

THIS OPINION WAS NOT WRITTEN FOR PUBLICATION

The opinion in support of the decision being entered today (1) was not written for publication in a law journal and (2) is not binding precedent of the Board.

Paper No. 17

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte PAUL C. CHIENG, DEBORAH J. BRAME
and ALEXANDER H. T. CHU

Appeal No. 94-2414
Application 07/911,504¹

HEARD: June 11, 1997

Before JOHN D. SMITH, PAK and THIERSTEIN, Administrative Patent Judges.

JOHN D. SMITH, Administrative Patent Judge.

DECISION ON APPEAL

This is an appeal pursuant to 35 U.S.C. § 134 from the final rejection of claims 32 and 34-46. Claims 31 and 33 stand withdrawn from further reconsideration as directed to a non-elected invention.

¹ Application for patent filed July 10, 1992. According to appellants, this application is a division of Application 07/342,547 filed April 24, 1989, now U.S. Patent No. 5,158,758 issued October 27, 1992.

The subject matter on appeal is directed to silica gel defined in part by its method of preparation. Representative claims 32, 34, 35 and 38 are reproduced below. To understand the subject matter defined by product-by-process claims 32 and 34, non-elected method claims 31 and 33 are also reproduced.

31. A method for producing silica gel suitable for use in high pressure liquid chromatography comprising:

(a) mixing a solution of silicon tetrafluoride and a solvent selected from the group consisting of C₁-C₃ aliphatic alcohols with a quantity of water sufficient to form a reactant mixture having a mass ratio of water to silicon tetrafluoride of between about 4:1 and 10:1 and to hydrolyze the silicon tetrafluoride to silica;

(b) permitting the silica to gel;

(c) separately recovering the silica gel from the remainder of the reactant mixture;

(d) washing said silica gel in water until the pH of the gel is at least above about 3;

(e) drying said washed gel to obtain dry gel having essentially one form of silanol moiety; and

(f) reacting said dried gel with a quantity of organochlorosilane (sic, to bond the organochlorosilane) to said dry gel.

32. Silica gel produced by the method of claim 31.

33. A method for producing silica gel suitable for use in high pressure liquid chromatography comprising:

(a) mixing a solution of silicon tetrachloride and a water-soluble, non-aqueous solvent with at least a stoichiometric

quantity of water containing fluoride ion to form a reactant mixture and to hydrolyze the silicon tetrachloride to silica;

(b) permitting the silica to gel;

(c) aging the gel for about 4 hours;

(d) separately recovering the silica gel from the remainder of the reactant mixture;

(e) washing said silica gel until the pH of the gel is at least above about 3;

(f) drying said washed gel to obtain dry gel having essentially one form of silanol moiety; and

(g) reacting said dried gel with a quantity of organochlorosilane to bond the organochlorosilane to said dry gel.

34. Silica gel produced by the method of claim 33.

35. A porous silica gel having a specific surface area between about 200 and 600 m²/g, average pore diameters in the range of about 50 to about 200 Angstroms and a narrow pore diameter distribution wherein said distribution has a standard deviation in pore diameter less than about 25 percent of the average pore diameter,

wherein said silica gel is produced by the method comprising:

(a) mixing a solution of silicon tetrafluoride and a water-soluble, non-aqueous solvent with at least a stoichiometric quantity of water to form a reactant mixture and to hydrolyze the silicon tetrafluoride to silica;

(b) permitting the silica to gel;

(c) separately recovering the silica gel from the remainder of the reactant mixture and;

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(d) washing said silica gel until the pH of the gel is at least above about 3.

38. A silica gel having a specific surface area of between about 200 and 600 m²/g, average pore diameters in the range of about 50 to about 200 Angstroms and a narrow pore diameter distribution wherein said distribution has a standard deviation in pore diameter less than about 25 percent of the average pore diameter,

wherein said silica gel is produced by a method comprising:

(a) mixing a solution of silicon tetrachloride and a water-soluble, non-aqueous solvent with at least a stoichiometric quantity of water containing fluoride ion to form a reactant mixture and to hydrolyze the silicon tetrachloride to silica;

(b) permitting the silica to gel;

(c) aging the gel for about 4 hours;

(d) separately recovering the silica gel from the remainder of the reactant mixture; and

(e) washing said silica gel until the pH of the gel is at least above about 3.

The references of record relied upon by the examiner are:

Jacques et al. (Jacques)	4,504,595	Mar. 12, 1985
Hench et al. (Hench)	4,849,378	Jul. 18, 1989
Okamura et al. (Okamura)	4,975,405	Dec. 4, 1990

Colin et al. (Colin), "Introduction to Reversed-Phase High-Performance Liquid Chromatography," Journal of Chromatography, 141 (1977), pp. 289-312.

The appealed claims stand rejected under 35 U.S.C. § 103 over Jacques or Okamura in view of Colin and Hench.

