ACIDIFIED NMP COMPOSITIONS STABILIZED WITH RESPECT TO COLOR FORMATION THEREIN

Acidified NMP is stabilized against color formation by including a chelating agent in the composition.
FOR THE PURPOSES OF INFORMATION ONLY

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ACIDIFIED NMP COMPOSITIONS STABILIZED
WITH RESPECT TO COLOR FORMATION THEREIN

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to paint remover formulations, and, more particularly to acidified N-methyl-2-pyrrolidone (NMP) compositions which are stabilized with respect to color formation therein.

2. Description of the Prior Art

Acidified NMP formulations are effective paint strippers; see U.S. Patents 4,749,510; 4,812,255; and 5,049,300. However, these compositions do not exhibit long-term color stability. Usually such acidic compositions turn yellow to dark brown after a period of time. Accordingly, it is an object of this invention to stabilize acidified NMP formulations with respect to color formation therein; and, more particularly, to improve the long-term color stability of acidified NMP solutions.

SUMMARY OF THE INVENTION

What is provided herein is a composition for stabilizing acidified NMP formulations to decrease undesired color formation therein. The invention is characterized by the presence in the composition of a chelating agent, suitably in an amount of at least about 10 ppm, preferably about 50 to 1000 ppm. A preferred chelating agent is ethylenediaminetetraacetic acid (EDTA), and its salts, particularly the sodium and potassium salts.
DETAILED DESCRIPTION OF THE INVENTION

In accordance with the present invention, there is provided a method of stabilizing acidified NMP against undesired color formation by including a chelating agent therein, suitably in an amount of at least about 10 ppm, preferably 50 to 1000 ppm. Suitable chelating agents are selected among aminocarboxylic acids, for example, ethylenediaminetetraacetic acid, hydroxyethylethylene-diaminetriacetic acid, and their salts, such as the sodium and potassium salts, and mixtures thereof.

The acidified NMP formulation is prepared as described by Fusiak in U.S. Patent 5,049,300, generally by including an inorganic or organic acid having a pKa < 4.0, preferably formic acid, sulfuric acid or phosphoric acid, usually in an amount of about 1-25% by weight of the composition, preferably about 5-10%. Alternatively, NMP may be acidified by treatment with a strong acid ion-exchange resin, or by treatment with carbon dioxide or other acidic gases.

Other conventional additives for such NMP paint stripper compositions may be included herein, as described in the above mentioned patent, e.g. diluents, activators, surfactants, thickeners, flavoring agents and bittering agents.

A typical composition of the invention comprises NMP, 7% by weight formic acid and 50 ppm EDTA. Such formulation has an APHA-color rating of 12.9, which increases to only 36.9 after 48 hours at 60°C. The same formulation, without the EDTA stabilizer, also has an initial color rating of 12.9; however, it increases to 144.6 after a similar test period.
The chelators used herein may be included in the acidified NMP compositions as dry powders or as an aqueous solution, e.g. those available as Versene® or Versenol® (Dow Chemical).

The stabilized compositions of the invention have the added advantage during use as paint strippers of decreasing the degree of "black spot" formation on wood substrates that may result when paint is removed. Such black spots are believed to arise from the presence of metals or metallic ions in the coating or from iron leached from stripping tools. Such metallic ions are chelated by the EDTA stabilizer component in the composition herein and black spots do not appear on the treated wood.

EXAMPLE 1

To approximately 100 g. N-methyl-2-pyrrolidone (NMP), 7 g. formic acid (95%) were added and thoroughly mixed until homogeneous. The sample was divided into 50 ml aliquots. To one aliquot, 50 ppm of ethylenediamine-tetraacetic acid (EDTA) was added and thoroughly mixed until homogeneous. The color (APHA) of the samples was measured initially and after 48 hours at 60°C. using a Hunter Colorimeter Model D25P9. Results are listed below:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Initial</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>as is (no EDTA)</td>
<td>12.9</td>
<td>144.6</td>
</tr>
<tr>
<td>with 50 ppm EDTA</td>
<td>12.9</td>
<td>36.9</td>
</tr>
</tbody>
</table>
EXAMPLE 2

