RECENT REALIZATIONS OF THE SF_6 AND CO_2 TRIPLE POINTS AT THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

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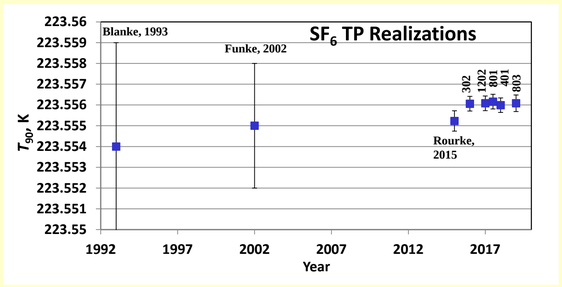
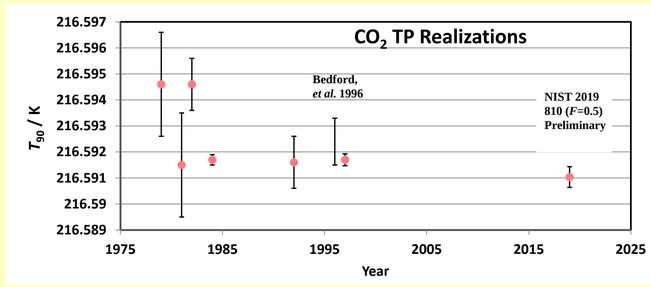
Abstract / Introduction

International restrictions on the trade of mercury-based products have created a near-term need to develop alternative materials for temperature standards. The triple points of SF_6 and CO_2 are good candidates for replacing the Hg triple point (TP) as a defined fixed point on the International Temperature Scale of 1990 (ITS-90). We have constructed a series of immersion cells for realizations of both the SF_6 TP and CO_2 TP that are suitable as 'drop-in-replacements' for Hg TP cells as currently used in conventional refrigerated baths. These immersion cells are pressurized, with no external manifold, and refillable via a conventional service valve. Realizations are performed in a quasi-adiabatic mode inside of a small vacuum chamber that is immersed in an ethanol bath. We report recent measurements using capsule-type standard platinum resistance thermometers (SPRTs) that have been calibrated on the subrange 4 of the ITS-90. Melting plateaus are presented versus both melted fraction F and inverse fraction F^{-1} scales. We compare the interpolated T_{90} temperatures as extrapolated to F=1 from these realizations with those reported in our prior work and from the published literature.



Recent History of SF_6 TP realizations.



History of CO_2 TP realizations since 1979.

Triple Point Cell Design and Construction

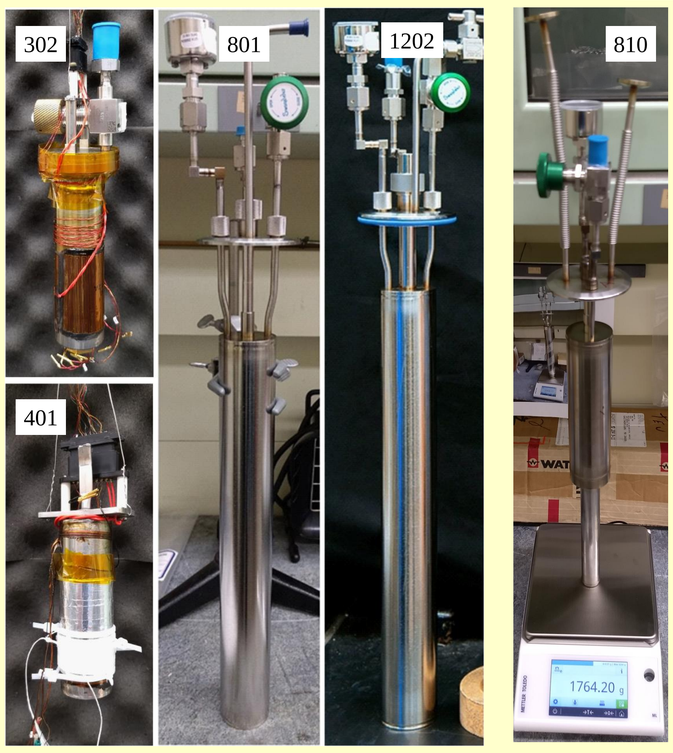
	T_{tp}	p_{tp}	ρ_l	ρ_s	Δh_f	dp_m/dT	dT/dz	A^{-1}
	(K)	(MPa)	(kg/m^3)	(kg/m^3)	(J/cm^3)	(MPa/K)	mK/m	uK/ppm
Hg	234.3156	1.7×10^{-10}	13,690	14,184	156	18.5	8.6	199
SF_6	223.556	0.231	1845	2281	66.7	1.56	11.6	79
CO_2	216.59	0.55536	1178.5	1532	232	4.69	2.47	45

