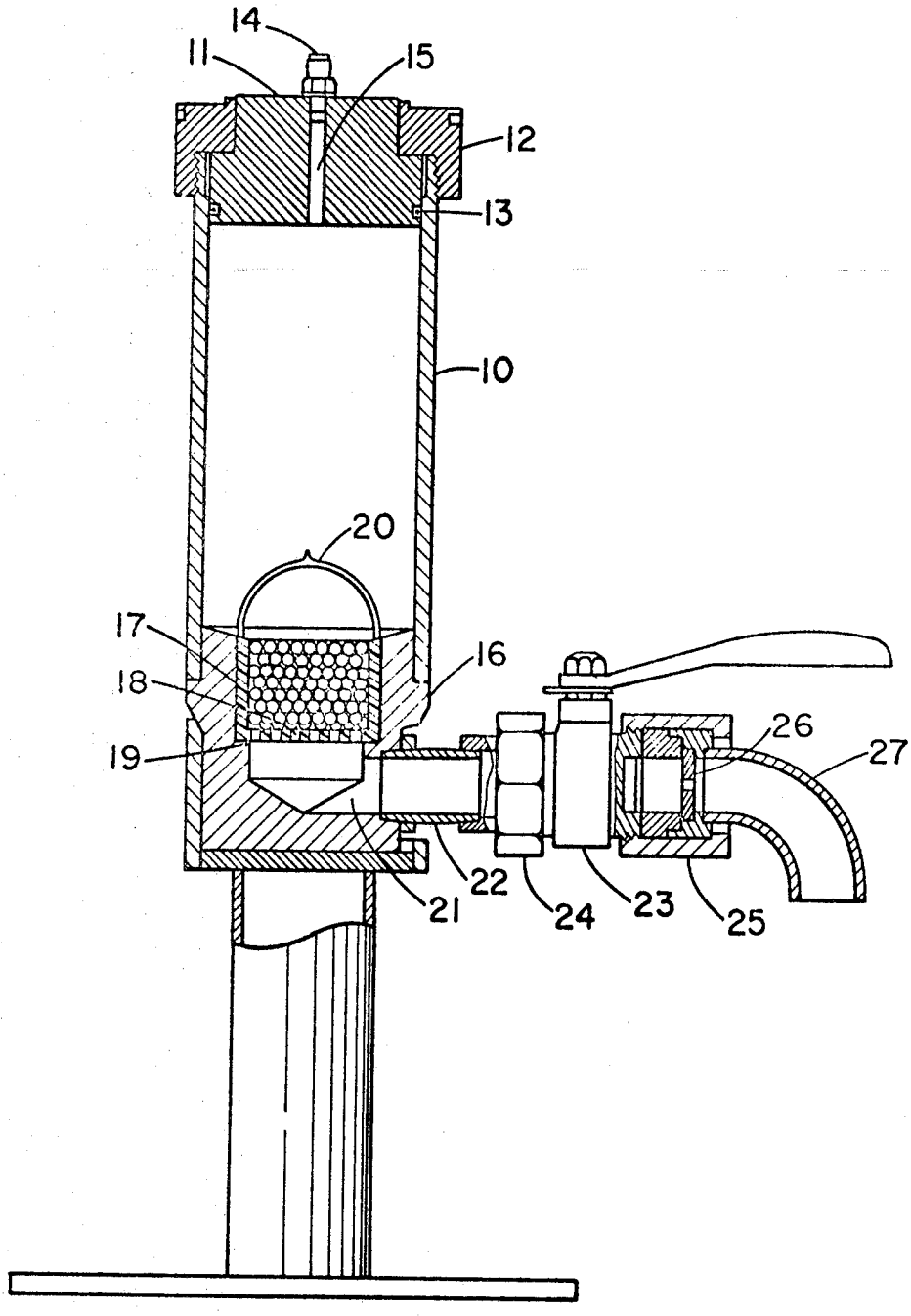
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STOPPING LOST CIRCULATION IN WELL DRILLING

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STOPPING LOST CIRCULATION IN WELL DRILLING

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ABSTRACT OF THE DISCLOSURE

A mixture of asbestos fibers and granular particles within critical limits is used to stop loss of drilling fluid to drilled formations.

In a recent industry-wide study of lost circulation agents, it was found that lost circulation materials which satisfy some tests do not satisfy others. None of the materials satisfied all the tests. Tentative standard tests have now been proposed to permit uniform testing of additives to determine which types of lost circulation a given material will stop and how effective it is for that purpose.

Two types of tests seem to simulate most nearly conditions in the field. These may be called the brass marbles test and the slot test. These will be described in detail below. It is, of course, desirable to find an additive suitable for sealing both the marbles and the slots. In general, fibers and flakes seem most effective for sealing marbles, and granular hard plant seed particles seem most effective for sealing slots. If granular particles are mixed with fibers, flakes or both, both types of particles generally lose some of their effectiveness so they are not very satisfactory for sealing either marbles or slots. By observing several critical limitations, however, a mix can be prepared which is rather surprisingly effective for sealing both marbles and slots. This material is described in U.S. Pat. 3,375,888, issued Apr. 2, 1968, to James L. Lummus and Billy V. Randall. The additive has received widespread commercial acceptance.

