

PROVISIONAL SPECIFICATION

Specification No. 115204. Application No. 115204 dated 30th March, 1968. Complete Specification left on 13th December, 1968 (Application accepted 23rd September, 1969)

Index at acceptance -31A[LVIII(2)]

IMPROVEMENTS IN OR RELATING TO ELECTROLYTE FOR TANTALUM ELECTROLYTIC CAPACITORS

COUNCIL OF SCIENTIFIC & 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 describes the nature of this invention:—

This is an invention by Shri BALKUNJE ANANTHASENOI, Scientist, Shri KANDADAI RAJAGOPALACHARI NARASIMHAN, Scientist, Shri VENKATASUBRAMANIAN LAKSHMINARASIMHAN, Junior Scientific Assistant, Shri ANANTHARAYANAN ANANTHARAMAN, Senior Laboratory Assistant and Shri DEVARAJ KANAGARAJ, Senior Laboratory Assistant, all are Indian Nationals employed in Central Electrochemical Research Institute, Karaikudi-3 Tamil Nadu, India.

This invention relates to improvements in or relating to electrolyte for tantalum electrolytic capacitors.

Hitherto it has been proposed to use electrolyte for capacitors containing dimethyl formamide either alone or in mixture with solvents like water, alkyl alcohols, ethers, amines with film forming compounds selected from phosphate, formate, thiocyanate, picrate, nitrate, lactate, borate, oxalate, tartrate with cation species selected from the group consisting of hydrogen, ammonium or substituted ammonium ions. Even though ethylene glycol based electrolytes are known the constituents present are covered by patents.

This is open to the objection due to the following reasons:

1. The solvent such as dimethyl formamide is very poisonous and highly corrosive.
2. Since it has good solvent action on washers like rubber, neoperin and sealing compound like Araldite—the common materials used in the fabri-

cation of capacitors—it is necessary to choose special type of washers such as teflon elastomer, silicon rubber. Such special type of washers are not available in our country and have to be imported.

The objective of this invention is to obviate these disadvantages by using non-corrosive, cheap, readily available chemicals for the preparation of electrolyte for tantalum electrolytic capacitors.

The following typical examples illustrate the nature of this invention :

Example 1

Ethylene glycol	..	60% by weight
Ammonium citrate	..	5% by weight
Deionised water	..	35% by weight
Resistivity of the electrolyte	..	160 ohm cm

Example 2

Ammonium citrate	..	4% by weight
Water	..	30% by weight
Ethylene glycol	..	66% by weight

Dated this 26th day of March, 1968.

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COMPLETE SPECIFICATION

COUNCIL OF SCIENTIFIC & 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 particularly describes and ascertains the nature of this invention and the manner in which it is to be performed:

This is an invention by Shri BALKUNJE ANANTHASENOI, Scientist, Shri KANDADAI RAJAGOPALACHARI NARASIMHAN, Scientist, Shri VENKATASUBRAMANIAN LAKSHMINARASIMHAN, Junior Scientific Assistant, Shri ANANTHARAYANAN ANANTHARAMAN, Senior Laboratory Assistant, and Shri DEVARAJ KANAKARAJ, Senior Laboratory Assistant all are employed in the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, and all are Indian Nationals.

This invention relates to improvement in or relating to electrolyte for tantalum electrolytic capacitors.

The advantages of using tantalum electrolytic capacitors consist in their long shelf-life, low D.C. leakage current, low power factor, wide temperature range of operation. The performance of the electrolytic capacitor is largely determined by the nature of the electrolyte used. The electrolyte should have substantially uniform and consistently reproducible electrical properties for the production of capacitors possessing standard and uniform characteristics.

Hitherto it has been proposed to use electrolyte for capacitors containing dimethyl formamide either alone or in mixture with solvents like water, alkyl alco-

hols, ethers, amines with film-forming compounds selected from phosphate, formate, thiocyanate, picrate, nitrate, lactate, borate, oxalate, tartrate with cation species selected from the group consisting of hydrogen, ammonium or substituted ammonium ions. Other patented ethylene glycol based electrolytes are known but they are covered by patents and they are intended for use in low voltage ratings only.

