

G-4 Concentration	Aspergillus niger	Penicillium citrinum	Trichoderma T-1
0.001M	100	100	100
.0001	67	20	100
.00001	31	3	52

Compound	Conc., % by wt.	Amount of Growth		Soil Burial, % Strength Retained		
		Aspergillus Niger	Chaetomium globosum	3 wks.	6 wks.	9 wks.
G-4	0.5	None	None	100	55	10
G-4	1.0	None	None	100	93	59
G-4	1.5	None	None	100	100	97
Copper naphthenate	0.5Cu*	Slight	None	78	46	12
Copper naphthenate	1.0Cu*	None	None	94	83	61

*Concentration in terms of % of copper (conc. of copper naphthenate in fabric equal to 12.5 times the copper conc.)

Organism	Concentration of G-4		Time Exp.	
	for 20°C.	for 37°C.	5 min	10 min.
Micrococcus pyogenes var. aureus (No. 209)	1—2,500	1—9,000	+	—
Salmonella typhosa (Hopkins strain)	1—6,000	1—12,500	+	—
Corynebacterium diphtheriae (avirulent)	1—9,000	1—30,000	+	—
Pseudomonas aeruginosa	1—1,100	1—3,000	+	+
Salmonella paratyphi A	1—20,000	1—55,000	+	—
Shigella dysenteriae (Flexner)	1—7,000	1—14,000	+	—
Streptococcus hemolyticus (Lancefield Group A)	1—7,000	1—14,000	+	—
Neisseria gonorrhoeae	1—35,000	(at 30°)	+	—

+ indicates growth; — indicates no growth

% pickup	lbs. of G-4-40 per 100 gals. of water	
	for 0.5% on fabric	for 1.0% on fabric
50	23 lbs., 0 ozs.	47 lbs., 4 ozs.
60	19 lbs., 0 ozs.	39 lbs., 1 ozs.
70	16 lbs., 4 ozs.	33 lbs., 4 ozs.
80	14 lbs., 4 ozs.	28 lbs., 15 ozs.
90	12 lbs., 10 ozs.	25 lbs., 10 ozs.
100	11 lbs., 7 ozs.	23 lbs., 0 ozs.

[3]

bath which converts the soluble sodium salt of G-4 to the insoluble free phenol. Intermediate drying is not required; the acetic acid should be fed at a rate which will insure that the goods leave the squeeze in an acid condition; indicator paper can be used to check this factor. The goods are then dried in the usual manner.

When a water repellent treatment is desired also, the above procedure is modified in either of two ways depending upon the type of water repellent.

A. Emulsion-type repellent.

After applying the alkaline G-4 solution in the first bath, the material is dried; about 2% of acetic acid is added to the second bath which in this case would contain the water repellent emulsion. After padding, the material is dried in the usual manner.

B. Two-bath repellent; first bath soap and wax, second bath aluminum salts.

The alkaline G-4 solution is added to the soap and wax and the mixture padded on as recommended by the manufacturer of the water repellent. After partial, or preferably complete drying, the cloth is run through the aluminum salt solution to which 2% of acetic acid has been added. The cloth is then dried in the regular way.

C. Solvent Solution:

G-4 may also be applied to textiles by means of its solution in organic solvents. On the basis of a 50% wet pick-up, the following solution is prepared:

G-4 Technical	3.0 lbs.
Isopropyl alcohol (99%)	4.0 gals.
V.M.P. naphtha or Stoddard solvent	44 gals.

G-4 is dissolved in the isopropyl alcohol; this solution is then added to the petroleum solvent with constant stirring. The final mixture is padded on at 50°C. Adequate precautions must be taken against health and fire hazards. Solvent applications are advantageous on heavy or tightly woven fabrics, such as stiff webbings, rope, heavy ducks, etc., as the fibers are quickly penetrated. For water repellency, a suitable solvent-soluble repellent should be dissolved in the petroleum solvent in the proportion normally recommended by the manufacturers. The solvent method is the only suitable procedure for treatment of textiles with a combination of G-4 and copper naphthenate.

From Aqueous Emulsion:

G-4 Technical can be formulated into aqueous emulsion, such as the following:

- A. 25 lbs. G-4 Technical
 15 gals. Pine Oil
 15 gals. Water (180 F.)
 10 lbs. Nekal® AEMA (Antara Chemicals)
 Dissolve the G-4 in the pine oil and add slowly with constant stirring to the Nekal-water solution

- B. 10 lbs. G-4 Technical
 10 lbs. Isopropyl alcohol
 1.4 lbs. Caustic soda flakes
 4 lbs. Igepal® CA extra conc. (Antara Chemicals)
 9 gals. Water
 While the use of the above emulsions is satisfactory for some purposes, they do not contain water repellents and are not recommended where water leaching is involved.

