

# Solar thermogravimetry experiments at PSI HFSS facilities

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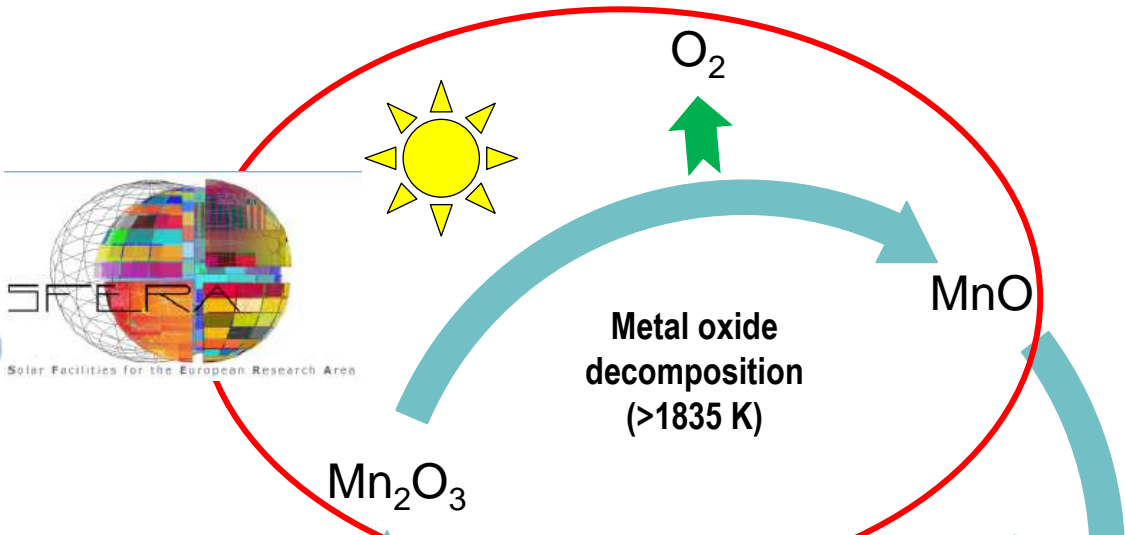
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[www.energy.imdea.org](http://www.energy.imdea.org)

- Context
- Solar thermogravimetry – Experimental set-up
- Analysis of Manganese oxides reduction
- Conclusions



## Manganese oxide based-on thermochemical cycle

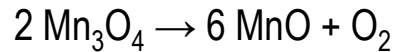


More details on 2<sup>nd</sup> and 3<sup>rd</sup> step in session THU-2-D: Solar Fuels and Chemical Commodities next Thursday.

*Factors Influencing Hydrogen Production and Sodium Recovery in the NaOH-MnO Thermochemical Cycle, A. Bayón, IMDEA Energy*

Sturzenegger, M., Ganz, J., Nüesch, P., Schelling, T., 1998. Solar hydrogen from manganese oxide- A new thermochemical cycle. Hydrogen energy progress XII 1-3,801-805.

- First-step manganese thermochemical cycle based for hydrogen production:



- First proceeds at the temperature range between 800 °C and 1060 °C in nitrogen, air or oxygen (Tinsley and Sharp, 1971).
- Solar reduction tests have been previously carried out by using concentrated radiation in a directly irradiated reactor exposed to a solar furnace at above 1627 °C (Frey et al., 2001).
- Thermogravimetry and a non-solar aerosol flow reactor have been used (Francis, 2008; Francis et al. 2008). The Avrami-Erofeev mechanism was the best at describing the kinetic model of the chemical reaction (fractional extent of the reaction,  $\alpha$ , between 0.15 and 0.85).