The commercial additive contains a rather low concentration of granular nutshells. A mixture containing a larger percentage of the low-cost granular nutshells but still suitable for sealing both marbles and slots would obviously be desirable. Such a composition would also, of course, be desirable since it would retain more of the effectiveness of the nutshells for sealing slots. A mixture not requiring so many ingredients would also be desirable.

An object of this invention is to provide a drilling fluid additive which will seal both marbles and slots in lost circulation tests. A more specific object is to provide a lost circulation mixture containing a high concentration of ground granular nutshells but still able to seal marbles in a lost circulation test. Still other objects will be apparent from the following description and claims.

We have found that when small amounts of certain grades of long, flexible asbestos fibers are mixed with ground nutshells, the mixture seals marbles in lost circulation tests. The very high concentration of ground nutshells insures excellent slot-sealing ability, of course, if the nutshell particle size and particle-size distribution are within certain limits. There are several other limitations

which will become apparent from the test results presented below.

The drawing is a view, partly in cross-section, of the appartus used in the tests.

In the drawing, a container **10** has a top plug **11** held in place by a cap ring **12**. A seal **13** is provided between the container and top plug. A fitting **14** permits connecting a source of high pressure gas, such as a cylinder of nitrogen to passage **15** in the top plug. The passage permits applying high gas pressure to the top of liquid in container **10**. A bottom section is permanently attached to container **10**. This bottom section has a bore of smaller diameter than that of container **10**. A transparent plastic sleeve **17** with a metallic perforated bottom plate **18** fits into the bore of bottom portion **16**. The perforated plate rests on a shoulder **19** which supports the plate. The plate and sleeve together form a basket for about 95 brass marbles about 9/16-inch in diameter. A bail **20** is provided for placing the basket of marbles in the container and removing the basket from the container. A horizontal passage **21** connects the space below plate **18** to a pipe section **22**. To the outer end of pipe **22** valve **23** is connected through union **24**. The valve is a full-opening valve with a passage as large as the internal cross-section of pipe **22**. Beyond valve **23** is an assembly **25** which can hold a slotted plate **26**. Assembly **25** also holds a curved conduit or spout **27** which carries liquids passing through the apparatus to a graduated container (not shown).

When a test using marbles was run, slotted plate **26** was removed. When a slotted plate test was run, the basket of marbles was removed. Otherwise, the test procedure was the same. With the top of the container off, mud containing the additive to be tested was poured into the top of the container. The amount which ran out of spout **27** before a seal was formed was then recorded. Next, the top was put on the container and gas pressure was applied in the top of the containers. The pressure was slowly increased to 100 pounds per square inch over a period of one minute, and the amount of mud which ran out of the spout was recorded. Then, the pressure in the top of the container was slowly increased over a period of about two minutes to 1,000 pounds per square inch. The volume of mud through the apparatus at this pressure was noted. A pressure of 1,000 pounds per square inch was allowed to remain on the mud for 10 minutes. The volume of mud through at the end of this time was recorded.

It will be noted that in this test volumes of liquid through the apparatus at several pressure and times are available. These values permit comparison of various materials. However, the commercially available mix of granules, flakes and fibers, previously referred to, sealed both a large slot and marbles at 1,000 pounds per square inch. Therefore, in order to be competitive with this commercial mix, it seemed that any additive to be considered satisfactory should do as well. For this reason, a test was considered a falure if it did not hold 1,000 pounds per square inch for 10 minutes. For a seal not to be maintained for 10 minutes, the seal had to be incomplete permitting liquid to continue leaking through the partial seal until the high pressure gas began blowing through.

The drilling fluid used in all the tests was prepared by mixing about 7 percent Wyoming bentonite in fresh water,

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allowing the mud to age at least 72 hours and then adjusting the viscosity to an apparent viscosity of 25 plus or minus 2 centipoises using a Fann viscosimeter. That is, the mud was diluted until the Fann 600 r.p.m. reading was about 50.

An explanation of the asbestos classification is necessary. In the first edition of the Encyclopedia of Chemical Technology by Kirk and Othmer, Interscience Publishers, vol. 2, page 138, a classification of asbestos fibers is given on the basis of screen analysis to determine fiber length. There has been some disagreement regarding this classification and it is not repeated in the second edition of the Encyclopedia. In order to avoid any confusion regarding the materials tested, they are listed in Table I by sample letters. The manufacturer's classification is also given in parentheses, although this classification may differ from that of other manufacturers and may even change from time to time for any given manufacturer.

TABLE I

Type	Screen analysis 2M	4M	10M	PAN
A (3F)	10.5	3.9	1.3	0.3
B (3K)	7.0	7.0	1.5	0.5
C (3R)	4.0	7.0	4.0	1.0
D (3Z)	1.0	9.0	4.0	2.0
E (4A)	0.0	8.0	6.0	2.0
F (4K)	0.0	4.0	9.0	3.0
G (4T)	0.0	2.0	10.0	4.0
H (5K)	0.0	0.0	12.0	4.0
I (6D)	0.0	0.0	7.0	9.0
J (8T)	(1)			

[1] No test.

In the analysis presented above, 16 ounces of asbestos is subjected to screen analysis. The number in the 2M column indicates the number of ounces of the sample retained on a screen having two meshes to the inch. Under the 4M heading, the number of ounces through a 2-mesh screen and retained on a 4-mesh screen is given. In the column headed 10M the number of ounces through a 4-mesh and retained on a 10-mesh screen is presented. The last column, headed "PAN," gives the number of ounces through the 10-mesh screen.