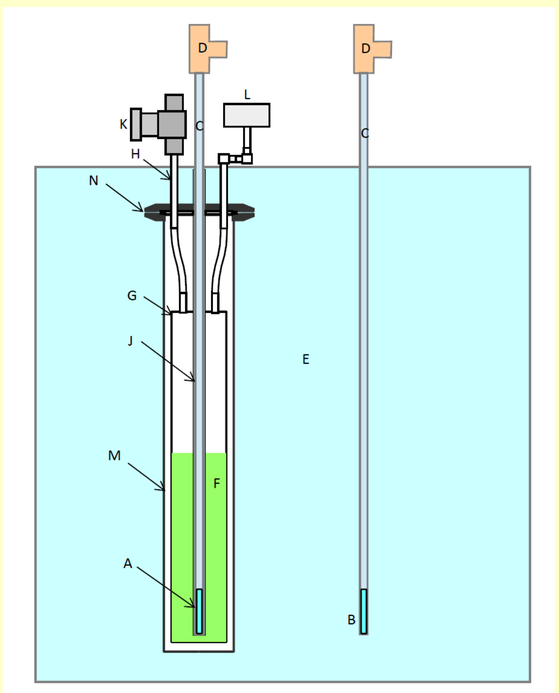
Five SF_6 TP cells and one CO_2 TP cell are in this study. Four cells (SF_6 serial numbers 1202, 801, 803 and CO_2 cell 810) are larger immersion types and two others (SF_6 Cell serial numbers 302 and 401) are smaller 'adiabatic' type cells. The cell design parameters are summarized in the below Table. The cells are shown in the photographs below.

Cell	Volume V_{cell}	SF_6/CO_2 mass, m_{cell}	Density ρ_{cell}	CDR ρ_{cell}/ρ_{cr}	Effective Immersion	Enthapy ΔH_f
s/n	cm^3	g	g·cm^{-3}		cm	kJ
302	42	12.9	0.307	0.41	N/A	0.47
401	14	9.1	0.650	0.87	N/A	0.33
801	580	590	1.017	1.37	16	21.3
1202	550	467	0.849	1.14	13	17.0
803	537	573.3	1.068	1.44	18.3	20.7
810	352	68.55	0.195	0.42	16.8	13.5

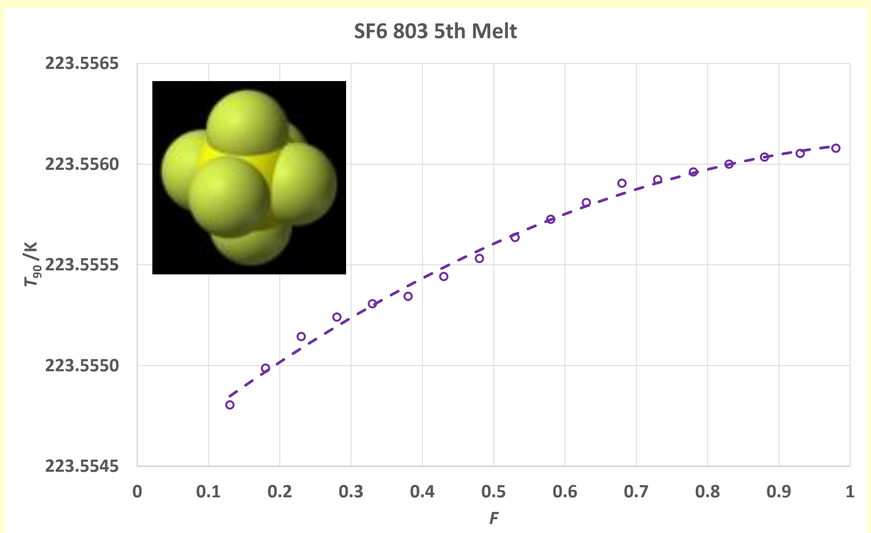
Cell 803 is similar to cell 801, but with the addition of an internal set of 30 copper baffles (shown below) to equalize radial gradients and support the solid SF6 mantle.

