Aggregate Physical Properties of Wastes – Melting Point and Specific Gravity

1. SCOPE AND APPLICATION

This SOP describes procedures for the determination of the melting point of solids and the specific gravity of liquids. These tests are generally used for the characterization of solid or liquid wastes. Pure (organic) solids and liquids can frequently be characterized by their melting point or specific gravity. The applicable temperature range for the melting point is 30 to 300 °C. Most organic liquids are lighter than water; the applicable range for specific gravity is 0 to 1. The tests covered by this SOP are S-MELT-PT and WS-SP-GRAV. Both melting point and specific gravity are intrinsic physical properties of solids and liquids respectively. Their values are independent of the amount or volume of the sample. Both measurements provide presumptive information, rather than conclusive information about the identity of unknowns.

2. SUMMARY OF METHODS

2.1 Melting Point: The sample is heated at 0.2 to 5.0 °C/min. to a maximum of 300 °C in order to determine the melting point, or the temperature at which the substance passes from the solid state to a liquid phase. During this transition, the optical properties of the substance change; this change is automatically detected by an optical sensor in the furnace. The furnace temperature is usually higher than the actual temperature of the sample. The melting point apparatus automatically corrects for this temperature lag.

2.2 Specific Gravity: The Specific Gravity of a substance is a dimensionless number that is defined as the ratio of the density of a material at a stated temperature to the density of water at that temperature. The density of the sample is calculated by measuring the weight of a known volume of sample. This is then compared to tabulated values of the density of water to calculate the specific gravity.

3. INTERFERENCES

3.1 Melting Point

3.1.1 Sample moisture may impede accurate measurement by the evolution of gas at various temperatures causing the sample to shift its position. Sample shifting may appear as a change in optical properties and give an erroneous melting point. When possible, the sample should be dried in a desiccator prior to melting point determinations.

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3.1.2 Samples that are dark or highly colored may not allow sufficient light transmission through the sample to detect a change during the solid to liquid transition. Violet, blue, and green samples usually cause the most problems.

3.1.3 Sample particulate size should be uniform when possible. Samples consisting of particles of varying sizes may yield differing results between replicates. Transition from solid to liquid may be more difficult for the instrument to detect with larger particles. Larger particles are also problematic when loading sample capillary tubes. To avoid particle size problems sample should be pulverized in agate mortar.

3.2 Specific Gravity

3.2.1 There are no significant interferences for the measurement of specific gravity.

4. APPARATUS AND EQUIPMENT

4.1 Instrumentation – Mettler Toledo FP62

4.2 Mettler ME-18552 Melting Point Capillary Tubes

4.3 Melting Point Tube Packing Rod (in the same container as the capillary tubes)

4.4 Analytical Balance accurate to 3 decimal places

4.5 Volumetric flask, Class A, 10 mL

4.6 Thermometer

5. REAGENTS AND CHEMICALS

5.1 Benzoic Acid Check Standard (MP 122.3 °C)

5.2 De-ionized water

6. SAMPLE ANALYSIS AND QUANTITATION

6.1. MELTING POINT

6.1.1 The Instrument should be switched on, with sample holder empty, 30 minutes prior to starting analysis to allow furnace temperature to equilibrate.

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6.1.2 Samples should be logged out of the receiving room and allowed to warm to room temperature.

6.1.3 After preparing the samples as directed in section 3, samples and QC are loaded into sample tubes to a height of ≈ 4cm.

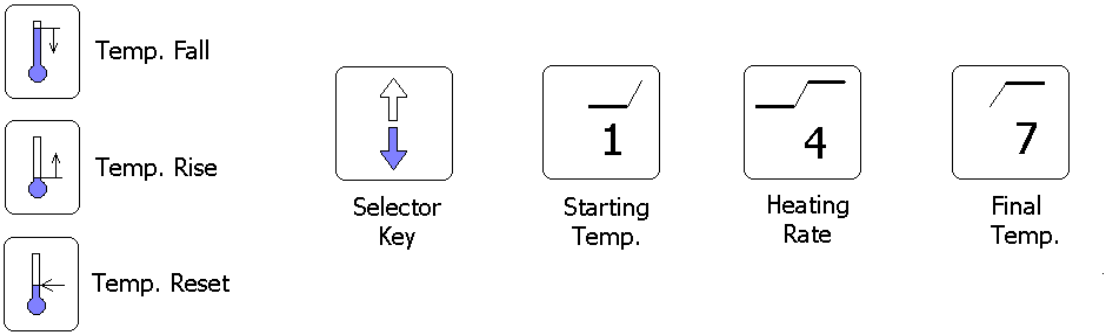


Figure 1 - Instrument Setup Keys

Note: Some instrument control buttons are only marked by icons or pictograms; descriptions of these buttons can be found in Figure 1 and in the FP62 manual on pp. 7-8.

