second massecuite boilings. The low second fraction, consisting substantially entirely of mother liquor from second massecuite, is passed to storage at C^1 where it is combined with the high third fraction to form liquor for third massecuite boilings, as already described. The sugar produced at B^4 is of a high quality comparable to the first sugar, and it attains a purity of at least 99. It is marketed with first sugar as plantation white sugar, or melted with first sugar and refined, without requiring affination treatment.

From the foregoing description of preferred embodiments, it will be understood that the present invention provides processes in which all massecuites are made from materials that are continually produced at controlled purities, and that the cyclical operation of the processes may take place without requiring testing, selection and apportionment of varying materials in order to prepare liquors for massecuite boilings. In keeping with the character of the product required from each stage, the operations at each stage are carried out to give a product of optimum qualities and to effect the maximum segregation of impurities from crystallized sugar values at minimum effort and expense.

The several stages are correlated and the qualities and distribution of recirculated process material are controlled so that the most direct and the most efficient route is followed from the incoming meladura to the low purity end material, the final molasses. In this correlation there is minimum intermingling of relatively impure with relatively pure materials and minimum reintroduction of once-concentrated impurities into higher-purity stages of treatment. Consequently, the volume of massecuite to be boiled and otherwise treated per unit output of desired products is reduced to a minimum, and losses of sugar values through inversion and other chemical changes are minimized.

This invention is not destricted to the use of any particular equipment so long as the equipment to be used possesses the requisite operating capacities. Nor is the invention restricted to features or details of the preferred embodiments, which have been disclosed for purposes of illustration, except as required by a fair construction of the appended claims.

I claim:

1. A three-massecuite process for producing raw cane sugar and final molasses from meladura which comprises boiling first massecuite from incoming meladura, centrifuging the massecuite to produce raw first sugar of at least 96° polarization and first molasses, boiling second-massecuite from first molasses, centrifuging second massecuite to produce raw second sugar of at least 96° polarization and second molasses, boiling third massecuite from second molasses and a run-off fraction from third massecuite purgings of about third massecuite purity, cooling and further crystallizing the boiled third massecuite, reheating the same just before centrifuging, purging the reheated third massecuite to extract mother liquor from third sugar and washing the sugar to a polarization of at least 96°, and sharply separating the streams of mother liquor and wash syrup expelled from the sugar to obtain final molasses of usual low purity and the aforesaid run-off fraction, respectively.

2. A three-massecuite process for producing raw cane sugar and final molasses from meladura which comprises boiling first massecuite from incoming meladura, centrifuging the massecuite to produce raw first sugar of at least 96° polarization and first molasses, boiling second massecuite from first molasses, centrifuging second massecuite to produce raw second sugar of at least 96° polarization and second molasses, boiling third massecuite from second molasses and a run-off fraction from third massecuite purgings of about third-massecuite purity, cooling and further crystallizing the boiled third massecuite, reheating the same just before centrifuging, purging the reheated third massecuite to extract mother liquor from third sugar and washing the sugar to a polarization of at least 96°, sharply separating the streams of mother liquor and wash syrup expelled from the sugar to obtain final molasses of usual low purity and the aforesaid run-off fraction, respectively, and blending said first, second, and third raw sugars to obtain raw sugar of at least 96° polarization having good refining and keeping qualities.

3. A three-massecuite process for producing raw cane sugar and final molasses from meladura which comprises boiling first massecuite from incoming meladura on seed sugar of at least 97 purity, centrifuging the massecuite to produce first sugar of at least 96° polarization and first molasses, boiling second massecuite from first molasses on seed sugar of at least 97 purity, centrifuging second-massecuite to produce second sugar of at least 96° polarization and second molasses, boiling third massecuite,

on seed provided by a portion of said second massecuite, from second molasses and a run-off fraction from third massecuite purgings of about third massecuite purity, cooling and further crystallizing the boiled third massecuite, reheating the same just before centrifuging, purging the reheated third massecuite to extract at least 90% of the mother liquor from the third sugar and then washing the sugar to a purity of at least 97, sharply separating the streams of mother liquor and wash syrup expelled from the sugar to obtain final molasses of usual low purity and the aforesaid run-off fraction, respectively, and using said third sugar of at least 97 purity as the aforesaid seed sugar.

4. The process as claimed in claim 3, mingling said third sugar of at least 97 purity with meladura to form a seed magma and graining first and second massecuites with said magma to provide the aforesaid seed sugar.

5. A three-massecuite process for producing raw cane sugar and final molasses from meladura which comprises boiling first massecuite of at least meladura purity from incoming meladure, on seed sugar of at least 97 purity, and centrifuging the massecuite to produce first sugar of at least 96° polarization and first molasses of about 20 or more points lower purity than the first massecuite, boiling second massecuite from first molasses on seed sugar of at least 97 purity, and centrifuging the same to produce second sugar of at least 96° polarization and second molasses of about 20 or more points lower purity than the second massecuite, boiling third massecuite, on seed provided by a portion of said second massecuite, from said second molasses and a run-off fraction from third massecuite purgings of about third-massecuite purity, cooling and further crystallizing the boiled third massecuite, reheating the same just before centrifuging, purging the reheated massecuite to extract at least 90% of the mother liquor from the third sugar and then washing the sugar to a purity of at least 97, sharply separating the streams of mother liquor and wash syrup expelled from the sugar so as to obtain final molasses of usual low purity and the aforesaid run-off fraction, respectively, and using said third sugar on the aforesaid seed sugar.

6. In the production of cane sugar and final molasses by a succession of massecuite boiling and centrifuging operations in three stages at successively lower purities, the steps which comprise washing the sugar in the centrifugals at the lowest-purity, or crystallizer, stage to a purity of at least 97, separating centrifugal run-offs into a lower-purity fraction consisting substantially entirely of mother liquor and a higher-purity fraction of a purity near the crystallizer massecuite purity consisting essentially of wash syrup, withdrawing the lower-purity fraction from process as final molasses of optimum low purity; and returning the higher purity fraction for boiling into further stripes of crystallizer massecuite.

7. In the production of cane sugar and final molasses by a succession of massecuite boiling and centrifuging operations in stages at successively lower purities, the steps which comprise boiling the lowest-purity, or crystallizer, massecuite to produce a massecuite substantially free of very small grain, cooling and further crystallizing the massecuite, reheating successive portions of the massecuite before centrifuging, purging more than 90% of the mother liquor from the reheated massecuite in a centrifugal, thereafter applying a fixed amount of washing liquid to the sugar in the revolving centrifugal and washing the sugar to a purity of at least 97, sharply separating the resulting wash syrup from the preceding mother liquor as they flow from the centrifugal curb and collecting the same, respectively, as a centrifugal run-off fraction of a purity near the crystallizer massecuite purity and as final molasses, and recirculating said run-off fraction for boiling into crystallizer massecuite.

8. In the production of high purity cane sugar and final molasses from meladura by a succession of massecuite-boiling and centrifuging operations in three stages at successively lower purities, the steps which comprise passing meladura to the highest-purity stage, washing the sugar in centrifugals at each stage to a purity of at least 97, separating centrifugal run-offs at each stage into higher-purity and lower-purity fractions of predetermined controlled purities, withdrawing the lower-purity fraction at the lowest stage from process as final molasses, passing the lower-purity fractions from the other stages to the stages next below for boiling into massecuites, and returning the higher purity fractions for boiling into massecuites at the respective stages.

9. A three-massecuite process for producing high purity cane sugar and final molasses from meladura which comprises fixing the purity of the third massecuite for the production of final molasses of optimum low purity, washing the sugar from each massecuite in centrifugals, making the first massecuite from meladura and process materials of considerably greater purity than the meladura including high purity seed sugar and a run-off fraction consisting substantially entirely of high purity wash syrup from the first purgings, maintaining the second massecuite at a normal purity in relation to the fixed purity of the third massecuite by making the same from high purity seed sugar, a run-off fraction consisting essentially of mother liquor from the first purgings, and a run-off fraction of regulated purity from the second purgings consisting of predetermined proportions of mother liquor and wash syrup, making said third massecuite from a run-off fraction consisting of mother liquor from the second purgings, a portion of said second massecuite as seed and a run-off fraction consisting essentially of wash syrup from the third purgings, and using washed third sugar at a purity of at least 97 as the aforesaid high purity seed sugar.

10. A three-massecuite process for producing high purity cane sugar and final molasses from the meladura which comprises boiling first massecuite, on seed sugar of high purity, from incoming meladura and a "high first" run-off fraction, purging mother liquor from the massecuite, washing the first sugar in centrifugals and continually fractionating centrifugal run-offs to produce first sugar of at least 99 purity, a "low first" run-off fraction consisting essentially of mother liquor and said "high first" run-off fraction consisting substantially entirely of wash syrup, boiling second massecuite, on seed sugar of high purity, from said "low first" fraction and a "high second" run-off fraction, purging mother liquor from the second massecuite, washing the second sugar in centrifugals and continually fractionating centrifugal run-offs to produce second sugar of about 99 purity, a "low second" run-off fraction consisting substantially entirely of mother-liquor and said "high second" fraction consisting essentially of wash syrup, boiling third massecuite, on seed provided by a portion of said

The present invention relates to improvements in sugar crystallization.

It is an object of the invention to improve the process for producing crystalline sugar.

Another object is to improve the rate of crystallization of sugar from supersaturated solutions.

Another object is to increase the yield of crystals in a sugar crystallization process.

A further object is to improve the quality of sugar crystals in respect of color, texture and dryness.

The above and other objects will become apparent in the course of the following description.

The conventional procedure of producing sugar from cane or beet involves repeated concentrations and crystallization. Some of these steps require extended lengths of time, as much as 72 hours, to attain practical completion of crystallization. The length of time is a major objection to the existing processes requiring large capital investment in materials in process and processing equipment. It has also been recognized that even holding the crystallization step over a prolonged period does not remove all of the valuable sugar and much of the sugar in the mother liquor is sacrificed as a low-grade by-product of sugar refining.

Generally similar processes are employed in the production of the various commercial forms of crystalline dextrose. The improvement of these processes is also within the scope of this invention.

A related problem is recognized in the so-called soft sugar phase of sugar manufacture in that these soft sugars or brown sugars are difficult to obtain in a satisfactory condition. A common objection to the soft sugars is their tendency to block into a solid mass. Other

objections noted are loss of bloom and absence of free flowing characteristics identified as "crawl" in the trade.

In accordance with the present invention it has been found that sub stantial improvements in crystalline sugar products are obtained when the mother liquor or adherent molasses is removed from the crystals in the presence of certain non-ionic surface active agents hereinafter described. To obtain these improvements the said surface active agents can be introduced at any stage of the process up to and including the crystal purging step. In addition to the improvement of the ultimate crystalline product, other advantages in the crystallization itself are obtained where the surface active agent is introduced before or during crystallization. Thus, the invention can be practised by adding the surface active agent during the boiling of the sugar liquor, during crystallization, during the dumping or spinning of the massecuite, or during the crystal purging (by addition to the purge water).

In the presence of small amounts of the surface active agents here employed sugar crystallizes from solution at an increased rate and has been observed to form a drier and whiter product. The improvement in the crystallization process therefore makes it possible to reduce the time required for growing a given quantity of sugar crystals or, under some circumstances, to grow a larger crop of sugar crystals in a given length of time. The crystalline sugar product is of better quality particularly in the soft or brown grades.

The nonionic surface active agents employed in the practice of the invention may be selected from the following groups: (1) partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule; (2) polyoxyalkylene ethers of partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; (3) polyoxyalkylene esters of long

chain fatty acids wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; and (4) long chain fatty acid esters of polyoxyalkylene ethers of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each. The term "long chain fatty acid" is used herein to identify the fatty acids which have from 12 to 18 carbon atoms per molecule.

Examples of surface active agents of the class identified as (1) above include glycerol monooleate, pentaerythritol monolaurate, mannitol dioleate, sorbitan monolaurate, sorbitan monostearate, sorbitan monopalmitate and glucose monostearate. The polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule, therefore, include the polyhydric alcohols, the cyclic inner ethers of polyhydric alcohols and the simple sugars.

Surface active agents of the type identified as (2) above include 10 polyoxyethylene glycerol monopalmitate, 15 oxyethylene pentaerythritol monomyristate, 10 polyoxyethylene-10 polyoxypropylene sorbitan monestearate, 20 polyoxyethylene sorbitan monostearate, 40 polyoxyethylene mannitol distearate, 20 polyoxypropylene sorbitan monostearate , 10 polyoxyethylene sorbide monolaurate, and 30 polyoxyethylene glucose monostearate. The Arabic numeral preceding the name of the compound indicates the average number of oxyalkylene groups per molecule of the compound. As well understood in the chemical arts, compounds of this type are prepared by reacting the partial ester with ethylene oxide propylene oxide or mixtures of ethylene and propylene oxides in proportions to produce the average number of oxyethylene groups desired per molecule of the finished polyoxyalkylene ether. Alternatively, a preformed polyalkylene glycol of an average oxyalkylene unit content corresponding to that desired is condensed by conventional etherification reaction with the partial ester.

Surface active agents of the type identified as (3) above include 8 polyoxyethylene monostearate, 15 polyoxyethylene distearate, 20 polyoxyethylene monooleate, 20 polyoxyethylene stearate oleate, 6 polyoxyethylene monolaurate, 10 polyoxypropylene monooleate, and 5 polyoxyethylene-5 polyoxypropylene monolaurate. Compounds of this type may be prepared by the esterification of a polyalkylene glycol containing the required number of oxyalkylene groups or by the reaction of alkylene oxide and the indicated fatty acid. The latter reaction produces a monoester which can, if desired, be completely esterified by reaction with another mol of the same or a different fatty acid.

