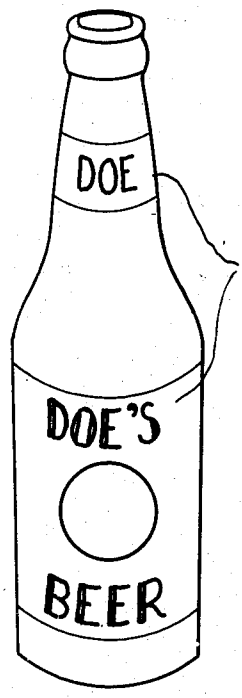
Oct. 6, 1953 L. R. NESTOR 2,654,170

DISINTEGRABLE LABEL AND WASHABLE CONTAINER LABELED THEREWITH

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Fig. 1.



Disintegrable Label.

Fig. 2.



Pigmented printable surface coating.

Fibrous base containing aluminum particles.

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DISINTEGRABLE LABEL AND WASHABLE CONTAINER LABELED THEREWITH

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This invention relates primarily to removable water-resistant labels for returnable containers such as beverage bottles.

Bottled beverages are commonly conditioned and temporarily stored in ice-chests, in contact with cold water, before being sold to consumers. Printed labels, either of paper or of a laminated construction, are adhered to the bottles as a means of identification. These labels are required to remain in place and in good condition under the conditions existing in the ice-chest, in order to fulfill their intended purpose of identifying and maintaining a demand for the bottled product. They must be firmly adhered to the bottle, and must have sufficient "wet strength" to keep from being torn and mutilated as they are jostled about in the ice-chest.

An object of this invention is the provision of a disintegrable label for beverage bottles. The label must be desirably decorative and attractive in appearance, and must remain in good condition during active storage of the bottled beverage in an ice-chest. The label must also have the ability to come away from the bottle, and to disintegrate, when passed through a warm 5% caustic cleaning solution in a bottle-washing machine. Another object is the provision of labeled bottles which remain attractively labeled during ice-chest conditioning and active storage, and which when passed through the typical bottle-washing process herein described are rapidly and completely freed of labels or portions thereof. Other desirable features and objects of the invention will become apparent on consideration of the specification and the appended claims.

In the drawing, Figure 1 represented a beverage bottle labeled with a neck label and a body label made in accordance with my invention, and Figure 2 represents a cross-section of a portion of one of said labels. The labels remain intact even after prolonged active storage of the bottles in ice-water. They are sufficiently water-resistant so that the conventional water-soluble label adhesives provide adequate bonding. On passing the bottle through weak (5%) caustic in a bottle-washing machine, the label completely disintegrates, exposing the adhesive, and remains dispersed in the solution as substantially separate paper fibers, which are easily rinsed from the bottle and removed from the machine.

One way in which I have prepared labels having the above-described as well as other advantageous characteristics will now be described.

The fibrous pulp required for the paper-making operation was conveniently obtained by re-

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pulping a preformed flat unsized sheet of saturating paper, a product made of bleached sulphite pulp having a high content, at least about 90%, of alpha-cellulose. The sheet had a basis weight of 35 lbs. per ream of 320 square yards, a lengthwise tensile strength of 35 lbs. per inch and a porosity (Gurley Densometer reading, 400 cc. of air through one thickness of the paper) of 20 seconds. A one-inch strip of the paper was suspended with the bottom dipping into water; the water was drawn upward in the strip by capillarity, reaching a height of 1 inch in 3½ minutes. "Duracel" paper has the indicated properties and is one example of a suitable source of pulp.

Three hundred pounds of paper as above described were mixed with water in a paper beater to a concentration of approximately 3½%. The paper rapidly broke up into a loose pulp. To this pulp was then added 30 lbs. of fine aluminum powder, No. 30 XD Standard Lining, and the stock was given a minimum beating action for one-half hour. Where the powdered aluminum was added as a dry powder, it was found desirable to add a dispersing agent, e. g., three pounds of "Triton R–100" was added in the beater; however, a more desirable method involves forming a thick slurry of the powder in alcohol and adding the slurry to the stock in the beater. In either case, the pulp and powder are lightly beaten together for a short time.

