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PHARMACEUTICAL SCREENING TEST COMPOSITION

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13 Claims

ABSTRACT OF THE DISCLOSURE

A two-part analytical reagent composition for determining the general pharmacological classification of a pharmaceutical preparation or a generally protein-free body fluid obtained from a person suspected of having a systemically active amount of a narcotic, stimulant, depressant, salicylate or hallucinogenic lysergic acid diethylamide. The first reagent comprises a reactant product solution of a major proportion of sulphuric acid together with minor proportions of solutions of formaldehyde, ammonium chloride, arsenic trioxide, lead acetate, and calcium hypochlorite together with minor amounts of potassium dichromate, pyridine and para-dimethylaminobenzaldehyde and optionally ferric ammonium sulfate or cobalt thiocyanate. The second reagent comprises an aqueous solution of a surfactant, such as an alkyl aryl sulfonate-polyphosphate composition, together with minor amounts of dinitrophenol and formaldehyde solution. Upon the addition of a pharmaceutical substance or a relatively protein-free body fluid, such as urine or filtered blood serum, to the reagents, a characteristic coloration is produced which is qualitatively determinative of the broad pharmacological groupings set forth hereinabove.

This application is a continuation-in-part of my copending application Ser. No. 165,782, filed July 15, 1971 for Pharmaceutical Screening Test Composition now abandoned, which copending application was a continuation of my application Ser. No. 699,348 filed Jan. 22, 1968 for Pharmaceutical Screen Test Composition, now abandoned.

The present invention relates generally to a simple method of rapidly qualitatively determining the broad pharmacological grouping of certain pharmaceutical preparations. More particularly, the present invention relates to a rapid method of qualitatively determining within a matter of minutes whether an unknown solid or liquid substance contains a narcotic, stimulant, depressant, salicylate, or the hallucinatory drug LSD. More specifically, by observing a color change that occurs when the unknown solid or liquid is added to a reagent system as set forth herein, within a matter of a few minutes, generally within a matter of seconds, it may be determined whether such unknown contains any substance falling within one of the aforementioned pharmacological groupings, and even more specifically which group.

Numerous methods of analysis for the identification of pharmaceutical preparations falling within the above-mentioned pharmacological groupings have been proposed. Generally speaking, however, such methods of analysis have either been very time-consuming and required highly trained personnel and sophisticated laboratory facilities, etc., so as to render such methods impractical or even undesirable for the rapid qualitative grouping of pharmaceuticals or the nature of pharmaceuticals within a body fluid such as urine or relatively protein-free blood serum. In addition, the previously recognized analytical reagent

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for natural narcotics, i.e., Marquis' Reagent (8-10 drops of 40% formaldehyde solution to 10 ml. pure concentrated sulphuric acid) cannot be used for the analytical identification of synthetic narcotics such as codeine or morphine. More significantly, if the Marquis' Reagent is not freshly prepared, it may develop a red, blue or purple color upon the addition of an opium-containing substance depending upon the age of the reagent.

A large number of situations arise where it becomes extremely important to make a rapid qualitative analysis of an unknown solid or liquid substance suspected of containing a pharmacologically active component which falls into the aforementioned broad groupings. An example of one situation involves a case of poisoning by the use of either a stimulant or conversely a depressant drug. In order to be able to apply rapid proper treatment to the poisoning victim, a doctor is dependent upon a rapid qualitative analysis of the patient's urine or blood serum. While it is appreciated that there are existing methods of making a qualitative analysis of such body fluids for stimulants and depressants, the tests are relatively laborious, time-consuming and require extensive laboratory equipment. Another situation which frequently arises involves the field of law enforcement where a law enforcement officer requires a rapid determination as to whether an unknown substance, including a body fluid, contains a narcotic, stimulant, depressant, salicylate or LSD component. This is especially important in the control of narcotic traffic in the underworld.

It is therefore an object of the present invention to provide a simple, rapid, reliable means for the qualitative identification of pharmaceutical substances generally falling within the nature of a narcotic, stimulant, tranquilizer, salicylate or LSD-containing substance whereby a doctor or a law enforcement officer may within a matter of minutes, or even seconds, determine whether a particular substance contains a pharmacologically active component falling within these broad groupings.

