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SMALL QUANTITY FIELD DISINFECTION

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Globaline and halazone have been used as disinfectants since the second World War. Currently, both are undergoing re-evaluation to find a disinfectant tablet that can dissolve fast and still pass the tests of storage and time.

THERE are many occasions when water disinfection must be practiced on a small scale and under adverse conditions. The need for a ready-to-use disinfectant is greatest during military operations or at times of natural disasters when small groups of people or even individuals have to depend upon sources of water which might be contaminated. Even during peace time, campers, sportsmen and adventurers have need for a packaged, instant-disinfectant such as a tablet. This need was first recognized more than 50 years ago.[1] However, only a few preparations, usually containing either iodine or chlorine, were in use at the beginning of the Second World War. At that time a team of scientists and engineers conducted an extensive investigation at Harvard University under a contract with the Committee on Medical Research of the Office of Scientific Research and Development. The Harvard researchers [2] listed the following desirable properties of chemical disinfecting agents which are intended for use under field conditions:

1. The disinfectant should be made available as a tablet of such size as to permit use of a single or at most two tablets for a small quantity of water.
2. The method of application should be simple, substantially foolproof, and not unduly time consuming.
3. The tablet should disintegrate or dissolve quickly and liberate its active ingredient rapidly in order to allow as much time as possible for the kill.
4. Disinfectant dosages should be sufficient to ensure disinfection of all kinds of natural waters without testing for residual concentrations of the disinfectant.
5. The treated water should be acceptable to the user. Its odor, taste and appearance should not be objectionable and foods and beverage powders or concentrates placed in the water should not be changed in normal appearance or flavor.
6. The treated water should not be toxic or otherwise undesirably physiologically active over reasonable periods of use. The water, furthermore, must not interfere with essential prophylactic or therapeutic medication.
7. The treated water should not be corrosive to water containers.
8. The disinfecting agent should be stable under conditions of storage and actual use.
9. The ingredients required in compounding the disinfectant should be economically and strategically available.
10. Manufacture of the chemical agent should lend itself to large scale preparation with normally available chemical and pharmaceutical equipment.

Ingredients of a Disinfecting Tablet

In addition to a disinfectant, a tablet must contain substances which aid in its manufacture, its dissolution, and which confer buffering properties which promote the process of disinfection. These ingredients may be classified as follows:

Filler. A filler or an excipient is always required in pharmaceutical practice to give the tablet adequate bulk. A number of fillers are available but a disinfecting tablet must employ one which is soluble in order to preserve the clarity of the treated water. At the same time, the filler should not be hygroscopic and should be inert to the disinfecting chemical. As shall be discussed later, the halazone tablet has sodium chloride as an excipient. A number of soluble nitrates, phosphates, acetates and sulfates may also be employed.

Buffer. The selection of a buffer to promote disinfection is very important. For any agent which releases chlorine, it is desirable that the pH of the chlorinated water be less than 8. Above pH 8, the predominance of hypochlorite ion seriously reduces the disinfection capability. Similarly, when iodine is used, the pH of the solution should not be less than 7. Otherwise, the viricidal efficiency of hypoiodous acid will be sacrificed. Often, the buffer will also serve as a filler. This is true of globaline tablets which employ disodium dihydrogen pyrophosphate as a buffer as well as an excipient.

Lubricant. The function of this ingredient is to lubricate the punches of tablet-making machines. Talc is a popular lubricant. Calcium or magnesium stearate are also sometimes added. The function of a lubricant may sometimes be performed by the filler.

Swelling Agent. Certain colloidal clays, such as bentonite, promote the disintegration of tablets by swelling in water and causing the tablet to burst. The clay is chemically inert but physically very active.

