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(54) **Title:** AMORPHOUS CAESIUM ALUMINIUM FLUORIDE COMPLEX, ITS PRODUCTION AND USE

(54) **Bezeichnung:** AMORPHER CÄSIUMALUMINIUMFLUORID-KOMPLEX, DESSEN HERSTELLUNG UND VERWENDUNG

(57) **Abstract:** The subject matter of the present invention is an amorphous caesium aluminium fluoride complex, a method of producing it and the use of the complex as a flux, in particular for the soft soldering of aluminium.

(57) **Zusammenfassung:** Gegenstand der vorliegenden Erfindung ist ein amorpher Cäsiumaluminiumfluorid-Komplex, ein Verfahren zu dessen Herstellung und die Verwendung des Komplexes als Flussmittel, insbesondere zum Weichlöten von Aluminium.

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AMORPHOUS CAESIUM ALUMINIUM FLUORIDE COMPLEX, ITS PRODUCTION AND USE

TECHNICAL FIELD OF THE INVENTION

The present invention provides an amorphous caesium aluminium fluoride complex, a process for its production and the use of the complex as a flux, in particular for the soft soldering of aluminium.

BACKGROUND TO THE INVENTION

The use of caesium fluoroaluminates as fluxes for soldering aluminium materials is known. The low melting point and hence the ability to solder even magnesium-containing aluminium alloys opens up many applications and developments. In the automotive industry, for example, the trend in recent years has been towards ever smaller radiators, which operate at ever greater pressures. This has led to increased strength requirements for the radiators, and this has been reflected in a higher magnesium content in the aluminium alloys. This development has also given rise to greater requirements for fluxes. In order to meet these requirements, it must be possible to tailor a flux specifically to the individual application. A critical requirement here is the ability to adjust the melting point of a flux across a broad range and above all also to allow a low melting point or melting onset below 440°C. The flux should be resistant, to oxidation for example, and be able to be processed into pastes and soldering rods.

The prior art offers fluxes made from crystalline complexes of caesium/ aluminium/ fluorine (Cs/Al/F), which are known as caesium fluoroaluminates. A disadvantage of these crystalline substances for use as fluxes is that they are defined compounds with narrow melting ranges or even precise melting points. Moreover, additional substances such as additives for example, with which the basicity or acidity can be adjusted and which have a positive influence on the soldering characteristics, cannot be bound to the crystalline complex.

US-A-4689092 describes a flux consisting of a caesium fluoroaluminate complex which with an appropriate ratio of caesium fluoride (CsF) to aluminium fluoride (AlF_3) begins to melt at 440°C. The material exhibits strong crystallinity in the X-ray diffractogram and is not hygroscopic. The material is technically simple to produce by a hydrothermal process from caesium fluoride and aluminium fluoride. However, melting points below 440°C cannot be achieved; melting points can only be established in the range from 440 to 460°C. Furthermore, the flux does not exhibit good properties for the soldering of materials whose magnesium content is greater than 1%. Moreover, due to its ready oxidisability and its rapid degradation, this flux is not suitable for flame soldering (US-A-5171377).

US-A-4923530 describes the production of an oxygen-containing suspension of caesium fluoroaluminate, which has good stability and a low melting point of 414°C. However, hydrofluoric acid at temperatures of up to 90°C is used in its production. The handling of the highly toxic hydrofluoric acid under these conditions calls for special requirements for materials and occupational safety. The use of caesium carbonate (Cs_2CO_3) in the reaction generates CO_2, which forms aerosols containing hydrofluoric acid and fluoride. Furthermore, the aim of this process is to produce a suspension. However, this is unsuitable for the production of soldering rods or anhydrous pastes. A dry powder is needed for that purpose.

US-A-5171377 describes the production of a flux from caesium fluoride, aluminium fluoride and crystalline aluminium hydroxide or aluminium oxide. The complex that is formed permits a broad melting range and is suitable for soldering magnesium-containing aluminium alloys. However, a melting onset below 440°C cannot be achieved. The use of aluminium hydroxide or aluminium oxide as additives means that no acidity can be established in the flux. The acidity of the flux counteracts the oxidation of the flux.

EP-A-0785045 describes the production of a flux consisting of a caesium fluoroaluminate reaction product with varying ratios of CsF to AlF_3. Depending on the ratio of CsF to AlF_3, melting points of less than 440°C down to 427°C can be achieved with this flux. However, the flux has clearly defined melting points, which shows that it is a crystalline substance.

