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Tisdel et al.

[11] **3,762,880**

[45] **Oct. 2, 1973**

[54] **APPARATUS FOR CRYSTALLIZATION AND RECOVERY OF ELEMENTAL SULFUR FROM SOLVENTS**

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[22] Filed: **Dec. 30, 1971**

[21] Appl. No.: **214,320**

Related U.S. Application Data

[62] Division of Ser. No. 837,113, June 27, 1969, Pat. No. 3,679,371.

[52] **U.S. Cl.**..... **23/273 R,** 23/312 S, 23/270 R, 23/300, 165/111

[51] **Int. Cl.**..... **B01d 9/02,** C01b 17/08

[58] **Field of Search** ..... 23/308 S, 312 S, 23/270, 270.5, 273 R, 273 F, 300; 196/14.52, 14.5; 165/111, 139

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Assistant Examiner—S. J. Emery
Attorney—Kellard A. Carter

[57] **ABSTRACT**

Apparatus for the crystallization and recovery of sulfur including a crystallizer vessel with inlet means for introducing a solvent with sulfur dissolved therein into the vessel at one level, and means for injecting an immiscible heat-exchange liquid at a different temperature from that of the solvent into the vessel at at least one other level different from the first level. The apparatus includes individual means for withdrawing heat-exchange liquid from the vessel, means for withdrawing crystalline sulfur slurry therefrom, and means for separating sulfur from this slurry.

12 Claims, 4 Drawing Figures

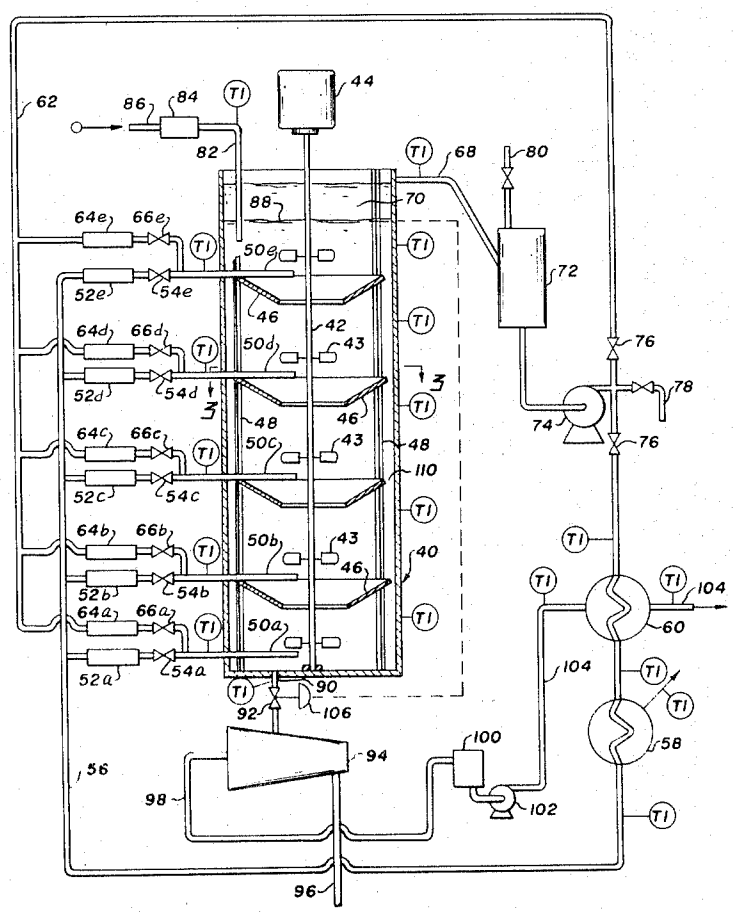
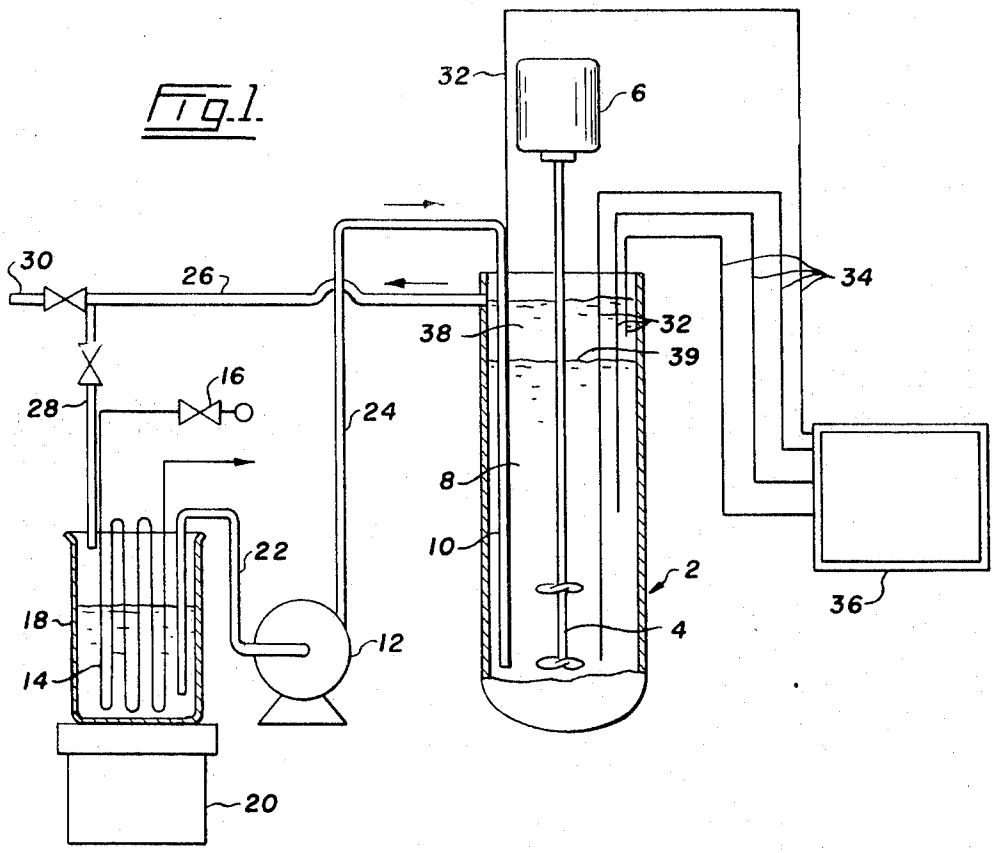


