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(54) **HIGH-PRESSURE, HIGH-TEMPERATURE MAGIC ANGLE SPINNING NUCLEAR MAGNETIC RESONANCE DEVICES AND PROCESSES FOR MAKING AND USING SAME**

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(58) **Field of Classification Search**

CPC G01R 33/30; G01R 33/302; G01R 33/305

See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

3,585,301	A *	6/1971	Newman	175/300
5,325,059	A	6/1994	Doty	
5,333,994	A	8/1994	Doty et al.	
6,054,857	A *	4/2000	Doty	324/321
2012/0146636	A1	6/2012	Hoyt et al.	

OTHER PUBLICATIONS

Hoyt, D. W., et al., High-pressure magic angle spinning nuclear magnetic resonance, Journal of Magnetic Resonance, 212, 2011, 378-385.

(Continued)

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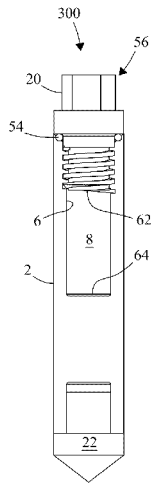
(57) **ABSTRACT**

Re-usable ceramic magic angle spinning (MAS) NMR rotors constructed of high-mechanic strength ceramics are detailed that include a sample compartment that maintains high pressures up to at least about 200 atmospheres (atm) and high temperatures up to about least about 300° C. during operation. The rotor designs minimize pressure losses stemming from penetration over an extended period of time. The present invention makes possible a variety of in-situ high pressure, high temperature MAS NMR experiments not previously achieved in the prior art.

7 Claims, 16 Drawing Sheets