Mn<sub>2</sub>O<sub>3</sub>



Mn<sub>3</sub>O<sub>4</sub>



MnO



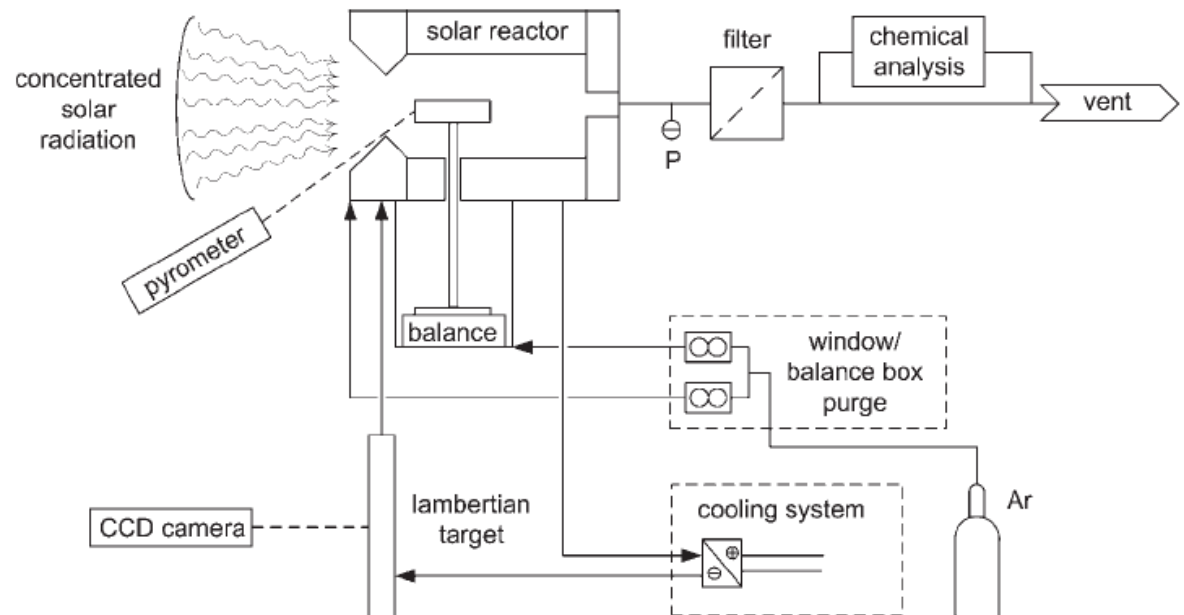
# Characterization Of Solid-Gas Chemical Reactions by Solar TG Context



A deep knowledge of any solar thermochemical process requires a detailed analysis of chemical reactions involved at operating conditions close to those found under concentrating solar radiation.

Thermal desorption in furnaces and thermogravimetric balances are commonly used for that.

Solar thermogravimetry (in solar furnace) has been applied for analyzing solid-to-gas chemical reactions



Schunk, L., Steinfeld, A., 2009. Kinetics of the thermal dissociation of ZnO exposed to concentrated solar irradiation using a solar-driven thermogravimeter in the 1800 - 2100 K range. *AIChE Journal*, vol. 55, no. 6, pp. 1497–1504.



# Experimental set-up



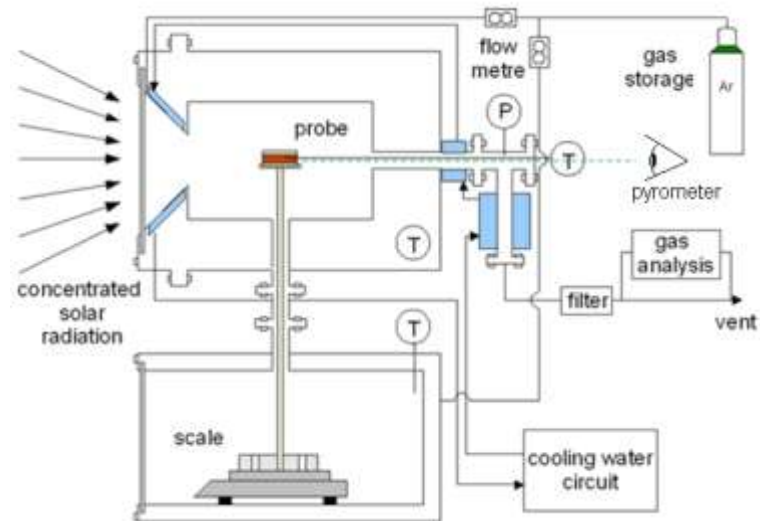
<http://solar.web.psi.ch/data/facilities/?pss>

## New adaptation for using in the HF solar simulator:

- Temperature probe measurement by pyrometer on the rear side after effective emissivity determination.
- Simultaneous gas analysis and mass measurement.
- Solar simulator focal plane placed on reactor aperture.