The lightly beaten pulp was then formed into a sheet on a cylinder machine, removed from the cylinder on a felt, removed from the felt in the wet state and wound into a roll, for temporary storage and convenience in handling, and finally rewound over steam drums to yield a dried paper.

The cylinder was fitted with a 70-mesh wire. Suction was applied from beneath the felt, and a good pressure was applied on the press roll, to produce a wet sheet containing approximately 70% water. The sheet at this point had a thickness of 0.012 inch. It was produced at 100 feet per minute, using a pulp at about ¼% concentration. The concentrated pulp from the beater was diluted to this figure by recycling the white-water from the cylinder.

Analysis of the dried product at this point showed it to contain 6–8% aluminum, fairly evenly distributed throughout the thickness of the sheet, although the wire side was not quite as bright in appearance as the felt side of the sheet.

The dried paper was then coated with a solution of zein (alcohol-soluble and alkali-soluble

corn protein) in ethylene glycol monoethyl ether, decoratively pigmented with aluminum flake. The formula of this solution was as follows, in parts by weight:

Zein	30
Ethylene glycol monoethyl ether	120
Aluminum flake	3.6
Triethylene gylcol (plasticizer—may be omitted)	10

This solution was roll-coated on the felt side of the dried paper in an amount just sufficient to provide a smooth finished appearance; the dry weight was found to be 3–4 grains per 24 square inches. Drying was carried out at 175° F.

The above-described procedure provided a label stock which printed well, and which when tested in the form of printed labels on bottles was found to remain in place and in good condition on the bottles in the ice-chest, and to disintegrate completely in the bottle-washer. In the laboratory dipping test described earlier, this label was removed and disintegrated within 25 dips, as previously noted hereinabove. It represents a preferred example of the product of my invention.

The same base sheet, without the added sizing coat, distintegrated rapidly in the caustic solution. However, it was weakened considerably by prolonged contact with cold water, and also had a mottled and less desirable appearance. Additionally, it was found that certain types of printing inks penetrated the sheet and bonded the fibers together to such an extent that disintegration in caustic was considerably delayed. With some base sheets and some classes of ink, disintegration was actually completely prevented by the coating of ink. The same inks could be used in printing my surface-sized label stock with no significant effect on the subsequent disintegration of the label in caustic.

Zein, an alcohol-soluble and alkali-soluble protein obtained from corn, is a preferred example of an alkali-soluble film-forming binder or adhesive for the decorative pigment of the sizing coat. Zein is non-discoloring on ageing, provides a firm and flexible bond, and is rapidly soluble in warm dilute caustic. Casein, another alkali-soluble film-forming protein, is also useful. Rosin may be used as a binder where sunlight ageing is not a factor, and where somewhat less toughness and brittleness is permissible. Carboxy methyl cellulose provides an alkali-soluble but water-insoluble binder which has been found useful. "Petrex" resin, the highly acidic resinous adduct of maleic anhydride and terpenes, is another example of an alkali-soluble film-forming binder or adhesive having some utility in my surface coatings.

Aluminum flake is a preferred example of a decorative pigment; in conjunction with the binder it forms a metallic film which provides an attractive appearance and also assists in the disintegration and solution of the binder component. Aluminum foil, adherently laminated to the fibrous base by means of the soluble binder, is another type of metallic film which also provides an excellent surface for printing, and is even more decorative than the aluminum flake. Porous sputtered or evaporated films of aluminum or other metals are also useful. The presence of aluminum particles in the binder accelerates its disintegration in the caustic, and I prefer to include a percentage of such particles in all cases.

