Datasheet for the decision
of 13 February 2012

Case Number: T 1932/08 - 3.3.07
Application Number: 01927774.8
Publication Number: 1268058
IPC: B01J 29/04, C07D 301/12,
C07C 249/04
Language of the proceedings: EN

Title of invention:
Process for the production of a titanium silicalite shaped article

Patent Proprietor:
Evonik Degussa GmbH

Opponent:
BASF SE

Headword:
-

Relevant legal provisions:
EPC Art. 100(a)(b)

Keyword:
"Disclosure - sufficiency (yes)"
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-
Case Number: T 1932/08 - 3.3.07

DECISION
of the Technical Board of Appeal 3.3.07
of 13 February 2012

Appellant: BASF SE
(Opponent) D-67056 Ludwigshafen (DE)

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Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 24 July 2008 rejecting the opposition filed against European patent No. 1268058 pursuant to Article 101(2) EPC.

Composition of the Board:
Chairman: J. Riolo
Members: D. Semino
M.-B. Tardo-Dino
I. The appeal of the opponent lies against the decision of the opposition division announced at the oral proceedings on 1 July 2008 to reject the opposition against European Patent 1 268 058. The granted patent comprised 11 claims, independent claims 1 and 8 reading as follows:

"1. Process for the production of a titanium silicalite shaped article, the titanium silicalite having MFI or MEL crystal structure, by:
   a) preparation of a synthesis gel containing a SiO$_2$ source, a TiO$_2$ source, tetra-n-propyl ammonium hydroxide or tetra-n-butyl ammonium hydroxide as a template compound and water,
   b) crystallisation of the synthesis gel under hydrothermal conditions,
   c) drying of the titanium silicalite from step b) at a temperature below the decomposition temperature of the template compound,
   d) preparation of a formable mass containing the product from step c), a binder selected from any of compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium and a paste-forming agent, 
   e) forming of the mass from step d) into a green shaped article,
   f) optionally drying, and
   g) calcination of the green shaped article."

"8. Process for the production of a titanium silicalite shaped article the titanium silicalite having MFI or MEL crystal structure, by:
a) preparation of a synthesis gel containing a SiO$_2$ source, a TiO$_2$ source, tetra-n-propyl ammonium hydroxide or tetra-n-butyl ammonium hydroxide as a template compound and water,
b) crystallisation of the synthesis gel under hydrothermal conditions,
c) concentration of the crystal suspension from step b),
d) preparation of a formable mass containing the product from step c) and a binder selected from compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium,
e) forming of the mass from step d) into a green shaped article,
f) optionally drying, and
g) calcination of the green shaped article.

II. A notice of opposition had been filed against the granted patent requesting revocation of the patent on the grounds of insufficiency of disclosure, lack of novelty and lack of inventive step in accordance with Article 100(a) and (b) EPC. The opposition was inter alia supported by the following documents:

D1: DE-A-196 23 972
D5: WO-A-00/12432
III. The decision under appeal can be summarised as follows:

(a) The objections under Article 100 (b) EPC that the drying and concentration steps could not be performed by a skilled person did not prejudice maintenance of the patent, because they had not been substantiated by verifiable facts.

(b) The method of granted claim 1 was novel with respect to D1, because that document did not disclose preparation of a formable mass containing a dried, but not yet calcined titanium silicalite, the use of a binder selected from compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium and the use of a paste-forming agent. Similarly, the method of granted claim 8 was novel with respect to D1, because that document did not disclose a concentration step for the crystal suspension of titanium silicalite, preparation of a formable mass containing the concentrated titanium silicalite and the use of a binder selected from compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium.

(c) The methods of granted claims 1 and 8 were inventive with respect to D3, which was the closest prior art, in combination with D1, because, although there was an indication in D1 that the product might or might not be calcined before shaping, no technical effect was described as related to that choice. A combination of D1 or D3 with D5 would not be obvious, because that document disclosed totally different titanium
silicalite products used for different catalytic reactions. Moreover, the skilled person could chose D5 as the closest prior art only with hindsight. Therefore the subject-matter of claims 1 and 8 involved an inventive step.