G-4 is available from several textile chemical suppliers for one bath application in the form of aqueous emulsions of the G-4 containing a water repellent. Such formulations have been found to be particularly useful on felt products.

VI. Application of G-4 to other Materials

Rubber Coated Fabrics:

The treatment of coated fabrics may involve two distinct problems, protection of the base fabric and/or protection of the coating. In the case of rubber-coated material, the protection of the base fabric is usually of paramount importance since mildew and bacteria have but little effect on rubber compounds in most uses. To achieve maximum effectiveness, G-4 should be applied to the fabric prior to coating. G-4 is particularly well suited for use in fabrics and other materials which will be in contact with rubber since it does not have any deleterious effect on natural or synthetic rubber.

Rope:

While G-4 can be impregnated into rope and cordage from solvent solution, it is usually preferred to add the G-4 to the cordage oil used in the manufacturing process. G-4 may not be soluble in the cordage oil in some cases; solutions can be achieved by adding the G-4 to the oil as a 20% solution in dipropylene glycol or similar solvent; the use of woolgrease in the oil increases the solubility of the G-4. Also, the G-4 may be satisfactorily dispersed in the cordage oil provided continuous agitation of the oil can be accomplished.

Under government specifications, a combination of copper naphthenate and G-4 is prescribed for the treatment of rope; it is required that 0.125±0.025% copper (calculated as metal) and 0.125±0.025% G-4 based on the weight of the rope be deposited in the material. If we assume that the pickup of cordage oil by the rope fibers is 12%, the cordage oil must contain 1.0% G-4; this is equivalent to adding 5 lbs. of the 20% solution of G-4 in dipropylene glycol to 95 lbs. of cordage oil containing the proper amount of copper naphthenate.

The use of this combination of G-4 and copper naphthenate is covered by Sular's U. S. Patent 2,468,068 under which the Government has been granted a royalty-free, non-exclusive license.

[4]

VAUDA

Chemical
Corp.

TECHNICAL BULLETIN D-1A

ACCEPTED

March 2, 1951

G-4[®] Technical

(Brand of Dichlorophene Technical)

As a Fungicide and Bactericide

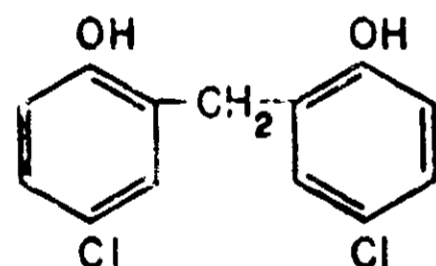
I. Introduction

G-4 is a potent fungicide and bactericide which is used to preserve cotton and woolen textiles and other materials. Mold, mildew, rot, mustiness and some types of rancidity are common expressions for the various types of deterioration caused by fungi and bacteria. G-4 is particularly effective against such deterioration.

II. Chemical and Physical Properties

Name: 2,2'-dihydroxy-5,5'-dichloro-diphenylmethane or 2,2'-methylenebis (4-chlorophenol) or bis (5-chloro-2-hydroxy phenyl) methane.

Structure



Melting point:	163 C. minimum
Appearance:	Light tan, free-flowing powder
Color:	White phenolic
Vapor pressure:	10 ⁻⁴ mm. of mercury at 100 C. about 10 ⁻⁵ mm. at 25 C. (extrapolated value)
Solubility:	Water 0.003 gram in 100 ml. Ethyl alcohol 53 of solvent at 25 C. Isopropyl alcohol 54 n-Butyl alcohol 43

Our recommendations for use of this product are based on tests believed to be reliable. The use of this product, being beyond the control of the manufacturer, no guarantee, expressed or implied, is made as to the effects of such or the results to be obtained if not used in accordance with directions and established safe practice. The buyer must assume all responsibility, including injury or damage, resulting from its misuse as such, in accordance with instructions.

t-Butyl alcohol	60
Propylene glycol	45
Acetone	80
Methyl ethyl ketone	75
Benzene	1.6
Toluene	1.7
Xylene	1.5
Stoddard solvent	0.2
Mineral Spirits	0.1

Soluble, with heat, in fatty acids and vegetable oils.

To obtain completely clear solutions of the technical grade of G-4, it may be necessary to filter the solutions.

III. Toxicity

Using the rabbit skin irritation technique, a petrolatum jelly containing 5% of G-4 was applied twice daily for 10 days. This high concentration was selected to increase the severity of the test and the margin of safety for the interpretation of the results. It was concluded from this work that G-4 was not a primary irritant.