Thus the fluxes known from the prior art do not correspond to the requirements described in the introduction. In particular, the prior art describes no amorphous caesium fluoroaluminates as fluxes.

The present invention aims to provide a flux based on a caesium fluoroaluminate which can be obtained as a dry powder, in which a melting onset below 440°C can be established, the position and above all the breadth of the melting range can be varied and the acidity or basicity of which can be adjusted using additives. Melting onset is understood here to be the lower end of the melting range. Melting range is understood to be the temperature range extending from the first onset of melting through to complete liquefaction of the substance.

SUMMARY OF THE INVENTION

According to a broad aspect of the invention, an amorphous caesium aluminium fluoride complex is provided which can be produced according to one or more of the methods and preferred embodiments described herein.

The invention provides, a caesium aluminium fluoride complex, or CsAlF complex for short, which melts over a relatively large temperature range, i.e. which has a broad melting range. This is possible because the complex according to the invention is amorphous, i.e. it is not crystalline. In amorphous solids the molecular structural elements are arranged not in crystal lattices but randomly. This means that amorphous solids do not have a defined melting point but rather a more or less wide melting range.

In an aspect of the invention there is provided a process for the production of an amorphous CsAlF complex which is characterised by the following process steps:

a) reacting an aqueous caesium fluoride solution with solid aluminium fluoride, and establishing a desired amorphicity through the addition of acid or alkaline compounds as additives which are selected from the group consisting of $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride; the reaction time being between 15 minutes and 6 hours;

b) establishing a desired basicity or acidity in the end product through the addition of said acid or alkaline compounds; and wherein during process steps a) and b) the temperature is between 95 and 175°C, the pressure is between 0.4 and 2.5 bar absolute, and the molar ratio of said additive to AlF_3 is between 0.0001 : 1 and 0.3 : 1;

c) on completion of the reaction, applying a vacuum in order to evaporate water present in the reactor and obtain a solid; wherein the pressure is reduced to values of between 10 and 100 mbar for between 5 minutes and one hour; and

d) dewatering the solid of step c) at a temperature between 80 and 300°C and a pressure between 10 and 900 mbar.

An elemental analysis of the amorphous CsAlF complex gives substantially the following composition (in wt.%):

Cs:	45-65
Al:	5-15
F:	20-40

It is known that aluminium fluoride forms complex salts with metal fluorides, for example caesium fluoride. These complex salts are synthesised from AlF_6 octahedra. These octahedra group together to form different packing arrangements according to the size and number of the metal ions that are present. Crystals of the compounds $CsAlF_4$, Cs_2AlF_5, Cs_3AlF_6 are formed in this way. These then show the typical properties of crystalline compounds.

Through skilful reaction control, this aggregation and compound formation is prevented according to the invention so that an amorphous caesium aluminium fluoride complex is formed.

A vacuum mixing dryer known per se has proved to be a suitable reactor for this purpose. It allows multiple process steps to be performed in succession; all process steps of the production process are advantageously performed in one vacuum mixing dryer. A caesium fluoride solution and aluminium fluoride are used as the starting materials. The performance of the process steps is described below by way of example, without limiting the invention.

DETAILED DESCRIPTION OF THE INVENTION

The invention is hereby described with reference to the following process step and preferred embodiments.

<u>Process step 1:</u>

Caesium fluoride dissolved in water is bound to the aluminium fluoride in solid form. The product properties can be controlled during the reaction through the addition of additives. As has been demonstrated, the addition of alkaline or acid compounds can be used to:

1. control the reaction time
2. adjust the amorphicity of the product
3. change the colour of the product from red-brown to bright white
4. specify the properties of the product for paste production
5. achieve suitability for various soldering processes.

The temperature is between 95 and 175°C, preferably between 105 and 150°C. The pressure is between 0.4 and 2.5 bar absolute, preferably between 0.9 and 2.0 bar absolute. The reaction time is between 15 minutes and 6 hours, preferably between 30 minutes and 5 hours.