In connection with the coating compositions in which aluminum flake is present in an alkali-soluble film-forming binder, it has been observed that dried films of such compositions will themselves provide water-resistant, rapidly alkali-disintegrable base sheets for labels. In a further modification, aluminum foil is laminated to the film. The foil provides a printable surface, and also prevents any possible stretching of the label and provides for accurate register during printing.

Another method of preventing deformation or stretch of the film is by the introduction of loose or unbonded fibers into the mixture of alkali-soluble binder and aluminum particles. Short segments of loose fibers, e. g. nylon, "Saran," or other natural or synthetic high-strength fibers, may be added and pressed into the still sticky film prior to complete removal of solvent. Fibers and film-forming components may be mixed together and simultaneously formed into a sheet. Lightly bonded felted webs or batts of short non-hydrated fibers may replace the pre-formed paper sheets hereinbefore described; the powdered aluminum may be held in such sheets either by a separate adhesive binder or by adhesion to synthetic solvent-activated or heat-activated fibers such as cellulose acetate.

In the specific construction in which aluminum foil is laminated to a disintegrable fibrous base, the foil serves as an alkali-soluble base surface for printing, and also provides an effective water barrier. In this case, water-soluble adhesives, e. g. starch (which is soluble also in dilute caustic solution), may be employed to laminate the foil and paper together, since the foil effectively prohibits excessive penetration of water to the laminating adhesive while the labeled bottle is in the ice-chest. Here also, it is found desirable to add a small percentage of aluminum powder to the water-soluble adhesive.

Alkali-insoluble components may be included in the coatings. For example, titanium dioxide or other white or colored pigment may be added to produce a desired appearance. Where aluminum powder is included, some alkali-insoluble binder may be used in conjunction with the alkali-soluble type, although ordinarily in minor proportions.

Another example of a water-resistant distintegrable label having somewhat improved appearance as contrasted with the coated label described hereinabove was produced as follows. The low-wet-strength fibrous base made, as before described, from lightly hydrated alpha-cellulose fibers and aluminum flake, was first coated with an amount of a 20% solution of zein in ethylene glycol monoethyl ether sufficient to provide approximately ¾ grain dry weight per 24 square inches. This coating, applied with rolls, penetrated the sheet material and reduced its porosity. The sheet was then additionally coated, from the same side, with about 1 grain per 24 square inches, dry weight, of an aluminum varnish. A drying oil-Bakelite (phenolic) resin long-oil varnish was used, and 12 parts by weight of aluminum flake were added (to 100 parts of varnish on the dry basis) just prior to coating. The aluminum flake "leafed out" to provide a shiny and uniform surface appearance, closely approaching that of the laminated foil type label. The resulting printed labels were fully satisfactory in their resistance to cold water, and also in their ability to disintegrate rapidly under mild agitation in warm dilute caustic. The varnish type

binder, which is ordinarily only slowly affected by caustic, rapidly disintegrated, due presumably to the action of the aluminum as well as the presence of the alkali-soluble sub-coating.

Throughout the specification and claims, where aluminum is specified, other alkali-reactive and gas-evolving amphoteric elements, of which zinc and silicon are examples, are contemplated as fully equivalent thereto. Reaction of the aluminum with caustic alkali solution is extremely rapid at the temperatures generally employed in bottle-washing machines. The hydrogen produced by the reaction provides internal pressure within the sheet or label, tending to separate the components thereof. The soluble components rapidly dissolve, leaving the insoluble components, largely the cellulosic fibers. Since, in my novel label stock, these fibers have an extremely low degree of inter-bonding, they are rapidly and completely separated under these conditions.

In the specific examples of paper base labels given above, minimum fiber inter-bonding is obtained by holding the amount of beating of the paper-making fibers to an absolute minimum. It is well recognized that paper-making fibers which have been beaten (hydrated), formed into a sheet, and dried are no longer hydrated and do not regain their hydration when simply dispersed in water. The half-hour beater operation with the bars set for minimum beating action, i. e. a so-called "light-brush" contact, appears to be sufficient to grind or embed the particles of aluminum into or on to the fibers, but does not provide even the minimum amount of hydration previously considered essential for label-stock formation. This is particularly true where, as in the present invention, it is desired to retain a considerable volume of pigment; retention of pigments would ordinarily be accomplished by providing rather heavily beaten, highly hydrated pulp.