IV. The opponent (appellant) appealed that decision.

V. With the reply to the statement of grounds the patent proprietor (respondent) provided counterarguments to the ones of the appellant. With a later letter dated 12 January 2012 the respondent filed three set of claims as auxiliary requests 1 to 3.

VI. Oral proceedings were held on 13 February 2012.

VII. The arguments of the appellant (opponent) can be summarised as follows:

Sufficiency of disclosure

(a) The drying step according to the process of claim 1 was not sufficiently disclosed in the patent in suit. Only spray drying was mentioned in the examples and even for that mode of realisation no detail was given. The only citation in the description relating to drying was clearly wrong (since the cited patent concerned loudspeakers) and neither a possible alternative document which should have been meant in place of that citation, nor the few documents mentioned in the appealed decision gave sufficient information about how to dry a titanium silicalite suspension.
(b) There was no example in the patent in suit falling under the process of claim 8, namely no example in which a concentrated suspension was submitted to a forming step without any intermediate step, so that the process of claim 8 was not sufficiently disclosed. The allegation in the appealed decision that the concentration of a crystal suspension was a common and basic industrial operation was not substantiated by sufficient evidence.

Novelty

(c) Document D1 disclosed a process according to granted claim 1, including the three features which had been indentified as distinguishing in the appealed decision. D1 disclosed unambiguously the sequence of a washing step and a drying step followed by calcination; furthermore it contained the information that a forming step could take place before or after calcination. Those disclosures amounted to the anticipation of a process containing all the steps of claim 1 in the given order. No choice from multiple lists was necessary to come to that conclusion. The use of a binder selected from compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium was a matter of course in view of the disclosure in D1 to use binders known from the state of the art and taking into account the disclosure of prior art documents, such as D8 and D2, the latter being also cited in D1. The use of a paste-forming agent, when preparing a formable mass, was also a matter of course, which the skilled person would consider as implicitly
disclosed, when taking into account the shaping of titanium zeolites by means of appropriate binders.

(d) D1 disclosed also a process according to granted claim 8. The filtration, centrifugation or decantation of the crystal suspension disclosed therein anticipated the concentration step according to claim 8. In this respect it was to be noted that the claim did not specify that the product of the concentration had still to be a suspension, nor gave a quantitative indication of the water content after concentration. As far as the sequence of steps and the use of specific binders were concerned, the same arguments were applicable as for granted claim 1.

Inventive step

(e) The closest prior art was represented by D1 and not by D3, since that document disclosed the crucial feature of the process of granted claim 1, namely that forming took place between drying and calcination and addressed a problem similar to the one of the patent in suit. D1 disclosed that forming could take place before calcination and gave several example of possible forming steps, including extrusion, which clearly showed that there was no different understanding of what was meant by forming in D1 and in the patent in suit. Since calcination was the process step meant to eliminate the template from the catalyst, forming could take place also on an intermediate product still containing the template. D1 moreover specified the possibility of accomplishing drying
before calcination. Those disclosures in D1 amounted to a direct and unambiguous disclosure of the sequence of process steps according to granted claim 1. The only possible differences between the process of granted claim 1 and the one disclosed in D1 were that in D1 no explicit disclosure was given of specific binders and of the use of a paste-forming agent. While it was clear from D1 that the properties of the produced catalyst had to be good, no relevance could be given to the mechanical properties of the product obtained by the method claimed in the patent in suit, since similar results were obtained for some examples and some comparative examples in the patent. Moreover, no comparative example was meant at reproducing the process of D1 and comparing it with the claimed process. For those reasons, the problem to be solved was simply that of providing an alternative process. The choice of the listed binders and the use of a paste-forming agent, which included the simple addition of water, could not provide any inventive step, since they were usual measures, known e.g. from D3 or also from D5, which disclosed the same process, even if the obtained product had a RUT-structure. The process of granted claim 1 was therefore not inventive.

(f) Similar arguments applied to the process of granted claim 8. D1 was still the closest state of the art and it disclosed the complete sequence of process steps according to granted claim 8 as well. Example 4 in the patent showed that the step of drying at least a part of the suspension of the crystals was necessary, so that the process as
claimed did not provide any advantage. Moreover, the wording of claim 8 did not exclude the presence of an intermediate washing step and filtration, centrifugation and decantation as possible concentration steps. Since the choice of the listed binders was obvious in view of the prior art, as represented e.g. from D3, lack of inventive step resulted also for the process of granted claim 8.