Below: Experimental setup for immersion-type cells : capsule-type SPRTs to measure the cell (A) and bath (B) temperatures; (C) borosilicate glass adapter tubes; (D) nylon gas-tight fittings; (E) 50 l ethanol bath volume; (F) condensed (shown in green) portion of SF6 (G) pressure wall of cell volume; (H) SF6 fill line; (J) central thermowell; (K) bellows valve; (L) pressure gauge; (M) vacuum chamber; and (N) ISO-63 vacuum flange with aluminum gasket.





Results using SF_6



SF_6 Cell 803 , 2019
- Direct Heating Only
- Pulse Melting
- 5 % ΔF / heat pulse
- 2 pulses / day
- 6 hrs. ON, 6 hrs. OFF
- Stable over all F values

SF_6 Source Cylinder
Concorde Specialty Gas†
Report of Analysis:
- CO_2 - ND*
- SO_2 - ND*
- $N_2 + O_2$* = 3.3 ppm
- H_2O < 1 ppm*
- CF_4 = 0.3 ppm

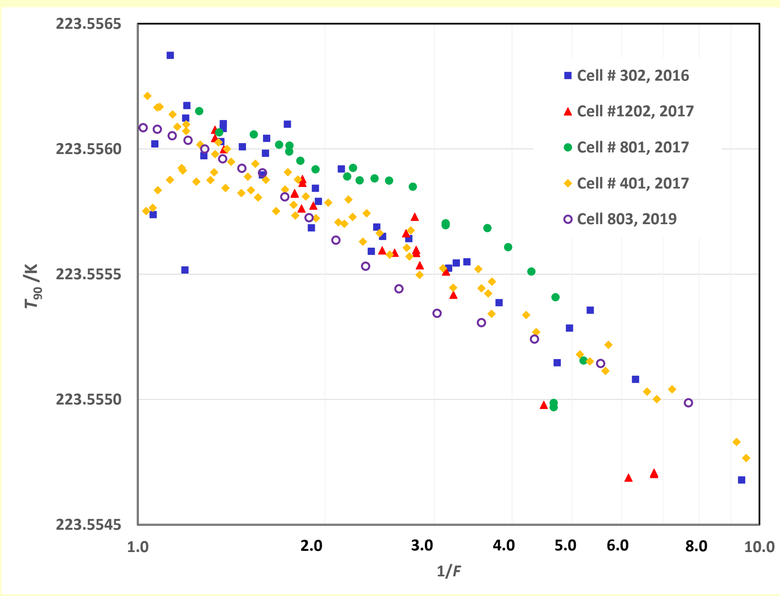
*Removed by in-line Getter

Complied SF_6 TP Realizations
Cells 302/401: Adiabatic 2016-17
- Pulsed Direct Heating
- Kapton Thermofoil Heaters
- 500 mW , 1000 mW

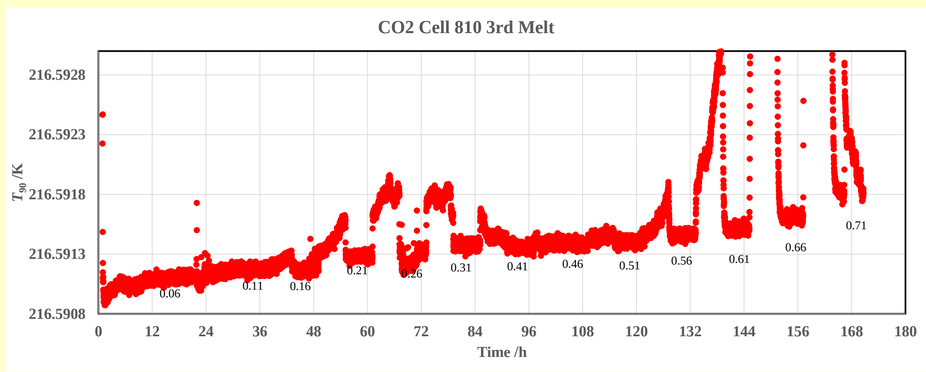
Cells 801/1202: 2016-17
- Pulsed Indirect Heating
- Gas-coupled to Bath Heat
- <≈ 10 mW

