Feb. 1, 1955 R. R. COATS 2,701,266

SEPARATION OF P-XYLENE BY CRYSTALLIZATION

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FIG. 1.

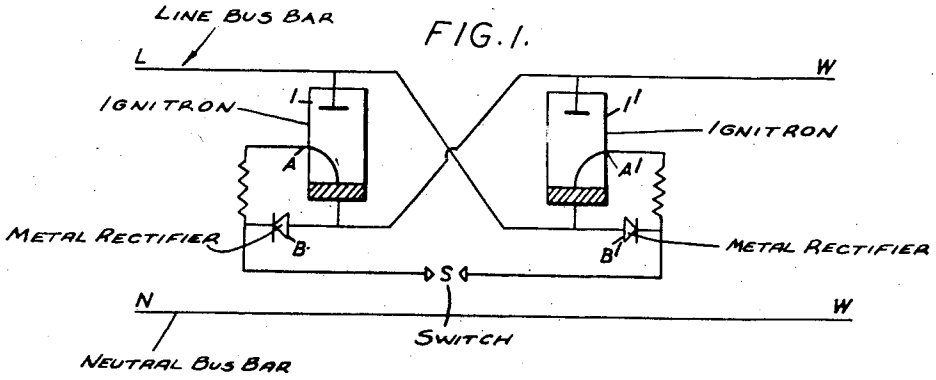

FIG. 2.

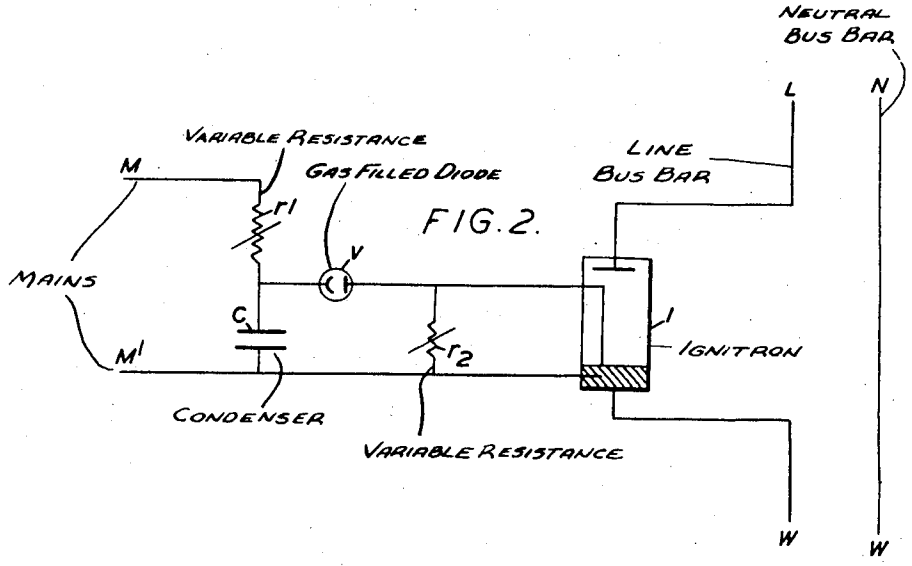

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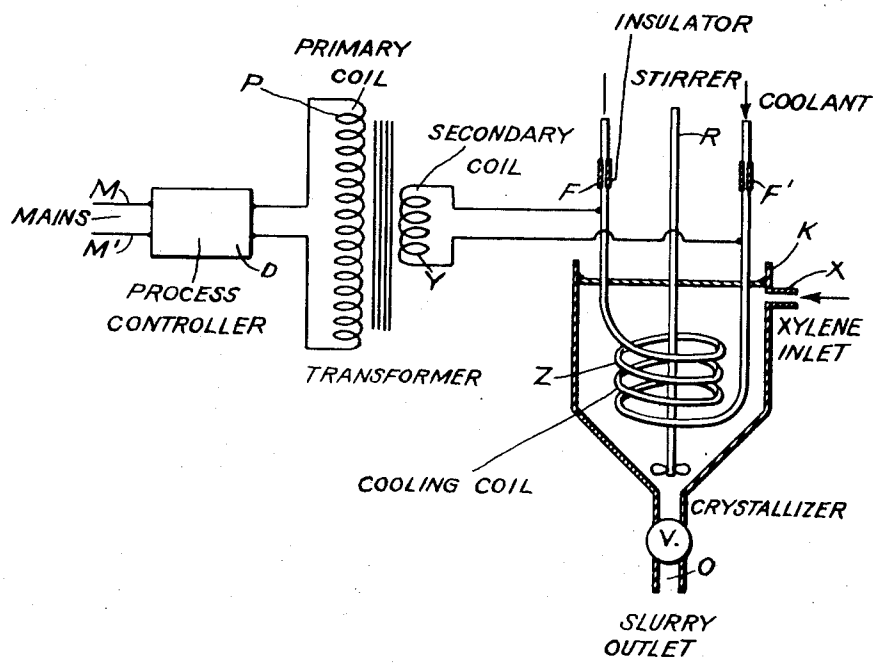
FIG. 3.



