

COMMISSIONER'S DECISION

OBVIOUSNESS: Dehalogenation Process

Some claims of this application, which related to the conversion of metal halides were refused for failing to define a patentable advance in the art over the reference cited. The rejection against claim 19 was withdrawn.

Final Action: Affirmed in part.

This decision deals with a request for review by the Commissioner of Patents of the Examiner's Final Action dated May 27, 1975, on application 126,631 (Class 23-224). The application was filed on November 2, 1971, in the name of Leonard N. Brigham et al, and is entitled "Dehalogenation Process."

The general subject matter of this invention is the conversion of metal halides, represented by MX_4 , to the corresponding oxides MO_4 . To accomplish this by prior art processes using hydrogen as the dehalogenating agent required temperatures in excess of 2000°F . The use of such high temperatures produced oxide powders with less than optimum surface characteristics. The present inventors disclosed that they could convert oxyhalides, represented by MO_2X_2 , to MO_4 at temperatures from 600°F . to 1600°F . using a vapourized alcohol as a dehalogenating agent and subsequently filed a supplementary disclosure widening the scope of the materials treated from oxyhalides to halides in general.

In the Final Action the examiner refused claims 13-18 and 20-23 of the supplementary disclosure for lack of invention in view of the following United States Patent:

3,000,703

Sept. 19, 1961

Brugger

Claim 19 was also refused on the ground of lack of support in the disclosure.

In that action the examiner stated (in part):

Applicant, in his letter of February 3, 1975, has argued against the relevance of United States patent 3,000,703 as applied in the

rejection of claims 13 to 18 and 20 to 23 because he alleges that there are significant differences between the present invention as set forth in revised claims and the prior art.

The first "difference" discussed by the applicant relates to the surface area and the bulk density of the zirconia product formed by the dehalogenation reaction. Applicant's specification teaches on page 2 lines 2 to 30 "To dehalogenate a metallic oxyhalide such as uranium oxyfluoride one needs, when using hydrogen, a temperature in excess of about 2000°F to have a practical rate of dehalogenation. Such a temperature produces undesirable properties in the resulting ceramic including the loss of the ability to make dense compacted ceramic bodies from the resulting powder due to a deadening of the powder (loss of surface area of the powder) at the temperature required for the dehalogenation process. In order to lower the temperature for conducting successful dehalogenation of metallic oxyhalides a wet hydrogen atmosphere has been used which has the effect of increasing the rate of dehalogenation at any given temperature when compared to the use of dry hydrogen. This also has a practical effect of lowering the temperature needed to achieve a practical rate of dehalogenation of a metallic oxyhalide. The dehalogenation process using wet hydrogen gives economies of operation and an increased powder activity in that there is greater ability of the powder to be compacted and sintered to dense structures.

In spite of the foregoing, it has still remained desirable to lower the temperature for achieving a practical, rapid rate of dehalogenation of metallic oxyhalide containing compositions. The lower temperature of dehalogenation of metallic oxyhalides enables even greater powder activity, enables greater economies of operation and gives more dense structures after the powders are compacted and sintered." And on page 6 lines 10 to 31 "While any temperature achieving dehalogenation can be employed by utilizing heated furnaces receiving the alcohol-containing atmosphere, the temperature is generally under about 1600°F and preferably under about 1100°F where it is desired to have a powder of high surface area with the range of temperature being about 600 to about 1600°F and a preferred range of temperature being about 600 to about 1100°F. This range gives a rapid rate of dehalogenation while preserving high surface area of the dehalogenated powder. In general the higher the temperature used the greater the rate of dehalogenation achieved with greater hydrocarbon impurity content of the dehalogenated powder. The lower temperatures in the foregoing range give the highest surface areas for the defluorinated powder. Where the atmosphere used in the furnace contains only vaporized alcohol, a higher temperature up to about 1600°F can be utilized for the dehalogenation step without loss of activity of the dehalogenated powder with a particularly preferred range of temperature being about 1200 to about 1600°F. Again the rate of dehalogenation and the surface area of the dehalogenated powder vary with the particular temperature in the foregoing range as noted above."

Both of these quotations indicate that low temperatures for the dehalogenation reaction promote the formation of products of high surface area and high bulk density. Thus, the Brugger process, which recommends the use of lower temperatures than those of the instant specification, should result in the production of high surface area,

high bulk density oxide products. The Brugger teaching confirms this contention in column 1 lines 9 to 11, column 2 lines 3 to 34, column 2 line 65 to column 3 line 7 and column 5 lines 51 to 55 of the patent. Each of the specified passages refers to a method of obtaining a high-purity zirconium oxide having a high bulk density in a porous granular form, ideal for making refractory objects as well as for use in the ceramic and glass industry, and thus precisely the type of product applicant desires to obtain.