Surface active agents of the type indicated as (4) above include the monolaurate of hydroxyethyl glycerol, the monopalmitate of 10 polyoxyethylene pentaerythritol, the dioleate of 6 polyoxyethylene mannitol, the dioleate of 6 polyoxypropylene mannitol, the hexacleate of 20 polyoxyethylene sorbitol, the pentastearate of 40 polyoxyethylene sorbitol and the monestearate of 6 polyoxyethylene glucose. The compounds of this group are distinguished from those of type (2) in that the polyhydroxylic compound is first reacted with alkylene oxide or an alkylene or polyalkylene glycol to form the oxyalkylene or polyoxyalkylene ether which is thereafter esterified with the indicated fatty acid.

Mixtures of surface active agents of one or more of the types described are contemplated for use in practising the invention.

Preferred surface active agents for use in accordance with the invention are the polyoxyethylene compounds of types (2), (3) and (4), which contain at least 6 oxyethylene groups for each fatty acid radical. These compounds are readily dispersible in the sugar solutions.

The amount of surface active agent required to accomplish the intended improvement varies with the particular agent selected, the

particular process in which it is used and the manner in which it is applied. From 0.01% to 0.5% surface active agent based on sugar solids is generally preferred. In order to facilitate mixing of the surface active agent with the concentrated liquors it may be found desirable to disperse or dissolve the surface active agent in a small quantity of water or a more dilute sugar solution and distribute this throughout the concentrated solution. Additional stirring or mixing may be desirable to hasten the dispersion of the added material.

Where the surface active agent is added to the purge water the preferred concentrations are from 0.1% to 1.0% in the water.

The following examples are illustrative of the process of this invention.

EXAMPLE I

The effect of a surface active agent of the classdefined on the crystallization of sucrose is illustrated by the following small scale laboratory test:

A sucrose syrup 1.04 supersaturated at 25°C. was modified by adding to it 0.3% of 20 polyoxyethylene sorbitan monostearate, based on the weight of sugar in the syrup. A 10 gram sample of the treated syrup was placed in a test tube and seeded with 6X sugar at a 1:30 ratio. The change in concentration of the syrup as crystallization occurred at 25°C. was determined by observing the refractive indices of the syrup at 1 minute intervals. After 15 minutes the syrup showed that 66.5% of the sugar in excess of the saturation value at 25°C. had crystallized.

A similar test run without the addition of the surface active agent showed only 19% crystallization in the same time.

EXAMPLE II

A test similar to that described in Example I was performed using 0.3% of 20 polyoxyethylene sorbitan monolaurate instead of the monostearate of Example I. After crystallizing for 15 minutes, 44% of

the sugar in excess of the saturation value had crystallized.

EXAMPLE III

The invention can be employed as follows in a cane sugar process:

The mother liquor from a second strike of crystals is boiled down in the conventional manner for a final crystallization and placed in a stirred tank type crystallizer. A quantity of 20 polyoxyethylene sorbitan monostearate equal to 0.1% of the weight of sugar solids in the massecuite is added to the crystallizer and dispersed throughout the massecuite. After the usual quantity of sugar crystals are obtained the massecuite is spun and purged in the conventional manner.

EXAMPLE IV

In a beet sugar process the invention can be practiced as follows:

To a green syrup in the vacuum pan 0.1% sorbitan monostearate is added based on the sugar solids in the pan. The syrup is boiled and the agitation produced thereby serves to disperse the surface active agent. The crystalline sugar product is recovered in the conventional manner and the mother liquor is passed on to be boiled for another strike of sugar. The mother liquid contains the added surface active agent and its functions are therefore carried through to the subsequent stages of the process.

EXAMPLE V

In a corn sugar refining process the invention can be practiced as follows:

To a corn syrup being boiled in a vacuum pan is added 0.05% of 8-polyoxyethylene monostearate based on the weight of sugar in the pan. After concentration the syrup is drawn off, seeded, crystallized and spun.

EXAMPLE VI

In the production of brown sugar the invention can be applied thus:

A cake of brown sugar in the centrifuge is purged with water containing 0.2% by weight of 20 polyoxyethylene monostearate. The sugar is then recovered from the centrifuge in the usual manner.

Many variations in the details of the process will be apparent to those skilled in this art without departing from the invention which is defined in the following claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. In the process for producing crystalline sugar the improvement which consists in removing the mother liquor from the crystals in the presence of a surface active agent selected from the group consisting of: (1) partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule; (2) polyoxyalkylene ethers of partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkenylene groups have from 2 to 3 carbon atoms each; (3) polyoxyalkylene esters of long chain fatty acids wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; and (4) long chain fatty acid esters of polyoxyalkylene ethers of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each.

2. The process of Claim 1 wherein the sugar is cane sugar.

3. The process of Claim 1 wherein the sugar is beet sugar.

4. The process of Claim 1 wherein the sugar is dextrose.

5. The process of Claim 1 wherein the sugar is brown sugar.

6. In the process for producing crystalline sugar the improvement which consists in growing the sugar crystals in the presence of a surface active agent selected from the group consisting of: (1) partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6

carbon atoms per molecule; (2) polyoxyalkylene ethers of partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; (3) polyoxyalkylene esters of long chain fatty acids wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; and (4) long chain fatty acid esters of polyoxyalkylene ethers of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each.

7. The process defined in Claim 6 wherein the said surface active agent contains at least 6 oxyethylene groups per fatty acid radical.

8. The process of Claim 6 wherein the surface active agent is 20-polyoxyethylene sorbitan monostearate.

9. The process of Claim 6 wherein the surface active agent is introduced into the crystallizer.

10. The process of Claim 6 wherein the surface active agent is present in the amount of 0.01 to 0.5% by weight of sugar solids.

11. In the process for making a soft sugar the improvement which consists in purging the spun cake with water containing a surface active agent selected from the group consisting of: (1) partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule; (2) polyoxyalkylene ethers of partial long chain fatty acid esters of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each; (3) polyoxyalkylene esters of long chain fatty acids wherein the

oxyalkylene groups have from 2 to 3 carbon atoms each; and (4) long chain fatty acid esters of polyoxyalkylene ethers of polyhydroxylic compounds which have from 3 to 6 carbon atoms per molecule wherein the oxyalkylene groups have from 2 to 3 carbon atoms each.

12. The process of Claim 11 wherein the surface active agent contains at least 6 oxyethylene groups per fatty acid radical.

13. The process of Claim 11 wherein the surface active agent is 20-polyoxyethylene sorbitan monostearate.

14. The process of Claim 11 wherein the surface active agent is present in the amount of 0.1% to 1.0% in the water.

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The present invention relates to a process for the crystallization of sugar in the sugar making industry and sugar refineries.

The crystallization of sugar from beet sugar or cane sugar syrups is generally obtained by evaporation and cooling. These operations are carried out in cooking or boiling apparatus and in crystallizers or mixers.

In the course of the cooking of the syrups germs are created in the latter or germs are introduced in the latter and these germs are enlarged during the cooking by a continuous supply of syrups from which a part of the water is removed by evaporation so as to maintain a given degree of supersaturation of the sugar within its mass during the cooking. The cooked mass obtained, which contains sugar crystals in suspension in the syrup having a low sugar content, is then poured into crystallizers or mixers where it is cooled so as to obtain a crystallization by cooling. When the mass has reached a given temperature generally 40-60°C, it is introduced into a turbine where it is centrifuged so as to separate the crystallized sugar formed, and a residue is collected which still contains sugar in solution and is once more subjected to the same series of operations. This series of operations is called one run. Normally, a plurality of successive runs are effected, the duration of which increases each time owing to the diminishing amount of sugar in the residues, and at the end of the last run there is obtained as residue a molasse which still contains a rather large amount of sugar.

The total cooking time in sugar making in respect of an operation comprising three runs is usually about between 17 and 22 hours with a high consumption of a large amount of steam which varies widely with respect to time and a utilization of a large number of cooking apparatus.

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This cooking, which lasts several hours, results in substantial losses of sugar which is destroyed with the by-products, so that the quality of the sugared juice is impaired. Further, the non-sugar itself undergoes changes which increase the viscosity. Now it is known that the fluidity of the mass is a function of the viscosity of the residues and a certain fluidity is necessary to permit the transfer of the cooked mass from one apparatus to the other and the turbining of this cooked mass. The higher the sugar crystal content of the cooked mass the greater the necessity of a high water content so as to obtain this fluidity. On the other hand, the crystallization yield upon cooling in the mixer is the lower as the water content is higher. Since in order to obtain a good crystallization there must exist a certain relative mobility of the crystals and the residue when cooling to permit a good diffusion of the sucrose through the residue, it is current practice to dilute the mass in the crystallizer with saturated residue which does not crystallize and only acts as a diluting agent, or with water which lowers the speed of crystallization of the sugar and results in a dissolution of the latter in the mother liquor where it remains in solution at the end of the treatment in the mixer. Consequently, in conventional processes it is impossible to obtain in each run an overall sugar yield exceeding 70% (this yield is the ratio between the weight of the sugar collected and the weight of the sugar present in the cooked mass, the yield usually being of the order of 55-65%). It is therefore necessary, as mentioned hereinbefore, to employ a plurality of successive runs.

Apart from the aforementioned drawbacks, the cooking is a delicate operation if it is desired to obtain crystals having a well regular and constant size, this being particularly so in the last run where viscosity is high owing to in particular the

modification of the non-sugars, fine crystals being obtained which block the interstices between the large crystals and render the centrifuging operation difficult.

The object of the present invention is to provide a process which markedly increases the crystallization of the sugared juice in the sugar-making industry or sugar refineries and consequently permits a decrease in the number of runs, which may be as much as 50%, a decrease in the consumption of fuel and a more efficient utilization of the apparatus of the plant, or a reduction in the required number of cooking apparatus.

The invention provides a process for crystallizing sugar from syrups from the sugar-making industry or sugar refinery of the type comprising at least one series of operations including a concentrating stage and/or a cooling stage and a turbining stage, wherein at least a part of the mass containing sugar crystals is withdrawn at one moment of the process selected from the concentrating and cooling stages and the passages between the concentrating, cooling and turbining stages, the sugar crystals contained in said part are removed, and the sugar liquid from which the crystals have been removed in one of said concentrating, cooling and turbining stages is returned to and admixed with the rest of said mass.

Further features and advantages of the invention will be apparent from the ensuing description, with reference to the accompanying drawings to which the invention is in no way limited.

In the drawings:

Figs. 1-4 are flow diagrams showing various manners of carrying out the process of the invention;

Fig. 1 shows a first manner of carrying out the invention. The sugar syrup from a prior stage of the sugar-making plant or refinery is progressively introduced into a conventional

cooking or boiling apparatus 1 in which the level of the mass is raised in the usual manner after having initiated the crystallization, for example by the introduction of germs. In this period the sugar crystals are formed and grow in accordance with the usual process, the temperature being held at about 80°C. At the end of the cooking or boiling, when the cooked mass reaches the usual level, a fraction of this cooked mass, which contains sugar crystals in suspension in a concentrated sugar residue is withdrawn through a pipe 2 provided with a valve 3 (or through a draining valve of the apparatus) and sent to a turbine 4 in which the sugar is separated from the residue by a centrifuging operation and collected at 5. The residue leaves the turbine 4 by way of a pipe 6 and can be very slightly diluted at 6a so as to dissolve the sugar crystals still in suspension (this added water can be removed in a subsequent concentrating stage, for example in a flash evaporator, without the sugar once more crystallizing, it remaining in supersaturation). Advantage can also be taken of this stage for modifying the pH of the residue so as to bring it to a value permitting the obtainment of the optimum yield of crystallization. The residue is de-aerated and heated at 7 to a temperature of the order of the cooking temperature and returned to the cooking apparatus by way of a pipe 8 where it joins the rest of the cooked mass. A new cooked mass is thus obtained in which the equilibrium between the residue and the sugar crystals is modified. The sugar crystals are immersed in a concentrated residue but the mass is more fluid than when the aforementioned fraction of the cooked mass was withdrawn by way of pipe 2 since the sugar crystals contained in this fraction have now been eliminated. Consequently, a certain amount of sugar once more crystallizes in the cooking apparatus, and it is possible to withdraw the residue by way of the pipe 2

until obtainment of a mother liquor which is such that, after the conventional compacting and subsequent cooling in a mixer, a residue is obtained in the final turbining in the form of molasse. When the cooking stage is considered finished, the cooked mass is evacuated by way of a pipe 9 into a conventional mixer 10 whence, after cooling, it is sent by pipe 11 into a turbine 12 where the sugar is separated at 13 and the residue at 14 in the usual manner. In this way it is possible to carry out the complete crystallization operation in a single run. The duration of the cooking in the cooking apparatus is much shorter than in the case of a plurality of successive runs. During this operation, a progressive rise in the ratio $\frac{\text{non-sugar}}{\text{water}}$ is observed, this is very important as concerns the improvement in the subsequent crystallization by cooling since for this operation it is advantageous to have a water content which is as low as possible compatible with sufficient fluidity. The latter is insured in the present invention by the fact that the $\frac{\text{amount of crystals}}{\text{amount of residue}}$ is lower than in the conventional process.

The total amount of sugar provided by the process is the sum of the sugars obtained in the turbine 4 and turbine 12.