It should be noted that the screens used for the screen analysis of asbestos are not the U.S. Standard Sieve Series used for the screen analysis of granular particles. The screens used for asbestos are those specified in "The Quebec Standard Asbestos Test." The widths of screen openings are as follows:

	Inch
2-mesh screen	0.500
4-mesh screen	0.187
10-mesh screen	0.053

Both the length of the asbestos fibers and the particle size and particle-size distribution of the granules are important. The tests are divided into two sections. In the first section, granules were used with a narrow particle-size distribution which is poor for our purposes. Nutshells of this sort are widely available in the field. The purpose of these tests was to determine what grade and amount of asbestos fibers had to be used in combination with such granules. In the second section of tests, the nutshell particle-size distribution was broad, a condition greatly preferred by us. These tests were run to determine how little asbestos could be used and of how short a fiber length if the particle-size distribution of the granules was more carefully controlled. This is important from an economic standpoint because of the great difference in costs of ground nutshells and the longer fiber grades of asbestos.

The ground nutshells used in the first group of tests had the particle-size distribution shown in Table II. The mesh sizes are U.S. Standard Sieve sizes. For example, −8+12 means the fraction which passed a No. 8 sieve and was retained on a No. 12 sieve.

Table II

Mesh size:	Percent by weight
+4	0.30
−4+8	48.20
−8+12	24.65
−12+16	19.10
−16+20	7.10
−20+30	0.45
−30	0.20
	100.00

A few tests were made using only the fraction retained on a No. 12 sieve. Additives prepared with this fraction also sealed both slots and marbles but did not seem to function quite as well as when the granules had a wider particle-size distribution. The granules should have at least about 20 percent retained on a No. 8 sieve, however, if wide fractures, crevices and the like are to be sealed.

Results of tests using these nutshells with a limited range of particle sizes are presented in Table III. The asbestos grades are those given in Table I.

TABLE III

	Asbestos Type	Asbestos Percent in mix	Lb./bbl. mixture in mud	Ml. through after 10 minutes at 1,000 p.s.i.
Test Number:				
1	A	15	15	2,450.
2	B	15	15	2,100.
3	C	20	15	2,125.
4	D	33	15	Failed.
5	A	5	30	Do.
6	B	5	30	Do.
7	B	7	30	Do.
8	B	3	30	Do.
	G	7		
9	B	5	30	1,600.
	G	5		
10	C	10	30	1,600.
11	A	15	30	1,120.
12	B	15	30	1,550.
13	C	15	30	1,300.
14	D	15	30	2,150.
15	C	20	30	950.
16	E	25	[1] 30	1,600.
17	F	50	30	2,500.
18	H	50	30	Failed.
19	C	6	50	Do.
20	C	10	50	1,200.

[1] With only the +12 fraction of nutshells.

A study of the results shown in Table III, together with the asbestos screen analyses in Table I, shows that the longer asbestos fibers are most important, the ones passing a 4-mesh screen being of little value, with one possible exception to be explained later. A further study of the effects of asbestos fiber length indicated the fibers retained on a 2-mesh screen are about 4 times as effective as those passing a 2-mesh and retained on a 4-mesh screen. Fibers passing a 4-mesh and retained on a 10-mesh screen probably have some effects but the effects are too slight to be given weight in setting limits on what should and

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should not be used. The following Table IV represents results of the tests in Table III, together with figures on the pounds of asbestos of the two longest fractions per barrel of drilling fluid. The table also presents a figure in which the 2- to 4-mesh fraction has been divided by 4 to provide a weight of 2-mesh fibers equivalent to the shorter length. Thus, the factor $2M$ plus $4M/4$ is the weight of fibers retained on a 2-mesh sieve plus one-fourth the weight retained on a 4-mesh screen.

TABLE IV

	Asbestos		Lb. bbl. mix/in fluid	Asbestos in drilling fluid				Ml. through after 10 minute at 1,000 p.s.i.
				Lb./bbl.				
	Type	Percent of mix		Total	2M	4M	2M+4M/4	
Test No.:								
1	A	15	15	2.25	1.47	0.55	1.61	2,450.
2	B	15	15	2.25	0.98	0.98	1.23	2,100.
3	C	20	15	3.00	0.75	1.31	1.08	2,125.
4	D	33	15	4.95	0.31	2.78	1.00	Failed.
5	A	5	30	1.5	0.98	0.37	1.07	Do.
6	B	5	30	1.5	0.66	0.66	0.83	Do.
7	B	7	30	2.1	0.92	0.92	1.15	Do.
8	B	3	30	0.9	0.39	0.39	0.55	Do.
	G	7		2.1	0.00	0.26		
9	B	5	30	1.5	0.66	0.66	0.87	1,600.
	G	5		1.5	0.00	0.19		
10	C	10	30	3.0	0.75	1.31	1.08	1,600.
11	A	15	30	4.5	2.95	1.10	3.23	1,120.
12	B	15	30	4.5	1.97	1.97	2.46	1,550.
13	C	15	30	4.5	1.12	1.97	1.61	1,300.
14	D	15	30	4.5	0.28	2.53	0.91	2,150.
15	C	20	30	6.0	1.50	2.62	2.16	950.
16	E	25	30	7.5	0.00	3.75	0.94	1,600.
17	F	50	30	15.0	0.00	3.75	0.94	2,500.
18	H	50	30	15.0	0.00	0.00	0.00	Failed.
19	C	6	50	3.0	0.75	1.31	1.08	Do.
20	C	10	50	5.0	1.25	2.19	1.79	1,200.