Test Organism

The disinfecting agent employed for waters obtained under emergency conditions must be capable of killing the most resistant waterborne pathogen. The Harvard Report [2] states that "Leaving out of consideration the virus of infectious hepatitis, the cysts of *Endamoeba histolytica* appear to be the most resistant waterborne pathogens that must be dealt with in the water disinfection and so appear to determine the pattern of accomplishment that must be established both in the laboratory and in the field." Much work has since been carried out on various enteroviruses and the results confirm the earlier observations that cysts of *Endamoeba histolytica* offer greater resistance than any enteric virus, including infectious hepatitis, to the disinfecting action of chlorine.[2] Morris,[3] for instance, quotes other investigators who state that the concentrations of HOCl needed to achieve a 99 per cent kill in 10 min at 5°C for virus and cysts are 0.002–0.4 ppm and 10 ppm respectively. Chang [4] presents

data for iodine which shows that, for a contact period of 10 min at 18°C and for a 99.9 per cent kill, the concentrations of I_2 and HOI needed for poliovirus Type I and *E. histolytica* are those given in Table 1. Table 1 indicates that, for effective disinfection of cysts and virus with iodine, both molecular iodine and hypoiodous acid should be present in solution. At pH 7, a dilute solution of iodine contains almost equal percentages of molecular iodine and hypoiodous acid.[5]

Tablets in Use

There are currently two tablets being used for water disinfection in the United States. The halazone tablet has been in use prior to and during World War II. The disinfectant employed is a chlorine compound. The other tablet, globaline, which contains an iodine-based disinfectant, is used by the US Armed Forces for the disinfection of canteen waters.

The composition of the halazone [2] tablet is given in Table 2.

The chemical name of halazone is p-dichlorosulfonamidobenzoic acid. It reacts with water to release hypochlorous acid up to 50 per cent of the titrable chlorine present. One tablet dissolved in a quart of water liberates a titrable chlorine concentration of 2.3 ppm and a maximum concentration of HOCl (as Cl_2) of 1.1 ppm. Titrable chlorine is defined [2] as the total oxidizing power of the material or solution under consideration which is effective in oxidizing iodine ion to iodine in dilute acetic acid solution, expressed as ppm of elemental chlorine. The reaction of the halazone in water is:

$$Cl_2NO_2SC_6H_4COOH + H_2O \rightarrow ClHNO_2SC_6H_4COOH + HOCl$$

In this tablet sodium chloride is the filler and the remaining two compounds form an alkaline buffer.

The globaline tablet derived its name from a chemical compound which was developed at Harvard. Globaline was first identified as triglycine hydroperiodide, $(NH_2CH_2COOH)_3:HI:I_2$ (2). The formulation was later modified to tetraglycine hydroperiodide $(NH_2CH_2COOH)_4:HI:1.24I_2$. This compound provides 42.32 per cent titrable iodine and 59.42 per cent total iodine. The composition of the globaline tablet is given in Table 3. One tablet dissolves in a quart of water to give 8 ppm of titrable iodine.

The talc is employed as a lubricant and disodium dihydrogen pyrophosphate serves as an acid buffer as well as an excipient. This acid buffer serves to lower the pH of natural waters for, at the time the tablet was developed, it was believed that elemental iodine was more germicidal than its main hydrolysis product, hypoiodous acid.

TABLE 1

Concentrations of I_2 and HOI Needed for Poliovirus Type I and E. Histolytica

Species	Poliovirus Type I	*E. histolytica*
Iodine, mg/l	20	2.5
Hypoiodous acid, mg/l	0.45	4

TABLE 2

Composition of Halazone Tablet

Component	Amount *mg*
Halazone	5.30
Soda ash, dried	5.18
Boric acid	11.92
Sodium chloride	114.00
Weight/tablet	136.40

Comparison of the Globaline and Halazone Tablets

An exhaustive study of the properties of both globaline and halazone has been conducted.[2] The following is a summary of some of the results:

Dissolution Time. Field studies employing soldiers in acceptability tests indicated that they considered rapid solubility of tablet as a primary criterion for acceptability. They were impatient with agents that required a waiting period of more than 10 min.

For field simulation, the tablet to be tested was placed in a liter volumetric flask containing tap water at 23°C. The stoppered flask was then inverted end-over-end continuously, causing the tablet to drop through water until it was dissolved. These tests showed that while globaline disintegrated and dissolved in less than one min, standard halazone tablets dissolved in 7.5 min. Thus, in the case of halazone, the actual contact time between the disinfectant and the organism would be 2.5 min if 10 min is taken as the total waiting time.