By way of example, to which the invention is not intended to be limited, the following results might be mentioned:

A cooked mass having a purity of 93 (the purity being the ratio $\frac{\text{sugar}}{\text{sugar + non-sugar}}$), a Brix of 91.5 (the Brix being the ratio $\frac{\text{dry substances}}{\text{dry substances + water}}$) and a ratio $\frac{\text{non-sugar (NS)}}{\text{water}}$ of 0.75 is prepared in the cooking apparatus 1. At the end of the cooking 50% of the cooked mass is withdrawn at 2 and turbined at 4 at a temperature of about 70°C, the sugar obtained is withdrawn and the residue has the purity of 84.8 when leaving the turbine at 6. It is heated to 80-85°C and is returned to the cooking apparatus. The conventional compacting is then effected and the

cooked mass which leaves the apparatus 1 at 9 at the end of the operation has a ratio $\frac{NS}{water}$ of 0.18, a residue purity of 79, and the total sugar yield of the run from the different turbining operations is 71.7%, whereas in the conventional process the sugar yield is 58%. Therefore, by means of the process of the invention, the sugar yield is markedly increased. Owing to this increase in the yield, it is possible in the case under consideration to finish the operation in a single run which is carried out in the same manner as that described with reference to Fig. 1.

The amount of cooked mass which is withdrawn by way of the pipe 2 varies preferably between about 25 and 65%, although this is not critical. The most advantageous amount in this manner of carrying out the invention is about 50%.

This withdrawal of the cooked mass can be effected at the end of cooking or during the cooking. It is also possible to operate in a continuous manner and continuously withdraw a cooked mass from the cooking apparatus during cooking and continuously return the residue to the cooking apparatus.

Fig. 2 shows diagrammatically a second manner of carrying out the process of the invention. The sugar syrup is cooked in a conventional cooking apparatus 20 in the usual manner. At the end of the cooking and after concentrating or compacting (serrage), the apparatus is drained by way of a pipe 21 and there is withdrawn by way of a pipe 22 part of the cooked mass in its path through the pipe 21 so as to send it to a turbine 23, the rest of the mass being sent by way of the pipe 21 to a conventional mixer 24 for its crystallization by cooling. If desired, a cooling mixer could be inserted in the pipe 22. In the turbine 23, the mass is centrifuged, the sugar is collected at 25 and the residue issues by way of a pipe 26 and is, if desired,

subjected to diluting and refining operations at 7, de-aerating and heating operations at 28 and a concentrating operation at 29 (these operations can be eliminated if desired but they are preferred in that they tend to insure a sound operation of the process). The residue is returned by way of a pipe 30 to the mixer 24 where it is admixed with the rest of the cooked mass. In this way the cooked mass which is cooled, contains a smaller amount of sugar crystals which are immersed in a supersaturated residue which imparts good fluidity to the mass and insures a good mobility of the residue around the crystals. Consequently, crystallization is effected under conditions which are better than those prevailing in the conventional process and the temperature in the mixer can be lowered (at least down to, for example, 30°C instead of the conventional 50-60°C) while still providing at the end of the mixing sufficient fluidity for the final turbining carried out in the apparatus 31 in which the sugar is collected at 32 and the residue at 33.

In the conventional process, instead of sending a concentrated residue to the mixer, it would have been necessary to dilute the cooked mass either with a saturated residue which only has a diluting function and therefore does not crystallize, or with water so as to obtain a fluidity suitable for the turbining operation with the resulting disadvantages mentioned hereinbefore.

The concentrating operation at 29 has the advantage of increasing the ratio $\frac{NS}{water}$ of the residue returning to the mixer 24 and this still further enhances the crystallization in this mixer. However, it must not be pushed to the point of once more obtaining crystals in the residue.

The amount of cooked mass withdrawn through 22 can advantageously vary between 25 and 60% of the cooked mass; it is

usually 30-40%. However, as a variant, it is also possible to withdraw the whole of the cooked mass and turbine it at 23, concentrate the residue and send it to the mixer 24. In this case, sugar crystals are introduced into this mixer whose crystal size can be so chosen as to obtain optimum crystallization.

In withdrawing for example 50% of the cooked mass by way of 22, it is possible to obtain a crystallization yield in the run of 70% whereas in conventional processes the yield is 64% in respect of the same cooked mass, the residue issuing from the mixer having a purity of 80, whereas in the conventional processes its purity exceeds 82.5.

Fig. 3 shows a modification of the manner of carrying out the invention just described, the cooked mass which has substantially not received an extreme concentrating or compacting leaving a cooking apparatus 40 by way of a pipe 41 and being divided by a pipe 42 into a fraction which is fed to a turbine 43 and a fraction which is fed directly to a concentrating mixer 44 operating under a vacuum, the latter being created in this mixer by way of a pipe 45. The sugar crystals are withdrawn from the turbine 43 at 44 and a pipe 46 including a concentrating apparatus 47 returns the mother liquor to the concentrating mixer 44. In this embodiment, the compacting is carried out in the concentrating mixer 44 and there are obtained in the run sugar yields of about 90%, which is a considerable increase over conventional yields. A pipe 48 feeds at the end of the crystallization the cooked mass from the mixer 44 to a conventional turbine 49 which separates the sugar at 50 and the residue at 51. Moreover, one or more cooling mixers could be disposed between the concentrating mixer 44 and the turbine 49 so as to still further increase the crystallization yield.

The manner of carrying out the invention shown in Fig. 4 comprises withdrawing a fraction of the cooked mass in the course

of mixing (crystallization by cooling) and returning the mother liquor resulting from the turbining of this fraction to the same mixing stage.

The cooked mass issues from the conventional cooking apparatus 60 after compacting and is supplied by way of a pipe 61 to an assembly of mixers constituted by two mixers in series relation, 62 and 63, which are interconnected by a pipe 64. The cooked mass starts to cool in the mixer 62 and then passes while still sufficiently fluid into the mixer 63 where it finishes its cooling and crystallization. A branch pipe 65 connected to the pipe 64 withdraws a part of the cooked mass and supplies it to a turbine 66 where it is centrifuged. The sugar is withdrawn at 67 and the residue is sent by way of a pipe 68 - if desired after having been diluted, refined, de-aerated, brought to a given pH and concentrated - to the mixer 63. The cooled cooked mass leaves the mixer 63 at the end of crystallization and is supplied by way of a pipe 69 to a turbine 70 where the sugar is withdrawn at 71 and the residue at 72.

In this manner of carrying out the invention, there is withdrawn at 65 a sufficient amount of cooked mass for obtaining at the end of crystallization a sugar content which is less than 45% of the weight of the cooked mass and preferably 25-40% of this mass, which permits the maximum reduction in the temperature of the mass at the end of mixing and consequently a more complete crystallization without adversely affecting the subsequent turbining, since the mass will remain sufficiently fluid and will comprise regular crystals.

Note that as in the preceding embodiment, 100% of the cooked mass issuing from the mixer 62 could be turbined at 66. In this case, sugar crystals from the pipe 67 or from another stage are added so as to reconstitute a cooked mass in the mixer 63.

In this way, it is also possible to reduce the number of runs owing to the improved crystallization yield. It will be obvious that each run is carried out in accordance with the invention.

It is also possible to withdraw a fraction of the cooked mass between the mixer and the turbine placed at the end of cooling stage and to return the separated residue obtained in the rest of the cooked mass in a waiting mixer before the final turbine operation.

In the manners of carrying out the invention shown in Figs. 2-4, it is possible, if desired, to introduce in the mixer in addition to the residue from the turbining of the fraction removed from the cooked mass, a residue from a previous stage of manufacture which has a lower purity and which was or was not previously diluted, refined, heated and concentrated. This is possible owing to the very favourable conditions of crystallization afforded by the process of the invention. This recycling of the residue of lower purity than the residue into which it is introduced permits increasing the ratio $\frac{\text{non-sugar}}{\text{water}}$ of the residue in course of cooling and this still more increases the crystallization. This residue of lower purity can even be molasse in the crystallization cooling stage of the last run or the next to the last run, since the residue obtained at this stage has not been subjected to as much cooking as in conventional processes and consequently does not have as high a viscosity; moreover, it contains a smaller amount of fine crystals in suspension, this molasse can also have an origin different from that obtained in the plant employing said process.

For reasons of simplicity, manners of carrying out the process of the invention in a discontinuous or intermittent manner have been described with reference to Figs. 2-4. However,

it will be understood that it is perfectly possible to operate in a continuous manner, if desired, without departing from the scope of the invention. In this case, it suffices to remove the fraction of the cooked mass continuously and to continuously return the residue from which the crystals have been removed to the required stage.

Owing to a judicious control of the operations, the process of the invention permits the elimination of any cooking (crystallization by evaporation in a cooking apparatus) of a juice whose purity is lower than about 82. How, it is known that it is just these cookings which are the longest, the most delicate to carry out and the most detrimental owing to the changes they create in the sugar and in the non-sugars. Consequently, it is possible to limit all the operations to one or two runs and to reduce the total cooking time to less than about half the usual time.

These remarkable results are possible since, in the process of the invention, an artificial or non-artificial cooked mass can be obtained at any point of the process which, for a given fluidity, has a residue having a migh higher concentration of sugar than in conventional processes. It is possible to obtain a ratio $\frac{HS}{water}$ much higher than that usually obtained and this has for result an improved crystallization during the series of operations and consequently a possible reduction in the number of runs.

Therefore, the process of the invention results in an important saving in fuel,,an improved utilization of the evaporation and cooking or boiling units, a very improved sugar yield of the latter, a reduction in man power and higher productivity. The turbining, heating and concentrating operations in the process of the invention are very easily carried out in conventional apparatus or in merely improved apparatus of sugar making

plants and sugar refineries and are very economical owing to the considerable saving in equipment afforded by the process of the invention.

Although specific examples of manners of carrying out the invention have been described with reference to the drawings, it must be understood that the scope of the invention is not intended to be limited thereto.

Thus, a particularly interesting manner of carrying out the invention concerns the continuous crystallization by concentrating without cooking a sugared liquid mixed with sugar crystals followed by a cooling and then a turbining of the mass; this manner of carrying out the process eliminates the continuous or discontinuous cooking in a cooking apparatus where a part of the water contained in the sugared liquid is evaporated in the presence of sugar crystals. In this case, the sugared liquid to be refined is concentrated without creating crystallization, preferably in rapid evaporators, a given amount of sugar having a given grain size is intimately admixed with the liquid leaving the evaporator, and a part of the mass is thereafter withdrawn in the manner indicated hereinbefore. The series of operations mentioned hereinbefore can be effected continuously in a manner which can be fully automatic.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. Process for the crystallization of sugar from syrups from sugar works and sugar refineries of the type comprising at least one series of operations including a plurality of stages selected from the group consisting of a concentrating stage, a cooling stage and a turbining stage, said process comprising withdrawing at least a part of the mass containing sugar crystals at one moment of the process selected from the concentrating and cooling stages and the passages between the concentrating, cooling and turbining stages, withdrawing the sugar crystals contained in said part and returning the sugar liquid from which sugar crystals have been removed to one of said concentrating, cooling and turbining stages so as to admix it with the rest of said mass.

2. A process as claimed in claim 1, wherein the part of the mass withdrawn is between 25 and 75% of the total mass.

3. A process as claimed in claim 2, when said part is 30-50% of the total mass.

4. A process as claimed in claim 1, wherein the sugar crystals contained in said part are removed by a centrifuging operation.

5. A process as claimed in claim 1, wherein said part of the mass is withdrawn in the course of a concentrating stage and the sugar liquid from which sugar crystals have been removed coming from said part is returned to the same concentrating stage so as to mix it with the rest of the mass.

6. A process as claimed in claim 1, wherein the pH of said sugar liquid is modified before returning it to the rest of the mass.

7. A process as claimed in claim 1, wherein said part of the mass is withdrawn at the end of the concentrating stage.

8. A process as claimed in claim 1, wherein said part of the mass is withdrawn between a concentrating stage and the consecutive cooling stage, and the sugar liquid from which sugar crystals have been removed is returned to the rest of the mass at said cooling stage.

9. A process as claimed in claim 8, wherein a concentrated sugar-containing residue having a purity lower than that of said sugar liquid is also introduced in the mass from which a part of its sugar has been removed.

10. A process as claimed in claim 1, wherein said part of the mass is withdrawn in the course of a cooling stage and the sugar liquid from which sugar crystals have been removed is returned to the rest of the mass at the same cooling stage.

11. A process as claimed in claim 10, wherein a concentrated sugar-containing residue having a purity lower than that of said sugar liquid is also introduced into the mass from which a part of its sugar has been removed.

12. A process as claimed in claim 11, wherein said residue is molasse.

13. A process as claimed in claim 1, wherein said sugar liquid from which the sugar crystals have been removed is concentrated before it is returned to the rest of the mass.

14. A process as claimed in claim 1, wherein a concentration of the mass under a pressure lower than atmospheric pressure is effected in the course of said cooling stage.

15. A process as claimed in claim 1, wherein said part of the mass is withdrawn continuously and the sugar liquid from which sugar crystals have been removed is continuously returned to the rest of the mass.

16. A process as claimed in claim 1, wherein the crystals are removed from the whole of the mass, the sugar liquid from which sugar crystals have been removed is concentrated, and sugar crystals are added thereto so as to continue the crystallization at the stage to which said sugar liquid is returned.

