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SAMPLING OF CRUDE TALL OIL

Object

This sampling procedure relates to a sample, either single or one of a set, that, while being suitable for testing, still constitutes an average sample as regards composition and properties of the lot of crude tall oil from which it has been drawn.

Crude tall oil is often a heterogeneous mixture of two liquid phases, tall oil and water, and a solid phase, crystallized rosin. All three phases may contain flocculated lignin, which prevents their separation but allows the formation of two or more layers. Special care is therefore indicated in the sampling procedure.

Definitions

A *point sample* is any small sample drawn from a lot of crude tall oil. Such a sample cannot be expected to be representative of the lot as regards the composition; it can only indicate the characteristics of the product in the vicinity of the sampling point. By mixing the oil lot before a point sample is drawn, the volume that the sample may be expected to be representative of will be greatly increased. It is, however, not easy to check the effect of mixing; moreover, mixing alone will not ensure that a point sample is a true *average sample*. To take into account the strong tendency for settling in crude tall oil, it is therefore necessary to draw *layer samples*, each representative of a liquid or congealed layer of the lot (in a large tank for instance).

A *surface layer sample* is one drawn at a level of one tenth of the depth below the surface.

A *middle layer sample* is one drawn at a level of one-half the depth below the surface.

A *bottom layer sample* is one drawn at a level of nine tenths of the depth below the surface.

A *foots sample* is one drawn at the lowest point of the tank i.e. from the bottom valve.

Several point samples or layer samples may be combined in specified proportions to form a *composite sample*. The foots sample, which often consists mainly of water, shall not be included in the composite sample.

An *average sample* is a sample comprising 200 ml—1000 ml, prepared by stirring a composite sample thoroughly and then dividing it into smaller portions, one of which can be tested immediately, while the others are stored for use as references. The lot to be sampled may be either a *drum lot*, consisting of several drums (small containers), or a *tank lot* in one or more tanks (each of which shall be sampled separately), e.g. tank cars or ships' tanks. The sampling equipment and procedure will be different in the two cases.

A. SAMPLING A DRUM LOT

Principle

A specified number of drums are chosen at random from the lot, and their contents heated gently and stirred thoroughly; from each one a point sample is drawn. The point samples are combined to provide a composite sample.

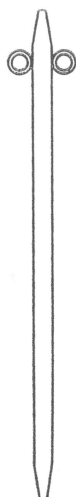


Figure 1. Sampling tube.

Apparatus

1. Heating device. A suitable method is to cover the drums with a tarpaulin to form a «tent» into which hot low pressure steam is led. The surface temperature of the drum shall not exceed 100°C.
2. Mixer, for mixing the contents in the drum; e.g. two motor driven rolls carrying the container.
3. Sampling tube (Figure 1), consisting of a tube with a conical constriction at each end, and with a diameter small enough for it to pass the bung opening of the container. To use, first close the upper opening air-tight with the thumb, lower the tube into the oil and remove the thumb from the upper end to allow oil to enter through the lower end. Close the upper end again with the thumb and transfer the sample so obtained to a sample bottle.
4. Mixing equipment, suitable for stirring a ca. 5 l composite sample.
5. Sample bottles of clear glass, 200, 500 or 1000 ml, with wide mouths that can be tightly closed with cork stoppers or plastic caps. If more than 20 point samples are to be drawn, smaller bottles of 50 or 100 ml may be required.

Procedure

If the number of drums (or other small containers) is:

- (a) 4 or less, sample all of them
- (b) 5—20, sample 4 of them
- (c) 21—100, sample 20 % of the drums but not less than 5
- (d) 101 or more, sample 10 % of them but not less than 20 and use 50 or 100 ml bottles.

The drums to be sampled should be drawn at random — for instance when the lot is being unloaded.

Heat the drums to 40°C—60°C and mix the contents of each drum. Immediately after mixing and while the drums are still warm open each one carefully and take a point sample with the sampling tube. The pressure in a hot drum may be considerable. To open a drum place it so that the bung is upwards; at first unscrew a few threads to release the

pressure and then continue unscrewing so that the bung can be opened without splashing.

Check by visual inspection whether a water layer has formed in any point sample. If any sample should contain more than ca. 5 % of water, draw point samples from a larger number of the drums to ascertain whether any of them contains excessive amounts of water. If this proves to be the case the water content of the lot cannot be calculated from that of the point samples. To get a better estimate stand any drums that apparently contain more than 5 % of water in an inclined position for a few hours after heating. Draw off the settled water either through the small bung near the edge of some drum bottom or by means of a siphon or hand pump. Measure and record the volume of the water removed (Note 1).

