(19) World Intellectual Property Organization International Bureau





(43) International Publication Date 9 June 2005 (09.06.2005) (10) International Publication Number WO 2005/051839 A3

- (51) International Patent Classification⁷: C04B 35/14, 35/16, B01J 21/08, 21/12, 21/14, C01B 33/32, 33/12, 6/24, 3/02, 3/08, C07C 5/00, C10G 35/00, 35/04, 19/00, 45/00, 17/00
- (21) International Application Number:

PCT/US2004/039304

(22) International Filing Date:

24 November 2004 (24.11.2004)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

60/524,038	24 November 2003 (24.11.2003)	US
60/561,886	14 April 2004 (14.04.2004)	US
60/578,818	14 June 2004 (14.06.2004)	US
60/611,700	22 September 2004 (22.09.2004)	US
60/611,701	22 September 2004 (22.09.2004)	US

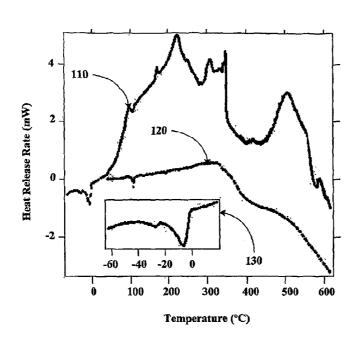
- (71) Applicant and
- (72) Inventor: LEFENFELD, Michael [US/US]; 629 West 115 Street, Apt. 3A, New York, NY 10025 (US).
- (72) Inventor; and
- (75) Inventor/Applicant (for US only): DYE, James, L.

[US/US]; 2698 Roseland Avenue, East Lansing, MI 48823 (US)

- (74) Agent: LINDEMAN, Jeffrey, A.; Nixon Peabody LLP, 401 9th Street, N.W., Washington, DC 20004 (US).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

[Continued on next page]

(54) Title: SILICA GEL COMPOSITIONS CONTAINING ALKALI METALS AND ALKALI METAL ALLOYS



(57) Abstract: The invention relates to Group 1 metal/silica gel compositions comprising silica gel and an alkali metal or an alkali metal alloy. The compositions of the inventions are described as Stage 0, I, II, and III materials. These materials differ in their preparation and chemical reactivity. Each successive stage may be prepared directly using the methods described below or from an earlier stage material. Stage 0 materials may, for example, be prepared using liquid alloys of Na and K which are rapidly absorbed by silica gel (porous Si02) under isothermal conditions, preferably at or just above room temperature, to form loose black powders that retain much of the reducing ability of the parent metals. When the low melting Group 1 metals are absorbed into the silica gel, a mild exothermic reaction produces Stage I material, loose black powders that are indefinitely stable in dry air. Subsequent heating to 400°C produces Stage II materials, which are also loose black powders. Further heating above 400°C forms Stage III material with release

of some Group 1 metal. It is believed that Stage I, II and III materials represent reductions of the silica gel after absorption of the Group 1 metal. Preferred Group 1 metal/silica gel compositions of the invention are those containing sodium, potassium, or sodium-potassium alloys with sodium and sodium- potassium alloys being most preferred. Each stage of the Group 1 metal/silica gel composition of the invention may be used as a reducing agent reacting with a number of reducible organic materials in the same manner known for alkali metals and their alloys.

WO 2005/051839 A3



Published:

- with international search report
- (88) Date of publication of the international search report: $$27\ \mathrm{April}\ 2006$

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US04/39304

A. CLASSIFICATION OF SUBJECT MATTER IPC(7) : C04B 35/14, 35/16; B01J 21/08, 21/12, 21/14; C01B 33/32, 33/12, 6/24, 3/02, 3/08; C07C 5/00; C10G 35/00, 35/04, 19/00, 45/00, 17/00. US CL : 501/133; 502/233, 237, 243, 344; 423/332, 333, 334, 335, 338, 644, 648.1, 657; 585/267; 208/133, 134						
US CL	SEARCHED	, 224, 222,	336, 611, 616.2, 657, 562, 267	,		
		1 • • • •				
Minimum do U.S. : 50	cumentation searched (classification system followed l/1/133; 502/233, 237, 243, 344; 423/332, 333, 334, 33	5, 338, 644	, 648.1, 657; 585/267; 208/133	3, 134, 226, 230, 262.1.		
Documentation	on searched other than minimum documentation to the	extent that	such documents are included	in the fields searched		
Electronic da	ta base consulted during the international search (nam	e of data ba	ase and, where practicable, sea	rch terms used)		
C. DOC	UMENTS CONSIDERED TO BE RELEVANT					
Category *	Citation of document, with indication, where a	ppropriate,	of the relevant passages	Relevant to claim No.		
X Y	US 5,856,262 A (FLICK et al) 05 January 1999 (05.	01.1999), s	ee entire document.	1, 4-7, 10-13, 16-18, 20, 23-25, 27-29, & 31- 32		
				19, 26, 30, & 33-53		
X 	US 6,022,823 A (AUGUSTINE et al) 08 February 20	000 (08.02.	2000), see entire document.	1, 4-7, 10-13, 16-18, 20, 23-25, & 27-33		
Y				19, 26, & 34-53		
A	US 2,731,326 A (ALEXANDER et al) 17 January 19	956 (17.01.)	1956), see entire document.	1-53		
Α	A US 2,765,242 A (ALEXANDER et al) 02 October 1956 (02.10.1956), see entire document. 1-53			1-53		
A	US 3,489,516 A (KUMMERLE) 13 January 1970 (13.01.1970), see entire document.			1-53		
A	US 4,975,405 A (OKAMURA et al) 04 December 19	990 (04.12.	1990), see entire document.	1-53		
				<u> </u>		
Further	documents are listed in the continuation of Box C.		See patent family annex.			
* Special categories of cited documents: "T" later document published after the international filing date or priorit and not in conflict with the application but cited to understand the			out cited to understand the			
	"A" document defining the general state of the art which is not considered to be of principle or theory underlying the invention particular relevance					
"E" earlier application or patent published on or after the international filing date "X" document of particular relevance; the claimed inverse considered novel or cannot be considered to involve when the document is taken alone						
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)		"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious				
"O" document	referring to an oral disclosure, use, exhibition or other means		to a person skilled in the art	_		
"P" document published prior to the international filing date but later than the "&" document me priority date claimed		document member of the same patent fa	amily			
	Date of the actual completion of the international search 2 November 2005 (22.11.2005) Date of mailing of the international search report 0 6 JAN 2006					
22 November 2005 (22.11.2005) Name and mailing address of the ISA/US Authorized officer						
	1 Stop PCT, Attn: ISA/US					
Commissioner for Patents		Cam N. Nguyen DEBORAH A. THOMAS				
	Box 1450	Telephone No. 571-272-1700				
	Alexandria, Virginia 22313-1450 Telephone No. 571-272-1700 Facsimile No. (571) 273-3201					

INTERNATIONAL SEARCH REPORT

International application No. PCT/US04/39304

ategory *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	
A	US 3,527,563 A (SHANKLIN) 08 September 1970 (08.09.1970), see entire document.	1-53	
A	US 3,079,234 A (JENKINS et al) 26 February 1963 (26.02.1963), see entire document.	1-53	
A	US 3,165,379 A (SCHAWARTZ) 12 January 1965 (12.01.1965), see entire document.	1-53	
Y	US 3,493,341 A (PAGE et al) 03 February 1970 (03.02.1970), see entire document.	2-3, 8-9, 14-15, & 2 22	
	,		
	,		
		-	