The reactants CsF and AlF_3 are used in a molar ratio of CsF to AlF_3 of between 0.9 : 1 and 3.0 : 1, preferably in a molar ratio of between 1 : 1 and 1.5 : 1. Additives such as $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride, preferably dilute hydrofluoric acid or CsOH, can be used to steer the reaction. These substances can be used individually or as a mixture. The amount of additive that is used is based on the AlF_3 that is used. The molar ratio of additive to AlF_3 is between 0.001 : 1 and 0.2 : 1, preferably between 0.008 : 1 and 0.1 : 1.

Process step 2:

Towards the end of the reaction time of process step 1, the desired basicity or acidity of the end product can be established by the addition of acid or alkaline compounds. The aforementioned additives $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride, preferably dilute hydrofluoric acid or CsOH, are used to this end. The amount of additive that is used is based on the AlF_3 that is used. The molar ratio of additive to AlF_3 is between 0.0001 : 1 and 0.3 : 1, preferably between 0.01 : 1 and 0.11 : 1. The temperature and pressure ranges correspond to those of process step 1.

Process step 3:

On completion of the reaction in process step 2, the water present in the reactor is evaporated very rapidly by application of a strong vacuum in order firstly to maintain the amorphicity of the product as far as possible and secondly to prevent the formation of undesirable secondary products. The reaction suspension changes into a solid in this process. The pressure is reduced to values of between 10 and 100 mbar, preferably between 20 and

40 mbar absolute. The time for which the water is evaporated to obtain the solid is between 5 minutes and one hour, preferably between 15 and 30 minutes.

Process step 4:

Once the solid has formed, the product is dehydrated to the desired extent at elevated temperatures and under a reduced vacuum. It is possible in this way to achieve residual moisture contents of less than 0.1%. The temperature to this end is between 80 and 300°C, preferably between 100 and 180°C. The pressure to this end is between 10 and 900 mbar, preferably between 20 and 100 mbar. In this way any crystalline hydrates that are formed, which reduce the amorphicity of the product, are destroyed again. Instead of a vacuum, a carrier gas for example can also be used to remove the moisture.

The production process can also be performed in reactors other than a vacuum mixing dryer. For process steps 1 and 2, stirred-tank reactors, kneaders or similar apparatus can also be used, for example. For process step 3, all types of drying apparatus that allow rapid drying can be used, for example spray dryers, belt dryers, fluidised-bed dryers and similar units. For process step 4, all types of drying apparatus that allow drying to be performed at elevated temperature, i.e. above 80°C, can be used, for example drying ovens or rotary kilns.

The production process according to the invention offers clear advantages over the prior art, firstly in terms of the possibility of adjusting the product properties and secondly regarding operational safety and plant costs, preferably if the process is performed in just one reactor and particularly preferably in only a vacuum mixing dryer.

The process according to the invention allows an amorphous CsAlF complex to be produced. Using an X-ray diffractometer this complex can be demonstrated to contain only traces of crystalline $CsAlF_4$ or $Cs_2AlF_5xH_2O$.

The reactant CsF cannot be detected in the product, AlF_3 only in small amounts.

The melting range of the complex can be adjusted according to the invention by controlling the reaction and varying the ratio of CsF to AlF_3. A melting onset below 420°C can be established. The product is hygroscopic and absorbs moisture slowly from the air.

These advantages over the prior art allow the CsAlF complex according to the invention to be used in a versatile manner. Owing to the multiplicity of different soldering applications, it is necessary to provide specific suitable fluxes corresponding to the individual soldering processes. With the present invention it is possible to produce a wide variety of products.

The potentially disruptive hygroscopicity of the complexes according to the invention can optionally be offset by means of processing measures: For example:

1. In the production of filler wires the flux can be shielded from the environment by the solder;

2. By compressing the flux a small surface area can be achieved and hence the moisture absorption reduced;

3. The moisture absorption can be suppressed by using organic liquids to produce suspensions.

The invention provides in detail:

- A process for the production of an amorphous CsAlF complex which is characterised by the following process steps:

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a) Binding the caesium fluoride dissolved in water to the aluminium fluoride in solid form and establishing the desired amorphicity through the addition of acid or alkaline compounds as additives,

b) Establishing the desired basicity or acidity in the end product through the addition of acid or alkaline compounds as additives,

c) Applying a strong vacuum on completion of the reaction in order to evaporate very rapidly the water present in the reactor,

d) Dewatering the product at elevated temperatures and under a reduced vacuum.

