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" IMPROVEMENTS IN OR RELATING TO THE ELECTROCHEMICAL  
PROCESS FOR THE PRODUCTION OF BROMOFORM FROM  
ACETONE "

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH,  
Rafi Marg, New Delhi - 1, India, an Indian  
Registered body Incorporated under the Registration  
of Societies Act ( Act XXI of 1860)

The following specification ~~particularily~~ describes ~~the nature of this invention~~

This is an invention by Handady Venkatakrishna Udupa,  
Director, Mysore Seshaiyer Venkatachalapathy, Scientist,  
and Sankaranarayanaier Chidambaram, Scientist, all of  
the Central Electrochemical Research Institute, Karaikudi, *Tamil Nadu*  
all Indians.

PRICE: TWO RUPEES

144480

This is an invention by Handady Venkatakrishna Udapa, Director, Mysore Seshaiyer Venkatachalapathy, Scientist, and Sankaranarayanaier Chidambaram, Scientist, all of the Central Electrochemical Research Institute, all Indians.

This invention relates to improvements in or relating to the electrochemical process for the production of bromoform from acetone.

Hitherto it has been proposed to produce bromoform chemically by employing generally bromine for the bromination of acetone and also electrolytically employing costly anode material like platinum under stationary conditions.

This is open to objection that free bromine employed in the preparation of bromoform is costly and all the bromine is not effectively utilised for the conversion of acetone into bromoform. The use of platinum as anode material is prohibitive due to its high cost.

The object of this invention is to obviate the disadvantages by the use of an electrochemical method employing cheaper anode materials like graphite. According to the present invention, acetone is anodically brominated to bromoform using sodium/potassium bromide solution as an electrolyte containing acetone.

To these ends the invention broadly consists in the preparation of bromoform by electrolysis of a solution of sodium or potassium bromide, sodium bicarbonate, sodium dichromate and acetone in water using graphite as stationary anode and stainless steel as cathode. Current density in the range of 5 to 15 A/dm<sup>2</sup> may be employed preferably 10 A/dm<sup>2</sup>. Temperature of electrolysis is kept between 15 and 25°C and pH of the electrolyte is maintained between 8.5 and 10. Bromoform formed is collected at the bottom.

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The following typical example is given to illustrate the invention:

EXAMPLE

A 400 ml pyrex beaker provided with bottom discharge was used as the cell. A cylindrical graphite rod was used as the stationary anode and cathode were two stainless steel plates. The inter electrode distance was 2 cm. Electrolyte was prepared by dissolving 50 gms of sodium bromide, 3 gms of  $\text{NaHCO}_3$  and 0.3 gms of  $\text{Na}_2\text{CrO}_4$  in 210 ml of water and to this 40 ml of acetone was added. A current density of  $10 \text{ A/dm}^2$  was employed. 12 A.hrs current was passed through the electrolyte. The temperature was maintained between 20 and 22°C and the pH was maintained between 9 and 9.5 by supplying  $\text{CO}_2$  gas through the electrolyte. Bromoform was collected at the bottom which was removed, washed with bicarbonate solution and then with water and dried. Weight of bromoform separated was 17.1 gms with a current efficiency of 92.5%. Voltage of the cell was 5.0-5.5.

The main advantage of the invention is that the costly platinum anode is replaced by cheaper anode material graphite. The costly brominating agent, free bromine, is avoided and the tedious handling of free bromine is eliminated.

