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(54) **METHOD FOR ELECTROCHEMICAL PRODUCTION OF A CRYSTALLINE POROUS METAL ORGANIC SKELETON MATERIAL**

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C07F 15/00 (2006.01)

(52) **U.S. Cl.** **556/118**; 556/136

(58) **Field of Classification Search** 556/118, 556/136

See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,104,140	A	8/1978	Eisenbach et al.	
4,599,150	A	7/1986	Mullin et al.	
5,648,508	A *	7/1997	Yaghi	556/9
6,617,467	B1	9/2003	Mueller et al.	
6,624,318	B1	9/2003	Mueller et al.	
6,893,564	B2	5/2005	Mueller et al.	
6,929,679	B2	8/2005	Mueller et al.	
7,008,607	B2	3/2006	Mueller et al.	
2003/0078311	A1	4/2003	Muller et al.	
2003/0148165	A1	8/2003	Muller et al.	

(Continued)

FOREIGN PATENT DOCUMENTS

DE 202 10 139 6/2003

(Continued)

OTHER PUBLICATIONS

Eddaoudi, Mohamed et al., "Systematic Design of Pore Size and Functionality in Isoreticular MOFs and Their Application in Methane Storage", Science, vol. 295, pp. 469-472, 2002.

Chen, Banglin et al., "Interwoven Metal-Organic Framework on a Periodic Minimal Surface with Extra-Large Pores", Science, Vo. 291, pp. 1021-1023, 2001.

(Continued)

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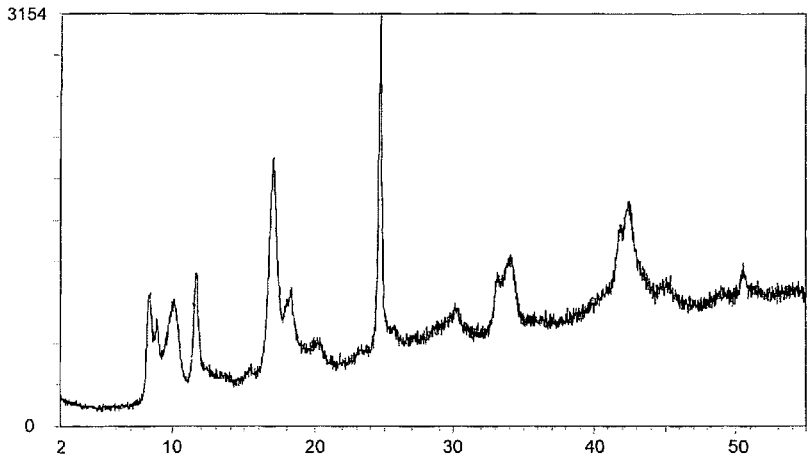
(57) **ABSTRACT**

A method of electrochemically preparing a crystalline, porous, metal-organic framework material comprising at least one at least bidentate organic compound coordinately bound to at least one metal ion, in a reaction medium comprising the at least one bidentate organic compound, wherein at least one metal ion is provided in the reaction medium by the oxidation of one anode comprising the corresponding metal.

14 Claims, 5 Drawing Sheets