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STABILIZED PHENYL-HYDRAZINE

Reinhard Böker and Hans Georg Fuchs, Frankfurt am Main, Germany, assignors to Farbwerke Hoechst Aktiengesellschaft vormals Meister Lucius & Bruning, Frankfurt am Main Hochst, Germany

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The present invention relates to stabilized phenyl-hydrazine.

The best and simplest characteristic for the determination of the degree of purity of phenyl-hydrazine is its solidification point, since already minute impurities result in a considerable drop of the solidification point of the phenyl-hydrazine. Pure phenyl-hydrazine which, for instance, is used in considerable quantities for carrying out syntheses on an industrial scale in the pharmaceutical industry, retains its theoretical solidification point of 19.6° C. only for a short period of time. It soon starts falling to reach only 11° C. after a 6 months' storage.

Now we have found that phenyl-hydrazine can be stabilized for a longer period of time by the addition of hexamethylene tetramine, preferably in proportions up to 3 per cent. strength. The addition of the said stabilizing agent may take place after the manufacture has been terminated or during the distillation for purity of the phenyl-hydrazine by adding dropwise a solution of hexamethylene tetramine saturated in the warmth.

Phenyl-hydrazine stabilized in this manner by the addition of hexamethylene tetramine of 2 per cent. strength has a solidification point of 19.3° C. and retains, even in the presence of metals which catalytically accelerate the decomposition, a solidification point of 19.3° C. to 18.1° C. (=100 per cent. to 98.5 per cent. of phenyl-hydrazine) for a period of more than 6 months at summer-like temperatures (25° C. to 35° C.) and a lighter shade, whereas non-stabilized phenyl-hydrazine turns slowly darker when being stored.

This stabilized product allows of a storage for a longer period of time which was hitherto impossible, and a shipping in metallic containers without substantial reduction of the percentage of phenyl-hydrazine. Furthermore, it is always advisable to dispatch stable phenyl-hydrazine if it is used as preliminary product for important syntheses of remedies.

The following example serves to illustrate the invention.

Example

In 500 grams of freshly distilled phenyl-hydrazine solidifying at 19.5° C. containing 99.5 per cent. of pure phenyl-hydrazine, there are dissolved 2 per cent. of hexamethylene tetramine; into the clear solution obtained a sheet of zinc is laid and the whole is allowed to stand at 25° C. to 35° C. in a closed, brown, glass-stoppered bottle. From time to time samples are taken from the bottle for the determination of the solidification point and the degree of purity of the phenyl-hydrazine, the results obtained being compared with samples of non-stabilized phenyl-hydrazine subjected to the same conditions.

The following table indicates the solidification points and degrees of purity of the two samples depending on the time of storage.

Time (months)	Phenyl-hydrazine non-stabilized		Phenyl-hydrazine stabilized	
	Solidification Point	Content Percent	Solidification Point	Content Percent
0	19.5	99.5	19.3	99.5
1	18.5	98.5	19.0	99.3
2	16.7	97.3	18.9	99.0
3	14.5	95.7	18.8	98.9
4	14.3	94.4	18.7	98.7
5	12.5	93.2	18.4	98.6
6	11.0	91.7	18.1	98.5
7	10.1	89.5	18.0	98.5

After a 7 months' storage under closure the non-stabilized material sustained a loss of about 10 per cent. of its efficacy, whereas the stabilized material sustained a loss of about 1 per cent. under the same conditions. The rapidity of decomposition of the stabilized material after a 7 months' storage has thus been reduced to about one tenth of that of the non-stabilized product. At an increased access of air as well as at elevated temperatures an even greater difference is to be expected.

After a 12 months' storage under exclusion of air, stabilized phenyl-hydrazine still has a degree of purity of 96.1 per cent. solidifying at 17.3° C., whereas the non-stabilized product contains only 65.7 per cent. of phenyl-hydrazine.

We claim:

1. A stable phenyl-hydrazine, wherein hexamethylene tetramine is present in a proportion of 1 to 3 per cent.

2. A stable phenyl-hydrazine, wherein hexamethylene tetramine is present in a proportion of about 2 per cent.

3. A process for the manufacture of a stable phenyl-hydrazine, comprising addition of hexamethylene tetramine in a proportion of 1 to 3 per cent. to phenyl-hydrazine.

4. A process for the manufacture of a stable phenyl-hydrazine, comprising addition of hexamethylene tetramine in a proportion of 1 to 3 per cent. during the distillation for purity of phenyl-hydrazine.

5. A process for the manufacture of a stable phenyl-hydrazine, comprising addition of hexamethylene tetramine in a proportion of 1 to 3 per cent. after the manufacture of phenyl-hydrazine.

No references cited.