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Thermal Analysis

Compatibility of Calibration Substances with Various Crucible Materials

The table below corresponds to our most current information. No claim is made that it is exhaustive. The user should regard it only as a guide.
Because this is a compilation of data from the literature and from our own experience, it is not possible to cover all possible combinations of measurement conditions. In case of doubt, it is advisable to run preliminary tests in a separate furnace.

	Calibration Substance																				
Crucible Material	Cyclopentane	Water	Gallium	Indium	Tin	Lead	Zinc	Lithium Sulfate	Aluminum	Silver	Gold	Barium Carbonate[2)]	Potassium Perchlorate[2)]	Potassium Chromate[2)]	Silver Sulfate[2)]	Rubidium Nitrate[2)]	Nickel	Bismuth	Cyclohexane	Cesium Chloride[2)]	Mercury
Corundum (Al_2O_3)	−	−	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	−	+	+
Graphite	−	−	+	+	+	+	+	+	+	+	−	?	?	?	?	?	−	?	−	?	?
Fused Silica	+	+	+	+	+	+	+	+	−	+	+	−	?	?	?	?	−	?	?	?	+
Aluminum[1)]	+	+	−	+	+	+	+	+	−	−	−	−	+	−	+	+	−	+	+	+	+
Silver	+	+	−	−	−	−	−	?	−	−	−	?	?	?	?	?	−	−	?	?	−
Gold	+	+	!	!	−	−	−	+	−	−	−	?	?	?	?	?	−	−	?	?	−
Stainless Steel	+	+	!	+	!	+	−	?	−	+	−	?	?	?	?	?	−	+	?	+	+
Platinum	+	+	!	!	−	−	−	+	−	−	−	+	+	+	+	+	−	−	?	+	−
Tungsten	−	−	!	?	?	!	+	?	!	+	+	?	?	?	?	?	?	+	?	?	+

+ No solubility or effect on the melting temperature to be expected.

! Corrosion processes possible with negligible changes in the melting temperature.

− • Crucible melts.
- Melt or transformation product reacts with the crucible material, resulting in greater changes in the melting temperature.
- Combination not practicable.

? Compatibility unknown.

[1)] In air, aluminum forms a thin oxide layer on the surface, which improves the corrosion resistance of the metal. Similar is true for tungsten.

[2)] Solid-solid transformation

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Thermal Analysis

Chemical Behavior of Platinum, Aluminum Oxide and Graphite Crucibles

The following are critical for **PLATINUM**:

- halogens (Cl_2, F_2, Br_2), aqua regia
- $LiCO_3$, prior to emission of CO_2
- HCl with oxidizing agent (e.g. chromic acid, manganates, iron(III) salts, molten salts)
- reducing atmospheres
- Pb, Zn, Sn, Ag, Au, Hg, Li, Na, K, Sb, Bi, Ni, Fe, steel, As, Si, (alloy formation)
- P, B
- Se above 320°C (immediate cooling and removal of the sample at the end of the measurement recommended to prevent evaporation of selenium)
- metal oxides with reducing substances such as C, organic compounds or H_2
- sulfur → roughening the surface
- alkali hydroxides, alkali carbonates, alkali sulfates, alkali cyanides and alkali rhodanides at higher temperatures
- $KHSO_4$ at higher temperatures
- carbon black or free carbon above 1000°C
- SiO_2 under reducing conditions
- HBr, KCN solution at higher temperatures
- high-temperature resistant oxides above 1000°C

no resistance to:

- $NaPO_3$ in air
- mixtures of KNO_3 and NaOH at 700°C under exclusion of air
- mixtures of KOH and K_2S at 700°C under exclusion of air
- KPO_3 in air atmosphere
- LiCl at 600°C
- Na_2O_2 at 500°C under exclusion of air
- $MgCl_2$, $Ba(NO_3)_2$ at 700°C
- HBr, HJ, H_2O_2 (30%) and HNO_3 at 100°C
- KCl (the decomposition products which form during melting are damaging, melting point: 768°C)

limited resistance to:

- KHF_2, LiF, NaCl at 900°C
- mixtures of NaOH and $NaNO_3$ at 700°C under exclusion of air

The following are critical for **ALUMINUM OXIDE**:

- N_2 in the presence of carbon: formation of AlN, therefore it is dangerous to measure carbon black at higher temperatures with Al_2O_3 crucibles in N_2 atmosphere
- F_2: formation of AlF_3 and O_2
- Cl_2: formation of $AlCl_3$ above 700°C
- sulfur: no reaction with liquid sulfur, in the presence of carbon in the gas phase, formation of sulfides at higher temperatures
- H_2S: formation of up to 3% Al_2S_3 when heated
- C: formation of carbides and Al when heated
- HF: quantitative reaction to AlF_3 and H_2O at higher temperatures
- metal flourides: attack by the melt, formation of $[AlF_6]^{3}$ anions and salts similar to cryolites
- glass: glass melts dissolve Al_2O_3
- hydrogen sulfates of alkali metals and alkaline-earth metals
- HCl: no reaction to 600°C, at higher temperatures increased reaction in the presence of carbon
- B_2O_3 or Borax: melt dissolves Al_2O_3, formation of aluminum borates and aluminum borides
- alkaline and alkaline-earth oxides and their salts with volatile anions: melts form aluminates or double oxides, important, for example, for hydroxides, nitrides, nitrates, carbonates, peroxides, etc.
- CaC_2: formation of Al_4C_3 when heated
- PbO: reaction from 700°C on, also important for higher lead oxides and lead salts with anions of volatile acids
- UO_3: reaction begins at 450°C, analogous to PbO
- $Me^{II}O$: Me = Fe^{2+}, Co^{2+}, Ni^{2+}, etc., formation of spinels
- Alkaline and alkaline-earth ferrites: melt dissolves Al_2O_3
- LiF
- zirconium alloys with melting range between 800°C and 1200°C: slow, weak reaction
- some metal alloys, e.g. Fe with 4% Al

The following are critical for **GRAPHITE**:

- O_2, reaction above 400°C
- molten metals, such as Co, Ni, Na
- N_2, reaction at 1700°C (formation of small amounts of cyanide)
- oxides (probable reduction upon direct contact)
- water vapor
- F_2, Br_2 at room temperature
- sulfur
- Si at approx. 1400°C (formation of SiC)
- chromic acid (aqueous)
- chlorosulfonic acid $Cl\text{-}SO_3H$
- SiO_2 → formation of SiC via intermediate product SiO (technical production of SiC above 1800°C; i.e. the reaction between SiO_2 and C definitely begins at lower temperatures)
- nitrous gases (NO, NO_2)
- concentrated sulfuric acid H_2SO_4 at approx. 150°C, highest concentration H_2SO_4 at room temperature
- diluted nitric acid HNO_3 at approx. 90°C, highest concentration HNO_3 at room temperature
- SO_3, from approx. 100°C
- danger of explosion with perchloric acid $HClO_4$
- NaOCl, from approx. 50°C

No claim is made that this overview is exhaustive.
The temperatures given were not derived from thermal analysis measurements. Therefore, the temperatures can shift to lower values under experimental conditions. In any case, preliminary tests in a separate furnace are advisable.