

**OIL SLUDGE OF HYDRATION**

**TECHNICAL SPECIFICATIONS**

**Technical Specifications**

**9147-005-55514306-2003**

## 1. RANGE OF APPLICATION

These technical specifications refer to oil sludge of hydration (hydro oil sludge), which is generated through hydration or physical refining of different kinds of vegetable oils at oil processing manufactures.

Hydration of unrefined oil takes place at certain temperature with addition of citric acid and alkali therefore there is hydrated oil and hydro oil sludge is generated.

Oil sludge of hydration is a complex system which consists of neutral fats, phosphorus-containing compounds and other non-fatty impurities.

Oil sludge of hydration is used in soap industry and for other technical purposes. Requirements of these technical specifications are obligatory.

Identification code while ordering: Oil sludge of hydration under technical specifications 9147-005-55514306-2003.

## 2. REQUIREMENTS TO QUALITY AND SAFETY

Oil sludge of hydration should meet the requirements of these technical specifications approved in accordance with the established procedure.

Oil sludge of hydration is generated through processing of various kinds of unrefined vegetable oils in accordance with State Standards.

As for organoleptical and for physicochemical parameters oil sludge of hydration should meet the requirements specified in table 1.

Table 1

Item number	Parameter name	Characteristic of oil sludge of hydration
1	Physical form	from liquid to the salve-like
2	Smell	specific, sharp
3	Color	from light brown to dark brown
4	Mass fraction of crude fat (ether-soluble substances), %	25-60
5	Mass fraction of moisture and volatile substances, %	40-75

Oil sludge of hydration is a complex system because of multiplicity of generating components, heterogeneity of their properties and their ability to interact among themselves in the course of storage. Therefore it is recommended to take samples from the stream directly at the moment of shipment to the truck tank.

It is not allowed to add all kinds of other fats to oil sludge of hydration. Occurrence of extraneous fatty products is not allowed.

### **3. MARKING**

Marking of shipping containers should be made in accordance with State Standard 14192-92 with marking additional notations which characterize production:

- the trade mark or the name of the enterprise of the manufacturer, its site;
- the product name;
- net mass;
- quantity of packing units;
- the shipment date;
- numbers of these technical specifications;

While shipping oil sludge of hydration in rail tank cars and truck tanks the marking, which characterize production, is specified in the accompanying quality documents.

### **4. PACKAGING**

4.1. Oil sludge of hydration should be shipped to the consumer in rail tank cars with the bottom discharge in accordance with State Standard 10674 and in truck tanks with tightly closed hatches in accordance with State Standard 9218, in flasks in accordance with State Standard 5037, in barrels in accordance with State Standard 17366.

### **5. ACCEPTANCE PROCEDURES**

5.1. Oil sludge of hydration is shown to delivery by lots. The lot is any quantity of oil sludge of hydration received after oil processing, prepared for single delivery-acceptance and issued with one certificate of quality. When oil sludge of hydration is shipped in rail tank cars, the lot means every tank car.

5.2. While shipping and receiving oil sludge of hydration in the liquid state in rail tank cars and tank trucks, samples are taken from every truck by intersection of the jet in the process of pumping-over of oil sludge of hydration.

For continuous sampling while pumping-over oil sludge of hydration with the pump, the sampling cock is positioned in the vertical part of the pipe, the nipple neck is screwed on the cock, the calibration orifice of which provides the continuous jet of oil sludge of hydration when the cock is open completely and excludes any correction of the jet.

The sample is positioned in the dry pure tank which contents is the average sample representing this lot of oil sludge of hydration.

5.3. Acceptance tests are carried out in the sample of oil sludge of hydration in amount of 0.05 % from mass of the shipped oil sludge of hydration but not less than 4 liters. 3 samples are taken from the average sample not less than 0.2 liter each.

One sample is sent for analysis and the remained two are sealed up. Corresponding labels are glued on jars (bottles) with samples. The label specifies:

- the manufacturer's name;
- the enterprise-addressee's name;
- the product name;
- the number of the lot and the generation date;
- the date of the sample collection;
- the post, surnames and signatures of people who take sampling.

One sealed up sample is stored within a month in the densely covered tare in the event of evaluation disagreements of the product quality (arbitration sample) and the other intended for the consumer is send to him/her on demand as in view of instability of the generated system (hydro oil sludge), the control sampling made in the course of loading of the tank, gives the chance to estimate qualitative characteristics of oil sludge of hydration most objectively.