The "hydration" of the cellulosic fibers may, according to various theories, involve chemical hydration, fibrillation or other physical action, or some combination of the two. Regardless of theory, the term is here used to signify the quality obtained by beater or Jordan action in the paper mill, and which is commonly measured in terms of reduced "freeness" of the pulp.

In a laboratory dip test, in which labeled bottles were repeatedly dipped into and withdrawn from narrow cylinders containing violently agitated 5% caustic soda solution at 140–150° F., and in which about 235 dipping cycles were estimated to be equivalent to a pass through a single section of a typical bottle-washing machine, conventional labels were not disintegrated at 1000 dips; whereas my preferred labels required only 25 dips for complete disintegration. Such a label is found to be substantially weakened and at least partially disintegrated on simple immersion in hot 5% caustic. In practice, commercial large-scale bottle-washing operations involve a considerable amount of agitation, and under these conditions it is not ordinarily necessary to employ a structure which is as nearly spontaneously disintegrable as my preferred product. Since increased hydration of pulp makes for less difficulty in forming and handling a paper web on the machine, it is sometimes desirable to go in this direction at the expense of some of the ability to spontaneously disintegrate. For example, a lightly beaten kraft pulp containing 13 parts by weight of aluminum powder and one-half part of alkali-soluble alum-precipitated rosin size for

each 100 parts of dry paper-making fibers was formed on a Fourdrinier paper-making machine into a sheet containing 4–5% of aluminum. This sheet was coated on the felt side with plasticized zein-and-aluminum sizing as described in connection with the previous example, and printed as labels. The individual labels were adhered to bottles and tested in the laboratory dip test. The labels quickly came away from the bottles and broke down into loose ½-inch portions within 250 dips in the hot dilute caustic, which represents an ability to disintegrate sufficient for many applications.

This same stock could, however, be converted to a spontaneously disintegrable and hence preferred type of sheet. For example, re-pulping of the aluminum-containing base sheet of the above example, addition of further aluminum powder, and re-formation of the sheet by a procedure analogous to that described in connection with the alpha-cellulose sheet, followed by impregnation and coating as before, produced a label which was fully equivalent to the alpha-cellulose label both in water-resistance and in its ability to disintegrate in dilute caustic. Other paper-making fibers which have been hydrated only to an analogous limited degree are found to be equally useful.

In confirmance of these results, a large number of bottles were labeled with the two varieties of my novel printed labels which in the laboratory test disintegrated in 25 dips and in 250 dips, as hereinabove defined. The labels were applied in a commercial bottle-labeling machine using a water-soluble starch base label adhesive. After preliminary ageing of the adhesive bond, the labeled bottles were cleaned in a commercial brewery type bottle-washing machine having five compartments each charged with warm 5% caustic soda solution. All of the labels were completely removed and disintegrated in the first two compartments. After a continuous run of eight hours, the mass of pulp in the bottom of these compartments was readily flushed out through a 2-inch drainpipe. Previous experience with the usual type of paper label in an equivalent run would have indicated the necessity of closing down the machine, draining the tanks, and then removing the pulp, through the manholes provided for that purpose, by hand operations. This tedious and expensive practice was entirely eliminated through the substitution of my novel disintegrable paper labels for the previously known paper labels. Continuous operation of the washer was made possible.

In addition, the amount of caustic soda required to charge the washer was decreased, since only two tanks were required in place of the customary five; the pulp was concentrated in the first two tanks; no labels were carried over into succeeding tanks by the bottles or chains after having once been removed from the bottles; and no labels or fragments of labels remained attached to or lodged in the bottles after the final rinsing.