The applicant in his response dated August 15, 1975, to the Final

Action stated (in part):

The Brugger reference is directed to a two-step method of conversion of a halide to an oxide, and is limited in its teaching to a particular halide compound, namely zirconium tetrachloride. Brugger's process involves heating in the presence of a hydrolysing agent, followed by a calcination step conducted at a substantially higher temperature than the hydrolyzing step.

A two stage heating cycle is not employed in the subject invention.

In the matter of rejection of claim 19, attention is directed to page 7 lines 5 through 11, which deals with the dehydration step referred to by the Examiner.

So far as application of the Brugger reference is concerned, attention is drawn to the case Canadian General Electric Co. Ltd. v Fada Radio Ltd. 1927 Supreme Court Report (SCR) 520 "....The true construction of a patent specification must be based on the specification alone, to derive the intentions of its authors...." Also "....Any information as to the alleged invention given by any prior publication must be for the purpose of practical utility equal to that given by the subject patent." In the foregoing, for "patent" substitute --application--.

The Brugger reference shows the conversion of zirconium chloride to zirconium oxide by heating the zirconium chloride in the presence of a vapourized alcohol. The temperature range taught by Brugger is based on the practical considerations which would appear necessarily to apply to anyone carrying out this reaction: high enough to maintain the alcohol in the vapour state and low enough to prevent excess loss of the halide through volatilization. More specifically he describes a method of dehalogenating zirconium tetrachloride comprising the step of heating the halide at temperatures in the range from about 120°C to 1200°C (248°F to 2192°F) in a dehalogenating atmosphere having as the dehalogenating component a vaporized alcohol.

Brugger also describes certain inherent advantages including: (a) the potential for employing lower dehalogenation temperatures than those recorded for other prior art processes; (b) the retention of active surface characteristics on the metal oxide product; (c) the production of a high purity, high density product which can easily be processed into highly refractory articles or structural material; and (d) the recovery of a highly concentrated hydrohalic acid by-product which are attainable through the use of a vaporized alcohol dehalogenation agent. These specified advantages correspond substantially to the desired objectives of the inventors of the instant application.

Claim 1 of the Brugger citation reads as follows:

A process for the production of zirconium oxide which comprises:

- (a) introducing solid zirconium tetrachloride into a reaction zone,
- (b) simultaneously introducing into the reaction zone a hydrolyzing agent,
- (c) said hydrolyzing agent having the formula ROH wherein R is selected from the group consisting of hydrogen and alkyl radicals containing between one and five carbon atoms,
- (d) maintaining the temperature in the reaction zone above the boiling point of the hydrolyzing agent but below the sublimation point of the zirconium tetrachloride,
- (e) maintaining the said reactants in the reaction zone for from about 1-5 hours in order to substantially eliminate the chloride from the resulting zirconium oxide product, and
- (f) thereafter calcining the resulting product to convert hydrous zirconia to zirconium oxide.

This application relates to the conversion of metal halides represented by MX_4 , to the corresponding oxides MO_4 at temperatures from 600°F to 1600°F using a vaporized alcohol as a dehalogenating agent. A supplementary disclosure was filed to broaden the scope of the materials treated from oxyhalides to halides in general.

Claim 13 (supported by supplementary disclosure) reads as follows:

A method of dehalogenating a composition including a metallic halide wherein the metallic portion is selected from the group consisting of uranium, plutonium, titanium, zirconium, silicon, tungsten, gadolinium, aluminum and mixtures thereof comprising the step of heating the composition at a temperature in the range of about 600°F to about 1600°F in a dehalogenating atmosphere having as the dehalogenating component a vaporized alcohol.

The question to be considered is whether claims 13 to 18 and 20 to 23 are too broad in scope in view of the Brugger citation.