VIII. The arguments of the respondent (patent proprietor) can be summarised as follows:

Sufficiency of disclosure

(a) Drying was a common process step in the field of shaped catalyst production and information was given in paragraph [0017] and in the examples of the patent in suit about how to carry it out, so that the process of claim 1 was sufficiently disclosed. Moreover, there was no difficulty in finding the right document which was meant in the description despite the error in the citation, as confirmed by the fact that the appellant had found it promptly.

(b) Example 4 of the patent in suit described an embodiment of the process of claim 8, which did not exclude that additional components were used together with the concentrated suspension and the binder in the preparation of the formable mass. In any case the presence of a concrete example was not a precondition of sufficiency of disclosure, which was a requirement of the whole patent. The
patent gave sufficient information regarding the concentration step in paragraph [0016] and that step was in any case a well known process step in the field. In the absence of evidence on the side of the appellant that the invention could not be reproduced, sufficiency of disclosure should be acknowledged.

Novelty

(c) As far as claim 1 was concerned, document D1 disclosed a drying step only in relation to a directly following calcination and not with a forming step in between. Moreover, forming was intended in a much broader sense in D1 than in the patent in suit, since it included also processes not leading to a shaped product, such as spray drying. No specific binder was mentioned in D1, nor the binder disclosed in D2 could be considered as part of the disclosure of D1, since D2 was cited in D1 in a completely different context. Moreover, the forming step including the use of binder was disclosed in D1 only in the context of forming of a calcinated product. In addition, no disclosure of a paste-forming agent was present in D1, nor was any value given for the temperature of the drying step.

(d) With regard to claim 8, the filtration, centrifugation and decantation steps of D1, which resulted in a separation of the crystals from the mother liquor and were followed by a washing step, could not be considered as a concentration of the crystal suspension, which was intended to produce
a more concentrated suspension of the titanium silicalite crystals. In any case, neither the sequence of a concentration step directly followed by a forming step and a subsequent calcination, nor the use of the specific binders listed in claim 8 were disclosed in D1.

Inventive step

(e) The scope of the patent in suit was that of providing an economic process for the production of titanium silicalite shaped articles, while improving the mechanical properties of the product without affecting its other properties. While D3 addressed the same problem, D1 aimed at a simple process for the production of titanium silicalite from cheap starting materials. Forming was a side aspect in D1 and did not appear in its examples; moreover, the relevance of the mechanical properties of the shaped product was not mentioned therein. For those reasons, D3 had to be considered as the closest prior art under application of the criteria commonly accepted in the case law. The process of claim 1 differed from the one disclosed in D3 mainly in that forming took place before calcination. That difference rendered one calcination step superfluous and therefore the process more economic. Moreover, a proper analysis of the examples and comparative examples showed that by means of that difference the mechanical properties of the product were improved. In that analysis each example had to be compared with the corresponding comparative example in which only the distinguishing feature
had been changed. The skilled person, looking for a more efficient process by means of which products with improved mechanical properties were obtained, would not find a hint to the proposed solution in the available prior art. In particular, the sequence of process steps according to granted claim 1 was not disclosed in D1 and D5 related to titanium silicalite with a different structure and did not mention the improvement of the product mechanical properties. Therefore, the process of granted claim 1 was inventive with regard to the available prior art. Even if D1 were taken as the starting point for the analysis of inventive step, the same conclusion would be obtained. The process of granted claim 1 differed from the disclosure of D1 in three aspects as outlined in the analysis of novelty. In particular, the production of a green shaped article was disclosed in D1 only starting from an already calcined product from which the template had been eliminated. While it was not sensible to try to reproduce any of the examples of D1 as comparative example, since none of them included the preparation of a green shaped product, the comparative examples on file were still relevant to show the effect of at least one distinguishing feature. In view of this, the same problem as formulated for D3 was solved with respect to D1. Neither D3, which suggested the use of a mixture of water and alcohol as paste-forming agent, nor D5, which did not address the improvement of mechanical properties, provided any hint towards the proposed solution.
(f) The same reasoning applied to the process of granted claim 8. Example 4 of the patent in suit was representative of that process as detailed in the context of sufficiency of disclosure and concentration had to be understood as a process step aimed at obtaining a concentrated suspension as explained in the context of novelty. D3 was still to be considered as the closest prior art, as D1 was more remote and did not disclose the critical sequence of process steps. Therefore, following the same line of arguments, the presence of an inventive step had to be acknowledged.