Another object of the present invention is to provide a simple, rapid, reliable method for qualitatively identifying a narcotic substance whether it be natural or synthetic in nature.

A further object of the present invention is to provide a novel analytical reagent composition for the qualitative identification of certain groups of pharmaceuticals whereby such identification can be rapidly and simply effected by a doctor, medical technician, law enforcement officer, etc., without the necessity of using elaborate laboratory equipment.

Still another object of the present invention is to provide a novel two-part analytical reagent composition for the qualitative identification of narcotics, stimulants, tranquilizers, salicylates and LSD and wherein the reagent composition is characterized by a relatively long shelf-life without significant loss of ability to rapidly identify substances containing components falling within the pharmacological groups set forth hereinabove.

Further objects and aspects of this invention will become apparent from the more detailed description thereof which follows hereinafter.

Briefly, the reagent composition, used in the method also set forth herein, is specifically utilized to screen-out and qualitatively determine whether an unknown substance, be it in a dry pulverulent or liquid form, contains a narcotic, stimulant, depressant (tranquilizer), salicylate or the hallucinatory drug LSD. The analytical reagent composition is not utilized to establish whether such unknown solid or liquid has a specific narcotic or other dangerous drug within it, but is used to classify the sample being tested within a pharmacological grouping as either a narcotic, stimulant, depressant, salicylate or the

hallucinatory drug LSD. Therefore, for example, if the utilization of the present analytical reagent composition qualitatively establishes that a substance of narcotic pharmacological effect is present in an unknown sample, then a complete laboratory analysis, in a manner as conventionally proposed heretofore, of the unknown is necessary to determine the specific narcotic in the unknown sample.

The analytical reagent composition of the present invention consists of two solutions broadly referred to hereinafter as Solution A and Solution B. If the unknown to be analyzed is in a solid, generally non-particulate form, it must be reduced to a powder, and assuming it is of a volumetric mass approximately equivalent to a pharmaceutical tablet, then introduced into approximately 2 ml. of Solution A and the admixture gently agitated after which the reactant vessel i.e. vial or test tube, is allowed to stand for approximately one to two minutes to permit a color change, i.e. characteristic color development. If the unknown imparts a purple color to Solution A, the unknown sample has a component within it which has a narcotic pharmacological effect. If the unknown imparts a deep red color to Solution A the presence of a component which has the pharmacological effect of a salicylate is indicated. If, however, a brown color is imparted to Solution A the unknown contains a component which has a pharmacologically stimulating effect. If the unknown is a liquid, i.e., either in dosage form or suspected to be within a body fluid such as urine or relatively protein-free blood serum, the unknown is introduced slowly in approximately 2 ml. of Solution A in such a manner so as to provide heterogeneous layers whereby a reaction, and characteristic color change, may occur at the site of the interface between Solution A and the unknown liquid.

If the mixture of Solution A and the solid unknown, or the area of the interface between the Solution A and a liquid unknown, fail to undergo a color change after approximately 1 to 2 minutes several drops of Solution B are introduced into the Solution A-unknown system, and the Solution A-unknown-Solution B system observed after approximately 2 minutes for a characteristic color change. If the addition of Solution B, in the aforementioned manner, causes a color change of the mixture from amber to dark brown, then such color indicates that the unknown being tested contains a depressant drug, i.e., tranquilizer within it. If the addition of Solution B, to the Solution A-unknown system, causes the system to change to a bluish color, the unknown material contains the hallucinatory drug LSD within it. If the addition of Solution B to the Solution A-unknown system brings about the development of a deep red color, then the presence of, and generally predominance of, a salicylate in the unknown is indicated.

Furthermore, the colorimetric test of the present invention differentiates between a depressant drug and a stimulant drug in that a stimulant drug produces a brown color upon admixing it with Solution A, but, a depressant drug will not impart a brown color to Solution A until after the addition of several drops of Solution B into the Solution A-unknown system.

The preparation of the two-part analytical reagent composition of the present invention requires the utilization of the following reagents.