Fig. 1.


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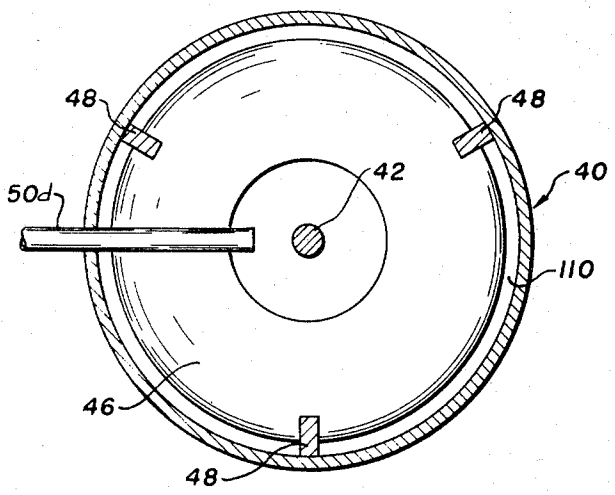

48
48
42
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50d
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46
48

Fig. 3.

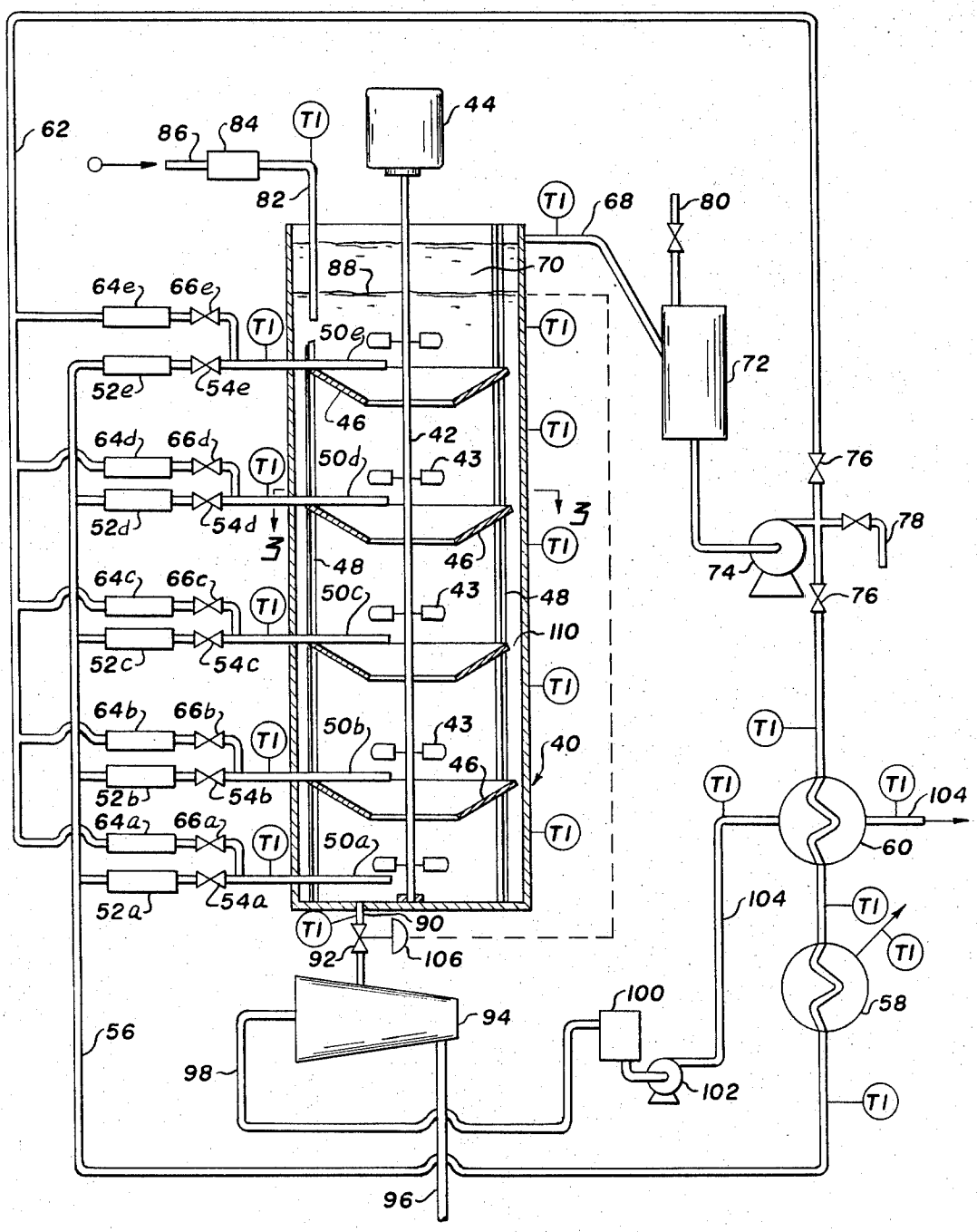

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52e
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52c
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40
52b
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64a
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106
104
60
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56
98
94
100
102
96

Fig. 2.

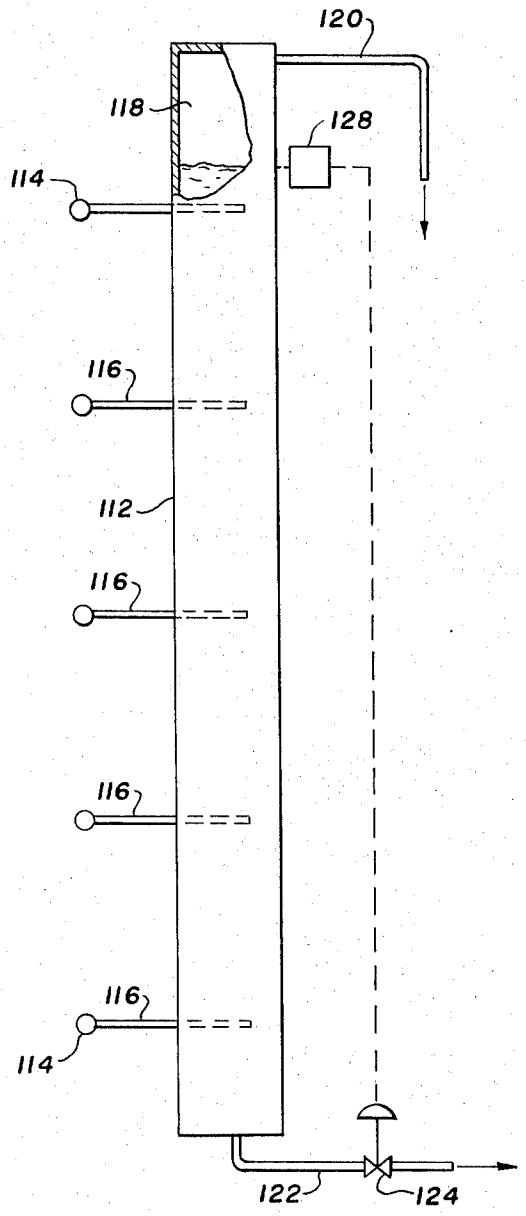


Fig. 4.

APPARATUS FOR CRYSTALLIZATION AND RECOVERY OF ELEMENTAL SULFUR FROM SOLVENTS

This is a division of application Ser. No. 837,113, filed June 27, 1969, now U.S. Pat. No. 3,679,371 granted July 25, 1972.

The supplies of sulfur obtainable from the well known Frasch sulfur mining process are rapidly dwindling. In terms of the world's increased demand for sulfur, the remaining known deposits of elemental sulfur are not amenable to the Frasch technique but, rather, are ideally situated for sulfur recovery.

These measured deposits occur throughout the world and major orebodies have been proven in the Philippines, Costa Rica, Bolivia, Guatemala, the United States, Canada and elsewhere.

These major sulfur-bearing orebodies, not amenable to Frasch mining techniques, are usually either volcanic in origin and/or deposited at or near the earth's surface. Normally they are too shallow in depth or their matrix is too fractured or too weak to permit the Frasch technique to be used economically. However, with the supply of sulfur being at its present level and with the demand increasing daily, these huge deposits become of increasing importance. Therefore, a great deal of attention has recently been given to processes for the recovery of their sulfur content. Among the operable processes for the recovery of elemental sulfur from these non-Frasch deposits are flotation concentration, hot water pressure autoclaving, thermal distillation, and solvent extraction techniques. It is with the latter process that the instant invention is concerned.

