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# Plastics — Differential scanning calorimetry (DSC) —

# Part 8:
Determination of amount of absorbed water

*Plastiques — Analyse calorimétrique différentielle (DSC) —*

*Partie 8: Détermination de la quantité d'eau absorbée*



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11357-8 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 11357 consists of the following parts, under the general title *Plastics - Differential scanning calorimetry (DSC):*

— *Part 1: General principles*



— *Part 2: Determination of glass transition temperature*

— *Part 3: Determination of temperature and enthalpy of melting and crystallization*



— *Part 4: Determination of specific heat capacity*

— *Part 5: Determination of temperatures, times, heat of reaction and degree of conversion*

— *Part 6: Determination of oxidation induction time*

— *Part 7: Determination of crystallization kinetics*

— *Part 8: Determination of amount of water sorbed by polymers*

Annex A is for information only.

# Plastics — Differential scanning calorimetry (DSC) — Part 8: Determination of amount of absorbed water

## 1 Scope

This International Standard specifies the testing method for determination of amount of adsorbed water in polymers by differential scanning calorimetry in a dynamic mode.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11357. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11357 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.



ISO 472, *Plastics - Vocabulary*

ISO 11357-1:1996, *Plastics - Differential scanning calorimetry (DSC) - Part :1 General principles.*



ISO 11357-3:1998, *Plastics - Differential scanning calorimetry (DSC) - Part 3: Determination of temperature and enthalpy of melting and crystallization.*

## 3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 11357-1:1996, clause 3 and the following apply.

**3.1**
**Water content ($W_c$)**
mass of water in the polymer divided by the mass of the dry sample, expressed in $g \cdot g^{-1}$

**3.2**
**Free water content ($W_f$)**
grams of bulk water in gram of dry sample.

NOTE Bulk water is calculated from heat of first order transition: crystallization or melting

**3.3**
**Bound water content ($W_b$)**
water species exhibiting large differences in transition enthalpy and temperature or those for which no phase transition can be observed by calorimetric methods

**3.4**
**Non freezing water content ($W_{nf}$)**
water fractions closely associated with the polymer matrix where no crystallization exotherms or melting exotherms are observed

**3.5**
**Freezing bound water content ($W_{fb}$)**
water species whose melting/crystallization temperatures and enthalpies are not significantly different from those of normal water

## 4 Principle

Determination of the mass of water which is tightly restrained by the hydrophilic groups of polymers using enthalpy of melting and crystallization of water.

## 5 Apparatus

### 5.1 DSC apparatus

See ISO 11357-1:1996, clause 5.1

### 5.2 Pan



The sample pan shall be capable of being sealed hermetically. If alminium pans are to be used, they should first be placed in an autoclave with a small amount of pure water at 100 ˚C for 3-5 hours in order to eliminate the possibility of formation of alminum hydroxide in the pan during the measuring cycle.



### 5.3 Analytical balance



See ISO 11357-1:1996

## 6 Temperature calibration and validation of transition enthalpy of water

### 6.1 Temperature calibration

See ISO11357-1:1996

### 6.2 Enthalpy calibration

1) Weigh sealing type DSC sample pan including the lid (±0.01 mg)

($m_{sample\ pan}$)

2) Put 0.5-2.0 mg deionizedwater (±0.01 mg) in the sample pan

3) Seal hermetically and weigh the sealed pan

This is necessary to avoid errors due to evaporation of water

4) Place the sample pans with water in the centre of the sample holder

5) Cool the sample holder from room temperature to -70 °C at the rate 10 K·min-1

6) Maintain at -70 °C for 5 min.

7) Heat at 10 K·min-1 to 60 °C.

8) Calculate heat of crystallization and heat of melting

{Ref. DIS/11357-3:1998 (DSC part 3)}

9) Heat of crystallization and heat of melting should be 334 J·g-1

10) If the heat of melting is changed more than ±10 J·g-1, the apparatus should be calibrated.

NOTE Heat of crystallization is always smaller than that of heat of melting due to super cooling.

## 7 Test specimen

A test specimen shall be powder, pellet, flake, filament or film shape. The test specimen shall be prepared by cutting it into a size appropriate for the apparatus.