5.4. If there is an unsatisfactory result regarding at least one of parameters, the test of the doubled sample from the same lot is performed. Results of retests are final and applied to the whole lot.

5.5. The certificate of quality should specify:

- the manufacturer's name,
- the site of the enterprise, its trade mark;
- the product name;
- the mass of the lot;
- quality parameters;
- the number of the lot and the shipment date;
- the number of these technical specifications.

## 6. TESTING METHOD

6.1. Physical form, smell and color of oil sludge of hydration are tested organoleptical and should meet the requirements specified in table 1.

### 6.2. Testing of mass fraction of crude fat

6.2.1. Apparatus and reagents:

- Soxhlet apparatus;
- a drying electrical cabinet;
- analytical scales of the 2nd class and technical in accordance with State Standard 24104-88;
- a beaker for 100 ml, a conical flask for 250 ml, a funnel, a condenser, exiccator, water bath, porcelain cups, clock glass, glass rods, everything in accordance with State Standard 23923;
- filter paper in accordance with State Standard 12026;
- river sand treated by hydrochloric acid, calcined and sifted through a sieve with meshes of 1 mm;
- ethyl ether, dried up over just calcined calcium chloride and distilled over at the temperature 34-36<sup>0</sup>C;
- absorbent cotton in accordance with State Standard 5556, defattened with diethyl ether.

*Testing preparation.*

The sample of oil sludge of hydration is carefully mixed in a glass container in order to receive homogeneous consistence.

*Test operation.* Extraction method (arbitration method).

3-4 g of the washed out and calcined sand are placed in a porcelain cup, then it is weighed with the sand and a glass rod on the analytical scales. Then 4-5 g of oil sludge of hydration are weighed to the cup and carefully mixed with the sand.

If the hydration sediment is very wet, the batch weight with the sand is dried before extraction within 30 minutes at 100-105<sup>0</sup>C. Then the mixture of oil sludge of hydration with the sand is carried over lost-free to an extraction cartridge. The surface of the cup and of the glass rod is wiped 2-3 times with scraps of the defattened cotton wetted with ether and placed in the cartridge. While wiping the rod, cotton scraps should be held with forceps.

A small layer of pure cotton is placed in the top part of the cartridge, the edges of the cartridge are turn and it is placed in the extractor of the Soxhlet apparatus. The extractor is joined with the dried and weighted receiving flask and solvent is poured into it in such quantity in order to overflow it through a siphon tube into the flask. Then a little more surplus of the solvent is added and the extractor is joined with the condenser fixed in the support stand. The mounted device is put on the hot water bath. The extraction end is tested by the absence of oil in the solvent which is in the extractor of the apparatus.

The extractor is separated from the condenser to test the end of extraction, then it is inclined towards the siphon tube and the whole solvent is poured from the extractor into the flask through the siphon tube, the last drops of the solvent are put on the dry pure glass. If after evaporation of the solvent there are no oil traces on the surface of the clock glass, then it means that extraction is finished.

Otherwise the device is mounted again and extraction is continued until final extraction of oil.

Duration of extraction of hydration sediment in the Soxhlet apparatus is 3-4 hours with 7-8 siphonages per hour. After extraction is finished, the device is unloaded and the solvent is distilled from the flask with micelle. The flask with fat is dried in the drying cabinet at the temperature 100-105° C to receive the constant weight. Within drying time the first weighing is done in an hour, the following ones are in half an hour.

*Analysis of results.*

Mass fraction of crude fat in oil sludge of hydration (X%) is calculated under the formula:

Where  $P_1$  - weight of fat extracted from oil sludge of hydration, in g

P - the batch weight of oil sludge of hydration, taken for analysis, in g

The final result is the arithmetic average of two parallel tests. Allowable discrepancy between two parallel tests should not exceed 0.5 %.

*6.2.2. Test operation by accelerated method.*

5g of the batch weight of oil sludge of hydration accurate within 0.01 g is placed in the beaker for 100 ml and 25 ml of ether is added, then the mixture is carefully stirred with a glass rod and let to settle.