The disintegration of the tablets is primarily controlled by the filler and expanding agent used in the tablet. Since halazone contains sodium chloride which hardens or "sets up," it suffers from a low rate of solution. The solubility of halazone itself varies with pH. It is low and constant up to a pH of about 4. Above this pH, the solubility increases rapidly, either because of hydrolysis of the dichlor group or through ionization of the carboxylic acid group.

pH	3.8	5.5	5.6
halazone solubility—g/l	0.09	0.83	1.200

It is possible that a change in the filler now employed in halazone might improve the dissolution time of the tablet markedly.

TABLE 3

Composition of Globaline Tablet

Component	Amount *mg*
Tetraglycine hydroperiodide	19.3 to 21
Disodium dihydrogen pyrophosphate ($Na_2H_2P_2O_7$)	82.5 to 92.3
Talc	not more than 6
Weight/tablet	110 to 120

The solubility of globaline is far greater than that of halazone; about 380 g/l of distilled water. The disintegration of the tablet, however, limits the rate of solution of the globaline.

Decreased water temperature increases the time required for solution for both tablets in accordance with the Van't Hoff-Arrhenius formulation. Some of the dissolution times observed were:

	10°C	20°C	30°C
Globaline	1.9 min	1.2 min	0.8 min
Halazone	9.5 min	8 min	6.5 min

In general, tests showed that storage at 60°C and room humidity did not affect the solution properties of either tablet.[2]

Comparing iodine and chlorine as disinfectants for small water supplies,

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the former appears to have certain advantages for the following reasons:

1. On a molar basis, iodine is more cysticidal than hypochlorous acid.
2. Iodine has very little organic demand as compared to chlorine.
3. Chlorine has a strong affinity for nitrogenous matter, whereas iodine has almost none.
4. Both predominant forms of iodine, molecular iodine and hypoiodous acid, are efficient germicides. They form an excellent combination for cysts and enterovirus. On the other hand, where chlorine is used, hypochlorous acid alone is a good germicide.

Cysticidal Dose. Cysticidal doses of globaline and halazone tablets were determined in Cambridge tap water alone or with the addition of interfering substances that might be present in natural polluted water.[2] The cyst density employed, 60/ml of water, was considered to be far higher than the concentration of cysts in sewage. It was estimated that in an area of high endemicity, the ratio of amoebic cysts to *E. coli* would be of the order of 1 to 100,000. The number of coliform organisms discharged by an individual is estimated to be about 400 bil/day. An infected individual would discharge cysts in numbers varying from several hundred to some 10 mil/day. This ratio would make the number of cysts in concentrated sewage about 10/ml.

Table 4 indicates that one tablet of globaline should be able to disinfect all cysts, pathogenic bacteria, and spores. No conclusive tests were carried out against organisms of infectious hepatitis and other enteroviruses. However, from the work of Chang[4] it is now possible to speculate on the viricidal capacity of waters disinfected with globaline. At 10°C, the pH of tap water was lowered to 6.6 by the addition of one globaline tablet which left a residual of 6.9 ppm of iodine. At pH 6.6, about 5 per cent of the titrable iodine is in the form of HOI.[6] As a result, the hypoiodous acid concentration is about 0.35 ppm. This amount of HOI may not be sufficient to be viricidal. The high concentration of titrable iodine and the use of an acidic buffer result, therefore, in a high cysticidal but low viricidal efficiency.

Tests with halazone tablets showed that at room temperature about 5 tablets/qt of water were required to destroy all cysts in 10 min, whereas 2.5 tablets were effective in 30 min. In moderately to heavily polluted water at the same temperature, 7 tablets were needed for disinfection in 10 min and about 5 for disinfection in 30 min. Larger dosages of these tablets are required because:

1. The halazone tablet can release a maximum concentration of titrable chlorine equal to 2.3 ppm and HOCl equal to 1.1 ppm (as Cl_2). This is far less than the dose required under adverse conditions. Morris[3] reports that 10 ppm of HOCl are required for 99 per cent kill of *E. histolytica* in 10 min at 5°C.
2. The halazone tablet has an alkaline buffer to aid in dissolving the compound. Unfortunately, at high pH the predominant species of chlorine is OCl^- which is about 100 times less cysticidal than HOCl.
3. The dissolution time of the halazone tablet is slow; 7.5 minutes at room temperature.