Table IV was prepared to determine if a critical concentration of asbestos is required in the drilling fluid to make to make it seal the marbles when the granules have only a narrow range of particle sizes. The results show that in general the asbestos concentration factor should be about 0.9 pound per barrel, this factor being determined by adding the portion retained on a 2-mesh screen to one-fourth the portion passing a 2-mesh and retained on a 4-mesh screen. A comparison of tests 3, 10 and 19 shows other factors are also involved.

The relatively poor results with an additive concentration of only about 15 pounds per barrel show this concentration is near the minimum which should be used with the narrow range of granule sizes. This is certainly one important factor. The other factor becomes apparent from a comparison of tests 10 and 19. The concentration of asbestos in both cases is exactly the same. The only difference is that in test 19, which failed, there was a higher concentration of nutshells. It is obvious from these tests, and others such as 5 and 7, that there is a critical minimum ratio of asbestos fibers to granular particles having a narrow range of sizes. In test 7, the additive, containing 7 percent asbestos, was very nearly successful in forming a seal. Therefore, the minimum concentration of asbestos in the mix with nutshells having a narrow range of particle sizes is about 8 percent by weight of the mixture. In test 9, it is interesting to note that this minimum can be met by the addition of very short fibers. This is the one possible exception to the statement that short fibers are not important. The usefulness of short fibers for this purpose is important from an economic standpoint since the shorter fibers are much less expensive than the longer ones. Preferably, the asbestos concentration should be from about 10 to about 20 percent by weight of the mixture with granular particles of limited size range. The concentration of asbestos in the mixture should not be more than about 50 percent by weight to avoid excessive dilution of the granules and thus insure a good slot-sealing ability.

The asbestos fibers and granular particles can, of course, be added separately to the drilling fluid if desired. In view of the critical minimum ratio, however, it is greatly preferred that the granules and fibers be pre-mixed to form a single dry additive mix. In accordance with the figures given above, the additive mix should contain from about 8 to about 50 percent by weight of asbestos and preferably from about 10 to about 20 percent by weight. In other terms, the ratio of asbestos to granules should be between 1 to 12.5 and about 1 to 1, and preferably from about 1 to 10 to about 1 to 5. The concentration of asbestos fibers retained on a 2-mesh screen plus one-fourth the asbestos passing a 2- and retained on a 4-mesh screen should equal at least about 3 percent by weight of the total additive when the granules have a poor particle-size distribution.

The concentration and ratio limits given above were determined using black walnut shells as the granular material. These shells had a density of 1.3 grams per cubic centimeter. If granular materials having densities widely different from this value are used, the concentration and ratio limits should be adjusted to take this change into consideration. In general, the ratio and concentration value should be multiplied by the factor $1.3/d$ where d is the density of the granular material in grams per cubic centimeter.

As noted above, a minimum concentration of about 15 pounds of the additive mix per barrel of drilling fluid should be used. When reference to a barrel is made, one containing 42 U.S. gallons is intended. Most of the work was done at an additive concentration of about 30 pounds per barrel since a concentration of other lost circulation additives near this value is often used. The reason is that mud containing higher concentrations of other lost circulation additives often is too difficult to pump. An advantage of our additive is that a much higher concentration can be used, thus providing the improved results shown by a comparison of tests 20 and 10 in Table IV. Even 50 pounds per barrel is far from the maximum concentration which can be pumped if the asbestos con-

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centration in the mix is low. The upper limit on the concentration is, of course, a concentration which forms a pumpable mixture with the drilling fluid.

In the tests reported in Tables III and IV, some rather arbitrary limits were used to determine whether a given additive mix failed or was successful. As explained above, these limits were selected principally on the basis of comparison to a commercially successful additive and not on the basis of what would produce improved results in field use. In almost all tests, for example, the additives greatly decreased the rate of flow of mud through the brass balls. As a more specific example, in test 7, the stream of liquid passing through the marbles was so small that two minutes were required for the mud to leak through so nitrogen gas blew through the apparatus. Obviously, use in the field of the additive in test 7 would provide very greatly improved results and would probably be considered a success. The point is that the limits described above should not be considered as strictly as in the usual case.

The same reasoning applies to the asbestos fibers. Obviously, if an asbestos fiber strikes a screen so that the long axis of the fiber is perpendicular to the screen, the fiber will go through the screen even if the fiber is much longer than the spacing between the wires of the screen. Therefore, the screen analysis, on which one of our limits is based, depends to some extent on the way in which the screen analysis is made. The preferred method is described in "The Quebec Standard Asbestos Test." The concentration factor of about 0.9 pound per barrel based on the screen analysis must be rather liberally interpreted in view of test 9 in Table IV, where a composition with a factor below 0.9 was quite satisfactory.

Chrysotile asbestos was used in the tests. This was partly because this was the principal type available in North America. The main reason, however, was because chrysotile asbestos is so much more flexible than other types. The asbestos fibers must bend to conform to curved surfaces in order to form a seal between these surfaces and the granular particles. Of the other types of asbestos, only amosite and crocidolite should be considered even fairly satisfactory. All other types are much too brittle. Chrysotile asbestos is certainly very greatly preferred.