To 750 g. NMP, 0.9 g. Amberlyst® 15 ion exchange resin (strong acid, para-toluene sulfonic acid type resin manufactured by Rohm and Haas) were added and stirred for 3 hours at 80°C. After 3 hours, the NMP was flashed distilled using a one-plate distillation set-up at 100 mm Hg. To 100 g. of the distillate, 100 ppm of EDTA were added and thoroughly mixed until homogeneous. The color (APHA) of the samples was measured initially and after 48 hours at 60°C. using a Hunter Colorimeter Model D25P9. Results are listed below:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Initial</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>as is (no EDTA)</td>
<td>9.0</td>
<td>180.9</td>
</tr>
<tr>
<td>with 100 ppm EDTA</td>
<td>9.0</td>
<td>34.8</td>
</tr>
</tbody>
</table>

EXAMPLE 3

Carbon dioxide was bubbled through NMP at 450 cc/min to remove residual methylamine for 24 hours. To 100 g. CO₂ treated NMP, 885 ppm Versenol® 120 (38% trisodium hydroxyethylhexamethylenediaminetriacetic acid aqueous solution sold by Dow Chemical U.S.A.) were added and thoroughly mixed until homogeneous. The color (APHA) of the samples was measured initially and after 48 hours at 70°C. using a Hunter Colorimeter Model D25P9. Results are listed below:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Initial</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>as is (no EDTA)</td>
<td>6.4</td>
<td>74.4</td>
</tr>
<tr>
<td>with 885 ppm Versenol® 120</td>
<td>6.5</td>
<td>30.9</td>
</tr>
</tbody>
</table>
EXAMPLE 4

A paint stripping composition identical to U.S. Patent 5,049,300 was prepared. To 100 g. of the paint stripper, 2000 ppm Versenol® 120 were added and thoroughly mixed until homogeneous. Three grams of untreated and EDTA treated paint stripper were placed onto a freshly sanded red oak panel. A section of steel wool was placed on each sample. A 22 oz. weight was placed upon the steel wool to insure complete contact. After 1 hour at ambient conditions, the steel wool was removed, and the red oak panel evaluated for black spot formation.

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Black Spot Formation</th>
</tr>
</thead>
<tbody>
<tr>
<td>as is (no chelator)</td>
<td>&gt; 95</td>
</tr>
<tr>
<td>with 2000 ppm Versenol® 120</td>
<td>&lt; 5</td>
</tr>
</tbody>
</table>

While the invention has been described with particular reference to certain embodiments thereof, it will be understood that changes and modifications may be made which are within the skill of the art. Accordingly, it is intended to be bound only by the following claims, in which:
WHAT IS CLAIMED IS:

1. A composition which is stabilized against significant long-term color formation therein consisting essentially of acidified NMP and a stabilizing amount of a chelating agent therein.

2. A paint remover composition according to claim 1 in which the chelating agent is present in an amount of at least 10 ppm.

3. A paint remover composition according to claim 2 wherein said amount is about 50 to 1000 ppm.

4. A paint remover composition according to claim 1 wherein said acid is formic acid.

5. A paint remover composition according to claim 1 wherein said chelating agent is ethylenediaminetetraacetic acid, hydroxyethylendiaminetriacetic acid, their sodium or potassium salts, and mixtures thereof.
A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : C09D 9/00; C11D 3/28, 7/32, 7/52
US Cl. : 252/143, 153, 162, 170, 171, 542, 546, DIG. 8, DIG. 11
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)

U.S. : US 134/38

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>US.A, 4,749,510 (Nelson) 07 June 1988 See abstract, column 3, line 8-col. 4, line 4.</td>
<td>1-4</td>
</tr>
<tr>
<td>X</td>
<td>US.A, 5,015,410 (Sullivan) 14 May 1991 See abstract, column 5, lines 16-29, example 24.</td>
<td>1-5</td>
</tr>
<tr>
<td>Y</td>
<td>US.A, 5,049,300 (Fusiak et al) 17 September 1991 See abstract and examples.</td>
<td>1-5</td>
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<tr>
<td>Y</td>
<td>US.A, 4,812,255 (Suwola) 14 March 1989 See abstract and examples.</td>
<td>1-5</td>
</tr>
</tbody>
</table>

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

Date of the actual completion of the international search: 02 MARCH 1993

Date of mailing of the international search report: 21 APR 1993

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</thead>
<tbody>
<tr>
<td>A</td>
<td>US, A, 2,723,222 (Stark) 08 November 1955 See column 2, lines 9-61.</td>
<td>1-5</td>
</tr>
</tbody>
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