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STABILIZED FURFURAL

Rock L. Comstock, Weeks, La., assignor to Bay Chemical Company, Inc., New Orleans, La., a corporation of Louisiana

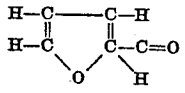
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6 Claims. (Cl. 260—347)

This invention relates to the stabilization of furfural.

Furfural is an organic liquid which is normally clear or colorless. It has many uses in modern technology, among which are its employment as a solvent or distributing agent and also as a base for certain synthetic resins. Furfural readily condenses with various reagents into larger molecular aggregates to form resinous plastics. However, furfural is normally unstable so that its tendency to resinify persists to an undesirable degree in the absence of the employment of stabilizing agents. Furfural becomes altered spontaneously even in storage, to produce gummy derivatives. These derivatives are dark in color and darken the furfural in which they are formed, even when present in considerable dilution. The dark color of furfural is, therefore, evidence of deterioration of the furfural.

It has heretofore been difficult to prevent this chemical alteration of furfural to form dark gummy products because the nature of this change is not known with certainty. It has been supposed to be due to oxidation. It is known that furfural is an organic compound of a heterocyclic system, and that its structure is that of an aromatic-like, five membered ring containing an oxygen atom in the ring, in which the carbon atoms attached to the oxygen are attached to other carbon atoms by double bond, so that furfural in structure is an unsaturated ring. The accepted chemical structure of furfural is:



Since unsaturated bonds tend to oxidize, it is obvious that furfural is susceptible to oxidation, and in the past, efforts to stabilize furfural have consisted in the use of agents intended primarily to offset the tendency to oxidize. Thus, phenolic compounds, such for example as pyrogallol, hydroquinone or catechol, well-known as reducing agents, have been used.

However, experiments underlying the subject invention indicate that the darkening of furfural during storage is not primarily an oxidation phenomenon, but is the result of polymerization, and that effective means of control is not primarily a matter of avoiding oxidation, but of inhibiting polymerization.

An object of this invention is to stabilize furfural against alteration during storage. A further purpose is to maintain the clarity or colorless nature of furfural even in the presence of air.

A particular object is to prevent spontaneous formation of dark gummy derivatives in furfural through inhibition of the normal tendency of furfural to polymerize.

This invention accomplishes stabilization of furfural by incorporating therein a small proportion of butyramid, sufficient to prevent polymerization. The amount of butyramid which it is necessary to add to the furfural is relatively small, its function being probably that of a catalyst rather than as an active reagent. A range of from 0.01% to 1.0% of butyramid is effective to inhibit undesirable chemical deterioration of furfural, but a range of from 0.01% to 0.1% is preferred.

The following examples are presented, as the result of tests, as showing the efficacy of butyramid as a stabilizer of furfural compared with certain other compounds:

Test No.	Furfural	Prospective stabilizer
	Cc.	
1	20	None.
2	20	.0023 gm. acetamid.
3	20	.0023 gm. furfuramid.
4	20	.0023 gm. butyramid.
5	20	.0023 gm. formamid.
6	20	.0023 gm. diphenylamine.
7	20	.0023 gm. benzidine.
8	20	.0023 gm. benzamid.

These preparations were prepared in glass test tubes, stoppered, exposed to ultraviolet light, and frequently observed thereafter. At the time of preparation, Test No. 3 became slightly yellow in color; no change was noted in the color of the others. At the end of four days exposure, all had assumed the same color, being the same slight yellow as Test No. 3. At the end of eight days exposure (total) the comparison was as follows, in order of lack of change:

Test No.	Prospective stabilizer	Results
4	Butyramid	Yellow (no change).
3	Furfuramid	Faint darkening.
8	Benzamid	Appreciable darkening.
6	Diphenylamine	Considerably darker.
7	Benzidine	Still darker.
5	Formamid	Do.
1	No stabilizer	Do.
2	Acetamid	Darkest (molasses color).

The compounds chosen for the above tests are all amine derivatives and were selected as prospective stabilizers of furfural due to the proved efficacy of one of them, furfuramid, as the result of previous experiments. The results, however,

shown that only two of them, butyramid and furfuramid, are useful as stabilizers of furfural, butyramid being the most effective, from which tests it is demonstrated that it is a false premise to attribute stabilizing properties with respect to furfural to a class of compounds, but that the stabilizing effect of a compound can only be determined as a matter of trial and error.

What I claim as my invention is:

1. Process of stabilizing normally unstable furfural comprising incorporating in the furfural a modicum of butyramid to substantially inhibit deterioration.

2. Process of stabilizing furfural comprising incorporating therein approximately 0.01% to 1.0% of butyramid.

3. Process of stabilizing furfural comprising incorporating therein approximately 0.01% to 0.1% of butyramid.

4. A stabilized furfural comprising normally unstable furfural containing approximately 0.01% to 1.0% of butyramid.

5. A stabilized furfural comprising normally unstable furfural containing approximately 0.01% to 0.1% of butyramid.

6. A stabilized furfural comprising normally unstable furfural containing a modicum of butyramid.

ROCK L. COMSTOCK.