With regard to slot-sealing tests, all additives tested seaeld at least a 0.16-inch wide slot when used in a concentration of about 30 pounds per barrel. In many cases, a 0.20-inch wide slot was sealed. As noted above, this was expected from the known high effectiveness of strong granular particles in sealing slots, providing a good particle-size distribution is present and the mix contains a high percentage of granules.

To this point, the tests and discussion have concerned use of asbestos with granular particles having the rather narrow distribution of sizes shown in Table II. Since such nutshells may be available and may be used with the asbestos, it seemed advisable to present the data and discusion even though such narrow ranges of particle sizes are not preferred. The reasons for preference of more widely distributed particle sizes will be apparent from the following data and discussion.

In one series of tests, ground nutshells having the particle-size distribution shown in Table II were mixed with nutshell flour having the particle-size distribution shown in Table V.

Table V

Mesh size:	Percent by weight
−100+120	3.1
−120+170	19.0
−170+200	16.3
−200+230	9.8
−230+270	8.4
−270+325	11.7
−325	31.7
	100.0

Results of test using both coarse nutshells and nutshell flour are shown in Table VI. In all these tests, the asbestos was type "B" shown in Table VI.

TABLE VI

	Percent asbestos	Percent nutshells: Coarse	Percent nutshells: Flour	Pounds per barrel mix in mud	Ml. through after 10 minutes at 1,000 p.s.i.
Test No.:					
1	7	84	9	30	1,020.
2	7	84	[1] 9	30	1,030.
3	4	86	10	30	Failed.
4	6	80	14	30	2,100.
5	6	84	6	30	1,800.
6	4	90	6	30	Failed.
7	10	82	8	30	1,500.
8	8	82	10	30	1,010.
9	8	82	10	50	1,300.
10	8	82	10	20	1,950.
11	8	82	10	10	Failed.

[1] −20+100 mesh nutshells.

Comparison of the results in Table VI with those in Table IV shows clearly the improved results obtainable by broadening the range of sizes of nutshell particles in the mixture with asbestos. Results in tests 1, 2, 4 and 5 of Table VI show that use of nutshells having a wide particle-size distribution makes possible the use of considerably lower concentrations of the relatively expensive asbestos fibers. Test 6 of Table VI shows that even with nutshell flour present, it is not possible to use less than about 5 or 6 percent by weight of asbestos in the mix with coarse ground nutshells. Tests 10 and 11 show that the inclusion of nutshell flour did not permit use of as little as 10 pounds per barrel of the mixture in drilling fluid if the marbles were to be sealed.

Table VII shows the results of slot-sealing tests using the composition of tests 8 to 11 in Table VI.

TABLE VII

	Concentrate mix lb./bbl.	Slot size, inches	Ml. through after 10 minutes at 1,000 p.s.i.
Test No.:			
1	50	.20	600.
2	30	.20	1,015.
3	20	.20	1,000.
4	20	.16	425.
5	10	.20	[1] Seal.
6	10	.16	950.
7	10	.13	850.

[1] Broke at 450 p.s.i.

These results show that as little as 20 pounds of the mixture per barrel of drilling fluid easily sealed a 0.20-inch slot. A concentration of only 10 pounds per barrel did not seal a 0.20-inch slot, but easily sealed one 0.16-inch wide.

In view of the data shown in Tables VI and VII, it seemed advisable to try ground nutshells with particles distributed fairly uniformly throughout the range from −4 down to less than 100 mesh in size. Therefore, ground black walnut shells were obtained from a commercial hammermill which used only a No. 4 sieve to remove the very coarse particles. Otherwise, the ground nutshells were unmodified. They were just as they came from the mill. The particle-size distribution of this material is shown in Table VIII.

Table VIII

Mesh size:	Percent by weight
+4	0.4
−4+5	2.1
−5+6	5.5
−6+10	35.6
−10+12	9.9
−12+16	15.1
−16+20	8.6
−20+40	10.0
−40+80	6.5
−80+100	1.2
−100	5.1
	100.0

Results of tests using these ground nutshells and various amounts and various grades of asbestos are shown in Table IX.

TABLE IX

	Asbestos		Percent nutshells	Concentrate mixture, Lb. bbl.	Ml. through after 10 min. at 1,000 p.s.i.	Filter cake
	Type	Percent				
Test No.:						
1	B	8	92	30	700	No.
2	B	6	94	30	800	No.
3	B	4	96	30	775	No.
4	B	2	98	30	1,550	No.
5	B	1	99	30	1,400	1/2" cake.
6		0	100	30	Failed at 100 p.s.i.	
7	F	6	94	30	1,050	No.
8	H	6	94	30	1,750	1/2" cake.
9	I	6	94	30	2,400	1" cake.
10		0	[1] 100	30	2,100	1/2" cake.
11	B	6	94	15	1,625	No cake.
12	F	6	94	15	2,000	3/8" cake.
13	D	6	94	15	Failed, borderline.	
14	H	6	94	15	Failed at 50 p.s.i.	
15		0	100	15	Failed explosively at 770 p.s.i.	
16	F	6	88	15	Failed explosively at 870 p.s.i.	
	J	6				
17	B	6	94	10	2,550	No.