17. A process as claimed in claim 1, wherein the crystallization of the sugar in said syrup is effected in one run.

18. A process as claimed in claim 1, wherein the crystallization of the sugar in said syrup is effected in two runs.

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Fig. 1

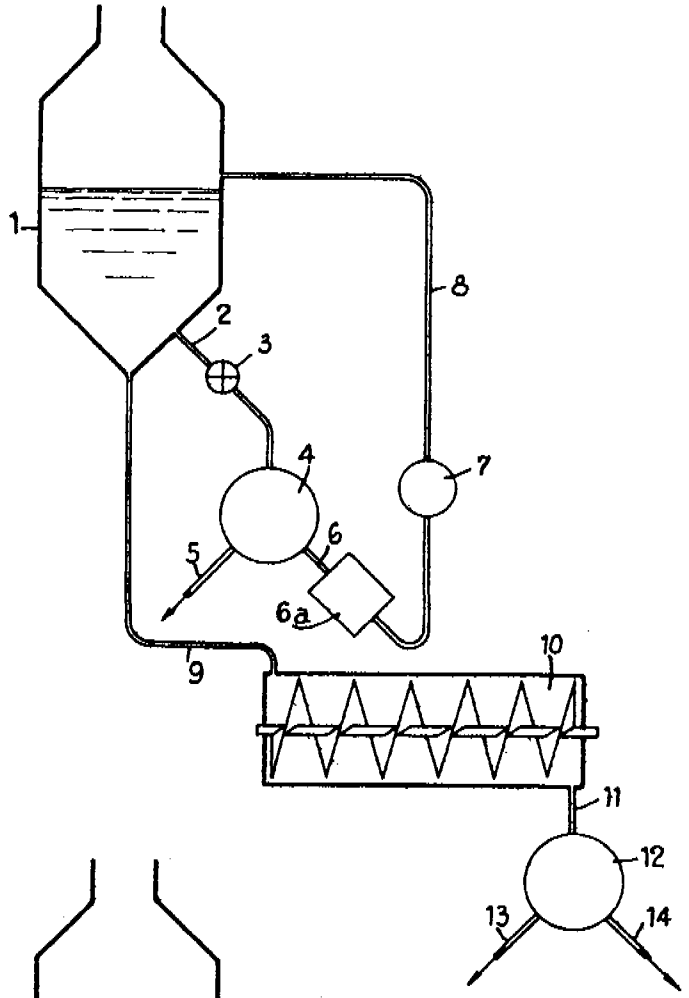

Fig. 2

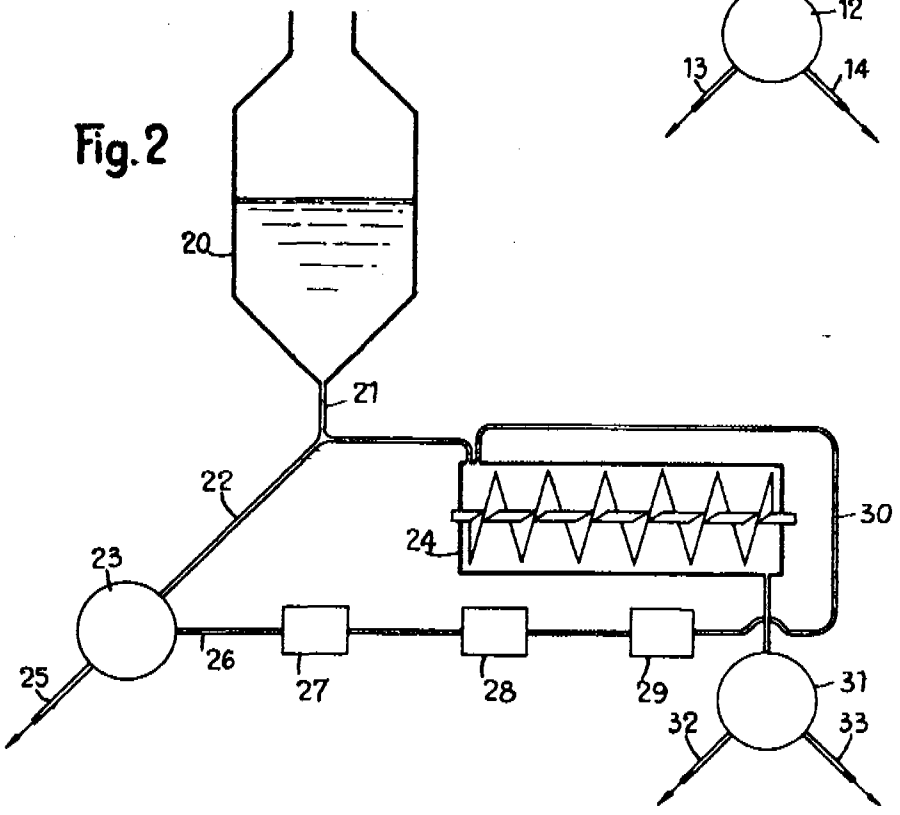

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Fig. 3

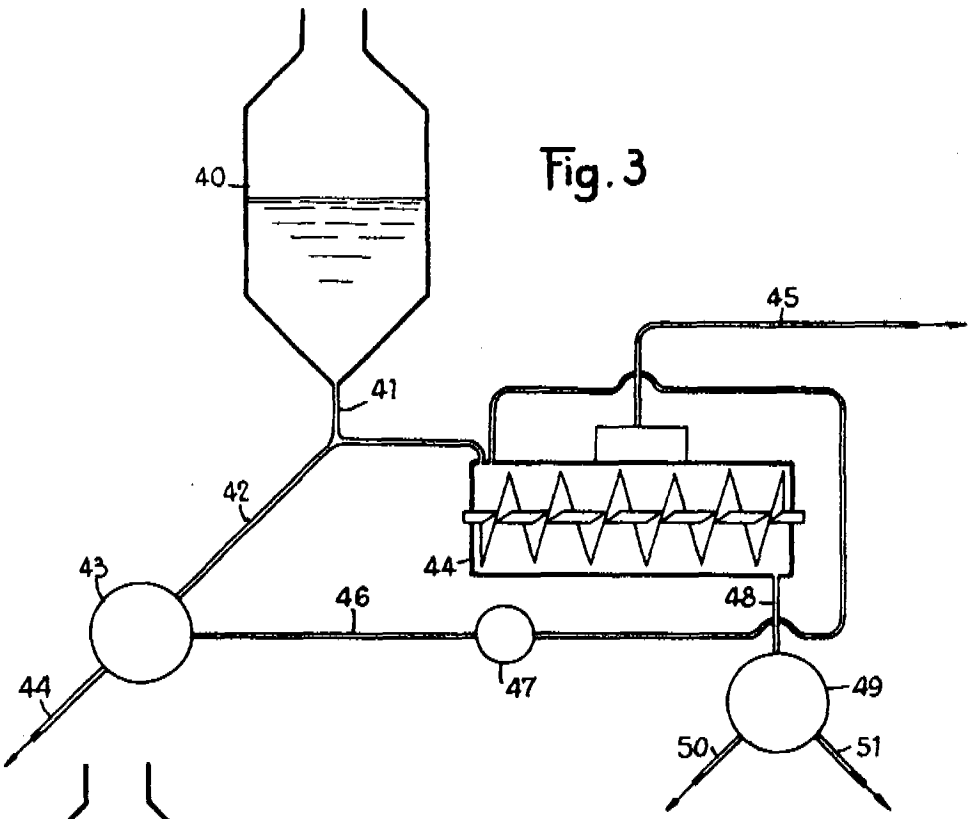
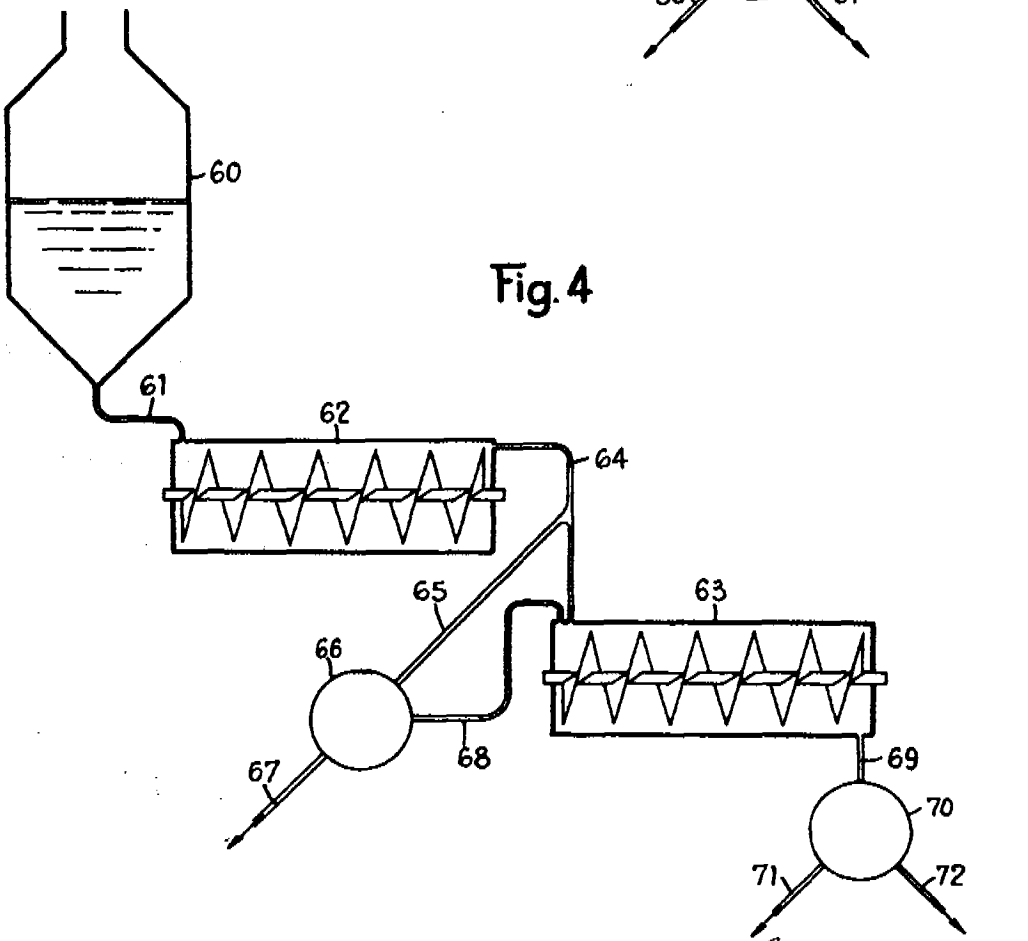


Fig. 4



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United States Patent [19]

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[11] **4,164,429**

[45] **Aug. 14, 1979**

[54] **PROCESS AND INSTALLATION FOR THE PRODUCTION OF SELECTED CRYSTALLIZATION SEEDS FOR USE IN A SUGAR REFINERY**

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[73] Assignee: **Fives-Cail Babcock**, Paris, France

[21] Appl. No.: **860,043**

[22] Filed: **Dec. 13, 1977**

[30] **Foreign Application Priority Data**

Dec. 14, 1976 [FR] France 76 37581

[51] **Int. Cl.**[2] **C13F 1/02**; C13F 1/06

[52] **U.S. Cl.** **127/15**; 23/301; 127/16; 127/19; 127/60; 127/61; 127/62; 422/245; 422/253

[58] **Field of Search** 127/15, 16, 19, 60, 127/61, 62, 24; 23/301; 422/245, 253

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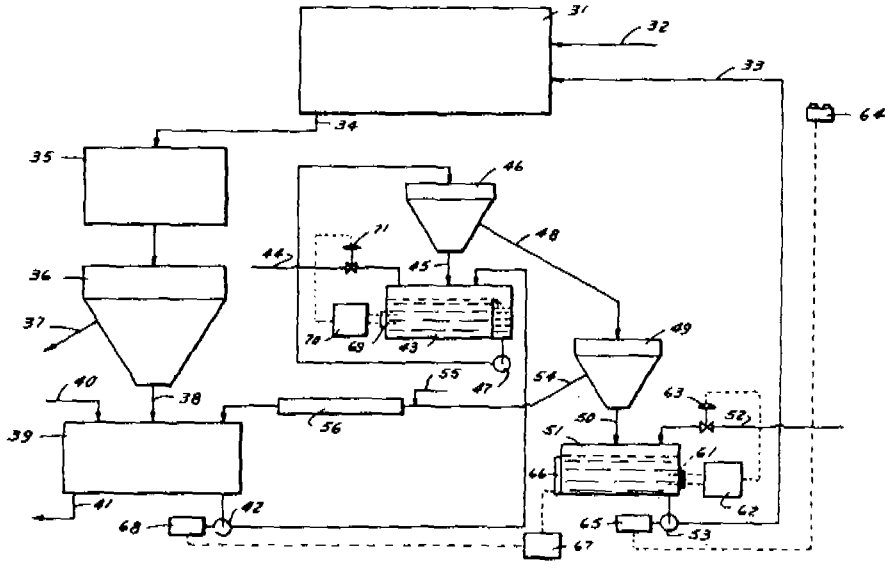
Primary Examiner—Sidney Marantz
Attorney, Agent, or Firm—Kurt Kelman

[57] **ABSTRACT**

Process, centrifuge and installation for producing crystallization seeds, as for the sugar industry. A suspension of crystals produced from the seeded solution is subjected to a scheme of centrifugal separations whereby seeds falling within a predetermined size range are obtained for seeding the solution.