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SEPARATION OF P-XYLENE BY CRYSTALLIZATION

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3 Claims. (Cl. 260—674)

This invention relates to processes of crystallisation.

In crystallisers operated by cooling the liquid to be treated by cooling surfaces such as coils, a common difficulty experienced is the building up of the crystallising substance as solid on the cooling surfaces. This reduces the cooling effect and consequently the output of crystals, and is often troublesome because the crystallised solid can only be removed from the cooling surface with great difficulty. In the past it has therefore frequently been necessary to operate at low rates or to provide an extremely large cooling surface so that the temperature difference between cooling medium and cooled liquid is small, but this involves relatively high capital cost and does not give high efficiency.

According to the present invention in processes of crystallisation employing a cooled surface or surfaces, production of the desired crystalline product is facilitated by intermittent heating of a cooling surface or surfaces by electrical means, e. g. by passing an electrical current through it or them, at selected time intervals, for short periods sufficient in duration to melt deposited solid adjacent to the aforesaid cooling surface or surfaces and removing said solid therefrom, e. g. by sloughing it off, to prevent deposition thereon.

According to the invention the heating of the cooling surface is effected so rapidly that the amount of heat transferred to cooling fluid or cooled liquid during the heating period is kept as low as possible compatible with efficient operation. This can be achieved by selecting suitable voltages, time intervals and incidence of heating.

One suitable method of operation is to make the cooling coil part of an electric circuit and to pass a heavy electric current through it for a very brief interval so that heat is generated in the mass of the coil. By using an alternating current of radio frequency it is possible to confine the heating effect substantially to the surface of the metal whereby it is more efficiently utilised and the coil more quickly resumes its own function of cooling. If a jacketed crystalliser is used the current can conveniently be produced inductively in the vessel wall.

One suitable arrangement comprises a jacketed cooling vessel provided with a stirrer and a worm coil of stainless steel, for example that sold under the registered trademark "Staybrite," forming part of an electric circuit which includes the secondary coil of a transformer. Current is supplied to the worm coil by closing the primary circuit of the transformer by means of a switch. Alternatively, the current may be applied to the worm coil by means of a thyratron-switched circuit or an ignitron circuit triggered by a suitable timing means, for example a time switch or a thyratron. An ignitron circuit is desirable when high amperage currents are to be used.

The principle on which the invention is based is the application, at intervals, of short electrical pulses applied so that the heat developed and available for melting the solid interferes to the minimum extent with the cooling function of the coils

The invention is applicable to crystallisation processes conducted over a wide range of temperature, e. g. from +100° C. down to −100° C., and is especially suitable for the separation of p-xylene from mixed xylenes at temperatures from +13° C. downwards.

Preferably the cooling elements, e. g. coils, are of low total heat capacity; the duration of each pulse should be brief, e. g. up to 10, and preferably less than 5, seconds; the input of energy should be sufficient but not greatly

in excess of that required to free the coil from deposition; and the pulses should be sufficiently frequent to prevent the deposition of hard, thick incrustation, which would not readily slough off.

When pulses of high amperage are employed it is preferred to employ an ignitron instead of a mechanical switch or contactor on account of the mechanical wear and burning of contacts which occur with the latter.

A suitable ignitron circuit for single phase operation is shown schematically in Figure 1 and comprises two ignitrons II', one to conduct each half cycle, with their igniters AA' and metal rectifiers BB', and a suitable switch S. L and N represent the line and neutral bus bars and WW the output to the coil in the cooling vessel or transformer feeding said coil.

When the duration of the pulse is of the order of a second, the switch S may suitably be a cam operated contact, preferably driven by a synchronous motor.

When a pulse of less than 1 second duration is required it is preferred to employ in place of switch S a condenser discharged circuit including a diode.

An example of a suitable condenser discharged circuit, for simplicity shown only for a half wave, is given in Figure 2 in which MM' represent direct current mains, C a condenser r_1, and r_2 variable resistances, of which r_2 is less than r_1, V a gas-filled diode, I an ignitron, LN the line and neutral bus bars, and WW the output to the coil in the cooling vessel or transformer feeding said coil. If desired the resistance r_2 could be replaced, although in general with not so good results, by a magnetic relay operating a mechanical switch controlling the ignitron.