The top layer is poured along the rod through the dried filter into the weighted conical flask for 250 ml. Ether extraction in the beaker is done until ether extracts are colorless. Then glass contents is moved quantitatively to the flask with ground-in microsection, the beaker and the filter are washed out to complete defatting.

The condenser is inserted into the flask with the filtered ethereal solution of the fat and it is placed on the water bath when the temperature of the bath is about 60°C and the ether is completely distilled. Then the flask with fat is dried in the drying cabinet at the temperature 100-105° C to receive the constant weight. Within drying time the first weighing is done in an hour, the following ones are in half an hour.

*Analysis of results.*

Mass fraction of crude fat in oil sludge of hydration (X%) is calculated under the formula:

$$X = P_1 \times 100/P$$

Where  $P_1$  - mass of fat extracted from oil sludge of hydration, in g

P - the batch weight of oil sludge of hydration, taken for analysis, in g

The final result is the arithmetic average of two parallel tests. Allowable discrepancy between two parallel tests should not exceed 0.5 %.

### **6.3. Testing of moisture and volatile substances in accordance with State Standard 11812-66.**

*Apparatus and reagents:*

- a drying electrical cabinet;
- analytical scales of the 2nd class and technical in accordance with State Standard 24104-88;
- chemical beakers for 100 ml, aluminium weighing bottles, exiccator.

*Testing preparation.*

The sample of oil sludge of hydration is carefully mixed in a glass container in order to receive homogeneous consistence.

*Test operation.*

About 3-4 g of the batch weight of oil sludge of hydration is weighed on the analytical scales to the weighted in advance glass or aluminium weighing bottle with 2-3 g of sand. The batch weight of oil sludge of hydration is dried to receive the constant weight at the temperature 105° C, periodically stirring it with the glass rod which is weighed together with the weighing bottle. Within drying time the first weighing is done in 2 hours, the following ones are in an hour.

*Analysis of results.*

Mass fraction of moisture and volatile substances in oil sludge of hydration (X%) is calculated under the formula:

$$X = (m_1 - m_2) \times 100/m$$

Where m - the batch weight of oil sludge of hydration in g.

m<sub>1</sub> - mass of the weighing bottle with oil sludge of hydration before drying in g.

m<sub>2</sub> - mass of the weighing bottle with oil sludge of hydration after drying in g.

The final result is the arithmetic average of two parallel tests. Allowable discrepancy between two parallel tests should not exceed 0.04 %.

#### **Accelerated method.**

About 3-4 g of the batch weight of oil sludge of hydration is weighed on the analytical scales to the weighted in advance glass or aluminium weighing bottle with 2-3 g of sand and glass rod. The batch weight of oil sludge of hydration is dried on the one-hotplate electric stove with constant careful stirring with the glass rod. Thereat spitting is not allowed.

The end of evaporation of moisture is tested if there is no weeping of the clock glass being over weighing bottle.

*Analysis of results.*

Mass fraction of moisture and volatile substances in oil sludge of hydration (X%) is calculated under the formula:

$$X = (m_1 - m_2) \times 100/m$$

Where m - the batch weight of oil sludge of hydration in g.

$m_1$  - mass of the weighing bottle with oil sludge of hydration before drying in g.

$m_2$  - mass of the weighing bottle with oil sludge of hydration after drying in g.

The final result is the arithmetic average of two parallel tests. Allowable discrepancy between two parallel tests should not exceed 0.04 %.

## **7. TRANSPORTATION AND STORAGE RULES.**

7.1. Oil sludge of hydration can be transported in rail tank cars in accordance with State Standard 10674 and in truck tanks in accordance with State Standard 9218 according to "Cargo Transportation Rule" valid for this type of transport as well as by open-top vehicles in steel barrels with detachable upper bottom in accordance with State Standard 13950 and in metal containers in accordance with State Standard 15102.

7.2. Rail tank cars and truck tanks are examined. There should not be water, dirt, foreign substances in rail tank cars and truck tanks.

7.3. While transporting by open-top vehicles, barrels and flasks should be protected from atmospheric precipitation.

## **8. MANUFACTURER'S WARRANTIES**

8.1. The manufacturer guarantees conformity of oil sludge of hydration to requirements of these technical specifications under the compliance of conditions of transportation and storage prescribed by the technical specifications.

8.2. In the course of storage of oil sludge of hydration there is possibility that its color and smell are changed.

8.3. Warranty period of storage is 2 months.