6.1.4. Turn the instrument on. Set the starting temperature by pressing the selector key followed by the starting temperature key [#1]. Enter the desired starting temperature and press the enter key [E]. The appearance of the keys is shown in Figure 1 above.

6.1.5. Next, set the heating rate by pressing the selector key followed by the heating rate key [#4]. Enter the desired heating rate and press enter [E]. Note: Increasing the rate of heating results in decreasing the accuracy of the measurement. Samples may first be scanned at 10 °C/min to obtain a rough estimate of the melting point range; the actual determination should be done at a heating rate of 0.2 °C/min for accurate measurements.

6.1.6. Set the final temperature by pressing the selector key followed by the final temperature key [#7]. Enter the desired final temperature (usually 300 °C) and press enter [E].

6.1.7 Press the reset key so that furnace returns to the previously set starting temperature.

6.1.8 Insert sample tube into the furnace and press the temperature rise button to start melting point determination. The instrument will stop automatically when the melting point is reached or the furnace reaches 300 °C. The finished analysis light will blink and the melting point will be displayed until the enter button [E] is pressed. The instrument will automatically return to the correct starting

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temperature at which time the instrument ready light will come on. Proceed to the next sample.

6.2 SPECIFIC GRAVITY

6.2.1 If the sample is normally stored in a refrigerator, remove the sample to the lab where the measurement will be conducted and allow the sample to warm up to room temperature. This is important since the specific gravity measurement is temperature dependent. The volumetric flask should also be in the same lab at room temperature. Calibrate the balance and thermometer prior to use.

6.2.2 Record the equilibrium (room) temperature of the liquid sample T (°C) to 3 significant figures.

6.2.3 Place a clean, dry 10 mL volumetric flask on the balance and record its weight or tare if necessary.

6.2.4 Fill the flask with the sample to its mark and record its weight. Let $D_S(T)$ denote the density of the sample. The density is given by

$$D_S(T) = \frac{\text{Weight of Sample(g)}}{\text{Volume of Sample (ml)}} = \frac{\text{Weight of Sample (g)}}{10}$$

6.2.5 Look up the density of water $D_W(T)$ at the temperature of the sample (T) from Table 1 at the end of this document. The specific gravity of the sample is calculated from the formula shown below. Report the specific gravity to three significant figures.

$$\text{Specific Gravity} = \frac{D_S(T)}{D_W(T)}$$

7. DATA ARCHIVAL

7.1 The two analyses described in this SOP are seldom performed in the laboratory (generally no more than one or two events per year). Due to the presumptive nature of the information provided by these tests, samples requiring these two tests are almost always assigned complementary tests that provide more conclusive information, including flashpoint, and tests for organic parameters. The raw data for these tests are recorded in existing lab notebooks; the Infrared Data notebook for Melting Point and the Flashpoint data notebook for specific gravity respectively. This is done due to the locations of instruments devoted to these tests and the frequency of the analyses.

7.2 Enter the melting point results into the “Template_MeltingPoint.xls” spreadsheet located at \\tlhlab3\Nutrient\Wet_Chem\ and save the results as a tab-delimited text (.txt) file. The file should follow the following naming convention:

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YYYYMMDDmp1.txt where the first four characters represent the year, followed by the month and day of analysis. An example is "20080305mp1.txt". The saved text file is ready to be uploaded to the LIMS' QC Manager program.

7.3 Enter the specific gravity using the results editor in the LIMS with a descriptive comment.

8. QUALITY CONTROL

8.1. For the melting point test, a benzoic acid standard should be run at the beginning, end, and following each set of ten samples during the analysis. Standard recovery should be within three standard deviations of the reported confidence interval at 0.2 °C/min (± 0.5 °C × 3σ = 1.5 °C).

8.2. For the melting point test, a duplicate sample should be run for each batch of ten samples to monitor instrument precision. RPD acceptance will be set at ±20% until enough data can be acquired to set more appropriate limits.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Read the MSDS information on benzoic acid for safe handling instructions.

9.2. When handling unknown samples use extreme caution due to presence of potential hazards.

10. REFERENCES

10.1 Mettler Toledo FP62 Operating Instructions

APPENDIX OF SIGNIFICANT CHANGES

04/07/2008	This SOP has been modified to include an additional test for specific gravity. The SOP has also been renamed to reflect this addition. Changes have been made to all sections to include the new test.
03/30/2012	The SOP was renamed and reclassified as a general organic SOP. The name was changed from NU-065-1.9 to OG-002-1.0

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TABLE 1: Density of Water (g/mL) vs. Temperature (°C)
(from Handbook of Chemistry and Physics, 53rd Edition, 1972)

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	0.999841	0.999847	0.999854	0.999860	0.999866	0.999872	0.999878	0.999884	0.999889	0.999895
1	0.999900	0.999905	0.999909	0.999914	0.999918	0.999923	0.999927	0.999930	0.999934	0.999938
2	0.999941	0.999944	0.999947	0.999950	0.999953	0.999955	0.999958	0.999960	0.999962	0.999964
3	0.999965	0.999967	0.999968	0.999969	0.999970	0.999971	0.999972	0.999972	0.999973	0.999973
4	0.999973	0.999973	0.999973	0.999972	0.999972	0.999972	0.999970	0.999969	0.999968	0.999966
5	0.999965	0.999963	0.999961	0.999959	0.999957	0.999955	0.999952	0.999950	0.999947	0.999944
6	0.999941	0.999938	0.999935	0.999931	0.999927	0.999924	0.999920	0.999916	0.999911	0.999907
7	0.999902	0.999898	0.999893	0.999888	0.999883	0.999877	0.999872	0.999866	0.999861	0.999855
8	0.999849	0.999843	0.999837	0.999830	0.999824	0.999817	0.999810	0.999803	0.999796	0.999789
9	0.999781	0.999774	0.999766	0.999758	0.999751	0.999742	0.999734	0.999726	0.999717	0.999709
10	0.999700	0.999691	0.999682	0.999673	0.999664	0.999654	0.999645	0.999635	0.999625	0.999615
11	0.999605	0.999595	0.999585	0.999574	0.999564	0.999553	0.999542	0.999531	0.999520	0.999509
12	0.999498	0.999486	0.999475	0.999463	0.999451	0.999439	0.999427	0.999415	0.999402	0.999390
13	0.999377	0.999364	0.999352	0.999339	0.999326	0.999312	0.999299	0.999285	0.999272	0.999258
14	0.999244	0.999230	0.999216	0.999202	0.999188	0.999173	0.999159	0.999144	0.999129	0.999114
15	0.999099	0.999084	0.999069	0.999054	0.999038	0.999023	0.999007	0.998991	0.998975	0.998959
16	0.998943	0.998926	0.998910	0.998893	0.998877	0.998860	0.998843	0.998826	0.998809	0.998792
17	0.998774	0.998757	0.998739	0.998722	0.998704	0.998686	0.998668	0.998650	0.998632	0.998613
18	0.998595	0.998576	0.998558	0.998539	0.998520	0.998501	0.998482	0.998463	0.998444	0.998424
19	0.998405	0.998385	0.998365	0.998345	0.998325	0.998305	0.998285	0.998265	0.998244	0.998224
20	0.998203	0.998183	0.998162	0.998141	0.998120	0.998099	0.998078	0.998056	0.998035	0.998013
21	0.997992	0.997970	0.997948	0.997926	0.997904	0.997882	0.997860	0.997837	0.997815	0.997792
22	0.997770	0.997747	0.997724	0.997701	0.997678	0.997655	0.997632	0.997608	0.997585	0.997561
23	0.997538	0.997514	0.997490	0.997466	0.997442	0.997418	0.997394	0.997369	0.997345	0.997320
24	0.997296	0.997271	0.997246	0.997221	0.997196	0.997171	0.997146	0.997120	0.997095	0.997069
25	0.997044	0.997018	0.996992	0.996967	0.996941	0.996914	0.996888	0.996862	0.996836	0.996809
26	0.996783	0.996756	0.996729	0.996703	0.996676	0.996649	0.996621	0.996594	0.996567	0.996540
27	0.996512	0.996485	0.996457	0.996429	0.996401	0.996373	0.996345	0.996317	0.996289	0.996261
28	0.996232	0.996204	0.996175	0.996147	0.996118	0.996089	0.996060	0.996031	0.996002	0.995973
29	0.995944	0.995914	0.995885	0.995855	0.995826	0.995796	0.995766	0.995736	0.995706	0.995676
30	0.995646	0.995616	0.995586	0.995555	0.995525	0.995494	0.995464	0.995433	0.995402	0.995371

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