Apparatus of the present invention relates directly toward a solvent extraction technique for the recovery of elemental sulfur as a crystalline product from either a sulfur flotation process concentrate or from the raw elemental sulfur bearing ore deposit as mined.

The solvent extraction recovery technique involves the steps of slurrying a finely divided elemental sulfur containing material with a solvent for the sulfur, contacting intimately the sulfur containing material with the solvent either by means of trickling contactors or efficient agitation or other means known to the art. This contact is maintained for a period of time sufficient to completely solubilize the elemental sulfur in the sulfur solvent.

In the solvent extraction process it is common practice to bring out the contact between the sulfur bearing material and the solvent at an elevated temperature, since elemental sulfur is ordinarily soluble to a greater degree in hot solvent than it is in cold solvent or in solvent at ambient temperatures or below. Thus when the relatively hot sulfur pregnant solvent — that is solvent which contains the maximum amount of sulfur dissolved therein — is clarified from undissolved matter and cooled or heated and evaporated, the sulfur crystallizes from the pregnant solution and can be recovered from the solvent so that an extremely pure sulfur product results. The recovered solvent, freed from dissolved sulfur, commonly referred to as the "lean" solvent, can then be recovered and recycled to the process.

It is an object of the present invention to provide apparatus for the recovery of elemental sulfur from sulfur bearing materials in a crystalline form wherein crystals are of a controlled size.

It is a further object of this invention to present apparatus for the recovery of sulfur as a crystalline product either from a sulfur flotation process or from a raw elemental sulfur bearing ore.

A further object of the invention is to provide apparatus for the recovery of crystalline sulfur from a hot sulfur pregnant solvent wherein the crystalline sulfur does not come in contact with a solid heat exchange surface and thereby avoids impairment of heat exchange efficiency resulting from scaling of such solid surfaces.

Another object of the invention is to provide apparatus wherein a sulfur crystal of desired size may be obtained by controlling the temperature differential between the hot sulfur pregnant solvent and the heat-exchanging material, by controlling the rate of temperature change of the pregnant solvent, and by controlling the temperature gradient of the pregnant solvent as it flows through the system.

A still further object of the invention is to provide apparatus which eliminates over-nucleation and resulting production of undesirable small sulfur crystals and the resulting crystallization agglomeration formation.

A still further object of the invention is to provide apparatus which results in a recovered sulfur product from a slurry of crystalline sulfur and solvent which is free flowing and does not tend to plug pipes or valves as is usual in prior art techniques.

Parent application Ser. No. 837,113 discloses and claims a process for the recovery of sulfur in the crystalline form from a solvent having sulfur dissolved therein. The process comprises generally directing sulfur-laden solvent into a vertical crystallization zone, and directing an immiscible heat-exchange liquid at a different temperature from that of the solvent into the zone at a different level to cause a countercurrent flow between the solvent and this liquid. The spacing between the first and second levels is sufficient to ensure adequate direct heat exchange between the solvent and the liquid to cause formation of crystalline sulfur by virtue of the heat exchange contact. The solvent and the heat exchange liquid are separately withdrawn from the zone, and the crystalline sulfur is recovered from the slurry in which it is contained.

Apparatus in accordance with the present invention comprises a crystallizer vessel in the form of a column, inlet means for introducing a solvent with sulfur dissolved therein into the vessel at a first level, means for injecting an immiscible heat-exchange liquid at a different temperature from that of the solvent into the vessel at at least one other level different from the first level to cause a countercurrent flow between the solvent and said liquid, the spacing between the first level and said other level being sufficient to insure adequate direct heat exchange between the solvent and said liquid to cause formation of crystalline sulfur by virtue of the heat exchange, means for withdrawing heat-exchange liquid from the vessel after said contact with the sulfur-pregnant solvent, means for withdrawing lean solvent and crystalline sulfur from the vessel, and means for separating the withdrawn sulfur from the phase in which it is contained.

Examples of this apparatus are illustrated in the accompanying drawings in which:

FIG. 1 illustrates a laboratory batch apparatus;

FIG. 2 is a detailed drawing of a crystallizer unit suitable for commercial operation,

FIG. 3 is a view in cross-section of FIG. 2 taken along the line 3—3 of FIG. 2, and

FIG. 4 is a drawing of an alternate crystallization zone for the crystallizer unit of FIG. 2.

Turning now to the drawing of FIG. 1, reference numeral 2 indicates a crystallizing vessel which is equipped with agitating device 4, driven by motor 6. This vessel 2 represents a crystallizing zone and is charged with the desired amount of a solvent containing dissolved sulfur therein as represented by numeral 8. An immiscible heat-exchange liquid having a temperature below that of the solvent is introduced into crystallizer 2 by means of inlet pipe 10, line 24 and recycling pump 12. The temperature of the heat-exchange liquid is controlled as desired by means of heat-transfer coils 14, through which a cooling medium is circulated by means of valve 16 and a cooling and circulating system, not shown. A heat-exchange liquid reservoir indicated at 18 is also equipped with a heating device 20, in order to increase the temperature of the liquid if required.