On the other hand, the tablet has a great advantage over the globaline tablet in that HOCl is an excellent viricidal agent.[3,7] It appears safe to assume, therefore, that if a certain dose of HOCl is cysticidal, it is also sufficient for all types of enterovirus. From information available, it appears that several improvements may be possible in the preparation of halazone tablets. The first is the inclusion of an acidifying agent which will promote the formation of HOCl but which will not affect the solubility of the compound. The second is the substitution of a filler which will increase the rate of solution of the tablet.

Acceptability of Tablets by Users. The acceptance of the disinfecting agent by the user is probably as important as its germicidal action. The user may hesitate to use the agent because of unpleasant taste, odor or color; adverse physiological reaction; or excessive time for disinfection.

TABLE 4

Effectiveness of Globaline

Kind of Water	Temp °C	Contact Time *min*	pH		Cysticidal Dose *tablet/qt*	Cysticidal Residual *I₂-ppm*
			Initial	Final		
Tap	3	25	8.0 to 9.0	6.5	1	7.5
Tap	10	15	8.0 to 9.0	6.6	1	6.9
Tap	23	10	8.0 to 9.0	7.3	1	7.5
Tap	28	5	8.0 to 9.0	6.65	1	7.5
Tea infusion	23	5	7.2	6.4	2	8.7

1. Unpleasant Taste, Odor or Color.

Tastes and odors may be caused either by the tablet itself in water or by its combination with beverage powders. For purposes of comparison, the Whipple scale of intensity of odors and tastes[8] was adopted as a yardstick to determine the relative palatability of the tablets. Investigators[2] used 4 tablets of halazone providing about 10 ppm of titrable chlorine and 1 tablet of globaline providing 8 ppm of titrable iodine per liter of boiled distilled water at 23°C. The pH was varied with citric acid, dihydrogen disodium pyrophosphate, and sulfuric acid. The water was tested by seven to fourteen subjects. The results obtained indicate that in the "pH range commonly encountered," the globaline was more acceptable than halazone. In fact, in this range of pH, globaline produced "faint" to "distinct" intensity of odor and taste whereas halazone treated water was rated "decided" to "very strong" range on the Whipple scale. The "objectionable thresholds" were also determined in boiled distilled water at 23°C and the results are shown in Table 5.

It is apparent that globaline would reach the "objectionable threshold" only if 2 tablets were used as is prescribed for heavily polluted waters. As for the effect of pH upon tastes and odors, it was deduced, though not conclusively, that tastes and odors were minimal at the pH values attained when the tablets are added to neutral, unbuffered waters. To study the effect

TABLE 5

Per Cent of Normal Cysticidal Dose at Which "Objectionable Threshold" Is Reached

Compound	Percentage					
pH	4	5	6	7	8	9
Halazone	50	40	25	25	25	25
Globaline	—	200	—	200	—	—

of temperature on the tastes and odors, tests were made at temperatures of 15°C, 23°C, and 30°C. The results indicate that the intensity increased with temperature but did not become objectionable even at 30°C, although at that temperature no water is pleasant to drink. All observers agreed that the coldest drink was the most palatable.

With regard to the effect of disinfection on beverage powders, no specific tests were made with either of the tablets.

2. Adverse Physiological Reaction.

The physiological response of the use of iodinated water has long been a matter of concern. A number of laboratory as well as field studies have been reported. Studies were conducted at Department of Pharmacology, Harvard University (O. Krayer); Division of Pharmacology, Food and Drug Administration; Army Medical Research Laboratory, Fort Knox; and Naval Installations, Marshall Islands.

All of these investigations were performed using iodine in concentrations equivalent to or in excess of those used in the field purification process. The tablets themselves were not used in these tests. The first three studies or their conclusions have been described in the Harvard Report [2] whereas the fourth study has been reported by Morgan and Karpen.[9] While the first three studies indicate that the ingestion of iodine-disinfected water by healthy male adults should have no injurious effect, the analysis of data in the fourth study revealed no evidence of weight loss, failure of vision, cardiovascular damage, altered thyroid activity, anemia, bone marrow depression, renal irritation, sensitization to iodine, predisposition to diseases of the skin, or impaired wound healing.

