

1

2,737,532

STABILIZATION OF PERCHLOROETHYLENE WITH ALKOXYALKYLNITRILES

Franklin Strain, Barberton, and Bernard J. De Witt, Akron, Ohio, assignors to Columbia-Southern Chemical Corporation

No Drawing. Application February 17, 1953, Serial No. 337,441

12 Claims. (Cl. 260—652.5)

This invention relates to the stabilization of perchloroethylene.

It is known that perchloroethylene has a tendency to decompose during use, storage, and shipment. This tendency is known to be more pronounced when it is in contact with metallic surfaces, such as iron or copper. Such decomposition renders these compounds objectionable when they are employed for certain commercial operations.

One of the important commercial uses of perchloroethylene is as a solvent, for example, in dry cleaning of wearing apparel, rugs, and other fabric materials. It is an excellent solvent for oils and greases. However, when it decomposes, the products of decomposition interfere to such an extent as to materially injure its beneficial effects. When employed in metal structures, the problem of corrosion of the metal caused by decomposition of perchloroethylene is quite serious.

Various chemical compounds have heretofore been proposed for use as stabilizers for halogenated aliphatic hydrocarbons and perchloroethylene. Among those which have been proposed are, benzaldehyde, cyclohexane, toluene, benzyl chloride, amylene hexylresorcinol, butyl mercaptan, ethyl acetate, guanidine, pyridine, and others. For certain purposes, these above stated compounds are unsatisfactory in that they are ineffective stabilizers for perchloroethylene, or their odor or color is objectionable.

In accordance with this invention, it has been discovered that perchloroethylene is satisfactorily stabilized when a small stabilizing amount of an alkoxyalkylnitrile containing 1 to 3 carbon atoms in the alkoxy group and 1 to 3 carbon atoms in the alkyl group, such as β -ethoxypropionitrile, is added thereto. Such stabilized compositions have acceptable odor and color characteristics. These alkoxyalkylnitriles have a boiling point not over about 200° C. They include, for example, β -ethoxypropionitrile, β -methoxypropionitrile, β -isopropoxypropionitrile, β -methoxyacetoneitrile, β -ethoxyacetoneitrile, β -isopropoxyacetoneitrile, etc. These compounds perform as satisfactory stabilizers even when the composition has been distilled many times.

The amount of the alkoxyalkylnitrile stabilizer which is required to stabilize perchloroethylene is exceedingly small, about 0.01 to 0.1 per cent by weight of alkoxyalkylnitrile being desirable to obtain optimum results. However, larger amounts up to approximately 1 to 2 per cent are effective and may be employed if economically feasible. Perchloroethylene so stabilized remains stable over many months even after repeated distillations.

The stabilizing effect of the alkoxyalkylnitriles on perchloroethylene has been established, according to the invention, by means of a test conducted over a 72-hour period. The test is performed by refluxing perchloroethylene in a flask in the presence of water, strips of copper, and the stabilizer. One hundred milliliters of perchloroethylene is placed in a 300-milliliter flask and 0.2 milliliter of water is added. The flask is attached to a small Soxhlet extractor and a bulb type Allihn con-

2

denser is attached to the extractor. One weighed copper strip is placed in each of the flask, extractor, and condenser. The copper strip which is placed in the condenser is placed so that the perchloroethylene condenses on the strip. The condenser is connected to a water scrubber to absorb any HCl evolved during the test. The perchloroethylene is heated to boiling point, the heating being carried out so that the extractor empties at 8 to 10 minute intervals. A 100-watt incandescent bulb is placed one inch from the vapor line of the extractor to furnish energy for photochemical oxidation.

After the test is completed, part of the water in the scrubber is poured through the condenser and extractor and finally into the flask. The water and perchloroethylene are poured into a beaker, phenolphthalein is added, and the water layer is titrated with 0.01 N NaOH. The copper strips are weighed to determine their weight loss during the test.