Patch tests were also conducted on 194 humans using G-4 at a concentration of 4% in a petrolatum base ointment. The patches were applied to the inside of the forearm and were removed after 48 hours.

Out of the 194 persons tested, 191 gave negative reactions and 3 were positive.

The acute oral toxicity in animals has been determined to be as follows:

LD ₅₀ Guinea Pigs	1.25 gms/Kg
LD ₅₀ Dogs	2.0 gms/Kg

In chronic toxicity studies on rats, 0.2% of G-4 was added to the food for a period of ninety days; this dosage corresponds to a daily intake of approximately 100 mg/Kg of body weight. The animals were autopsied and histopathological studies were made on various tissues. There was no evidence of toxicity after 90 days. At a concentration of 0.5% daily in the diet, there was evidence of kidney changes at the end of ninety days.

IV. Biological Activity

G-4 exhibits both fungicidal and bactericidal properties which is an important advantage since both fungi and bacteria may be contributing factors to deterioration. The effectiveness of G-4 has been well established by the Armed Forces who consume large quantities for the protection of their equipment.

Fungicidal Properties:

The literature on the fungicidal properties of G-4 is so voluminous that only a few examples can be cited here to illustrate its activity.

Various laboratories have tested G-4 against fungi in nutrient agar medium. In these tests, the center of the agar plate was inoculated with a drop of a spore suspension of the test organism and periodic measurements of the size of this colony were made for several days. The ratio of these measurements to those of a control plate which did not contain G-4 was recorded as percentage inhibition (See Table I, page 3).

Using Trichophyton interdigitale in a standard agar plate method, a zone of 6 mm. was obtained with filter paper impregnated with a 2% solution of G-4 in alcohol; at a solution strength of 0.2% only a trace of a zone was noted.

The results of laboratory tests on cotton duck treated with G-4 are given in Table II (Page 3). Since certain of these tests are not standardized procedures, fabric samples treated with copper naphthenate were used for control purposes. These samples were tested to check the beneficial effect of a water repellent treatment.

A concentration of 0.25% of G-4 in a fabric has been found to be the minimum concentration which will pass the Aspergillus niger and Chaetomium globosum tests.

Bactericidal Properties:

Table III (page 3) shows the dilution of G-4 which will kill the various micro-organisms in 10 minutes, but not in 5.

The AOAC method of test was used with modification necessary for growing the different bacteria since G-4 is not soluble in water, necessitating the solution containing 1% G-4

was employed. 0.1 ml. of G-4 was dissolved in 1 ml. of 95% alcohol and 0.75 ml. of 0.5 N-alcoholic potassium hydroxide and, to this solution, water was added to make a total volume of 100 ml.

The data in Table III may also be expressed as phenol coefficients as follows:

	30 C. 10 min.	35 C. 10 min.
Salmonella typhosa	7	100
Micrococcus pyogenes var. aureus	12	100

Note: When G-4 is used as incorporated into substrates other than water, bactericidal efficacy tests must be conducted to prove claims for bactericidal activity. If bacteriostatic activity claims are to be made for the treated product, other appropriate tests must be conducted.

V. Methods of Application to Textiles

Recommended Concentrations:

For outdoor use, it is recommended that G-4 be applied with a water-repellent finish to obtain maximum effectiveness.

The following concentrations of G-4 are suggested:

0.25-0.5% for textiles not used out-of-doors.

0.8-1.0% for textiles subject to weathering.

Most government specifications on mildew-proofing with G-4 require that the treated fabric contain about 1% G-4 based on the dry weight of the goods.

From Alkane Soluble

While G-4 is not readily soluble in water, an aqueous solution of the sodium salt can be readily prepared. Such a solution, in a concentration of 10% of the sodium salt, is called G-4-40 and can be prepared as follows:

G-4	160 lbs
Caustic soda flakes (Tech.)	18 lbs
Water	18 gals

The G-4 and caustic soda are mixed together and put into a large container until solution is complete. The color of solution of the caustic soda is a pinkish tint; if the G-4 into solution, additional heating may be desirable to speed the process. The stock solution is then diluted with water to the desired strength. To eliminate the pinkish appearance of this solution, filter with an aid such as Super Cel (Johns-Manville Corp.).

Knowing the percentage pickup of the pad liquor by the fabric and the percentage of G-4 that should be dissolved in the fabric, one can determine, from Table IV (page 3), the dilution of the stock solution (G-4-40) that is required.

The diluted solution should be padded on at a temperature of 140-180 F. The material must then be passed through a roller which will