A more exhaustive study is now under way at Gainesville, Fla., under A. P. Black where far lower dosages of iodine are being used. Partially reported results indicate that there is no evidence that iodine, under the experimental conditions employed, has had any detrimental effect on general health or thyroid function.[5, 10]

3. Time for Disinfection.

As mentioned earlier, the acceptability tests show that the soldiers in the field are impatient with disinfectants which take more than 10 min to complete their action. In other words, the dissolution should preferably take place in a matter of sec to leave a full 10 min contact time for sterilization.

4. Use of Globaline in Vietnam.

In 1968, Hurst and Bird conducted a survey of the reaction of marines in Vietnam to the use of the globaline "water purification" tablets.[11] The survey indicated that over two-thirds of the marines added a soft drink mix, such as Kool-Aid, Fizzies, Twist, Fla-Vor-Aid, or Funny Face, to their canteen water to conceal the taste imparted by the globaline tablet. Subsequent laboratory studies by these investigators showed that the ascorbic acid used in the preparation of some of the soft drink mixes will react to reduce elemental iodine to iodide. Therefore, if the tablet and the mix are added simultaneously, no persistent iodine residual is obtained and the disinfecting capacity of the globaline tablet is seriously reduced.

Other results of the survey indicated that the majority of the marines were using 2 globaline tablets per canteen. On the other hand, 11 per cent were using none. The majority of the latter group cited "bad taste" or "no time" as reason for not using the purification tablet.

The survey made an attempt to find out how the marines were applying the soft drink mix. About two-thirds of those marines who employed soft drink mixes added the globaline tablet first and "waited" before adding the mix. The other one-third added the mix prior to or simultaneously with the globaline tablet.

When asked to recommend a means for improving the purification tablets, the marines suggested that:

1. A better tasting purification tablet be prepared.

2. Another method of purification be devised "without delay."

3. That a soft drink mix be added to C-rations.

Hurst and Bird also conducted laboratory studies on the rate of solution of the globaline tablets.[11] They observed that tablets manufactured in 1961–1965 required from 2 to 10 min to dissolve. As a result they prepared new tablets using the standard tetraglycine hydroperiodide tablet, sodium bicarbonate, and citric acid. These reportedly dissolved "in less than 30 sec."

The method of packaging iodine tablets, 50 per bottle, was subjected to criticism. "When a marine has wet hands, the current method of packaging makes it difficult to dispense 1 or 2 tablets from a bottle without getting water into the bottle. . . ."

In conclusion, these investigators recommended that "a tablet . . . be prepared which contains tetraglycine hydroperiodide, goes into solution rapidly, has a flavoring agent which does not contain a reducing agent, and is packaged in a manner which will give long-time protection to the tablet and provide for easy dispensing."

Thermal Stability of Tablets. Water disinfecting tablets designed for global use must be capable of withstanding extremes in air temperature as well as heat developed in storage warehouses. To test the stability of globaline, accelerated storage tests at 140°F and room humidity were carried out to determine the rate at which tablets decompose and active ingredients are dissipated.[2] Tests on globaline powder indicate that it lost about 30 per cent of its iodine after 1 month and about 60 per cent after 2 months. On the other hand, results of experiments with halazone tablets indicated that no appreciable loss of available chlorine occurred after 20 days at 140°F. Therefore, the halazone tablet can be described as thermally stable.

Resistance to Humidity. To determine the relative stability of tablets in humid atmospheres, they were subjected to tests at humidities of 100, 79 and 55 per cent at room temperature. The gain in weight and the loss of active ingredient after certain time intervals at room temperature was measured.

At 100 per cent humidity globaline appeared to be more stable than halazone as the former retained 37 per cent of original iodine and the latter 24.7 per cent of original chlorine after the same number of days.

At 79 per cent humidity as well as 55 per cent humidity, globaline appeared to be less hygroscopic. Over long periods of time at 32 per cent humidity, globaline again proved to be a stable substance.