[1] 94 percent of material with Table VIII analysis, 6 percent of material with Table V analysis.

Probably the most remarkable test of this group is test 10. By adding a little nutshell flour to the broad range of ground nutshell particle sizes, it was possible to make the nutshells alone seal the marbles. As shown in test 6, the ground nutshells without the added flour and with only about 5 percent passing a No. 100 sieve did not come even close to sealing the marbles. In test 10, two points should be noted. One is the large volume of mud which ran through before a seal was formed. This indicates the result was borderline. The other, and more important point, is the thick filter cake which formed.

A thick filter cake is obviously undesirable, since it is removed from the formation every time the bit is pulled out of the well or run back into the well. This reinitiates loss of mud to the formation. In addition, thick filter cakes can stick the drill string in the well. An advantage of the asbestos mixture with ground nutshells is that the asbestos seems to lubricate the nutshells into the formation. Once in the formation, the asbestos fibers tie the nutshell particles together to form a rigid structure which actually cements the marbles together. Little cake is deposited on the surface of the well bore. The seal is formed within the formation.

Another difficulty should be considered in connection with tests 15 and 16. When these tests failed, they did so explosively. That is, as soon as a break formed in the thick filter cake, the mud jetted through the marble bed very violently. As a matter of fact, the laboratory room was well splashed with mud. Thus, when the bit scrapes a thick filter cake off the well wall, the resulting jet of mud can damage the formation to such an extent that stopping mud loss again is very difficult. For the above reasons, the tests in Table IX, such as tests 5, 8, 9, 10 and 12, where a thick filter cake formed, are looked on with considerable suspicion and distrust. Field use of the compositions employed in these tests can hardly be recommended. Therefore, it is recommended that not less than 2 percent of asbestos should be used even when one of the long fiber grades of asbestos is employed. This is because of the thick filter cake which formed in test 5, when only 1 percent asbestos was used. Test 17 shows that when a fairly high concentration of a long-fiber grade of asbestos is used, as little as 10 pounds per barrel of the asbestos-nutshell mixture will seal marbles. It is important to note that in spite of the high volume of drilling fluid which passed through the marbles in this case before a seal was formed, there was little filter cake. Tests 7, 8 and 9 indicate there is a minimum limit on asbestos fiber length which could be observed even with a good nutshell particle-size distribution. A comparison of tests 7 and 12 indicates that asbestos which is suitable for use in a mix at high concentrations in a mud may not be satisfactory for use when the mixture with nutshells is used at low concentrations in the drilling fluid. A seal may form but the filter cake may be so thick that the seal becomes dangerous.

Considering the data in Table IX, the same 3 interrelated factors seem to be present, as in the tests producing the data in Table IV. These 3 factors are fiber length of the asbestos, concentration of asbestos in the mixture with nutshells and concentration of this mixture in the drilling fluid. When the nutshell particle-size distribution is added to this list, 4 variables are found to be interrelated in such a way that limits on each variable are dependent on the status of each other variable.

The discussion following Table IV sets limits on the amount and type of asbestos which should be used if granular particles with poor particle-size distribution are used. Referring to the data in Table IX, the same factor $2M+4M/4$ used in connection with Table IV can be determined. Both tests 7 and 17 of Table IX indicate a minimum $2M+4M/4$ value of at least about 0.1 should be observed, even when granular particles having a good particle-size distribution are employed. Test 17 of Table IX also indicates that the concentration of the mixed granules and asbestos should be at least about 10 pounds per barrel even when a good grade of asbestos and granules with a good particle-size distribution are used in a good ratio. These values permit setting minimum limits when good quality granular materials are used. Preferably, asbestos should be used of a type and in an amount to give a $2M+4M/4$ factor of at least about 0.9 together with granules having a good particle-size distribution.

The slot-sealing ability of the composition shown in test 2 of Table IX was tested. The results are presented in Table X.

TABLE X

	Concentrate mix lb./bbl.	Slot size, inches	Ml. through after 10 min. at 1,000 p.s.i.
Test No.:			
1	50	0.20	Seal broke at 680 p.s.i.
2	30	0.20	Seal broke at 730 p.s.i.
3	20	0.20	Seal broke at 250 p.s.i.
4	20	0.16	1,900.
5	15	0.16	2,450.
6	10	0.16	Seal broke at 640 p.s.i.
7	10	0.13	850.

Failure of the composition to seal a 0.20-inch slot even at high concentrations shows the ground nutshells had insufficient coarse particles to be most effective for this purpose. This might have been expected from the small amount of coarse particles in the ground nutshells as indicated in Table VIII. Since the composition sealed

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a 0.16-inch slot at a concentration of only 15 pounds per barrel of drilling fluid, it is obvious that the material is quite satisfactory for commercial use. For better slot-sealing ability, as shown in Table VII, however, the amount of coarse particles in the ground nutshells should be increased over that present in the nutshells used in the tests of Table X.

The shape, strength and other physical properties of the granular material should be as described in U.S. Pat. 2,943,680 Scott et al. The principal requirements are that the granules have a compressive strength of at least about 5,000 pounds per square inch, should be substantially insoluble in oil and water and should have a density between about 0.8 and about 2.0 grams per cubic centimeter. The particle-size distribution, however, need not be as described in the patent. Not more than about 1 or 2 percent of the granules should be retained on a No. 4 sieve of the U.S. Standard Sieve Series to avoid difficulty in pumping mud containing the granules.