## High Flux Solar Simulator (HFSS), PSI, Switzerland.

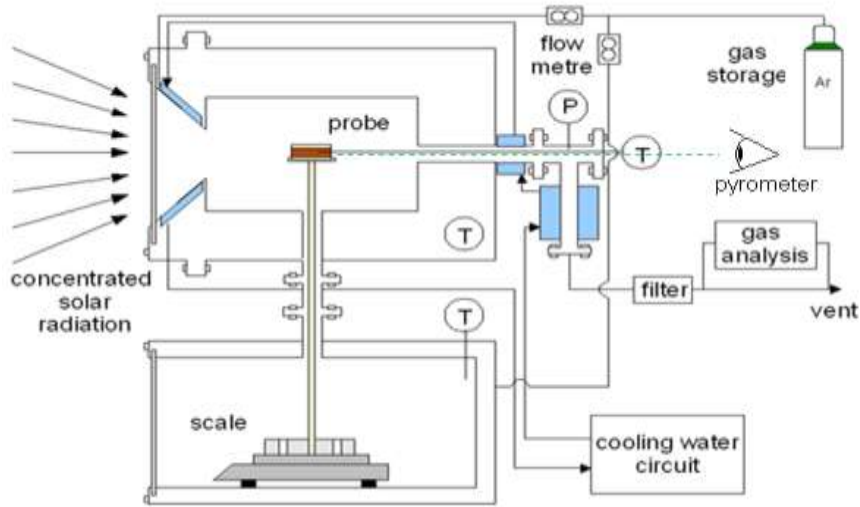
- 10 high-pressure Xe arcs, each close-coupled to elliptical specular reflectors,
- Capable of delivering 50 kW of continuous radiative power, mostly in the visible and IR spectra
- Peak flux intensities up to 11,000 suns.
- Temperatures exceeding 2000 °C at heating rates exceeding 1000 °C/s are achievable.







# Experimental set up



Reactor cavity

Reactor cavity

Scale cavity



Reactor gas outlet

Pyrometer



# Mn<sub>2</sub>O<sub>3</sub> reduction



- Sample holder: Al<sub>2</sub>O<sub>3</sub> 99 % purity; inner diameter, 17 mm; outer diameter, 25 mm; length, 45 mm
- Mn<sub>2</sub>O<sub>3</sub> powder 99 % purity, -325 mesh, Sigma-Aldrich.
- Samples were prepared by mixing the oxide powder with water in order to obtain a thick paste that was introduced inside the alumina tube and dried at 130 °C for 1 hour.



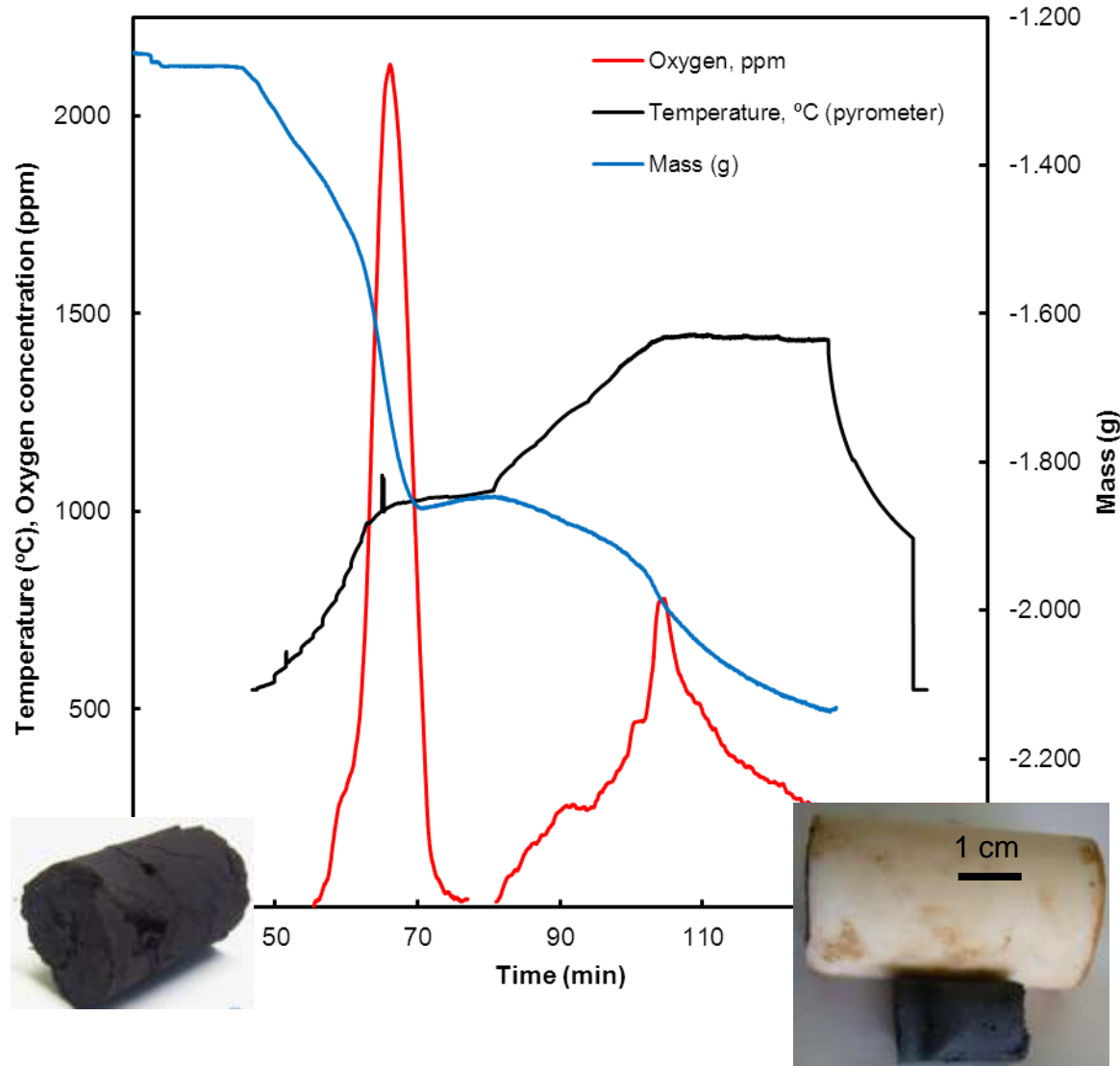
- Complete conversion after treatment (confirmed by XRD).
- Melting on sample front partial melting due to strong radiation transfer.