The invention also provides for a process for the production of an amorphous CsAlF complex which is characterised by the following process steps:

a) reacting an aqueous caesium fluoride solution with solid aluminium fluoride, and establishing a desired amorphicity through the addition of acid or alkaline compounds as additives which are selected from the group consisting of $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride; the reaction time being between 15 minutes and 6 hours;

b) establishing a desired basicity or acidity in the end product through the addition of said acid or alkaline compounds; and wherein during process steps a) and b) the temperature is between 95 and 175°C, the pressure is between 0.4 and 2.5 bar absolute, and the molar ratio of said additive to AlF_3 is between 0.0001 : 1 and 0.3 : 1;

c) on completion of the reaction, applying a vacuum in order to evaporate water present in the reactor and obtain a solid; wherein the pressure is reduced to values of between 10 and 100 mbar for between 5 minutes and one hour; and

d) dewatering the solid of step c) at a temperature between 80 and 300°C and a pressure between 10 and 900 mbar.

Further preferences are as follows:

- In process step a) a reaction time of between 15 minutes and 6 hours, preferably between 30 minutes and 5 hours,
- In process step a) and/or b) the addition of additives to control the product properties,
- In process step a) and/or b) the addition of acid or alkaline compounds as additives to establish the desired basicity or acidity in the end product,
- In process step a) the addition of acid or alkaline compounds as additives to adjust the amorphicity in the end product,
- In process step a) and/or b) the addition of additives $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride, particularly preferably dilute hydrofluoric acid or CsOH,

- In process step a) and/or b) a molar ratio of additive to AlF_3 of between 0.0001 : 1 and 0.3 : 1, particularly preferably between 0.008 : 1 and 0.11 : 1,

- In process steps a) and b) a temperature of between 95 and 175°C, particularly preferably between 105 and 150°C,

- In process steps a) and b) a pressure of between 0.4 and 2.5 bar absolute, particularly preferably between 0.9 and 2.0 bar absolute,

- In process step c) the application of a strong vacuum on completion of the reaction in order to evaporate very rapidly the water present in the reactor,

- In process step c) the reduction of the pressure to values of between 10 and 100 mbar absolute, particularly preferably between 20 and 40 mbar absolute,

- In process step c) the conversion of the reaction suspension to a solid,

- In process step c) a time of between 5 minutes and one hour, particularly preferably between 15 and 30 minutes, for which the water is evaporated to obtain the solid,

- In process step d) an elevated temperature and a reduced vacuum in order to dehydrate the product to the desired extent,

- In process step d) the obtaining of residual moisture contents of less than 0.1%,

- In process step d) a temperature of between 80 and 300°C, particularly preferably between 100 and 180°C, to obtain residual moisture contents of less than 0.1%,

- In process step d) a pressure of between 10 and 900 mbar, particularly preferably between 20 and 100 mbar,

- In process step d) the use of a carrier gas to remove the moisture instead of a vacuum;

- A process for producing an amorphous CsAlF complex wherein the process is performed exclusively in a single reactor, preferably in a vacuum mixing dryer;

- A process for producing an amorphous CsAlF complex wherein process steps a) and b) are performed in stirred-tank reactors and/or kneaders;

- A process for producing an amorphous CsAlF complex wherein process step c) is performed in a drying apparatus which allows rapid drying;

- A process for producing an amorphous CsAlF complex wherein process step c) is performed in a spray dryer, belt dryer or fluidised-bed dryer;

- A process for producing an amorphous CsAlF complex wherein process step d) is performed in a drying apparatus which allows drying at temperatures above 80°C;

- A process for producing an amorphous CsAlF complex wherein process step d) is performed in a drying oven or rotary kiln;

- A caesium-aluminium fluoride complex which is amorphous, obtainable by one of the processes according to the invention;

- An amorphous CsAlF complex with a melting onset below 440°C, preferably below 430°C, particularly preferably below 420°C;

- An amorphous CsAlF complex with a melting range breadth of at least 30°C, preferably at least 50°C, particularly preferably at least 60°C;

- An amorphous CsAlF complex with a melting range breadth of 30 to 90°C, preferably 30 to 80°C, particularly preferably 30 to 70°C;
- An amorphous CsAlF complex with a melting range of between 400 and 500°C, preferably between 410 and 490°C, particularly preferably between 415 and 480°C;
- An amorphous CsAlF complex containing 45 to 65 wt.%, preferably 50 to 60 wt.%, particularly preferably 55 to 60 wt.% of caesium, 5 to 15 wt.%, preferably 7 to 13 wt.%, particularly preferably 8 to 12 wt.% of aluminium and 20 to 40 wt.%, preferably 25 to 35 wt.%, particularly preferably 27 to 33 wt.% of fluorine;
- The use of an amorphous CsAlF complex as a flux for the soft soldering of aluminium.