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THE REJECTION OF PRODUCT-BY-PROCESS CLAIMS 32 AND 34

Appellants characterize the present invention as directed to a silica gel made according to a "novel method" which is presented in U.S. Patent No. 5,158,758. No method claim in that patent, however, is directed to the specific method which now defines the product of claims 32 and 34 in this appeal. As evident from respective steps (f) and (g) of method claims 31 and 33, the silica gel defined by product-by-process claims 32 and 34 is, in actuality, a silica gel bonded by a quantity of organochlorosilane. This bonded form of silica gel is said to be especially useful for reverse phase liquid chromatography (RPLC). See the specification at page 13, lines 9-11.

Appellants submit (brief, page 9) that the silica gel defined by claims 32 and 34 is distinguishable from Jacques, Hench and Colin "singly or in combination" because the claims require a "dry gel having essentially one form of silanol moiety". This language appears in the respective processes as step (e) of claim 31 and step (f) of claim 33 as part of a claimed drying step, i.e. "drying said washed gel to obtain dry gel having essentially one form of silanol moiety". It is noted, however, that the dried gel is then subjected to reaction with a

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quantity of organochlorosilane to bond organochlorosilane to the dry gel thus forming a bonded silica gel structure. Thus, it is questionable whether the claim language "having essentially one form of silanol moiety" which describes an intermediate structure of an unreacted dry gel constitutes a structural limitation of the claimed gel, which is a bonded gel. In this regard, see Colin at page 298 which describes the structure of silica gel after bonding with a monochlorosilane.

Even assuming that an unreacted dry gel structure "having essentially one form of silanol moiety" survives the bonding step when the silica gel is reacted and bonded with monochlorosilane, it is not apparent that the claimed bonded silica gel product is distinguished from prior art bonded silica gel products as described or suggested by Colin. In this regard, in describing prior art techniques appellants explain in their specification at page 13, lines 1-4 that

[D]ilute hydrofluoric acid wash typically is used to condition silica before bonding stationary phases thereto. Such conditioning improves separation efficiency and sample recovery because the surface silanols are fully hydroxylated, i.e., form individual silanol groups (emphasis added).

Moreover, Colin teaches at page 297 that maximum coverage density of the bonded phase is obtained when a maximum number of "free silanols" are available on the surface of the silica gel.

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Although Colin teaches that it is generally accepted that the surface of a silica gel particle is covered with a monolayer of silanol groups with an irregular distribution, Colin clearly teaches that it is necessary to activate or dehydrate the silica before carrying out monochlorosilane bonding. Compare, for example, the disclosure of Colin at page 297, first full paragraph, and Colin's teaching that a "completely hydroxylated silica" has been obtained by prior art workers by activating or dehydrating silica gel on heating at 250E under a vacuum. Further, we observe appellants' acknowledgment (specification, page 11, line 34 to page 12, line 28) that appellants' drying step is effected by method known in the art. There is therefore no reasonable basis to argue, as appellants have, that appellants' dry gel having essentially one form of silanol moiety differs at all from prior art "completely hydroxylated" silica gels which have been bonded by reaction with an organochlorosilane.

Where, as here, the prior art organochlorosilane bonded silica gel appears to be either identical or slightly different from the organochlorosilane bonded silica gel defined by product-by-process claims 32 and 34, the burden is on appellant to establish that the prior art gels do not necessarily or

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inherently possess the characteristics of the claimed product. See, for example, In re Marosi, 710 F.2d 799, 803, 218 USPQ 289, 292-93 (Fed. Cir. 1983). Here, appellants have failed to provide any evidence demonstrating that the unreacted prior art dry gels disclosed by Colin do not possess "essentially one form of silanol moiety" thereon, much less evidence demonstrating any difference between the prior art bonded silica gels and the claimed bonded silica gels.

In light of the foregoing, we affirm the rejection of product-by-process claims 32 and 34.

THE PRIOR ART REJECTIONS OF CLAIMS 35-46

Claims 35-46, although drafted as product-by-process claims, stand on a different footing than claims 32 and 34. These claims define a silica gel having a specific surface area between about 200 and 600 m²/g, average pore diameters in the range of about 50 to about 200 Angstroms, and importantly, a narrow pore diameter distribution wherein said distribution has a standard deviation and pore diameter less than about 25 percent of the average pore diameter.

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We have carefully reviewed each of the relied upon prior art references, particularly Hench, that form the basis of the examiner's rejections.

As persuasively argued by appellants, the narrow pore diameter distribution is an integral limitation of these claims, and none of the relied upon references, including Hench, teach one of ordinary skill in the art how to make a silica gel having a pore diameter distribution having a standard deviation and pore diameter less than about 25 percent of the average pore diameter as claimed. Particularly, see the brief at page 5.

Essentially, for the reasons set forth in appellants' briefs, we agree that the examiner has failed to establish a prima facie case of obviousness for the subject matter defined by appealed claims 35-46. We, therefore, reverse the examiner's rejections of these claims.

In summary, the rejection of claims 32 and 34 is affirmed. The rejections of claims 35-46 is reversed. Accordingly, the decision of the examiner is affirmed-in-part.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 CFR § 1.136(a).

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AFFIRMED-IN-PART

JOHN D. SMITH)	
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Administrative Patent Judge))	APPEALS AND
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