This is open to the objection due to the following reasons:

The solvent such as dimethyl formamide is very poisonous and highly corrosive. Since it has good solvent action, it is very difficult to choose right material for washers and sealing compounds. The material recommended for washers are silicone rubber, teflon and butyl rubber. These materials are not readily available in our country and have to be imported.

The objective of this invention is to obviate these disadvantages by using non-corrosive, cheap, readily available chemicals for the preparation of electrolyte for tantalum electrolytic capacitors.

The electrolyte for use in the tantalum electrolytic capacitors must have the following essential properties :

1. It should be capable of forming the required dielectric film on the anode at the voltage within the temperature range specified.
2. There should not be any chemical reaction between the electrolyte and any part of the capacitor over the temperature range of operation except the electrolytic process of formation of oxide film at the anode.
3. The change in resistance and viscosity of the electrolyte within the temperature range of operation should be minimum.
4. The electrolyte should remain liquid over a wide range of temperature, high polarity and good solvent properties for the ions to be employed.

The above mentioned characteristics for the electrolytic are attained by the use of ethylene glycol as a solvent, having the good solvent action on certain organic acids and salt of the acids and salt of the acid derived from ammonium or substituted ammonium ions. Examples of the anion species are citrate, oxalate, picrate.

Thus, the present invention consists of a formulation for preparing an electrolyte for use in tantalum electrolytic capacitors which comprises a solution containing citrate ion and a cation containing at least one nitrogen atom dissolved in a mixture of ethylene glycol and water solvent. The citrate ions are present to impart both film-forming property and conductivity to the electrolyte.

Tantalum electrolytic capacitor was fabricated out of 12.5 micron thick tantalum foil of 30 sq.cm area and electrolytically formed at 140 volts. A similar dimensioned plain tantalum foil was used as cathode. The electrolyte having the following composition as shown in Table 1 gives a capacity of 5 microfarad at 25°C and a satisfactory operational temperature range between -55°C and +85°C as evidenced by the following data given in Table 2 with respect to the low temperature operation:

Table 1

Ethylene glycol	..	60% by weight
Ammonium citrate	..	5% ..
Water	..	35% ..
Resistivity	..	160 ohm cm

Table 2

Temperature	Capacity in mfd.	Power Factor at 50 cps.	Leakage current in μ A	Impedance at 120 cps.
+25°C	5	0025	2	..
+85°C	5	0025	1	..
-55°C	4.35	0060	Nil	320

After life-test of 2000 hours at the rated temperature of +85°C and the rated voltage of 100 volts, a change in capacity was found to be 4%. The change in leakage current at 85°C had dropped from the initial value of 0.003 A/ μ F-V to 0.001 A/ μ F-V during the life-test.

We Claim

1. The composition for use in tantalum foil type electrolytic capacitors which comprises ethylene glycol and water wherein an ammonium or substituted ammonium salt of an organic acid where the anion is selected from the group consisting of citrate, oxalate or picrate is present in the new composition.
2. An electrolyte for use in tantalum electrolytic capacitor comprising a solution of Claim 1 consist of a mixture of ethylene glycol and water wherein the said resulting mixture possesses good solvent properties for the organic acid anions.
3. An electrolyte for use in tantalum electrolytic capacitor comprising a solution of Claim 1 wherein water is present in an amount of up to about 48% to 12% of the electrolyte composition.
4. An electrolyte for use in tantalum electrolytic capacitor comprising a solvent as in Claims 1 and 2 where the organic acid anion species is selected from the group consisting of citrate, oxalate or picrate.
5. An electrolyte for use in tantalum electrolytic capacitor comprising a solvent of Claims 1 and 2 where ethylene glycol is present at a concentration of from about 50% to 80% of the total solvent.
6. An electrolyte for use in tantalum electrolytic capacitor comprising a solvent of Claim 1 where the anion species is selected from the group consisting of citrate, oxalate or picrate and is present in an amount of from 2% to about 8% of the electrolyte composition.

Dated this 9th day of December, 1968.

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