10 Claims, 4 Drawing Figures



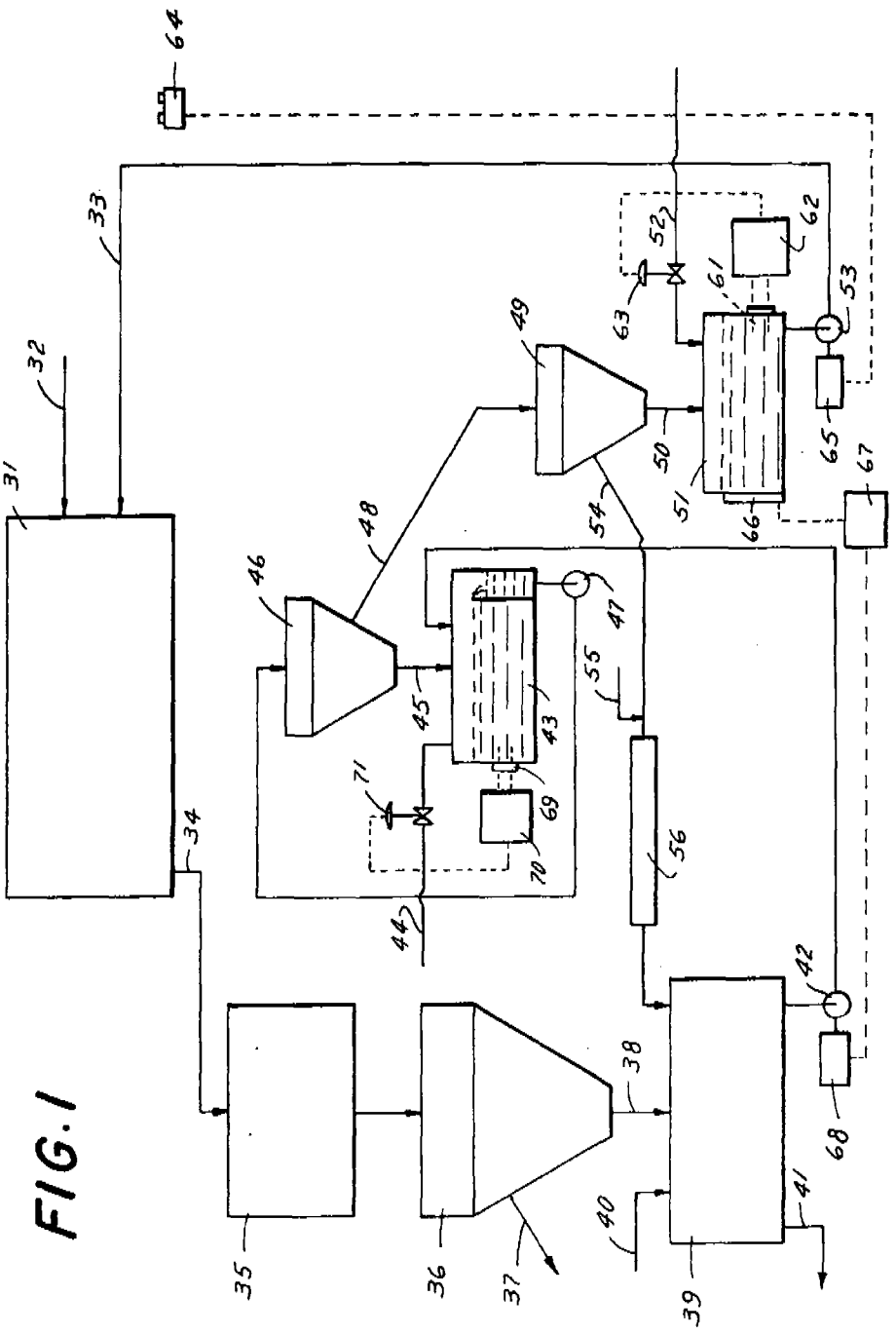

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FIG. 1

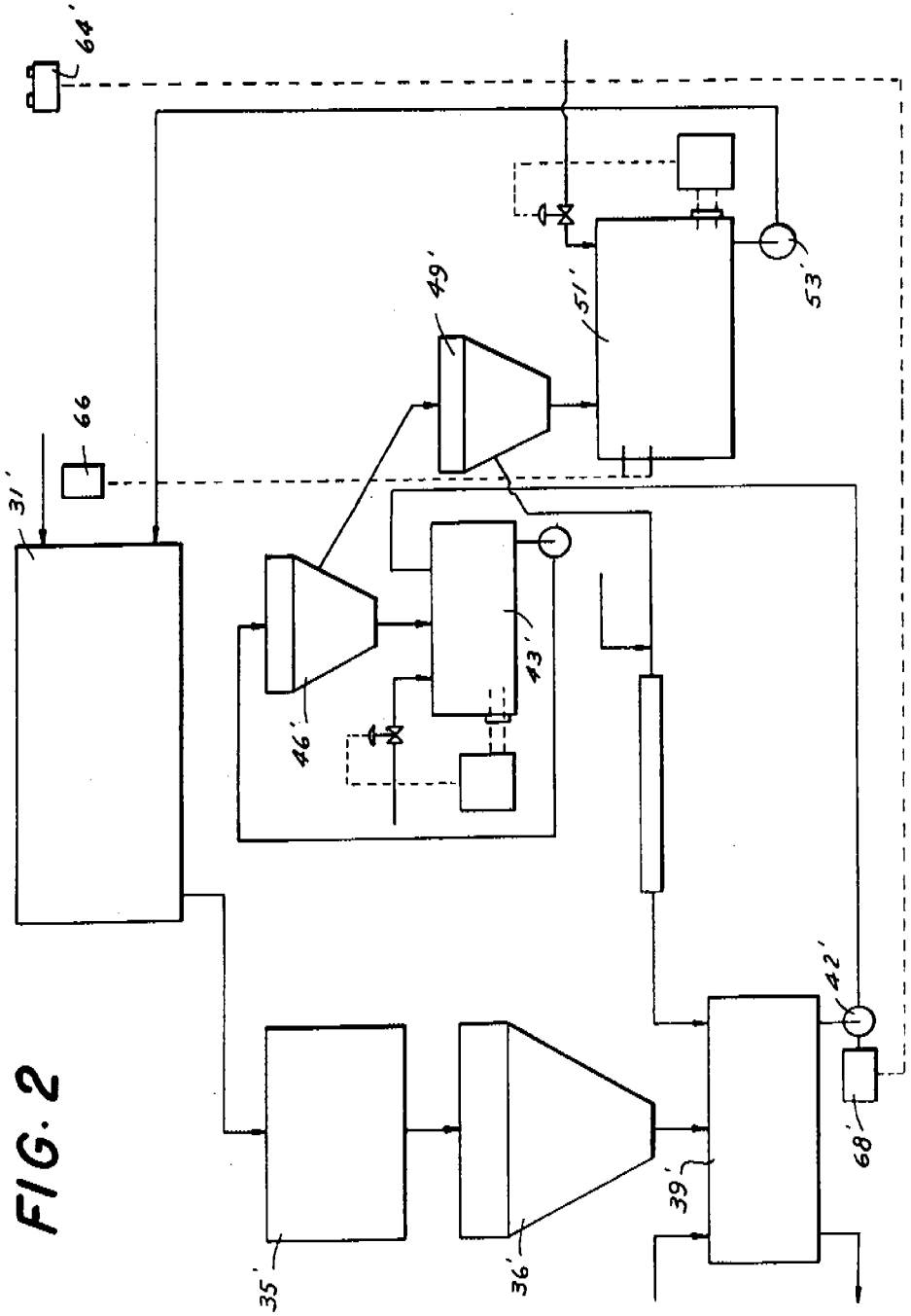

FIG. 2

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FIG. 3

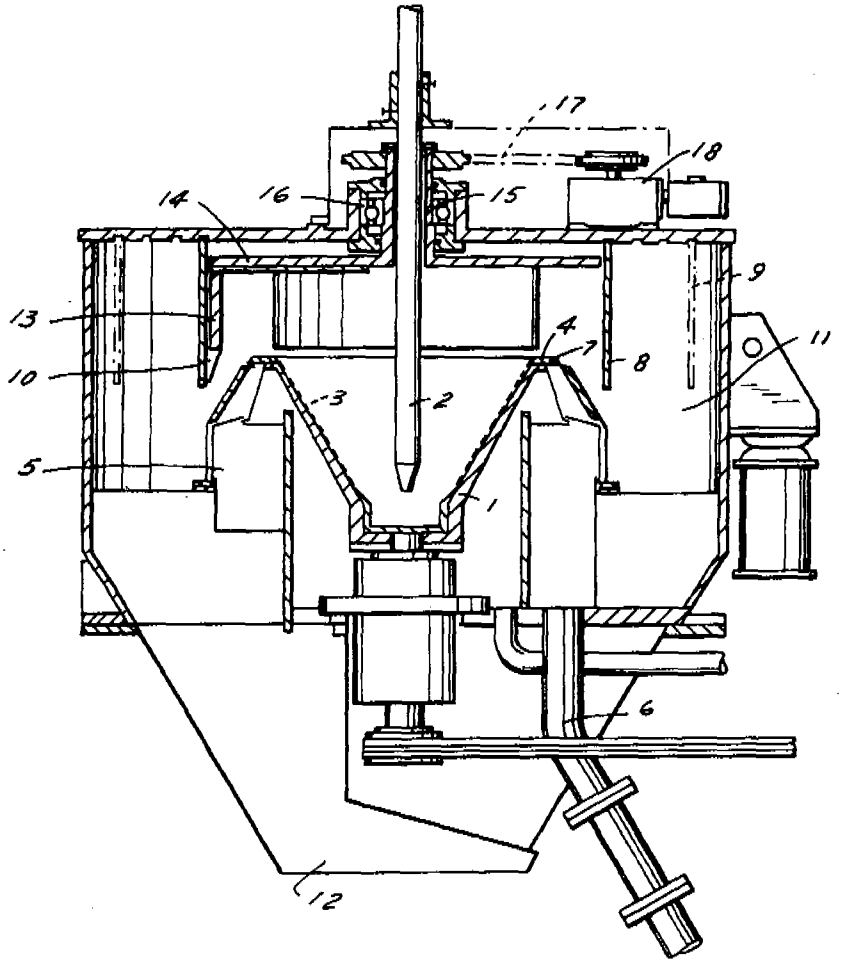
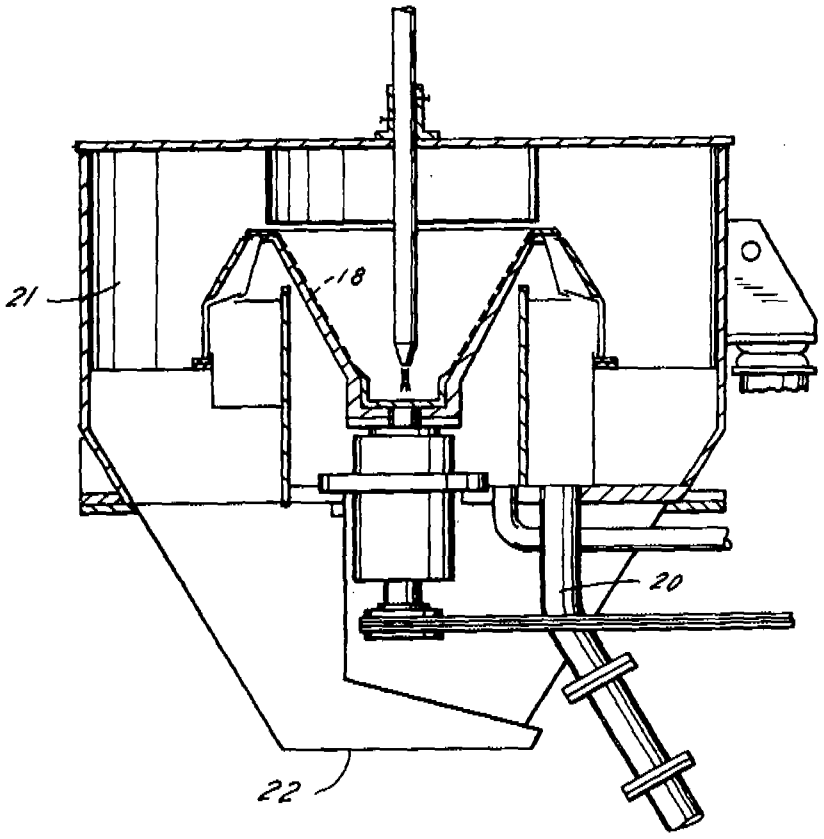


FIG. 4



PROCESS AND INSTALLATION FOR THE PRODUCTION OF SELECTED CRYSTALLIZATION SEEDS FOR USE IN A SUGAR REFINERY

A crystallizing apparatus, which has as its object the extraction of the mother liquor and the production of crystals of a predetermined dimension, must initially be fed with a suitable number of crystallization seeds to assure the realization of these conditions. When the apparatus is for continuous production, the number of seeds delivered must be proportional to the output of the apparatus.

This first crystallization phase, which consists of the introduction of seeds and the beginning of their growth is difficult to effectuate, considering the very small size of the introduced seeds and the risk of significant variations in the initial crystal population by local remelting of fines (crystals of very small dimensions) or by agglomerations.

This risk is particularly high in the last strike of crystallization of a sugar refinery, considering the low purity of the treated molasses, their high viscosity and the correspondingly low speed of crystallization.

Frequently, in discontinuous crystallization, this difficulty is reduced by effectuating the operation in two stages:

In the first stage, starting with crystallization seeds, one obtains crystals of a sufficient size to be certain that they will retain their identity in the following operations (of the order of 100 to 180μ). For this stage, one uses molasses of higher purity (for example molasses of the first strike or refinement instead of the molasses of the second strike for a scheme of three strikes) and one effectuates the operation starting with an excess of seeds and by producing partial remelts in the course of the cycle to restore them empirically to the envisaged crystal population; this manner of operation, however, does not avoid the existence of insufficient crystal populations, leading to the formation of fines in the second stage of crystallization.

In the second stage, the massecuite obtained in the preceding stage, which is called footings, serves as seeds for the continuation of the crystallization operation, the crystallizer having been fed by the normal molasses to extract (in the preceding case, the molasses of the second strike).

This manner of proceeding has the disadvantage that it requires the introduction of relatively pure molasses for the last strike of crystallization (to constitute the footings), hence a less favorable extraction of molasses is attained at the crystallization plant, all other conditions being equal. On the other hand, such a process is difficult to reproduce in viable fashion in an entirely continuous process, considering the previously indicated difficulties for controlling the first part of the crystallization (growth of the seeds to 100 to 180μ).

The invention has as object a process and installation for the continuous production crystals in a narrow range of dimensions which, when suspended at a constant content in a saturated solution, permits feeding a crystallization apparatus with a controllable population of crystals substantially proportional to the volume of the solution to be treated.