1% aqueous solution of arsenic trioxide
1% aqueous solution of ammonium chloride
2% aqueous solution of lead acetate
0.05% aqueous solution of calcium hypochlorite
96% sulfuric acid (95.5% to 96.5%) dinitrophenol
37% formaldehyde solution U.S.P. (with 10%–15% methanol inhibitor)
pyridine
potassium dichromate
p-dimethylaminobenzaldehyde
surfactant (preferably Coleo which is a registered trademark of Colgate-Palmolive-Peet Co. for a proprietary

product which is understood to contain: 15% sodium docecylbenzene sulfonate, 50% sodium tripolyphosphate, 5% sodium pyrophosphate, and 30% sodium hexametaphosphate).

However, it will be understood that the surfactant set forth is merely exemplary and that other surfactant compositions, having analogous ionic activity, i.e., strongly alkaline surfactants which, upon addition of Solution B to Solution A, react to generate heat, may be substituted for the specific surfactant set forth, without departing from the scope of the invention.

Optionally, the two-part analytical reagent may also include the following reagents: 1% aqueous solution of ferric-ammonium sulfate, or cobalt thiocyanate.

Analytical reagent Solution A is prepared as follows:

- (a) to about 47.9 to 50 ml. 37% formaldehyde solution, U.S.P.
- (b) add approximately 0.020 to 0.025 ml. 1% aqueous solution of ammonium chloride, 0.018 to 0.023 ml. 1% aqueous solution of arsenic trioxide, 0.018 to 0.030 ml. of 2% aqueous solution of lead acetate, 0 to 0.020 ml., preferably 0.010 to 0.020, of 1% aqueous solution of ferric-ammonium sulfate, and 0.008 to 0.012 ml. of 0.05% aqueous solution of calcium hypochlorite

Intimately admix the above solutions by thoroughly washing the measuring pipette, or other measuring device, with the formaldehyde solution.

- (c) add the above (a)–(b) solution slowly to 925 to 970 ml. of 96% sulfuric acid (95.5% to 96.5%).

The admixture of solution (a)–(b) with the sulfuric acid is effected in a manner so as to create a minimum amount of heat, such as by the utilization of a glass rod within the vessel containing the acid whereby the (a)–(b) solution is slowly layered beneath the sulfuric acid by pouring downwardly along the glass rod.

- (d) with the glass rod still in place inside of the reaction vessel, about 0.25 to 0.48 gm. of potassium dichromate is added, preferably at or near the glass rod, after which the reactant solution is stirred thoroughly and allowed to cool to ambient temperature.
- (e) after cooling, 2 to 2.8 ml. pyridine are dissolved in the above system.
- (f) to the approximately 1000 ml. of (a)–(b)–(c)–(d)–(e) is added 2095 to 2175 mg. of p-dimethylaminobenzaldehyde after which the so-completed Solution A is stored and tightly sealed in a receptacle.

As can be seen in step (b), the use of ferric ammonium sulfate is optional. If it is eliminated, the color changes of the solution become slightly less vivid. In lieu of the ferric ammonium sulfate, an equivalent amount of cobalt thiocyanate may be substituted. Use of a cobalt thiocyanate permits the specific identification of the narcotic cocaine. When Solution A includes cobalt thiocyanate in place of ferric ammonium sulfate, and the unknown is cocaine, instead of the solution turning purple, a light blue precipitate will form and settle out of the solution. It will be appreciated, however, that Solution A may include either cobalt thiocyanate or ferric ammonium sulfate or, alternatively, may contain neither component.

Solution B of the analytical reagent composition of the present invention, also referred to hereinafter as the activator solution, is prepared by:

- (a) adding 1500 to 1800 mg. dinitrophenol, 1 to 2 liquid oz. surfactant (preferably Coleo—as set forth hereinabove), 26 to 34 ml. of 37–40% formaldehyde U.S.P.
- (b) to sufficient water to dilute to one liter (about 900 ml.)

After preparation, the resultant Solution B placed in a tightly sealed receptacle.

