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(54) Title: STABILIZED MAGNESIUM HYDROXIDE SLURRY

(57) Abstract

A method for producing a stabilized magnesium hydroxide slurry comprising physically deflocculating the magnesium hydroxide solids in a starting slurry to produce a stabilized magnesium hydroxide slurry. Depending on the chloride level of the starting slurry, a cationic polymer may be added to the slurry. Optionally a thickening agent may be added to the deflocculated slurry. Also disclosed is a stabilized magnesium hydroxide slurry produced by the method which may be transported and stored without substantial agglomeration of the magnesium hydroxide solids.
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STABILIZED MAGNESIUM HYDROXIDE SLURRY

FIELD OF THE INVENTION:

The present invention relates to a stabilized magnesium hydroxide slurry that may be transported, pumped and/or stored without substantial agglomeration. The present invention also relates to a method for producing the stabilized magnesium hydroxide slurry.

BACKGROUND OF THE INVENTION:

Magnesium hydroxide, Mg(OH)_2, is widely used in industry as a neutralizing agent, as a gas absorber, in waste water treatment, in power plant applications and in other utilities. Magnesium hydroxide has also recently replaced sodium hydroxide in many applications due to the increased price of sodium hydroxide and to shortages of sodium hydroxide.

Magnesium hydroxide may be derived from three basic sources: seawater, well brines and magnesite ore. In a typical process a magnesium hydroxide slurry is produced from the chemical reaction of dolime (CaO-MgO) and well brine. The well brine comprises primarily calcium chloride but also includes magnesium chloride. The chemical reaction of dolime and well brine produces a slurry of magnesium hydroxide in a chloride containing liquor. The slurry is then further processed to reduce the chloride level, and concentrated to approximately 40-55% magnesium hydroxide solids. If desired the magnesium hydroxide slurry may be further processed in various types of furnaces to produce a magnesium oxide product.

The viscosity of a magnesium hydroxide slurry increases with increasing magnesium hydroxide solids loading (concentration) and does so dramatically as the loading exceeds 45%. Under normal conditions the 40-55% solids magnesium hydroxide slurry will have a relatively high viscosity, possibly between 3000 and 4000 centipoise. In many heretofore known magnesium hydroxide slurries, if agitation of the slurry is absent, the solids tend to settle and agglomerate. The settling and agglomeration results in an extremely hard mass which, after a period of time, is extremely difficult, if not impossible, to re-suspend.

A magnesium hydroxide slurry having these characteristics could not be shipped for long distances, or stored for more than a short time without agitation without the slurry ceasing to be in pumpable
form. Furthermore, even if a portion of the transported slurry could be transferred from the transport vehicle, generally a truck or rail car, the hard mass of agglomerated slurry ("heel") remaining in the vehicle could not easily be resuspended and therefore would need to be manually removed from the transport vessel by shoveling and/or scraping. Additionally, the portion of the slurry that could be transferred would need to be maintained under substantially constant agitation to prevent agglomeration of the solids.

For these and other reasons, it would be advantageous to have a stable, high solids content, magnesium hydroxide slurry that may be transported, pumped and/or stored without substantial solids agglomeration. It would also be advantageous to have a stable, high solids content, magnesium hydroxide slurry that would permit any solids that did agglomerate to be easily resuspended. Further, it would be advantageous to have a stable, high solids content, magnesium hydroxide slurry that could be transported in trucks or rail cars for a period of up to 14 days and evacuated from the truck or rail car leaving only a minimal "heel" of agglomerated solids.

The present invention achieves these advantages as well as other advantages that will be apparent from the following description.

SUMMARY OF THE INVENTION:

The present invention is directed to a method for producing a stable magnesium hydroxide slurry, and the slurry produced by the method.

According to the method of the present invention, a magnesium hydroxide slurry, produced by conventional methods such as from well brines, having the desired solids content, generally between 50 and 65% solids, by weight, is subjected to a physical deflocculation step to produce a stabilized magnesium hydroxide slurry. Depending on the chloride level of the starting magnesium hydroxide slurry, the method may also include treating the slurry with a cationic polymer to lower the viscosity of the slurry to a point which enables the physical deflocculation step to proceed to substantial completion. In a preferred method of the present invention, a thickening agent is added to the slurry during or after the physical deflocculation step.
Physical deflocculation refers to a process wherein the magnesium hydroxide solid particles are subjected to mechanical forces which break up small loose agglomerations of the particles.