It is well known that, for an optimum extraction of the molasses, it is necessary to have a high crystal population to increase the surface of crystallization and to reduce the average distance between crystals; on the other hand, the smaller crystals must be big enough not to run the risk of remelting in the treatment cycle and to limit the granulometric dispersion of the resultant sugar. These two imperatives require lower and upper limits in the range of the granule sizes of the crystal products according to the invention.

The process of the invention uses the crushing and screening of crystals in a humid state after which they are suspended in a saturated solution to form a magma.

The first selection is made in a first drier by passing the magma over a screen with large openings. The liquid phase entraining the crystals of small size is directed to a second drier which effectuates the second selection with a screen having smaller openings.

The oversized crystals from the first drier are crushed and then recycled. The oversized crystals from the second drier constitute the selected crystals and are suspended in a saturated solution to be utilized as crystallization seeds. The molasses from this drier containing fines is placed into a state of under-saturation by the addition of water and, after heating, is recycled.

Even if a dispersion is produced in the growth of the crystals in the crystallizer, it is impossible, due to the process of the invention, to obtain fines passing through the screen of the drier with the molasses, which reduces the losses in the last stage of crystallization.

The crushing of the sugar may be effected by any suitable apparatus but for the oversized crystals ejected at high speed during the first drying, which must be crushed, one will preferably use crushing by projection. A device is added to the first drier to assure this function; it is constituted essentially by a receiving metal sleeve which is constantly cleaned by rotating wipers so disposed that the crystals are forcefully projected and subjected to an impact capable of breaking them. This manner of crushing has a selective action. In effect, the crystals are slowed down in their trajectories by the friction in the air. This slowing down is the more pronounced as the specific surface of the crystal is large and, therefore, its dimension is small. Starting with a given initial speed and the selected length of the trajectory, it is possible to collect the small crystals without breaking them while the large crystals retain an impact speed capable of causing their rupture.

The preparation of the magma which is to be subjected to the two dryings and of the magma for seeding is made in mixers which are advantageously provided with control systems for the content of crystals in the magma and level controls.

The invention has the following advantages:

No delivery to the continuous crystallization apparatus of a solution richer than that to be extracted.

Easy management of the crystallization starting with an optimum proportion of seeds permitting to obtain the best conditions of speed and extraction for a given growth coefficient.

The possibility of obtaining crystals of a size superior to the cut-off of separation by the centrifugal drying, thus permitting avoidance of the enrichment of the dried mass.

The following description refers to the accompanying drawings which illustrate the invention and wherein:

FIG. 1 is a schematic view of an installation for carrying out the process of the invention applied to a continuously operating crystallizer;

FIG. 2 is a schematic view of another installation for carrying out the process of the invention in the case of its application to a batch crystallizer;

FIG. 3 is an axial section of a drier for use in the installation of FIGS. 1 and 2; and

FIG. 4 is a section of another drier used in the installation of FIGS. 1 and 2.

The schematic view in FIG. 1 shows the principle of a continuous crystallization of the third strike of a sugar refinery, with continuous delivery of selected crystallization seeds. It is understood that the details of the schematic view used for the understanding of the process in no way limit the scope of the process of this invention to sugar refining, nor is this process limited to the nature of the treated product or the type of apparatus described herein. To indicate the general character of the process, the sugar refinery terms are indicated in parentheses in the following description to exemplify the generic terms.

The schematic view of FIG. 1 shows a continuous crystallization apparatus 31 (continuous vacuum pan of the third strike) fed at 32 with a solution to be extracted (poor molasses of the second strike) and at 33 by a suspension of crystallization seeds (magma 3). in the apparatus, the solution is maintained at super-saturation by evaporation under vacuum, which leads to crystallizable material collecting on the crystallization seeds, which grow. The suspension of crystals (massecuite of the third strike) is extracted at 34. The crystallization is followed by cooling in a mixer 35. The cooled product from mixer 35 is dried in a continuous centrifugal drier 36 which produces, on the one hand, a liquid phase 37 (molasses) and, on the other hand, crystals 38 (sugar from the third strike). These crystals fall into a mixer 39 whereinto a purer solution (low purity molasses of the second strike) is delivered at 40 to form a suspension (magma 2). This suspension is recycled at 41, either to obtain refined crystals or to serve as crystallization seeds at a stage of increased purity (continuous vacuum pan of the second strike). A part of the suspension is removed by a metering pump 42 to be directed to a mixer 43 and there to be mixed with a solution delivered at 44 and of the same nature as that utilized in the previously mentioned mixer, and with crushed products 45 coming from a continuous centrifugal drier 46 which will be described hereinbelow with reference to FIG. 3. This drier is designed to assure the impact crushing of crystals which are too large to pass through the openings of the screen of the drier.

In the case of sugar refineries where a remelting of the sugar of the third strike (in the particular case of beet sugar refineries) is effected directly, a controlled output of sugar crystals coming from drier 36 is introduced directly into mixer 43 before remelting.

The suspension obtained in the mixer 43 is recycled by a metering pump 47 to the drier 46. The crystals passing through the openings of the screen of this drier are removed at 48 with the liquid phase and are fed to a second continuous centrifugal drier 49 represented in FIG. 4. The crystals whose dimensions are larger than those of the openings of the screen of the drier 49, which are smaller than those of the screen of the drier 46, are removed at 50. These are the crystals whose dimensions are encompassed by the fixed range. They fall into a mixer 51 which receives at 52 a solution of the same purity (molasses of the second strike) as that whose extraction by crystallization is to be assured. The thus constituted magma (magma 3) is recycled by a metering pump 53 to be injected into the continuous crystallization apparatus (vacuum pan of the third strike). The crystals of dimensions smaller than the openings of the screen of drier 49 contained in the output 54 of the drier are remelted by injection of a small proportion of water at 55, are reheated at 56 bringing the suspension to below saturation; the obtained solution is recycled to the mixer 39.

In the industrial realization of this process, the management may be facilitated by an automatic control. By way of non-limiting example, a preferred control will be described.

The content of crystallization seeds in the magma in mixer 51 is measured by a conductivity meter 61. The measurement is transmitted to a control 62 which actuates a valve 63 disposed in conduit 52 in such a manner as to maintain this content equal to a fixed operating value.

By a manual control 64 acting upon the pump speed regulator 65, the output of the suspension of seeds (magma 3) extracted from mixer 51 is so controlled that a desired crystal population is obtained in the crystallizer. A level meter 66 acting upon the control 67 controls a governor 68 which adjusts the output of the pump 42 which removes the suspension of crystals (magma 2) from the mixer 39. A conductivity meter 69 acting upon the control 70 regulates the output of a valve 71 disposed in the conduit 44 so as to maintain the content of crystals in the magma in the mixer 43 constant.

FIG. 2 shows an installation which is analogous to the preceding one for a crystallization apparatus 31' which operates discontinuously. In this case, the output of the pump 42' is adjusted by the speed governor 68' remote-controlled by a switch 64' so as to constitute a reserve of the seed suspension (magma 3). The level of the mixer 51' is recorded on level meter 66. The pump 53' is operated each time magma is required.

The drier 46 shown in FIG. 3 comprises a conical basket 1 rotating at high speed and to which the suspension of the product to be dried is fed through the delivery tube 2. The liquid phase passes through the screen 3 whose mesh size is selected to retain crystals of a size larger than that of the desired crystals which are entrained with the liquid passing through the screen. This product circulates over the wall of the basket and is then centrifugally ejected through orifices 4 disposed at the periphery of the basket. It is collected in a circular receptacle 5 whence it is removed by an output pipe 6. The solid phase retained on the screen is ejected practically at the tangential speed of the basket above the flange 7 to be projected against a cylindrical metallic sleeve 8. A certain number of sleeves of different diameters are provided to permit the length of the trajectory to be selected as a function of the desired size of the crushed product, the sleeve 9, in chain-dotted lines, being that of the largest diameter. The peripheral speed of the basket and the length of the trajectory are so selected that the largest projected crystals arrive at the sleeve with a speed such that they are broken by the effect of the impact and that the small crystals, moreover, slowed down by the friction in the air are not broken or are subjected only to a partial erosion. The products, although energetically dried, are sticky in the pulverized state; to avoid an accumulation of the products, which would modify the rupturing conditions of the crystals, the sleeve is cleaned by rotating wipers 10.

The number and the speed of rotation of these wipers are such that the impact conditions vary little. It would also be possible to use a rotating sleeve on which fixed wipers acted. The crushed products fall into the annular chamber **11** to be removed through orifice **12**. The wipers are mounted on supports **13** fixed in operating position on platform **14** which entrains them into rotation. The entrainment shaft **15** of this platform is hollow so as to permit passage of the delivery tube; it is journaled in bearing **16** integral with the cover of the drier and is rotated by transmission **17** and a reduction motor assembly **18**.

The drier **49** represented in FIG. **4** is of a conventional construction and will not be described in detail. It receives the dried product of the drier **46** which contains crystals whose dimensions are inferior to those of the openings of the screen of the latter. The screen **18** of the drier **49** has smaller openings. The crystals whose dimensions are inferior to those of these openings are removed with the liquid phase through pipe **20**. The larger crystals, which are selected to constitute the crystallization seeds, are collected in the tank **21** and removed through the orifice **22**.

By way of example, in the case of a third strike of a sugar refinery, the dimensional limits of the crystallization seeds may be fixed between 0.050 mm and 0.125 mm; the drier **46** will have a screen with meshes of 0.130 mm and the drier **49** a screen with meshes of 0.60 mm. If one allows a volumetric growth of **15** to **16** in the crystallization apparatus, the crystals of the massecuite product will have dimensions comprised between 0.125 and 0.315 mm and an average width of 0.220 mm.

I claim:

1. Process of producing crystallization seeds for seeding a solution of a crystallizable product, which comprises forming a suspension of crystals produced from the seeded solution in a fraction of the said solution, subjecting the said suspension to a first centrifugal separation wherein crystals whose size is superior to a predetermined maximal dimension are separated from the said suspension, subjecting the said suspension to a second centrifugal separation wherein crystals whose size is inferior to a minimal predetermined dimension are removed with the suspension, and seeding the said solution with the crystals separated from the suspension in the course of the second separation.

2. Process according to claim **1**, wherein the crystals removed with the said suspension in the second separation are dissolved and the obtained solution is recycled to the said suspension.

3. Process according to claim **1**, wherein the crystals separated from the suspension in the course of the second separation are mixed with another fraction of the said solution to form a seeding magma and the seeding magma is metered to the said solution.

4. Process according to claim **3**, wherein the crystallization seeds are produced continuously in the second separation and the crystals are fed continuously to the said solution for continuously crystallizing the product, and the output of the suspension subjected to separation is maintained proportional to the output of the solution of the crystallizable product, the percentage of crystals in the said suspension being maintained constant.

5. Process according to claim **3**, wherein the crystallization seeds are produced continuously in the second separation and the seeding magma is stored, the crystallizable product being crystallized discontinuously and batches of the stored seeding magma being fed to the said solution during crystallization.

6. Process according to claim **1**, wherein the crystals separated from the suspension in the course of the first separation are crushed and the crushed crystals are recycled to the said suspension being subjected to the first separation.

7. Process according to claim **6**, wherein the crystals are crushed by projecting them at high speed against an impact surface.

8. Installation for carrying out the process of claim **6**, comprising a first mixer for forming the said suspension of crystals, a first centrifugal separator arranged to receive the said suspension and having a screen to separate crystals, the mixer having an input receiving the crystals from the separator, a second centrifugal separator arranged to receive the suspension of crystals having passed across the screen of the first separator, the mesh size of the screen of the second separator being smaller than that of the screen of the first separator, and a second mixer arranged to receive crystals separated from the suspension in the second separator, the second mixer having an output for the crystals, and a variable output pump connected to the output for feeding the crystallization seeds to a crystallization apparatus for the solution.

9. Installation according to claim **8**, wherein each mixer comprises an element for measuring the crystal content of the product contained in the mixer, conduit means for feeding the suspension thereto, a valve in the conduit means, and a control controlling the valve in response to indications of the measuring element to maintain the content of crystals equal to an operating value.

10. Installation according to claim **8**, wherein the second mixer comprises a level control which controls the feeding of the first mixer.

* * * * *

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(54) Manufacture of sugar by crystallisation by cooling

(57) Sugar is manufactured by crystallisation by cooling, in several stages. Each of these stages envisages, in cascade formation, a concentration of the sugar solution, preferably in a vacuum, at a temperature of about 80°C, and then crystallisation obtained by cooling the solution slowly down to about 35°C. Crystallisation is preceded by the addition of microcrystals and followed by the removal of the crystals which have formed during the process.