Examples

The invention is described in more detail below by means of a number of embodiment examples, without being limited thereto:

<u>Example 1</u>: CsAlF complex having a narrow melting range

Process step a: 81 kg of an aqueous 71.4% caesium fluoride solution are placed in a vacuum mixing dryer with vapour condenser and vacuum system and 21 kg of AlF_3 are added. The mixture is stirred under normal conditions until a homogeneous suspension is formed. Then 50 kg of 0.2% hydrofluoric acid are added to the suspension as an additive. The mixture is heated to boiling under normal pressure with stirring and is refluxed for 4.5 hours.

Process step b: 5 kg of a 0.1% caesium hydroxide solution are added to the boiling suspension as an additive. 80 kg of water are evaporated off from the suspension within one hour. The vapour is condensed and the volume used to determine the end point of evaporation.

Process step c: A vacuum is applied. A residual pressure of 200 mbar is reached within 5 minutes. The remaining water evaporates very rapidly. After 30 minutes the pulp turns into a free-flowing powder. The residual pressure at the end is 35 mbar.

Process step d: The product is then dried with stirring under maximum vacuum. A product temperature of 160°C is reached. The drying process is completed after 12 hours and the powder is cooled to 30°C and removed.

The CsAlF complex obtained has a pH of 6.9, a residual moisture content of 0.8% and a melting interval as follows: onset = 474°C, end = 478°C. Figure 1: DSC (differential scanning calorimetry).

Example 2: CsAlF complex having a broad melting range

Process step a: 76 kg of an aqueous 75.9% caesium fluoride solution are placed in a vacuum mixing dryer with planetary mixer, vapour condenser and vacuum system and 22 kg of AlF_3 are added. The mixture is stirred under normal conditions until a homogeneous suspension is formed. Then 0.9 kg of caesium carbonate are added to the suspension as an additive. The mixture is heated to boiling under normal pressure with stirring and is refluxed for one hour.

Process step b: 5 kg of 0.1% hydrofluoric acid are added to the boiling suspension as an additive. The suspension is heated to boiling under normal pressure.

Process step c: A vacuum is applied. The pressure drops continuously down to 50 mbar. After one hour so much water has evaporated off that a dry powder has been obtained. The residual pressure at the end is 20 mbar.

Process step d: The product is then dried further with stirring under maximum vacuum. A product temperature of 180°C is reached. The drying process is completed after 8 hours and the powder is cooled to 40°C and removed.

The CsAlF complex obtained has a pH of 6.6, a residual moisture content of 0.1 % and a melting interval with multiple peaks from 419 to 472°C; Figure 2: DSC.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A process for the production of an amorphous CsAlF complex which is characterised by the following process steps:

   a) reacting an aqueous caesium fluoride solution with solid aluminium fluoride, and establishing a desired amorphicity through the addition of acid or alkaline compounds as additives which are selected from the group consisting of $CsHCO_3$, Cs_2CO_3, CsOH, dilute hydrofluoric acid or caesium bifluoride; the reaction time being between 15 minutes and 6 hours;

   b) establishing a desired basicity or acidity in the end product through the addition of said acid or alkaline compounds; and wherein during process steps a) and b) the temperature is between 95 and 175°C, the pressure is between 0.4 and 2.5 bar absolute, and the molar ratio of said additive to AlF_3 is between 0.0001 : 1 and 0.3 : 1;

   c) on completion of the reaction, applying a vacuum in order to evaporate water present in the reactor and obtain a solid; wherein the pressure is reduced to values of between 10 and 100 mbar for between 5 minutes and one hour; and

   d) dewatering the solid of step c) at a temperature between 80 and 300°C and a pressure between 10 and 900 mbar.

2. A process according to claim 1, characterised in that during process steps a) and b) the temperature is between 105 and 150°C.

3. A process according to claim 1 or 2, characterised in that during process steps a) and b) the pressure is between 0.9 and 2.0 bar absolute.