The method of the present invention advantageously produces a stabilized magnesium hydroxide slurry that can have a magnesium hydroxide solids content of up to 65%, by weight, and can be shipped long distances for periods of 7-14 days without agitation and arrive in a state wherein the slurry can be removed from the transporter by pumping, pressure or gravity without leaving a significant "heel" in the transport container.

The method of the present invention also advantageously produces a stabilized magnesium hydroxide slurry that will remain stable and substantially free of agglomerated magnesium hydroxide solids for an indefinite period of time with agitation, or for at least 30 days without agitation.

The method of the present invention further advantageously produces a stabilized magnesium hydroxide slurry that will not plug up transfer lines and/or metering pumps.

The stabilized magnesium hydroxide slurry of the present invention is characterized by having a magnesium hydroxide solids content of 50 to 65%, by weight wherein substantially all of the solids are deflocculated. Preferably the stabilized magnesium hydroxide slurry of the present invention also has one or more of the following properties: a viscosity of 50-1000 centipoise (cps); a density of 1.4-1.6 grams/cubic centimeter (g/cc); a mean particle size of the magnesium hydroxide solids of 1.7-5.0 microns, preferably 3-4 microns; and a chloride level of 0.30-0.75% by weight on a MgO basis, preferably 0.35-0.60 by weight on a MgO basis. The stabilized magnesium hydroxide slurry may also include 1 to 5000 parts per million (ppm) of a cationic polymer. A preferred stabilized magnesium hydroxide slurry of the present invention additionally includes 1 to 100 ppm of a thickening agent.

The stabilized magnesium hydroxide slurry of the present invention has the aforementioned advantages as well as other advantages that will be apparent to those of ordinary skill in the art from the following more detailed description.
BRIEF DESCRIPTION OF THE DRAWINGS:

Figure 1 is a schematic of a possible embodiment of the method of the present invention.

DETAILED DESCRIPTION OF THE INVENTION:

The method of the present invention for producing a stabilized magnesium hydroxide slurry comprises the following steps:

determining the chloride level of a starting magnesium hydroxide slurry;

adding between 0 and 5000 parts per million ("ppm"), of a cationic polymer to the starting slurry; wherein if the chloride level of the starting slurry is less than or equal to 0.42%, by weight on a MgO basis, 0 ppm of the cationic polymer is added to the starting slurry; if the chloride level of the starting slurry is 0.43 - 0.52%, by weight on a MgO basis, 1000 ppm of the cationic polymer are added to the starting slurry; if the chloride level of the starting slurry is 0.53-0.65%, by weight on a MgO basis, 2500 ppm of the cationic polymer are added to the starting slurry; and if the chloride level of the starting slurry is greater than or equal to 0.66%, by weight on a MgO basis, 3500- 5000 ppm of the cationic polymer are added to the starting slurry;

physically deflocculating the magnesium hydroxide solids in the slurry, without homogenizing the slurry, to produce a stabilized magnesium hydroxide slurry.

In a preferred embodiment of the method of the present invention, the method includes the additional step of adding a thickening agent in an amount of 1-100 ppm to the slurry to raise the viscosity of the slurry to a desired level.

The stabilized magnesium hydroxide slurry of the present invention comprises:

50 - 65%, by weight, deflocculated magnesium hydroxide solids; wherein the slurry will remain substantially free of agglomerated magnesium hydroxide solids for at least 30 days.