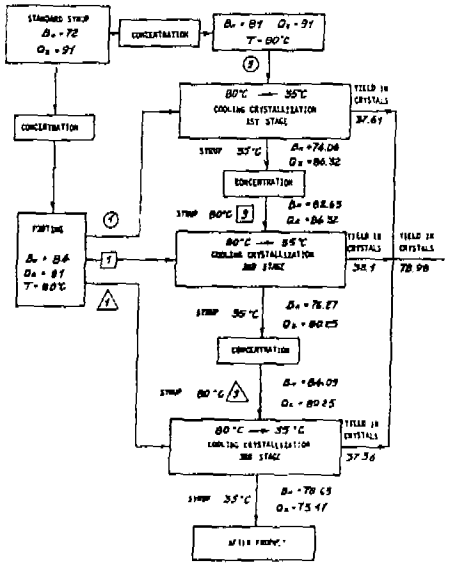

Fig 2

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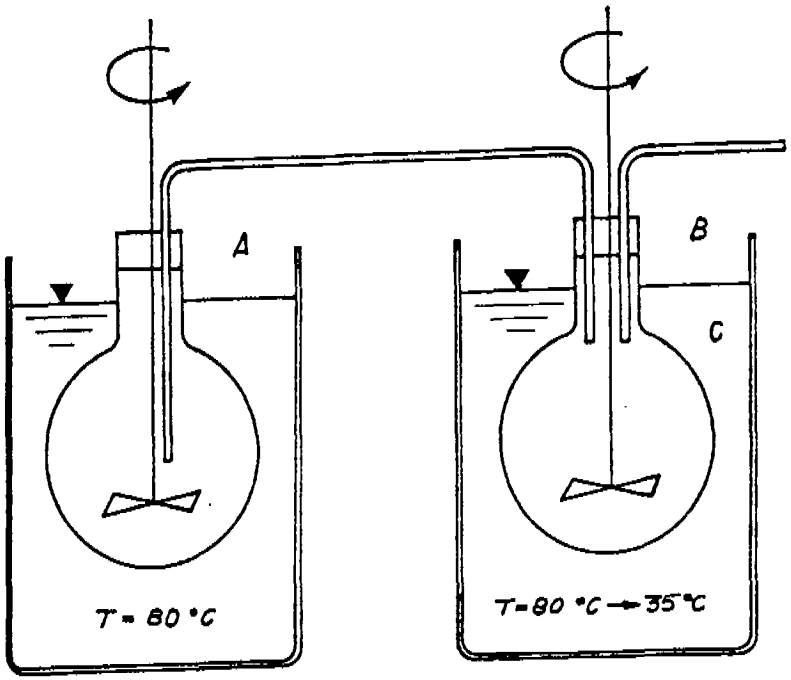

Fig. 1

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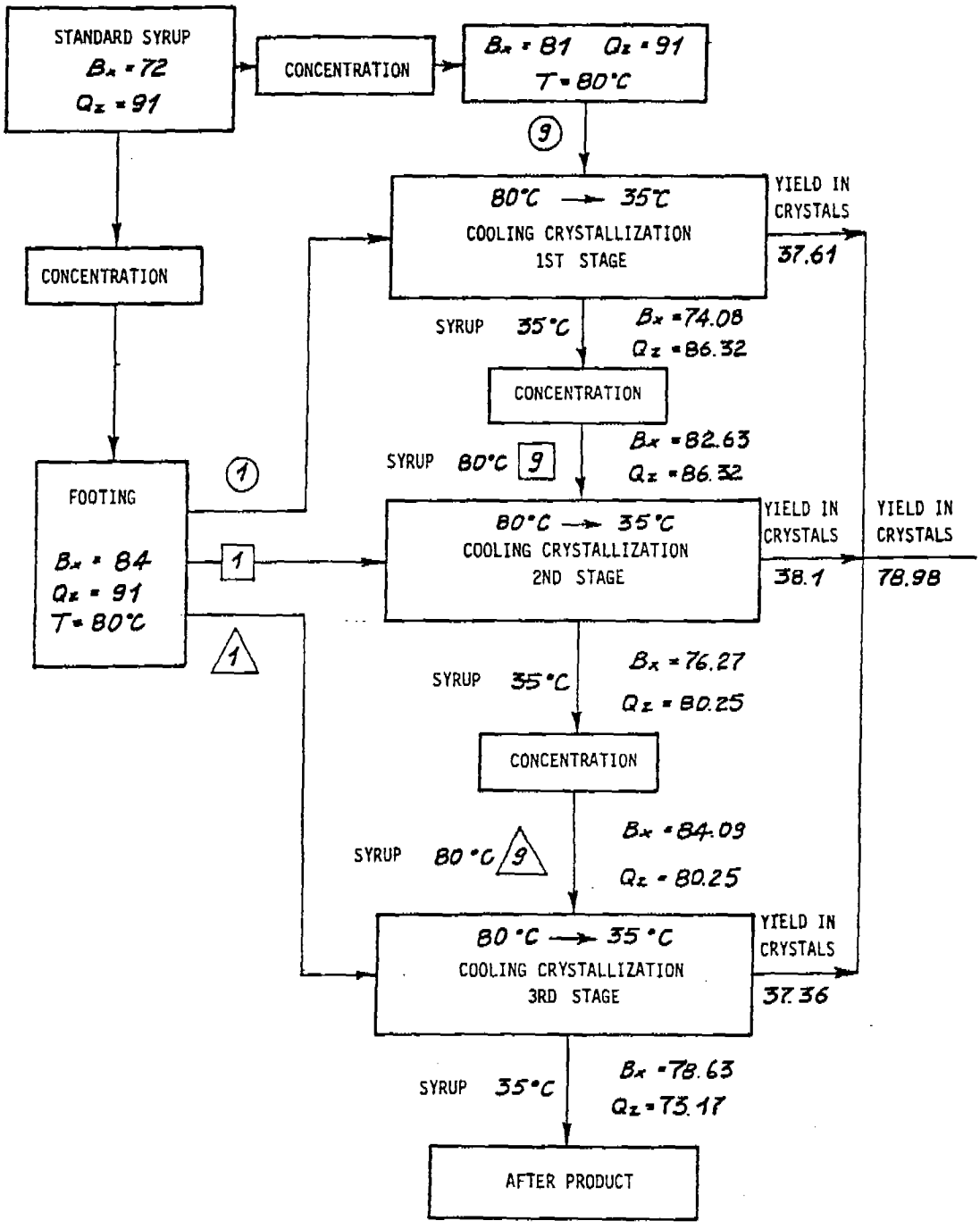

Fig. 2

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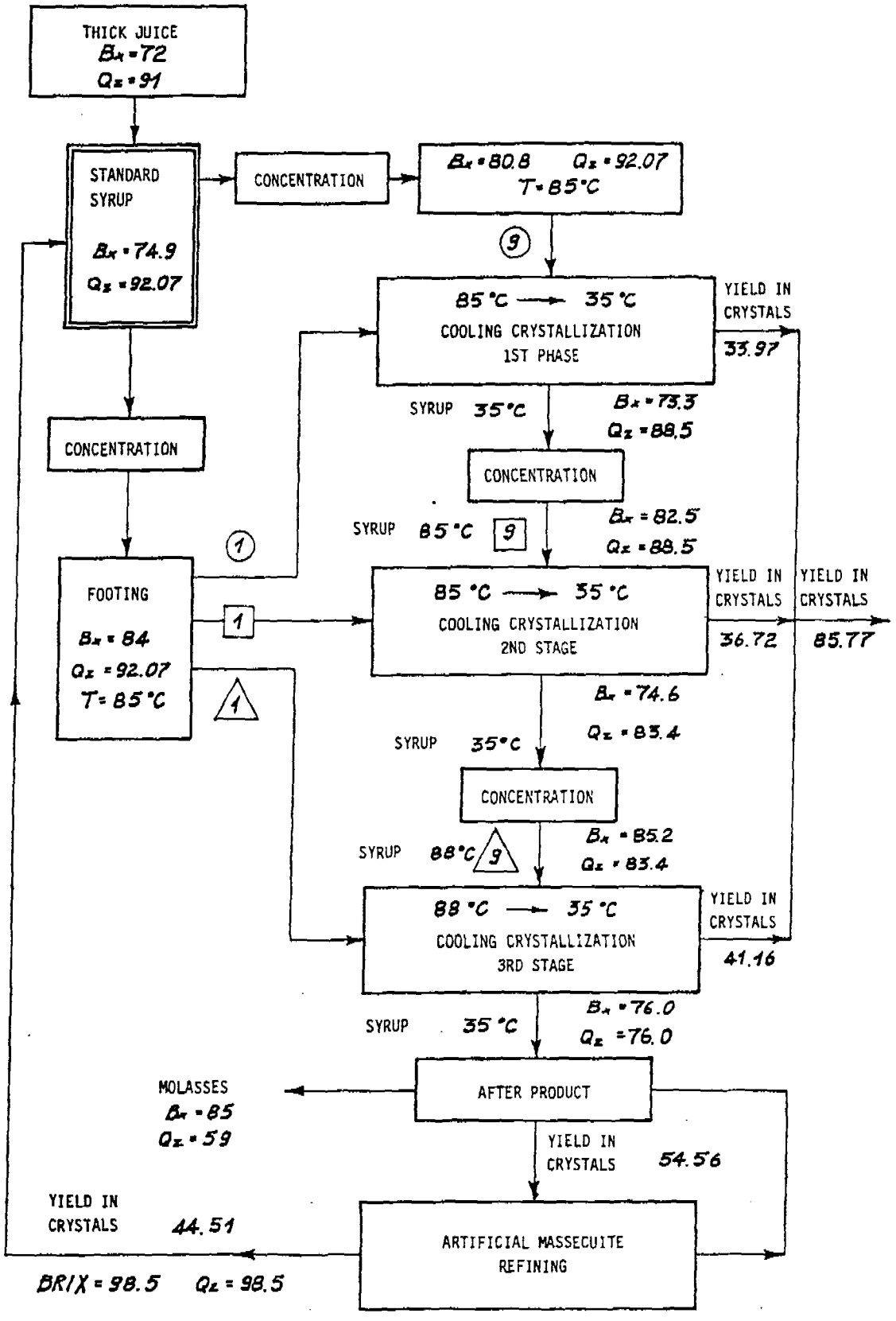

Fig. 3

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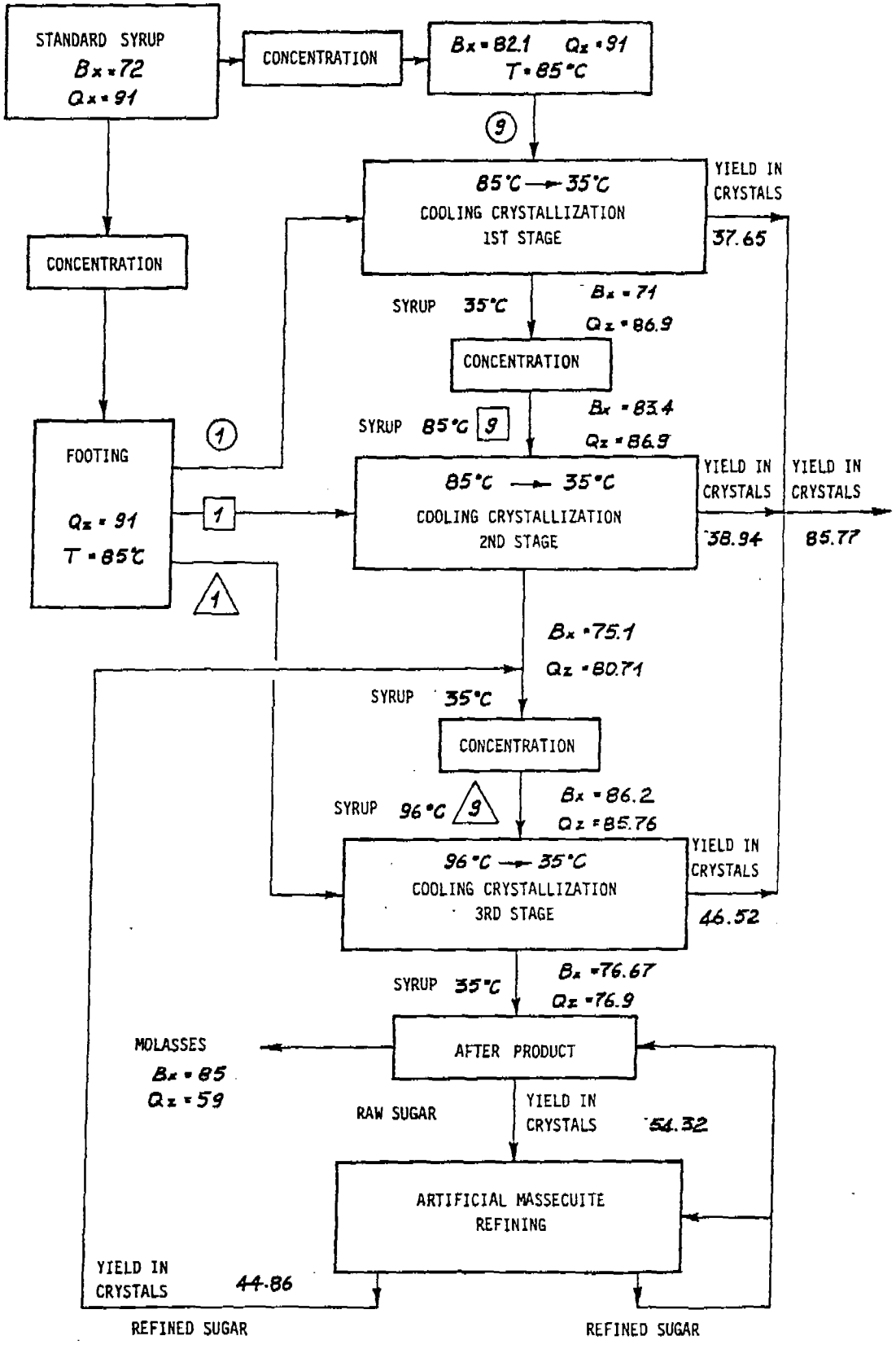

Fig. 4

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MANUFACTURE OF SUGAR BY CRYSTALLIZATION BY COOLING OF SUGAR SOLUTIONS

This invention relates to a sugar manufacturing process involving crystallisation by cooling.

In the manufacture of sugar the crystallisation stage is crucial to the production of sugar which is not undesirably discoloured. The more extreme the temperature, supersaturation and solution viscosity the more marked are the inclusions of discolouring matter and ash.

Known crystallisation processes do not yield in practice a sufficiently white sugar and although suggestions have been made concerning crystallisation by cooling (De Vries, Accinelli) no industrially viable procedure has yet been developed.

The present invention sets out to provide such a process.