For pulses of still shorter duration and period a thyratron triggered ignitron could be used.

The general arrangement of a simple and effective method of carrying the invention into practice is shown in Figure 3, which is self explanatory. Alternating current from mains MM' is supplied to the primary coil P of a transformer through an electrical process controller D which is set to give impulses of desired duration at specified time intervals, a suitable program being one impulse of one second duration per minute. This causes a corresponding surge of current in the circuit comprising the secondary coil Y and the cooling coils Z which are electrically insulated by insulators F.

The liquid mixture of hydrocarbons containing p-xylene is introduced into crystallizer K provided with stirrer R through the pipe X. The liquid mixture is cooled by cooling coils Z through which a coolant such as pentane is passed. Crystals rich in p-xylene are formed and removed from the crystallizer through the valved outlet pipe O. Build-up of these crystals on the cooling surface of the coils Z is prevented by the periodic surges of current, sent as above described, through said cooling surface.

The invention is illustrated by the following example.

Example

Four litres of a mixed xylene fraction containing 42% of the para-isomer, 25% of the ortho-isomer and 33% of the meta-isomer, was introduced into a 3 litre Dewar vacuum flask of 6" inside diameter x 10" depth, in which was situated a 3" inside diameter x 4" overall depth cooling coil of stainless steel tubing of ¼" outside diameter and 3/16" inside diameter through which a coolant could be circulated. This coil was connected to the secondary windings of a transformer which could supply to it a current of 200 amps. at 20 volts. The flask was provided with a paddle type stirrer the spindle of which was located within the coil, the arrangement being such that the liquid in the neighbourhood of the coils was strongly agitated. In order to stimulate running conditions in a continuous process, involving removal of crystalline p-xylene by centrifuging and feeding fresh xylene mixture, there was provided a small heater of 100 watts maximum output controlled by a rheostat and situated near the bottom of the flask.

The mixture of xylenes contained sufficient p-xylene to give a slurry containing 20% by weight solids at −38° C., which was the operating temperature. Methanol was pumped through the coil at a rate of 60 litres per hour

to cool the xylene mixture. The stirrer was rotated at 200 revolutions per minute. Under these conditions the temperature difference between the mixture and the methanol entering the coil could be readily controlled at 20 degrees by varying the heat input from the heater. The duration of the pulses of the surge current and the number of pulses per minute were controlled by a press button contactor in the primary circuit of the transformer. The pulses were applied at equidistant time intervals.

Operating in the above described manner, the following data were obtained.

Run No.	Description of pulse		Average temperature of xylene, ° C.	Av. temp. diffs. xylene and inlet methanol	
	Duration, Secs.	Number per Minute		Min.	Max.
1			−36	10	20
2	3	1	−37	10	25
3	2	1	−38	10	20
4	1	1	−38	11	15
5	1	2	−38	13	16

The following observations, which bring out the advantages of using the surge type of heating, were made:

Run 1.—Thick deposition of p-xylene on the outer surface of the coils was present at 30 minutes and very heavy deposition at 45 minutes.

Run 2.—The outer surface of the coils was clear of solid after 2 hours running. The temperature rise of the pot contents caused by a single 3 second surge was approximately 1° C.

Run 3.—The outer surface of the coils was free from deposition after 2 hours.

Run 4.—The outer surface of the coils was free from deposition after 60 minutes, but the critical condition had nearly been reached as indicated by a certain amount of incrustation remaining on the coil above the surface of the liquid, where splashing and creep of the liquid had occurred.

Run 5.—The condition of the coils after 60 minutes was similar to that in Run 4 after the same time.

While amperages of 10 and more may be used, preferably the amperage of the current employed in the process is 100 or more.

By total heat capacity in this specification is meant water equivalent.

I claim:

1. In the process of separating p-xylene from a liquid mixture of hydrocarbons containing the same by crystallization employing at least one cooled surface immersed in the liquid mixture for cooling the mixture to a temperature at which crystallization occurs, the improvement which consists in relatively strongly and directly heating the cooled surfaces by periodically passing an electric current having an amperage of at least 100 through said cooled surface for a short interval of time less than 5 seconds.

2. A process as recited in claim 1 in which the electric current is an alternating current of radio frequency and the time interval does not exceed 1 second.

3. A process as recited in claim 1 in which the electric current passing through said cooled surface is an alternating current produced inductively in the surface.

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