Preferably the stabilized magnesium hydroxide slurry of the present invention also has one or more of the following properties: a viscosity of 50-1000 cps; a density of 1.4-1.6 g/cc; a mean particle size of the magnesium hydroxide solids of 1.7-5.0 microns, preferably
3-4 microns; and a chloride level of 0.30-0.75% by weight on a MgO basis, preferably 0.35-0.60 by weight on a MgO basis. The stabilized magnesium hydroxide slurry may also include up to 5000 parts per million (ppm) of a cationic polymer, wherein if the chloride level of the slurry is less than or equal to 0.42%, by weight on a MgO basis, the slurry includes 0 ppm of the cationic polymer; if the chloride level of the starting slurry is 0.43 - 0.52%, by weight on a MgO basis, the slurry may include 1000 ppm of the cationic polymer; if the chloride level of the starting slurry is 0.53 - 0.65%, by weight on a MgO basis, the slurry may include 2500 ppm of the cationic polymer; and if the chloride level of the starting slurry is greater than or equal to 0.66%, by weight on a MgO basis, the slurry may include 3500-5000 ppm of the cationic polymer. A preferred stabilized magnesium hydroxide slurry of the present invention additionally includes 1 to 100 ppm of a thickening agent which raises the viscosity of the slurry to a desired level.

The chloride level of the starting magnesium hydroxide slurry may be determined in any manner known to those of ordinary skill in the art including X-Ray Fluorescence Spectrometry. The %, by weight on a MgO basis refers to the %, by weight chloride in comparison to the calculated %, by weight of MgO in the slurry solids.

The particle size of the magnesium hydroxide solids may be determined in any manner known to those of ordinary skill in the art, including X-Ray Monitored Sedimentation.

Suitable cationic polymers for use in the method and stabilized magnesium hydroxide slurry of the present invention include but are not limited to: Holly Fix I, a cyano-guanidine polymeric resin manufactured and sold by Holly Oak Chemical, Inc., of Fountain Inn, South Carolina and having a boiling point of 212° F and a specific gravity of 1.186 @ 25° C. Additional suitable cationic polymers are known to those of ordinary skill in the art. While not wishing to be bound by any particular theory, it is believed that the cationic polymer assists in the deflocculation of higher chloride content starting magnesium hydroxide slurries by reducing the viscosity of the starting slurry to a point where the slurry may be physically deflocculated.
The physical deflocculation of the magnesium hydroxide solids in the starting magnesium hydroxide slurry may be accomplished by any means known to those of ordinary skill in the art, provided that minimal homogenization of the slurry occurs during the physical deflocculation step. Preferably, the slurry is physically deflocculated using an homogenizer wherein the operating conditions are controlled to minimize homogenization of the slurry but deflocculate substantially all of the magnesium hydroxide solids. A preferred homogenizer is an APV Gaulin Homogenizer, manufactured and sold by APV Gaulin, Wilmington, Massachusetts. In order to physically deflocculate the magnesium hydroxide solids, the slurry is passed through the APV Gaulin Homogenizer, generally at a pressure of 1000-8000 pounds per square inch (psi). The slurry may be passed through the APV Gaulin Homogenizer 1-10 times. The term "pass" refers to a complete turnover of the slurry in the vessel. In the preferred method of operation, the slurry is passed through the APV Gaulin Homogenizer 3 times at approximately 2500 psi. An alternative method of operation is to recycle the slurry through the APV Gaulin Homogenizer instead of using discrete passes. A preferred recycling time, at approximately 2500 psi, is equivalent to 3.25 passes.

Suitable thickening agents for use in the method and stabilized magnesium hydroxide slurry of the present invention include, but are not limited to: CMC (carboxymethylcellulose), guar gum and xanthum gum.

The process and stabilized magnesium hydroxide slurry of the present invention will be described in more detail with reference to Figure 1. Although Figure 1 depicts a particular embodiment of the process of the present invention, it should be understood that the process of the present invention is not limited to the particular embodiment depicted in Figure 1.

Referring to Figure 1, a starting magnesium hydroxide slurry having a solids content of approximately 50-60%, produced by a conventional process such as from well brines, is transferred from a drum filter unit, 2, utilized in producing the starting slurry, to slurry storage tank, 4. Slurry storage tank 4 contains agitation means 5, to minimize the agglomeration of the magnesium hydroxide solids in the starting slurry.
The chloride content of the starting slurry may be determined in the slurry storage tank, 4. The starting slurry is pumped from slurry storage tank 4, to process tanks 8 and 10. Optionally, the starting slurry may be pumped through drum or disc filter 6, prior to entering process tanks 8 and 10. Drum or disc filter 6, may be utilized if it is desired to further concentrate the starting slurry to raise the level of magnesium hydroxide solids.