The preferred compositions for Solutions A and B are set forth below:

Solution A, for each approximately 1000 ml., comprises the following active components in the amounts indicated:

37-40% aqueous formaldehyde solution	ml.	47.9	5
1% aqueous solution of ammonium chloride	ml.	0.025	
1% aqueous solution of arsenic trioxide	ml.	0.020	
2% aqueous solution of lead acetate	ml.	0.025	
1% aqueous solution of ferric ammonium sulfate or cobalt thiocyanate	ml.	0.020	10
0.05% aqueous solution of calcium hypochlorite	ml.	0.010	
96% sulfuric acid	ml.	950	
Potassium dichromate	gm.	0.35	
Pyridine	ml.	2.0	15
p-Dimethylaminobenzaldehyde	mg.	2160	

Solution B, for each approximately 1000 ml., comprises the following active components in the amounts indicated:

Water	ml.	900	
Surfactant	oz.	1 liquid	
37-40% aqueous formaldehyde solution	ml.	30	
Dinitrophenol	mg.	1680	

The following examples are included to specifically illustrate the utilization of the preferred compositions of the two-part analytical reagents of the present invention for the colormetric identification of substances pharmacologically active as narcotics, stimulants, tranquilizers, salicylates and the hallucinatory drug LSD.

EXAMPLE I

A capsule known to contain a narcotic, i.e., codeine, was admixed with approximately 2 ml. of Solution A, formulated as set forth hereinabove, and allowed to stand for a color change to develop. After approximately 1 to 2 minutes Solution A developed a purple color, this indicating the presence of a narcotic in the capsule. If the unknown were in the form of a tablet it would have been necessary to first crush the tablet before admixing it with Solution A.

EXAMPLE II

A tablet known to contain a salicylate and a placebo tablet were subject to testing as in Example I, and the salicylate containing tablet imparted a deep red coloration to Solution A and the placebo did not impart any coloration to the Solution A.

EXAMPLE III

A test somewhat similar to Examples I and II were repeated with a meprobamate tablet, a placebo and a caffeine containing tablet in separate test vials. The meprobamate i.e., tranquilizer, imparted no coloration to Solution A and four drops of Solution B were added thereto and in approximately 2 minutes the test solution changed from amber to dark brown as characteristic of the presence of tranquilizer drugs. The placebo failed to impart coloration to Solution A and failed to impart any coloration to mixture of Solution A as well as Solution B. The caffeine containing drug imparted a brown coloration to Solution A, without the addition of Solution B as is necessary with a tranquilizer.

EXAMPLE IV

The contents of a capsule known to contain LSD was added to approximately 2 ml. of Solution A and in the absence of any development of color therein approximately four drops of Solution B was admixed therewith. After approximately 1 to 2 minutes the solution became bluish in color as has been found to be characteristic of the presence of LSD. It will be noted that should a deep red color occur subsequent to the addition of Solution B to a generally uncolored Solution A-unknown, the predominance of a salicylate in the unknown is indicated.

Although not specifically set forth by example, it will nevertheless be understood that the two-part analytical reagent composition of the present invention may be utilized to determine whether a sample of urine or protein-free filtered blood serum contains a toxic dose of a narcotic or other dangerous drug. This test may be used in a clinic, hospital, or wherever the urine or blood samples are readily available. In this regard, it is to be remembered that when blood samples are being tested, they must be protein-free filtered. If the material to be tested is in a liquid form, a small amount is dropped or poured slowly down the side of a vial containing approximately 2 ml. of Solution A so as to layer the unknown into the vial without actually admixing the unknown-containing liquid. When testing liquid unknowns a colormetric ring indicating the pharmacological group to which the unknown belongs may form at the interface between the present solution and the unknown solution.

The foregoing is considered as illustrative only of the principles of the invention. Further, since modifications may occur to those skilled in the art, it is not desired to limit the invention other than by the scope of the invention as claimed.

I claim:

1. A two-part analytical reagent composition comprising:

(1) a first solution which, for each approximately 1000 ml., comprises in the approximate amounts set forth as active components.