The particles should preferably be well distributed throughout all sizes down to and including those passing a No. 325 sieve. As shown in Table IV, however, by using at least about 8 percent by weight of long-fiber asbestos with ground nutshells, it is possible to seal beds of large marbles even when the nutshells have a rather narrow particle-size distribution. Ideally, there should be a substantially uniform particle-size distribution from 4-mesh particles down to and beyond 325-mesh particles. From a practical standpoint, however, it is generally best to use the natural distribution of sizes produced by a grinder. The data presented in Table IX show that excellent results can be obtained by using such materials together with rather remarkably small amounts of asbestos. The screen analysis in Table VIII indicates the granular particles can be considered to be well distributed throughout the range from about 4 to about 325 mesh if they contain from about 5 percent to about 25 percent, and preferably between about 10 and about 20 percent, in each of the following fractions:

−4+6
−6+8
−8+10
−10+12
−12+16
−16+20
−20+40
−40+100
and −100

It has been shown that by use of at least about 8 percent of long-fiber asbestos, wide variations in nutshell particle-size distribution are possible. Two limitations are important, however. In order to seal wide fractures, crevices and the like, it is important that the granular materials contain at least about 20 percent by weight particles retainable on a No. 8 sieve. The other limitation concerns drilling with jet bits. When jet bits with ports as a small as $\frac{9}{32}$- of $\frac{10}{32}$-inch are used, a compromise must be made. The composition shown in test 10 of Table IV sealed a $\frac{9}{32}$-inch jet bit port and almost sealed a $\frac{10}{32}$-inch port. In order to insure against sealing of such ports, the amount of granular particles retainable on a No. 6 sieve should not exceed about 2 percent, even though this means the composition may be less capable of sealing wide fractures, crevices and the like. Commercially, two compositions are advisable, one containing a high concentration of large granules to seal large fractures and the other containing a low concentration of large particles to avoid sealing jet bit ports.

Natural resins, such as rosin, or synthetic resins, such as the thermosetting phenol-formaldehyde resins, or thermoplastic polymers, such as the methyl methacrylates, may be used as the material of which the granular particles are made. Hard rubber is another satisfactory material. Ground hard plant seed particles are greatly preferred, however, particularly ground black walnut shells.

Alternates to asbestos fibers do not seem to exist. When other fibers, such as nylon, hemp, cotton, rock wool, and the like, are made long enough to meet our requirements, these fibers tangle together to form mats which are ineffective. Asbestos seems to be quite unique, not only because the long fibers do not tangle badly into mats, but because of the ability of the asbestos, as mentioned above, to tie the granular particles together. The result is a strong mass within the pores between the large granules, such as gravel, to cement the granules together and form a strong impermeable section of formation frequently extending several inches away from the well. No other fibers have been found which will perform this function. The inertness of asbestos to fermentation is another advantage of asbestos over organic fibers.

When the nutshells and asbestos are added to drilling fluid, the resulting composition should be agitated as vigorously as possible. It has been found that the ability of the preparations to seal marbles is improved by such agitation. Apparently, the agitation separates the asbestos fiber bundles into smaller bundles which are more effective. The asbestos should be milled before use to decrease the amount of agitation required in the drilling fluid.

The combination of nutshells and asbestos can be used in several ways. For example, about 15 pounds per barrel of the mixture can be added to the entire circulated drilling fluid volume, and this volume can then be circulated in the well. It is important in such cases to bypass the shale shaker mud screens in order to avoid removing the larger nutshell particles and asbestos fibers from the drilling fluid. It is greatly preferred, however, to add a high concentration of the mixture to a small batch or "pill" of the drilling fluid in a concentration of about 30, 50 or even more pounds per barrel of the small batch. This small batch is then circulated through the well to seal the zone to which mud is being lost. It may be desirable to determine the position of the zone to which mud is being lost. The batch of drilling fluid containing the nutshells and asbestos is then spotted opposite this zone. The top of the well may then be closed in and pressure applied to squeeze the nutshells and asbestos into the zone to which mud is being lost. While the additive is referred to as a drilling fluid additive, it will be apparent that it can also be used in cement slurries or the like. The additive can also be used in combination with diatomaceous earth, with diesel oil, bentonite and cement and the like in processes known in the art.

The additive can be used in water, oil or mixtures of the two. A thickening agent, such as clay in the water phase, or an oil-dispersible soap in the oil phase, should be present in an amount sufficient to support the additive and maintain it in dispersed condition in the liquid.

Descriptions given above are presented principally as examples only. Many variations and alternates will be apparent to those skilled in the art. Therefore, we do not wish to be limited to the examples but only by the following claims.

We claim:

1. A drilling fluid additive for decreasing loss of the drilling fluid to drilled formations, said additive consisting essentially of flexible milled asbestos fibers and granular hard plant seed particles, said asbestos fibers being present in an amount equal to from about $8\times1.3/d$ to about $20\times1.3/d$ percent by weight of the total additive and having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch and retained on a 4-mesh per inch screen equals at least about $3\times1.3/d$ percent by weight of the additive, where d is the density of the granular material in grams per cubic centimeter, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and at least about 20 percent by weight of

said granular particles being retained on a No. 8 U.S. standard sieve, said granular particles having a compressive strength of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

2. The additive of claim 1 in which said asbestos is chrysotile asbestos.

3. The additive of claim 1 in which said granular particles are principally ground black walnut shels.

4. The additive of claim 1 in which said granular particles are distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20, −20+40, −40+100, and −100, all sizes being U.S. Standard Sieve sizes.