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Dated this 3rd day of October, 1975

*M. Kumar*  
Asst. Patent Officer  
Council of Scientific & Industrial Research

144480

**THE PATENTS ACT. 1970**

**COMPLETE SPECIFICATION**

(Section—10)

**" IMPROVEMENTS IN OR RELATING TO † ELECTROCHEMICAL  
PROCESS FOR THE PRODUCTION OF BROMOFORM FROM ACETONE "**

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH,  
Rafi Marg, New Delhi - 1, India, an Indian Regis-  
tered body Incorporated under the Registration  
of Societies Act ( Act XXI of 1860)

The following specification particularly describes  
and ascertains the nature of this invention and the manner  
in which it is to be performed :-

This is an invention by Handady Venkatakrisna Udupa, Director,  
Mysore Seshaiyer Venkatachalapathy, Scientist, and Sankaranarayanaiah  
Chidambaram, Scientist, all of the Central Electrochemical Research  
Institute, Karaikkudi, all Indians.

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This invention relates to the improvements in or relating to the electrochemical process for the production of bromoform from acetone.

Hitherto it has been proposed to produce bromoform chemically by employing generally bromine for the bromination of acetone and also electrolytically employing costly anode material like platinum under stationary conditions.

This is open to objection that free bromine employed in the preparation of bromoform is costly and all the bromine is not effectively utilised for the conversion of acetone into bromoform. The use of platinum as anode material is prohibitive due to its high cost.

The object of this invention is to obviate the disadvantages by the use of an electrochemical method employing cheaper anode materials like graphite. According to the present invention, acetone is anodically brominated to bromoform using sodium/potassium bromide solution as an electrolyte containing acetone.

To these ends the invention broadly consists in the preparation of bromoform by electrolyzing a solution of sodium or potassium bromide, sodium bicarbonate, sodium dichromate and acetone in water using graphite as stationary anode and stainless steel as cathode. Current density in the range of 5 to 15 A/dm<sup>2</sup> may be employed, preferably 10 A/dm<sup>2</sup>. Temperature of ~~about~~ electrolysis is kept between 15 and 25°C and pH of the electrolyte is maintained between 8.5 and 10. Bromoform formed is collected at the bottom.

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The following typical example is given to illustrate the invention and does not limit the scope of invention.

EXAMPLE

A 400 ml pyrex beaker provided with bottom discharge was used as the cell. A cylindrical graphite rod was used as the stationary anode and cathodes were two stainless steel plates, placed equidistant on either side of the anode. The inter-electrode distance was 2 cm. Electrolyte was prepared by dissolving 50 g of sodium bromide, 3 g of  $\text{NaHCO}_3$  and 0.3 g of  $\text{Na}_2\text{CrO}_4$  in 210 ml of water and to this 40 ml of acetone was added. A current density of  $10 \text{ A/dm}^2$  was employed. 12 A hrs electricity was passed through the electrolyte. The pH was maintained between 9 and 9.5 by supplying  $\text{CO}_2$  gas through the electrolyte and the temperature was maintained between 20 and  $22^\circ\text{C}$ . Bromoform was collected at the bottom which was removed, washed with bicarbonate solution and then with water and dried. Weight of bromoform separated was 17.1 g with a current efficiency of 92%. Voltage of the cell was 5.0 - 5.5 V.

The main advantage of the invention is that the costly platinum anode is replaced by cheaper anode material-graphite.

We claim:

1. A process for the electrochemical production of bromoform from acetone using a graphite anode and stainless steel cathodes at a current density of 5 to  $15 \text{ A/dm}^2$ , a temperature of 15 to  $25^\circ\text{C}$  and the pH of the electrolyte of 8.5 to 10.
2. A process as claimed in claim 1 wherein a current density of  $10 \text{ A/dm}^2$  is preferably employed.
3. A process as claimed in claim 1 wherein a solution consisting of 25% sodium bromide, 1.5% sodium bicarbonate, 0.15% sodium dichromate, and 20% acetone in water is used as electrolyte.

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4. A process as claimed in claim 1 wherein the temperature is preferably 22°C.
5. A process as claimed in claim 1 wherein the pH of the electrolyte is kept in the range of 8.5- 10.0 by passing CO<sub>2</sub> gas through the electrolyte.

Dated this 19th day of August 1976.

sd/-

(PATENTS OFFICER)

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH