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IMPROVEMENT OF WATER SOLUBLE TEST FOR HNS

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DEVELOPMENT DIVISION

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IMPROVEMENT OF WATER SOLUBLE TEST FOR HNS

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ABSTRACT

The analytical procedure now utilized for the determination of the water-soluble material content in HNS is cumbersome and time consuming. This procedure, if possible, should be supplanted by an equivalent but less involved method.

DISCUSSION

The material specifications for HNS I and HNS II requires testing of samples, selected at random, from each inspection lot. Among the tests required are those for determining (1) the surface moisture and volatiles, with a maximum allowable content of 0.05 weight percent, and (2) the water-soluble material content, wherein a maximum of 0.03 weight percent is established.

The test procedure, as specified for the determination of the water-soluble content, includes introduction of an accurately weighed sample, of about 10 grams of HNS, directly into a tared 100 to 150 mg fine porosity filter crucible. The sample is washed by adding 80 mg of water containing a 5-volume percent of methyl alcohol to the crucible and stirring the contents, with a previously weighed stirring rod, to

aid in wetting the sample and breaking up lumps. The HNS is allowed to remain in contact with the wash solvent for 20 minutes, then the crucible is positioned over a filter flask and the contents filtered by application of vacuum. The sequence is repeated two additional times.

The crucible and its contents along with the stirring rod are subsequently dried at 100 to 105 C for four hours, then placed in a dessicator and allowed to cool before reweighing. The loss in weight of the HNS is reported as water soluble. On a 10 g sample the maximum water soluble content is set at 0.003 g.

Erroneous water soluble content values can be reported as a result of (1) high sample surface moisture, or (2) material loss during handling. On a 10 g HNS sample the maximum permissible loss due to surface moisture and volatiles is 0.005 g.

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If this loss is attributed entirely to moisture, this loss will be considered as water-soluble content and it will exceed the maximum allowable water-soluble content (0.003 g). Although utmost care and diligence are exercised during the sample processing, sample loss equal to or in excess of the maximum allowable loss may result from accidental spattering. If the spattering is noticed the test can be repeated. If it goes unobserved and the weight loss is high it leads to consternation.

Considerable thought has been given to the feasibility of developing an equivalent test procedure to supplant the current testing technique.

Increasing the mass of the test sample to 30 or 40 g was considered, but found impractical. This would entail the use of a 350 to 400 ml fine porosity filter crucible since the solvent volume per wash must also be increased. This proves impractical since the combined weight of the crucible and the HNS exceed the load capacity (200 g) of the analytical balance.

Determination of the water soluble content from the filtrate was proposed as a logical approach. This would involve combining all three filtrates from each respective wash. If the filtrate is homogeneous, evaporation to dryness of an accurately measured volume, in a tared distillation flask, should yield a representative residue in g/ml. The solvent removal can be accelerated by distillation of the major portion of the fluid followed by slow evaporation to dryness of the balance.

If one assumes a water soluble content of 0.003 g from a 10 g sample of HNS, the solute in the combined filtrates (240 ml) would be $1.25 \times$

10^{-5} g/ml. Subjecting a 100 ml filtrate volume to the distillation and evaporation to dryness treatment would yield 1.25×10^{-3} g of residue from which the total soluble content can be calculated.

Nine 10 g samples of HNS (Chemtronics, Lot 105-48), of known high water soluble content, were washed per the existing test procedure (SOP 6-1725). The three 80 ml/wash filtrates from each respective sample were combined for subsequent distillation and evaporation to dryness of 100 ml volumes. The washed HNS samples were dried and the loss (weight percent) for each sample was determined (Table I).

Identical volumes (100 ml) of each filtrate were distilled and evaporated to dryness in previously weighed distillation flasks. The distillation, over an oil bath, was discontinued after most of the filtrate had been removed. The balance was slowly evaporated to dryness. The exterior of each flask was thoroughly cleaned and subsequently dried in an oven at 100 to 105 C for 30 minutes.

After cooling in a dessicator the flasks were reweighed. Any gain in weight of the flasks was attributed to residue from the water solubles. From the weight of the residues (g/100 ml filtrate) the water soluble material content in the 10 g HNS samples was determined (Table I).

The water soluble material content (wt. %) in HNS as determined by the two analytical techniques are listed in columns 3 and 6, Table I. Examination of these columns reveals that the values established per the procedure outlined in SOP 6-1725 are higher by a factor of two than the corresponding values determined from the filtrate-residues.

Table I. HNS I, Chemtronics Lot 105-48; Water Soluble Content

Sample	Analysis					
	SOP 6-1725			Filtrate Residue		
	Mass (g)	Weight Loss (g)	Loss Weight (%)	Residue (g/100 ml)	Residue Calculated (g/240 ml)	Loss Weight (%)
1	10.3366	0.0106	0.10	-	-	-
2	10.0724	0.0136	0.14	-	-	-
3	10.0056	0.0099	0.10	-	-	-
4	10.0312	0.0103	0.13	0.0014	0.0034	0.03
5	10.0116	0.0126	0.13	0.0070	0.0168	0.17
6	10.0129	0.0101	0.10	0.0030	0.0072	0.07
7	10.0090	-	-	0.0019	0.0046	0.05
8	10.0142	0.0121	0.12	0.0018	0.0043	0.04
9	9.9981	0.0134	0.13	0.0016	0.0038	0.04
10	10.0001	-	-	0.0033	0.0079	0.08
11	10.0001	-	-	0.0025	0.0060	0.06
12	9.9998	-	-	0.0027	0.0065	0.04
Mean ($\bar{X}$)			0.115			0.064
Std. Dev. (σ)			0.02			0.04

The HNS involved in this endeavor was evaluated for "surface moisture and volatiles" content. This value was established at 0.015 weight percent. Column 3, Table I, reveals a "water soluble material" content of 0.115 weight percent (mean). This implies that only a minor portion of the water soluble content can be attributed to surface moisture.

The water soluble material values listed in column 6, Table I, were determined from the filtrate residues (100 ml aliquot/sample). These values should correspond with the values in column 3 but are found to be only half the value. One can assume that approximately one-half

of the water soluble content is material with a low vapor pressure and is consequently extracted during the filtrate reduction through distillation.

The discrepancies in the test values above indicate that the content of water soluble materials in HNS cannot be determined quantitatively from the residue obtained through distillation and evaporation of the waste filtrates.

No equivalent test, to supplant the procedure now employed in establishing the water soluble content in HNS, has been developed and none can be proposed at this time.