IX. The appellant (opponent) requested that the decision under appeal be set aside and the European patent be revoked.

X. The respondent (patent proprietor) requested that the appeal be dismissed or, alternatively, that the patent be maintained on the basis of the set of claims of one of auxiliary requests 1 to 3 as submitted with the letter of 12 January 2012.

Reasons for the Decision

1. The appeal is admissible.

2. Sufficiency of disclosure

2.1 According to the case law (Case Law of the Boards of Appeal of the European Patent Office, 6th edition 2010, II.A.7) lack of sufficient disclosure presupposes that there are serious doubts, substantiated by verifiable
facts, and in order to establish insufficiency, the burden of proof is upon an opponent to establish on the balance of probabilities that a skilled reader of the patent, using his common general knowledge, would be unable to carry out the invention.

2.2 In the present case the appellant-opponent alleges that the drying step according to the process of claim 1 and the concentration step followed by the forming step of the process of claim 8 are not sufficiently disclosed, but does not provide any evidence in this respect. In particular, the appellant has neither attempted to carry out the processes of the invention to show that they cannot be reproduced without undue burden, nor provided any verifiable facts that this should be the case.

2.3 The Board considers drying and concentration as well-known unit operations in the field of processing of chemical products and does not see any reason to presuppose that there may be serious doubt that they cannot be carried out. In the absence of evidence on the side of the party who bears the burden of proof, it can only conclude that the objections under Article 100(b) EPC do not prejudice maintenance of the patent as granted.

2.4 This conclusion cannot be affected by an error which took place in the citation of a prior art document (see the citation of DE-A-197 31 672 in paragraph [0017] of the patent in suit, whereby that document relates to loudspeakers and therefore is obviously unrelated to the present invention), since there is no requirement that prior art documents are cited for the realisation
of any step of a claimed process, in particular when, as in the present case, these steps are well-known. Neither is it relevant whether example 4 is an embodiment of the process of claim 8 (which is indeed the case, see point 4.6, below), since, independently of the details of that example, it cannot be put in doubt without verifiable facts that the skilled person is able to accomplish a well-known concentration step of a crystal suspension.

2.5 Finally, it is noted that it is not up to the opposition division, as alleged by the appellant, to provide a sufficiently large number of documents to support the view that the contested steps are well-known in the field, when, as in the present case, the party who bears the burden of proof has not been able to discharge it.

3. **Novelty**

3.1 Document D1 discloses a process for the production of a titanium containing crystalline molecular sieves of chemical formula \((\text{SiO}_2)_{1-x}(\text{TiO}_2)_x\) (page 2, lines 3-4), by preparation of a synthesis gel from \(\text{SiO}_2\) as silicium source, a titanium source and a template compound in an aqueous solution, crystallisation of the synthesis gel under hydrothermal conditions, separation of the solid, washing and calcination, wherein the solid can be formed before or after calcination (page 3, lines 3-11; claim 1). As template tetra-n-propyl ammonium hydroxide is preferably used, when a MFI crystal structure is desired, and tetra-n-buty ammonium hydroxide is employed, when a MEL structure is aimed at (page 3, lines 29-35).
3.2 In detail, it is disclosed that the crystals are separated from the mother liquor by means of filtration, centrifugation or decantation and are washed with a suitable fluid, preferably water (page 4, lines 2-4). Thereafter the crystals are optionally dried and then they are calcined at a temperature between 300 and 1000°C to remove the template (page 4, lines 4-5). No forming is mentioned in this context.