The invention consists in a cooling crystallisation process for manufacturing sugar, comprising in sequence:

a) rapidly concentrating a starting syrup of BRIX value Bx between 65 and 75, and purity quotient Qz between 85 and 95 up to saturation at a temperature between 75° and 100° C;

b) mixing crystallisation nuclei with the concentrated syrup;

c) gradually cooling the syrup to promote growth of sugar crystals on the nuclei; and

d) centrifuging off the crystals formed by cooling.

Vacuum heat evaporators are preferably used to concentrate the starting syrup, so that it remains only briefly at high temperatures, and hence discolours to a minimal extent.

The crystallisation nuclei may be present in a footing to which the concentrated syrup is added e.g. at a footing: syrup volume ratio of 1:8 to 1:12, preferably 1:9 to 1:11. Alternatively, the crystallisation nuclei can be added as a slurry of powdered sugar. Cooling preferably takes place at a temperature gradient of about 10°C (e.g. 5°-15°C) per hour and for over about four hours (e.g. 2 to 6 hours), to terminate at 30°-40°C.

Most particularly, however, the invention provides a cooling crystallisation process for manufacturing sugar in which the sequence of stages defined above is repeated two or more times, and modified in that the starting syrup for second or subsequent repetitions is the mother liquor remaining after centrifuging of crystals from the next earlier such procedure.

Conveniently, the mother liquor from the final repetition of the four stages is subject to conventional crystallisation to produce sugar and molasses, and the sugar produced from such conventional crystallisation is incorporated into a starting syrup for one of the four-stage procedures.

The invention will be further described with reference to the accompanying drawings in which:-

Figure 1 is a diagram of an experimental device by means of which the invention can be practiced on a laboratory scale;

Figure 2 is a block diagram of a working process of the invention;

Figure 3 is similar to Figure 2 but modified in operational features; and

Figure 4 is again similar to figure 2, but differently modified in operational features.

For the purpose of laboratory-scale tests, the simple device shown in figure 1 was used.

The abbreviations used below in the description have the following meanings:-

"Brix" is the dry-substance contents of the solution;

"Qz" is the quotient of purity, equal to the ratio of the sugar substance contained in the solution to the total dry substance (sugar and non-sugar) x 100;

"CS" is saturation coefficient of the impure solution as compared to the pure solution;

"BETA" is the supersaturation coefficient;

"C.V." is the variability coefficient;

"M.A." is the mean opening.

First Crystallisation

A first series of tests was carried out on portions of standard factory-produced syrup having Brix = 72 and Qz = 91, which was first concentrated beforehand up to Brix = 81 and kept at a temperature of 80° in storage flask A. In these conditions, considering the CS of 1.05, the syrup is saturated.

Part of the same standard factory syrup was concentrated in flask B until it reached Brix = 84 at a temperature of 80°C. Assuming a CS of 1.05, in these conditions the syrup has a supersaturation coefficient BETA = 1.23. A small quantity of the seeding solution normally used for industrial production (one or two drops) was then added to crystallisation vessel B.

Once the formation of the grains of sugar had been ascertained, slow cooling was started, with slow addition at the same time of syrup from A to B by means of a suction device. The ratio of the quantity of footing initially added to B to the feed syrup was 1:9.

Feeding was terminated after about 40 minutes and cooling after a further four hours. At the end of the crystallisation, the massecuite was centrifugated using a laboratory-scale basket-centrifuge, and the sugar was

carefully washed in order to remove completely the layer of adhering syrup. The syrup obtained had a Brix of 74, confirming the theoretical yield discussed below.

For comparison purposes, samples of sugar obtained by centrifugation of factory-produced massecuite made from the standard syrup were taken, and were washed in the same manner used for the samples of sugar obtained by cooling as above.

The comparision between the characteristics of average samples of both types of sugar is shown in Table 1. The difference between the two types of sugar in colour and grain size were noticeable by visual inspection.

Second Crystallisation

The drained syrup obtained from this first crystallisation with Brix = 74 at 35°C and Qz = 86 still contained a considerable quantity of potentially crystallizable saccharose.

Further crystallisation could be achieved by bringing said drained syrup from the first crystallisation to saturation at a high temperature (for example 80°C), rapid evaporation and repeating the cooling crystallisation as described above. In order to effect this on a laboratory scale it was however necessary to

use mixtures of standard syrups and factory-produced green syrups so as to obtain a sample of syrup reproducing the syrup to be used in practice for the second cooling crystallisation.

Using the same device as shown in figure 1 and preparing the footing in exactly the same way as with the first crystallisation, the second cooling crystallisation was carried out to obtain a drained syrup having Brix = 76 and Qz of about 80, being effectively saturated at a temperature of 35°C.

Third Crystallisation

In order to have a sufficiently exhausted drained syrup to proceed with traditional low-product boiling, it was first necessary to carry out a third crystallisation, repeating the process as above, that is to say saturating again by rapid evaporation the drained syrup at 80°C and carrying out another cooling crystallisation. In this case again, in order to produce a sufficient quantity of the solution to be crystallised, it was necessary to prepare the mixture using factory-produced syrups and molasses.

The third crystallisation was carried out using footing prepared in the same way and maintaining, as in the

previous two cases, the 1:9 ratio of footing/feed/syrup. This third stage of cooling crystallisation was carried out to obtain a syrup with a Brix of about 78.5 and a Qz of about 73. With this type of draining syrup it is then possible to proceed with the final exhaustion to molasses using a traditional type of boiling.

The analytical characteristics of the three types of sugar produced are shown together in Table 2.

The colour and the ash content of the sugar obtained from the second and especially from the third crystallisation are in fact inferior to those of the first crystallisation stage because the feed syrup mixtures were factory-produced draining syrups which had previously undergone boiling at a high temperature. If syrups obtained from earlier cooling crystallisations were available, the ash content and the colour especially of crystals originating from the third crystallisation would be considerably lower. In three-stage crystallisation, the deepening of the colour during the three stages is very limited since the syrups do not remain at high temperatures for very long, unlike the syrups in traditional boiling.

Comparison of the C.V. values of the three sugars with the C.V. of factory-produced raw sugar indicates the

uniform size of the crystals obtained. Also, such crystals (obtained by cooling) are especially shiny because they have not undergone any particular thermal shock.

On the basis of the results obtained in the laboratory tests described above, a three-stage cooling crystallisation process was set up, as shown in Figure 2. On examining the Brix and Qz values of the various syrups it is seen that the crystallisation yields obtained make it impossible to exhaust the various syrups to a very satisfactory degree.

The crystallisation yield in the three stages varies between 37 and 38%, thus providing very fluid massecuites to make centrifugation and washing easy.

The total yield in crystals is of about 73% of the initial saccharose. The distribution of the percentage of crystals between the three stages is 45.6% for the first stage, 32.3% for the second and 22.1% for the third.

In the diagram of Fig. 2, neither the use of the sugar obtained by crystallisation of the low product nor the refining drain-off deriving from the centrifugal washing of the crystals have been considered.

In the diagram of Figure 3 the refining of the first-product sugar, its dissolving and addition to the second syrup, and the dissolving of 8% of the crystals during centrifugal washing is taken into account. These differences entail a different quantity of sugar to be crystallised in the various stages, and this requires different temperature distributions for the cooling.

In the diagram of Figure 4, the low-product sugar is sent after refining to the last cooling crystallisation stage, again with a dissolving of 8% of the crystals during centrifugal washing. The temperature distribution among the various stages is different again in order to achieve the best possible degree of exhaustion.

A comprehensive examination of the three diagrams shows that depending on the type of solution chosen, it is possible to achieve the planned degree of exhaustion in the three crystallisation stages based on temperature distribution.

Table 3 shows a smaller quantity of sugar dissolved during centrifugation (5%), a Qz of the thick juice of 90 instead of 91, and a hypothetical crystallisation diagram in four stages rather than three.

In order to evaluate the whole cooling crystallisation process from the point of view of energy consumption and plant engineering, the arrangement of Figure 3 was compared with the arrangement of a refinery and with that of a free-boiling white sugar process. The data obtained, and set forth in Table 4 shows that the energy balance is clearly in favour of cooling crystallisation as compared to a refinery cycle (energy consumption is more than halved). The comparison with a free-boiling white-sugar arrangement shows comparable energy consumption levels, but the sugar produced is of a much poorer quality from free-boiling. The total evaporating surface area is about four times smaller for cooling crystallisation than for the traditional method. The volume of the crystallisation vessels required for cooling crystallisation is about 1.5 times that for traditional crystallisation.

The advantages of the invention specifically described are

(a) as to starting material; thick juice can be made directly into commercial grade granulated sugar

(b) as to procedures; the crystallisation times need not exceed four hours, the temperature drop is variable in a range between say 80° and 35° to accomodate

alterations in feed and procedures, the energy requirements are less, and the evaporating surface is greatly reduced with a manageable increase in crystallisation volume

(c) As to product handling; the sugar is easily centrifugable and washable because of the fluidity of the mother liquor and the syrups do not discolour to the same extent as in the traditional processes

(d) as to the final product, the sugar is white, shiny and of good M.A. and C.V. values.

T A B L E 1

	First-product factory sugar	1st cooling crystallization
Colour in solution (MEC points)	19.3	5.3
Ash (MEC points)	27.8	8.3
Farbtype (MEC points)	11	4
Total MEC points	58.1	17.3
M.A.	0.57	0.61
C.V.	40	27

Comparison between characteristics of sugar obtained by cooling (1st stage) and first-product factory manufactured sugar.

TABLE 2

	1st cooling crystalliz.	2nd cooling crystalliz. *	3rd cooling crystalliz. **
Colour in solution (MEC points)	5.3	5.5	10.5
Ash (MEC points)	8.3	8.9	17.7
Farbtype (MEC points)	4	4	8
Total MEC points	17.3	18.4	36.2
M.A.	0.61	0.56	0.56
C.V.	27	27	29

Analytical characteristics of the three types of sugar produced on a laboratory scale in the three cooling crystallization stages.

* Artificial draining syrup obtained by mixing thick juice and factory-produced green syrup.

** Artificial draining syrup obtained by mixing thick juice with factory produced molasses.

TABLE 3

	Thick Qz	Temperature in degrees C							
		1st crystal. beg.	end	2nd crystal. beg.	end	3rd crystal. beg.	end	4th crystal. beg.	end
Remelting of raw sugar to thick juice	91	80	35	80	35	80	35	-	-
Refining of raw sugar, last stage, sol. 5% xx to white	91	80	35	80	35	101	35	-	-
As above but with sol. 8% xx	91	85	35	85	35	96	35	-	-
As above but in 4 stages	91	80	40	80	40	80	40	81	35
	91	80	40	80	40	80	40	92	40
Refining of raw sugar, remelting to thick juice, sol. 5% xx to white	91	80	35	80	35	87	35	-	-
As above but with sol. 8% xx	91	85	35	85	35	88	35	-	-
As above but in 4 stages	91	80	40	80	40	80	40	80	51
As above but in 3 stages, Qz d. 90	90	85	35	85	35	72	35	-	-
	90	85	35	85	35	85	52	-	-

Various possibilities for implementing cooling crystallization.

TABLE 4

	Crystallization by cooling	Traditional refinery cycle crystallization	Traditional free-boil. white-sugar crystalliz.
Total evaporating surface (sq.m.)	700	2700	2600
Crystallizing vessels requ.(cu.m.)	450	300	250
No. centrifuges needed (1000 kg each	10	12	10
Steam consumed (in t/d) for :			
a) Water to be evaporated	508.9	1408.7	601.1
b) Heating	113.5*	-	-

Comparison between cooling crystallization, a refinery cycle and a free-boiling white-sugar process.
Production of 1000 t/d sugar.

* Assuming that the draining syrups are heated by steam only; they could be heated at least partially using hot recirculation water.

CLAIMS

1. A cooling crystallisation process for manufacturing sugar, comprising in sequence:

(a) rapidly concentrating a starting syrup of Brix value Bx between 65 and 75, and purity quotient Qz between 85 and 95 up to saturation at a temperature between 75^{o} and 100^{o}C;

(b) mixing crystallisation nuclei with the concentrated syrup;

(c) gradually cooling the syrup to promote growth of sugar crystals on the nuclei; and

(d) centrifuging off the crystals formed by cooling.

2. A process as claimed in Claim 1 in which vacuum heat evaporators are used to concentrate the starting syrup.

3. A process as claimed in Claim 1 or 2 in which the crystallisation nuclei are present in a footing to which the concentrated syrup is added.

4. A process as claimed in Claim 3 in which the volume ratio of the footing to the syrup is between 1:8 and 1:12.

5. A process as claimed in Claim 4 in which the said ratio is between 1:9 and 1:11.

6. A process as claimed in Claim 1 or 2 in which the crystallisation nuclei are added as a slurry of sugar powder.

7. A process as claimed in any one proceding claim in which cooling takes place at a temperature gradient of about $10^{\circ}C$ per hour and is terminated when the temperature of the supersaturated solution achieved is 30°-$40^{\circ}C$.

8. A process as claimed in any one preceding claim in which cooling takes place over about four hours and is terminated when the temperature of the supersaturated solution achieved is 30°-$40^{\circ}C$.

9. A process as claimed in Claim 1 and substantially as herein described.

10. A cooling crystallisation process for manufacturing sugar in which the sequence of stages as claimed in any one preceding claim is repeated two or more times, and modified in that the starting syrup for second or subsequent repetitions is the mother liquor remaining after centrifuging of crystals from the next earlier such procedure.

11. A cooling crystallisation process as claimed in Claim 10 in which the mother liquor from the final repetition of the four stages is subject to conventional crystallisation to produce sugar and molasses, and in which the sugar produced from such conventional crystallisation is incorporated into a starting syrup for one of the four-stage procedures.

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