Cell 803: 2019
- Pulsed Direct Heating
- Kapton Thermofoil Heaters
- 48 mW

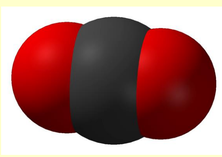
† Certain commercial equipment, instruments, or materials are identified in this poster in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

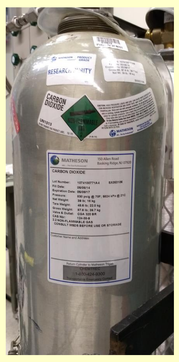


Results using CO_2



CO_2 Cell 810 , 2019
Direct Heating
Pulse Melting
5 % ΔF / heat pulse
Stable Plateau F < 0.56

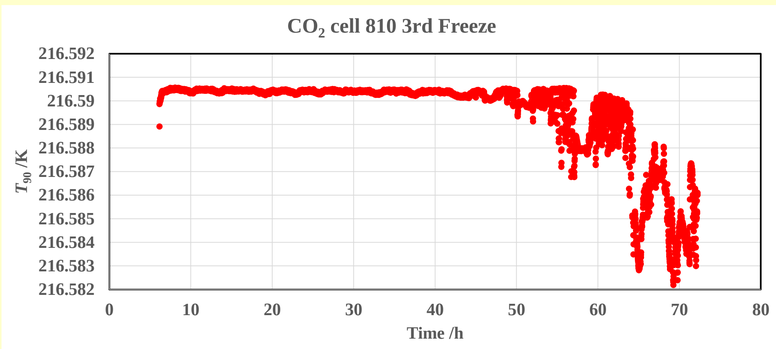


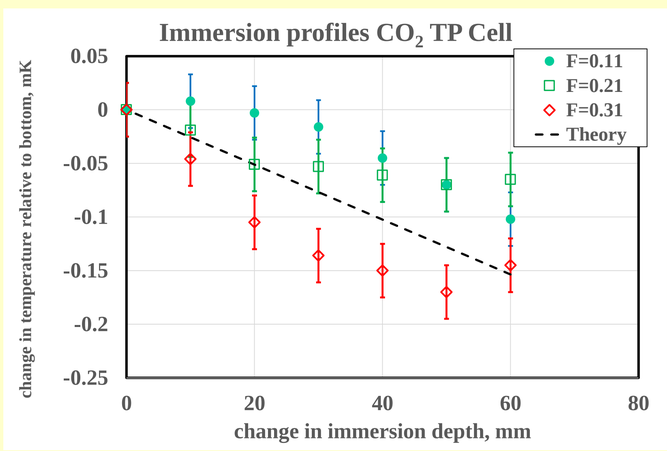


CO_2 Source Cylinder
NIST Gas Metrology Group
Measured:
- Ar: 0.30 ± 0.03 ppm
- N_2 : 1.05 ± 0.03 ppm

Specification:
- CO < 0.2 ppm *
- H_2 < 1 ppm*
- N_2 < 5 ppm
- O_2 < 2 ppm*
- H_2O < 2 ppm*
- THC (as CH_4)* < 4 ppm

*Removed by in-line Getter





Immersion Profiles, CO_2 Cell 810 , 2019
- Some distortions observed
- 10 mm steps downward
- Predicted from Clapeyron Equation :
  - 0.0247 mK/cm
- assumes melting line: 4.69 MPa/K

$$\left.\frac{dT}{dz}\right|_{T=T_{tp}} = \frac{\rho g}{\left.\frac{dp}{dT}\right|_{T=T_{tp}}}$$

Summary

- Results using SF_6 :
  - average of four cells (#302, 1202, 801, 401) 2017 [1]
    - 223.55587(33) K , F=0.5 ; 223.55607(33) K , F=1
  - cell #803 (immersion type), 2019, *preliminary*
    - 223.5556(5) K , F=0.5 ; 223.55608(50) K , F=1
  - Pressure head correction: 0.116(16) mK/cm [1] ; Clapeyron Eqn. : 0.116 mK/cm
- Results using CO_2 , Cell 810, 2019 *preliminary*:
  - 216.5910(5) K , F=0.5
- References
  - [1] W. Tew and K. Quelhas, *J Res NIST*, Vol 123, Art. No. 12013 (2018) https://doi.org/10.6028/jres.123.013