The desired flow of the heat-exchange liquid is then circulated by means of pipe 22, circulating pump 12 through line 24 and line 10 into crystallizer vessel 2. A heat-exchange liquid overflow pipe shown at 26 is provided to withdraw heat-exchange liquid and for recirculating to reservoir 18 through valved line 28. Makeup heat-exchange liquid may be added to the system through valved line 30.

The crystallizer zone is provided with thermowells 32 connected to thermocouples 34 and a multipoint temperature recorder shown at 36. The thermowells are provided at locations which allow for the recordation of the various temperatures necessary for process control and, as shown in FIG. 1, measure and record the temperature at the bottom of the crystallizer zone, at the middle of the solvent phase, the middle of the heat-exchange liquid phase and at the point of introduction of the heat-exchange liquid into the crystallizer zone.

In operation, crystallizer vessel 2 is charged with the sulfur pregnant solvent, an immiscible heat-exchange liquid is injected into direct contact therewith by means of circulating pump 12, lines 24 and 10, impeller 44 is activated by means of impeller motor 6 and the direct intimate contact between the sulfur pregnant solvent and the heat-transfer liquid takes place.

As illustrated in FIG. 1, an immiscible heat-transfer liquid which is less dense than the sulfur solvent is introduced into the bottom of the crystallizer zone, is thoroughly admixed with the sulfur solvent by means of impeller 4, moves upwardly through the solvent phase as a dispersed phase and separates therefrom as a separate coolant liquid phase, as shown at 38. The interface between the solvent and liquid phases is shown at 39.

The separated heat-exchange liquid phase is continuously withdrawn through lines 26 and 28 into reservoir 18, is subjected to the requisite heating or cooling as may be necessary in reservoir 18 and is recirculated to the crystallization zone through line 22, circulating pump 12, line 24 and line 10.

When the desired separation of crystalline sulfur from the sulfur pregnant solvent has been obtained in crystallizer vessel 2, the process is stopped and the crystalline sulfur is recovered from the slurry in crystallizer vessel 2. The practice of the instant invention is described by referring to the following examples in which the laboratory batch apparatus of FIG. 1 is used.

EXAMPLE I

This typical experiment was designed to study sulfur crystalline size growth under conditions of the carefully controlled and uniform heat transfer rendered possible by this invention. 2,500 g of trichloroethylene having a boiling point of 179°F at 620 mm HG were saturated at 150°F with 144.5 g elemental sulfur analyzing 99.982 percent sulfur. This system was then placed in an experimental glass apparatus arranged as per FIG. 1. Water varying in temperature over the period of operation from 150°F was then introduced into the bottom of the hot sulfur pregnant trichloroethylene as a dispersed phase during continuous agitation at a rate of 72 g/min. to result in a solvent cooling rate of 0.73°F per minute. During the period of operation, a temperature difference (ΔT^1) of 10° to 13°F was maintained between inflowing dispersed heat-exchange water at the point of inflow and the solvent, while a temperature differential ΔT^2 of 1° to 3°F was measured between solvent layer and coalesced heat-exchange water layer. The average of ΔT^1 and ΔT^2 is calculated and represents ΔT^{avg}, and, as explained above, along with solvent cooling rate correlates directly with crystalline particle size.

At the end of the operation, the inflowing heat-exchange water temperature was 60°F, the solvent-sulfur crystal slurry temperature was 70°F, and the supernatant heat-exchange water layer temperature was 73°F. A total of 109.9 g of sulfur crystals were recovered from the apparatus and 34.6 g of sulfur remained dissolved in the residual solvent. The crystals had the following screen analysis.

Tyler Mesh	%, %m direct	Wt %, cumulative
+10	0.0	0.0
+28	25.1	25.1
+35	36.3	61.4
+48	24.2	85.6
+65	9.8	95.4
+100	4.1	99.4
−100	0.6	100.0

With a solvent cooling rate of 0.73°F per minute and with a ΔT^{avg} of 7°F, 85.6 percent of the crystalline sulfur was retained on the +48 mesh screen.

EXAMPLE II

This typical series of experiments using the apparatus of FIG. 1 and the procedures described in Example I were designed to study control of crystalline size growth through control of crystallizer temperature differentials and control of solvent cooling rate.