3.3 As to the forming of crystalline, titanium containing molecular sieves in powder form, D1 discloses that the powder catalysts can be brought into a form suitable for their use as oxidation catalysts by a known forming process, such as granulation, spray drying, spray granulation or extrusion (page 4, lines 31-35). Binders according to the state of the art can be used in the forming step for the processing of powder catalysts (page 4, lines 38-40). Preferably use is made of binders, which after forming and through calcination are left in a form, which does not show any acid reaction and is inert with respect to hydrogen peroxide (page 4, lines 40-42). None of the examples contains a forming step (pages 4-7).

3.4 Summing up, while document D1 does mention in claim 1 and in a passage of the description where the wording of claim 1 is repeated that the solid crystals can undergo a forming step before or after calcination, it does not contain any concrete teaching of how to put into practice the embodiment in which forming is accomplished before calcination. Indeed, when a more detailed process including drying is described, there is no information about when a forming step should be
accomplished. Moreover, forming is described only for molecular sieves in powder form (which are understood to be already calcined products) and several forming processes are listed which may or may not require the use of a binder. Finally, no forming is present in any of the examples.

3.5 Under such circumstances, it is concluded that document D1 does not contain a direct and unambiguous disclosure of the sequence of process steps drying, preparation of a formable mass containing the dried product and a binder, forming that mass into a green shaped article and calcining. A different conclusion could be reached only as a result of an ex-post facto analysis, namely by means of a reading of the document in the light of the invention in the patent in suit.

3.6 Therefore, novelty of the process of granted claim 1 with respect to the disclosure of D1 can be acknowledged on the basis of that difference alone.

3.7 In addition, it is noted that the generic disclosure of binders in D1 (see point 3.3, above) cannot be interpreted as a disclosure either explicit of implicit of binders selected from compounds of silicon, aluminium, boron, phosphorus, zirconium and/or titanium. The fact that binders belonging to that list are mentioned in documents, which are either not cited in D1 (such as D8) or cited in a different context (such as D2, cited on page 2, line 28 of D1), and that their use may form part of the common general knowledge in the field is of no relevance in the analysis of novelty, so that the indication of specific binders constitutes
3.8 Finally, D1 does not mention the use of any paste-forming agent. Here again, the fact that such a use may belong to the common general knowledge in the field does not affect the analysis of novelty, since in the absence of any sort of information that feature cannot be considered as disclosed in D1 either explicitly or implicitly.

3.9 Similar considerations apply to the novelty of the process of granted claim 8 with respect to the disclosure in D1. In view of the fact that D1 does not contain any concrete teaching of how to put into practice the embodiment in which forming is accomplished before calcination (points 3.1 to 3.4, above), it does not contain a direct and unambiguous disclosure of the sequence of process steps concentration, preparation of a formable mass containing the concentrated product and a binder, forming that mass into a green shaped article and calcining.

3.10 In that respect it is noted that, contrary to the allegation of the respondent, the Board is not able to distinguish a generic concentration step from the specific steps of filtration, centrifugation and decantation disclosed in D1 (point 3.2, above), whose result is undoubtedly a product in which the concentration of the solid crystals is higher than in the starting suspension. The analysis whether the product of such steps could still be considered as a suspension is of no relevance, since claim 8 does not
specify what the result of the concentration step should be, so that filtration, centrifugation and decantation fall under the generic concentration step of claim 8. In any case, the fact that the Board considers the step of concentration as disclosed in D1 does not have any bearing on the analysis of novelty, since the specific sequence of process steps of claim 8 is not disclosed in D1 (see point 3.9, above).

3.11 In addition, the process of claim 8 is novel with respect to the disclosure of D1 in view of the indication of specific binders (see point 3.7, above).

3.12 For these reasons, both the process of granted claim 1 and the one of granted claim 8 are novel with respect to the disclosure in D1.

4. Inventive step

4.1 The critical issue on which the appellant contests the appealed decision on the analysis of inventive step concerns the choice of the closest prior art, based on the fact that D1 and not D3 has to be chosen, since that document discloses the crucial feature of the process of granted claim 1, namely that forming takes place between drying and calcination.

4.2 The Board, however, in the analysis of novelty with respect to claim 1 has come to the conclusion that D1 does not disclose the critical sequence of process steps, namely drying, preparation of a formable mass containing the dried product and a binder, forming that mass into a green shaped article and calcining. The whole argumentation of the appellant, which is based on
the false premise that the sequence of process steps is disclosed in D1 and the difference lies only in the choice of a specific binder and in the use of a paste-forming agent, can therefore not be followed by the Board.