Depending on the chloride content of the starting slurry a cationic polymer may be added to the slurry while the slurry is being pumped from storage tank 4 to process tanks 8 and 10. The addition of cationic polymer to the slurry is shown by box 7, in Figure 1. If the chloride level of the starting slurry is less than or equal to 0.42%, by weight on a MgO basis, 0 ppm of the cationic polymer is added to the starting slurry; if the chloride level of the starting slurry is 0.43 - 0.52%, by weight on a MgO basis, 1000 ppm of the cationic polymer are added to the starting slurry; if the chloride level of the starting slurry is 0.53-0.65%, by weight on a MgO basis, 2500 ppm of the cationic polymer are added to the starting slurry; and if the chloride level of the starting slurry is greater than or equal to 0.66%, by weight on a MgO basis, 3500-5000 ppm of the cationic polymer are added to the starting slurry. The cationic polymer is evenly distributed throughout the starting slurry in process tanks 8 and 10 and during passes through the homogenizer.

From the process tanks 8 and 10 the slurry is pumped through homogenizer 12, which is preferably a APV Gaulin Homogenizer, manufactured and sold by APV Gaulin of Wilmington, Massachusetts. The homogenizer 12, deflocculates the magnesium hydroxide solids in the slurry. The operating conditions of the homogenizer, and the number of times the slurry is passed through the homogenizer, should be sufficient to deflocculate substantially all of the magnesium hydroxide solids in the slurry. As described above, the slurry is passed through the APV Gaulin Homogenizer, generally at a pressure of 1000-8000 pounds per square inch (psi). The slurry may be passed through the APV Gaulin Homogenizer 12 and back into process tanks 8 and 10, one to ten times. In the preferred method of operation, the slurry is passed through the APV Gaulin Homogenizer 3 times at approximately 2500 psi. An alternative method of operation is to
recycle the slurry through the APV Gaulin Homogenizer instead of using
discrete passes. A preferred recycling time, at approximately 2500
psi, is equivalent to 3.25 passes.

Optionally, a thickening agent, such as CMC may be added to the
slurry as the slurry is being passed through the homogenizer. The
addition of a thickening agent to the slurry is shown by box 14 in
Figure 1. The amount of thickening agent added to the slurry is
generally between 1 and 100 ppm, depending on the viscosity desired in
the final stabilized magnesium hydroxide slurry.

After deflocculation has been completed, the stabilized
magnesium hydroxide slurry is transferred from the homogenizer to
final product storage tank 16. The final product storage tank 16 may
contain means for agitating, 17, however such means are not necessary
to keep the stabilized magnesium hydroxide slurry from settling. From
storage tank 17 the stabilized magnesium hydroxide slurry of the
present invention may be transferred to trucks or rail cars for
transport.

The process described above, and depicted in Figure 1, may be
utilized to produce a stable magnesium hydroxide slurry having a
solids content of 50 to 65%, by weight that will remain substantially
free of agglomerated solids for at least 30 days. The slurry may also
be characterized by one or more of the following characteristics: a
viscosity of 50-1000 cps, a density of 1.4-1.6 g/cc, and a mean
particle size of the magnesium hydroxide solids of 1.7-5.0 microns,
preferably 3-4 microns; and a chloride level of 0.30-0.75% by weight
on a MgO basis, preferably 0.35-0.60 by weight on a MgO basis; wherein
substantially all of the magnesium hydroxide solids are deflocculated.
The stable magnesium hydroxide slurry will remain substantially solid
agglomerate free without agitation during transport. After a
transportation period of up to 14 days the slurry will arrive at its
final destination in a state wherein the slurry may be removed from
the transport vessel by pumping, pressure or gravity without leaving a
significant "heel" in the container. After transfer to a storage tank
at the final destination, the stable magnesium hydroxide slurry will
remain stable for up to 30 days without agitation, and remain stable
indefinitely with agitation.
The following example will further illustrate the process and stabilized magnesium hydroxide slurry of the present invention.