We observe that the Brugger reference is restricted in its teaching to the dehalogenation of zirconium tetrachloride for the production of zirconia, whereas this application relates to the dehalogenation of metallic halides in which the metallic portion is selected from the Markush group consisting of uranium, plutonium, titanium, zirconium, silicon, tungsten, gadolinium, aluminium and mixtures thereof. The metallic halides encompassed by the applicant's claimed processes have a wide range of physical and chemical properties and under conditions of dehalogenating may require non-uniform processing techniques to assure the retention of desirable surface characteristics on the metal oxide products. These possible anomalies are not disclosed in applicant's specification but the adequacy of the supplementary disclosure for the support of the presently rejected claims was not challenged since it has been made evident that in the course of preparing a pure uranium oxide, the dehalogenating technique employing alcoholic reagents, utilized in the process of the principal disclosure for the dehalogenation of metal oxyhalides, was also found effective to dehalogenate the metallic halides of claim 13. The single example included in the supplementary disclosure describes to a highly restricted embodiment wherein the halides are present only in trace amounts. No teaching of particular temperature conditions or handling requirements specific to the treatment of the various metallic halides has been supplied. It is therefore clear that the only possible invention encompassed by the refused claims rests entirely in the selection of a vaporized alcohol to serve as the active agent in a dehalogenation reaction. This feature, however, is obvious in view of the Brugger citation.

The applicant, in his response to the Final Action, has relied upon the apparent difference in the defined dehalogenation temperature to distinguish the processes of the rejected claims from the teaching of the reference.

However, it is observed that the main thrust of applicant's disclosure relates to the development of a dehalogenation process which operates at temperatures sufficiently low to avoid producing undesirable surface characteristics in the resulting ceramic product. This desired improvement was effectively achieved through the use of low initial reaction temperatures in the process described by the Brugger patent. Furthermore it may be reemphasized that once the sublimation temperature of the partially dehalogenated zirconium chloride was sufficiently elevated to prevent any significant loss of zirconium tetrachloride through volatilization, the reaction temperature in the Brugger process was raised to a range from about 500°C. to 550°C. (932°F. to 1022°F.) for the completion of the reaction. This value lies within the range of useful temperatures defined by rejected claim 13.

The applicant argued that "a two stage heating cycle is not employed in the subject invention." We note, however, that Brugger shows all aspects of the process of converting $ZrCl_4$ to ZrO_2 taught by the present applicant. The one stage heating cycle is not, in our view, patentably significant over a two stage heating cycle in the present circumstances.

The applicant relied upon a Supreme Court decision, Canadian General Electric Co. Ltd. v Fada Radio Ltd., and quoted a passage from it to support his argument against the refusal of certain claims. The passage, however, "... Any information as to the alleged invention given by any prior publication must be for the purpose of practical utility equal to that given by the subject patent," does not appear in that case. It is, however, in The King v Uhlemann Optical Company (1950) Ex. C.R. 142 at 157 where we find: "The requirements that must be met before an invention should be held to have been anticipated by a prior publication have been discussed in many cases and may be stated briefly. The information as to the alleged invention given by the prior publication must, for the purpose of practical utility, be equal to that given by the subsequent patent." (underlining added) It is clear, however, that

the court was concerned with anticipation and not with obviousness or lack of invention. Notwithstanding the above, the Brugger citation does describe a process for the dehalogenation of zirconium tetrachloride to yield zirconium oxide. The patented technique has a practical utility substantially equal to that of the process in refused claim 13, which process encompasses the same reaction for the same purpose.

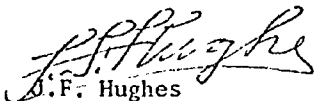
We are satisfied that the similarity of the process defined by claim 13 to the teaching of the Brugger citation makes it clear that there is no patentable advance made in the art. The remainder of the refused claims, namely claims 14 to 18 and 20 to 23, which are essentially dependent on claim 13, contain minor restrictions which are not deemed inventive.

It is also noted that claim 19 was refused for lack of support in the disclosure.

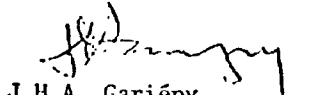
The claimed embodiment is, however, supported by the disclosure on page 7, lines 5 through 11. That rejection is therefore withdrawn. It is noted that this claim should appear with those claims supported by the principle disclosure rather than under the heading "Claims Supported by Supplementary Disclosure."

We recommend that the decision in the Final Action to refuse claims 13 to 18 and 20 to 23 be affirmed, and that the ground for refusing claim 19 be withdrawn.

Claim 19 should therefore be renumbered "claim 13."


J.F. Hughes
Assistant Chairman
Patent Appeal Board

I concur with the recommendations of the Patent Appeal Board. Accordingly, I refuse to grant a patent on claims 13 to 18 and 20 to 23. The ground for the rejection against claim 19 is withdrawn. The applicant has six months within which to submit an appropriate amendment cancelling the refused claims, or to appeal this decision under the provision of Section 44 of the Patent Act.


J.H.A. Gariépy
Commissioner of Patents

Dated at Hull, Quebec
this 3rd. day of May, 1976

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