When all the point samples required have been drawn and any excess water has been separated and accounted for by the procedure as described above, combine them without delay in amounts approximately proportional to the quantities in the drums. Then stir the composite sample thoroughly with a mechanical mixer until there is no visible sign of settling. Distribute the mixed sample immediately among the required number of bottles to give average samples. Close the bottles tightly and label them (Note 2).

B. SAMPLING A TANK LOT

Principle

If heating equipment is available, a tank with crude tall oil is heated to 40°C—60°C. The oil is mixed as thoroughly as possible and a specified number of layer samples are drawn by means of special tank-sampling equipment. The layer samples are combined to form a composite sample.

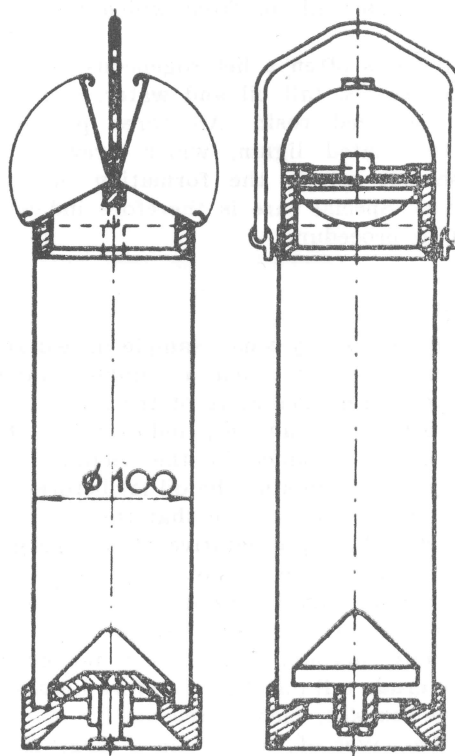


Figure 2. Liquid sampler for deep tanks.

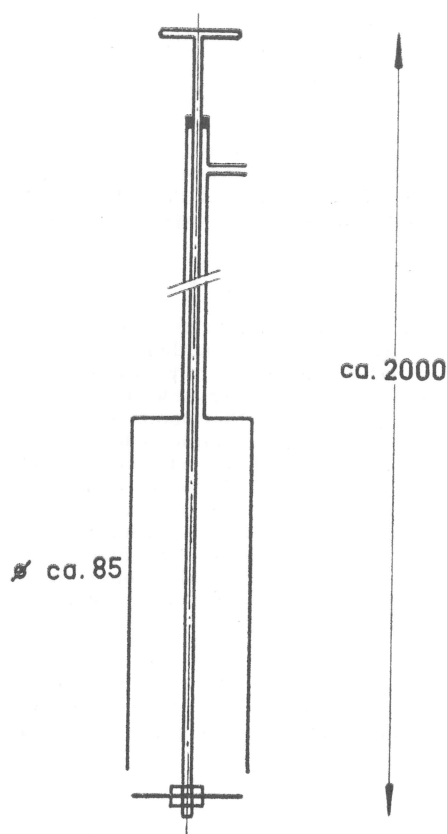


Figure 3. Semi-liquid sampler in principle.

Apparatus

1. Liquid sampler (Note 3) for deep tanks (ships' tanks) that can be heated and stirred continuously during storage and transportation. The sampler (Figure 2) is in the form of a cylindrical stainless steel vessel with a capacity of ca. 1 litre, attached to a long chain. In the bottom of the vessel is a self-opening valve, and at the top are two hinged lids. On lowering the sampler into the liquid, the valve and lids open, and when it is moved upwards they close and trap a sample.
2. Semi-liquid sampler (Note 4) for tanks containing settled and semi-solid rosin sludge at the bottom (unheated railway or trucks tanks). The sampler (Figure 3) can be lowered into the crude tall oil by means of a thin-walled steel tube, which serves as a hollow shaft and a guide for a steel rod for opening and closing the

bottom of the sampler. It can be operated from the top.

3. Sampling pails, 2 to 3 l, of stainless steel, and wide enough to allow a sampler to be lowered into them and emptied.
4. Mixing equipment, suitable for stirring a ca. 5 l composite sample.
5. Sample bottles of clear glass, 200, 500 or 1000 ml, with wide mouths that can be tightly closed with cork stoppers or plastic caps.