5. A drilling fluid additive for decreasing loss of the drilling fluid to drilled formations, said additive consisting essentially of flexible milled asbestos fibers and granular hard plant seed particles, said asbestos fibers being present in an amount equal to from about $2\times1.3/d$ to about $20\times1.3/d$ percent by weight of the total additive and having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch and retained on a 4-mesh per inch screen equals at least about $3\times1.3/d$ percent by weight of the additive, where d is the density of the granular material in grams per cubic centimeter, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and being distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20, −20+40, −40+60, −60+100 and −100, all sizes being U.S. standard sieve sizes, said granular particles having a compressive strnegth of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

6. A drilling fluid additive for decreasing loss of the drilling fluid to drilled formations, said additive consisting essentially of flexible milled asbestos fibers and granular hard plant seed particles, said asbestos fibers being present in an amount equal to from about $8\times1.3/d$ to about $20\times1.3/d$ percent by weight of the total additive and having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch and retained on a 4-mesh per inch screen equals at least about $0.4\times1.3/d$ percent by weight of the additive, where d is the density of the granular material in grams per cubic centimeter, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and being distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20, −20+40, −40+60, −60+100 and −100, all sizes being U.S. standard sieve sizes, said granular particles having a compressive strength of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

7. A pumpable composition suitable for decreasing loss of drilling fluid to formations penetrated by a well consisting essentially of a liquid selected from the group consisting of water, oil and mixtures of the two, flexible milled asbestos fibers, granular hard plant seed particles and sufficient of a thickening agent for said liquid to permit said liquid to support said asbestos fibers and granular particles, said asbestos fibers being present in an amount equal to from about $8\times1.3/d$ to about $20\times1.3/d$ percent of the combined weight of asbestos fibers and granular particles, said asbestos fibers having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch screen and retained on a 4-mesh per inch screen equals at least about 0.9 pound per barrel of said liquid, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and at least about 20 percent by weight of said granular particles being retained on a No. 8 U.S. standard sieve, the combined concentrations of said asbestos and granular particles being at least about $15\times d/1.3$ pounds per barrel of said liquid, where d is the density of the granular material in grams per cubic centimeter, said granular particles having a compressive strength of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

8. The compositions of claim 7 in which said asbestos is chrysotile asbestos.

9. The composition of claim 7 in which said granular particles are principally ground black walnut shells.

10. The composition of claim 7 in which said granular particles are distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20, −20+40, −40+100 and −100, all sizes being U.S. standard sieve sizes.

11. A pumpable composition suitable for decreasing loss of drilling fluid to formations penetrated by a well consisting essentially of a liquid selected from the group consisting of water, oil and mixtures of the two, flexible milled asbestos fibers, granular hard plant seed particles and sufficient of a thickening agent for said liquid to permit said liquid to support said asbestos fibers and granular particles, said asbestos fibers being present in an amount equal to from about $2\times1.3/d$ to about $20\times1.3/d$ percent of the combined weight of asbestos fibers and granular particles, said asbestos fibers having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch screen and retained on a 4-mesh per inch screen equals at least about 0.1 pound per barrel of said liquid, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and being distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20, −20+40, −40+60, −60+100 and −100, all sizes being U.S. standard sieve sizes, the combined concentrations of said asbestos and granular particles being at least about $15\times d/1.3$ pounds per barrel of said liquid, where d is the density of the granular material in grams per cubic centimeter, said granular particles having a compressive strength of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

12. A pumpable composition suitable for decreasing loss of drilling fluid to formations penetrated by a well consisting essentially of a liquid selected from the group consisting of water, oil and mixtures of the two, flexible milled asbestos fibers, granular hard plant seed particles and sufficient of a thickening agent for said liquid to permit said liquid to support said asbestos fibers and granular particles, said asbestos fibers being present in an amount equal to from about $8\times1.3/d$ to about $20\times1.3/d$ percent of the combined weight of asbestos fibers and granular particles, said asbestos fibers having sufficient long fibers so that the fibers retained on a 2-mesh per inch screen plus ¼ the fibers passing a 2-mesh per inch screen and retained on a 4-mesh per inch screen equals at least about 0.9 pound per barrel of said liquid, at least about 98 percent by weight of said granular particles passing a No. 4 U.S. standard sieve and being distributed in size so that from about 5 to about 25 percent by weight falls within each of the following size ranges: −4+6, −6+8, −8+10, −10+12, −12+16, −16+20,

—20+40, —40+60, —60+100, and —100, all sizes being U.S. standard sieve sizes, the combined concentrations of said asbestos and granular particles being at least about $10 \times d/1.3$ pounds per barrel of said liquid, where d is the density of the granular material in grams per cubic centimeter, said granular particles having a compressive strength of at least about 5,000 pounds per square inch, being substantially insoluble in oil and water, and having a density between about 0.8 and about 2.0 grams per cubic centimeter.

13. A method for stopping loss of drilling fluid to formations penetrated by a well comprising circulating down the well to the loss zone the composition of claim 7.

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