Experiment	A	B	C
Operating Conditions			
1. Charge to crystallizer			
Trichloroethylene, gm	2500.0	2500.0	2500.0
Sulfur, gm	144.5	144.5	144.5
2. Heat-exchange liquid flow rate, ml/min	15	70	368
3. ΔT^1, inflowing heat-exchange liquid vs. solvent, °F	42	10	3
4. ΔT^2, solvent layer vs. coalesced heat-exchange liquid layer, °F	4	2	1
5. ΔT^{avg}, average of ΔT^1 and ΔT^2, °F	23	6	2
6. Solvent cooling rate, °F/min	0.44	0.83	1.80
7. Sulfur balance at end of run			
Recovered crystalline sulfur, gm	104.3	109.9	106.9
Dissolved sulfur in residual solvent, gm	40.2	35.4	37.6
8. Screen analysis of crystalline sulfur			
Tyler Mesh	Cumulative, Wt %		
+10	0.0	0.0	0.0
+28	0.3	13.6	4.3
+35	8.7	60.8	30.3
+48	37.8	82.1	73.4
+65	88.3	95.1	93.8

+100	96.3	99.4	98.9
−100	100.0	100.0	100.0

Of the three experiments of this typical series, the operating conditions of experiment B, in which $\Delta T^{avg.}$ was controlled at 6°F and solvent cooling rate was controlled at 0.83°F/min, resulted in a superior sulfur crystal size distribution.

From many similar experiments of which the foregoing examples are typical, it is readily apparent that the novel crystallization system of this invention for recovery of sulfur from a sulfur pregnant solvent is extremely simple and highly efficient.

FIGS. 2 and 3 illustrate one form of apparatus in accordance with the present invention.

In FIGS. 2 and 3 reference numeral **40** indicates a vessel which comprises a crystallization zone. Vessel **40** is equipped with agitating means illustrated herein by an impeller shaft **42**, agitators **43** and motor drive **44**.

Crystallizer vessel **40** is equipped with a plurality of inclined ring baffles shown in cross-section at **46** which are supported within the crystallizer vessel by a plurality of vertical side baffles illustrated at **48**.

Crystallizer vessel **40** is provided with a plurality of heat-exchange liquid inlet dispersing pipes shown in the drawings as **50.***a–e*. Heat-exchange liquid is injected into the crystallizer zone through the inlet dispersion pipes through flowmeters **52.***a–e* and flow control valves **54.***a–e*, the heat-exchange liquid being distributed by manifold line **56** after having passed through heat exchangers **58** and **60**.

A system for the careful control of the temperature of the heat-exchange liquid being introduced in the crystallizer vessel is accomplished by provisions for the introduction of heat-exchange liquid at a different temperature, such as, for example, a warmer temperature, by means of manifold line **62**, flowmeters **64.***a–e* and flow control valves **66.***a–e*.

An upper phase liquid overflow pipe is provided as shown at **68** to continuously remove overflow of the upper phase **70** into a surge tank **72**. From surge tank **72** the upper phase is recycled to the system by means of circulating pump **74**, valves **76** and lines **56** or **62**. A sampling line or a bleed valve is provided in line **62** as shown at **78**. Makeup heat-exchange liquid may be added to surge tank **72** through line **80**.

Heat is recovered by exchange between the two phases in heat exchanger **60**. Heat is further removed as required from the recycle heat-exchange liquid by exchange with a medium external to the process in heat exchanger **58**.

The crystallizer vessel **40** is provided with an inlet line **82**, flowmeter **84** and line **86** for the addition of the more dense phase of the crystallizer system, for example, the sulfur-pregnant solvent, and is normally added to the crystallizer vessel beneath the interface **88** of the two-phase system within the crystallization zone.

The more dense phase of the two-phase system and the sulfur crystals are withdrawn from the bottom of the crystallization zone through outlet line **90**, control valve **92** and into separation device **94**.

Separation device **94** is preferably a centrifuge and is used to separate, in the normal operation of the process, crystalline sulfur from the phase in which it is contained. The separated crystalline sulfur is removed from the separation device **94** through line **96**. The separated recovered phase is removed from the separation vessel through line **98**, passed through surge tank **100**, through pump **102**, line **104** and through heat exchanger **60**. After passing through heat exchanger **60** the recovered material is delivered by means of line **104** to storage or recycled to the process at an earlier stage thereof.

The apparatus is provided with various temperature indicators illustrated in the drawing as TI and with various other flowmeters and flow control valves which are not shown.

Flow control valve **92** operates by means of liquid level controller **106** which is connected to an interface sensor at interface level **88** in order to carefully control the rate of bottom draw off.

In operation, using, as an example, a heat-exchange liquid of less density than the solvent, in this case trichloroethylene as the sulfur solvent and water as the heat-exchange liquid, the sulfur-pregnant trichloroethylene is continuously added to the crystallizer vessel **40** by means of line **86**, flowmeter **84** and line **82**. Water at a temperature below that of the sulfur-pregnant solvent is circulated through the crystallizing vessel as a dispersed phase by means of flowmeters **52.***a–e*, flow control valves **54.***a–e* and inlet pipes **50.***a–e*. Intimate contact between the sulfur pregnant solvent and the water is assured by the agitation provided by agitators **43**.