In order to gage resistance to humidity and thermal stability during actual use in the field, bottles of globaline and halazone tablets, with and without cotton plugs, were placed in a control room held at 80 to 90 per cent humidity and approximately 80°F. Every 2 hours during the day each bottle was opened for 1 min and a tab-

let was drawn. Over a 3-week period, none of the compounds showed an appreciable loss of strength, and there was little variation between the bottles with or without cotton plugs.

Corrosion of Metals. To see the effect of halazone and globaline disinfected waters on the materials of canteens, a series of experiments was conducted on aluminum and steel canteens. To perform accelerated tests, the strength of solutions was quadrupled. Thus, the globaline solution contained 32 ppm of titrable iodine and the halazone solution had 20 ppm of titrable chlorine. Two types of tests were conducted, drip tests and immersion tests.

In the drip tests, the solutions were allowed to drop upon the experimental metal and run down it for about 9 hr each day over a period of 36 to 50 days. The same solution was used over and over again, but it was freshly reconcentrated each day with the respective tablets. The loss in metal was assumed to be an indication of corrosion. The results showed that the steel canteen metal was much more resistant to corrosion than the aluminum metal. Upon aluminum, globaline appears to be more corrosive than halazone, although upon steel, the action of globaline is less pronounced than that of halazone.

In immersion tests with the same solutions of globaline and halazone tablets, the former was less corrosive than the latter on steel canteens but the reverse was true in case of aluminum canteens.

Summary and Conclusions

The globaline tablet was developed as a result of a tremendous effort on the part of scientists and engineers at Harvard during the Second World War. It has satisfied most of the criteria set for a disinfecting tablet.

The globaline tablet was produced on the assumption that molecular iodine alone is germicidal (and not its hydrolysis products) and that cysts of *E. histolytica* represent the test organism. Molecular iodine is known to be an extremely good cysticidal agent but several researchers [4, 7] have shown that it is much less viricidal than hypoiodous acid. The earlier assumption that "the destruction of virus by disinfectants appear to be of the same order of magnitude as that of most pathogenic nonsporulating bacteria" [2] has therefore been invalidated.[3, 7] Thus it would be useful to evaluate the viricidal power of globaline tablets. Because the acidic buffer in globaline lowers the pH, the amount of hypoiodous acid liberated may be insufficient to kill any virus present. The substitution of an alkaline buffer (pH 8) would yield about 40 per cent HOI and 60 per cent molecular I_2.

At room temperature the globaline tablet was expected to dissolve in less than a min. Recent studies [11] have indicated that it may take longer, perhaps from 2 to 10 min. However, these tests were performed on tablets which had been manufactured a few years earlier. The discrepancy may be ascribed either to the adverse effect of storage on the solution properties of the tablet or the pressure exerted by tableting machines. Since these factors are difficult to control, further studies to find a more suitable swelling agent are indicated. An alternative would be an effort to make the tablet effervescent.

Color, taste, and odor problems associated with the use of globaline tablets are reportedly moderate.[2] In fact, these characteristics might be taken as a qualitative indication of the presence of an iodine residual. However, the reaction of marines in Vietnam to the tablet has been surprisingly strong.[11] In fact, a large majority of the marines surveyed found it necessary to mask the taste imparted by the globaline tablet by adding soft drink mixes to their canteen waters. In some cases this resulted in a reduction in the disinfecting capacity of the purification tablets.

The halazone tablet, at present, is considered unsuitable for military use, not only because little titrable chlorine is released, but because most of the chlorine released is present as OCl^- due to the presence of an alkaline buffer. Since OCl^- is about 100 times less cysticidal than HOCl, the efficiency of the chlorine is greatly reduced. The alkaline buffer is added to increase the rate of solution of halazone. It might be possible, however, to prepare a reasonably soluble tablet buffered at a pH of 6 rather than 8 or 9.

Finally, the practice of packaging 50 tablets of globaline in a single bottle may also be subject to improvement. Once opened for the use of the first tablet, the remaining tablets may start to "set up" or harden. In addition, the disinfecting agent may be lost. With modern improvements in packaging techniques and materials, it may not be difficult to devise a package which contains 1 or 2 tablets. Alternately, the use of powder pillows may be a solution to the problems of stability and solubility.

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