Procedure

Sample each tank separately. If the contents have not been mixed for some time, first draw a foots sample through the bottom valve. If this consists mainly of water, run off the water through the bottom valve until oil or rosin appears. Measure and record the volume of water removed (Note 1). If no water appears or when it has been removed, and if it is possible, heat the tank to 40°C—60°C, for instance by means of steam coils, and mix the contents of the tank. Immediately after mixing, draw at least 3 layer samples (surface, middle and bottom layer samples) from the top of the tank. Lower the sampler near the middle of the tank. In the case of very deep tanks increase the number of layer samples to be drawn so that at least every third metre of liquid is sampled. If no heating or mixing has been performed, draw samples at 1-metre intervals.

Check the samples, and especially the bottom layer sample, for settled water. If this exceeds ca. 1 % of the volume of the sample, draw a foots sample from the bottom valve. If this consists mainly of water, run off the water from the tank and measure and record its volume (Note 1). If the water does not reach the level of the bottom layer (nine tenths of the depth below the surface), the water in the layer samples shall not be removed. By the following procedure combine the layer samples in amounts proportional to the quantity of crude tall oil in the respective layer to form a composite sample (see also Table 1).

If the tank is in the form of a vertical cylinder (a common type of storage tank) or otherwise has a constant horizontal cross-section, and if the number of layer samples is at least 5, take equal portions of each.

If the tank is in the form of a horizontal cylinder or if the number of samples is less than 5, take a greater quantity from the middle layer sample or

Table 1.

| Number of layer samples | Parts by volume of layer samples to be combined | | | | | |
|-------------------------------|---|---|---|----------------------------------|---|---|
| | Constant cross-section tank | | | Full horizontal cylindrical tank | | |
| | 5 | 4 | 3 | 5 | 4 | 3 |
| 10 % below surface | 1 | 2 | 1 | 1 | 1 | 1 |
| upper middle | 1 | 3 | | 2 | 4 | |
| middle | 1 | | 3 | 4 | | 8 |
| lower middle | 1 | 3 | | 2 | 4 | |
| 10 % above bottom | 1 | 2 | 1 | 1 | 1 | 1 |

samples than from the surface and bottom layer samples. The cross-section of some ships' tanks, though irregular, may be considered to be regular for the purpose of this sampling procedure.

Stir the composite sample thoroughly until there are no visible signs of settling and divide it into the required number of average samples. Close the sample bottles tightly and label them (Note 2).

Report

Report the sampling procedure and observations. The sampling report shall include the labelling data, the origin of the crude tall oil (name of producer), identification of storage and means of transport for the oil lot, and the time of arrival and of sampling.

Report also the amount of water removed from the oil lot at different stages of the sampling procedure. If water has been removed from any layer sample this must be clearly indicated to ensure that the above instructions have been followed.

The sampling report may be combined with a report on the *quantity* of a crude tall oil lot (Note 3).

Additional information

This method is essentially an adaption of several mineral oil sampling methods, ASTM D 270-65 (1), DIN 51750 (2) and SIS 02 02 02 (4).

Note 1

Unless otherwise expressly stated by trade agreement, the salable quantity of a tall oil lot containing more than 1.5 % of water is taken as the tall oil present after

reduction of the water content and augmented by 1 % of the total quantity, this being the permissible fraction of water.

Note 2

Some trade agreements require that the samples be sealed by a specially authorized person or public official as a guarantee of the identity of the samples. The same applies to endorsement of the report on the sampling procedure.

Note 3

A liquid sampler for deep tanks described in DIN 51750, is commercially available. Another type, also commercially available and similar in principle but less convenient to use, is the flushing case sampler (3). This sampler is equipped with a thermometer to indicate the temperature of the trapped sample (which remains constant until the sampler has been removed from the tank), and is therefore suitable when the procedure includes determination of the *quantity* of oil, which involves measuring the volume — known as an *ullage* measurement, density (SCAN-T 2) and temperature. Both types of samplers are small enough to enter the special ullage openings on the hatches of ships' tanks.

Note 4

The semi-liquid sampler has been used for many years for sampling crude tall oil in tank cars. A similar sampler, a «Verschluss-Stechheber», is described in DIN 51750.

Literature

1. American Society for Testing and Materials, 1967 Book of ASTM Standards, part 18, p. 39.
2. Deutscher Normenausschuss, DIN 51750, 1963.
3. American Society for Testing and Materials, 1966 Book of ASTM Standards, part 18, p. 202.
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