47.9 to 50 ml. 37%-40% aqueous formaldehyde solution

0.020 to 0.025 ml. 1% aqueous solution of ammonium chloride

0.018 to 0.23 ml. 1% aqueous solution of arsenic trioxide

0.018 to 0.030 ml. 2% aqueous solution of lead acetate

0 to 0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate

0.008 to 0.012 ml. 0.05% aqueous solution of calcium hypochlorite

925 to 970 ml. 96% sulfuric acid

0.25 to 0.48 gm. potassium dichromate

2.0 to 2.8 ml. pyridine

2095 to 2175 mg. p-dimethylaminobenzaldehyde;

(2) and a second solution which for each approximately 1000 ml. comprises in the approximate amounts set forth as active components:

1 to 2 liquid oz. surfactant

26 to 34 ml. 37%-40% aqueous formaldehyde solution

1500 to 1800 mg. dinitrophenol

water to dilute to 1000 ml.

2. The composition of claim 1 wherein said surfactant consists essentially of: sodium dodecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate.

3. A two-part analytical reagent composition, as claimed in claim 1 wherein said first solution has the following composition for each approximately 1000 ml. of solution:

47.9 ml. 37%-40% aqueous formaldehyde solution

0.025 ml. 1% aqueous solution of ammonium chloride

0.020 ml. 1% aqueous solution of arsenic trioxide

0.025 ml. 2% aqueous solution of lead acetate

0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate

0.010 ml. 0.05% aqueous solution of calcium hypochlorite

950.0 ml. 96% sulfuric acid

0.35 gm. potassium dichromate

2.0 ml. pyridine

2160 mg. p-dimethylaminobenzaldehyde; and said second solution has the following composition for each approximately 1000 ml. of solution:

900.0 ml. water
1 liquid oz. surfactant
30.0 ml. 37%–40% aqueous formaldehyde solution
1680 mg. dinitrophenol.

4. The composition of claim 3 wherein said surfactant consists essentially of: sodium dodecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate.

5. A method of forming a two-part analytical reagent composition which comprises forming a first solution which, for each approximately 1000 ml., comprises the following active components in the approximate amounts set forth, by the procedure comprising adding:

- (a) approximately 0.020 to 0.025 ml. 1% aqueous solution of ammonium chloride, 0.018 to 0.023 ml. 1% aqueous solution of arsenic trioxide, 0.018 to 0.030 ml. of 2% aqueous solution of lead acetate, 0 to 0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate, and 0.008 to 0.012 ml. of 0.05% aqueous solution of calcium hypochlorite, to 47.9 to 50 ml. 37%–40% aqueous formaldehyde solution,
- (b) slowly adding the solution resulting from (a) to approximately 925 to 970 ml. of 96% sulfuric acid,
- (c) dissolving approximately 0.25 to 0.48 gm. potassium dichromate in the solution resulting from (b),
- (d) adding 2 to 2.8 ml. pyridine to the solution resulting from (c),
- (e) dissolving approximately 2095 to 2175 mg. of p-dimethylamino benzaldehyde in the solution resulting from (d);

and forming a second solution which, for each approximately 1000 ml., comprises in admixture

1500 to 1800 mg. dinitrophenol
1 to 2 liquid oz. surfactant
26 to 34 ml. 37%–40% aqueous formaldehyde solution, and
water to dilute to 1000 ml.

6. The method of claim 5 wherein said surfactant consists essentially of as active components: sodium dodecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate.

7. A method, as claimed in claim 5, wherein said first solution, for each approximately 1000 ml. is formed by the procedure comprising adding:

- (a) approximately 0.025 ml. 1% aqueous solution of ammonium chloride, 0.020 ml. 1% aqueous solution of arsenic trioxide, 0.025 ml. of 2% aqueous solution of lead acetate, 0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate, and 0.010 ml. of 0.05% aqueous solution of calcium hypochlorite, to 47.9 ml. 37%–40% aqueous formaldehyde solution,
- (b) slowly adding the solution resulting from (a) to approximately 950 ml. of 96% sulfuric acid,
- (c) dissolving approximately 0.35 gm. potassium dichromate in the solution resulting from (b),
- (d) adding 2 ml. pyridine to the solution resulting from (c),
- (e) dissolving approximately 2160 mg. of p-dimethylaminobenzaldehyde in the solution resulting from (d);

and said second solution, for each approximately 1000 ml., is formed by admixing

900 ml. water
1680 mg. dinitrophenol
1 liquid oz. surfactant and
30 ml. 37%–40% aqueous formaldehyde solution.