## 8 Procedure

The procedures of measurement are as follows:



### 8.1 Method 1 (One point method)

1) Weigh sealing type DSC sample pan including the lid (±0.01 mg) ($m_{\text{sample pan}}$);



2) Weigh 5-10 mg specimen (msample);

NOTE 1 Mass of dry samle is not determined in this stage.

3) Add 1-5 mg of pure water;

NOTE 2 Water content is approximate in this stage.

4) The sample pan is hermetically sealed and allowed to equilibrate for several days, preferably for one week to assure uniform hydration of the sample prior to testing. The equillibration time should be longer for hydrophobic polymers;

5) Weigh the sealed pan ($m_{\text{sample pan}} + m_{\text{sample}} + m_{\text{water}}$);

6) Place the test specimen in the centre of the sample holder;

7) Determine the enthalpy of the specimen under the same conditions as given in 6.2 and the same scan rate as used for calibration;

8) Record the DSC heating and cooling curves;

9) Remove the sample pans from the sample holder;

10) Pierce a hole on the lid of sample pan using a pin;

11) Anneal the sample pan in an oven at 120˚C for 30 min;

*NOTE 3* When the sample is thermaly unstable at the above conditions, temperatureand time should be changed.

12) Remove sample pans from an oven and weigh immediately ($m_{\text{sample pan}} + m_{\text{dry sample}}$);Calculate enthalpy ($q$) in Joule from the peak integration.

### 8.2 Method 2 (Extrapolation methods)

Prepare 5-7 specimen with various amounts of water. Procedure is the same as the method 1,

NOTE Water content is approximate in this stage.

## 9 Calculation of amount of bound water

### 9.1 Method 1 (One point measurement)

1) Calculate

$W_c = m_{water}/m_{sample}$ $(g \cdot g^{-1})$ (1)

$W_b = W_c - q/334/m_{\text{dry sample}}$ $(g \cdot g^{-1})$ (2)

2) If cooling and/or melting shows one peak,

$W_b = W_{nf}$ (3)

3) If two peaks are observed either or both heating and cooling curve, calculate each peak. (See Annex A)



### 9.2 Method 2 (Extrapolation methods)

1) Calculate $W_c$ and

$q/334/m_{\text{dry sample}}$ $(g \cdot g^{-1})$ (4)

2) Relationship between Wc and q/334/m dry sample shall be established

(Ref. Figure 1)

3) The extrapolated value is $W_b$

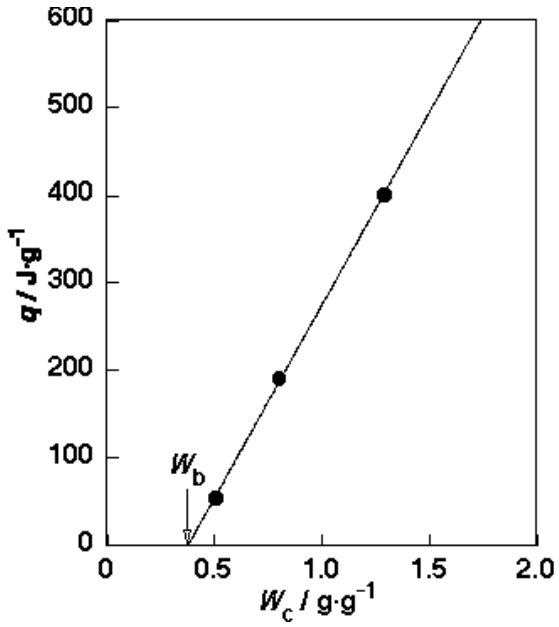


**Figure 1 — Relationship between water content and enthalpy of free water.**



## 10 Report



a) Reference to this International standard;

b) Date of the test;

c) Identification of the sample tested, including the thermal history;

d) Manufacturer, the model and type of DSC apparatus;

e) The materials, shape and dimensions of the pan and lid;

f) Materials used for enthalpy validation (water);

g) Heating and cooling rate;

h) Shape, dimensions and mass of the test specimen;

i) Sampling of the test specimen;

j) The test results including the water content (Wc, ) and the bound water content (Wb);

k) Other items required if any.