**EXAMPLE**

This example illustrates the production of a stabilized magnesium hydroxide slurry of the present invention utilizing the process of the present invention.

A starting magnesium hydroxide slurry was produced by the chemical reaction of dolime and well brine. The dolime was obtained from dolomitic limestone which was quarried, crushed and calcined in kilns to produce dolime as follows:

\[ \text{MgCO}_3 \cdot \text{CaCO}_3 \rightarrow \text{MgO} \cdot \text{CaO} + \text{CO}_2. \]

The dolime was reacted with well brine to produce a magnesium hydroxide slurry as follows:

\[ \text{MgO} \cdot \text{CaO} + \text{H}_2\text{O} \rightarrow \text{MgO} + \text{Ca(OH)}_2 \]
\[ \text{Ca(OH)}_2 + \text{MgCl}_2 \rightarrow \text{Mg(OH)}_2 + \text{CaCl}_2 \]
\[ \text{MgO} + \text{H}_2\text{O} \rightarrow \text{Mg(OH)}_2. \]

The magnesium hydroxide slurry which was at this stage in an enriched calcium chloride liquor was thickened/settled, washed relatively free of chlorides, further concentrated to approximately 56%, by weight, solids and transferred to a storage vessel. At this stage the viscosity of the slurry was approximately 2000 centipoise. The magnesium hydroxide slurry thus produced, formed the starting slurry for the method of the present invention.

The chloride concentration of the starting slurry was determined by X-Ray Fluorescence Spectrometry to be 0.48%, by weight on a MgO basis. Based on this chloride level, 1000 ppm of Holly Fix 1 cationic polymer were added to the starting slurry as the slurry was pumped from the storage vessel into process vessels. From the process vessels, the magnesium hydroxide solids in the cationic polymer containing slurry were deflocculated using an APV Gaulin homogenizer at 2500 psi pressure. The slurry was passed from the process vessels through the APV Gaulin homogenizer three times. While the slurry was being passed through the homogenizer, 10 ppm of carboxymethylcellulose was added to the slurry.
After the final pass through the homogenizer, the stabilized magnesium hydroxide slurry thus formed was transferred to a storage vessel. The final stabilized magnesium hydroxide slurry comprised 56%, by weight, deflocculated magnesium hydroxide solids, 1000 ppm cationic polymer and 10 ppm CMC and had a viscosity of 150 centipoise and a density of 1.48 g/cc. The mean particle size of the magnesium hydroxide solids in the stabilized magnesium hydroxide slurry was 3.48 microns as determined by X-Ray Monitored Sedimentation using a MicroMeritics Model 5100 Sedigraph.

The stabilized magnesium hydroxide slurry could be transferred to rail cars, which may be fitted with an air sparger system, and transported for up to 14 days without agitation. After arriving at its final destination, the stabilized magnesium hydroxide slurry could be removed from the rail cars by pumping. If any settling has occurred the rail cars may be sparged prior to pumping. After the stabilized magnesium hydroxide slurry is pumped out the rail cars will be substantially free of agglomerated magnesium hydroxide solids ("heel").

After transport, the stabilized magnesium hydroxide slurry could be stored in a storage vessel wherein it will remain substantially agglomeration free, without agitation, for 30 days.
CLAIMS:

1. A method for producing a stabilized magnesium hydroxide slurry comprising:
   providing a starting magnesium hydroxide slurry including loose agglomerations of magnesium hydroxide solids and having a viscosity which enables substantially all of the magnesium hydroxide solids in the slurry to be physically deflocculated and stably suspended within the slurry; and
   physically deflocculating the magnesium hydroxide solids to produce a stabilized magnesium hydroxide slurry.

2. The method of claim 1 further comprising:
   measuring the chloride level of the slurry, and
   adding cationic polymer to the slurry to adjust the viscosity of the slurry.