4.3 The Board agrees completely with the approach on the analysis of inventive step which was followed in the appealed decision and in the arguments of the respondent during appeal proceedings, namely that D3 is the closest state of the art, that the process of granted claim 1 differs from the one disclosed in D3 in that forming takes place before calcination, that the problem to be solved is to provide a more efficient process by means of which products with improved mechanical properties are obtained, that the problem has been effectively solved by the claimed process in view of the examples and comparative examples in the patent and that neither D1, nor D5 provide a hint to the claimed solution as a solution to the posed problem, so that inventive steps needs to be acknowledged. Since the appellant has not contested that the skilled person, starting from D3, would come to that conclusion, it is not the Board's duty to investigate further on this issue.

4.4 Even starting from document D1, which is a more remote starting point than D3 in view of the analysis of the differences with respect to D1 which has been accomplished above (see points 3.1 to 3.8), the same conclusion is obtained.

4.4.1 In view of the absence of any concrete teaching in D1 of how to put into practice the (formally mentioned)
embodiment in which forming is accomplished before calcination, the embodiment of D1 which comes closer to the process of granted claim 1 whose result is a titanium silicalite shaped article is the one in which forming with a binder is accomplished on (calcined) molecular sieve powder (point 3.3, above).

4.4.2 With respect to that embodiment, the problem to be solved is still to be seen (as when starting from D3) that of providing a more efficient process by means of which products with improved mechanical properties are obtained.

4.4.3 As far as the improvement of mechanical properties is concerned, the examples and comparative examples in the patent can be accepted as a convincing evidence that such a result is obtained by means of the claimed process. In that respect it is noted that, contrary to the submissions of the appellant, each of examples 1, 2 and 3 in the patent in suit has to be compared with the corresponding comparative example, which differs only in the sequence of process steps (comparative examples 1, 2 and 3 respectively), and that in all cases it is shown that by means of the claimed process an improvement in the lateral breaking strength is obtained. While it is true that the comparative examples do not reproduce examples of D1, it must be noted that none of the examples of D1 includes a forming step and allows to obtain a shaped product, so that a closer reproduction of D1 could not be accomplished by the respondent and what is available has to be taken as a sufficient evidence that the posed problem has been effectively solved.
4.4.4 The skilled person would not find any hint to the claimed solution of the posed problem in the available prior art, since D3 suggests the use of a mixture of alcohol and water as paste-forming agent in order to obtain catalysts with satisfactory mechanical stability (page 3, line 29 to page 4, line 16) and D5 does not deal with the mechanical properties of the obtained catalyst.

4.4.5 For these reasons, the presence of an inventive step for the process of granted claim 1 is obtained also when starting from document D1.

4.5 As far as the process of claim 8 is concerned, the appellant contests the appealed decision on the same argument that D1 has to be taken as the closest state of the art instead of D3 due to the disclosure in D1 of the critical sequence of process steps. Since the Board does not agree with the presence of such a disclosure in D1 (see points 3.9 and 3.10, above), the arguments of the appellant on lack of inventive step are not found convincing for the same reasons as for granted claim 1 (points 4.1 to 4.4, above).

4.6 The only additional argument of the appellant concerning claim 8 concerns the absence in the patent in suit of any example according to the process of claim 8, based on the fact that in example 4, which is the only example in the patent where a concentration step is accomplished, the concentrated product is mixed with some spray dried product before preparation of a formable mass and extrusion (paragraph [0048] of the patent in suit). The Board does not agree with that argument, since in claim 8 the formable mass is defined
as containing the product from the concentration step, which does not exclude that other products, such as a spray dried product, is present therein. For that reason, an example of the process according to granted claim 8 is present in the patent in suit, which shows the good results in terms of lateral breaking strength of the obtained product also in that case (paragraph [0049]), so that all the arguments used for showing the presence of an inventive step for the process of granted claim 1 are equally valid for the process of granted claim 8.

5. In view of this, all the objections of the appellant against the patent in suit in the granted version fail.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar

The Chairman

S. Fabiani

J. Riolo