

# (12) United States Patent

#### Cammarano et al.

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## (54) METHODS FOR THE PREPARATION OF BIOLOGICALLY ACTIVE COMPOUNDS IN NANOPARTICULATE FORM

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	A61K 31/192	(2006.01)
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	A61K 9/14	(2006.01)

(52) U.S. Cl.

CPC ...... A61K 31/192 (2013.01); A61K 31/496 (2013.01); **A61K 31/4535** (2013.01); **A61K** 31/551 (2013.01); A61K 9/1617 (2013.01); A61K 9/143 (2013.01); A61K 31/196 (2013.01) USPC .......... 424/493; 514/420; 514/567; 514/569;

## (58) Field of Classification Search

See application file for complete search history.

#### (56)References Cited

#### U.S. PATENT DOCUMENTS

5,145,684 A * 5,202,129 A		Liversidge et al 424/489 Samejima et al.
5,298,262 A *	3/1994	Na et al 424/489
2002/0047058 A1*	4/2002	Verhoff et al 241/26
2003/0137067 A1*	7/2003	Cooper et al 264/5
2003/0228357 A1*	12/2003	Johnson et al 424/465
2004/0173696 A1*	9/2004	Cunningham et al 241/21
2007/0059356 A1*	3/2007	Almarsson et al 424/464

#### FOREIGN PATENT DOCUMENTS

0600528 6/1994 WO2007/070851 WO 6/2007

#### OTHER PUBLICATIONS

Tsuzuki, T.; Pethick, K.; McCormick, P. Synthesis of CaCO3 nanoparticles by mechanochemical processing. Journal of Nanoparticle Research, vol. 2, p. 375-380, 2000.\*

Tsuzuki, T.; Pirault, E.; McCormick, P. Mechanochemical Synthesis of Gadolinium Oxide Nanoparticles. Nanostructured Materials, vol. 11, No. 1, p. 125-131, 1999.\*

Tsuzuki, T.; McCormick, P. Mechanochemical synthesis of nanoparticles. Journal of Materials Science, vol. 39, p. 5143-5146, 2004.\*

Grigorieva, T. F.; Barinova, A. P.; Lyakhov, N. Z. Mechanosynthesis of nanocomposites. Journal of Nanoparticle Research, vol. 5, p. 439-

Tsuzuki, T.; McCormick, P. Mechanochemical Synthesis of Metal Sulphide Nanoparticles. Nanostructured Materials, vol. 12, p. 75-78,

Office Action in corresponding Canadian Application 2,653,384, dated Mar. 10, 2014, pp. 1-3.

Juhnke, M. et al., "Nanoparticles of soft materials by high-energy milling at low temperatures," 7th world congress of chemical engineering, Glasgow:pp. 1-10 (2005).

#### \* cited by examiner

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## **ABSTRACT**

A method for producing a composition comprising nanoparticles of a biologically active compound, comprising the step of: dry milling a solid biologically active compound and a millable grinding compound in a mill comprising a plurality of milling bodies, for a time period sufficient to produce a solid dispersion comprising nanoparticles of the biologically active compound dispersed in an at least partially milled grinding compound is described as are various compositions produced using such methods.

#### 14 Claims, 26 Drawing Sheets

**LUPIN EX. 1020** Lunin v iCeutica



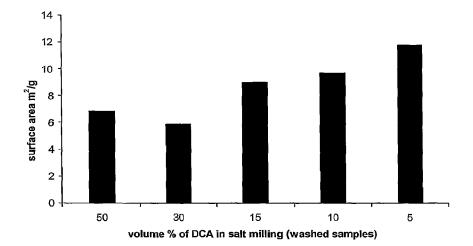
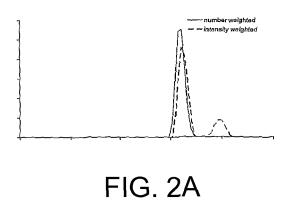


FIG. 1



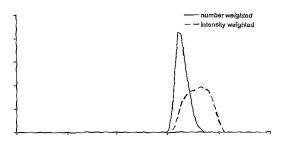
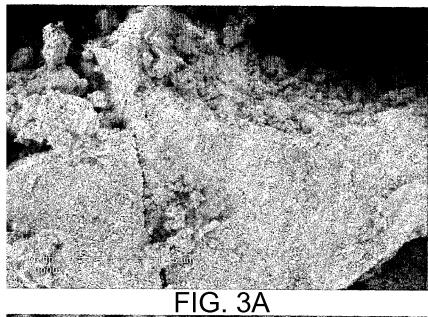


FIG. 2B



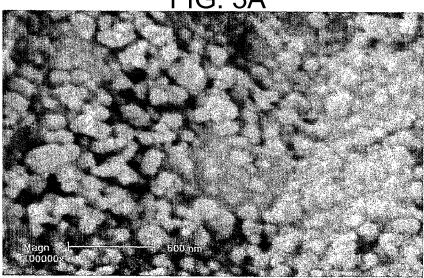


FIG. 3B

Vol %	SEM post washing	TEM post washing
5		
10		0.5 til.
15		<u>Maun</u>
30		Tipu
50	Magn. 1 um. 1 um.	

FIG. 4



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