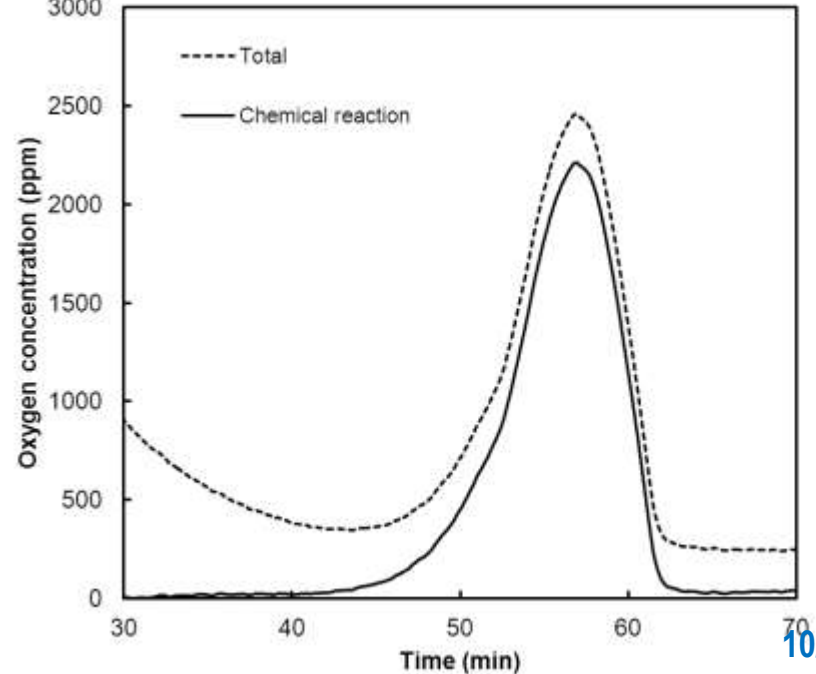
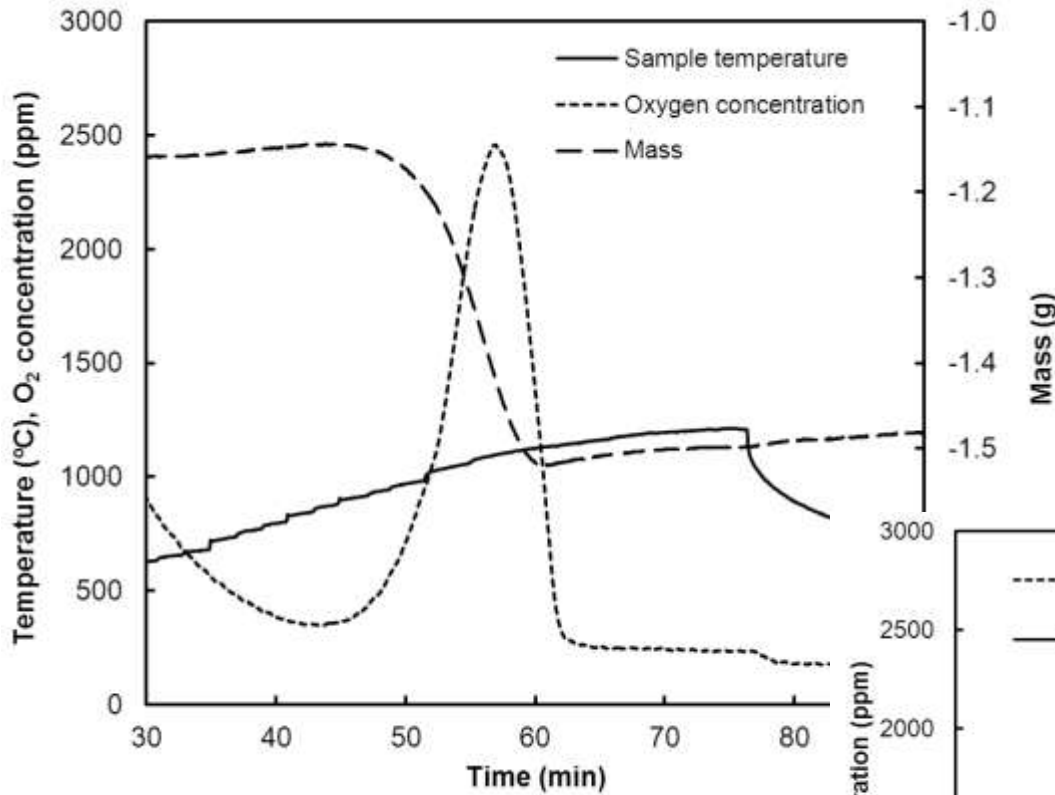