The various factors which influence the disintegration of the label, such as degree of hydration of the fibers, amount of aluminum, thickness and density of sheet, type and quantity of coating, etc. must be balanced against each other in producing the desired product. As already noted, the degree of hydration must be very low in order that the fibrous sheet may be disintegrated; but increased agitation in the washer will permit somewhat increased hydration in the pulp, since

additional energy is then available for separating the fibers once they have been loosened by the action of the evolved gas. The amount of aluminum may be within the preferred range of 5–10% as indicated by the examples, but papers having as low as 2–3% aluminum have shown rapid disintegration, and higher percentages are also contemplated. The percentage of aluminum required for adequate disintegration of the sheet is found, for example, to be dependent on the size and distribution of the aluminum particles, the greatest efficiency being obtained with label stock in which extremely finely divided particles of aluminum, having a high surface-to-weight ratio, are uniformly distributed throughout the sheet. Excessive amounts of aluminum are uneconomical and may result in excessive or dangerous amounts of evolved hydrogen.

Although considerable variation in the thickness may be tolerated in my disintegrable sheet material, labels as employed for bottled beverages are normally standardized at about 2–4 mils, or somewhat greater in the case of paper-and-foil laminations. Extremely thin papers are either lacking in strength and hence difficult to print and apply, or if made stronger, e. g. by longer beating of the pulp, do not completely disintegrate during washing. One reason for the latter effect is believed to be that the evolved gas is rapidly released from the extremely thin paper without providing much disruptive force between individual fibers or layers of fibers. On the other hand, very thick papers, which ordinarily must be made in several plies, tend to come apart in layers rather than as loose fibers, and in general are not as satisfactory as those of the general range of thickness above indicated.

Where in the above-described specific examples I have applied my alkali-dispersible sizing to but one surface of my disintegrable backing, it will be apparent that the same or a different coating composition may be applied to the reverse side if desired. Such a construction offers certain advantages, e. g. it provides a still higher degree of water-resistance, but the increased cost is not justified for many applications.

The presence of aluminum powder in the surface sizing composition has been found to increase the rate of disintegration of my novel label structure, as mentioned hereinbefore. Additional advantages are to be obtained by adding a small percentage of aluminum to the label adhesive, whether of the water-soluble or alkali-soluble and water-insoluble variety. In the case of the water-soluble starch base adhesives commonly employed for labeling, the presence of aluminum flake has been found to increase the water-resistance of the dried adhesive film. At the same time, it increases the rate at which the film is attacked and removed in caustic.

I provide by means of my invention a novel and useful disintegrable label. My new label retains its original appearance even after prolonged contact with cold or room-temperature water in an ice chest, yet is rapidly and completely disintegrated during the cleansing process in the bottle-washer. The resulting pulp is easily rinsed from the bottle and from the washer. Containers such as beverage bottle, carrying my improved labels, retain their identity under active conditioning and storage, yet are easily and completely freed of all traces of the labels when run through the conventional bottle-washing processes.

What I claim is:

1. A washable container carrying and adherently attached label and being substantially unaffected in appearance by active conditioning and storage in an ice-chest, said label being capable of rapid removal and complete disintegration on washing of said container with warm dilute caustic, and said label comprising a low-wet-strength fibrous base sheet and a water-resistant printable surface coating, said base sheet comprising a mixture of aluminum particles and lightly bonded fibers, and said coating comprising a binder and a decorative pigment, at least one of said binder and pigment being rendered soluble on contact with warm dilute caustic.

2. A refillable bottle carrying an adherently attached label and being substantially unaffected in appearance by active conditioning and storage in an ice-chest, said label being capable of rapid removal and complete disintegration on washing of said bottle in warm dilute caustic, and said label comprising a low-wet-strength fibrous base sheet and a water-resistant printable surface coating, said base sheet comprising a mixture of lightly hydrated paper-making fibers and aluminum flake, and said coating comprising an alkali-soluble, water-insoluble film-forming binder and a decorative pigment, said pigment comprising aluminum flake.

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