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# Chemical Behavior of Platinum, Aluminum Oxide and Graphite Crucibles

The following are critical for **PLATINUM**:

- halogens ($Cl_2$, $F_2$, $Br_2$), aqua regia
- $Li_2CO_3$, prior to emission of $CO_2$
- SiC from approx. 1000°C
- PbO, $FeCl_3$
- Be alloys (begin to evaporate just above the melting point)
- HCl with oxidation agents (e.g. chromic acid, manganates, iron(III) salts, molten salts)
- reducing atmospheres
- Pb, Zn, metalls such as Pb, Zn, Sn, Ag, Au, Hg, Li, Na, K, Sb, Bi, Ni, Fe, steel, As, Si
- 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 $H_2$
- oxide in an inert gas atmosphere at higher temperatures (reduction)
- 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
- SiC and $Si_3N_4$ at temperatures above 100°C (release of elementary Si)
- HBr, KCN solution at higher temperatures
- high-temperature resistant oxides above 1000°C

**no resistance to:**

- 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
- 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 in a nitrogen 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 from approx. 1400°C
- HF: quantitative reaction to $AlF_3$ and $H_2O$ at higher temperatures
- $CuSO_4$: diffusion through the bottom of the crucible from approx. 1000°C
- compounds containing silicium, e.g. $MoSi_2$: contamination of $Al_2O_3$ from approx. 1200°C in inert gas; reactions at the contact points in air atmosphere
- metal fluorides: attack by the melt, formation of $[AlF_6]^3$ anions and salts similar to cryolites
- $SiO_2$ glass: glass melts dissolve $Al_2O_3$
- hydrogen sulfates of alkaline metals and alkaline-earth metals
- HCl: no reaction to 600°C, increased reaction in the presence of carbon at higher temperatures
- $B_2O_3$ 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, 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 = $Fe^{2+}$, $Cu^{2+}$, $Ni^{2+}$, etc., formation of spinels; CaO above approx. 1200°C
- alkaline and alkaline-earth ferrites: melt dissolves $Al_2O_3$
  -zirconium alloys with melting range between 800°C and 1200°C: slow, weak reaction
- titanium alloys (very high oxygen affinity)
- reaction with iron-nickel and magnesium alloys possible at higher temperatures

The following are critical for **GRAPHITE**:

- $O_2$, reaction above 400°C
- molten metals, such as Fe, Co, Ni, Na
- $N_2$, reaction from 1700°C (formation of small amounts of cyanides)
- 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 $ClSO_3H$
- $SiO_2$ →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, $NO_2$)
- sulfuric acid, $H_2SO_4$, concentrated at approx. 150°C, fuming $H_2SO_4$ at room temperature
- nitric acid, $HNO_3$, diluted at approx. 90°C, fuming $HNO_3$ 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 are primarily literature values. Therefore, the temperatures can shift to lower values under experimental conditions. In any case, it is advisable to run tests in separate furnaces.

# 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 (H. K. Cammenga et al., Thermochimica Acta, 219, 1993) and from our own experience, it is not possible to cover all possible combina-tions and measurement conditions. In case of doubt, it is advisable to run preliminary tests in a separate furnace.
For platinum crucibles with $Al_2O_3$ inliners, basically the same data like for corundum crucibles applies. The only exception is nickel as a calibration substance. The melting point of nickel with 1455°C is above the recommended maximum working temperature for Pt crucibles on a type S sample carrier.

| | Calibration Substance | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Crucible Material** | Cyclopentane | Water | Gallium | Indium | Tin | Lead | Zinc | Lithium Sulfate[2] | Aluminum | Silver[3] | Gold[3] | 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.

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- Crucible melts.
- Melt or transformation product reacts with the crucible material, resulting in greater changes n 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.
[3] Gold and Silvery stick in $Al_2O_3$ crucibles, but can normally be removed mechanically.

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