4. A process according to any one of claims 1 to 3, characterised in that during process step a) the reaction time is between 30 minutes and 5 hours.

5. A process according to any one of claims 1 to 4, characterised in that during process step a) and/or b) the additives are dilute hydrofluoric acid or CsOH.

6. A process according to any one of claims 1 to 5, characterised in that the

molar ratio of additive to AlF_3 is between 0.008 : 1 and 0.11 : 1.

7. A process according to any one of claims 1 to 6, characterised in that during process step c) the pressure is reduced to values of between 20 and 40 mbar absolute.

8. A process according to any one of claims 1 to 7, characterised in that during process step c) the time for which the water is evaporated to obtain the solid is between 15 and 30 minutes.

9. A process according to any one of claims 1 to 8, characterised in that during process step d) a residual moisture content of less than 0.1% is achieved.

10. A process according to any one of claims 1 to 9, characterised in that during process step d) the temperature is between 100 and 180°C.

11. A process according to any one of claims 1 to 10, characterised in that during process step d) the pressure is between 20 and 100 mbar.

12. A process according to any one of claims 1 to 11, characterised in that during process step d) a carrier gas can be used to remove the moisture instead of a vacuum.

13. A process according to any one of claims 1 to 12, characterised in that the process is performed exclusively in a single reactor, preferably in a vacuum mixing dryer.

14. A process according to any one of claims 1 to 13, characterised in that process steps a) and/or b) are performed in stirred-tank reactors and/or kneaders.

15. A process according to any one of claims 1 to 14, characterised in that process step c) is performed in a drying apparatus which allows rapid drying.

16. A process according to any one of claims 1 to 15, characterised in that process step c) is performed in a spray dryer, belt dryer or fluidised-bed

dryer.

17. A process according to any one of claims 1 to 16, characterised in that process step d) is performed in a drying apparatus which allows drying at temperatures above 80°C.

18. A process according to any one of claims 1 to 17, characterised in that process step d) is performed in a drying oven or rotary kiln.

19. An amorphous CsAlF complex obtained by the process according to any one of claims 1 to 18.

20. A CsAlF complex according to claim 19, characterised in that the onset melting point of the complex is below 440°C.

21. A CsAlF complex according to claim 20, characterised in that the onset melting point of the complex is below 430°C.

22. A CsAlF complex according to claim 20, characterised in that the onset melting point of the complex is below 420°C.

23. A CsAlF complex according to claim 19, characterised in that the melting range of the complex is between 400 and 500°C.

24. A CsAlF complex according to claim 23, characterised in that the melting range of the complex is between 410 to 490°C.

25. A CsAlF complex according to claim 23, characterised in that the melting range of the complex is between 415 and 480°C.

26. A CsAlF complex according to any one of claims 19 to 25, characterised in that it contains 45 to 65 wt.% of caesium, 5 to 15 wt.% of aluminium and 20 to 40 wt.% of fluorine.

27. A CsAlF complex according to claim 26, characterised in that it contains 50 to 60 wt.% of caesium, 7 to 13 wt% of aluminium and 25 to 35 wt.% of fluorine.

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28. A CsAlF complex according to claim 26, characterised in that it contains 55 to 60 wt.% of caesium, 8 to 12 wt.% of aluminium and 27 to 33 wt.% of fluorine.

29. Use of a CsAlF complex according to any one of claims 19 to 28, as a flux, preferably for the soft soldering of aluminium.