# Complete solar thermogravimetry





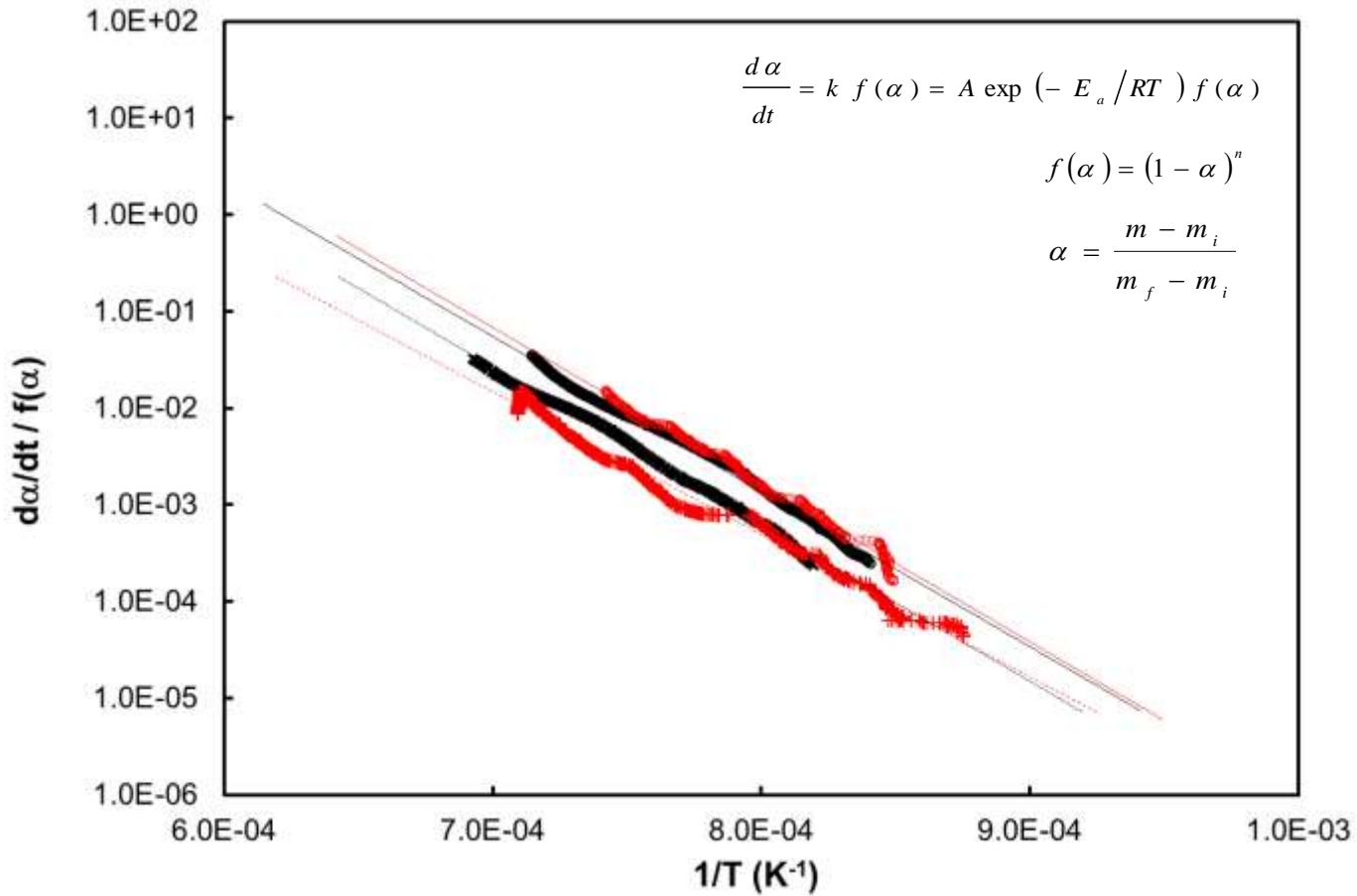
# First reduction





# Mn<sub>2</sub>O<sub>3</sub> reduction

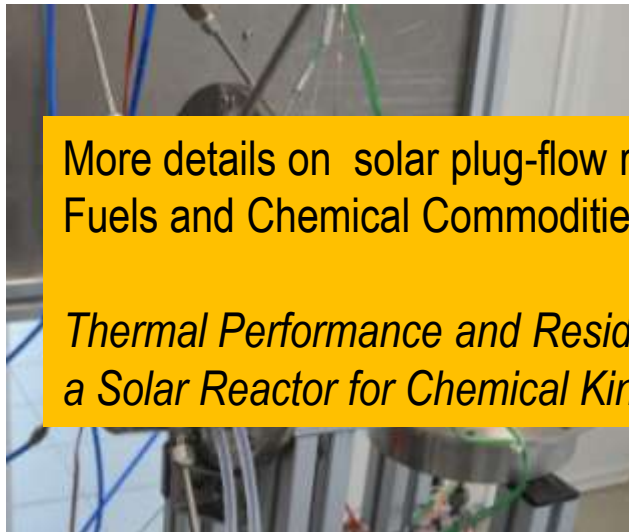
Best fitting for an nth order kinetic rate with  $n = 0.93$  for a conversion range between 1 and 99%.



Pre-exponential factor compatible with a temperature uncertainty of 50 °C.

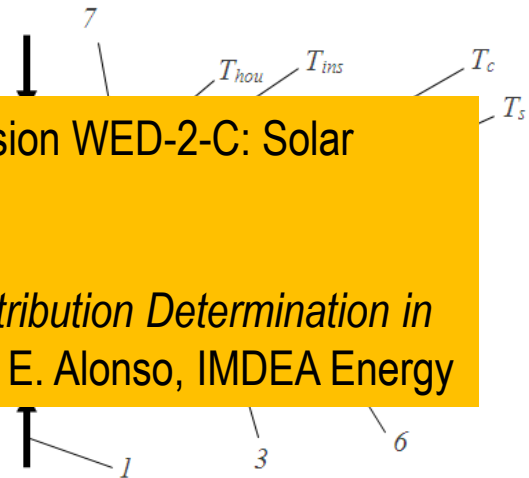
# Conclusions

- Kinetics of reduction chemical reaction of manganese oxides been successfully determined.
- New improvements on sample holders design are necessary for solar thermogravimetry.
- Gas analysis is enough for characterizing chemical reactions if plug-flow is assured.



More details on solar plug-flow reactors in session WED-2-C: Solar Fuels and Chemical Commodities tomorrow.

*Thermal Performance and Residence Time Distribution Determination in a Solar Reactor for Chemical Kinetics Analysis, E. Alonso, IMDEA Energy*



# Characterization Of Solid-Gas Chemical Reactions by Solar TG

## Acknowledgments

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