3. The method of claim 2 wherein the viscosity of the starting slurry is reduced by the steps comprising:
   determining the chloride level of the starting magnesium hydroxide slurry;
   adding between 0 and 5000 ppm, of a cationic polymer to the starting slurry; wherein if the chloride level of the starting slurry is less than or equal to 0.42%, by weight on a MgO basis, 0 ppm of the cationic polymer is added to the starting slurry; if the chloride level of the starting slurry is 0.43-0.52%, by weight on a MgO basis, 1000 ppm of the cationic polymer are added to the starting slurry; if the chloride level of the starting slurry is 0.53-0.65%, by weight on a MgO basis, 2500 ppm of the cationic polymer are added to the starting slurry; and if the chloride level of the starting slurry is greater than or equal to 0.66%, by weight on a MgO basis, 3500-5000 ppm of the cationic polymer are added to the starting slurry.

4. The method of claim 2 further comprising: adding a thickening agent to the deflocculated slurry in an amount of 1 to 100 ppm.
5. The method of claim 4 wherein the thickening agent is selected from the group consisting of carboxymethylcellulose, xanthum gum and guar gum.

6. The method of claim 2 wherein the cationic polymer is a cyano-guanidine polymeric resin.

7. A stabilized magnesium hydroxide slurry produced by the process of:
   providing a starting magnesium hydroxide slurry including loose agglomerations of magnesium hydroxide solids and having a viscosity which enables substantially all of the magnesium hydroxide solids in the slurry to be physically deflocculated and stably suspended within the slurry; and
   physically deflocculating the magnesium hydroxide solids in the slurry to produce the stabilized magnesium hydroxide slurry.

8. The stabilized magnesium hydroxide slurry of claim 7 wherein the viscosity of the starting slurry is reduced by the steps comprising:
   determining the chloride level of a starting magnesium hydroxide slurry;
   adding between 0 and 5000 ppm, of a cationic polymer to the starting slurry; wherein if the chloride level of the starting slurry is less than or equal to 0.42%, by weight on a MgO basis, 0 ppm of the cationic polymer is added to the starting slurry; if the chloride level of the starting slurry is 0.43-0.52%, by weight on a MgO basis, 1000 ppm of the cationic polymer are added to the starting slurry, if the chloride level of the starting slurry is 0.53-0.65%, by weight on a MgO basis, 2500 ppm of the cationic polymer are added to the starting slurry; and if the chloride level of the starting slurry is greater than or equal to 0.66%, by weight on a MgO basis, 3500-5000 ppm of the cationic polymer are added to the starting slurry.
9. The stabilized magnesium hydroxide slurry of claim 7 wherein the process for producing the slurry further comprises adding a thickening agent to the deflocculated slurry in an amount of 1-100 ppm.

10. The stabilized magnesium hydroxide slurry of claim 8 wherein the cationic polymer is a cyano-guanidine polymeric resin.
A. CLASSIFICATION OF SUBJECT MATTER

IPC 5 CO1F5/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 CO1F B01F C09C A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database consulted during the international search (name of database and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>DATABASE WPI Week 8705, Derwent Publications Ltd., London, GB; AN 033915 SHIN NIPPO N KAGAKU 'Stable magnesium hydroxide dispersion is made with specified thixotropic index and concentration.' &amp; JP,A,61 291 413 (SHIN NIPPO N KAGAKU KOGYO) 22 December 1986 see abstract</td>
<td>1,7</td>
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<td>US,A,4 430 248 (P.R.RAY) 7 February 1984 see column 1, line 43 - column 2, line 6; examples</td>
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</table>

Further documents are listed in the continuation of box C. Patent family members are listed in annex.

Date of the actual completion of the international search

23 August 1994

Date of mailing of the international search report

09.09.94

Name and mailing address of the ISA

European Patent Office, P.B. 31818 Patentlaan 2 NL - 2230 HN Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016

Authorized officer

Zalm, W

Form PCT/ISA/210 (second sheet) (July 1992)
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<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<td>US,A,2 335 373 (T. WOODWARD) 30 November 1943 see page 2, right column, line 69 - page 3, left column, line 33; claims 1,2,4-7,9,10</td>
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<td>A</td>
<td>US,A,3 957 674 (SANO ET AL.) 18 May 1976 see column 4, line 8 - line 14; claims</td>
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