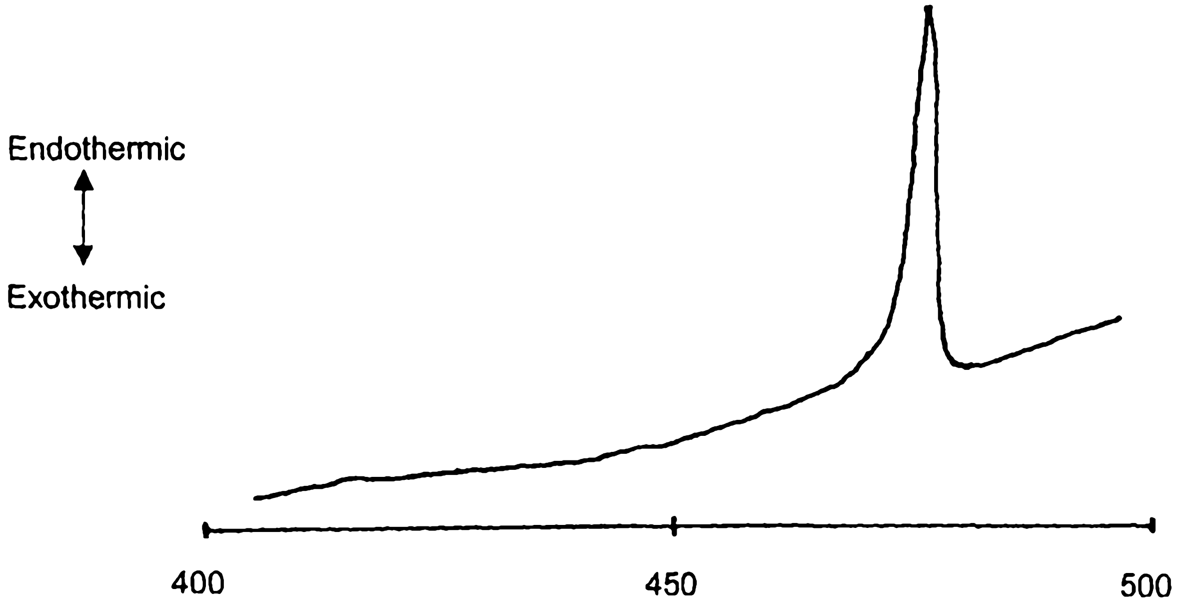
30. A process for the production of an amorphous CsAlF complex substantially as hereinbefore described with reference to the Examples.

CHEMETALL GMBH

WATERMARK PATENT AND TRADE MARKS ATTORNEYS

P30497AU00

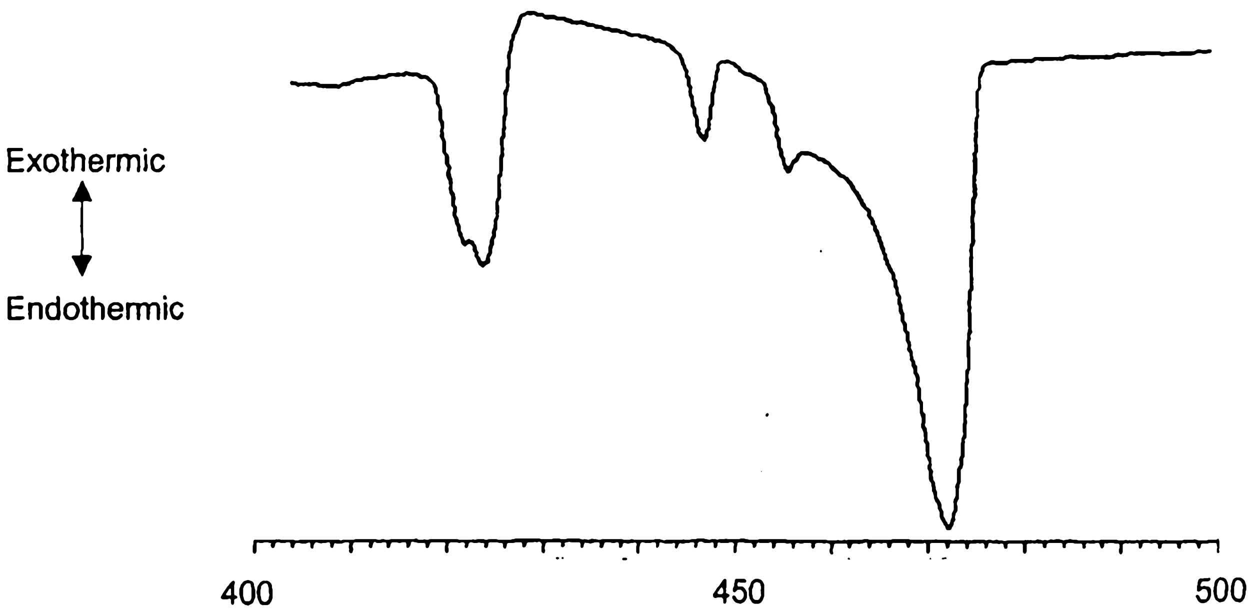
Figure 1:



Abcissa: Temperature [°C]

Ordinate: Heat flow

Figure 2:



Abcissa: Temperature [°C]

Ordinate: Heat flow