The dispersed phase, that is the water phase, moves gradually upward, because of its lesser density, through the body of the sulfur pregnant solvent in the crystallization zone, and cools the sulfur pregnant solvent. The water after coalescing into layer **70** is removed continuously by overflow through line **68**, into surge tank **72** and is recirculated to the system by pump **74**. The water discharging from pump **74** is split into two flows by means of lines **62** and **56**. Line **62** carries warm heat-exchange water to inlet pipes **50.***a–e* for blending with cold heat-exchange water to provide temperature control within crystallization zone **40**. Line **56** carries water through heat exchanger **60** where it is cooled by exchange with cold barren solvent issuing from pump **102** and thereby effecting heat recovery by heating the barren solvent, through heat exchanger **58** where the temperature of the coolant water is further reduced by heat exchange with a cooling medium, external to the process, and finally through flow meters **52.***a–e*, control valves **54.***a–e*, and inlet pipes **50.***a–e* into crystallization zone **40**. In the crystallization zone **40** the process of cooling the sulfur-pregnant solvent with the dispersed water is repeated.

Continuously removed from the bottom of the crystallizer zone through line **90** and control valve **92** is the slurry of barren solvent and crystalline sulfur obtained as a result of the heat exchange relationship occurring in the crystallizing zone of crystallizer vessel **40**. This slurry passes into separator **94** where the solid crystals are separated from the solvent liquid and removed through line **96** and from the process. The recovered lean solvent is recirculated through line **98**, surge vessel **100**, circulating pumps **102**, and line **104** and heat exchanger **60** to an earlier stage of the process.

FIG. **4** illustrates an alternative type of crystallization zone, which is used in a continuous manner. In this Figure, reference number **112** indicates a column-type vessel which comprises this alternate crystallization zone. In operation, using as an example, trichloroethylene as the sulfur solvent and water as the heat-exchange liquid, the sulfur-pregnant trichloroethylene

is continuously introduced into the crystallization zone **112** by means of line **114.** Water at a temperature below that of the sulfur-pregnant solvent is introduced into the crystallization zone as a dispersed phase by means of inlet pipes **116.***a–d.* The sulfur-pregnant solvent flows downward and the dispersed coolant water flows upward by virtue of the difference in densities. The coolant-water after upward coalescing into layer **118** is removed continuously by overflow through line **120.** The slurry of crystalline sulfur and trichloroethylene formed by virtue of the direct-contact cooling with the dispersed water is removed continuously from the crystallization zone through line **122** at a controlled rate equal to that of the input pregnant-solvent by means of interface level-control valve **124** connected to interface sensor-controller **128.**

The instant invention is further illustrated by the following specific example of results obtained with particular operating conditions using a crystallizer unit similar to that shown in FIG. **2** and FIG. **3.** It will, of course, be understood that the following description as given primarily for purposes of illustration and is not to be construed in any way to limit the invention in its broader aspects.

EXAMPLE IV

The typical practice of the instant invention using apparatus similar to that shown in FIG. **2** and FIG. **3** having a vessel volume of approximately 370 gallons is described herein.

Over a period of **78** hours, 203,000 lbs. of sulfur-pregnant trichloroethylene at a temperature of 159°F was fed to the crystallizer unit. During the period of operation, 4,343 lbs. of crystalline sulfur were produced. The typical screen analysis of this product after being processed through a centrifuge and a solvent evaporator was:

Tyler Mesh	Cumulative Wt. %
+10	5.7
+28	9.1
+35	25.8
+48	53.2
+65	77.5
+100	92.0
−100	100.0

The typical purity of this product was 99.98 wt. % elemental sulfur.

Average operating conditions during the period of operation were:

Pregnant Solvent Feed Rate, lb/hr.	2,600
Pregnant Solvent Temperature, °F	159
Heat-Exchange Liquid Flow Rates, lb/hr	
Inlet Pipe **50***a*	600
Inlet Pipe **50***b*	500
Inlet Pipe **50***c*	200
Inlet Pipe **50***d*	150
Inlet Pipe **50***e*	0
Heat-Exchange Liquid Inlet Temperature, °F	
Inlet Pipe **50***a*	50
Inlet Pipe **50***b*	65
Inlet Pipe **50***c*	85
Inlet Pipe **50***d*	89
Inlet Pipe **50***e*	—
Crystallizer Volume Temperature Gradient, top to bottom, °F	
Coalesed Water Layer	92
Top Stage-Compartment	95
Next Stage-Compartment	87
Next Stage-compartment	78
Bottom-Stage-Compartment	75
Crystalline Sulfur Product Rate, lb/hr.	56

It is thus clearly evident that the apparatus of this invention provides for the recovery of a crystalline sulfur of controlled size, efficiently and economically, without the inherent disadvantages of the prior art. The desired results are obtained by controlling the temperature differential between the sulfur pregnant solvent and the heat-exchange liquid, which by the inventive intimate is conveniently and accurately accomplished. solubilized By means of the apparatus just described, the sulfur crystal particle size may be varied as desired, and the resulting slurry of substantially sulfur free solvent and crystalline sulfur can be efficiently separated by any of any of the well known separation techniques. The heat-exchange liquid, illustrated for example above by water, is continuously recycled to the crystallization process and is easily and efficiently controlled as to its temperature by means of the apparatus illustrated. An additional advantage of the process is the utilization of waste heat by means of the heat-exchanger circuits so that the heat which would otherwise be lost is added to the barren solvents as it issues from the crystallization process.