8. The method of claim 7 wherein said surfactant consists essentially of as active components: sodium didecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate,

9. A method of colorimetrically qualitatively determining the presence of a narcotic, stimulant, tranquilizer, salicylate substance and LSD in a sample which comprises the steps of:

- (a) adding the sample to a first test solution comprising a reaction product of:

aqueous formaldehyde solution
aqueous solution of ammonium chloride
aqueous solution of arsenic trioxide
aqueous solution of lead acetate
a reagent selected from cobalt thiocyanate and an aqueous solution of ferric ammonium sulfate
aqueous solution of calcium hypochlorite
sulfuric acid
potassium dichromate
pyridine
p-dimethylaminobenzaldehyde;

- (b) observing the said first solution-sample system for a characteristic color development wherein the development of a purple color indicates the presence of a narcotic, the development of a deep red color indicates the presence of a salicylate and the development of a brown color indicates the presence of a stimulant;

- (c) in the absence of the development of any coloration in the said first solution-sample system, adding several drops of a second test solution to said first solution-sample system, said second test solution comprising:

water
surfactant
aqueous formaldehyde solution
dinitrophenol;

- (d) after a short time period observing the said first solution—said second solution—sample system for a characteristic color development wherein the development of a dark brown color indicates the presence of a tranquilizer, the development of a bluish color indicates the presence of the hallucinatory drug LSD and the development of a deep red color indicates the predominance of a salicylate in the sample.

10. The method of claim 9 wherein said surfactant consists essentially of, as active components: sodium dodecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate.

11. The method of claim 9 wherein said first test solution comprises, for each approximately 1000 ml.,

47.9 to 50 ml. 37%–40% aqueous formaldehyde solution
0.020 to 0.025 ml. 1% aqueous solution of ammonium chloride
0.018 to 0.023 ml. 1% aqueous solution of arsenic trioxide
0.018 to 0.030 ml. 2% aqueous solution of lead acetate
0 to 0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate
0.008 to 0.012 ml. 0.05% aqueous solution of calcium hypochlorite
925 to 970 ml. 96% sulfuric acid
0.25 to 0.48 gm. potassium dichromate
2.0 to 2.8 ml. pyridine
2095 to 2175 mg. p-dimethylaminobenzaldehyde;

and said second test solution comprises, for each approximately 1000 ml.,

1 to 2 liquid oz. surfactant
26 to 34 ml. 37%–40% aqueous formaldehyde solution
1500 to 1800 mg. dinitrophenol
water to dilute to 1000 ml.

12. The method of claim 11 wherein said surfactant consists essentially of, as active components: sodium dodecylbenzene sulfonate; sodium tripolyphosphate; sodium pyrophosphate; and sodium hexametaphosphate,

13. The method of claim 12 wherein said first test solution comprises, for each approximately 1000 ml.,
 47.9 ml. 37%–40% aqueous formaldehyde solution
 0.025 ml. 1% aqueous solution of ammonium chloride
 0.020 ml. 1% aqueous solution of arsenic trioxide
 0.025 ml. 2% aqueous solution of lead acetate
 0.020 ml. of a reagent selected from cobalt thiocyanate and a 1% aqueous solution of ferric ammonium sulfate
 0.010 ml. 0.05% aqueous solution of calcium hypochlorite
 950.0 ml. 96% sulfuric acid
 0.35 gm. potassium dichromate
 2.0 ml. pyridine
 2.160 mg. p-dimethylaminobenzaldehyde;

and said second test solution comprises, for each approximately 1000 ml.,
 900.0 ml. water
 1 liquid oz. surfactant
 30.0 ml. 37%–40% aqueous formaldehyde solution
 1680 mg. dinitrophenol.

References Cited

William P. Butler, "Methods of Analysis," IRS Publication No. 341, 77–78, revised June 1967.

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