The following table shows the results of tests of various alkoxyalkylnitrile stabilizers on perchloroethylene and compared them with the stability of perchloroethylene without any stabilizer under similar conditions:

	Amount Percent by weight of the composition	Acidity, Milliliters, of 0.1 N NaOH per 100 milliliters of C_2Cl_4	Copper Weight Losses (Milligrams)		
			Flask	Ex-trac-tor	Con-denser
β -methoxypropionitrile...	0.01	6.4	0.6	8.6	9.4
Do.....	0.03	3.5	1.4	11.8	6.0
Do.....	0.1	2.6	0.8	3.7	4.5
β -ethoxypropionitrile.....	0.01	4.4	0.5	7.7	3.7
Do.....	0.03	2.5	1.2	8.3	5.7
Do.....	0.1	6.2	0.6	5.1	3.4
No stabilizer (perchloroethylene alone).....		2,850.0	8.7	109.5	179.0
β -methoxyacetoneitrile.....	0.1	-----	1.3	7.5	13.7

These tests show the improvement in stability of perchloroethylene caused by the addition of an alkoxyalkylnitrile such as described above. Other halogenated aliphatic hydrocarbons containing up to 3 carbon atoms, such as carbon tetrachloride chloroform and trichloroethylene, may be stabilized in accordance with this invention by adding a stabilizing amount of such an alkoxyalkylnitrile.

Although the present invention has been described with reference to specific details of certain embodiments thereof, it is not intended that such details shall be regarded as limitations upon the scope of the invention except insofar as included in the accompanying claims.

This application is a continuation-in-part of our co-pending application Serial No. 167,258, now abandoned, filed June 9, 1950.

We claim:

1. The method of stabilizing perchloroethylene which comprises adding thereto a small stabilizing amount of an alkoxyalkylnitrile having up to 3 carbon atoms in the alkoxy group and up to 3 carbon atoms in the alkyl group.
2. The method of stabilizing perchloroethylene which comprises adding thereto a small stabilizing amount of β -ethoxypropionitrile.
3. The method of stabilizing perchloroethylene which comprises adding thereto a small stabilizing amount of β -methoxypropionitrile.
4. The method of stabilizing perchloroethylene which comprises adding thereto a small stabilizing amount of β -isopropoxypropionitrile.
5. The method of stabilizing perchloroethylene which comprises adding thereto an alkoxyalkylnitrile in amount corresponding to 0.01 to 0.1 per cent by weight of the mixture.
6. The method of stabilizing halogenated aliphatic hy-

drocarbons containing up to 3 carbon atoms which comprises adding thereto a small stabilizing amount of an alkoxyalkylnitrile having up to 3 carbon atoms in the alkoxy group and up to 3 carbon atoms in the alkyl group.

7. A composition of matter comprising perchloroethylene and a small stabilizing amount of an alkoxyalkylnitrile having up to 3 carbon atoms in the alkoxy group and up to 3 carbon atoms in the alkyl group.

8. A composition of matter comprising perchloroethylene and a small stabilizing amount of β -ethoxypropionitrile.

9. A composition of matter comprising perchloroethylene and a small stabilizing amount of β -methoxypropionitrile.

10. The method of stabilizing halogenated aliphatic hydrocarbons containing up to 3 carbon atoms which comprises adding thereto a small stabilizing amount of an alkoxypropionitrile having up to 3 carbon atoms in the alkoxy group and having a boiling point below 200° C.

11. A composition of matter comprising a halogenated aliphatic hydrocarbon containing up to 3 carbon atoms and a small stabilizing amount of a methoxyalkylnitrile having up to 3 carbon atoms in the alkyl group.

12. A composition of matter comprising a halogenated aliphatic hydrocarbon containing up to 3 carbon atoms and a small stabilizing amount of an ethoxyalkylnitrile having up to 3 carbon atoms in the alkyl group.

References Cited in the file of this patent

UNITED STATES PATENTS

2,043,260	Missbach	June 9, 1936
2,422,556	Klabunde	June 17, 1947

FOREIGN PATENTS

512,458	Great Britain	Sept. 15, 1939
620,296	Great Britain	Mar. 23, 1949