To summarize briefly, the instant invention relates to apparatus for the recovery of elemental sulfur from a solvent containing sulfur dissolved therein, and embodying a novel heat transfer technique wherein a liquid of a density different from that of the solvent for the sulfur and at a temperature different from that of said solvent is admixed an intimate contact therewith, causing the temperature of the sulfur containing solvent to change and/or causing said solvent to vaporize and thereby causing the solubilized sulfur to crystallize therefrom in a controlled crystal size.

We claim:

1. Apparatus for the crystallization and recovery of sulfur from a sulfur-pregnant solvent, which comprises

a crystallizer vessel in the form of a column and having upper and lower ends,

a plurality of vertically spaced inlets for the vessel, the uppermost one of which is spaced below the upper end of the vessel, a plurality of vertically spaced downwardly sloping inclined baffles to insure stage mixing and enhance the temperature gradient of flowing solvent, each baffle being disposed below one of said inlets, a plurality of agitators in the crystallizer vessel, at least one said agitator being disposed adjacent each of said inlets, separate means for directing a light phase heat-exchange liquid into the vessel through each of said inlets,

means for controlling the flow of the light phase heat-exchange liquid to each of said inlets, means for controlling the temperature of the light phase heat-exchange liquid at each of said inlets,

inlet means for introducing a heavy phase sulfur-pregnant solvent into the vessel near the upper end thereof,

the temperature of the heat-exchange liquid being different from that of the solvent and the heat-exchange liquid flowing upwardly and the solvent flowing downwardly to the lower end of the vessel,

the spacing between said inlet means and said inlets and between the individual inlets being sufficient to insure adequate direct heat exchange between the solvent and the heat-exchange liquid to cause formation of crystalline sulfur by virtue of the heat exchange,

means for withdrawing heat-exchange liquid from near the upper end of the vessel after said contact with the sulfur-pregnant solvent,

means for withdrawing lean solvent and crystalline sulfur from the vessel near the lower end thereof, and

means for separating the withdrawn sulfur from the withdrawn solvent.

2. Apparatus as claimed in claim **1** including means for recycling said withdrawn heat-exchange liquid to said inlets of the crystallizer vessel.

3. An apparatus according to claim **1** wherein said crystallizer vessel is equipped with a plurality of vertically spaced and downwardly inclined ring baffles to insure staged mixing and enhance the temperature gradient of the flowing solvent.

4. An apparatus according to claim **1** wherein said crystallizer vessel is equipped with circumferentially spaced vertical baffles to insure intimate contact with between said solvent and said heat-exchange liquid.

5. Apparatus as claimed in claim **1** including means for directing heat-exchange liquid withdrawn from the upper end of the vessel back to said inlets.

6. Apparatus as claimed in claim **5** including means for transfering heat between said heat-exchange liquid withdrawn from the upper end of the vessel and said lean solvent separated from said withdrawn sulfur.

7. Apparatus as claimed in claim **1** including means for directing a first portion of the heat-exchange liquid withdrawn from the upper end of the vessel back to said inlets, means for cooling a second portion of said withdrawn heat-exchange liquid, and means for directing said cooled second portion of the heat-exchange liquid back to said inlets.

8. Apparatus as claimed in claim **7** including means for intermixing said first and second portions of the heat-exchange liquid at each of said inlets to control the temperature of the heat-exchange liquid being directd into the vessel through said each inlet.

9. Apparatus as claimed in claim **1** including means for maintaining a predetermined level of sulfur-pregnant solvent in the vessel so as to control the level of an interface between said solvent and said heat-exchange liquid near the upper end of the vessel.

10. Apparatus as claimed in claim **9** in which said inlet means is positioned to direct sulfur pregnant solvent into the vessel below said predetermined interface level.

11. Apparatus as claimed in claim **9** including means for controlling the rate of withdrawal of lean solvent and crystalline sulfur from the vessel.

12. Apparatus as claimed in claim **9** including means responsive to the level of said interface for controlling the rate of withdrawal of lean solvent and crystalline sulfur from the vessel.

* * * * *

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,762,880 Dated October 2, 1973

Inventor(s) LAWRENCE C. TISDEL: HARRY B. SCOTT

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In the heading, column 1, item [73] should appear as follows:

Assignee: Brameda Resources Limited,
Vancouver, British Columbia,
Canada.

Signed and sealed this 5th day of February 1974.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

RENE D. TEGTMEYER
Acting Commissioner of Patents